# Idaho National Laboratory Directed Research and Development FY-2010

March 2011



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March 2011

Idaho National Laboratory Idaho Falls, Idaho 83415

http://www.inl.gov

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It is my pleasure to present the Idaho National Laboratory's (INL's) fiscal year (FY) 2010 Laboratory-Directed Research and Development (LDRD) Annual Report. This report demonstrates the types of cutting edge research the INL is performing to help ensure the nation's energy security. The research conducted under this program is aligned with our strategic direction, benefits the Department of Energy (DOE) and is in compliance with DOE order 413.2B. This report summarizes the diverse research and development portfolio with emphasis on the DOE Office of Nuclear Energy (DOE-NE) mission, encompassing both advanced nuclear science and technology and underlying technologies.

The INL's primary mission supports the four objectives contained in the DOE-NE Nuclear Energy Research and Development Roadmap. The INL's LDRD program emphasizes development of technical capability to support future DOE-NE research and development needs. The focus of these capabilities includes; advanced nuclear fuels and materials, separations and waste forms, modeling and simulation, and instrumentation and controls.

The INL's LDRD program also serves the nation through research and development that helps ensure homeland security with emphasis

on instrumentation for nuclear non-proliferation applications, cyber security systems, and telecommunications systems. Additionally, our science base is extended through research that advances hybrid energy systems and develops environmentally sound approaches and products for improved management of the hydrocarbon fuel cycle.

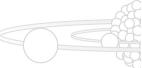
The LDRD research is instrumental in developing the scientific and engineering resource capabilities and collaborations needed to accomplish future DOE missions. It is an essential competency strengthening activity, attracting and maintaining world-class researchers, and cultivating collaborative networks with universities, industry and other laboratories required to fully support DOE's and other national priorities.

I am proud of the accomplishments and opportunities that the INL's LDRD projects provide to the nation, and encourage you to take the time to review these project narratives and reflect on these contributions.

John J. Grossenbacher Laboratory Director







ii

## Acknowledgments

This annual report provides an overview of the Laboratory Directed Research and Development (LDRD) Program at the Idaho National Laboratory (INL). Summarized in this report are 108 technical research projects conducted in fiscal year 2010. The LDRD Program at the INL is managed for the laboratory by Mr. John J. Grossenbacher, Laboratory Director, and Mr. John S. Martinell, LDRD Program Manager.

Many people contributed to this report; however, particular thanks go to the principal investigators who conduct the cutting-edge research so important to this laboratory.

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iv

# **Table of Contents**

| Acknowledgments  | ii |
|--|----|
| Table of Contents  |    |
| Overview and Highlights  |    |
| Highlights   |    |
| INL Collaborative Researchers Advance Foundation for Next-Generation Safety Analysis Codes                           |    |
| INL Advances Understanding of Control System Data Fusion   | 9  |
| INL Researchers Develop International Safeguards for Commercial Pyroprocessing Facilities                            | 1  |
| Project Supports Multi-Scale, Multi-Physics Simulations  | 13 |
| Research Advances Next Generation Hybrid Energy Systems  | 15 |
| Project Advances Water Purification System Research  | 17 |
| Nuclear Energy Science and Technology  |    |
| Reactor Physics Sensitivity Analysis, Uncertainty Quantification, and Data Assimilation Capability                   | 21 |
| Next-Generation Code for Nuclear System Safety Simulation  | 24 |
| Multiscale Modeling and Simulation of Nuclear Fuel Performance   | 25 |
| Nuclear Reactor Safety Case  | 29 |
| Process Modeling of Solvent Extraction Separations for Advanced Nuclear Fuel Cycles                                  | 3  |
| Reactivity of Radiolytically Produced Nitrogen Oxide Radicals toward Aromatic Compounds                              | 33 |
| Consolidated Electrochemical Technique for Separation and Recovery of Actinides from Fission Products in Oxide Fuels | 32 |
| Neptunium Redox Chemistry in Irradiated Aqueous Nitric Acid  | 35 |
| Reversible Gas Phase Reactions for Recovery of Graphite from Recycled HTGR TRISO Fuel                                | 36 |
| Comprehensive Thermodynamic Models for Aqueous Partitioning of Actinides from Used Nuclear Fuel                      | 37 |
| Advanced Burnup Measurement and Nuclear Forensics using ICP-MS Isotopics Analysis Techniques                         | 39 |
| Advanced Ceramic Nuclear Fuels   | 4  |
| Spatially Correlated Microstructural Characterization: From Centimeters to Nanometers                                | 44 |
| Viability Evaluations of Linear Variable Differential Transformers (LVDTs) for In-Pile Instrumentation               | 45 |
| Instrumentation for In-pile Detection of Thermal Conductivity  | 4  |
| Human Performance Assessment for Technology Neutral Evaluation   | 49 |
| Ultrasonic Transducer Sensors for In-Pile Detection of Dimensional Changes   | 51 |
| Smart Grid Impact on Commercial Nuclear Plants   | 53 |



| Characterization of Fluidized Beds via Pressure-Fluctuation Analysis   | 54  |
|--|-----|
| Extraction of Fission Products in Room Temperature Ionic Liquids and sc-CO <sub>2</sub> with Study of Radiolysis | 55  |
| Integrated Mesoscale Approach for the Simulation of Nuclear-Fuel Behavior  | 56  |
| Aleatory vs. Epistemic Uncertainty in Seismic Hazard Analyses  | 58  |
| In-Pile Temperature Monitor and Control for ATR  | 59  |
| Multi-Rate Shock Physics Simulation of Blast and Penetration Events in Concrete                                  | 60  |
| National & Homeland Security   |     |
| Methodologies for the Design, Analysis, and Validation for Operation of Complex Resilient Networks               | 65  |
| Computational Materials Characterization   | 68  |
| Next Generation Control System "Smart Grid" Simulation Environment   | 70  |
| Cognitive Intelligent Wireless Communication Devices   | 72  |
| Cyber Security for Protection of Critical Infrastructure: Expanding the Scientific and Engineering Bases         | 75  |
| Object Reconstruction Techniques for Use in Radiography  | 76  |
| Cognitive Network Engine and Simulation Framework  | 77  |
| Application of Dynamic Bayesian Networks to Systems with Ambient Intelligence                                    | 79  |
| Armor Research and Development in Support of the U.S. Army   | 83  |
| Advanced Multi-Layer Laminate Blast Mitigation System  | 85  |
| Enhanced Metal Ion Analysis  | 86  |
| Environmental and Energy Sciences  |     |
| Investigation of Low-Temperature Performance in Membrane Materials and Processes for Gas Separations             | 89  |
| High-Performance Polymer Membranes for High-Temperature Gas Separations  | 90  |
| Reducing Coalbed Methane (CBM) Water Discharge Volume  | 94  |
| Triazine-Based CO <sub>2</sub> Capture Agents  | 95  |
| Near-Field Impacts of In-Situ Oil-Shale Development on Water Quality   | 96  |
| Dynamic Impact Model and Information System to Support Unconventional Fuels Development                          | 99  |
| Advanced Remote Sensing for Energy and Environmental Applications using Unmanned Aerial Vehicles                 | 101 |
| Addressing the Spectrum of Nuclear-Related NDE Needs   | 103 |
| Water and Energy System Interdependency Modeling for Multicriteria Decision Analysis                             | 105 |
| Degradation and Conversion of Lignin using Extremophilic Systems   | 106 |



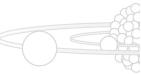
| Metabolic Engineering of <i>Alicyclobacillus acidocaldarius</i> for Lacit Acid Production from Biomass-derived Monosaccharides | 107 |
|--|-----|
| Network Interaction in the Thermoacidophile Alicyclobacillus acidocaldarius  | 108 |
| Specific Biological Responses to Nano Metal Oxides   | 109 |
| Development of Non-Lethal Methods for Enhanced Lipid Recovery from Microalgae  | 111 |
| Development of Thermally Generated In Situ Precipitation Barriers due to Subsurface Heat Injection                             | 112 |
| Hybrid Osmosis Water Purification Systems Research   | 113 |
| Biological Fixation of CO <sub>2</sub> for Fuel and Chemical Production Using Acetogen Intermediary Metabolism                 | 114 |
| Advanced Adaptive Algorithms in Phased-Array Ultrasonics for Materials Inspection  | 115 |
| Conducting Perovskite Materials for Catalytic Applications   | 116 |
| Acoustic Nanostructures  | 118 |
| A New Paradigm for Robust Catalysts  | 120 |
| Particle-discrete Element Model Simulation   | 121 |
| Scaling of Welding Processes   | 122 |
| Effect of Glycosylation on the Activity and Stability of Bacterial Enzymes   | 125 |
| Energy Security Initiative   |     |
| Generation and Expulsion of Hydrocarbons from Oil Shale  | 129 |
| Nuclear Pathways to Energy Security  | 131 |
| Altering Wettability by Chemical Amendments to Improve Gas Production from Tight Sands   | 134 |
| Adaptive Process Modeling of Hybrid Energy Systems   | 135 |
| Feedstock Processing & Energy Storage  | 136 |
| Mesocilicate Supported CO <sub>2</sub> Capture Material  | 137 |
| Membrane Separation System Research for the HYTEST Facility  | 138 |
| Electrochemical Reduction of CO <sub>2</sub> to CO and Hydrocarbon Fuels   | 139 |
| Hybrid Systems Process Integration and Dynamics Studies  | 140 |
| Conversion of Light Hydrocarbons to Fuels and Chemicals  | 141 |
| Nuclear Nonproliferation Initiative  |     |
| Active Interrogation Die-Away Assay Development Program  | 145 |
| Xe-135 Recovery from the Spontaneous Fission of Cf-252   | 148 |
| Developing a Next-Generation, Risk-Informed Approach for Robust and Resilient Design Development                               | 149 |
| Neutron Spectrometer Development   | 150 |



Idaho National Laboratory

vii

| Nuclear Material Detection Using Neutron Time-of-Flight   | 153 |
|---|-----|
| On-line Monitoring of Actinide Concentrations for Advanced Aqueous Separation Processes                   | 156 |
| Development of a Safeguards Approach for Pyroprocessing   | 157 |
| Radionuclide Collection-Detection Device for Remote Monitoring of Tc-99 as a Proliferation Indicator      | 158 |
| Institute for Nuclear Energy Science & Technology   |     |
| Design and Testing of a Mars Hopper   | 161 |
| In-situ Characterization of an Oxide Film for Prediction of Stress Corrosion Cracking Susceptibility      | 162 |
| Center for Advanced Energy Studies  |     |
| Investigation of Public Discourse Methods in Energy Policy Decision-making                                | 165 |
| Development of Lignocellulosic Ethanol Production Potential in Idaho                                      | 169 |
| Optimization of Ceramic Waste Forms Used for Electrochemical Processing of Spent Nuclear Fuel             | 172 |
| Prediction and Monitoring of CO <sub>2</sub> Behavior in Deep Reactive Geologic Formations                | 173 |
| Fabrication of Advanced ODS Alloys using Field Assisted Sintering   | 174 |
| Small Specimen Test Techniques for Evaluating Radiation-Induced Changes in Mechanical Properties          | 175 |
| Design and Operation of Two-Stage Manure Anaerobic Digestion Process                                      | 176 |
| Transmission Site Suitability Decision Support Technology   | 178 |
| CAES Vertical Axis Wind Turbine   | 179 |
| Center for Advanced Modeling & Simulation   |     |
| Study of Preconditioning Techniques for Krylov Solvers Applied to Hybrid Neutron Transport Calculations   | 183 |
| Fickian and Thermal Diffusion in Nuclear Materials from Linear Response Theory and Multiscale Simulations | 185 |
| Error Estimation for Stochastic Uncertainty Quantification  | 186 |
| Advanced Visualization using Immersive Environments   | 187 |
| Distinctive Signatures  |     |
| INSTRUMENTATION, CONTROL AND INTELLIGENT SYSTEMS DISTINCTIVE SIGNATURE                                    | 191 |
| Resilient Control System Network Agents   | 193 |
| Integrated Control Systems Data Fusion  | 194 |



| Wireless Sensor Testing   | 195 |
|---|-----|
| Anomaly Detection, Diagnosis, and Resilient Control in Complex Engineered Systems                                 | 197 |
| 3D Spatial Representation in Support of Design Inspection and Verification  | 200 |
| Resilient Condition Assessment Monitoring (ReCAM) System  | 201 |
| Automated Differential Equation-Based Identification  | 203 |
| MATERIALS FOR HARSH ENVIRONMENTS SIGNATURE  | 205 |
| Development of a Small Sample Volume Mechanical Properties Testing Technique for Irradiated Fuels and Materials   | 207 |
| Irradiation-Induced Evolution of Defects and Microstructures in Nanocrystalline BCC Mo                            | 208 |
| Microstructural Evolution of UO <sub>2</sub> and U under Irradiation  | 210 |
| Irradiation Testing and Molecular Modeling of Irradiation-Assisted Diffusion and Microstructural Evolution (FCCI) | 211 |
| SEPARATIONS & ACTINIDE SCIENCE SIGNATURE  | 213 |
| Evaluation of Covalent Interactions in Actinide Coordination Compounds  | 215 |
| Rapid Detection of Plutonium, Neptunium and Technetium in Water Samples   | 216 |
| Measuring Actinide Speciation in High pH Solutions  | 217 |
| Isotope Ratio Measurements Methods for Direct Analysis of Samples   | 221 |
| Investigation of Molten Bromide Salt Systems for Separation and Recovery of Actinides from Fission Products       | 222 |
| Appendices  |     |
| Appendix A — Authors  | 225 |
| Appendix B — Refereed Publications.   | 229 |
| Appendix C — Relevance to Major National Programs   | 239 |







# Overview







## Overview and Highlights

The FY 2010 Laboratory Directed Research and Development (LDRD) Annual Report is a compendium of the diverse research performed to develop and ensure the INL's technical capabilities can support the future DOE missions and national research priorities. LDRD is essential to the INL—it provides a means for the laboratory to pursue novel scientific and engineering research in areas that are deemed too basic or risky for programmatic investments. This research enhances technical capabilities at the laboratory, providing scientific and engineering staff with opportunities for skill building and partnership development.

Established by Congress in 1991, LDRD proves its benefit each year through new programs, intellectual property, patents, copyrights, publications, national and international awards, and new hires from the universities and industry, which helps refresh the scientific and engineering workforce.

#### Benefits of LDRD to INL

The benefits of INL's LDRD research are many as shown in the tables below. One of three R&D 100 Awards won by INL in FY-10, "Supercritical/Solid Catalyst (SSC) Biodiesel Production Process" had its beginnings in an LDRD initially funded in 1995. Fourteen patents were issued in FY-10 that were attributable to LDRD funding and 15 of the total invention disclosures submitted in FY 2010 were attributable to LDRD research. Fifty refereed journal articles and conference proceedings were published during the fiscal year as a result of LDRD research.

| LDRD Metrics – FY-10  |    |  |
|-----------------------|----|--|
| National Awards       | 1  |  |
| Invention Disclosures | 15 |  |
| Patents               | 14 |  |
| Copyrights            | 0  |  |
| Refereed Publications | 50 |  |
| INL Post Docs         | 10 |  |
| INL Students          | 24 |  |
| New S&E Hires         | 13 |  |

| FY10 LDRD Program Statistics |          |  |
|------------------------------|----------|--|
| LDRD Program Cost            | \$29M    |  |
| Number of Projects           | 108      |  |
| Largest Project              | \$1,570K |  |
| Smallest Project             | \$12.5K  |  |

#### The LDRD Process at INL

The LDRD Program is administered in accordance with requirements set in DOE Order 413.2B, accompanying contractor requirements, and other DOE and federal requirements invoked through the INL contract. The LDRD Program is implemented in accordance with the annual INL LDRD Program Plan, which is approved by the DOE. Nuclear Energy Program Secretarial Office. This plan outlines the method the laboratory uses to develop its research portfolio, including peer and management reviews, and the use of other INL management systems to ensure quality, financial, safety, security and environmental requirements and risks are appropriately handled. The LDRD Program is assessed annually for both output and process efficiency to ensure the investment is providing expected returns on technical capability enhancement.

The call for proposals and project selection process for the INL LDRD program begins typically in April, with preliminary budget allocations, and submittal of the technical requests for preproposals. A call for preproposals is made at this time as well, and the preparation of full proposals follows in June and closes in July. The technical and management review follows this, and the portfolio is submitted for DOE-ID concurrence in early September. Project initiation is in early October.

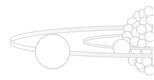
The technical review process is independent of, and in addition to the management review. These review processes are very stringent and comprehensive, ensuring technical viability and suitable technical risk are encompassed within each project that is selected for funding. Each proposal is reviewed by two or three anonymous technical peers, and the reviews are consolidated into a cohesive commentary of the overall research based on criteria published in the call for proposals. A grade is assigned to the



technical review and the review comments and grade are released back to the principal investigators and the managers interested in funding the proposals. Management criteria are published in the call for proposals, and management comments and selection results are available for principal investigator and other interested management as appropriate. The DOE Idaho Operations Office performs a final review and concurs on each project prior to project authorization, and on major scope/budget changes should they occur during the project's implementation.

#### Report Organization

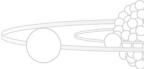
This report begins with several research highlights that exemplify the diversity of scientific and engineering research performed at the INL in FY 2010. Progress summaries for all projects are organized into sections reflecting the major areas of research focus at the INL. These sections begin with the DOE-NE Nuclear Science and Technology mission support area, followed by the National and Homeland Security and the Energy and Environmental Science and Technology areas. Major INL initiatives and distinctive Signatures areas complete the project summaries. The appendices provide an author index, as well as a list of refereed publications and information on project relevance to major national programs.



# Highlights

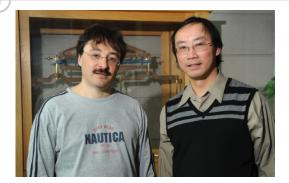






## INL Collaborative Researchers Advance Foundation for Next-Generation Safety Analysis Codes

Nam Dinh NE156



From left to right: Robert Nourgaliev and Nam Dinh.

Reactor safety is an area where Idaho National Laboratory (INL) is known throughout the world for its leadership in developing largescale experiments, knowledge base, and simulation methods that cultivated in a family of successful and widely used RELAP5 computer codes. Over the past few decades, as well as the future, the INL-enabled technology has served, and will continue to support, nuclear industry in plant design-basis safety analysis and licensing applications. However, as the current fleet of nuclear power plants ages, their continued safe and economic operation would benefit from advanced modeling and simulation capabilities that provide an improved characterization of plant safety margins.

Launched in 2009 with a mandate to develop advanced reactor safety simulation as strategic research and development (R&D) area in the Nuclear Science and Technology program, a three-vear Laboratory Directed Research and Development (LDRD) project embarked on the development and demonstration of the scientific basis and computational technology for a Next-Generation Safety Analysis Code (NGSAC), aka R7. Lead by principal investigator and INL Laboratory Fellow, Nam Dinh, this LDRD project brings together a team of multidisciplinary talents from INL and collaborating institutions. Differing from other national and international efforts aimed to advance the state of the art in reactor simulation, this INL LDRD project exercises a multi-pronged approach, concurrently advancing a novel analysis framework (so-called "risk-informed safety margin characterization" or RISMC), a

computational engine (NGSAC), and a verification and validation (V&V) infrastructure.

"In 2009, this LDRD project enabled us to formulate a basic concept of a next-generation safety analysis code as a computational engine for risk-informed safety margin characterization, and to identify both critical capability gaps and high-risk/high-reward developments," Dinh said. "Then in 2010, we were able to take these findings, investigate them, and make advances that significantly strengthen the technical basis for the development, demonstration, and validation of next-generation system safety codes."

The methods research conducted in this LDRD project provided technical insights that facilitated R&D planning for the U.S. Department of Energy (DOE)'s Light Water Reactor (LWR) Sustainability Program's RISMC pathway. In 2010, the LWR Sustainability Program started its support for the development of a β-version of R7 code, which is a prototype NGSAC aimed at plant life extension decisionmaking. Also in 2010, DOE's Nuclear Energy Advanced Modeling and Simulation program supported implementation and testing of advanced methods in code V&V and uncertainty quantification in R7, and DOE's Consortium for Advanced Simulation of LWRs selects R7 code as the advanced system simulation engine to be integrated into its VERA package. Recognizing the potential role of NGSAC in utility's deliberation on extending LWR plant life beyond sixty years, nuclear industry has strongly endorsed the R7 code project manifested through participation in the project by industry experts supported by the Electric Power Research Institute (EPRI). Thus, shortly after a year evolved in the INL LDRD project, the NGSAC concept has secured broad programmatic support.

Significant contributions to research tasks were made by INL researchers Robert Nourgaliev, Vincent Mousseau, Haihua Zhao, Scott Lucas, Ray Berry, Kimberlyn Mousseau, University of California-Davis' mathematics professor Steve Shkoller, Utah State University (USU)'s mechanical engineering professor Barton Smith, North Carolina State University's

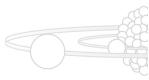


mechanical and aerospace engineering professor Hong Luo, and their graduate students.

The LDRD project research results were documented in fifteen technical reports, including seven conference papers and two journal publications. A paper on PIV uncertainty led by a USU student won the American Society of Mechanical Engineer's Knapp Award for outstanding original paper in fluid engineering. In addition, the project researchers shared their developments during the 2009 and 2010 Modeling Experimentation Validation (MeV) Summer School, which was used as training

materials for nearly 100 young researchers and engineers in the field of reactor thermal hydraulics and reactor safety. Dinh is the school's academic dean.

These technical advances, along with progress in the NGSAC concept development and a growing multi-institutional partnership in support of the R7 code project, mean that INL's Nuclear Science and Technology program's investment in "safety analysis" has successfully helped define a strategic R&D area with potential for transformative impact on nuclear power industry and engineering practice.



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### **INL Advances Understanding of Control System Data Fusion**

Timothy McJunkin IC107



Back row: Timothy McJunkin and David Gertman. Middle Row: Jason Wright, Lee Shunn, and Ronald Boring. Front Row: U of I professor Milos Manic and Miles McQueen.

Modern critical infrastructure control and security systems have the capability to provide facility mangers, operators, and security personnel with an abundance of monitoring data. It is critical for facilities to turn that data into information and decisions in a timely, actionable time frame.

This Laboratory Directed Research and Development (LDRD) project is led by Idaho National Laboratory (INL) principal investigator, Timothy McJunkin, and includes contributions from INL researchers Miles McQueen, Lee Shunn, David Gertman, Ronald Boring, and Jason Wright. The team is also collaborating with University of Idaho (U of I) professor, Milos Manic, and his students, Kevin McCarty and Ondrej Linda.

During the first year of this project, a very simplified process model and threat scenario was created to aid in the development of the initial data fusion conceptual approach. Then, in its second year, these research efforts were built upon by adding complexity to the process as well as adding a more sophisticated cyber-event to further define and test the data fusion capability.

"Basically, the end result of data fusion is to create situational awareness. Raising a red flag is great, but it is still time-critical to look at all the data to make a decision. We hope our research will help end users be more effective when making a decision," says McJunkin.

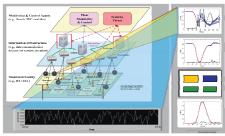
By bringing in experts in process information, cyber-security, and computational intelligence, this year's project became multidisciplinary. Ultimately, each member of the research team is trying to capsulate a lot of data into a usable form. For example, human factor experts Ronald Boring and David Gertman take the data and try to determine the best way to present that information and test the human performance in the monitoring and control of the system. In addition, the U of I collaborators are focused on the computational intelligence side of the research project in order to advance data mining tools for strategic optimization and tactically important early accurate detection of anomalies.

One way the research is currently being used is by collecting synthetic fuel model information such as temperature, pressure, flow rate and collecting data on the process. One application, on which the research is currently focused, is a complicated synthetic fuel generation plant model, which is a component of the INL's Hybrid Energy Test (HYTEST) platform. HYTEST forms a tightly coupled system between energy generation and multiple uses of the energy; thus, provides a data rich process requiring data fusion to be monitored and controlled effectively. The data and alarms measured in the process, such as temperature, pressure, flow rate and chemical composition are combined through data fusion to present the information of most interest of importance at a given moment to operators and/or supervisors of the plant. In addition, this type of research – gathering information so timely decisions can be made – is envisioned to assist in addressing human error introduced into plant system operations or to mitigate large scale cascading impacts of system degradation, e.g., the Northeast blackout of 2003. In addition, this type of research – gathering information so timely decisions can be made – is envisioned to assist in addressing human error introduced into plant system operations or to mitigate large scale cascading impacts of system degradation, e.g., the Northeast blackout of 2003.





"The information we are gathering is critical to understand where things can happen and hopefully minimizing these errors in the future, producing systems that provide greater resilience," explained McJunkin.



Visualization - Human Machine Interface

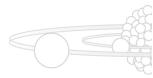
Data Fusion Provides Advanced Alarms, Organization and Highlighting of the Most Interesting Information

Cyber Security

Process

Physical Security

Most time-critical or interesting data is highlighted to operators or supervisors as expediently as possible.



# INL Researchers Develop International Safeguards for Commercial Pyroprocessing Facilities

Richard Metcalf
NN119



Richard Metcalf

In a world where security is a top priority, Idaho National Laboratory (INL) is stepping in to assist the International Atomic Energy Agency (IAEA) by offering their expertise in developing safeguards for pyroprocessing facilities. INL realizes the importance of developing a standard safeguards approach and its criticality to building a facility right the first time.

Principal investigator, Richard Metcalf, is leading the INL Laboratory Directed Research and Development (LDRD) project with coinvestigators Michael Simpson, Dee Vaden, Greg Teske, Robert Bean, and David Gertz.

INL's unique pyroprocessing facilities created an ideal opportunity to take the lead on this project. The primary objectives of the LDRD were to determine the validity of existing safeguards approaches for pyroprocessing facilities and exploit the unique attributes of the pyroprocess to identify new safeguards methods. INL development of these pyroprocessing safeguards will constitute a new sub-field in reprocessing safeguards and will bring INL recognition as the pre-eminent experts in international safeguards applied to pyroprocessing facilities.

"PUREX (wet chemistry reprocessing)-type safeguards are generally expected to be applied to pyroprocessing. This is a major misunderstanding; that's comparing apples to oranges," explained Metcalf. "We've spent significant time and effort to lay that foundation properly, so that the material can be secured using techniques that INL intends to develop."

Efforts in 2010 were undertaken to continue those that began in 2009. Specifically, the project effort was directed towards:

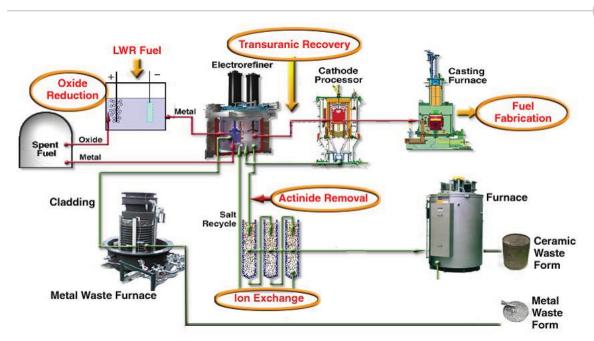
- Continuation and advancement of the diversion pathway analysis.
- Design and fabrication of the digital cloud chamber and further development of the inverse spectroscopy algorithm.
- Laboratory analysis of the Curium content of selected fuel samples from the INL pyroprocessing facility.
- Investigation of the effectiveness and expense of installation of IAEA safeguards equipment at the INL Fuel Conditioning Facility (FCF).
- Investigation of the application of goalsdriven safeguards principles to pyroprocessing.

Additionally, effort began to perform MCNP radiation modeling of detector response around the electrorefiner, one of the most complicated equipment pieces in the process. The project also worked with the INL classification officer to develop a project classification aid that drew from multiple classification guidance sources.

A solid basis has been formed to develop a safeguards approach using processing experience at FCF as a baseline and demonstration platform that can be applied to international commercial pyroprocessing facilities.

"No country is going to be told 'no,' that's not our place or our right," Metcalf said. "But we can provide them the tools to secure their material the right way, for the sake of our collective international security."





Pyro flowsheet.

## **Project Supports Multi-Scale, Multi-Physics Simulations**

Maria Okuniewski
AF107



Dr. Maria Okuniewski

Nuclear energy researchers at Idaho National Laboratory are working to understand the microstructural evolution in irradiated U and UO<sub>2</sub> with positron annihilation spectroscopy (PAS) and transmission electron microscopy (TEM). Moreover, a unique PAS system will be designed specifically to examine irradiated materials.

This Laboratory Directed Research and Development (LDRD) project is led by INL principal investigator, Dr. Maria Okuniewski, and includes contributions from Prof. Kelvin Lynn and Dr. Marc Weber from Washington State University, and INL researcher Doug Akers.

PAS and TEM is being utilized to analyze defects, determine defect energies, and diffusion lengths in  $\mathrm{UO}_2$  and  $\mathrm{U}$ .  $\mathrm{UO}_2$  is of particular interest since it is a model oxide fuel for nuclear reactors (e.g., light water and fast reactors), while U is a model metallic fuel for fast reactors. The results from their research will be used to understand the microstructural evolution in  $\mathrm{UO}_2$  and U as a function of various irradiation parameters, such as burn-up, type of irradiation particle, and temperature. The neutron irradiated model fuel will be utilized to test the newly designed PAS system for irradiated materials.

The success of this project can be attributed to the expertise of the entire team, which covers a large knowledge base ranging from positron physicist to positron systems designer to nuclear materials engineer. It has been a pleasure to work with the WSU team again, as well as with researchers across the INL campuses.

Within nuclear reactors, the fuels are subjected to irradiation damage from neutrons, fission products, both solid and gas, threshold reaction particles, electrons, gamma rays, etc. The radiation damage and decay processes produce point defects (vacancies and interstitials), as well as gas atoms. These point defects may annihilate or cluster and agglomerate. The gas atoms typically have low solubilities and are trapped within the various fuel matrices, hence they nucleate bubbles. Solid fission products may also precipitate out, form new phases, or react with the cladding to form wastage. This irradiation damage has deleterious effects on the thermal and mechanical properties of the fuels. INL is working to understand these irradiation effects within the fuels, specifically the irradiation-induced evolving microstructure and its relationship to the degradation of the thermal and mechanical properties is crucial to the understanding of fuel behavior and design, as well as to the development of multi-scale, multiphysics models.

Currently, an interest exists in conducting multi-physics, multi-scale experiments and modeling of nuclear fuels and materials to ultimately produce a predictive fuel performance code. These techniques are necessary to help quantify and eventually predict nuclear fuel performance since the present US nuclear infrastructure is no longer capable of generating the large irradiation performance database needed to develop and validate a fuel performance code based on the traditional approach. Historical fuel performance codes used empirical correlations that were fit to the data obtained from post-irradiation examinations of irradiated fuels. Although these codes were valuable, they could not predict fuel behavior. Therefore, a need for a new model exists that can take into account both previously accumulated data along with new data for validation purposes. The ultimate goal of this code would be to



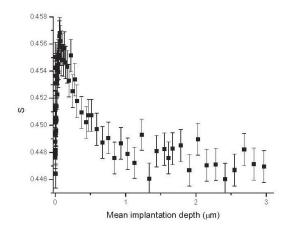
predict fuel behavior of new nuclear fuels with a minimal number of experiments necessary for validation. The experiments that are being carried out under this LDRD will directly support the validation (e.g., resultant irradiation-induced microstructure), as well as input parameters necessary for this new model (e.g., vacancy formation energy).

In the past, the details of the fuel microstructure were largely ignored for a few reasons: (1) there was a much greater focus on the overall fuel performance and the thermophysical properties of the fuel, which were primarily related to the heat transfer capabilities; and (2) there was a number of technological challenges related to handling and analyzing irradiated fuel that are gradually being overcome and solved. This research directly addresses the second point since it is creating a new tool at INL to examine irradiated materials.

Our future focus of the research will extend to linking these results to multi-scale, multi-physics models. The follow-on research will also include developing the capabilities to conduct spatially resolved PAS measurements of larger, irradiated specimens.

The significance of this research is that it will provide unique data (e.g., defect formation energies, as well as a qualitative and quantitative description of defects in the irradiated fuels),

lead to a better understanding of the impact of microstructure on the thermal and mechanical properties of fuels, and provide crucial input and validation data for multi-scale, multi-physics models. More specifically, this research will help to support the efforts of the Energy Frontier Research Center (Center for Materials Science of Nuclear Fuel) at INL, as well as modeling efforts being undertaken by the Nuclear Energy Advanced Modeling and Simulation program.



PAS Doppler broadening depth profiled results of a depleted U foil. The higher S parameter near the specimen surface indicates the presence of an oxide layer.

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### **Research Advances Next Generation Hybrid Energy Systems**

*Lee Shunn* EI112



Dr. Lee Shunn

In recent years, there has been a push in the United States to produce energy more cleanly and efficiently. Advanced hybrid systems that combine several conventional technologies into a flexible energy system are one solution to meeting our nation's energy needs. Research to effectively design, operate, and control these hybrid systems is the focus of the Idaho National Laboratory (INL) Laboratory Directed Research and Development (LDRD) project led by principal investigator, Lee Shunn, and supported by co-investigators, Dan Ginosar and Eric Larsen.

"We use a small-scale reactor system that we've built in the lab to examine how dynamic changes in operating conditions like temperature and pressure cascade through an energy system and affect processes downstream," said Shunn. "Characterizing the transient response of each component helps us design control systems to operate these very complicated systems safely." As part of this project, Shunn has developed a computer model for dynamic simulations of chemical processes and energy systems. This software includes models for catalyic synthetic fuel reactors and advanced membrane separations. Models for other process equipment, such as compressors, valves, storage tanks, heaters, coolers, and their controllers, have also been developed. The models are combined in an overall process simulation that describes fuel synthesis from gasified coal.

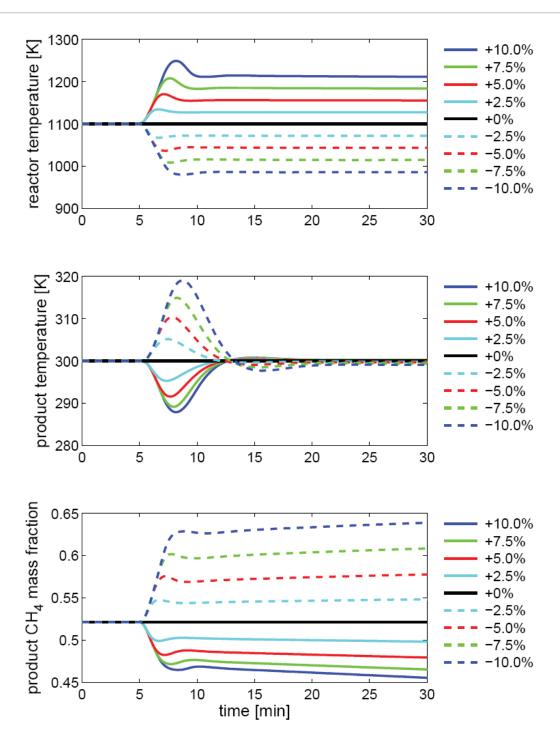


HYTEST laboratory-scale synfuels reactor system.

The computer model offers a fully transient description of an advanced chemical process and is being used to demonstrate adaptive modeling concepts for controller design. The process model is already being used by two additional LDRD projects and several university collaborations. This work has helped identify and address technological gaps and research needs in the field of instrumentation, monitoring, and control of advanced hybrid energy systems.

During the next couple of years, the research team will build new tools for adaptive modeling for process control, state assessment, and system prognostics in tightly coupled, interdependent reactor systems. These efforts contribute to our energy security by enabling systems that efficiently use domestic resources to deliver clean energy products.





Results from a computer model of methane synthesis showing the transient response of the system to changes in the reactor temperature system.

### **Project Advances Water Purification System Research**

Frederick Stewart ST144



From left to right: Michael Benson, Aaron Wilson, Frederick Stewart and Mark Stone

During the past year, advances were made in creating a high efficiency water purification system that is adaptable, deployable, and portable as part of an Idaho National Laboratory (INL) Laboratory Directed Research and Development (LDRD) project.

Using hybrid osmosis, principal investigator, Frederick Stewart, and his co-investigators, Mark Stone, Mike Benson, and Aaron Wilson, successfully demonstrated a relatively new technology that will help in the shortfalls of current membrane technology.

Hybrid osmosis, a dual system that uses a combination of forward osmosis (FO), reverse osmosis (RO), and ultrafiltration (UF) to create a system that is relatively insensitive to the applied feed stream, is the name applied to the INL concept of a two-stage water purification system based on membrane technology.

The initial activity for this project was the construction of a lab-scale forward osmosis system suitable for the analysis of membranes and draw solutions. Specifically, a number of membrane substrates have been characterized for bulk water flux using sodium chloride and magnesium chloride in the draw solution. Eight commercial membranes were used and screened against both salts.

"Our technology niche is in the draw solution where we have created new classes of draw solutes that drive the process," explained Stewart. "Commercial membranes are advanced enough that we wanted to focus on what the new INL salts would do with different membranes.

We found that our salts are as good as any and better that most."

The INL technology avoids many of the problems with RO by using a two stage system. The first stage is a FO system, which is a liquid separation process where feeds are circulated at low pressure across a membrane. The second stage can be an RO system, or a nanofiltration/ultrafiltration system that is specifically tailored to the draw solution. Thus, the FO system in front can reduce much of the downside of RO by replacing the pressure driving force with an osmotic driving force. Further, the draw solution can be formed using a solute that has little fouling potential, so the second stage can be specifically configured to one type of feed, the draw solution, thus optimizing this second stage.



Membrane cell.

The net result is a water purification system that is less sensitive to incoming feeds and extends the operating lifetime of the membrane by significantly reducing fouling and other deleterious behaviors. All that is required is a careful determination of the draw solution composition that must be tailored to the expected nature of the feed, and membrane selection.

Stewart added, "Ultimately, we wanted to lower the potential of membrane foul. We were able to show through our research that the system is relatively insensitive to the applied feed stream."

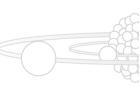






Nuclear Energy Science & Technology





# Reactor Physics Sensitivity Analysis, Uncertainty Quantification, and Data Assimilation Capability

Cristian Rabiti

**Provide a reactor design toolkit capable to perform uncertainty quantification** NE153

Sensitivity analysis and uncertainty quantification are essential tools in reactor physics design. They allow quantifying margins, performing optimizations, defining target accuracy requirements, evaluating relevance of experiments, and carrying out nuclear data adjustments. The goal of this laboratory-directed research and development (LDRD) project is to provide Idaho National Laboratory (INL) with the most advanced methodologies and tools to perform such a task: (1) developing the needed new software tools, (2) defining a scientifically based methodology for validating existing codes and data, (3) applying the tools and the methodology using available experimental data, and (4) creating both expertise and a culture among INL reactor physicists that could be eventually propagated to other fields (e.g., thermal-hydraulics, thermal mechanics, fuel behavior, etc.) for a multiphysics application.

#### Project Summary

The main objective of this project is to address the following issues:

- Create a state-of-the-art framework to perform V&U in reactor physics field
- Scope V&U new methodologies for reactor physics
- Extend V&U methodologies already used in reactor physics field to multi-physics applications
- Test new V&U methodologies in particular for non-linear problems.

How these tasks will be accomplished:

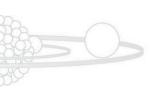
- A framework environment tailored to the development of a reactor sensitivity analysis code suite will be created. We are also planning to evaluate the possibility to align our effort with similar project ongoing at the division level.
- A new nodal parallel code with adjoint capability will be developed and interfaced with HELIOS.
- In cooperation with Texas A&M, the Krilov method will be applied to the solution of coupled transport solver algorithm. This approach will allow us to have a locally

- refined solution that is fundamental to the V&U process.
- In cooperation with North Carolina State University, the efficient subspace method (ESM) will be extended in order to be compatible with Monte Carlo codes.
- ESM implementation in connection with the ERANOS code will be tested and compared with other available methodologies.
- As part of the LDRD, we foresee the involvement of the Organization for Economic Cooperation and Development's light water reactor (LWR) uncertainty analysis modeling.
- We will evaluate the potential extension of the representativity concept in other fields, such as fluid dynamics and fuel behavior.

#### Results and Accomplishments

- Software able to perform sensitivity based on the ESM has been finalized in cooperation with North Carolina State University. The code perturbs microscopic cross sections according to user stochastic distributions and uses the ERANOS code suite to analyze the neutronic response of reactors. Both the manual and the software are available at INL.
- The application of the ESM methodology for stochastic code application has been fully derived and a module to perform the perturbation of the ACE cross section format is ongoing.
- The INSTANT (Intelligent Nodal and Semistrucured Treatment for Advanced Neutron Transport) code has been written and internally released. INSTANT is a neutron transport solver based on the second-order PN equation and hybrid finite element method. At this stage, INSTANT supports triangular, hexagonal, and Cartesian geometries in both 2-D and 3-D with reflecting, vacuum, and periodic boundary condition. The code has parallel implementation based on domain decomposition with MPI to take advantage of the development of supercomputing techniques.





- A StarCCM+ model of a seven-pin with wire wraps sodium-cooled reactor has been realized and interfaced with the DAKOTA Uncertainty Quantification (UQ) module. Presently we are collecting reliable information on the input uncertainty in order to perform a realistic analysis of the uncertainties. The variation on geometry was also investigated to show that varying positions of a pin, such as slanting or bending, could be taken into account. For the variation of the position of the center pin with respect to its nominal position, Sculptor mesh deformation software was used to deform the center pin slightly in the X and Y directions without the need to remesh. In addition, because we did not have enough uncertainty statistical data for a sodium reactor, we entered into cooperation with the Karlsruhe Institute of Technology (KIT) in Germany. Its researchers have completed many actual experimental tests and continue to test different methodologies and geometries for lead-bismuth-cooled reactors. Under a memorandum of understanding between INL and KIT, an INL representative traveled to Germany and spent two weeks with KIT personnel (July 10-24, 2010). He toured the many experiments they have assembled for both lead-bismuth-cooled studies as well as their soon-to-be-completed sodium-cooled reactor studies.
- During Fiscal Year 2010, several activities relative to nuclear data validation were carried out. These include:
  - Target accuracy assessment
  - Assessment of the impact of core minor actinide (MA) content on reactivity coefficients for advanced reactors
  - Coordination of the OECD/ Nuclear Energy Agency (NEA) subgroup on "Methods and Issues for the Combined Use of Integral Experiments and Covariance Data."

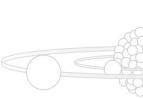
For the target accuracy assessment, the impact of correlations among a set of selected nuclear data has been studied.

In order to perform this study, the optimization code SNOPT was acquired and the improved methodology that takes into account the correlation was implemented. The impact of correlation terms is very significant in target accuracy assessment evaluation and produces very stringent requirements on nuclear data. For this type of study, a broader energy group structure should be used in order to smooth out requirements and provide better information to evaluators and cross section measurement experts. The results of the investigation also indicate that a careful analysis is still needed to define the most appropriate and effective strategy for data uncertainty reduction. It seems that, besides a further consolidation of the present covariance data libraries, a strategy of combined use of integral and differential measurements should be further pursued in order to meet future requirements.

The introduction of significant amounts of MAs can have an impact on the core reactivity coefficients (e.g., coolant void and Doppler coefficients) and, in the case of cores with very low conversion values, on the delayed neutron fraction. A study of this issue has shown that it is impossible to define rather theoretical "upper limits" in the MA content in the fuel of a fast reactor that would recycle transuranics in a homogeneous mode. However, a careful physics analysis. which should be performed system by system, can suggest appropriate design measures that would limit the potential impact on safety of the MA content increase in the core, accounting for both void reactivity and Doppler reactivity effects.

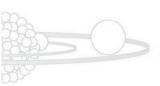
#### Benefits to DOE

The capabilities developed can eventually be used in the certification process for licensing future reactors with the Nuclear Regulatory Commission. Moreover, the sensitivity and uncertainty evaluation tools as well the



validation strategy developed in this LDRD could be directly applied to quantify and reduce margins in nuclear reactor design of interest to the U. S. Department of Energy (Generation IV [GEN-IV], Next Generation Nuclear Plant [NGNP], and advanced LWRs). This project introduces a new approach in the complex field

of uncertainty quantification. We aim, in the long term, to propagate and mix with already existing uncertainty methodologies the adjoint approach in the multi-physics field. This will enhance our confidence in simulation, reduce experimental needs, and decrease reactor design time.



### **Next-Generation Code for Nuclear System Safety Simulation**

Nam Dinh

To enable science-based simulation-driven safety analysis of nuclear reactor systems NE156

The project focuses on the basic capabilities needed to enable development, demonstration, and validation of next-generation system simulation and safety analysis codes, including fluid flow homogenization, high-order-accurate solution scheme, validation, and uncertainty quantification.

#### Project Summary

Single-phase flow modeling: A novel homogenization method, the so-called Lagrangian averaging model was developed, which successfully removes small-scale spurious oscillations from smooth flows and offers an order-of-magnitude scale-up in the computational mesh. This includes an extension, the so-called alpha-beta model, which uses the proposed homogenization approach in the smooth flow regime, and a new shock-capturing technique in the discontinuous flow regime.

Two-phase flow modeling: Different approaches to hyperbolization were studied, with focus on testing the potential of a seven-equation model and secrete element method. Applicability of the models to description of steam-water flows in a reactor safety code was analyzed. The study suggests that a modified six-equation model with adjustment for consistent closure relations is a preferred option.

Advanced numerical solution algorithms research focuses on high-order-accurate numerical methods in all-speed (all-Mach) compressible flow. The results confirmed the hypothesis about potential for significant improvement of computational efficiency while maintaining solution accuracy by means of the fully implicit JFNK algorithm and rDG scheme.

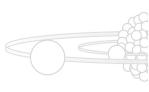
The experimental uncertainty quantification and validation support activity focused on development of automatic uncertainty

quantification for particle image velocimetry (PIV) and design and construction of a facility for computational fluid dynamics (CFD) validation. The work demonstrated convincingly that PIV uncertainty varies in space and can be computed with minimal expense to the user.

CFD verification and validation (V&V) data management was initiated at Idaho National Laboratory (INL) as part of 2010 activity in this project. This has led to development of a proof-of-concept configuration, acquiring and loading the first set of high-quality experimental data (supplied by Bettis and Utah State University) to the system. The activity has attracted a broad base of support from industry to academia and regulator.

#### Benefits to DOE

This research provided technical capabilities and expertise needed to develop analytical tools that transform the current practice in system simulations and safety analysis, as critically needed to support safety and economic improvements and life extension of the aging fleet of light water reactor (LWR) plants. The insights are instrumental to formulation of research and development (R&D) scope and planning for the Department of Energy's LWR Sustainability Program's Risk Informed Safety Margin Characterization pathway. Research results were used as teaching materials to contribute to the training of nearly 100 young researchers and engineers in the field of reactor thermal hydraulics and reactor safety (2009 and 2010 Nuclear Engineering Summer School "Modeling, Experimentation and Validation in Nuclear Thermal Hydraulics and Safety Analysis (MeV)").



### **Multiscale Modeling and Simulation of Nuclear Fuel Performance**

Glen Hansen

Developing a unique multiscale modeling and simulation capability that integrates engineering scale analysis with mesoscale microstructural evolution of irradiated fuel
NE159

The purpose of this project is to develop a unique multiscale modeling and simulation capability that integrates engineering scale analysis of nuclear fuel performance with the mesoscale microstructure evolution of the irradiated fuel. Radiation-induced microstructure evolution, such as fission gas production and nucleation and growth of voids, has a strong impact on the thermo-mechanical properties of fuel. Current approaches to modeling fuel performance use empirical correlations to determine these properties. However, these correlations are valid only for the experimental conditions under which they were obtained. To develop a predictive fuel performance model that is accurate in a large range of conditions, the engineering scale code must be informed by lower-length-scale simulations. Our approach achieves this by using a phase field model of irradiation-induced microstructure evolution to determine thermo-mechanical properties that reflect the current state of the fuel to an engineering scale simulation.

The FY-2010 work on this project was divided into three objectives: (1) Multiphysics Object Oriented Simulation Environment (MOOSE) framework development to facilitate creation of the multi-scale model; (2) the creation of MARMOT, a MOOSE-based mesoscale phase field code; and (3) the coupling of the engineering scale BISON fuel performance code to the MARMOT phase field code. During FY-2010, major progress was made on each objective, and an initial version of the multi-scale fuel performance code was developed.

Project Summary

The MOOSE framework has been under development at Idaho National Laboratory (INL) since 2008. It supports a wide range of scientific simulation tool development from groundwater reactive transport to nuclear fuels performance. The laboratory-directed research and development (LDRD)-funded MOOSE development included internal data structure reorganization to facilitate the linking of two MOOSE-based codes and framework upgrades to support building a phase field based mesoscale simulation tool.

MOOSE data structure reorganization focused on the ability to run two (or more) MOOSE-based simulations simultaneously and pass data between them. The bulk of the work involved compartmentalizing several large data structures so that multiple instances could be created simultaneously. The reorganization was a complete success, with MOOSE now being able to run any number of independent simulations and pass data between them. This new capability bore immediate fruit with the coupling of the BISON engineering scale nuclear fuels performance code and the MARMOT mesoscale simulation capability to do multi-scale analysis of a single nuclear fuel pellet.

Several pieces of functionality were added and optimized in MOOSE to facilitate building a phase field-based simulation capability. All of the enhancements are too numerous to list here, but a few will be summarized. Critical to the mesoscale work was the addition of periodic boundary condition support. Enhancements were also made to the initial condition capabilities in MOOSE, as well as the ability to do mesh adaptivity on those initial conditions; these are important for capturing initial details of the grain structure at the mesoscale level. Post-processing capabilities were also added to allow for the calculation of integrated quantities, such as thermal conductivity and porosity. The highly nonlinear structure of the free-energy functionals at the mesoscale level required the addition of Newton damping capabilities. Looking toward the future, Discontinuous Galerkin capabilities were developed to facilitate advanced solution techniques of the phase field equations. Numerous enhancements were made to the timestepping and execution system to allow the mesoscale simulation to permit more intelligent time-step selection. Numerous optimizations were also made to the framework, specifically to accelerate the solution process of the mesoscale

INL has been pioneering the development of mesoscale modeling of radiation-induced microstructure evolution using phase field models for several years. Previously, these phase field codes were explicit finite difference codes. These codes were limited to small time steps and could not be coupled to other physics, such as



heat conduction or solid mechanics, without adding an additional solution method. Since the phase field models are defined as a system of nonlinear equations, they can be solved using Finite Element Methods (FEM). As a part of this LDRD project, a new FEM-based mesoscale phase field simulation code was developed using MOOSE. This code, called MARMOT, leverages the considerable capabilities within MOOSE to solve fully coupled phase field equations with implicit time integration. Thus, MARMOT can take time steps four orders of magnitude larger than the explicit phase field codes. Further, MARMOT fully couples the phase field calculations to solid mechanics and heat conduction calculations. In FY-2010. MARMOT was used to model void nucleation and growth and grain growth. Also, MARMOT directly supports the calculation of irradiationinduced evolution of bulk properties, such as thermal conductivity and porosity within reactor fuel. Figures 1 and 2 demonstrate the current capabilities of the MARMOT mesoscale code.

During the fuel's lifetime, its microstructure changes drastically due to radiation damage. Using MARMOT, these changes can be modeled to predict the resultant changes in bulk material properties, such as thermal conductivity. The microstructural evolution results in changes in the macroscale fuel behavior, which, in turn, has a large effect on reactor performance. INL's BISON fuel performance code predicts fuel rod behavior, in which the effect of radiation on the material behavior is traditionally represented with empirical constitutive models. By coupling mesoscale MARMOT simulations to a macroscale BISON simulation, the effects of radiation-induced microstructure evolution are captured directly.

Models at different length scales can be connected with either hierarchical coupling or concurrent coupling. In hierarchical coupling, the mesoscale model is run initially at various operating conditions to construct a mesoscale-informed constitutive model, which is then used with the macroscale simulation. Hierarchical coupling is computationally efficient, but does not directly resolve the coupling. The Center for

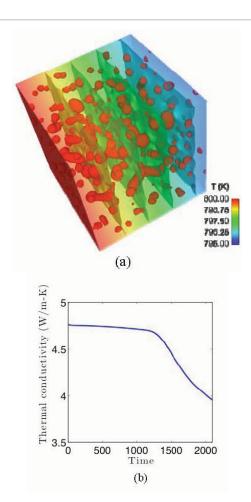


FIGURE 1. Phase field simulation of void nucleation in a 1 mm cube of UO<sub>2</sub> with a coupled heat conduction calculation to determine the bulk thermal conductivity. The final temperature profile and void structure in the cube is shown in (a) and the evolution of the bulk thermal conductivity with time due to the void formation in (b).

Advanced Vehicular Systems (CAVS) at Mississippi State University has a team that specializes in hierarchical multi-scale modeling. This team has several years of successful experience using multi-scale models to predict behavior in vehicular systems. Therefore, the LDRD project subcontracted with CAVS to assist in developing hierarchical models of the effect of microstructure evolution on thermal conductivity. The method they outlined, using experimentally validated models at each scale to feed information to the next higher scale, will be implemented in FY-2011.



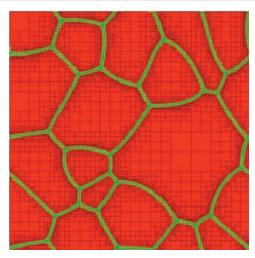


FIGURE 2. Phase field simulation of grain growth in a copper polycrystal. Mesh adaptivity is used to increase the computational efficiency of the simulation.

In concurrent coupling, the mesoscale simulations are run simultaneously with the macroscale simulation, and information is passed between the scales. Concurrent coupling is more computationally expensive than hierarchical coupling, but it directly resolves the interaction between the scales. In FY-2010, BISON was coupled to MARMOT to capture the effect of void nucleation and growth on the bulk thermal conductivity. As an initial effort, a single MARMOT simulation was coupled with a BISON single fuel pellet simulation. The average temperature in the pellet was passed to the MARMOT simulation and the average thermal conductivity was passed to the BISON simulation. The coupled model performed very well. Figure 3 shows the results from the simulation.

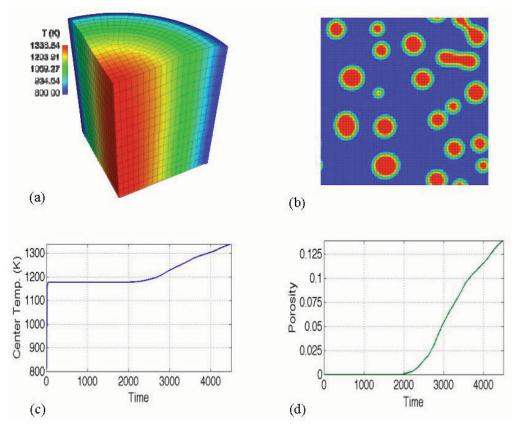


FIGURE 3. A simulation of heat conduction in a  $UO_2$  fuel pellet is concurrently coupled to a mesoscale simulation of void nucleation under irradiation. The average pellet temperature is used at the mesoscale to determine the vacancy mobility while the porosity is used at the macroscale to determine the thermal conductivity. The temperature profile of the eighth-section of the fuel pellet is shown in (a), the final void structure is shown in (b), the center pellet temperature is plotted versus dimensionless time in (c) and the mesoscale porosity versus time is shown in (d).



### Benefits to DOE

During FY-2010, an initial multi-scale model that captures the effect of radiation-induced void nucleation on the heat conduction within a  $\rm UO_2$  fuel pellet was developed. To accomplish this goal, upgrades were made to the MOOSE framework, and the MARMOT phase field code was created using MOOSE. Using the

multi-scale simulation methodology developed in this project permits the creation of a predictive multi-scale fuel performance code. Such a simulation tool will provide a means of developing innovative fuel designs that will assist in securing U. S. energy security in the future.

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### **Nuclear Reactor Safety Case**

Vince Mousseau

Producing INL-specific production software to augment RELAP5-3D

This project is focused on producing production software to augment the current Idaho National Laboratory (INL) nuclear reactor safety code RELAP5-3D. The goal is to capture the capability of the RELAP5 code in modern software with improved numerical methods. This software will then provide the foundation for future reactor safety work.

#### Project Summary

This project has only been funded for the last six months of Fiscal Year 2010. Because of that, there has mostly been startup work; most of the people funded are only working a fraction of a FTE.

The project has focused on the following areas:

- a. Choosing test problems to focus future work. The RELAP5 software has evolved for about 35 years. It now has a very wide range of applicability and to reproduce its capability will be a large job. Therefore, this project has adapted the strategy to pick one reactor and one transient to focus on first. This proof of principle on a real transient and a real reactor should provide an avenue to secure programmatic funding.
- b. Choose and learn a computational framework for the basis of the new software. The goal here is to minimize the amount of math and computer science required by the nuclear engineers who will be developing this software. Previous laboratory-directed research and development (LDRD) investments in the MOOSE software will be leveraged in this LDRD. The MOOSE framework will provide the temporal and spatial integration techniques as well as the mesh generation. This will leave the nuclear engineers to focus on the physics of the application.
- c. Develop a validation database. There is a strong push to employ computational fluid dynamics (CFD) codes as part of a nuclear reactor safety study. To utilize a CFD code this way, one needs to validate

- the applicability of the code. So work has progressed to leveraging off another LDRD this year to lay the ground work for a CFD database and to write a white paper to get funding from the National Institute of Standards and Technology. The work is also being marketed to the Department of Energy Office of Nuclear Energy, Science, and Technology (DOE-NE). Additionally, investigations have begun to create a similar database for system code validation.
- d. Initial work has begun to figure out what key improvements can be made to RELAP5 to make the transition to a new reactor safety code easier. Two important aspects are being investigated. The first is to add an iteration loop to the RELAP5 numerics to make the code more implicit. The second is to modernize (convert from PVM to MPI) the RELAP5 "executive" package. The "executive" software is how RELAP5 communicates with other software. The goal of this optimization process is to construct an interface between RELAP5 and the new thermal hydraulics code. This interface will enable code users to choose between the current RELAP5 models and the models in the new code.

#### e. External collaboration:

- Los Alamos National Laboratory –
  Work is proceeding with a senior
  scientist at LANL to learn data
  assimilation techniques and how to
  employ them.
- ii. *University of Utah* A Ph.D. student is learning about multi-physics simulation and how to use data assimilation as a tool for experimental design.
- iii. North Carolina State University A professor is studying the model uncertainty between two different two-phase flow models.

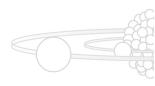


- iv. The Ohio State University A professor and a student are designing an experiment to measure the twophase flow sound speed with two different methods in the same experiment.
- v. Massachusetts Institute of
  Technology A professor and a
  student are working on development
  of a new method of uncertainty
  quantification for nuclear reactor
  safety codes.
- vi. *University of Michigan* A professor and a student are working on a new method of risk assessment.

#### Benefit to DOE

The RELAP5 code plays a major role in the nuclear power industry in the Unites States and throughout the world. It is used by the U. S. Nuclear Regulatory Commission (NRC) under the name TRACE; and the nuclear vendor, AREVA, under the name SRELAP. The work to maintain and improve the RELAP5 code capability will support projects in DOE-NE (light water reactors, small modular reactors, HUB, NEAMS, and Nuclear Energy Enabling Technology) as well as NRF, the NRC, and the nuclear power industry.

A database to validate CFD code and nuclear reactor safety codes will benefit all agencies that use CFD as part of their computational simulation effort.



**Idaho National Laboratory** 

30

# **Process Modeling of Solvent Extraction Separations for Advanced Nuclear Fuel Cycles**

Dr. Troy Tranter

Define and develop useable dynamic process models to simulate advanced solvent extraction processes under transient and steady-state conditions

A fresh process modeling approach that incorporates efficient numerical solution methods for the governing differential equations (in both temporal and spatial dimensions) is essential for simulating, understanding, and predicting the inherent transient behavior and feedback loops of such a complex series of unit operations. A capability for accurately simulating the dynamic behavior of advanced fuel cycle separation processes will provide substantial cost savings and myriad technical benefits to include predicting and evaluating transient response to off-normal or upset conditions, establishing operational safety envelopes, evaluating product purities, minimizing waste volumes, or even examining proliferation scenarios. The objectives of this project are to define and develop useable dynamic process models to ultimately simulate advanced solvent extraction processes under transient and steady-state conditions.

#### Project Summary

An executable model was developed in the first year to successfully simulate dynamic conditions in a PUREX-type flow sheet using conventional mixer-settler solvent extraction equipment. This model was expanded in the second year's work to simulate the timedependent changes, from startup to steady-state, in 30 stages of annular centrifugal contactors (ACC) running a TRUEX flow sheet. A pilot-scale demonstration was then performed and results compared to the model simulation. Work in the final year was focused on further refinement of equations to model concentration transients in an ACC. The ACC model was developed using a simplified combination of continuity and momentum conservation terms from the Navier-Stokes equations. Systems with forced convection terms (i.e., mass flow) are often modeled adequately by considering spatial gradients only in the direction of flow. Effects from flow vectors in other coordinates, i.e. mixing, are approximated by an overall dispersion coefficient. This approach is not technically correct in terms of modeling the real physics; however, it is common in engineering practice and has provided levels of fidelity that are adequate for research and development support. The ACC model uses three well-mixed

volume elements to simulate concentration changes in each phase occurring in the mixing zone and a continuous dispersed plug flow volume element to model concentration changes occurring in rotor.

The model provides reasonable predictions of the extractable metal as compared to the experimental data. Although every effort was taken to sample at precise time intervals, the samples were collected manually and thus a significant uncertainty was introduced by the sampling method. The ACC system reaches steady-state rapidly and a few seconds of deviation between sampling times could be expected to impact experimental results. Nevertheless, the simulation predicts dynamic and steady-state concentration profiles that agree quite well with expected system behavior, which indicates the structure and numerical solution of the model equations are correct. It was also shown that the model simulation converges to a solution quite rapidly. This work is novel in that a functional model for centrifugal contactors does not currently exist. This type of simulation is needed to accurately simulate candidate flow sheets under Fuel Cycle Research and Development Program (FCR&D). The modeling capabilities developed under this laboratorydirected research and development project have resulted in follow-on programmatic funding for Fiscal Year 2011 (Safeguards & Separations Modeling).

#### Benefits to DOE

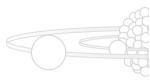
A sustainable, secure and safe nuclear energy source hinges on a variety of factors, including the development of an advanced. closed nuclear fuel cycle. Separation processes are key components in the development of advanced nuclear fuel cycles. Development of dynamic models for the advanced aqueous separation processes for the treatment of spent nuclear fuel will support the DOE Energy mission. These models will help to ensure the productive and optimal use of energy resources in recovering and using the energy remaining in spent nuclear fuel. This project will also contribute to the mission of reducing the nation's dependence on foreign oil by supporting the expansion of nuclear energy production in the United States.



The cleanup of legacy nuclear wastes, as well as environmental stewardship for future nuclear related missions, are important aspects of EM programs. Radionuclide chemistry and separations will continue to be important aspects of EM missions. Development of process models for advanced aqueous processes will form the basis of a predicative capability useful to minimize the amount of nuclear waste requiring disposal in a geological repository and extend the life of the geological repository. This will allow a greater amount of spent nuclear fuel (existing and future) to be dispositioned in the repository. This directly supports the EM mission to dispose of radioactive waste left behind as a byproduct of nuclear weapons production, nuclear powered naval vessels and commercial nuclear energy.

The project is integral to the development of an advanced nuclear fuel cycle. This project has the potential to greatly simplify an advanced aqueous processing strategy by using predicative process modeling. Since these models would be predicated on a solid understanding of the process chemistry and engineering technologies, dynamic process models would serve to identify the those areas where science and technology research would be the most fruitful.

The National Security mission includes advancing nuclear non-proliferation. Advanced reprocessing of spent nuclear fuel requires processes which minimize/eliminate proliferation potential in the nuclear fuel cycle. The proposed research focuses on developing new processes to separate the actinides and lanthanides into one or more products. This reduces proliferation concerns by grouping the Pu with other actinides and avoiding production of very pure forms of Pu.



## Reactivity of Radiolytically Produced Nitrogen Oxide Radicals toward Aromatic Compounds

Bruce Mincher

Determination of the kinetics and mechanisms of nitration of phenyl rings by radiolytically produced nitrogen-centered radicals in acidic aqueous solution

NE147

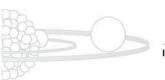
This was the final year of the three-year project. The mechanism, kinetics, and products of the nitration of aromatic compounds, using strongly activating anisole and non-activating toluene were characterized in acidic and neutral nitrate solution. These results allowed us to identify that different nitration mechanisms operated under different conditions, which in turn allowed us to predict products. For example, we found that in acidic solution, nitration occurs by nitrosonium or nitronium reaction mechanisms, depending on the activation hardness of the substrate molecule. Conversely, in neutral solution, nitration reactions occur by free radical addition to aromatic rings, with a different product distribution. Free radical nitration also occurs on alkyl substituents of aromatic rings. The practical result of these findings is that the yield and identity of nitration products can now be better predicted for nuclear solvent extraction ligands.

#### Project Summary

The objective was to define the kinetics and mechanisms of the nitration of phenyl rings by radiolytically produced nitrogen-centered radicals in acidic aqueous solution. The use of nitric acid as a solvent is ubiquitous in the nuclear industry, especially in spent fuel reprocessing. Understanding the mechanism of nitric acid radiolysis is key to controlling the degradation of the compounds used in nuclear fuel reprocessing and high-level waste treatment. These results are also significant to other disciplines interested in free radical chemistry, including the hazardous waste treatment community and the atmospheric chemistry community, where similar N-radical reactions are important.

#### Benefits to DOE

This project has provided information that allows intelligent design of molecules for use in fuel cycle separations. For example, aromatic ethers such as Cs-7SB (used in the Cs and Sr Solvent Extraction (CSSX) process) are preferentially nitrated in irradiated nitric acid by the nitrous acid catalyzed reaction. This nitration does not significantly impair solvent extraction performance, but probably does protect other species in solution from nitration.



## Consolidated Electrochemical Technique for Separation and Recovery of Actinides from Fission Products in Oxide Fuels

S. D. Herrmann

This research investigates electrolysis of dissolved oxide fuel in a molten salt electrolyte for separation and recovery of uranium

NE157

The purpose of this research is to characterize the fundamental chemistry and electrochemistry of a novel molten salt system for separation and recovery of actinides from fission products in oxide fuels. The primary objectives of this research are to measure (1) the extent of uranium oxide dissolution and (2) the electrochemical potentials and current densities for decomposition of dissolved uranium oxide as a function of system temperature and uranium fluoride composition.

#### Project Summary

Three series of tests were performed in Fiscal Year 2010 to characterize the dissolution and electrolytic decomposition of uranium oxide in molten fluoride salt systems with select anode and cathode materials. The first series of tests involved loading the molten salt furnace - III with a ternary eutectic mixture of LiF-NaF-KF to which uranium tetrafluoride was added for a 20 wt% uranium concentration. Uranium oxide was immersed in the salt pool, and cyclic voltammetry was performed on select anode and cathode materials within the molten salt at varying temperatures between 575 and 900°C to determine reaction potentials. Salt samples were taken at each temperature setting and analyzed for oxygen concentration. The molten salt was then subjected to constant current operations at 650, 725, and 800°C, during which uranium metal was deposited on a cathode rod and oxygen ions participated in reactions at select

anodes. Samples of uranium deposits were also taken for chemical analysis. The prescribed series of tests was repeated with a molten salt loading of LiF-UF4 eutectic, which involved a higher uranium concentration (62 wt%) to improve uranium oxide solubility. Salt sample analyses revealed generally higher uranium oxide concentrations in the LiF-UF<sub>4</sub> than those in LiF-NaF-KF-UF<sub>4</sub> A third series of tests was performed with a molten salt loading of LiF-UF<sub>4</sub> in which the uranium tetrafluoride was converted to uranium trifluoride. This latter salt system exhibited a more direct reduction of uranium to metal at the cathode than that with the previous salt systems. Salt samples from the third series of tests are awaiting analysis for uranium oxide concentrations.

#### Benefits to DOE

This research addresses Idaho National Laboratory's Fuel Cycle Research and Separation needs by investigating the fundamental electrochemistry of a novel fuel cycle concept, which could prove to be a simpler and more cost-effective electrochemical treatment of oxide fuels than the state-of-the-art approach. This research could benefit programs within the Department of Energy (e.g., Fuel Cycle Research and Development) and industry (e.g., General Electric – Hitachi) that are pursuing electrochemical treatment of nuclear oxide fuels.

Idaho National Laboratory

34

### **Neptunium Redox Chemistry in Irradiated Aqueous Nitric Acid**

Bruce Mincher

An investigation of the radiation-induced reactions affecting the valence state of neptunium in irradiated nitric acid

NE160

The metal ion separation schemes used in nuclear fuel reprocessing rely on known and stable metal oxidation states to effect separations. Neptunium is problematic because it can exist as extractable Np(IV) and Np(VI), or inextractable Np(V) within the range of redox conditions encountered in the process. Often, all three oxidation states are simultaneously present, and this has thwarted attempts to perform efficient neptunium coextraction. We have initiated an investigation of the parameters affecting the valence state of neptunium in irradiated nitric acid, with special attention to radiation-induced reactions. The long-term goal is to understand these reactions well enough to predict neptunium valence under various conditions of acid concentration and absorbed radiation dose.

#### Project Summary

This is the first year of the project. Samples of neptunium in varying concentrations of nitric acid were irradiated, and the change in oxidation states was observed using UV/Vis spectroscopy.

Higher nitric acid concentrations favor Np(VI), which is reduced to Np(V) by irradiation. No Np(IV) has been measured to date. We are investigating the possibility that the radiolytic reducing agent is HNO<sub>2</sub>, produced by HNO<sub>3</sub> radiolysis. Nitrous acid yields at various dose rates have been measured. Our collaborators at Oregon State University and Cal State University Long Beach have independently measured the reaction rate constant between nitrous acid and hydrogen peroxide, also produced by irradiation. The results are not in agreement and we are currently working to resolve the discrepancy.

#### Benefits to DOE

The ability to reliably co-extract neptunium with uranium and plutonium, or to coextract neptunium and plutonium separately from uranium could lead to a simplified and proliferation-resistant advanced fuel cycle. The factors that determine the valence state of neptunium under process conditions must be known in order to control the valence state of neptunium and to achieve this separation.



## **Reversible Gas Phase Reactions for Recovery of Graphite from Recycled HTGR TRISO Fuel**

R. S. Herbst, J. D. Christian, M. R. Greenhalgh

Experimental evaluation of potential carbon (graphite) reactions with specific reactant gases

NE161

High-temperature gas reactor (HTGR) fuels will consist of graphite blocks or pebbles containing tri-structural isotropic (TRISO) fuel particles of uranium in graphite pebbles or rods. This project will examine recovering the carbon from graphite fuels, including radioactive <sup>14</sup>C, as an alternative and improvement to burning the carbon to CO<sub>2</sub> with subsequent environmental release. This carbon recovery step would form the basis for the head-end processing of used HTGR fuel to recover valuable uranium and other irradiation products. Reaction kinetics of several reactive gases with graphite will be determined experimentally to establish practicality. Each reactant gas is predicted to react with carbon to form carbon-containing gaseous compounds that can be reversibly dissociated back into carbon and reactant gas. The test results will be used to derive mass balance and throughput rates of these processes for treatment of the HTGR fuels.

#### Project Summary

The first year was a steep learning curve involving setup and operation of the thermal gravimetric analyzer (TGA), vacuum and gas-handling systems for the project. All of the issues have been resolved and the equipment, including the TGA and sulfur vapor generator, is installed and operational. All required materials have been procured. Finally, we have identified and obtained fuel compact and graphite materials used in different nuclear reactors and HTGR fuels to conduct the experimental program on actual materials of interest (along with TRISO particles). Specific applications have been evaluated, and the results will be used to focus

the following experimental efforts: reaction of carbonized fuel compact matrix material to recover the carbon and expose the fuel matrix for processing; removing the SiC layer of the TRISO particles to access to the UO<sub>2</sub> for processing; and a proprietary application and process with large potential utilization, defining a potential pathway for disposal of graphite as low-level waste (LLW) as well as recycle of selected materials for reuse.

We have determined that the dissociation of carbon disulfide ( $CS_2$ ), a reaction product of graphite with sulfur, will not reversibly decompose at a practical rate in the 275–400°C temperature range as predicted thermodynamically; the alternative approach will involve converting the  $CS_2$  to carbon via reaction with  $H_2$ . We will look at reactions and reaction rates of graphite and carbonized fuel matrix material in the presence of sulfur, chlorine, and fluorine gases (individually) as indicated in the alternative hypotheses in the original proposal. We anticipate that initial graphite and fuel matrix reaction data with S and  $F_2$  gases will be collected during the next fiscal year (FY-11).

#### Benefits to DOE

This year was related to equipment setup and experimental design. Benefits of the research will be espoused next year with the actual collection of experimental data. In the meantime, Idaho National Laboratory scientists have become more learned in graphite properties and reactivity, which will guide the selection of gas reactants and development of potential process flowsheets.

atory

## **Comprehensive Thermodynamic Models for Aqueous Partitioning of Actinides from Used Nuclear Fuel**

Peter Zalupski
Modeling of solvent extraction equilibria using SXFIT computational package

This research effort focuses on careful experimental considerations of non-ideal solution chemistry of aqueous electrolyte mixtures and organic solutions to assemble a library of thermodynamic parameters required to accurately reproduce the results of a solvent extraction experiment. Knowledge—i.e., approximation of the non-ideal behavior of all components in the aqueous and organic solutions—is an important prerequisite to the predictive modeling efforts performed by the computer program SXFIT. The program matches the collected metal distribution data with the extraction equilibria suggested by the user. A successful agreement between the experimental data and that calculated by SXFIT produces a model, which is used in further predictive modeling. The thermodynamic library constitutes of series of Pitzer ion interaction parameters (aqueous) and Hildebrand solubility parameters (organic) applicable to mixtures representative of separation chemistries under study. The aqueous solutions are investigated using isopiestic equilibration measurements. Vapor pressure osmometric (VPO) experiments are conducted to study aggregation phenomena and activity effects in the organic phase. Successful collection of these thermodynamic parameters will help develop predictive modeling capabilities for liquid-liquid distribution processes, which are being considered as viable options for aqueous reprocessing of used nuclear fuel.

Project Summary

This project's experimental efforts focused on collecting fundamental thermodynamic data required to produce descriptors of non-ideal behavior in solutions pertaining to the TALSPEAK process, a solvent extraction process developed to separate actinides from lanthanide elements. The An/Ln inter-group separation employs an aqueous soft donor aminopolycarboxylate complexing agent, diethylenetriamine-N,N,N',N,"N"-pentaacetic acid (DTPA) to preferentially hold the actinides in aqueous lactate buffer solution, while lanthanides are extracted into an organic phase by a powerful cation exchanger, di(2ethylhexyl)phosphoric acid (HDEHP). The design of the thermodynamic studies intended to

gradually introduce progressively higher levels of complexity into the investigated solutions. This approach allows quantifying the magnitude of non-ideal behavior expected when combining, i.e., mixing components into a final working solution. To illustrate, the aqueous phase of TALSPEAK could contain up to millimolar concentrations of lanthanides and uranium, trace amounts of other actinides, 0.05 M DTPA and 1.0 M of total lactate. As the mixture is adjusted to an approximate pH of 3.6, this solution contains a variety of aqueous ionic species, all interacting, all skewing the solution behavior away from ideality. The simplest aqueous electrolyte mixture of relevance to TALSPEAK is sodium lactate in water, which constituted an initial test bed for the thermodynamic studies in the aqueous solutions. The ionic interactions in aqueous solutions have been investigated using isopiestic equilibration measurements. Vapor pressure osmometric experiments were conducted to study non-ideal solution behavior in the non-aqueous environment. The organic solutions pertaining to TALSEPEAK may contain up to 0.5 M HDEHP, which, due to its ability to form hydrogen bonds, aggregates in non-polar diluents, such as toluene or ndodecane. Such aggregative equilibria, as well as the activity effects originating from solute-solute interactions in the organic phase, mainly contribute to the observed non-idealities.

The operation of the isopiestic equilibration chamber was validated using equilibration studies of sodium chloride reference standard solutions. Excellent agreement between all solution molalities was achieved. This precision is required for isopiestic equilibrations to produce viable thermodynamic quantities. The isopiestic equilibrations between solutions of sodium lactate (unknown) and sodium chloride (reference standard) produced a thermodynamic curve of osmotic coefficient that describes the changes in the activity of water and solute-solute interactions that contribute to growing non-ideal behavior in solution. The sodium lactate isopiestic run was completed after 4.5 months of continuing equilibrations and mass determinations as samples progressively become more concentrated due to water removal. The collected osmotic coefficient data was represented by sets of equations describing non-



ideal behavior of 1:1 electrolyte mixture using a non-linear least-squares regression fitting routine to afford the first set of Pitzer ion-interaction parameters. The smoothed osmotic coefficient curve for sodium lactate has been generated. Interestingly, the curves for lactate and triflate salts exhibit are the most similar, which agrees very well with previous calorimetric investigations by Zalupski et al. The next set of equilibrations will introduce an additional degree of complexity to the system with the analysis of a buffer mixture of lactate and lactic acid. The concept of a thermodynamic measure of pH requires careful consideration. To this end, a new galvanic cell without liquid junction will be constructed to utilize a prototype hydrogen electrode to perform electromotive

To initiate the vapor pressure osmometric measurements, the new instrument was tested using a series of standard solutions of solutes known to exclusively exist as monomers. Sucrose octaacetate was used for studies in toluene and tetracosane for studies in *n*-dodecane. The expected dimeric aggregation has been observed for HDEHP in aromatic diluent, where the slope of the experimental VPO response was half that observed for the monomeric sucrose octaacetate. The studies of HDEHP interactions in a non-aqueous aliphatic environment yielded evidence of significant deviations from the "ideal" dimeric response.

measurements of hydrogen activity in solution.

Curvature was observed in the experimental readout, where the VPO signal subsides at high concentrations of HDEHP. Such deviations indicate either the existence of higher HDEHP aggregates or the non-ideal behavior of HDEHP dimers in solutions. Further experiments will distinguish between those possibilities and derive the thermodynamic descriptors (Hildebrand solubility parameters) for HDEHP dimer in aromatic and aliphatic diluent.

#### Benefits to DOE

These research activities promoted Idaho National Laboratory's (INL's) status as a sponsor of new scientific endeavors, including a much more fundamental approach to studying separations within the nuclear fuel cycle umbrella. Our approach to theoretical reproduction of the experimental results is perhaps the most thorough and systematic study of chemical processes that govern separations of radionuclides in solution. Pacific Northwest National Laboratory scientists have expressed interest in collaborative studies. Their initiation of similar studies on a different separation scheme will further enhance the recognition of this undertaking. The research developments are in line with Objective 1 of INL's Strategic Plan for FY 2009 – FY 2018, contributing to advancements and INL's leadership in fuel-cycle research, development, and demonstration.

## **Advanced Burnup Measurement and Nuclear Forensics using ICP-MS Isotopics Analysis Techniques**

Jeff Giglio

Leveraging the capability of ICP-MS to quantify multiple fission products and actinides in a single scan to develop a system of linear burnup equations

The Burnup Monitor Multiple Linear Regression Method was applied to the highburnup light water reactor oxide fuel. Based on the neutronics assessment, four major isotopes were responsible for more than 98% of all fission events in this type of fuel, making it one of the more complex systems considered. For independent measurement-based determination of burnup in the oxide fuel, a system of linear equations with four unknowns had to be constructed and solved. Earlier work with simple fuels showed that increasing the number of fission products in the system resulted in a better convergence of the system to a right solution. For this reason more than 50 fission-product isotopes were measured by inductively coupled plasma mass spectrometry (ICP-MS) in each oxide fuel sample. However, the data had to be pre-filtered owing to the presence of either significant non-linear effects (such as capture) in some isotopes or other measurement-related problems.

Project Summary

A set of 24 fission product isotopes was selected for each fuel sample to create an over-determined system of 22 linear equations with four unknowns, which was solved by the linear least-squared method. Though the total number of fissions in each sample could be determined reasonably well (within 5% of theoretical), the split of fissions between four major isotopes was incorrect. This is potentially a result of the uncertainties in the evaluated nuclear data file (ENDF) fission yield data (which serve as model coefficients and essentially define the model), uncertainties in the measured number of atoms, and/or numerical problems associated with the matrix. In other words, due to various uncertainties, the spread in the number of fissions determined from each individual fission product monitor in the system is significant enough to prevent the system from converging to a right solution. Singular value decomposition (SVD) of the matrix revealed a presence of the relatively small, close-to-zero singular values (diagonal values of the stretcher matrix) that are usually characteristic of the nearly rank-deficient or ill-conditioned matrices. Rank-deficient matrices either do not have a solution or have infinite solutions. The condition

number of the 22 × 4 matrix, which is a ratio of the maximum and minimum singular values, was high but not exactly that of a singular matrix. A singular matrix has an infinitely high condition number and a zero determinant; as a result, it cannot be inverted. Matrices with a large condition number are typically identified as illconditioned, indicating that the problem might not be stable numerically because small perturbations in the matrix or the right-hand vector result in large perturbations of the solution. Besides, such ill-conditioned and rankdeficient matrices are also known to be particularly susceptible to roundoff errors in computation; fortunately, this problem is more typical for the large-size problems with many unknowns. It was demonstrated, however, that when the difference in the number of fissions determined from various fission products is very small (within 1%), the system can be successfully solved for the four unknown fission fractions in oxide fuel.

In order to obtain a meaningful solution, nearly rank-deficient or badly conditioned problems typically benefit from regularization. Regularization, in general, involves introduction of additional information, such as supplementary data, restrictions for smoothness of the function, or bounds on the vector space norm. Such information helps to further constrain the system and make it a robust full rank. The condition number of the matrix is lowered and singular values increase as a result of regularization. One of the simplest forms of regularization is the linear least squares; however, this method is known to suffer from the outliers. Future efforts can be dedicated to either further increase the number of the matrix equations (number of measured fission-product isotopes) or filtering the outliers in an attempt to lower the condition number of the matrix and increase singular values.

Analysis of the measured fission-product data in the Reduced Enrichment Research and Test Reactor highly enriched uranium (>58% U235) U-Mo fuels plates has demonstrated that for 25 fission-product isotopes, thermal U235 fission yields were within 10% from the ENDF/B-VII values.



### Benefits to DOE

The work developed in the laboratory-directed research and development project has significant applications to national security missions. The generation of advance measurement techniques and the development of reactor characteristics from the data could be used to characterize origin of fuels or reactors used to irradiate the fuel.



#### **Advanced Ceramic Nuclear Fuels**

Dr. Paul A. Lessing, Dr. Pavel Medvedev, Prof. W.R. Cannon

Developing processing knowledge to fabricate advanced ceramic nuclear fuel forms that will withstand the rigors of long-time service in fast-neutron reactors

NU101

The overall goal of this project was to develop processing knowledge in order to fabricate advanced ceramic nuclear fuel forms (e.g., pellets or rods) that will withstand the rigors of long-time service in fast-neutron reactors. To support this goal, it was necessary to build expertise and new facilities at Idaho National Laboratory (INL) in the area of processing advanced ceramic nuclear fuels. A new, complete, functioning powder and granule processing laboratory was established. With the aid of laboratory-directed research and development (LDRD) and Indirect General Purpose Capital Equipment (IGPCE) Program funding, the total value of equipment residing in the processing lab is now quite significant. In this lab, rounded, "flowable" granules were produced that were used to press excellent "green" pellets. Research was conducted on several organic polymers for use as binders and pore-forming agents. This included green-strength and radiation-damage resistance measurements. Ultimately, this project produced TRU-MOX pellets in the confines of the Advanced Fuel Cycle Initiative (AFCI) glove box located at the Materials and Fuels Complex (MFC)/Fuel Manufacturing Facility (FMF) and developed a proto-type stoichiometry control system (SCS) for controlling oxygen partial pressure in sintering furnaces. This system will allow the future fabrication of TRU-MOX fuel pellets with well-controlled oxygen-to-metal (O/M) ratios. The O/M ratio is an important property for high-performance fuel whose control is necessary to prevent excess chemical reactions with fuel cladding. This project also supported INL's Funding Opportunity Announcement (FOA) project on spark plasma sintering by providing a series of "reference" pellets fabricated of many surrogate compositions using conventional "die press and sinter" technology.

Project Summary

The focus during Fiscal Year 2010 was to further develop advanced processing knowledge of the surrogate material cerium oxide (CeO<sub>2</sub>) and try to extend this knowledge to depleted uranium oxide (DUO<sub>2</sub>). During the course of the year the processing laboratory, consisting of special "lab-scale" experimental processing and

granulation equipment, was moved from MFC Lab 789 to the Materials Lab at the Center for Advanced Energy Studies (CAES). An exhaustive effort was necessary to move all equipment into CAES, including the newly obtained dry bag isostatic press that allows INL to fabricate a variety of homogeneous fuel shapes. Permission was gained at CAES to continue work on CeO<sub>2</sub>; however, due to a change in direction by management, we were not able to establish the capability to work with DUO<sub>2</sub> in CAES.

Two ground-breaking journal articles were written and a U.S. Patent application submitted documenting the technical progress of this project:

- "Dry Bag Isostatic Pressing for Improved Green Strength of Surrogate Nuclear Fuel Pellets," G. W. Egeland, W. R. Cannon, L. D. Zuck, P. A. Lessing, and P. Medvedev, Journal of Nuclear Materials, Online publication complete: 20-SEP-2010, DOI .2010.08.022.
- "Crack Model for Toughness of Green Parts with Moisture or a Fluid Binder," W. R. Cannon, P. A. Lessing and L. D. Zuck, which will be submitted to the *Journal of American Ceramic Society* in the near future

A U.S. Patent application (IDR BA-367, "Methods of Forming Nuclear Fuel Pellets, etc." P. A. Lessing) was submitted, gaining protection for our special techniques to isostatically press and shape homogeneous "green" pellets. Using these special techniques, CeO<sub>2</sub> powder was granulated, consolidated into a rod using the dry bag isostatic press, and sintered into a dense ceramic. Ceria rods, produced at CAES by dry bag isotatic pressing and the final sintered rodpieces, are shown in Figures 1 and 2 (next page). These rods were the very first produced at INL using the recently acquired dry bag isostatic press. The surface texture of the rods can be made smoother by inputting smaller, rounded granules, accomplished by using a recently purchased Caleva brand "Spheronizer." Rounding the granules will also improve the variations in rod diameter by improving the granule packing. Figure 3 (next page) shows



actual TRU-MOX pellets of proper dimensions that were pressed in a metal die and sintered in the AFCI glove box.



FIGURE 1. Dry Bag Isostatically pressed rods comprised of CeO<sub>2</sub>



FIGURE 2. Densified  $CeO_2$  rod pieces after sintering in a high temperature furnace.

The Journal of Nuclear Materials paper details the project's development of green pressing with organic binders. Dry bag isostatic pressing was proposed for mass production of nuclear fuel pellets. Dry bag isostatically pressed rods of a fuel surrogate (95% CeO<sub>2</sub>-5% HfO<sub>2</sub>) 200-mm long by 8 mm in diameter were cut into pellets using a wire saw. Four different binders and CeO<sub>2</sub> powder obtained from two different

sources were investigated. The strength of the isostatically pressed pellets for all binder systems, as measured by diametral testing (Fig. 4), was about 50% higher than pellets produced by uni-axial dry pressing at the same pressure (Fig. 5). It was proposed that the less-uniform density of uni-axially pressed pellets accounted for the lower strength. The strength of pellets containing CeO<sub>2</sub> powder with significantly higher moisture content was five times higher than pellets containing CeO<sub>2</sub> powder with a low moisture content even though they were 25% less dense. Capillary pressure of the moisture was thought to supply the added binding strength.



FIGURE 3. TRU-MOX pellets fabricated in the AFCI glove

The experimental results related to moisture content lead to the development of the Journal of the American Ceramic Society paper. In this paper, a theoretical model was developed to predict the toughness, K<sub>IC</sub>, of green ceramics based on the strength of meniscuses formed between particles near the tip of the critical flaw. The model considers capillary pressure, surface tension and the viscous flow of binder. Calculations were performed for moisture only, but the model can also be applied to binder meniscuses. Capillary pressure is highest at low moisture content. By using well-established force distance relationships for the meniscus between single particles, toughness was determined as a function of moisture content. K<sub>IC</sub> increases with moisture content and decreases with contact angle, and no particle size dependence was found. However, if particles are agglomerated and the meniscus is between agglomerated particles, the toughness is







FIGURE 4. Diametral strength testing of a green pellet.

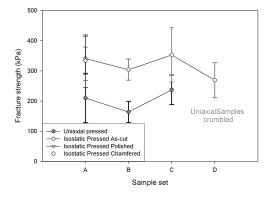


FIGURE 5. Fracture strengths of isostatically pressed pellets are higher than for die-pressed pellets.

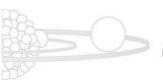
particle-size dependent and reaches a maximum at low moisture content. This model assumes that the capillary pressure is transmitted throughout the green part. The calculated values of  $K_{\rm IC}$  agrees approximately with the measured toughness of binderless green parts' two different specific surface areas (Table 1).

TABLE 1. Predicted and experimental results for pressed rods from high and moderate specific surface area powders.

|          | Water<br>Content | ESD* | K <sub>IC</sub> Predicted | K <sub>IC</sub> Measured |
|----------|------------------|------|---------------------------|--------------------------|
| Powder   | Volume%          | □m   | (MPam <sup>1/2</sup> )    | (MPam <sup>1/2</sup> )   |
| Inframat | 14.5             | ~ _  | 0.016                     | 0.049                    |
| TJTM     | 1.6              | ~ _  | 0.006                     | 0.013                    |

#### Benefits to DOE

During the course of this project, excellent progress has been made in fabricating ceramic fuel pellets, building advanced capabilities at INL, and developing basic knowledge necessary for successful high-performance ceramic fuel fabrication. Work has resulted in the submission of a U.S. Patent application (to protect commercial applications) and the completion of two papers for two journals (one accepted). The work has focused on developing methods and materials to control the density (porosity) and homogeneity necessary to enable ceramic fuels (TRU-MOX) to be used in a high neutron flux reactor (e.g., Advanced Recycling Reactor) where the fuel is to be irradiated to high burn-up (beyond 100 GWd/t). These reactors transmute trans-uranic elements (TRU) recycled from light water reactors (LWRs) to produce energy while reducing residual long-term radiotoxicity and decay heat for storage in a spent-fuel repository. The TRU material (Am and Np oxides) that we have added to UO2 and PuO2 ceramics are characteristic of re-cycled LWR fuel feedstocks being developed by the AFCI. Variations on these new fabrication techniques will also prove innovative and valuable to existing LWRs that use ceramic fuel pellets, including MOX and IMF for LWRs. A commercial fuel manufacturer has expressed great interest in the results of this project. This interest will increase as we begin to use our new techniques to process UO<sub>2</sub> (depleted) powder.



### **Spatially Correlated Microstructural Characterization: From Centimeters to Nanometers**

Dawn Janney

Developing a methodology to facilitate collection of chemical, structural, and microstructural data from the same locations on highly radioactive samples

NE163

The project's purpose is to develop a methodology that would facilitate using a suite of instruments to collect chemical, structural, and microstructural data from the same locations on highly radioactive samples. This will allow us to collect data on a continuum of spatial scales extending across seven orders of magnitude (from centimeters to nanometers). Although many characterization instruments are currently available at Idaho National Laboratory (INL), each instrument is typically used independently, with no method available to ensure that different instruments analyze the same areas or to easily correlate data between different instruments. The project includes developing both a methodology for locating the sample's desired area in each instrument and the data mining/fusion software to enable information acquired to be correlated with other instruments.

#### Project Summary

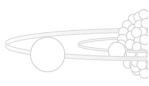
- a. Coordinate transformations and software were developed that allow us to find the same location in a sample repeatedly based on "fiducials" (special markers in each sample holder whose locations can be easily measured), even when the sample has been transferred to a different instrument.
- b. The software was tested by finding the same locations using the Bruker D8 Discover X-ray diffractometer in microdiffraction mode and several scanning electron microscopes.
- c. A database was designed and developed to I allow us to find all the information from a specific sample and area, no matter which instrument it was collected with.
- d. Optics in LOCI (a Fourier transform mass spectrometer) were modified and are techniques are currently being testted to find fiducials and sample locations based on these fiducials.

- e. A variety of samples, were manufactured, including two containing actual nuclear materials (depleted uranium and zirconium), and mounted these samples in holders with fiducials.
- f. We are currently identifying instruments at MFC, IRC, and CAES that might be incorporated into the LDRD in future years, if funding continues. We have started discussions with principal investigators and subject-matter experts associated with some of these instruments.
- g. A poster abstract on our results was submitted to the TMS Annual Meeting, which was accepted.

#### Benefits to DOE

The Department of Energy (DOE) is tasked with securing competitive and environmentally responsible nuclear technology to meet the energy needs of the U.S. A key for advancing nuclear technology is the ability to design and fabricate nuclear fuels and related materials, which requires an understanding of the phases and microstructures in nuclear materials, including fresh and irradiated fuels. Developing this understanding requires using a variety of instruments to collect chemical, structural, and microstructural data from the same locations on highly radioactive materials. This project involves developing a methodology for locating the sample's desired area in a variety of instruments and correlating the data for each area collected. It will facilitate comprehensive characterization of nuclear materials and fuels for acquisition of new knowledge and understanding, contributing to DOE's ability to carry out its mission.

Idaho National Laboratory



1

## Viability Evaluations of Linear Variable Differential Transformers (LVDTs) for In-Pile Instrumentation

Joy Rempe

**Development of a high-temperature sensor for detecting dimensional changes during irradiation testing** NE150

New materials are being considered for fuel, cladding, and structures in next-generation and existing nuclear reactors. Such materials can experience significant dimensional and physical changes during irradiation. Currently, such changes are measured at the Advanced Test Reactor (ATR) by repeatedly irradiating a specimen for a specified period of time and then removing it from the reactor for evaluation. The labor and time to remove, examine, and return irradiated samples for each measurement makes this approach very expensive. In addition, such techniques provide limited data, and handling may disturb the phenomena of interest.

This three-year project evaluated the viability of linear variable differential transformers (LVDTs) as in-pile real-time sensors for detecting dimensional changes in specimens during high-temperature (up to 500°C) ATR irradiation tests. As shown in Figure 1, LVDTs are electrical transformers with three coils placed end-to-end around a tube. A magnetically permeable cylindrical core, attached to the object whose position is to be measured, slides along the axis of the tube. Previous evaluations have shown that these sensors are robust, frictionless instruments for detecting dimensional changes in lower-temperature, non-irradiation environments. Evaluations completed in this project were designed to develop a much-needed high-temperature sensor for detecting dimensional changes during irradiation testing at

the ATR and other materials and test reactors. Hence, project evaluations offer the potential for a less expensive, more accurate in-pile sensor for detecting geometry changes during irradiation.

#### Technical Progress

Nuclear-grade LVDTs from the U.S. and foreign sources were evaluated as candidates for in-pile deployment. Initial efforts, which included calibration evaluations and long-duration, high-temperature testing, clearly indicated the superiority of LVDTs supplied by the Institute for Energy at the Halden Reactor Project (IFE/HRP). However, Curie point effects, due to the nickel contained in the LVDT coil material, were identified as a potential weakness (under certain operating conditions) in the IFE/HRP design. For that reason, INL has worked with IFE/HRP to develop and evaluate developmental LVDTs with an alternate coil material that is not susceptible to the Curie effect. Calibration and long-term, high-temperature testing of the developmental LVDTs performed by INL demonstrate that the new LVDTs can operate in a very stable manner for long periods (1000 h) at high temperatures (500°C). As shown in Figure 2 (next page), the degradation of the original LVDTs provided by IFE/HRP and by another nuclear-grade LVDT manufacturer was not observed in the developmental LVDTs provided by IFE/HRP. Hence, the developmental LVDTs are recommended for use in ATR high-temperature irradiation tests.

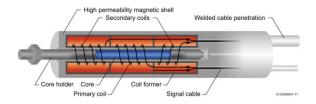
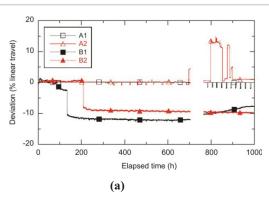




FIGURE 1. Schematic and photo of typical LVDT.



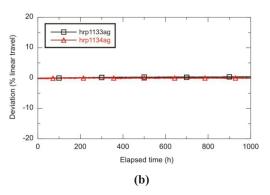


FIGURE 2. LVDT response during long duration (1000-hour) test at 500°C: (a) LVDTs originally provided by nuclear-grade vendors (LVDTs designated with an "A" were provided by IFE/HRP); (b) developmental LVDTs provided IFE/HRP.

#### Benefits to DOE

Results from this project have produced an LVDT design suitable for ATR irradiation tests. Hence, project results increase ATR instrumentation capabilities, which support DOE's energy security mission by developing and demonstrating much-needed sensors to enhance the accuracy of data obtained from, and reduce the costs of, irradiations in materials and test reactors such as the ATR. In addition, project results provide a much-needed capability for obtaining vital data required for key

Department of Energy-Office of Nuclear Energy research programs, such as the Generation IV and Fuel Cycle Research and Development programs, and enhance testing capabilities for the ATR National Scientific User Facility. Finally, recognition (e.g., publications, patents, and awards) from this research promotes INL's instrumentation capabilities, furthering INL efforts to develop the Instrumentation, Control, and Intelligent Systems Distinctive Signature.

### **Instrumentation for In-pile Detection of Thermal Conductivity**

Darrell Knudson, Joy Rempe

Exploring the benefits, limitations, and accuracy of two candidate in-pile thermal conductivity measurement approaches

NE158

Currently, changes in thermal conductivity of fuel or materials irradiated in the Advanced Test Reactor (ATR) are evaluated out-of-pile, where properties of materials are measured after samples have been irradiated for a specified length of time. The labor and time to remove, examine, and return irradiated samples for each measurement makes out-of-pile approaches very expensive. In addition, data may not be obtained under prototypical conditions because they only capture the sample's endstate after it is removed from the reactor, and removal and reinsertion into the reactor may disturb the phenomena of interest.

#### Project Summary

In this three-year project, Idaho National Laboratory (INL) and Utah State University (USU), with assistance from the Institute for Energy Technology at the Halden Reactor Project (IFE/HRP), are exploring the benefits, limitations, and accuracy of two candidate in-pile thermal conductivity measurement approaches: a multiple thermocouple steady-state approach and

a transient hot wire method (THWM) with the ultimate objective of providing ATR users a method for detecting changes in fuel or material thermal conductivity during irradiation.

#### Benefits to DOE

During Fiscal Year (FY) 2009 (the first year of this three-year project), efforts focused on evaluating the two-thermocouple approach. Results from this effort show that accuracies similar to those obtained with out-of-pile techniques could be achieved. During FY 2010, investigations primarily focused on the THWM approach because of the increased accuracies possible with this technique. The THWM, or line heat source method, is applied by embedding a line heat source in the material whose thermal conductivity is to be measured (Fig. 1). From a condition of thermal equilibrium, the heat source is energized and heats the sample with constant power. The temperature response of the sample is a function of its thermal properties. Following a brief transient period, a plot of the temperature versus the natural logarithm of time becomes

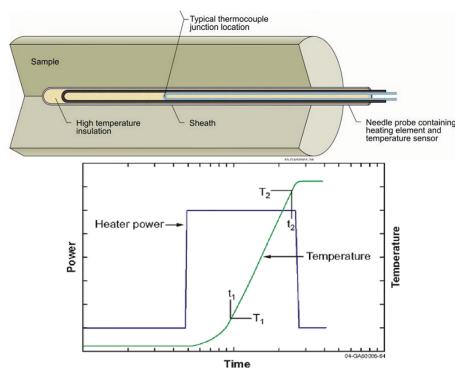


FIGURE 1. Hot wire needle probe design and thermal response of material with an embedded needle probe.





linear, and the thermal conductivity for the sample can be calculated from a relationship between (1) the power dissipated by the heater wire, (2) the thermocouple temperature at the time when the linear portion of the curve started,  $T_1$ , (3) the temperature when the linear portion of the response curve ended,  $T_2$ , and (4) the time period,  $t_2$ - $t_1$ . In this project, a new hot wire probe design containing a resistance heater and a thermocouple embedded in a single probe was developed. Testing has been performed with prototype needle probes on samples with thermal conductivities ranging from 0.2 W/m-K to 16 W/m-K in temperatures ranging from 20°C to 600°C. Thermal conductivity values measured using the needle probe match data found in the literature to within 5% for samples tested at room temperature, 6% for low thermal conductivity samples tested at high temperatures, and 12% for high thermal conductivity samples tested at high temperatures. Efforts are underway to obtain even higher accuracies by implementing improved signal processing capability. Experimental results also show that this sensor is capable of operating in various test conditions and of surviving long-duration irradiations. Efforts are also underway to prepare a prototype needle probe for an upcoming irradiation of hydride fuel in the Massachusetts Institute of Technology Research Reactor.

#### Results and Accomplishments

Results from this project increase INL instrumentation capabilities, which support the Department of Energy's energy security mission by developing and demonstrating high-temperature in-pile instrumentation needed to obtain data required for key Department of Energy-Office of Nuclear Energy research programs, such as the Generation IV, Fuel Cycle Research and Development (FCRD), and ATR National Scientific User Facility (NSUF) programs. Specifically, this research is enhancing INL's expertise for producing optimized, more reliable, and lower cost in-pile instrumentation. In addition, enhanced instrumentation from this research can increase testing capabilities of INL's ATR NSUF. Finally, recognition (e.g., publications, patents, and awards) from this research promotes INL's instrumentation capabilities, furthering INL efforts to develop the Instrumentation, Control, and Intelligent Systems Distinctive Signature.



### **Human Performance Assessment for Technology Neutral Evaluation**

David Gertman, Jeffrey Joe, Alan Mecham, William Phoenix Combining virtual and physical testing for design, development, and review of digital control systems and interfaces

NU100

Future control room design for nuclear facilities will incorporate advanced digital technologies, automation, and intelligent systems. Driven by these advances, the concepts of nuclear power plant (NPP) operations may be radically different for light water reactor (LWR) operation of nuclear power plants and at all levels of the fuel cycle. Design advances are necessary to meet complex control requirements, reduce staffing and maintenance costs, and reduce the burden of security costs. Prior to any change, however, research is needed to evaluate the impact of these new technologies and operational concepts for their effect on human performance and, ultimately, plant safety. This includes operator and crew performance in the presence of advanced instrumentation and control (I&C) systems for abnormal and emergency operations.

The driving research issue at hand that this laboratory-directed research and development (LDRD) project addresses is as follows:

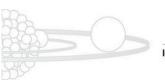
At this time, there does not seem to be an agreed-upon, effective methodology for designers, owner-operators, maintainers, and regulators to assess the overall impact of computer-based, human-machine interfaces on human performance in nuclear power plants. What methodology and approach should be used to assure proper consideration of human factors and human-machine interfaces? (National Research Council 1997).

The proposed research will develop the much needed methodology: human cognition models, human performance metrics, and laboratory infrastructure to support the design and evaluation of advanced digital-control methods and interfaces. The goal of this research is to establish a sound technical basis for advanced control room technology selection and deployment, regulatory approval, system integration, staffing levels, and meeting safety requirements.

Project Summary

The objective of this research is to integrate NPP simulation with human-in-the-loop performance testing in order to develop capabilities and methods that establish a sound technical basis for new control room technology selection, regulatory approval, and system integration decisions. This is being accomplished by developing an iterative methodology that utilizes various conceptual tools and methods from which baseline human- and equipment-performance measures in a technology-neutral framework can be derived. Specific objectives of this project include the following milestones:

- Design INL human system simulation laboratory to allow for collection of human in-the-loop performance testing
- Construct/configure laboratory and integrated advanced equipment for conducting physiological recording of control room response
- Train INL scientific and engineering staff in data collection for physical recording equipment, including vision, heart rate, and event logging
- Identify candidate NPP simulations, or surrogate simulations, for implementation in the human system simulation laboratory
- Develop three-dimensional visualization/programming capability
- Identify human cognitive simulations and integrating architecture
- Establish working relationship with National Aeronautics and Space Administration (NASA) Ames Research Center
- Obtain NASA man-machine interaction design and analysis system (MIDAS) software
- Establish university partners and conduct research studies evaluating human performance in advanced instrumentation and control environments expected for NPPs as a function of light water reactor sustainability

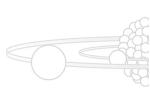


 Present findings at peer-reviewed national and international conferences.

#### Benefits to DOE

We expect that once simulations have been implemented, the laboratory will provide a needed resource for human performance assessment in the presence of advanced instrumentation and control for DOE and Nuclear Regulatory Commission regulators.

Because no national simulation capability for crew performance in advanced control rooms exists, INL will be uniquely poised to offer research and analysis and data collection capabilities. Although no immediate intellectual property has been identified, INL is also positioned to benefit from intellectual property related to methods, models, and data coming available from this research project.



## **Ultrasonic Transducer Sensors for In-Pile Detection of Dimensional Changes**

Steven C. Taylor

Investigating the feasibility of using ultrasonic transducers as in-pile sensors NE164

At Idaho National Laboratory's (INL's) Advanced Test Reactor (ATR), deformation of specimens during irradiation is currently evaluated out-of-pile, where the properties of materials are measured after the specimens have been irradiated for a specified length of time. The labor and time to remove, examine, and return irradiated specimens for each measurement makes the current out-of-pile approach very expensive. In addition, the out-ofpile measurements only capture the specimen's end state after it is removed from the reactor. Therefore, data are not obtained under prototypic conditions, and the removal and examination process may disturb the phenomena of interest. Although linear variable differential transducers (LVDTs) have been successfully deployed at other test reactors and are being investigated for ATR applications, ultrasonic transducers offer the potential for a more compact, higher temperature, more accurate, and multidimensional real-time sensor for detecting geometry changes of creep specimens during irradiation. This three-year project is investigating the feasibility of using ultrasonic transducers as in-pile sensors for detecting geometry changes in creep specimens by completing laboratory demonstrations (including bench top, furnace, and autoclave tests). Experimental results are also compared to theoretical predictions to further validate measurement capabilities.

#### Project Summary

During Fiscal Year 2010 (FY-10), efforts primarily focused on optimizing components required for using an in-pile UT sensor method and completing laboratory demonstrations. Efforts were initiated to identify and optimize key components of a candidate in-pile setup (Fig. 1) that included a magnetostrictive transducer (e.g., driver coil), a Remendur guide, a coupling between the Remendur guide and a long stainless steel wave guide that allows the transducer to be located outside of the reactor, and a creep specimen with an "acoustic horn" to optimize the signal. A coupling method was developed that allows the magnetostrictive transducer to be directly coupled to a 30-foot-long stainless-steel waveguide with minimal loss in signal. Welding methods were

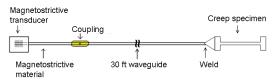


FIGURE 1. Key components required for an in-pile UT demonstration.

tested to determine a suitable way to attach the 30-foot-long waveguide to the creep specimens. Results indicate that butt-welding provides both good mechanical strength and minimal loss in the ultrasonic signal. The electronics for performing the measurements were tested to optimize measurement variables. Computer programs were written to perform data collection and increase system automation.

High-temperature testing was also initiated at INL's High Temperature Test Laboratory. Initial results appear to agree with finite element predictions. A design was developed for testing this setup in an autoclave, and key system components have been ordered so that autoclave evaluations can begin in FY-11.

A tube furnace at INL's High Temperature Test Laboratory was used to perform preliminary time-of-flight (TOF) measurements on two stainless -steel creep specimens at 525°F (302°C). Figure 2 compares the recorded waveforms from the 4-inch and 6-inch-long specimens. The data indicate a 58.4-µs TOF for the 4-inch gage length specimen and a 78.72-µs TOF for the 6-inch gage length specimen. Thus,

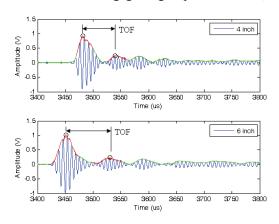


FIGURE 2. Comparison of waveforms from 4- and 6-inch specimens heated at 525°F (302°C).

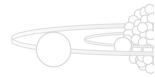


the ultrasonic velocity in the gage length of the specimen is approximately 5 mm/µs. This value is consistent with guided wave theory predictions for the cylindrical gage section at 525°F. In addition, the data demonstrates that the buttwelding of the specimen to the waveguide allows sufficient transmission of the ultrasonic signal.

#### Benefits to DOE

Results from this project can increase INL instrumentation capabilities, which support the Department of Energy's energy security mission by developing and demonstrating high-temperature in-pile sensors needed to obtain key data required for Department of Energy-

Office of Nuclear Energy research programs, such as the Generation IV, Fuel Cycle Research and Development (FCRD), and ATR National Scientific User Facility (NSUF) programs. Specifically, this research is enhancing INL's expertise for producing optimized, more reliable, and more compact in-pile instrumentation. In addition, enhanced instrumentation from this research can increase testing capabilities of INL's ATR NSUF. Finally, recognition (e.g., publications, patents, and awards) from this research promotes INL's instrumentation capabilities, furthering INL efforts to develop the Instrumentation, Control, and Intelligent Systems Distinctive Signature.



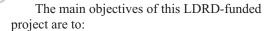
**Idaho National Laboratory** 

52

### **Smart Grid Impact on Commercial Nuclear Plants**

Magdy Tawfik

Studying the impacts of additional renewable power generation units on Nuclear Power Plants (NPP) active components and life-extension related issues



- (a) Examine key NPP active components that may be subjected to sudden electrical power grid perturbations and assess the impact on component health.
- (b) Consider the impact on the different modes of operation to be identified and studied in detail in the next year.
- (c) Identify existence or lack of regulatory material in existing nuclear regulations addressing the negative impacts (in particular) of external power perturbations on NPPs by:
  - 1. Considering the direct impacts on components.
  - 2. Considering the impacts on human factors in response to external events.
  - 3. Considering the possible effects on the plant's operation.

#### Project Summary

The impacts of the electric power grid on NPPs have been defined during this first fiscal year as follows:

- (a) The U.S. NPPs serve as base loading generation and are not suited to provide load following capabilities.
- (b) Auxiliary system loads (i.e., pumps, valves, etc.) are sensitive to power quality events, similar to many pieces of industrial equipment.
- (c) The U.S. NPPs 1E systems are designed to react to various electrical system events and respond by placing the plant into a safe shutdown mode.
  - 1. Examples of this shutdown include the momentary loss of external power and the momentary drop in external supply voltage.

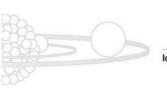
- (d) Primary electrical equipment in the NPPs (i.e., generator step-up unit transformer) was damaged due to external faults and transient events on the power system.
- (e) The damage modes and life-cycle characteristics of generator step-up unit transformers have been investigated.
- (f) The modeling of cascading power system failures and events that can impact the 1E safety systems have been defined:
  - 1. An analysis method for modeling vulnerabilities of external electrical supplies by insignificant/remote substation failures has been developed.

#### Benefits to DOE

Took the following technical steps to:

- (a) Establish the foundation of defining the limits of the smart grid installing equipment as a percentage of the overall generated capacity within a region.
- (b) Identify possible vulnerabilities to the installed NPP generation capacity from nonregulated and unlimited smart grid components.
- (c) Show that cascading events in the electrical grid can be a reality, given the combination of proposed smart grid equipment to be installed under the inherited grid conditions.
- (d) Show DOE's active involvement and interest in the development of the healthy energy policy for the nation.

This work can be the foundation for the application's usefulness to other organizations (i.e., NASA and a number of government organizations).



## **Characterization of Fluidized Beds via Pressure-Fluctuation Analysis**

Doug Marshall

Establishing a fluidization lab at CAES to study fluidization of solid particles in a gas stream PH103

Two 2-D fluidized bed models have been made with replaceable gas injector nozzles and side profiles to enable exploration of a wider variety of fluid bed configurations.

Facility modifications have enabled the use of compressed air, cooling water, and water discharge in addition to electrical power for a high-temperature furnace.

The high-temperature furnace has been relocated and the data collection system restored to enable data collection.

#### Project Summary

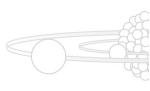
The objective is to establish a fluidization lab at the Center for Advanced Energy Studies (CAES) to study fluidization of solid particles in a gas stream in order to:

- a. Correlate fluidization patterns with pressure fluctuation patterns and structures as measured at the gas inlet
- Investigate the effect of high temperature on fluidization behavior and extend existing science up to 1500°C (about 700°C hotter than previously reported)

c. Modify existing empirical correlations according to high temperature data

#### Benefits to DOE

The project is poised to provide data that will be useful in designing fluidized bed systems for the production of coated-particulate nuclear fuels for very high temperature reactors (VHTRs) and space propulsion systems. Comparing computer model output with the physical model's behavior will expose the strengths and weaknesses of the computer models that may be used to understand fuel coater processes, which cannot be directly observed. No meaningful data have been collected during the configuration phase of the project. Data collection is expected to start by the end of October 2010.



54

## Extraction of Fission Products in Room Temperature Ionic Liquids and sc-CO<sub>2</sub> with Study of Radiolysis

Brian Harris

Developing alternative methodologies for EM clean-up and advancing a cleaner nuclear fuel cycle PH104

The proposed research aims to study the properties of a room-temperature ionic liquid (RTIL) extraction and sc-CO2 back extraction system for radionuclides—primarily actinides and lanthanides—in several matrices with the ultimate goal of developing alternative methodologies for EM clean-up and advancing a cleaner nuclear fuel cycle. The two primary goals are to (1) optimize the extraction efficiencies for separating lanthanides and actinides in two well-characterized soils by investigating the parameters of temperature, pressure, extraction time, and the addition of solvent modifiers and/or ligands, and (2) determine the radiolytic stability of the ionic liquid and the radiation chemical yields of identified products.

#### Project Summary

In Fiscal Year 2009 (FY-09), work was performed to assess the parameters necessary for the preparation of the tributyl phosphate (TBP):nitric acid complex TBP(HNO<sub>3</sub>)<sub>1.8</sub>(H<sub>2</sub>O)<sub>0.6</sub> by evaluating the concentration of "super acid" in the ionic liquid, looking at preliminary liquid extractions of lanthanides and actinides from various matrices, and assembling the sc-CO<sub>2</sub> apparatus. This work was continued into FY-10; we also worked on determining the parameters required to optimize the conditions necessary for the sc-CO<sub>2</sub> back extraction and extended to the conditions necessary to strip nuclides from the RTILs for analysis and possible RTIL recycling.

Extractions on spiked and un-spiked Radioactive Waste Management Complex (RWMC) lake-bed soil show that twenty six nuclides, including Pu and natural U isotopes, are extracted from the soils directly into the ionic liquids using the TBP(HNO<sub>3</sub>)<sub>1.8</sub>( $H_2O$ )<sub>0.6</sub>complex. The low extraction efficiency for many of the elements may be due, in part, to the fact that these were only single contact with small volumes of the RTIL containing the TBP(HNO<sub>3</sub>)<sub>x</sub>(H<sub>2</sub>O)<sub>v</sub> complex. Initial sc-CO<sub>2</sub> back extractions at 3000 psi and 40°C from the extracted Rocky Flats (NIST SRM 4353B) soil show that for light rare earth elements (REEs) La, Ce, Pr, Ce, Nd, and Sm, ~18% can be back extracted into the sc-CO<sub>2</sub> from the RTIL per cell volume of sc-CO<sub>2</sub> and collected. For actinides Th and U, ~14% can be similarly recovered.

Although an effective HPLC method has not been fully developed to assess degradation products of the RTIL, initial water strips on these samples were analyzed on an ion chromatograph (IC). Electrospray ionization mass spectrometry (ESI-MS) analysis is being evaluated to identify species from the radiolysis of the RTIL and to elucidate any differences between the RTILs produced in the lab at the University of Idaho (U of I) and those obtained commercially from Sigma-Aldrich. The initial sc-CO<sub>2</sub> apparatus has been modified and improved to allow for finer monitoring of reaction conditions and control of dynamic extractions without significantly increasing sc-CO<sub>2</sub> volume.

This spring a research plan supporting a Ph.D. candidate was approved through a presentation and visit to U of I. The subcontract for U of I providing purified RTILs was also continued.

#### Benefits to DOE

In Nuclear Science and Technology, our initial radiolysis data contributes to a sciencebased approach to better understanding the behavior of a possible ionic liquid-based extraction scheme to improve on existing separation technologies, as well as providing alternate approaches to simplify or provide more efficient or cost-effective processes applicable to the nuclear fuel cycle, waste cleanup, etc. In the Energy and Environment S&T Directorate, we have gained knowledge of the behavior of our ionic liquids in different matrices (soil, ores, and aqueous) and various conditions that will aid in understanding their potential application to understanding and dealing with environmental issues. The improved sc-CO<sub>2</sub> apparatus is a distinct resource in investigating the dissolution and extraction of radioactive metals in various matrices under controlled conditions. We continued our relationship with U of I through a subcontract in supplying purified RTILs.



### **Integrated Mesoscale Approach for the Simulation of Nuclear-Fuel Behavior**

Dieter Wolf

Develop an integrated microstructure-based, mesoscopic modeling and simulation capability

The main goal of this work is to develop an integrated microstructure-based, mesoscopic modeling and simulation capability for nuclear fuels and structural materials that incorporates the critical role of microstructure and its evolution under irradiation, as well as stress and temperature effects. The integrated code will combine two main building blocks within a single computational platform: (1) an atomistically informed phase-field modeling and simulation approach, and (2) a finite-element approach for the stress distribution within the evolving polycrystalline microstructure. These stresses will be used as input into the phase-field simulation approach. The combined simulation capability will enable materials-physics-based prediction of the thermo-mechanical behavior of polycrystalline fuel and cladding materials under the effects of irradiation, including the complex interplay between fission-gas swelling and release, high-temperature creep, and grain growth.

#### Project Summary

Traditionally, the prediction of swelling and fission-gas release behavior in both metallic and oxide fuel has been based on the classical rateequation approach that incorporates assumed key atomic mechanisms into an analytic framework. By their nature, such mean-field approaches ignore microstructural heterogeneity (e.g., distributions in the grain sizes, grain shapes, and anisotropic grain-boundary properties). Nevertheless, they have been quite successful as evidenced by the existence of well-known software packages that capture the nucleation and growth of inter- and intra-granular bubbles under the simultaneous destructive influence of irradiation-induced gas-atom re-solution. However, uncertain—yet critically important materials parameters of the model are usually adjusted by fitting to empirical data, rather than using a more rigorous materials-physics-based simulation approach. These uncertainties raise questions about the validity of the physics invoked to explain the observations.

The present effort aims to develop an integrated mesoscopic modeling capability that explicitly incorporates the critical role of microstructure and its evolution under the effects of irradiation and (internal and external) stresses.

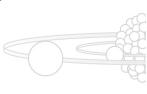
By combining a finite-element approach with a phase-field modeling approach, we will be able to model the thermo-mechanical behavior of both fuel and cladding in the presence of fissiongas bubbles and with explicit incorporation of microstructural discreteness and inhomogeneity. This approach represents a significant point of departure from the legacy models in that explicit consideration of the most critical microstructural processes in the fuel under irradiation makes possible the concurrent simulation of fission-gas behavior and fuel restructuring. Explicit consideration of the dynamic interplay between fission-gas bubble kinetics and microstructural processes, such as plastic deformation and grain growth, represents a critical aspect of this coupling. This will then enable prediction of the connection between bubble swelling and fissiongas release that naturally incorporates the effects of temperature and stress, and their gradients, on both fuel restructuring and fission-gas dynamics.

#### Results and Accomplishments

During Fiscal Year 2010, we made significant progress in matching phase-field and sharp-interface models and asymptotic analysis of the phase-field model to determine its consistency with the corresponding sharp interface model and to fix model parameters in terms of physical materials properties. This step is necessary in order to determine whether the assumed free-energy functional gives rise to defect and interface kinetic equations consistent with the classical sharp-interface description of the model. In the phase-field model the material parameters appear in the free-energy functional and in the kinetic evolution equation of the order parameters (via the Cahn-Hilliard and Allen-Cahn equations). This analysis has resulted in the determination of the gradient energy parameters and the kinetic mobility of the non-conserved order parameters in terms of actual material properties, such as the surface energy and constitutve kinetic paramters, including species diffusivities.

#### Benefits to DOE

The integrated mesoscopic, materialsphysics based predictive engineering-simulation capability to be developed for fission-reactor fuels and their cladding will represent a powerful



tool for the design and validation of new and established nuclear systems, benefiting the DOE's science and technology mission.

#### Conclusion

We continue to take important steps towards developing and testing a comprehensive conceptual and computational framework for a unified mesoscale approach capable of capturing all the coupled, concurrent microstructural processes that take place *under irradiation* within a single computational platform. Based on various considerations, we have chosen the

phase-field approach. The numerical solution of the Cahn-Hilliard and Allen-Cahn equations together with the corresponding free-energy functional uses finite-element and adaptive-mesh-refinement types of computational techniques. These are not only amenable to advanced, massively parallel computer architectures and algorithms, but also facilitate scale-bridging algorithms consistent with the prevailing engineering-code approaches. The asymptotic analysis described above has demonstrated that our approach correctly yields the front-tracking approach as a theoretical limit.



### Aleatory vs. Epistemic Uncertainty in Seismic Hazard Analyses

Suzette Payne

Addressing issues related to defining and reducing aleatory and epistemic uncertainty in seismic hazard analyses

EN101

This Idaho National Laboratory (INL) laboratory-directed research and development (LDRD) project addresses issues related to defining and reducing aleatory and epistemic uncertainty in seismic hazard analyses. We have identified three areas that are significant contributors to predicted ground motion levels where aleatory variability can be replaced by epistemic uncertainty. The study areas include: (1) estimation of earthquake recurrence (i.e., maximum magnitude and rate); (2) incorporation of measured GPS and process-level modeling of fault block rotations into the estimation of faultspecific slip rates; and (3) parameterization of site-specific ground motion attenuation models. The first effort is focused on performing analyses with multivariate relationships between magnitude (M) and fault dimensions (i.e., surface and subsurface displacement, rupture width, surface and subsurface rupture length, and rupture area) using the well-known "Wells and Coppersmith" (1994) dataset. Because their analysis assumed independence, when all five relationships are applied on a single fault, there is a resultant non-physical multi-modal  $M_{max}$ distribution, which leads to greater uncertainty about mean seismic hazard levels. The second effort takes an alternative approach to incorporate fault-slip rates into a seismic hazard analysis that may not be accounted for as a contributor to the artificial multi-modal  $M_{max}$ distribution. The third effort is focused on taking a new approach for how to incorporate direct or inferred subsurface data and their epistemic uncertainty into site-specific and empirical ground motion models rather than accounting for uncertainty through inter-model (irreducible) aleatory variability.

#### Project Summary

The first year focused primarily on efforts to perform multivariate analyses on magnitude-fault dimension data, and some progress was made on preparing for analyses to estimate fault-slip rates using GPS data and on parameterization of site-specific ground motion models. For the multivariate analyses, the 1994 data set was obtained from Don Wells (of Wells and Coppersmith). Univariate regression analyses were performed to authenticate and validate the original 1994 dataset, which took

some time since not all data details were documented in the published paper. Upon successful completion of this step, the data were then evaluated to determine whether multivariate analyses could be used and whether additional data (such as earthquakes from 1995 to 2010) would also be needed. The preliminary results are encouraging and indicate multivariate analyses improves estimating magnitude based on fault dimensions. Further work is needed to include more earthquake data to reduce scatter in the original dataset, evaluate the approaches used in estimating fault dimensions in the original work, and separate parameters to assess possible systematic biases. For estimating fault-slip rates using GPS data, the new program, "tdefnode" (McCaffrey, 2009) was obtained and implemented at Idaho National Laboratory (INL). This program inverts GPS horizontal velocities to simultaneously solve for angular velocities, slip-rate deficit, and strain rates. Additionally, we compiled fault slip rates for fault-specific earthquake sources closest to INL and earthquake slip vectors for the Snake River Plain region. For parameterization of the ground motion models, we compiled, analyzed, or identified other projects with data that can be used in our analyses. These efforts included collecting and analyzing data relevant to depth intervals of interest (<600 m), review of well information, and review of geostatistical analysis and spatial continuity of shallow structures.

#### Benefits to DOE

Although not complete, our results will benefit INL and the U. S. Department of Energy (DOE). The recent evaluation of INL probabilistic seismic hazard analyses indicated potentially higher seismic design levels may result from an update of the hazard, primarily arising from incorporation of aleatory variability in ground motion models. Results of this research will directly impact approaches that can be taken in future hazard analyses to reduce uncertainties in parameters and models. Reducing such uncertainties can lead to cost-effective data collection and can be used to minimize costly facility upgrades currently expected based on limited INL-specific data.

Idaho National Laboratory

58

### **In-Pile Temperature Monitor and Control for ATR**

Jian Gan

This project is to develop a fiber-optic temperature sensor for real time in-pile measurement  ${\tt EN102}$ 

The objective of this project is to develop a fiber-optic temperature sensor for in-pile real-time temperature measurement. The advantages of using a fiber-optic temperature sensor include (1) its small size, (2) no signal interference from electric and magnetic fields, (3) high sensitivity, and (4) reliability. Irradiation temperature is one of the most critical parameters that heavily influence the microstructural development under irradiation. Idaho National Laboratory's (INL's) Advanced Test Reactor (ATR) plays a strong role in nuclear materials and fuel development through in-pile irradiation experiments. The capability to record in-pile irradiation temperatures is essential to the success and the quality of these reactor irradiation tests. This project aims to develop a prototype fiber-optic temperature sensor along with a miniature heating element to measure and control the irradiation temperature.

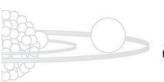
#### Project Summary

The major components for the experimental system for the sensor test have been identified and put to work. We evaluated different sensor configurations and identified three designs to proceed: (1) the original conceptual design based on an air gap Fabry-Perot (F-P) interferometer, (2) a sapphire F-P with sapphire fiber for the high-temperature section of the sensor head, and (3) a silica fiber with sapphire F-P. The bench-

top test has been performed, and all three designs work, which means these designs can produce the signal response to the temperature-induced F-P gap optical path change. A prototype sensor has been fabricated and tested successfully up to 600°C using only silica fiber with a sapphire disc F-P. The packaging and coupling of different parts and fibers to ensure good performance and measurement are the ongoing challenges for this project. A significant amount of work is required to refine the design, fabrication, and integration of the entire measurement.

#### Benefits to DOE

Research and development (R&D) on this sensor is important to INL's nuclear fuel and materials irradiation testing mission. It will significantly improve the ATR National Scientific User Facility (NSUF) program for the nation by providing high-quality irradiation test data with good temperature measurement, which is currently unavailable due to high costs and space limitation in the core. Our first year's result brings us one step closer to monitoring inpile irradiation temperatures. The experimental system developed by our collaborator, The University of Houston, is critical to test and fabricate the sensor with different designs. Continued work on this project will ensure INL's leadership in nuclear R&D.



# Multi-Rate Shock Physics Simulation of Blast and Penetration Events in Concrete

W. D. Richins, T. K. Larson, S. R. Novascone, W. F. Jones; Dr. C. Pantelides, U of Utah; Tim Garfield, Ph.D. Student, University of Utah

Enhancing capability for future research and development in event simulation for nuclear power safety and national and homeland security

EN103

The primary purpose of our research project is to establish and validate by experiment new methods and material parameters for simulating and enhancing the performance of critical concrete structures subjected to malevolent attacks and dynamic accident events. Our results will extend the application of simulation results into regimes where large scale experiments are too costly or otherwise impossible to conduct. The project began in October 2009 with identifying concrete test sample designs that would likely be relatively straight forward to build and simulate. We issued a subcontract to the University of Utah (U of U) to design and fabricate the concrete test samples. Since the concrete form work and materials were donated by Hanson Structural Precast, Salt Lake City, UT, we were able to increase the extent of our planned testing this year. We are supporting one graduate student at the U of U, Tim Garfield, who is completing the first year of his Ph.D. program on blast and penetration response of concrete.

We hypothesize that experimental data and application methods derived from our proposed studies will extend the application of simulation results into regimes where large scale experiments are too costly or otherwise impossible to conduct.

To test this hypothesis, we will:

- Model three benchmark problems to evaluate our ability to simulate dynamic concrete response: (1) high explosive blast loading, (2) high-speed penetrator, and (3) dynamic Brazilian test. Models for the dynamic Brazilian test are available from a previous study by the authors.
- Conduct small-scale concrete sample testing to provide high quality data to compare, validate, and improve simulation results.
- Design, construct, and test concrete samples to address the effects of concrete reinforcement, high temperatures expected in Next-Generation Nuclear Plant designs, standard steel rebar placement, and three potential performance-enhancing techniques: external fiber wrap, glass-based rebar, and synthetic fibers.

Project Summary

The blast test concrete specimens were delivered to the Idaho National Laboratory (INL) National Security Test Range (NSTR), and testing began in late September 2010. Three thicknesses were selected to represent a range in concrete failure mechanisms from bending to shear. The reinforcement of the test panels varies to sample possible building material configurations: (1) plain concrete with steel rebar reinforcement typical of structural concrete; (2) synthetic fibers with no steel reinforcement; (3) glass fiber reinforced polymer (GFRP) reinforcement bars; (4) steel rebar reinforcement retrofitted with an external GFRP jacket to simulate the retrofit of existing buildings; and (5) combinations of all of these. The blast test specimens were instrumented and tested September 27–29, 2010. Concrete cylinders were also tested to give us reliable concrete strength measurements.

Simulations of the response of the concrete panels to blast loading were undertaken to design the blast tests specimens using LS-DYNA, which is a multipurpose multi-physics simulation code designed primarily to simulate highly nonlinear physical phenomena such as large deformations due to blasts or impacts. The blast loading was selected using a combination of the simulation results and testing of one control panel. We used 10 to 20 lb of C4 explosive at a 1-m standoff from the center of the panel front face to ensure adequate damage without destroying the panels. Our Fiscal Year 2010 (FY-10) test plan for the blast panel tests included a dimensioned diagram showing the layout of the test panels, concrete block supports, instrumentation locations, explosive charge test stand, etc. An existing laboratory instruction, LI-135, "Explosive, Mechanical and Ballistic Breaching Research and Development," covered work activities of this type at the NSTR. Preliminary test results show significant reduction in concrete spalling and scabbing when using synthetic fibers in the mix or when the concrete test panels were retrofit with fiber wrap.

Using an established INL design, the U of U is building an impact test machine for conducting dynamic Brazilian tests on our concrete cylinders in FY-11. We requested and

Idaho National Laboratory

60

received supplemental funding to build the machine this year, allowing us to proceed with our concrete tension modeling and testing program early in Year 2. A Brazilian split-cylinder simulation model was created to match the planned dynamic split-cylinder experiments at the U of U.

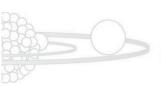
We are preparing two abstracts for the 2011 International Conference on the Mechanical Behavior of Materials.

### Benefits to DOE

This project brings new analytical tools and capability to INL, as well as a defined, peer-reviewed method to validate dynamic simulation of concrete structures in future safety analyses of nuclear or other safety-related systems. The fiber wrap process is likely to prove a viable method to retrofit buildings, increasing blast resistance.

Exposure to difficult problems strongly enhances our capability to perform future research and development in simulating dynamic events related to both nuclear power safety and national and homeland security. INL publications establish credibility in the technical community and contact with program managers who may need assistance in this area.

Collaboration with the U of U will lead to joint publications, proposals, and the potential for Ph.D. opportunities for INL researchers.







National & Homeland Security







# Methodologies for the Design, Analysis, and Validation for Operation of Complex Resilient Networks

Craig Rieger, Keith Daum, INL; Venkat Venkatasubramanian, Purdue University Developing a rigorous mathematical and computational framework to model and analyze complex resilient distributed control networks

NS165

Complex networks are highly organized sets of flows of entities such as material, energy, information, money, etc., that aim to deliver service levels that maximize some performance metric, such as profitability, service availability, etc. Examples of such networks include the electrical power grid, transportation networks, supply chains, advanced nuclear fuel cycles (NFCs), and so on. Such networks are national—even global—in scope, highly regulated, require traceability, integrated with power systems, and have to adjust to supply, demand, and technological disruptions to ensure competitive products, service, and prices on timely deliveries.

In such an uncertain environment, the ability to identify and monitor the complex interconnections and emergent properties, assess the current state, diagnose anomalous or abnormal events, and autonomously reconfigure the network is vital to guarantee service and manage profitability. When these disruptions occur, strategic, operational, and tactical decisions involving engineering, management, and information layers will have to be executed. In the design and analysis of such networks, efficiency, robustness, and cost are critical performance metrics. Efficiency measures how well the network performs under given constraints, robustness measures how well the network will perform when parts of it are taken down due to various disruptions, while cost considers not only the procurement cost of the system but also the cost to keep the system performing up to the requirements. Resilient networks are both robust and efficient.

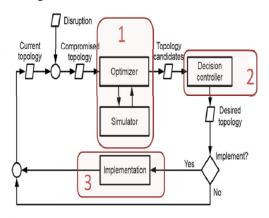
The technical objectives and the project deliverables for this three-year project would consist of the following:

- Understand how the topological features of a complex resilient network affect performance metrics such as efficiency, cost, and robustness
- Develop a general mathematical and computational framework for the design of complex resilient decentralized control networks
- Demonstrate these concepts and systems by implementing them in a testbed that will

- simulate resilient network prototypes and scenarios
- Produce papers, presentations, reports, and software to facilitate technology transfer to INL.

## Project Summary

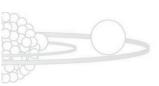
Building on the foundations laid in the first year, we enhanced the graph theoretical mathematical framework for modeling and analyzing network topologies to also include a decision framework. We developed RNEDE, a comprehensive framework for resilient network design. RNEDE environment consists of an optimizer and a decision controller, shown in Figure 1, which when subject to a series of disruptions determines a topology that achieves the functionality of the network and determine cost-efficient remedial measures. We developed a testbed prototype named RNEDESim, which includes capabilities for optimization, decision making, and visualization.



Objective 1: Optimizer problem
Objective 2: Decision control problem

FIGURE 1. Architecture of RNEDE.

Given a topology for a complex network and a sequence of incoming disruptions (resulting in a fault), RNEDE determines which remedial actions to take such that the network remains resilient to structural changes in the topology (due to a given disruption). In RNEDE a remedial action corresponds to determining the location and number of new nodes and/or edges that can be placed back on the network to make



it resilient to the disruption. The problem of determining the number and location of the remedial action is solved in two parts. First, RNEDE determines the set of optimal remedial action that can be taken, i.e., how many nodes/edges to add and where to add them. This determination is based on several factors, such as the level to which the complex network has been compromised, and the constraints associated in implementing the remedial actions on the network. Second, RNEDE decides whether it should choose to implement any of the suggested remedial actions. This decision-making is crucial, as a remedial action taken now may not be valid in the future. This part of RNEDE is based on the sequence of incoming disruptions and the cost associated with the remedial actions.

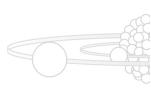
We now describe the problem mathematically. Let the complex network be represented by a topology T = (V, E), with V being the set of vertices and E being the set of edges. The topology specification T satisfies a set of constraints  $C = c_1, ..., c_n$ . The function S:  $T \rightarrow R^+$  determines the cost of maintaining a topology satisfying a set of constraints. Let  $f_i$  be a disruption that arises in the network which "compromises" the topology of the network. Let T' be the compromised topology. T' may or may not satisfy the set of constraints C. Let  $F: T \rightarrow$ R+ be a monotonic function that, given the original topology and the compromised topology, quantitatively measures the amount of the compromise. Let the system's knowledge base consist of a set of remedial actions, A = $a_1, ..., a_N$  and a cost function  $Q: A \to R^+$ . The two objectives in RNEDE then are to (1) obtain that set of remedial actions, such that when applied to T' result in a remedied topology T''such that the compromise is minimized, i.e.,  $F(T''; T) \le \epsilon$ , and (2) If  $\sigma = f_{1...}f_n$  is the sequence of disruptions that arise in the network, and the knowledge about each disruption arrives sequentially at the system, then minimize the cost of maintaining the compromised topology and the cost of making the change, i.e.,  $min \sum \sigma($  $a_i Q(a_i) + \mu S(T')$ ).  $\mu$  is the weight parameter that equates the two costs. For the supply chain case study considered, the topology satisfies a degree constraint, i.e.,  $|\{e|e \in E, (V,E) \in T\}| \le k$  for all  $V \subseteq T$ , and for a given constant k. The

function *F* is equal to the diameter of the topology. The cost function is application dependent and can be represented in any of the network quantities such as bandwidth or latency.

In RNEDE, the first part of the problem is solved by modeling it as an optimization problem, and the second part of the problem is solved by modeling it as a decision control problem. The framework is aided with a Visualizer, which allows the system administrator managing the network to diagrammatically see which changes in topology are being suggested. RNEDE evaluates a disruption one at a time and suggests a remedial action. This suggestion is based on the already-seen threats and assumes minimal knowledge about the future sequence of disruptions that may arise.

#### RNEDESim features:

- 1. The ability to visualize, create, edit and analyze large complex networks/graphs.
- 2. A modular design that makes it easy to add, remove, and extend functionality and new modules to the existing software.
- 3. A dynamic simulation platform for developing and evaluating methods for control of networked systems. In RNEDESim, each node of the graph represents an active process that generates events and responds to external events. We envision that RNEDESim will be dynamic with respect to its analysis capabilities.
- 4. The ability to load existing connected and disconnected networks, which are as large as 5000 nodes and edges (completely connected).
- 5. The ability to load a threat mode, i.e., a disruption and initiation schedule can be described. Also, nodes and edges are disabled for a given range of time.
- 6. On a large network, descriptions can be added for nodes, e.g., for producers/consumers/edge labels.
- 7. For the entire network, several metrics are available for computation and comparison, e.g., clustering coefficient, diameter,





centrality, coloring, strongly connected components, etc.

Figures 2 and 3 show an example of an optimized supply-chain network. In the third year, we plan to develop a decentralized control methodology for network resilience.

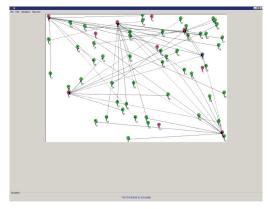


FIGURE 2. RNEDESim optimized network.

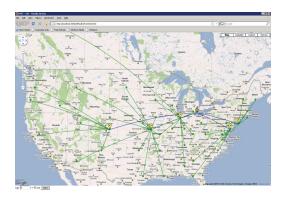


FIGURE 3. RNEDESim view on the map.

## Benefits to DOE

Given the above accomplishments, we are confident that the project is progressing well. The theoretical frameworks, dynamic simulations, and experimental campaigns are advancing the state-of-the-art in developing the next generation of highly automated resilient networks that can provide greater stability and efficiency while achieving graceful degradation by anticipating failures. Such an advance is required for a wide range of applications seen in energy, environment, and nuclear industries. The several papers that have already come out of this project as well as the simulation environment are beneficial to core Department of Energy and International Conference on Information Systems objectives.



# **Computational Materials Characterization**

Jeffrey Lacy

Rapid characterization of ballistic-resistant materials from sparse experimental data NS166

The Taylor impact test has for years been a popular method for verifying constitutive models and identifying a subset of model parameters, supplementing data provided by other experimental methods. Parameter identification methods typically compare measured deformation of the cylinder at a restricted number of locations to the deformations predicted by a hydrocode. For example, Johnson and Holmquist measured final length, maximum diameter, and one intermediate diameter to obtain the three yield- and strain-hardening constants (A, B, and n) of the Johnson-Cook Model. Allen, Rule, and Jones extended the method by employing a second-order polynomial response surface to minimize the difference between the experimental and computed cylinder profile at a single impact velocity. They had greatest success when beginning with parameter values calibrated to quasi-static test data.

## Project Summary

In the current work, we are extending the method to employ a multi-objective genetic optimization algorithm to minimize the cylinder profile errors simultaneously on three cylinders impacted at different velocities. No test data other than the three Taylor cylinders is employed in the parameter development; rather generic starting coefficients are employed. To ensure that the resulting coefficient set is applicable to other load states, both split Hopkinson pressure bar and axisymmetric expanding ring tests were conducted and compared to the resultant Johnson-Cook model. Excellent agreement was obtained.

A36 structural mild steel from 2 in. thick rolled plate was chosen as the subject material for this investigation. Because it is widely available and inexpensive, it is often used as a baseline or witness material in blast and ballistic studies. However, because it is never used directly in blast- or penetration-resistant systems, it is not well characterized in this load regime.

Cylinders, 1.27 cm in diameter by 4.23 cm in length, were impacted into a massive anvil of AR400 steel, at 3 velocities of 249.4, 269.4, and 284.5 m/sec. To measure the profile of deformed cylinders, a commercially available desktop 3-dimensional laser profilometer system was

employed. The full volume description allows us to quickly compare profiles around the specimen to evaluate ovality (material anisotropy) or asymmetry of the impact, and select the most appropriate profiles for comparison to computational results. Mathematica and Python scripts have been written to automate the profile generation process.

The DAKOTA toolkit, developed and maintained by Sandia National Laboratories, was employed as the framework for developing a J-C parameter set that minimized the error between computed and observed cylinder profiles simultaneously for three specimens impacted at different velocities (thus multi-objective). A genetic algorithm approach was selected to modify and evaluate the fitness of candidate parameter sets with 5 design variables (*A*, *B*, *n*, *C*, and *m*). 50 generations were allowed with a maximum of 2500 simulations. A mutation rate of 0.1 was employed.

The Eulerian finite-difference code CTH V9.1 was used to perform all impact simulations. Two-dimensional axisymmetry was employed, but the usual assumption of a perfectly rigid impact face was discarded as unnecessarily simplistic. Rather the anvil response was approximated using a Johnson-Cook model representative of rolled homogeneous armor steel. The domain was meshed with uniform cells, 0.1 mm  $\times$  0.1 mm, and all runs carried out to 160 µsec to ensure completion of the impact event without operator intervention.

Given that this parameter set was derived from only three experiments, we investigated the extent to which the results are applicable to loading conditions beyond the Taylor cylinder. To that end, specimens from the same plate of steel were subjected to two other load cases: the split Hopkinson pressure bar (SHPB) and the axisymmetric expanding ring (AER) tests. Data from both are compared to the final Taylor-derived parameter set as a way to check the validity of the parameter set. We did not incorporate these data into the parameter extraction process in order to maintain independence of the reference data from the measured quantities.

aboratory

68



True stress/true strain relationships derived from the extracted J-C parameters are plotted along with the experimentally-obtained data. Quasi-static tensile data (curve 1) is reported by Fisher and Iwankiw. Compressive SHPB and tensile AER data are from the current effort as described herein.

The extracted J-C parameter set does a good job of capturing the yield, hardening, and rate dependence observed in the SHPB test results. The initial data from the AER, reported as a single dynamic yield stress number, also compares well. It should be noted that current data does not indicate whether the thermal softening parameter is matched. Additional tests at high temperature are required to evaluate that parameter.

The classic Taylor impact test imparts temporally and spatially varying fields of strain, strain rate, and temperature through the specimen. It is possible to exploit this complexity by identifying constitutive model parameters that predict the final deformed shape of the specimen. We employed a multi-objective genetic optimization method to identify the bestmatch parameter set for a group of three specimens impacted at different velocities. No other experimental data was used to calibrate or guide the optimization process.

We find the method provides good agreement with experimental split Hopkinson pressure bar compression and axisymmetric expanding ring tensile results. Further work is necessary to confirm that thermal softening parameters are adequately captoured.

## Benefits to DOE

Development and deployment of armor and blast-resistant systems includes the elements of: threat characterization and evaluation; novel materials development and characterization; performance prediction and optimization using modeling and simulation; armor solution prototyping; full-scale, field performance testing and validation; and limited-scale production of deployable systems. This effort will enhance and accelerate the development cycle by inventing and validating new methods to identify material properties necessary for the design and characterization of armor and blast-resistant solutions, benefiting DOE's National Security mission.



# **Next Generation Control System "Smart Grid" Simulation Environment**

Lyle Roybal

Developing a simulation tool that will allow the testing of next generation control system concepts for resilience, including the aspects of cyber and smart grid automation

NS167

The proposed objective of this effort is the development of a simulation tool that will allow the testing of next generation control system concepts for resilience, including the aspects of cyber and smart grid automation specified in the call. Due to the extreme breadth of the work scope and the broad definition of Smart Grid and Micro Grid concepts, the work scope was narrowed to focus on the effects of renewable energy concepts such as wind and solar power generation on military bases and their effects on critical mission aspects of their operation. This is especially important in the development of Smart/Micro Grids, where distributed assets are representative of the physical system and the effects of failure can be regional or critical mission related.

Expected areas of development opportunities and goals include:

- Developing an understanding of control requirements for bulk power control in typical Smart/Micro Grid applications
- Developing and validating design criteria, design guides, and standards for successful implementation of Smart/Micro Grid concepts for commercial, industrial, military, and utility applications
- Developing an Idaho National Laboratory (INL) platform/test-bed for evaluating the use of Smart/Micro Grid control concepts on real world systems integrating renewable sources, Smart Grid controls, related cyber, communications, etc.

### Project Summary

Two separate modeling efforts were pursued concurrently during the course of this laboratory-directed research and development (LDRD) project. In one, a validated power system dynamic model was created on the Real Time Digital Simulator (RTDS) that integrates renewable energy into an existing U. S. Department of Energy (DOE) or U. S. Department of Defense (DOD) facility power system to enhance mission sustainability. Validation is achieved by comparing RTDS operational results to the data collected during a loss of commercial power test conducted at F.E. Warren Air Force Base, resulting in an islanding

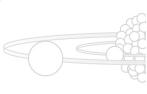
situation in which diesel and wind turbine generation were providing power to the base static load. Operational and dynamic test data were recorded and are to be compared with the dynamic simulation data available from the RTDS model.

With the completion of the validated model, sensitivity testing can now begin in earnest to determine the overall system dynamic sensitivities, control rates, stability margins, etc., needed to support the research and provide the basis for completing overall objectives.

In a second smaller effort, a systems dynamics approach was considered and a very primitive systems dynamics model of a theoretical military base or post was constructed. This included renewable concepts such as wind and solar power, and a crude model using the battery storage from electric and hybrid vehicles to augment the base energy needs and sustain the base/post energy needs during a time of distress. While the systems dynamic approach does not per se model Smart Grid control strategies specifically, it does allow the analyst to investigate different size mixes of renewable sources and load shed strategies that may be needed to keep critical base resources alive. Additionally, the system dynamics approach allows the analyst to express base operations in terms of "green energy" and "carbon footprint," which are quickly becoming important with the advent of Federal mandates for military installations in these areas. The systems dynamics is a stock and flow approach to modeling dynamic processes. In the case of electric usage strategies by a potentially islanded base or post, the stock is energy and the flow is power. This approach is not meant to model the detailed physics of the electric power grid in a base or post, but rather capture the general movement of power to various entities modeled and investigate sizing of backup sources, or other renewable storage strategies in a disruptive event such as the loss of a major energy source to the base.

# Benefits to DOE

Continuation of mission objectives during a loss of commercial power at DOE and DOD facilities is of extreme importance. Integration of



renewable energy provides an avenue for the facility to continue operations without becoming mission limited by the fuel available onsite or contributing to air emissions, resulting in a smaller carbon footprint. The validated RTDS model provides a real world system to study the Smart/Micro Grid phenomena, development, and testing of real world control strategies, equipment, system effects.

The work establishes the basis for expanding the current INL Power Test bed to include Smart/Micro Grid type field testing at INL. The RTDS Smart/Micro Grid modeling capability (combined with our power-system test-bed) continues to attract the interest of key DOD and university researchers looking for real-world test-beds that can support their research and test system needs.

The concept of the Smart/Micro Grid at INL leverages and unifies all the elements of the existing INL national security test-bed

capabilities and resources, i.e., cyber, powersystem, wireless communications, etc. Given that the INL test-beds exist, are robust, fully matured, integrated, and well staffed, a successful Smart/Micro Grid implementation is a logical next step in our continued development of the related INL signature areas.

Additionally, the use of systems dynamics approaches to answer bigger picture questions like the size of battery backups versus costs benefits, and the impact of renewable (and somewhat unreliable) energy sources of the operation of a critical DOD or DOE installation, and the impact of different energy strategies on the carbon footprint of an installation shows promise and should be investigated further during the coming years.



# **Cognitive Intelligent Wireless Communication Devices**

Hussein Moradi, Ph.D., P.E.

Establishing a leadership role in wireless communications
NS168

The National and Homeland Security Directorate (N&HS) of the Idaho National Laboratory (INL), through its laboratory-directed research and development (LDRD), is building technology research, development, demonstration, and deployment (RDD&D) capabilities to establish a national leadership role in wireless communications. This research complements the testing capabilities of the INL's Next Generation Wireless Test Bed. A key component of this initiative is completing proofof-principle research, development, and evaluation of a cognitive communication device prototype that can potentially satisfy future government and global needs. The LDRD started in second week of January 2010. As the research evolved, more focus was applied to modeling and demonstrating a point-to-point communication between two software-defined radios (SDR) as opposed to performing network topology simulation. This was a good decision because in the process of achieving the demonstration goal many fundamental problems needed to be resolved.

Project Summary

A SDR development platform was determined to be necessary to move wireless communication ideas and theories into reality. This approach would allow theories to be validated and provide a better understanding of how wireless designs are actually implemented in hardware, as well as provide insight into the design tradeoff decisions that must be made when implementing such designs in hardware.

A comparison was made of commercially available SDR platforms. A competitive analysis was made between several SDR providers such as Universal Software Radio Peripheral (USRP) from Ettus LLC, SDR-300 from National Instrument, and Small Form Facture (SFF) SDR from Lyrtech Corp. The Lyrtech platform was determined to be the best solution based on price, performance, ease of use, and availability of open user libraries. Two Lyrtech SFF SDR systems were purchased with LDRD funding.

The Lyrtech SDR system utilizes Field Programmable Gate Arrays (FPGAs) and a Digital Signal Processor (DSP). This allows the system to be easily reconfigured in order to quickly implement design changes. The system is "Software Defined" due to the flexibility of modifying the design by changing the software that runs on the DSP or the code that is loaded into the FPGAs.

The Lyrtech system makes use of several advanced software development tools such as: Matlab Simulink, Ti Code Composer, and Xilinx IDE. The team integrated these tools with the Lyrtech system to provide a model-based design environment. This environment, while complex, offers a tightly integrated simulation and development platform that can reduce and simplify the design cycle. A model-based design environment provides high level blocks that are easy to simulate and that can be quickly compiled into code. With the push of a few buttons, the same design can be simulated and then compiled and loaded onto the SDR platform. This design environment also provides a flexible platform where simulations can be done entirely in software on the host computer or split between the SDR hardware and the host computer.

The Lyrtech SDR design is complex and offers many interfaces and communications paths that can be configured in various ways to offer a flexible wireless development platform. We now understand the hardware as well as the reference Multiple-Input-Multiple-Output (MIMO) Orthogonal-Frequency-Division-Multiplexing (OFDM) transmit/receive design that was provided by Lyrtech. This reference design, which provided a point-to-point OFDM communication system, was successfully modified and simulated within the INL wireless laboratory. Next, a spectrum sensor using Welch's method was designed and implemented on the Lyrtech system. Successful spectrum sensing and various design trade-offs were verified and observed using the SDR system and a signal generator.

These initial first steps in implementing a SDR system helped the team gain a deeper understanding of the fundamentals of wireless communication and the complexities and tradeoffs when implementing such designs in hardware. These efforts have provided essential understanding and capabilities about these systems that will allow the team to quickly move



forward in researching new areas of wireless communications. These capabilities provide a basis that will allow new design concepts and ideas to be quickly implemented and tested in hardware.

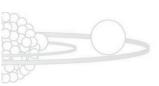
#### Benefits to DOE

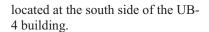
A platform for testing wireless communications has been established at INL that will enable further research and development of sophisticated wireless technologies. These capabilities will provide an environment to perform RDD&D of wireless communications technologies.

Below are listed the capabilities that have been achieved thus far:

- Lyrtech SFF SDR Hardware Platform:
  - This platform will allow theoretical design ideas to be implemented and tested on a physical radio. Using a ready-made SDR platform had significantly reduced the development costs and time that would otherwise be involved in obtaining this capability.
  - This platform integrates sophisticated third party tools such as:
    - Mathworks, Matlab, and Simulink tools
    - Xilinx FPGA Suite
    - Texas Instruments Code Composer Studio
  - This platform provides a model-based design environment that allows designs to be easily simulated and implemented, thus reducing development time.
- University contacts and cooperation that have contributed research expertise and consultive insight on the most current wireless research topics:
  - Brandon Lo (Georgia Tech Intern, Ph.D. Candidate): Provided wireless technology fundamentals, high- and low-level design implementation for MIMO OFDM design and spectrum sensor. The funding for Brandon's work was established through this LDRD

- through INL's 2010 summer internship program.
- Dr. B. Farhang (University of Utah Professor and Associate Chair): Provided tutorial wireless technology fundamentals and consultive input for future implementation of filter bank technology with SDR. The funding for Dr. Farhang's work was established through the 2010 Faculty Staff Exchange (FSE) program.
- Wireless testing lab and tools, which will greatly facilitate current and future wireless hardware design and development:
  - Spectrum Analyzer: Funded under amended 2010-LDRD
    - The Signal Analyzer is used to validate our spectrum sensing design. Essentially, once the design is correct after the debugging phase we will be able to observe the same signal on the spectrum analyzer and the output of our designs.
  - Simulation capable computer laptops: Funded under 2010-LDRD
    - We utilized these laptop PCs as our host computers and to run MatLab simulations. These PCs may not be adequate for our long term development due to the need for faster simulation processing times. As a result, high end computers have been identified and are in procurement as part of equipping the new wireless R&D laboratory (UB4), funded under IGCPE FY2010.
  - RF isolation locations, also known as RF screenroom: Funded under 2010-LDRD
    - The screenroom is 10 ft (W) × 17 ft (L) × 8 ft (H) and is doubly shielded to isolate the RF equipment for high fidelity testing of handheld prototypes against the simulation results. The screenroom will be assembled in wireless lab

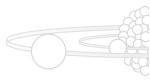




- Signal Generator: Funded under 2010-IGCPE
  - The signal generator is used to generate OFDM subcarrier frequencies tuned to the 2.5 GHz WiMAX spectrum. The generated signal is in turn sensed by our design.
- Intellectual Expertise. The team has gained expertise in the following areas:
  - Wireless communication: use of Fast Fourier Transform, modulation schemes, mixing, up/down converting, filtering, OFDM, and MIMO/SISO (Single-Input-Single-Output) design

- Matlab and Simulink application to wireless devices
- Model Based Design application to wireless device design
- FPGA design experience
- o Digital Signal Process code development.





# **Cyber Security for Protection of Critical Infrastructure: Expanding the Scientific and Engineering Bases**

Miles McQueen

Expanding the scientific and engineering bases for protecting Information Technology NS169

There is a clear need to expand the scientific and engineering bases for protecting Information Technology and other critical infrastructure from cyber security related attacks. Currently, with a few important exceptions such as cryptographic ciphers and protocols, security decisions at all levels of management and across cyber security disciplines are based on poorly understood or unrealistic models, ill-defined needs, urban legends, expert assertion, or wishful thinking. This project began the process of creating a fundamental shift in the practice of security from craftsmanship and art to science and engineering by focusing on measurements related to the vulnerability disclosure process. Vulnerability data was identified, collected, and analyzed to assess reasonableness of current disclosure timelines and to suggest new approaches.

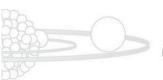
#### Project Summary

Three organizations which discover, purchase, or report cyber vulnerabilities have recently announced new timelines for public disclosure. TippingPoint, through the Zero Day Initiative, has announced they will provide a sixmonth window for a vendor to generate a workaround or patch; Rapid 7 will provide a 15-day window before reporting the vulnerability to CERT/CC, which then allocates another 45 days for a vendor fix before public announcement is made; and Google has announced a 60-day window for vendors to make

a fix before public announcement. After our detailed analysis of the timelines for 900 vulnerabilities, it is not at all clear how the individual organizations decided on their selected timelines for disclosure, nor whether the stated timelines will be more helpful than destructive. For example, with high confidence we know that the current mean and median time for a vendor to provide a fix to a privately reported vulnerability are approximately 200 and 150 days respectively; we also have evidence that highly publicized and visible indications of a vulnerability result in vendor diversion of resources to quickly provide a patch, but may still take well over 60 days; and we also have preliminary evidence that it is questionable whether individual software products become more resistant to attack due to vulnerability disclosure.

#### Benefits to DOE

The clear need to expand the scientific and engineering bases for protecting information technology and other critical infrastructure from cyber security related attacks has been initiated. By using empirical evidence to call into question the current vulnerability disclosure process and timelines in Information Technology, the appropriate processes for other critical infrastructure may now be more rationally addressed.



# Object Reconstruction Techniques for Use in Radiography

Clinton Van Siclen

Developing a method to turn 2D radiographs into a 3D image NS170

A national security customer is looking for "techniques to produce 3D images from one or more 2D radiographs." How few radiographs are needed to produce a useable 3D reconstruction? To answer this question, the interrogated object (e.g., a suspicious container) may be regarded as a 3D array of voxels, each characterized by an xray attenuation coefficient value. Knowing these values gives the desired 3D image of the object. These values are the "unknowns" that appear in the set of linear equations represented by the 2D radiographs. Unfortunately, because the resolution of the radiographs dictates the voxel size, in general there will be many more "unknowns" than equations. Thus there are an infinite number of solutions to this problem; that is, an infinite number of 3D image reconstructions that produce the given set of radiographs.

In view of the apparently poor prospects for success (high risk!), it should be emphasized that the (potential) customer has provided a very well defined problem, and further, one that is evidently important to our national interests. Thus, this is a worthwhile project to pursue.

#### Project Summary

A first version of a simple computer model of x-ray interrogation was completed, and a novel iterative method for calculating solutions

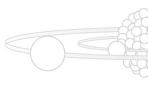
to the equations provided by the radiographs was developed. This method, which was verified by the computer model, is remarkable for (1) its simplicity; (2) its ability to incorporate information not related to the radiographs into the solution (i.e., the 3D image); and (3) its ability to produce *all* (infinite in number) of the solutions to the system of equations.

What then is the *best* solution? Consider that all solutions are points in a multi-dimensional space (the number of dimensions equals the number of voxels). These will more-or-less cluster in that space, reflecting the degree of variance among solutions. Then the best solution is arguably the centroid solution (the point at the center of the cluster) when the cluster size is sufficiently small.

Thus a methodology for addressing the project objective, namely, how many radiographs are enough, has been established.

#### Benefits to DOE

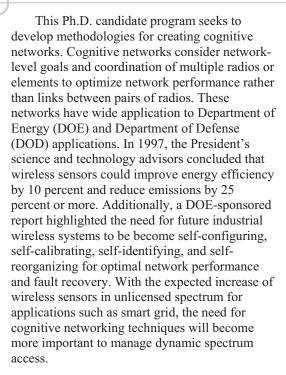
This project benefits DOE's national security mission, which is accomplished in large part by nuclear (neutron, gamma ray, x-ray) imaging. The iterative method developed under this LDRD project should become the method-of-choice for 3D image reconstruction.



# **Cognitive Network Engine and Simulation Framework**

Juan Deaton

Methodologies for creating cognitive networks
PH101



The DOD faces similar challenges. Next-generation warfighter communications should be capable of dynamically adapting communications to compensate for spectrum availability and interference. This is why the Army's Communications-Electronics Research, Development, and Engineering Center (CERDEC) issued a call for proposals for cognitive network concepts. Additionally, the Defense Advanced Research Projects Agency's (DARPA's) Next Generation (XG) and Wireless Network After Next (WNAN) are both programs that seek to advance cognitive networking concepts.

Therefore, leveraging the Ph.D. candidate at Virginia Tech, a lead university in cognitive networking research and Idaho National Laboratory's (INL's) unique capabilities with the wireless test bed, this project seeks to identify methodologies for developing cognitive networks. The Ph.D.'s expertise in an important research area will have applications for the next 10 years or more in areas relevant to homeland security missions with the wireless test bed and critical infrastructure protection. In terms of specific research, this involves researching a problem relevant to both DOE and DOD

missions: channel assignment. The channel assignment problem concerns how to assign available spectrum channels and transmit powers to a wireless network such that the network topology can be formed.

#### Project Summary

The objective of last year's work was to develop a method to compare disparate channel assignment algorithms in ad hoc networks. To accomplish this goal, we developed a system evaluation model and simulations for the evaluation. In addition, we also produced a new distributed algorithm for power control and channel assignment. This new algorithm was significantly less complex and had near performance to that of the best-centralized algorithms.

Additional work focuses on examining dynamic spectrum access in next-generation wireless networks specifically to examine a new framework and operational signaling procedures. This work created three new invention disclosures. The inventions describe a spectrum accountability framework as a means to solving many of the problems created by adding a dynamic spectrum access overlay in nextgeneration wireless networks. In addition to the spectrum accountability framework, the inventions also describe new operational signaling procedures. These signaling procedures are divided into three different embodiments. The first set identifies new or impacted service request supporting procedures to support a bearer service. The second set is for primary and secondary user alarms and responses. The third set is for the initialization of point-to-point devices to support use of dynamic spectrum access for wireless back haul. These signaling procedures form the basis for the operational basis for the spectrum accountability framework.

#### Benefits to DOE

This bulk of this year's research focused on the evaluation of dynamic channel assignment techniques for cognitive dynamic spectrum access networks. The results of this work were published in the *Journal of Wireless Personal Communications*. In addition, this work was also used to complete a Master of Science in Electrical Engineering degree and published as

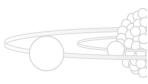


part of a Ph.D. dissertation thesis. Work this summer focused on examining dynamic spectrum access in next-generation wireless networks. This work produced three invention disclosure records (1862, 1863, 1864), which could result in valuable intellectual property. This work is targeted for the DySPAN conference to be held in May 2011 (submitted November 2010).

In the next decade, wireless network operators expect accelerated growth in smart phone devices and applications. Although these new devices and applications provide a welcome source of new revenue, the tremendous amount of data generated could overpower current operator spectrum capacity as early as 2014. As a result, operators must find new ways to increase their spectrum capacity. One option is to use the TV white space spectrum opportunistically (i.e., use vacant TV channels as secondary users). By deploying these TV white spaces as a dynamic spectrum access overlay, capacity-constrained operators could prevent loss of potential revenue by augmenting their licensed spectrum capacity.

While a dynamic spectrum access overlay can increase spectral capacity, it also brings significant challenges. The problem of identifying hidden receivers is a valid concern resulting in strict Federal Communications Commission (FCC) regulations for secondary use. In addition to avoiding interfering with primary users, the FCC also has yet to outline spectrum usage rules among secondary competitive operators. Furthermore, due to the possibility of many secondary competitive operators, detecting rule violations is vitally important in resolving spectrum feuding and judicious spectrum usage. This new work takes on the challenges of deploying a dynamic spectrum access overlay in next-generation wireless networks.

The wireless test bed along with this new research in understanding how a spectrum access overlay should work, creates a new INL asset. We are now in a unique position for performing full-scale research for dynamic spectrum access experiments at a network level. At this time, no other universities or test beds have this combined capability. Ph.D. research will continue to examine what work and experiments INL can carry out to further research in this area.



# **Application of Dynamic Bayesian Networks to Systems with Ambient Intelligence**

Dana Kelly

Demonstrate the practical application of dynamic Bayesian networks within an ambient intelligent system

PH102

The objective of this work is to demonstrate the practical application of dynamic Bayesian networks within an ambient intelligent system. Ambient intelligent systems possess the following attributes: First, they are contextaware, meaning they can recognize individuals within a particular situational context. Second, they are deeply customizable, tailored towards changing individual needs over time. Third, they are adaptive in their responses to inputs from the environment, allowing them to anticipate an individual's desires or intentions without conscious mediation. Finally, they are deeply embedded, integrated into the environment in a network context.

Project Summary

The funding provided for this research supported Ph.D. research at Technische Universiteit/Eindhoven. The research focused on the application of process mining techniques to the analysis of human reliability analysis (HRA) simulator data. An HRA that follows accepted good practices should incorporate data from a system simulator into the task analysis. Such data should include operator actions taken. process variable values as a function of time, because such variables influence operator behavior, and alarms and annunciators received by the operators. Put another way, what needs to go into an HRA is well understood. The problem the community has faced for many years is how. The resources required to analyze the resulting information are likely a reason for the infrequent use of simulator observations in support of the HRA task analysis. The goals of the qualitative analysis are to provide insights about process improvements that reduce risk, and to produce a model of operator performance for later quantification, in particular the principal process (and deviations from this process) followed by operators in responding to a plant upset condition. What is missing are tools to allow analysts to more efficiently and consistently make use of the sometimes vast amount of information gathered in the qualitative analysis, particularly during observations of operator responses in plant simulators. This research explored the potential of applying process mining to simulator data to aid in developing

models of human performance in support of the qualitative aspects of the HRA.

The following four research questions were explored and the answers to them are summarized below. The PhD dissertation draft was completed and submitted to the Ph.D committee. Expect to defend dissertation in early Calendar Year 2011:

- RQ1: What are the requirements for a tool to aid in the analysis of large amounts of simulator data in support of HRA?
- RQ2: How do current HRA methods approach the issue of simulator observations and are these approaches suitable for incorporating simulator observations into the HRA task analysis?
- RQ3: Are there tools in other domains that are more suitable and which, if adopted (and adapted to their new domain), could improve the state of the art in HRA modeling and task analysis?
- RQ4: What are the limits of applicability of these tools from other domains, and what improvements are needed in order to make them practical for use by an analyst who is not a specialist in using such tools?

### Results and Accomplishments

Research has provided input into development of crew-response models for the new hybrid human reliability analysis method being sponsored by the U.S. Nuclear Regulatory Commission. The findings with respect to each of the research questions above are summarized in what follows.

Ch. 2 of the dissertation, which examined the overall HRA process, listed the following characteristics to be considered in HRA modeling:

- Plant behavior and conditions
- Timing of events and the occurrence of human action cues
- Parameter indications used by the operators and changes in those parameters as the scenario proceeds



- Time available and locations necessary to implement the human actions
- Equipment available for use by the operators based on the sequence
- Environmental conditions under which the decision to act must be made and the actual response must be performed
- Degree of training, guidance, and procedure applicability.

The first three and the last of these are captured in simulator observations, which are an essential ingredient to a good HRA, as described also in Ch. 2. Simulators can produce very large output files, in a variety of formats. Manually analyzing such output data is very resource intensive, and has in the past limited the use of simulator experiments and observations in support of HRA. Thus, one requirement for the analysis tool is that it be capable of accepting data in a flexible format, and that it be able to handle large amounts of data, as process variable values may be recorded at millisecond intervals over a scenario lasting one hour or more. A second requirement is that the analysis cannot be purely statistical, because the HRA is concerned with the *process* followed by the operators, and not solely in statistical variables, such as the time at which a certain action is performed.

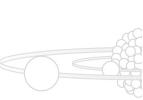
Ch. 2 examined two representative HRA methods, THERP and ATHEANA, both of which are considered complete methods, in that they address all three aspects of the HRA: identification, modeling, and quantification of HFEs. As noted in Ch. 2, most HRA methods address only HFE quantification, and thus are much more limited than THERP and ATHEANA. Both THERP and ATHEANA discuss the need to incorporate simulator observations into the HRA. However, neither method addresses how this is to be done. Ch. 2 also examined the TALENT approach, which was intended to constrain the variability observed in applying THERP (TALENT was developed before ATHEANA), by providing guidance for performing the HRA task analysis. However, TALENT does not address the question of how to incorporate simulator data into the task analysis. This led to the conclusion that there are no extant tools in the HRA community of practice that are suitable for analyzing large amounts of simulator data in the context of an HRA task analysis.

Ch. 3 of the dissertation, which provided an overview of business process mining tools, along with some selected industrial applications of these tools, showed that these tools have potential for application in support of HRA task analysis specifically, and simulator data analysis more generally. Tools developed for statistical data mining are of very limited use for HRA, because they are not concerned with the underlying process. The business process mining community appears to be the only one to have addressed data mining from a *process* rather than a *statistical* perspective.

Based on the industrial applications of process mining reviewed in Ch. 3, there was promise that simulator event logs could be mined from the control-flow perspective to provide information on the underlying process describing operator behavior, information that could be used to support existing approaches to HRA task analysis, as described in Ch. 2, or techniques such as the CRT proposed for use in the new hybrid HRA method described in Ch. 5, which is currently under development.

Ch. 3 examined some of the process mining tools and techniques in the context of simulator data. The most promising techniques appeared to be those developed for mining flexible processes, in particular the fuzzy model. Because simulator log files are typically very large, traditional process mining approaches can be expected to produce an overly complex "spaghetti model" that would be quite opaque to analysis. The fuzzy model abstracts away the irrelevant details, leaving the salient aspects of interest for the task analysis.

Ch. 4 began exploring how the tools of process mining might be applied to simulator data, beginning with a relatively small set of logs from the Halden empirical study. These logs had to be first converted to .csv format, and then to the .mxml format required by ProM. This file format conversion proved to be the first challenge, as a conversion plug-in for ProMImport had to be written in Java. A number



of difficulties were encountered during this conversion process, among them issues with timestamp formatting, inclusion of quotes in the event description field, and a mixture of characters used to delimit the fields in the data. These problems became even more severe in the application of Ch. 6, which involved much larger event logs.

Even for the relatively small event logs examined in Ch. 4, traditional process mining approaches, such as the alpha-algorithm for mining a Petri net, produced an overly complex model that would be of little use for task analysis. The heuristic miner produced a model that was significantly less complex, and showed some features that are immediately useful for HRA task analysis, such as alternative paths through the operating procedures taken by some of the crews who participated in the study. Converting the heuristic net to a fuzzy model within ProM allowed the fuzzy model to be animated to visualize the flow through the process model for each crew. The heuristic net can also be converted to a Petri net in ProM to allow more quantitative analysis.

Ch. 4 concluded that certain process mining tools, especially the fuzzy miner, appeared to be of use in support of task analysis, because they could clearly highlight differences in the underlying process governing each crew's performance. The heuristic miner proved to be useful as long as the event logs are relatively small, as they were for the analysis in Ch. 4. Ch. 6, which examined much larger and more typically sized event logs, concluded that the heuristic miner was of less practical use for logs of that size. Ch. 4 found that trace clustering, another technique for analyzing flexible processes, provided some insights as to highlevel similarities and differences among the 14 crews participating in the Halden simulator exercises. The trace-clustering technique may be helpful for follow-on simulator experiments involving large numbers of crews. However, its overall usefulness for HRA task analysis may be somewhat limited, as the number of crews in most facilities is much smaller than the 14 crews involved in the Halden exercise. For example, there were only four crews participating in the follow-on exercise described in Ch. 6, so trace

clustering and related tools were not explored further.

Ch. 6 examined the application of these process mining tools to simulator data collected at a U.S. plant during a follow-on to the Halden empirical study. Conversion of simulator logs to the .mxml format required by ProM proved to be a particularly severe problem to overcome. Thus, a significant amount of file preparation was required, including the development of a Java plug-in filter for ProMImport, before process mining could begin.

Such up-front file preparation introduces the opportunity for errors in the resulting mined models. Much of this preparation would be obviated if the log files were output from the simulator in .xml format, thus allowing easy conversion to the closely related .mxml format used by ProM. The process mining community has recently been working on making the tools more useful to the nonspecialist, and a conversion package called Nitro that has the potential to reduce the conversion burden significantly is now being marketed by Fluxicon (http://fluxicon.com/nitro/).

The fuzzy miner proved to be the tool of choice in ProM for mining the large event logs of Ch. 6. However, the imported simulator action event logs were very large after conversion to .mxml format, containing up to about 72,000 audit trail entries each. The resulting models were not useful, as they contained extremely large clusters, and these clusters contained both relevant and irrelevant actions. Lowering the node significance cut-off to reduce the cluster size produced models that were too complex, and too cluttered with insignificant actions, to be useful. Many of these audit trail entries were low-level actions, which are not at a high enough level to be of interest in the HRA task analysis. Thus, in order for tools such as the fuzzy miner to be useful for HRA task analysis, some considerable up-front manual filtering of the simulator action logs appears likely to be necessary, with more filtering being required the lower the level of the events that are logged. Such filtering has the potential to introduce errors into the resulting mined models, and so the analyst who does the filtering must have detailed knowledge of facility procedures and



operations, or have access to someone who does, to ensure that such errors are not introduced.

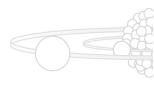
Applying the fuzzy miner to the filtered logs provided some especially useful insights for HRA task analysis, and particularly for construction of the crew response trees (CRTs) being considered for use in the new hybrid HRA method described in Ch. 5.

Process mining also provides a graphical illustration of the crew-to-crew process variability. From the standpoint of constructing a CRT to represent the procedural flow in the HRA task analysis, the process mining in Ch. 6 illustrates the additional complexity of the loosely controlled process followed by one of the crews. Thus, the structure of a CRT that is developed considering the performance of this crew will be more complex than had an analyst

not considered this crew, with additional branches required to represent the more complex process.

#### Benefit to DOE

The application of ambient intelligent systems to nuclear nonproliferation supports key projects in DOE-NE's mission of introducing the next generation of nuclear power plants. Electric grid reliability for transmission and distribution of electricity is also a vital concern for the nation, and there is great potential for ambient intelligent systems to help ensure high grid reliability and rapid detection and isolation of emergent problems. The work is directly relevant to the Advanced Instrumentation and Controls R&D pathway of the LWR Sustainability Program.



# **Armor Research and Development in Support of the U.S. Army**

Thomas Zagula

Developing a propellant-based reactive armor system

SM101

The objective of the proposed work is to demonstrate proof-of-principle concepts, focusing in four main areas:

- 1. Development of cost-effective bainitic steel as alternate armor steel.
- 2. Roll-bonded laminate armor system.
- Development of a ferroelectric material for EM armor system.
- 4. Research and development of a propellant-based reactive armor system.

Project Summary

#### Bainitic Steel

Recent research conducted at Cambridge University showed that low alloy bainitic steels, previously considered of limited value, can possess very high strength when processed correctly and can be produced at a low cost. The results of this research indicated that low-alloy steel with a final microstructure containing only nano-sized bainite and retained austenite phases can potentially be high-performance armor steel. This work is a continuation of current Idaho National Laboratory (INL) efforts to produce bainitic steel with the combination of high hardness, strength, and toughness required for armor applications. The bainitic steel effort will be directed towards identifying the optimum composition and heat treatment for armor applications, identifying the critical metallurgical characteristics required for ballistic performance. completing full-scale ballistic verification, and developing full-scale manufacturing processes.

#### Roll-Bonded Laminate Armor Systems

Past research at INL shows roll-bonded Ti/Ti alloy laminated plates have improved impact performance versus homogeneous plates with equivalent areal density, indicating the possibility of designing and fabricating multilayered laminated systems with improved ballistic and blast pressure performance. Additionally, reduced areal density can be achieved by roll-bonding more dissimilar constituent plates together. The behavior of the laminate under high impact loading is determined by a variety of material, processing, and design parameters, including high strain rate

properties, relative layer thickness, interfacial bond strength, number and order of layers, etc. Material systems will be chosen with consideration for density, high strain rate properties, ductility, formation of brittle intermetallic compounds between constituents, and material costs. Such data will also be useful design information to the survivability and the modeling and simulation communities. Rollbonding development will focus on the influence of relative layer thickness, surface preparation/finish, and the amount of deformation reduction on solid and blast pressure impacts. Targets taken from roll bonded plates as well as individual constituent plates will be subjected to impact testing using 20 mm FSP and high explosives to demonstrate improved performance.

# <u>Ferroelectric Material for Electromagnetic</u> <u>Armor System</u>

An electromagnetic (EM) armor system (also known as the Walker plate system) consists of two pre-charged metallic plates separated by an air gap. When a continuous shaped-charge (SC) jet bridges the two plates, an electric circuit is completed. Completion of the electric circuit causes a current to flow through one of the plates, through the jet, and to the opposite plate. The length of the jet that bridges the two end plates becomes a long straight conductor, and a highly compressive electromotive force (Lorentz force) is imposed on this length of the jet while ohmic heating of the jet occurs concurrently. When the capacitors are depleted, the EM field dissipates and the compressive force is released. The super heated jet then "rebounds" and jet material disperses violently and circumferentially into fine particles in a manner similar to a series of "smoke rings." The radial dispersion of the SC jet diminishes its subsequent penetration power into the base armor. The exploitation of EM field in the defeat of SC jet has been extensively researched and flash x-ray images have been taken of the formation of the smoke rings.

Sandia has demonstrated that high-pressure depolarization of a ferroelectric material can generate significant power pulses; but, to our knowledge, no specific research has been performed on utilizing ferroelectric materials for



EM armor. The objective of the ferroelectric material effort is to identify and develop a suitable ferroelectric material as a substitute to the conventional-charged capacitor banks of an EM armor system and demonstrate its performance potential with a small scale live-fire test

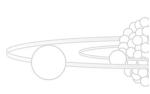
#### Propellant-Based Reactive Armor System

For this proposed R&D effort, we propose to investigate the potential use of a high-performance propellant chemical, such as guanidine nitrate (CH<sub>5</sub>N<sub>4</sub>O<sub>2</sub>), as a substitute driver material to high explosives (HE) in energetic reactive armor (ERA) systems. When blended with an appropriate proportion of oxidizer and a polymeric binder, guanidine nitrate (GUN) forms a composite that is environmentally stable and insensitive to incidental impact. GUN is normally available in powder form and, when blended with a room

temperature cured, catalyst-based polymer, can be cast and molded readily into net shape. A high-carbon-content-based polymer/binder such as polyurethane (PU) can be selected with the ultimate purpose of generating voluminous gaseous products, when shocked to a threshold level by the impact of an incoming, high-velocity projectile. Because GUN-based composite is a stable and insensitive material, the casting can also be trimmed, drilled, and pressed by mechanical means, and can be handled manually or with standard industrial equipment.

## Benefits to DOE

The work proposed for this laboratory-directed research and development (LDRD) project benefits INL and the U. S. Department of Energy (DOE) in establishing new capabilities and expertise that enhance the laboratory's ability to develop a propellant-based reactive armor system.



Idaho National Laboratory

1

# **Advanced Multi-Layer Laminate Blast Mitigation System**

Tom Lillo

Roll bonding shown to be a viable method for producing steel/aluminum plate composites with improved ballistic performance

BS110

The project sought to demonstrate that dissimilar metals could be bonded together, using an inexpensive rolling operation, to produce a laminate with ballistic properties superior to the constituent materials. The objective was to demonstrate roll-bonding on a commercially relevant scale and subject the bonded part to live-fire ballistic testing.

## Project Summary

Two roll-bonding campaigns using varied rolling parameters were carried out on 12 systems and produced four laminates suitable for ballistic testing. Ballistic testing using a Fragment Simulating Projectile (FSP), traveling at 6000 feet/second, showed the roll-bonded laminates to be well bonded with delamination occurring only near the point of impact. Also, a significant improvement in ballistic behavior was realized for the Al/steel laminate over that of just steel. The ballistic limit of 1-in.-thick Rolled Homogenous Armor (RHA) steel is given in literature as 3700 ft/second while our laminates did not fail even at a velocity exceeding 5000 ft/second (although our laminate was

substantially thicker than 1 in.—on the order of 2 in. thick—the RHA at 0.875 in. thick with the remainder of the thickness being aluminum alloy 6061). Additional laminates with a total thickness of 1 in. need to be made and subjected to ballistic testing to directly compare to 1 in. thick RHA armor steel. However, the laminates tested so far suggest that the concept of roll-bonded armor systems can significantly outperform monolithic armor materials, making the project successful in this regard.

## Benefits to DOE

The project provided a basic understanding for roll-bonding dissimilar materials and of the physics behind ballistic impacts on laminates of dissimilar materials, which will aid in the development of increased ballistic protection of critical components and personnel, thereby increasing national security. The Department of Defense will also benefit from improved armor designs that are lower in weight and higher in performance, reducing overall vehicle weight and increasing maneuverability and fuel efficiency.



# **Enhanced Metal Ion Analysis**

Mark Stone

Improving ionization efficiency and simplifying sample preparation FF107

Thermal ionization mass spectrometry (TIMS) is a standard method for analysis of geologic materials, isotope fingerprinting of pollutants, trace metals, medical measurements, groundwater, and chemical metrology. Sample preparatin is a tedious process and requires days to complete. We have developed a new technology for enhancing the ionization efficiency for TIMS analysis. It uses a microporous ion emitter (PIE), which is a mixture of metal powders scintered onto the filament. Using this new method, the detection limit has been reduced by orders of magnitude for praseodymium. This new approach reduces the loss of sample and allows for the analyte to be directly applied to the PIE, or the addition of an ion exchange resin top coat or other components such as silica gel for certain elements.

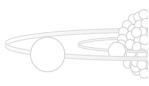
# Project Summary

The objectives involve improving ionization efficiency and simplifying sample preparation. This year's approach was three fold: (1) look for an enhancement in the analysis of metals such as uranium, (2) look for an enhancement in the analysis of still other metals by combining the PIE idea with the established silica gel technique, and (3) explore different sizes and shapes of PIEs to try to optimize their performance.

## Benefits to DOE

A porous TIMS emitter constructed from a Re/Pt alloy tightly bonded to a rhenium filament has been developed. This type of emitter is more reliable and shows sensitivity superior to the established resin bead method for uranium. It is rugged; none of the 100+ samples tested were lost. The overall efficiency ranged between 1% and 2.2% for uranium, an improvement over the resin bead method of 0.58%. In a related research and development project, the ionization efficiency of Sb by the traditional Si gel loading technique was compared with the Si gel method combined with the Pt/Re PIEs technology. The PIE technology provided an enhancement by a factor of ~4. Finally, some interesting differences were observed when using different shapes and sizes of PIEs, work that will continue.

This research directly relates to national security because it allows for more sensitive detection of ions related to nuclear nonproliferation. It also relates to environmental quality and science by permitting researchers to more easily and accurately detect and identify ions interest in the environment and to permit isotope fingerprinting of pollutants. Other geological and trace metal analysis could benefit from this work as well.



Energy & Environment Science and Technology







# **Investigation of Low-Temperature Performance in Membrane Materials and Processes for Gas Separations**

Frederick F. Stewart, Ph.D.

Development of more effective membranes for CO<sub>2</sub> and acid gas separation from permanent gases ST130

During the life of this project, three central tasks were completed. First, the relationship of structure and gas transport behavior was probed through selected syntheses where ten new polymers were formed. Second, the relationship between temperature and transport behavior was defined for numerous new polymers. Third, an apparatus was constructed to measure gas solubility in polymers. Also, a collaboration was funded with the Colorado School of Mines (CSM) in which a post-doctoral associate and a graduate student supported this project. The post-doctoral associate worked on studying the temperature-gas transport relationship in a high-performance perfluorinated polymer. This work led to a publication in late 2009. The graduate student completed his Master's thesis by studying the

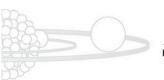
poly(trifluoroethoxy)phosphazene and a custom INL phosphazene that exhibits excellent CO<sub>2</sub>/N<sub>2</sub> separation factors for both permeability and solubility over a wide range of temperatures. The CSM group was instrumental in expanding INL's ability to measure gas solubility. INL has a state-of-the-art capability to measure permeability over a range of polymers from rubbers, which separate based on solubility, to glassy materials that exhibit size sieving. Permeability is a function of the solubility of the gas in the membrane and the diffusivity, which is a measure of the gas's ability to penetrate through polymer void volumes. To understand permeability, one must be able to measure two of the three variables. The new INL capability facilitates the direct measurement of solubility; this, coupled with the established capability to measure permeability, enables us to calculate diffusivity. Knowledge and understanding of the fundamental relationships in membrane transport allows us to develop and deploy membrane separation systems in highly demanding environments. This comprehensive capability aids the process for bidding new external work because it is needed for the appropriate matching of a membrane system with an application.

Project Summary

The objective of this project was to probe the relationship between polymer glass transition temperature (T<sub>o</sub>), membrane thickness, and process temperature on membrane performance to develop more effective membranes for CO<sub>2</sub> and acid gas separation from permanent gases. Membranes' processes are inherently low in energy: thus, we proposed to develop better systems to gain efficiency in terms of both productivity and cost. This work was done through three tasks: (1) membrane formation experiments using new techniques, (2) permeability experiments that investigated parameters such as temperature and pressure, and (3) sorption experiments in which we constructed and used an instrument that yields gas solubility data within membranes. Idaho National Laboratory (INL) has an established reputation in membrane science. This project was meant to enhance that capability through adroit experiments and partnership with academia that will ultimately lead to the enabling science and technology that will allow us to partner with industry and pursue development and commercialization. The novelty of this project lies in the relationship between T<sub>o</sub> and gas transport behavior and our unique materials; we can form a variety of low T<sub>g</sub> materials that can also operate at temperatures as high as 300°C.

#### Benefits to DOE

This project has led to a new capability and an enhanced understanding of membrane transport processes, which the project team is actively marketing. Other INL projects also are benefitting from a greater understanding of why membranes perform as they do. This is particularly valuable for DOE-sponsored applications such as carbon capture, hybrid energy systems, natural gas upgrading, and numerous other industrial processes.



# **High-Performance Polymer Membranes for High- Temperature Gas Separations**

John R. Klaehn, Christopher J. Orme, William J. Koros Increasing membrane gas throughput while maintaining selectivity at high temperatures ST131

High-performance (HP) polymers (Fig. 1) are glassy polymers that have high thermal stabilities with operating temperatures of 400°C, which allows their use in applications unsuitable for most organic polymers. These polymers are resistant to most organic solvents, and they are mechanically robust with high compressive strength. Their properties allow blending with copolymers and modifiers as well. Advances in polybenzimidazole (PBI) functionalization technology have facilitated further development of other HP polymers. Exciting high-temperature gas separation results were obtained from PBI and polyimides.

FIGURE 1. High-performance (HP) polymer structures.

The project's objective is to increase membrane gas throughput while maintaining selectivity at high temperatures by developing new HP polymeric membrane systems for gas separation applications. Products include heat and environmentally treated polymer membranes that have desired carbon dioxide (CO<sub>2</sub>) and/or oxygen  $(O_2)$  permeation properties. The approach that we proposed is based on "controlling" the polymer matrix and includes heat and environmental treatments. The heat treatments consisted of exposing the HP polymer membranes to carefully controlled temperatures in an inert gas environment in a furnace. This thermal treatment process gives highly selective membranes for gas separations. The literature lacks information regarding controlled heating under oxidizing conditions employing other gases, like nitrogen (N<sub>2</sub>) or CO<sub>2</sub>. Polymer membranes are well known to be swollen/plasticized by acid gases like CO<sub>2</sub>; however, it is not known what long-term affects may be induced into polymeric membranes by coupling the two processes. Idaho National Laboratory (INL) has conducted suite of

experiments in which membranes were processed using different treatment methods under different gas environments, attempting to enhance the membranes' gas transport properties.

### Project Summary

Our proposed approach was based on "controlling" the polymer matrix by heating under mildly oxidizing environments (thermal processing). INL's thermal processing methods were shown to give highly selective membranes for gas separations under inert atmospheres (argon). We evaluated several HP polymer membrane materials for this project, including Kapton® (polyimide), VTEC PI series (polyimides), PBI, and blends of these polymers, all of which were chosen for their high-temperature stability (>250°C) and their ability to effect gas separation for hydrogen (H<sub>2</sub>) and CO<sub>2</sub>. We have thermally processed multiple membranes under several different conditions, and our results show improvements over the parent polymers, especially with Kapton and VTEC. For example, our thermally processed Kapton membranes provided excellent separations for carbon dioxide verses methane (Alpha = 34 at 250°C), which has not been shown previously. We have successfully shown that gas transport properties of a polymer can be improved using our thermal processing methods that provide the desired high-temperature HP membrane transport properties. In addition, small molecule cross-linkers were found to greatly enhance the stability of the polymers. Certain polymer blends were shown to withstand very high temperatures (about 600°C) without noticeable physical changes of the polymer blend. During Fiscal Year (FY) 2010, a patent pending polymer blend ("MAL-PRO") was identified that has exceptional gas separations for hydrogen versus carbon dioxide (Alpha = 16 at 150°C). This was a major discovery for this project and high-temperature gas separations in general. Overall, our synthetic and processing pathways provide control (tuning) of the polymers' high-temperature gas transport properties—especially for temperatures of greater than 250°C.

ratory

The Laboratory's high temperature gas permeability testing apparatus was upgraded to provide mixed gas permeability experiments of greater than 400°C—a new, highly unique INL capability. With this apparatus, various glassy polymer membranes/films have been successfully tested up to 450°C. For example, a Kapton film using our thermal processing techniques gave good carbon dioxide separation from methane at 400°C, even after an extended period. In FY 2008, we postulated that using cross linking agents with HP polymers might provide enhanced gas separations. During FY 2009, we found one cross-linked, HP polymer blend that forms stable, high-temperature membranes. Remarkably, it provided even better gas separations than the parent polymer. The FY 2010 results demonstrate that cross-linked polymers can give impressive high-temperature stability while achieving excellent gas separations. Also during FY 2010, we expanded INL's gas permeability testing capabilities to include high-temperature water exposure (large quantities of steam). This is an invaluable resource for the Laboratory, since most of the critical high-temperature gas separations (watergas shift, flue-gas, and carbon-to-methanol conversion) will have high-temperature steam present. INL's polymer research group can now properly evaluate the high-temperature gas transport properties of polymeric membrane materials.

We have developed working relationships with academia and industry. INL's enhanced relationships include: (1) Georgia Institute of Technology (Georgia Tech) for gas permeability analysis and development of small polymer fiber membrane modules (contract research, supporting students and post docs) and (2) Texas Christian University using positron annihilation lifetime (PAL) spectroscopy for determining polymer void volume of the membranes (contract with faculty member). We have developed a collaborative research and development agreement (CRADA) with Compact Membrane Systems (CMS) for developing new blends with their fluorinated polymers. Finally, new funding sources are being generated by this research through the Department of Energy-Energy Efficiency and

Renewable Energy's (DOE-EERE's) Industrial Technologies Program (ITP), where we have partnered with General Electric and Western Research Institute (WRI).

Pyre-ML and Kapton are nearly identical polyimides when fully processed, and it can be postulated that Pyre-ML may give similar performance as the previously treated Kapton. Our work indicates that Pyre-ML is used best for gas separations in a hollow fiber membrane format. Because INL has not developed hollow fiber spinning methods, we subcontracted with Georgia Tech to spin fibers and assemble the needed demonstration modules. Georgia Tech also agreed to provide gas permeability data for the modules. Georgia Tech has delivered the initial hollow fiber membrane results (Fig. 2). Their gas testing results of the hollow fibers showed better gas separations than Kapton at room temperature. This is a major step towards larger scale gas separations.

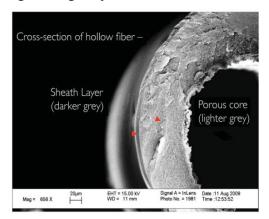


FIGURE 2. Pyre-ML hollow fiber.

The question about the changes in the polymers' void volume while being exposed to CO<sub>2</sub> was addressed through positron annihilation lifetime (PAL) spectroscopy (subcontract with Texas Christian University [TCU]) for various glassy polymers. An interesting trend with the positron (o-Ps) lifetime and intensity has been observed from these studies. Tight matrix glassy polymers (VTEC and PBI) show a decrease in o-Ps intensity at higher CO<sub>2</sub> concentrations, demonstrating that CO<sub>2</sub> can be trapped in the tight matrix and CO<sub>2</sub> fills the open voids in these polymers. However, o-Ps lifetimes for these



polymers do not change, indicating that the matrix does not expand (open up) greatly when exposed to higher CO<sub>2</sub> concentrations. Overall, these data suggest that the polymers are undergoing changes while being exposed to CO<sub>2</sub>, but the degree of change is small at modest pressures.

To conclude, we have the unique technical understanding that makes possible chemical synthetic modification of several of the HP polymers and provides the basis for enhancing these polymers' performance. In addition, we have developed several cutting-edge polymer processing techniques and obtained gas separations data for both pure and mixed gases. The data obtained during this project will offer the basis for intellectual property disclosures, several publications and presentations, and new partnership opportunities. Overall, we continue building an exceptional gas separation capability that will continue INL's leadership in the area of membrane technology development for years to

#### Benefits to DOE

Tangible accomplishments for this project for FY 2010 include:

- Awarded funding from DOE-EERE ITP with GE and WRI as our partner for \$2,400K.
- Submitted several invention disclosure records (IDRs). One IDR (BA-500) was elected by EES&T's BEST committee and another three are in preparation.

#### • Publications:

- Wrote a book chapter that will be published by Elsevier in Inorganic, Polymeric and Composite Membranes: Structure-Function and Other Correlations – Series: Membrane Science and Technology.
- Submitted a technical paper for publication at the *International* Conference on the Application of Accelerators in Research and Industry (CAARI 2010)

#### • Presentations:

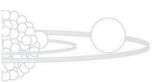
- American Chemical Society National Meeting (Spring 2010)
- Gordon Research Conference 2010
- International Conference on the Application of Accelerators in Research and Industry (CAARI 2010)
- Collaborations with universities:
  - Georgia Institute of Technology (William J. Koros); manufacture high-temperature membrane modules
  - Texas Christian University (Carroll A. Quarles); positron annihilation lifetime spectroscopy (PALS) on selected glassy polymers
- Future opportunities: collaborations and/or funding:
  - Compact Membrane Systems (CMS) has signed a CRADA to do a project with us.
  - General Electric (GE), INL, and Western Research Institute (WRI) are collaborating with INL's research group on a funded 3-year ITP project for high-temperature syngas/hydrogen/carbon dioxide separation.
  - New funding opportunities from DOE-Office of Fossil Energy (FE) (10–13 awards split from \$60 million).

This research provides a great benefit for INL, the region, the nation, and DOE by enabling new membrane technologies for high-temperature separation of carbon dioxide and other industrial gases. DOE-FE and DOE-EERE will benefit from the research direction for improved carbon dioxide and other relevant industrial separations. Overall, everyone can benefit from the possibility of truly cleaning up the environment and providing clean energy at reasonable costs. We have created the key steps for future partnerships and target future funding opportunities for INL. We have developed many working relationships/collaborations with industry and academia that will lead to



92

development of commercial polymer membranes. We have found several polymer manufactures for collaboration, including PBI Performance Products, Inc. (PBI), CMS and RBI, Inc. (VTEC—polymer manufacturers). In addition, we are putting together collaborations with U.S. membrane manufacturers (like Aire Liquide, Millipore, General Electric, CMS, and Air Products) and power plant companies (like WRI and Dominion). Furthermore, this project has specifically targeted expected DOE-FE and DOE-EE solicitations in the out years.



# Reducing Coalbed Methane (CBM) Water Discharge Volume

Eric P. Robertson

Decreasing surface water discharge while maintaining an economical methane production scheme GS101

The original hypothesis of this project was to show that it is possible to reduce or eliminate the discharge of produced water associated with the production of coalbed methane (CBM) production operations. By employing a production process called water-alternating-gas (WAG), the amount of produced water from CBM operations that are discharged to surface drainages could be dramatically reduced.

We proposed to investigate the prospect of re-injecting the produced water (common to most CBM reservoirs) back into the same formation along with a gas in an alternating sequence to economically reduce surface water discharge and maintain an economical methane production scheme. We proposed to investigate these by integrating laboratory experiments and measurements, reservoir simulation, economic analysis of field implementation, and field design.

## Project Summary

Laboratory experiments were done using fresh coal blocks collected from Powder River basin surface coal mines. The intent of the experiments was to generate data regarding desorption of methane from coal during successive slug-injections of nitrogen and water. Two-dimensional floods were designed to mimic a quarter 5-spot field pattern. We cut ½-inch blocks of coal and arranged them to form a two dimensional volume that was 1/2-inch thick by 6.5 inches long and 6.5 inches wide. We coated the outside surface of the miniature coal seam (cut coal blocks) with a non-sag epoxy that did not enter into the very small spaces between the

blocks to allow fluids to pass through. The epoxy-coated block was made with an inlet port, an outlet port, and three other ports to measure in situ pressures; the entire epoxy-sealed block was then placed into a pressure containment vessel, which was subsequently filled with pressurized water to contain the pressure within the epoxy-coated coal block.

Funding for the project in 2010 was dramatically cut, so not all project goals were completed. The original hypothesis stated that by alternating inert gas injection with water injection, a more efficient sweep of the adsorbed methane gas could be accomplished and that this process would be an economic alternative to the current practice of depressurization-desorption and water disposal. We are currently at the stage of experimentation to evaluate the efficacy of the alternating injection scheme, so no hard conclusions can be drawn at this time.

## Benefits to DOE

The research has led to numerous discussions with colleagues from interested parties including the University of Wyoming, Anadarko Petroleum, and mining companies where the coal was collected. A proposal written jointly with Anadarko Petroleum was submitted to the *Research Partnership to Secure Energy for America* (RPSEA) forum (funded by the Department of Energy-Office of Fossil Energy [DOE-FE]). Discussions and resulting proposals such as these have kept Idaho National Laboratory and DOE current on industry needs and provided opportunities for future external funding.

Idaho National Laboratory

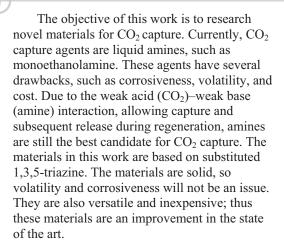
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# Triazine-Based CO<sub>2</sub> Capture Agents

Michael Timothy Benson

Investigating novel materials for CO<sub>2</sub> capture

ST132



## Results and Accomplishments

After elucidating an appropriate synthetic scheme for the synthesis of the materials, several promising materials were generated. A variety of protecting groups were initially investigated to protect the primary amines while the pendent groups were attached to the triazine. Without protecting groups, the primary amines crosslink, forming an insoluble polymeric structure. The protecting groups were unsuccessful, though, likely due to steric factors. Protecting groups tend to be large and bulky, and with six protecting groups required, there is not enough room around the triazine ring. Instead of a protecting group, nitrile compounds were utilized and then hydrogenated after pendent

group attachment. The materials synthesized are triazine rings with 3 pendent groups, either diethylenetriamine (Material 1) or dipropylenetriamine (Material 2), bonded through the center nitrogen. CO<sub>2</sub> capture for Material 1 was found to be 1.90 mol CO<sub>2</sub>/Kg

sorbent; for Material 2, CO<sub>2</sub> capture was 1.37 mol CO<sub>2</sub>/Kg sorbent. The commercial ethanolamines, such as monoethanolamine, capture CO<sub>2</sub> at 0.85 mol CO<sub>2</sub>/Kg sorbent, so a large improvement has been made in CO<sub>2</sub> capture. Materials 1 and 2 are not optimized, thus a higher CO<sub>2</sub> capacity may be attainable. To explore the mechanism, the phenyl derivatives have also been synthesized, specifically to investigate the need for the triazine nitrogens. The data indicates the triazine nitrogens are required for higher CO<sub>2</sub> capacity, although without the primary amines present in the system, the triazine nitrogens are ineffective at capturing CO<sub>2</sub>. Several possible mechanisms have been proposed for CO<sub>2</sub> binding to the triazine materials.

### Benefits to DOE

The cleaner energy production effort, especially lowering  $\mathrm{CO}_2$  emissions, continues. The development of new, more efficient  $\mathrm{CO}_2$  capture agents will benefit energy resources, improve environmental quality, and lower the cost of electricity.

DOE's Office of Energy Effiency and Renewable Energy (EERE), specifically through the Industrial Technologies Program (as one example) is interested in carbon capture/sequestration, especially concerning the cost and efficiency in power plants.

The DOE Office of Fossil Energy has a large effort in carbon capture and sequestration, such as The Carbon Sequestration Leadership Forum, the Regional Sequestration Partnerships, the FutureGen Initiative, and the Carbon Sequestration Core Program.



## **Near-Field Impacts of In-Situ Oil-Shale Development on Water Quality**

Thomas R. Wood, Carl Palmer, Hai Huang, Earl Mattson
Controlling and monitoring groundwater quality during oil shale development
ST134

Oil shale in the arid western states contains vast quantities of kerogen that could provide oil to significantly reduce the United States' dependence on foreign oil. However, many technical challenges remain to be solved before this world-class resource can be developed. including significant environmental concerns related to water. Available data to evaluate impacts to water resources are decades old and were collected under conditions that do not represent new oil shale in-situ technologies. Most current approaches are based on slow heating for long time periods at temperatures between 260 and 375°C to convert kerogen to oil. Formerly, oil-shale development involved fast heating to temperatures much greater than 600°C, producing different and often more hazardous waste products. Groundwater models of the impact of oil-shale retorts on aquifer water quality will require knowledge of the contaminant fluxes, which are primarily controlled by the concentration of contaminants at the source and the permeability of the retort zone. The objectives of this research are to understand the oil and gas produced and their associated contaminants, to determine the permeability during and after in-situ retorting. and to develop modeling and simulation tools to evaluate impacts to groundwater. Through careful laboratory testing of oil shale, we are defining the relationships between leachable contaminants and the applied heat and pressure. Additionally, we are developing quantitative relationships among permeability, retort heating history, and effective stress. These relationships are being incorporated into heat-flow models that will allow us to characterize the permeability field immediately surrounding the retort zone.

Project Summary

Controlling and monitoring groundwater during development is a key challenge for bringing unconventional fossil-fuel resources online to help secure the energy future for the United States (GAO, 2007). This proposal supports the DOE mission to accelerate development while simultaneously protecting groundwater resources. We will provide Idaho National Laboratory/ U. S. Department of Energy (INL/DOE) with capabilities for environmental protection of water resources by

creating the scientific models required to understand changes in groundwater flow fields and predict contaminant release and migration during and after oil-shale retorting. These understandings, when coupled with INL cutting-edge computational tools, will provide stakeholders with a means of evaluating potential impacts on groundwater and designing appropriate monitoring systems. In addition, the potential for industry collaboration is high, as the end product will be beneficial for facilitating successful permitting of developments.

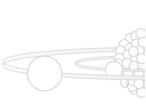
Our research is closely aligned with the Palmer, et al. (2007) LDRD, which is evaluating the character of shale oil recovered by modernday lower-temperature in-situ processes. The Palmer LDRD evaluates oil produced from oil shale heated across a range of temperatures, pressures, and water saturations. The waste material, or spent shale, from their experiments provides an ideal source of material for our leach tests and permeability tests of spent shale. Furthermore, equipment is shared between the two research projects. In addition to characterizing the leached contaminants from spent shale, this LDRD measures the permeability of oil shale that has been heated to temperatures below pyrolysis (the temperature required to produce oil from oil shale) to characterize the groundwater flow field adjacent to the retort chamber. Our project tasks are listed

Task 1: Obtain oil shale – Completed.

<u>Task 2</u>: Identify and determine concentrations of potential contaminants – *Completed for all oil-shale samples under various retort-heating scenarios, needed for environmental transport source terms.* 

<u>Task 2a</u>: Analysis of retort water and air fractions – *Completed. CO<sub>2</sub> was added to the original work scope, which was needed for an air source term transport model we presented at the Oil Shale Symposium.* 

<u>Task 2b</u>: Analysis of spent oil shale – Completed. Several additional specialized tests yielding data that was required for understanding long-term potential of abandoned retort to act as source to ground water completed as well.





<u>Task 2c</u>: Leach tests on spent oil shale after flushing – *Completed. The results of these tests are critical for ascertaining long-term environmental impacts.* 

<u>Task 3</u>: Permeability of oil shale – *Completed.*Several of these experiments were conducted to assess the evolution of fracture permeability with heating, which are needed for modeling near field release to aquifers.

<u>Task 3a</u>: Measure permeability of spent and unspent oil shale – *Completed. The results are critical for groundwater, air-flow, and transport simulations.* 

<u>Task 3c</u>: Measure changes in fracture permeability with temperature – *See Task 3 above*.

#### <u>Task 4</u>: Modeling – *Completed*.

<u>Task 4a</u>: Fracture heat flow and geotechnical model – *Completed. This model is critical for understanding how tightly coupled processes evolve and affect contaminant transport during retort heating and post abandonment. The GeoFracFH code has been copyrighted.* 

#### Results and Accomplishments

Several dozen oil-shale retorting experiments have been run this year to evaluate the toxic material produced by in-situ oil shale retorting and the toxicity of the spent oil shale remaining after retorting. The FY-2010 work advanced the laboratory testing initiated in FY-2008, primarily using instruments designed and built in FY-2008 and FY-2009. Experimental conditions were systematically varied in terms of maximum heat achieved during retorting, the duration of the retort, the grade (gallons per ton) of the oil shale ore, and the water content of the retort experiment (hydrous) to quantify relationships between retort conditions and contaminants produced. A suite of carefully controlled experiments using spent oil shale were also conducted to evaluate the chemical transformations, leaching characteristics and the environmental water quality impacts from in-situ oil shale retorting. A data set has been created that characterizes contaminants released under various retort conditions.

Retort experiments were conducted using intact cores of oil shale under compressive stress to evaluate the role of fractures and other geomechanical transformations on the mobility of contaminates at the boundary of in-situ retort zones. These experiments required engineering and machining specialized core holders to hold the core under confining pressure as a means of simulating in-situ conditions of pressure. Additional experiments were conducted evaluating how fracture permeability evolves during heating due to plastic deformation of the oil shale. A surrogate fracture was created with a known permeability, placed in the core holder, and then retorted. After retorting, the permeability of the surrogate fracture decreased by three orders of magnitude due to plastic deformation/annealing of the fracture. This has important implications for contaminant transport by ground water away from spent-shale zones.

Laboratory experiments were conducted for the explicit purpose of considering vapor or gas transport to the atmosphere from in-situ oil shale retorts. These experiments were similar to the water phase experiments, with the addition of a sorption test of produced gases by raw oil shale. Basically, the exhaust gases were directed into a chamber filled with chipped oil shale material and allowed to equilibrate. Samples of the gas from the chamber were compared to samples from the retort to evaluate if sorption of contaminants will occur on the surrounding native rock. The results of the laboratory experiments will serve as input to a vadose-zone transport model and ultimately an air-transport simulation, which will be presented at the 29th Oil Shale Symposium.

The modeling and simulation activity has continued to advance the now-copyrighted computer code, initiated in FY-2008, by including plastic deformation and adding parallel computing capability. The GeoFracFH Model is able to simulate heat, fluid flow, and geomechanical rock deformation during in-situ retorting. Of considerable importance is the fact that these processes are very tightly coupled, and this code accurately simulates the feedback between heat, geomechanical deformation (fracturing), and fluid flow. This capability is not available from any other source, and it is



required to simulate the evolution of rock permeability and porosity with heating, a critical environmental impact to groundwater. A brief summary of the advancements made to the

The GeoFracFH Model is a particle-based discrete-element model (DEM) that has been coupled with fluid flow and heat conduction/convection. The rock matrix material is represented by a network of DEM particles connected by mechanical bonds. During the simulation process, the mechanical bonds that have been stretched or bent beyond a critical strain – both tensile and shear failures are simulated – are broken and removed from the network in a progressive manner. Bonds can be removed from the network with rates or probabilities that depend on their stress or strain, or the properties of the discrete elements and bonds can be varied continuously to represent phenomena such as creep, strain hardening, and chemical degradation. Darcy flow and heat transport equations are solved on an underlying fixed-finite difference grid with evolving porosity and permeability for each grid cell that depends on the local structure of the discreteelement network (such as the DEM particle density). The fluid-pressure gradient exerts forces on individual elements of the DEM network, which then deforms and fractures the rock matrix. The deformation/fracturing, in turn,

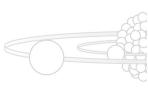
factor for investigating the potential

model in FY-2010 is provided below.

changes the permeability, which again changes the evolution of fluid pressure, thereby coupling the two phenomena. The intimate coupling between fracturing, fluid flow, and thermal transport makes the GeoFracFH model, rather than conventional continuum mechanical models, necessary for coupled hydro-thermalmechanical problems in the subsurface.

#### Benefits to DOE

This LDRD has been successful on several fronts: (1) adding a technical capability to the INL in line with DOE's energy security mission, particularly with the development of unconventional fossil energy in the western U.S.: (2) opening a new market niche for the INL in unconventional strategic fuels (important under the Energy Act of 2005) that is highly leveraged with several ongoing and new national, regional, and local efforts; (3) generated and disseminated new scientific findings and results that aid in understanding the environmental impacts from proposed energy developments; (4) elevated the technical reputation of the INL in the oil shale industry; and (5) made significant contributions to helping the DOE solve the nation's daunting problems with respect to imported fossil fuels. Many industry and university relationships have been developed over the year. Two competed research proposals have been awarded and two industry contracts are in place as a direct result of this LDRD.



## Dynamic Impact Model and Information System to Support Unconventional Fuels Development

Randy Lee, Dan Ames (Idaho State University)

Modeling the dynamic impact of unconventional fuel development

ST133

Development of unconventional hydrocarbon resources can enhance the energy security of the United States, with substantial resources in the Mountain West. For example, it is estimated that the equivalent of 1.5–1.8 trillion barrels of oil exist in oil shale deposits of the Uinta, Green River, and Washakie Basins. Undoubtedly, locating, extracting, and processing these resources will draw people to the area and increase demand on water. Therefore, development of unconventional fuel sources, such as oil shale, commonly face two principal challenges: (1) environmental, the most salient of which centers around water and (2) socio-economic, as it relates to rapid growth and development in rural areas. Each of these challenges has a broad range of temporal and spatial scales with their solutions being highly interdependent upon one another due to the interconnectedness of the entire system. Resource managers and stakeholders need tools and methods to identify, understand, anticipate, and mitigate the potential health and environmental effects of unconventional fuel development.

#### Project Summary

The objective of this research is to develop a dynamic impact modeling system to enable resource managers and stakeholders to understand and predict the potential environmental, social, and economic consequences of unconventional fuel development on the western United States across both spatial and temporal scales (Fig. 1). The foundation of this objective is to integrate existing and proven environmental models into a sophisticated state-of-the-art decision support system that will provide better and more accurate predictions by allowing the data from each model to update the other. The system will contribute to a better understanding of the interdependencies between natural and human systems and allow extensive evaluation of potential management options. A key capability is to provide a virtual test environment where the user will interact with a time-based simulation and visually perceive the consequences and effects of various management options across the landscape. The initial focus of this research was

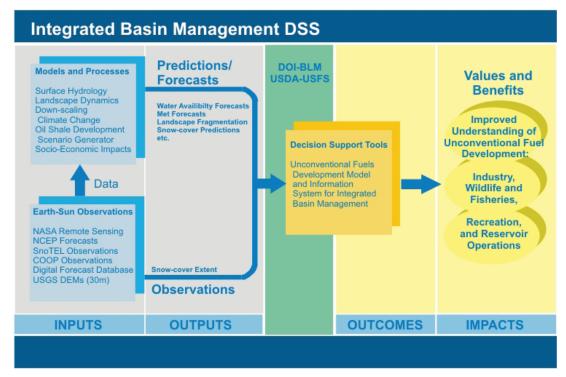
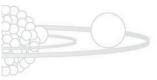


FIGURE 1. Integrated modeling systems approach.



centered on the availability of water resources and the impacts associated with this resource by developing an oil shale industry in the Piceance Basin in Northern Colorado.

#### Technical Objectives

Technical Objectives for fiscal year 2010 (third and final year of funding):

- Develop integration framework
- Build connection between ArcGIS tools and Powersim system dynamics modeling tool
- Integrate Powersim model from Laboratorydirected Research and Development (LDRD) project ST139 (Water and Energy System Interdependency Modeling for Multicriteria Decision Analysis – Robert Jeffers)

#### Results and Accomplishments

During the first several months of Fiscal Year 2010, a demonstration application was developed that enabled Powersim to run inside ESRI's ArcMap GIS software. The code was developed by ISU under subcontract. This was the first step in developing a framework for

performing system dynamics modeling using inputs directly from a geographic information system (GIS) application, and visualizing the results on a map interface.

The project was halted during the fourth month of the year due to a manpower shortage that would have precluded us from finishing the tasks for the year. Work did continue under the subcontract with ISU on connecting the existing system dynamics model in LDRD ST139. This work included using GIS tools to further enhance the model by developing precipitation data from PRISM (a precipitation and temperature model for the U.S. over the past four decades).

#### Benefit to DOE

The project realized the development of a connection between a GIS and system dynamics model, an accomplishment that had not been achieved before. Unfortunately, the project did not reach all goals due to a premature end. The accomplishments of this project have proven very useful for LDRD ST139 and will enable that project to make significant contributions to the water energy future of the U.S.

Idaho National Laboratory

100

## **Advanced Remote Sensing for Energy and Environmental Applications using Unmanned Aerial Vehicles**

Matthew O. Anderson, Ryan C. Hruska Utilizing UAVs for safe and efficient data collection and processing

This research project's two primary objectives are targeted at advancing the state-of-art in environmental remote sensing, which can be applied directly to watershed management applications using unmanned autonomous vehicles.

Project Summary

The first objective is to develop an unmanned air vehicle (UAV)-based hyperspectral imaging (HSI) system that will support current and projected needs of land management agencies and private industry needing rapid, low-cost, and effective environmental monitoring and mapping systems. The second objective is to integrate intelligent autonomous path planning and collision avoidance technologies into the UAVs to allow for optimized and safe operations throughout national airspace. The research is aimed at providing peer-reviewed scientific knowledge that will enhance Idaho National Laboratory's (INL's) work with programs and agencies focusing on utilizing UAVs for safe and efficient data collection and processing.

Results and Accomplishments

Year 1 and 2 objectives, which have been met, achieved the following:

- Hyperspectral sensor selection, calibration, and ground testing (NovaSol HSI vs. Resonon)
- Arcturus UAV T16 airframe characterization and flight readiness
- Sensor and subsystems integration into the UAV airframe
- Flight tests over control field using groundbased spectral measurements to validate the spectral quality of airborne image data
- Hyperspectral imagery of the UAV Test Area and control field
- Development of image processing methodology (radiometric corrections, spatial corrections, image classification, sensor fusion, etc.)
- UAV hyperspectral data collection capability applied to real-world problem of nitrogen content in sagebrush

- Identification and development of Sense & Avoid methods for UAV data collection
  - Researched current Federal Aviation Administration (FAA) directives on sensing requirements
  - Developed sensors and architecture
  - Developed on-board intelligent system
  - Performed (?) hardware in-the-loop simulation and ground-based testing with the goal to autonomously modify flight characteristics based upon environmental conditions
- Development of strategic partnerships with industry and university researchers.

INL and its partners have successfully demonstrated an affordable and organic (personally owned; easier to acquire data compared to manned or satellite HSI acquisition) remote sensing airborne solution and advanced obstacle avoidance capability. INL is becoming nationally recognized as a viable Department of Energy UAV research center with subject matter experts and resources. This year's research studies of FAA safety needs and technologies that meet these needs have allowed the INL to demonstrate our commitment to safe UAV operations while still developing advanced research strategies.

It is noteworthy that just having conducted the aforementioned calibration test flights has resulted in the request of hyperspectral data by ISU Geosciences and Biology Departments as well as Stoller LLC. Meetings have been held at INL with the Bureau of Land Management and U.S. Forest Services. Also of significance is that hyperspectral sensor vendor Resonon Inc. included INL as a partner in their Phase II Small Business Innovation Research (SBIR) program, resulting in INL receiving their \$75,000 nearinfrared sensor for this year's flight operations; also, Guided Systems Technology received \$300,000 in collaborative research funding from the Air Force Research Laboratory. The Robotics and Intelligent Systems Department is seeing an increase in UAV-related projects targeted at using the new T16-based UAV with increased payload capacity and operational flight times and the hyperspectral sensors associated with this project.

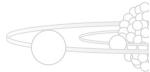




#### Benefit to DOE

A major element of the DOE and INL mission is to ensure productive and optimal use of our nation's energy resources in an environmentally sensitive manner. As a result INL's Energy and Environment Directorate has a vision of providing research leadership in clean energy options with a focus on providing science and engineering-based solutions to meet the Nation's environmental and energy/water resource management challenges. The development of advanced remote sensing capabilities utilizing unmanned aerial vehicles would contribute to a wide range of research

areas supported by DOE and INL. By performing this proposed research, INL would have the capability to provide a platform to collect data needed by scientists in order to understand and monitor complex/changing environmental issues related to energy management. Also the successful integration of intelligent collision avoidance technologies would potentially enable UAVs to be deployed into the national airspace, significantly growing research and funding opportunities with NASA, USFS, Bonneville Power Administration, DHS, and other asset and land management agencies.



### Addressing the Spectrum of Nuclear-Related NDE Needs

David Hurley, Zhandos Utegulov

Development of laser ultrasonic techniques to monitor the onset of melting and ablation in metallic materials

ST136

This laboratory-directed research and development (LDRD) project involves the development of the theoretical and experimental underpinnings of a hybrid nondestructive examination (NDE) approach that incorporates both laser ultrasound and eddy current inspection. A large portion of 2009's effort involved the development of novel eddy current inspection techniques that utilize giant magneto-resistive devices. This year's effort focused on the development of laser ultrasonic techniques to monitor the onset of melting and ablation in metallic materials. This approach has diverse applications ranging from localized determination of melting point to laser cleaning of surfaces. For NDE applications, identification of the ablation threshold is important for determination of the onset of material damage.

#### Project Summary

To investigate the onset of melting, we examined the laser ultrasonic waveform that propagates along the epicentral direction. The experimental setup is shown in the top left of Figure 1. A pulsed Nd:YAG laser is used to excite ultrasonic waves in a bulk tungsten sample. A photorefractive laser interferometer operating at 532 nm is used to detect out-of-plane surface motion associated with ultrasonic waves. The bottom left of Figure 1 shows typical epicentral waveforms with laser fluence as a parameter. The longitudinal and shear waves are clearly visible. The arrival time of the longitudinal wave relative to the trigger pulse does not change; however, the arrival time of the shear wave is delayed with increasing pump fluence above 135 MW/cm<sup>2</sup> (top right of Fig. 1). We believe this corresponds to the onset of melting. This phenomenon can be understood by considering generation conditions in detail. For thermoelastic generation, mode conversion of longitudinal waves at the free surface is responsible for the shear wave component of the signal. For a laser fluence above the melting threshold, there is a small melt pool located in the center of the generation volume. Longitudinal waves emanating from subsurface

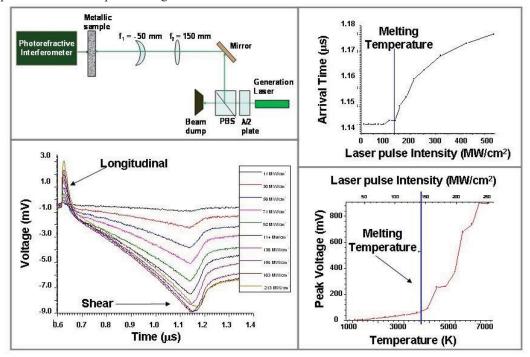


FIGURE 1 Top left: Experimental setup. A pulsed Nd:YAG laser is used for ultrasonic generation and a laser interferometer is used to detect out-of-plane surface motion associated with ultrasonic waves. Bottom left: Epicentral waveforms with generation laser fluence as a parameter. Top right: Arrival time of the shear wave versus generation laser fluence. Bottom right: Photodetector peak voltage due to white light generation versus surface temperature.

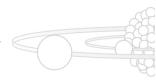
sources cannot mode convert upon reflection because the molten pool cannot sustain shear waves (i.e., zero shear modulus). As a result, there is no shear wave generation in this region of the generation volume. Thus shear waves emanate from an annular thermoelastic source that surrounds the melt pool. The propagation distance from these off-axis thermoelastic sources is larger than for the sources along the epicentral axis; hence, the arrival times will be greater. This results in weighting more heavily shear waves that arrive from the periphery, causing the shear arrival to move out in time.

To investigate the onset of ablation, we examined the white light generation due to plasma excitation. The white light intensity, which is proportional to the photodetector voltage, is shown in the bottom right of Figure 1. Below ablation there should be not white light generation. However, the data suggest that white light is being generationed and scales linearly with pump fluence in this region. We speculate

that this is due to preferential absorption at surface defects caused by polishing. As the pulse energy increases above the ablation threshold for tungsten, there is a rapid increase in white light generation due to radiation from the highly excited plasma. The change in functionality at  $\sim 3000 \text{ K}$  clearly illustrates the onset of ablation.

#### Benefits to DOE

The work conducted this year involved developing new laser ultrasonic capabilities at INL. Laser ultrasound is envisioned as a key NDE tool for next-generation nuclear energy systems and presently is an under-utilized tool for the current light water nuclear fleet. The work conducted under this LDRD has helped our group secure funding from the light water sustainability reactor program to investigate the efficacy of laser ultrasound to characterize and monitor the integrity of advanced SiC fuel cladding material.



## Water and Energy System Interdependency Modeling for Multicriteria Decision Analysis

Robert Jeffers

Developing a hydrologic model of water supply to couple with sector-specific electricity supply and demand

ST139

The primary initial hypothesis is that hybrid energy systems, when coupled with an appropriate geographic resource base, will provide added resiliency in the face of climate variability and/or economic instability over their traditional energy technology counterparts. Within the past year, hydrofracturing technologies have been heavily exposed as a potential option for keeping natural gas prices low. We have completed an initial literature review of the potential futures for natural gas and conclude that no prediction can be made regarding the future speed of hydrofracturing growth. In addition, we have completed the initial phase of a hydrologic model that successfully predicts streamflow with precipitation and temperature as the only inputs. Therefore, we have refined our proposal to investigate the effect of variable natural gas prices in addition to variable wind and water availability on the resiliency of a hybrid energy system involving these three technologies.

#### Project Summary

Work began with evaluating the available options for creating an energy-water system modeling framework. We decided that modeling work would initially progress using the Powersim and AnyLogic software in parallel, since models can be ported between the two tools. In addition, we concluded that an in-house geographical information system (GIS) tool would be used as the framework's interface. Powersim and the GIS client have been successfully linked with a database, which provides spatially explicit, temporally dynamic modeling capability of both water and energy systems. In addition to tool development, preliminary analysis was accomplished on the hydrologic system. The Upper Snake River Basin (USRB) is being used as a proof-ofconcept region. Models of the major reservoirs and watersheds in the USRB were developed, calibrated, and successfully linked, which provides insight into potential operational procedures under climate variations. Work to select and model hybrid energy systems is ongoing, and initial feedback points to a windhydro-natural gas coupled system.

#### Benefits to DOE

The research has led to interest from the Deputy Attorney General of Idaho to develop consensus (and avoid litigation) among the major stakeholders in Idaho's water resources. A letter of support has been signed by the stakeholders and a proposal is being developed to create consensus-building models of the USRB with INL as the technical lead. The process is already benefiting these stakeholders by bringing them together to talk about their shared water resource management issues.

Internally, this research funded an undergraduate internship, in which the student became well versed and excited about both system dynamics modeling and water resource issues. A subcontract with a recent M.S. student at the University of Idaho was also extended for the purpose of merging groundwater, surface water, and irrigation sub-models into a coupled model.

The coupling of GIS and system dynamics tools, with the addition of agent-based modeling methods and dynamic database integration, is a cutting-edge framework that provides a highly manageable spatial-temporal modeling suite. This combination of toolsets into a modeling framework is expected to provide a marketable capability in all areas where multiple scales of temporal and spatial dynamics are at work. Specific to the energy-water field, the hydrology sub-models that have been developed using the framework combine simplicity and accuracy and are in the process of being tested against results from other monthly water balance models. The hydrology model is expected to be unparalleled due to its low data requirements and ease of coupling with other systems. Finally, we have undergone further development with a Flex GIS web-based client that combines both speed and customizability to create stunning and insightful user interfaces to system dynamics models.



## **Degradation and Conversion of Lignin using Extremophilic Systems**

William Apel

The goal of this project is to assess the use of extremophilic systems for the bioconversion of lignin to feedstocks and value-added products

ST140

The objective of the proposed research is to demonstrate the presence and activity of bacterial lignin degradation enzymes from acidic and alkaline thermal environments and to isolate and characterize the activity of these enzymes related to enhanced biomass utilization and processing and production of aromatic building block molecules. An interdisciplinary approach consisting of general microbiology, enzymology, genomic (metagenomic), proteomic, and metabolomics will be used to determine the presence and activity of lignin-degrading enzymes in thermal features in Yellowstone National Park. A combination of these approaches for enzyme discovery will be used to ensure that enzymes from both cultivable and uncultivable bacteria are represented. Completion of this objective will lead to the isolation of industrially important thermoacidophilic enzymes for lignin degradation. In addition, depending on the activity of these enzymes, mechanisms to produce aromatic monomers and acids that can be "building blocks" for production of other value-added chemicals.

#### Project Summary

- Hot Spring Bioprospecting
  - Wood, sediment and water samples were taken from Elk Geyser, a hot spring with temperatures between 70 and 80°C and a pH near 3.5.
  - Water, sediment cores, and plant biomass samples were taken from Mickey Hot Springs, a feature with temperatures >70°C and pH near 9, in the Alvord Desert.

#### Microbial Enrichment

- Two thermoacidophilic cultures able to use Kraft Lignin for growth have been enriched from Elk Geyser wood samples. These cultures are able to grow at 70°C and pH 3.5.
- Four thermoalkaline cultures able to use Kraft Lignin for growth have been isolated from Mickey Hot Springs. These cultures can grow from 70 to 80°C at pH 9. Extracellular laccase production was demonstrated in these cultures, shown by the decolorization of remazol brilliant blue R dye.
- Metagenome Analysis
  - Microbial DNA was extracted and amplified from Elk Geyser and Mickey Hot Springs samples and will be used for sequencing metagenomes.
  - A subcontract was placed with Eurofins MWG|Operon for sequencing.

#### Benefit to DOE

Strategically, this project will continue to develop systems biology expertise at Idaho National Laboratory. This laboratory-directed research and development project will also expand our expertise in extremophilic enzymes related to the processing of biomass for fuel and chemical feedstocks, which supports the Department of Energy's energy security mission. In addition, the research is supporting a Master's of Science student at Montana State University.

Idaho National Laboratory

106

## Metabolic Engineering of *Alicyclobacillus acidocaldarius* for Lacit Acid Production from Biomass-derived Monosaccharides

Brady Lee

An interdisciplinary approach to develop a genetically modified strain of the gram-positive thermoacidophile for production of value-added chemicals GB104

The objective of this research project is study the ability of an industrially important gram-positive thermoacidophile to generate lactic acid from pentose and hexose sugars. We are accomplishing this objective by taking an interdisciplinary metabolic engineering approach, which combines aspects of genetic engineering, bioprocess engineering, and metabolomics to develop a genetically modified strain of the gram-positive thermoacidophile for production of value-added chemicals.

Results and Accomplishments

#### Metabolic Flux

- Established growth characteristics and optimized chemostat parameters for optimum growth of test organism on xylose and glucose under carbon- and nitrogen-limited conditions.
- Ran numerous chemostat experiments to determine effect of carbon- and nitrogen-limitation on the metabolic footprint of the gram-positive thermoacidophile.
- Determined that while small amounts of ethanol and organic acids such as lactate are produced during growth, production of a currently unidentified extracellular material appears to be the primary metabolite when growing on xylose or glucose.

#### Metabolic Modeling

Continued to refine and test metabolic model developed during previous fiscal years using elementary flux mode analysis. Through this research it was determined that the gram-positive thermoacidophile has 3,865 unique elementary modes when grown on glucose, whereas only 1,871 modes are available when grown on xylose.  Determined that the gram-positive thermoacidophile does have the potential to grow anaerobically, but would require expression of specific enzymes and production of organic acids or alcohols as redox sinks.

#### • Metabolic Engineering

- Determined that triparental mating between *Bacillus subtilis* and the grampositive thermoacidophile is one potential method of transforming this microbe.
- Initiated construction of an E. coli/B. subtilis shuttle vector for use with gram-positive thermoacidophile.

#### Benefit to DOE

Strategically, this project will continue to develop systems biology expertise at Idaho National Laboratory. This laboratory-directed research and development project will also expand our expertise in extremophilic enzymes related to biomass processing for fuel and chemical feedstocks, which supports the Department of Energy's mission of energy security. In addition, the research has supported eight summer students and is now supporting a Master of Science student in Microbiology at Idaho State University.



## Network Interaction in the Thermoacidophile *Alicyclobacillus* acidocaldarius

Brady Lee

Focus on carbohydrate active proteins to explore Alicyclobacillus acidocaldarius saccharide use, with attention to regulation of gene expression GB105

The goal of the proposed research is to understand carbohydrate-induced differential gene expression patterns by this gram-positive thermoacidophile during growth on a range of carbon and energy sources. Understanding the effect of carbon catabolite repression (CCR) on regulation of genes during growth on different carbon sources, and the ability to control this regulation, represents the first step in optimizing growth and/or enzyme production. The research approach will be targeted toward carbohydrate active proteins to explore issues related to use of saccharides by this bacterium, with a focus on regulation of gene expression.

Results and Accomplishments

- Genome Analysis
  - Continued in silico analysis of gene and operon structure for carbohydrate-related gene elements
  - Identified sequences (catabolite responsive elements) in promoter regions that allow CCR to occur.
- Analysis of CCR
  - Ran chemostat experiments to determine effect of glucose in establishing CCR in thermoacidophile of interest

 Extracted ribonucleic acid (RNA) for expression analysis using microarrays and quantitative polymerase chain reaction (qPCR).

#### Network Interaction

- Added task to begin looking at protein-DNA interactions. Binding of catabolite control protein A (CcpA) to promoters will be assayed using electrophoretic mobility shift assays and DNase footprinting studies.
- Began developing network interaction models using Cytoscape.

#### Benefits to DOE

The proposed research will develop expertise in systems biology at the Laboratory. This expertise will then be available for projects relating to all aspects of microbiology, including bioenergy, environmental, homeland security, and even medical microbiology. This research also will be used in support of a doctoral dissertation for the principal investigator.

### Specific Biological Responses to Nano Metal Oxides

James Henriksen

Laying the foundations for the development of useful assays of biological responses to engineered metal oxide NP

ST141

Nanoparticles (NP) are researched, developed, and manufactured by several Department of Energy (DOE) national laboratories. Industrial use of NP is growing rapidly, which comes with the potential for accidental or weathering release to the environment. A diverse array of synthetic NP exists, but little is known about their environmental impacts. A need for monitoring and screening technology and an improved understanding of the potential toxicity, transport, and fate of NP in the environment exists. The purpose of this project is to evaluate the biological response to engineered metal oxide NP. We hypothesize that the formation of intracellular reactive oxygen species (ROS) is a common mechanism underlying the response of biological systems to metal oxide NP. The whiterot fungus Phanerochaete chrysosporium and the soil bacterium Pseudomonas putida are model systems with environmental relevance that provide molecular tools, known responses to ROS and metals, initial evidence of NP effects, and unique biological characteristics to test these responses. We will examine biomolecular markers of ROS and metal response during treatment of both types of cells with ROS, bulk metal oxides, and well-characterized NP. The comparison of these responses will provide the scientific underpinnings for novel methods to detect bioavailable NP in complex environmental backgrounds, and identify characteristics and concentrations that indicate potential ecotoxicity. The characteristics of the NP following interaction with the biological cultures will also be evaluated in order to better understand the potential impacts on their subsequent environmental fate and transport.

#### Project Summary

Since the beginning of the project in March 2010, we have laid the foundations for the development of useful assays of biological responses to engineered metal oxide NP. In the areas of work procedures, NP characterization, fungal enzyme assays, and bacterial response to ROS, a base capability has been established. An initial suite of NP was selected and procured for research that vary in size, composition, and predicted ROS generation. Initial characterization of the NP has been performed,

and florescent indicators of ROS generation have been investigated. A series of experiments demonstrated that an enzyme of P. chrysosporium was differentially regulated under different ROS stresses that are potentially representative of NP levels of ROS generation. A series of experiments have been performed with the metal resistant bacterium P. putida that demonstrate ranges of ROS that the organisms can withstand. Initial toxicological ranges of ROS reactive NP were investigated. The NP characterization showed that while most NP had the expected characteristics, there was a good deal of agglomeration in many of the NP under the conditions that they will be used in biological assays and might be encountered in the environment. The initial florescent indicator selected to investigate the ROS generation properties of the NP in biological conditions was tested, and while providing adequate sensitivity at pH 7 for the bacterial systems, it was shown to have inadequate sensitivity to determine the NP ROS generation in the lower pH of fungal growth conditions. Other compounds are being investigated as replacements. The hypothesis that an enzyme activity of P. chrysosporium increases with increasing ROS stress was shown to be correct. Current experiments will determine the sensitivity range of this response, and the range over which this effect would be useful to detect the biological impact of ROS producing NP. A hypothesis that one of the most ROS active NP would be toxic to the bacterium P. putida at low levels even without light exposure was disproven, creating opportunities to investigate light-induced toxicity and the more sensitive genetic biological responses discussed in the proposal.

#### Benefits to DOE

We are benefiting the DOE missions by establishing a research program that will provide valuable information to industry and government for an improved understanding of the potential toxicity, transport, and fate of NP in the environment. This work supports the "Environmental Impacts of Energy" goal of the Energy Security theme. To speed adoption of nanomaterials developed for energy uses (such as fuel cells and catalysts in unconventional oil recovery or hydrogen storage), their safety must



environmental contaminant fate and transport, which focuses on nano- and biomolecular materials. DOE benefits from the establishment of research in this area of environmental, health, and safety (EHS) impacts of NP, as EHS impacts of NP are a funding priority of the cabinet-level National Science and Technology Council

(NSTC).

be ascertained. This work supports the environmental quality goal by increasing the understanding of the toxicology, fate, and transport of materials by providing information and tools to guide research and regulation in the developing field of the environmental impacts of nanomaterials. This work supports the BER objective to increase understanding of

### **Development of Non-Lethal Methods for Enhanced Lipid Recovery from Microalgae**

Deborah Newby

Developing an array of non-lethal methods for enhanced lipid recovery from algae ST142

This laboratory-directed research and development (LDRD) project, which was initiated at the end of March, was designed with the intent of adding an algal component to Idaho National Laboratory's (INL) renewable energy/alternative fuels portfolio. The focus is on the development of an array of non-lethal methods for enhanced lipid recovery from algae. Physicochemical milking and molecular/systems biology approaches to understanding algal lipid metabolism and excretion are being developed and employed. The end objective of this work is to exploit the physiological responses of microalgae to stress/shock conditions in order to improve the economic feasibility of production and recovery of lipids from microalgae.

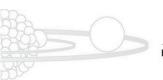
#### Project Summary

Algal biology in general is a new area of research for INL, so significant time and resources were allocated over these first 5 months to acquire and set up appropriate equipment and bioreactors and to become familiar with the different growth characteristics of our model algae: *Chaetoceros gracilis*, a diatom and USU-80, a green algae.

DNA from C. gracilis has been isolated, purified, and is currently being sequenced via a subcontract. Much effort was required to enrich and separate the C. gracilis from bacteria that it requires for growth prior to extracting the DNA. The sequence will be analyzed and will provide the foundation for the development of a microarray next year. This microarray will enable us to examine gene expression of C. gracilis under different growth and milking conditions, providing insight at the molecular level into lipid production and exocytosis (release of the lipids). Studies looking at lipid production and exocytosis under different salt concentrations and in the presence of a biocompatible solvent have been initiated. Small aliquots of unstressed USU-80 were subjected to preliminary milking experiments employing methylene chloride as the organic extractant. Algae were exposed to solvent for different time periods, organic solvent was removed by rotary evaporation, and culture returned to fresh media for viability testing. All cultures grew, but limited lipids were recovered, suggesting the need for pre-extraction shock conditions. Similar results were obtained for experiments where the shock was simply a rapid and significant change in salt concentration. Future experiments will focus on shock conditions and assay of a suite of water immiscible solvents to find the most effective milking protocols.

#### Benefits to DOE

This project served as a nucleation point for the development of an algal biofuels area at INL. Although still early in this project, we have developed strong regional partners in the algal biofuels arena, including several at Utah State University (USU). We have leveraged these relationships to bring in two DOE Office of Biomass Program projects, one looking at algal feedstock logistics and the other at algal dewatering using cross-flow filtration. In addition, we are currently working on two additional proposals in this area. One project is a United States Department of Agriculture CAP proposal in which we are partnering with several regional universities and engineering companies. The other proposal will focus on halophilic algae from the Great Salt Lake. Collaboration has been formed with several regional university partners, including the Energy Dynamics Laboratory at USU, to respond to the call when issued by the Department of Energy (DOE). These partnerships will have long-term value to INL, the region, the nation, and DOE. In addition, if we are successful with the milking strategies under development, the economic feasibility of algal biofuels will be greatly improved.



## Development of Thermally Generated In Situ Precipitation Barriers due to Subsurface Heat Injection

Earl Mattson

Evaluate if a ground water barrier can develop around the perimeter of a fractured/porous media that is heated to high temperatures

ST143

The primary technical objective of this proposed laboratory-directed research and development (LDRD) project is to understand how a chemical precipitation barrier may develop in fractured and porous media where groundwater is converted to steam by anthropogenic heating. In the context of energywater studies, this project focuses on in situ oilshale retorting, where development of a chemical precipitation barrier could effectively limit water ingress into an oil-shale retort. To evaluate in situ thermally induced precipitation, we will evaluate conditions necessary to create mineral deposition events through numerical simulations from modified multiphase transport models that are supported by laboratory validation experiments. If creation of a barrier is feasible, we can compare the energy and time required to develop it with alternative means of excluding groundwater from the retort to help identify the feasibility of creating a mineral deposition barrier for groundwater control.

#### Project Summary

We have been working on collecting and assembling the Piceance Basin information on fracture density, fracture aperture, ground water gradient, flow velocity distribution, and geochemistry. This data will be used to support the hypothesis that geochemical changes due to subsurface heating of the retort can occur in the vicinity of a subsurface oil shale retort. We have concluded that a Theis flow model could adequately represent leakage into an in situ retort, and are in the process of including this information in a system dynamic model to evaluate parameter uncertainty.

Two abstracts have been accepted to national meetings:

- Mattson, Earl D., Carl D. Palmer, and Robert B. Perkins, 2010, "Potential Geochemical Releases to Groundwater from an In Situ Oil-Shale Retort," Presentation evaluating geochemical changes that could affect permeability surrounding a retort to the Ground Water Protection Council, Pittsburgh, Pennsylvania, September 26–30, 2010
- Mattson, Earl D., Kara Eby, Jeremy Boak, and Carl D. Palmer, 2010, "Water Consumption Analysis for an IN Situ Oil-Shale Retort," Presentation evaluating the necessary permeability structure surrounding retort necessary to keep water consumption to acceptable levels submitted to the Geological Society of America 2010 Meeting, Denver, Colorado, October 31–November 3, 2010.

#### Benefits to DOE

The research has been on-going for 4 months and is too immature for immediate benefit. Expected benefits depend on the evaluation of the necessary effectiveness of subsurface barriers needed around an in situ retort and if we can create this barrier through the thermal manipulation of the geochemistry. When the U.S. oil-shale resources are developed, knowledge of the water consumption will be critical for making resource extraction management decisions. This research effort will allow Idaho National Laboratory (INL) to have the capability to assess water consumption and quality issues associated with in situ extractions. We are submitting abstracts to oil-shale and ground water conferences in an effort to establish INL as a leader in this field.

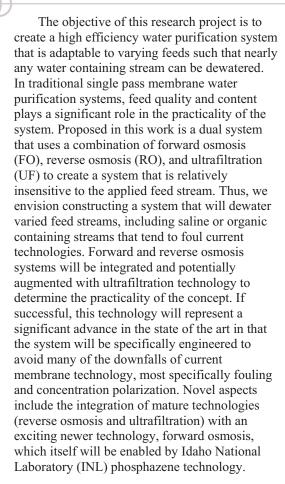
Idaho National Laboratory

112

### **Hybrid Osmosis Water Purification Systems Research**

Frederick F. Stewart, Ph.D.

Creating a high efficiency water purification system that is adaptable to varying feeds ST144



#### Project Summary

This project was a new start in May 2010. The initial activity for this project was the construction of a lab-scale forward osmosis (FO) system suitable for the analysis of membranes and draw solutions. The heart of the system is the cell that houses the membrane. Flux of water from the feed side into the draw solution is

measured gravimetrically. Experimental activities are focused on the first two tasks. A number of membrane substrates have been characterized for bulk water flux using NaCl and MgCl<sub>2</sub> in the draw solution. Eight commercial membranes have been acquired and screened against both salts. These membranes include four from Sterlitech Corp, which markets General Electric Osmonics membranes; two from Hydration Technologies, and two from Dow Chemical. In terms of bulk water flux, the Hydration Technologies' membranes appear to offer the highest performance. Initial data suggest rapid equilibration, on the order of 50 minutes to obtain steady-state data. A dependence on the salt contained in the draw solution is evident where MgCl<sub>2</sub> results in higher fluxes. It should be noted that the back diffusion of either salt to the feed side of the membrane has not been yet determined. Loss of flux as a function of time is expected since the dilution of the draw solution results in a lower driving force for transport. The magnitudes of the flux are quite high and rival the highest performance of more traditional RO/UF processes. These data will serve as a basis to judge process improvements, like new draw solutes, which are currently being developed.

#### Benefits to DOE

During the initial four months of this project, a new capability for water purification has been developed at INL, which has the promise of being valuable to the Department of Energy for a variety of water purification applications. Process improvements are underway and an invention disclosure record describing an improvement in the technology will be filed early in Fiscal Year 2011.



## Biological Fixation of CO<sub>2</sub> for Fuel and Chemical Production Using Acetogen Intermediary Metabolism

William Apel

The goal of this project is to use acetogenic systems to fix carbon dioxide ST145

This research is designed to identify and optimize the biological fixation of carbon dioxide (CO<sub>2</sub>) and corresponding production of value-added products using acetogenic bacteria. Acetogenic bacteria are known to possess flexible metabolisms capable of using many different substrates for catabolic and anabolic growth. As implied above, they are capable of CO<sub>2</sub> fixation. Also, as a part of their normal metabolism they produce a number of potentially valuable alcohols and organic acids. As such, they are excellent targets for development as a biological system that can be optimized for the conversion of atmospheric CO2 into value-added products such as fuels and industrial chemicals. Laying the groundwork for development of this system is the overall goal of this effort, with the following specific efforts for Fiscal Year 2010 (FY-10) goals: (1) begin metagenomic analysis of hot springs in Yellowstone National Park (YNP), and (2) collect environmental samples and enrich for acetogenic activity.

**Project Summary** 

This project was initiated early summer of 2010 at which time efforts were begun to meet the two FY-10 goals noted above.

Metagenomic data from five hot springs in YNP were obtained through a joint collaboration with the Thermal Biology Institute at Montana State University. These sequence data were

trimmed and assembled into contigs. These data are currently in the process of submission to NCBI for annotation. After the sequence data are annotated, they will be mined for information that may assist in identifying future sampling opportunities and identify useful correlations between acetogenic diversity and geochemical conditions.

In addition, environmental samples were collected from three hot springs (A, B, and M) in the Alvord Desert (AD) of Oregon and four hot springs (GS, EG, BC, and EA) in the Norris Geyser Basin in YNP. The temperature and pH of each sampling site was measured as well. Upon arrival at the laboratory, the environmental samples were enriched by using them to inoculate acetogenic media in serum bottles in an anaerobic glove box and incubating at 40, 60, 70, or 80°C. Samples that exhibited growth were transferred and tested for alcohol and organic acid production along with CO<sub>2</sub> consumption.

Benefits to DOE

This project is developing a system to produce biofuels and other value-added products while consuming atmospheric CO<sub>2</sub>, so it directly supports the energy resource and environment quality missions of the Department of Energy.

Idaho National Laboratory

114

## **Advanced Adaptive Algorithms in Phased-Array Ultrasonics** for Materials Inspection

Tim McJunkin, Dr. Milos Manic (UofI), Dr. Michael Moles (University of Toronto)

Automating ultrasonic examination technology
PH105

Ultrasonic examination is a technology used to view the insides of objects—including the human body—for medical applications or solid materials for characterization of structural integrity. Phased-array ultrasonic examination, as currently used, sets configuration for the examination entirely prior to use. This research is producing a novel, adaptive framework that reacts to the data available to automatically reconfigure an examination system. The hypothesis is that machine intelligence can provide both a more optimal collection and presentation of features contained within the examined subject at the current sensor location and instructions to the technician, or surrogate machine, for alternate locomotions to acquire better information or images. This improves on the paradigm to "collect as much data or information from as many points of view as achievable and then try to extract value" by turning the problem towards its inverse of evaluating a potentially small set of data and adjusting the point of view or depth of focus to arrive at more optimal information. Also included in the research will be a novel automated-intelligence scheme for adaptively combining the entirety of the capabilities of phased-array ultrasonic instruments that have traditionally been used independently. Untapped capabilities of an instrument vital to the powergeneration industry will be exploited, with potential benefits to efficiency, robustness, and accuracy in inspecting new and in-service components. Currently unresolved issues, such as imaged artifacts from off-angle reflectors and multiple propagation modes, are being put into reach with this research.

#### Project Summary

During FY10, the software interface developed during the first year of this project to phased array hardware for change instrument configurations (i.e., choice of apertures, delays, and gains) to receive real-time data from the system was enhanced. The interface was completed in a combination of C-sharp and

Labview. We continued to develop adaptive algorithms to, in real time, auto-focus on indications through genetic algorithms and gradient descent methods. This provides the means to interrogate any part of a time-based signal to find the optimal focusing laws. We derived the necessary expressions to define the characteristics of the focus signal from a linear phased-array transducer, resulting in the ability to determine the sound speed and position of the reflector in a pulse-echo configuration. This breakthrough achieves the means to resolve an issue with interpreting the ultrasonic signal to the origination and model of propagation, an advancement that provides the means for image artifact elimination and imaging enhancement by placing the returned signal at the correct location in space.

The framework for advising changes in position of the transducer for more optimal viewing of a target is in place with analysis of the shape of the focal laws. For example, the position of the sensor can achieve better focus if delay laws are symmetrical (i.e., less steering away from the natural direction of the transducer). Idaho National Laboratory (INL) ultrasonic focal law calculators were enhanced to allow them to be more general in that they can produce sector scans and be more effectively used. Also enhanced was the ability to add a framework for determining the change in position or orientation of an array for optimal interrogation of an indication. Each of the above and other specific benefits to applications are being captured in the draft of the PI's doctoral dissertation and associated publications.

#### Benefits to DOE

INL capabilities have been directly enhanced through the ability to utilize existing equipment in phased-array ultrasonics. This augmentation is available and will be exploited for oil and gas proposals, nuclear energy fabrication, and service inspection and containment of spent fuel at the end of the nuclear energy lifecycle.



### **Conducting Perovskite Materials for Catalytic Applications**

Sergey N. Rashkeev, Lucia M. Petkovic, Thomas M. Lillo (INL); Vivek Utgikar, (U of I) Investigating the catalytic activity of conducting lanthanum-based perovskite-type oxides

RS107

In this BlueSky project, we investigated the catalytic activity of lanthanum-based perovskitetype oxides  $(La_{1-x}A_xFe_{1-y}Co_yO_{3-\delta})$ , where A is an alkaline earth metal Sr or Ba). The large number of prospective metal-metal compositions embedded in these structures, their diverse properties, as well as their low oxygen permeability at low temperatures makes them candidates for many applications, including fuel cell electrodes, membranes, and partial oxidation of hydrocarbons. The variety of perovskite chemical compositions and their local crystal environments makes it possible to choose the material that will be the right catalyst for a specific chemical reaction. Though the electrical conductivity of these materials has not so far been considered important for applications, one could expect that their activity might be significantly enhanced when working under the NEMCA (non-Faradaic-modification-ofcatalytic-activity) conditions, in which catalyst properties are purposely modified by applying an electrical field. The electrical conduction of the framework guarantees that a charge can be delivered to catalytically active sites as well as removed from it.

The development of advanced catalytic systems with tunable functionalities opens new horizons for different industrial applications. Also, the state-of-the-art methods that were used and developed in this project can be employed for the rational design of other catalytic systems, including catalysts for nuclear waste separation. This project was directed towards highly innovated research and development (R&D) activities that can lay the groundwork for the Idaho National Laboratory (INL) missions in energy and environment.

In this one-year proof-of-concept project, we used several reactions as probes to determine the effect of perovskite composition on the reaction type and reaction rate. These reactions are the oxidation of soot and carbon monoxide (CO + O  $\rightarrow$  CO<sub>2</sub>) and the reduction of nitric oxide (2 NO  $\rightarrow$  N<sub>2</sub> + O<sub>2</sub>). These reactions offer the advantage of simplicity (requiring just few components) and represent typical cases of oxidation and reduction processes.

Project Summary

The modeling/calculation studies provided guidance for experimental measurements, suggesting some special combinations of the perovskite chemical composition and surface structure for which one could expect the highest reduction (oxidation) surface activity. The modeling studies have been done with and without applied electric field (the presence of electric field was simulated by playing with charges at different surface atomic sites) while the experimental measurements at this stage have been performed only without the NEMCA conditions. This is because the experimental setup for performing measurements of the catalytic activity in an external electric field requires significant funding for buying additional equipment, preparing high quality perovskite films with a high degree of stoichiometry, and definite surface orientation, etc., which were not available.

The experimental studies definitely indicated that the material may be of interest for catalytic applications—the soot oxidation temperature is significantly reduced in the presence of catalyst compounds synthesized in this work:  $La_0 {}_8Sr_0 {}_2Co_0 {}_4Fe_0 {}_6O_3$ , La<sub>0.8</sub>Sr<sub>0.2</sub>Co<sub>0.5</sub>Fe<sub>0.5</sub>O<sub>3</sub>, La<sub>0.8</sub>Ba<sub>0.2</sub>Co<sub>0.4</sub>Fe<sub>0.6</sub>O<sub>3</sub>, and La<sub>0.8</sub>Ba<sub>0.2</sub>Co<sub>0.5</sub>Fe<sub>0.5</sub>O<sub>3</sub>—by about 150–200°C in comparison with oxidation in air. Also, the modeling shows that NEMCA conditions may enhance its activity even further (i.e., the catalyst may be tuned by both the surface composition and an electric field). In addition to catalytic applications, these perovskites may be used as cathode materials for catalytic reduction of oxygen in solid oxide fuel cells (SOFC) and solid oxide electrolyzer cells (SOEC). These results are interesting for the high-temperature electrolysis for hydrogen production project (a part of the NGNP project in which INL is actively participating).

We investigated the dynamics of the structure reconstruction with oxygen loss during the regulated increase of temperature. We found that Sr-doped perovskites undergo a higher number of phase transformations than Ba-doped structures because of the higher mobility of Sr atoms in the perovskite matrix. The calculations are in very good qualitative agreement with



catalytic experimental measurements and allow identification of some special combinations of the perovskite chemical composition and local surface structures for which one could expect the highest catalytic activity for oxidation processes. In particular, we provided natural explanations why the soot combustion peak for Ba samples occurs at lower temperatures than for Sr samples and why Fe-rich compounds oxidize soot at lower temperatures than Fe-rich materials.

We plan to search for additional funding for future research work in this direction. For this purpose, we established strong collaboration with several universities. In particular, Dr. Rashkeev and Dr. Petkovic are collaborators in a National Science Foundation proposal (PI: Professor Vivek Utgikar) recently submitted from the University of Idaho (chemical engineering program). Dr. Rashkeev plans to be a collaborator in a Department of Energy (DOE) proposal (PI: Professor Maija Kukla) on structural properties of complex perovskite oxides that will be submitted from the University of Maryland in the near future. Wide collaboration with universities will attract graduate students and postdoctoral researchers into the projects.

#### Benefits to DOE

This project contributed to the following DOE missions:

- 1. Energy: This research widened capabilities in developing new, potentially revolutionizing materials with tunable characteristics for energy catalysis including biomass technologies and upgrading of Fischer-Tropsch hydrocarbons.
- 2. Environment: As a proof-of-concept, we considered reactions of the soot and carbon monoxide oxidation and nitric oxide reduction, which contribute to environmental projects. This understanding can be applied to other processes including nuclear waste separation.
- 3. <u>Science and Technology</u>: The project contributed to understanding oxygen diffusion through an oxide ceramics with impurity atom additions. This understanding can be applied to other systems.
- 4. <u>National Security</u>: Development of highly active, tunable catalysts for synfuels production will ultimately reduce the U.S.'s dependence on foreign energy sources.



#### **Acoustic Nanostructures**

David H. Hurley

Investigating an innovative method to provide lateral confinement of charge carriers BS108

This project involved investigation of an innovative method to provide lateral confinement of charge carriers, using high frequency acoustic phonon pulses. The confining potential associated with the acoustic phonon arises from a coupling between dynamic strain and electric field (piezoelectric coupling). The work performed built on a close collaboration between Idaho National Laboratory (INL) and researchers at the Center for Integrated Nanotechnologies at Sandia National Laboratory (CINT-SNL). The INL contribution involved generation and detection of picosecond surface acoustic phonon pulses as well as development of new capability to perform time resolved photoluminescence studies to gauge the strength of the confining potential. The SNL contribution involved fabrication of high frequency lithographic gratings used for acoustic phonon transduction.

#### Project Summary

The objective of this laboratory-directed research and development (LDRD) project was to demonstrate confinement of charge carriers using high frequency surface acoustic phonons. The duration of this project was for one year and was funded through the "Blue Sky" LDRD research initiative. While the ultimate goal of this project was not fully realized, several key new capabilities have been established that position INL to pursue new funding opportunities involving fundamental investigations of 5f electron materials. The work presented in the remainder of this report is divided into two coordinated activities.

Low temperature, ultrafast optical metrology of electron phonon coupling: Much of the work involving quantum confinement is performed at low temperatures. The primary reason for this is that electron phonon coupling at higher temperatures masks subtle changes brought about by nanometer-sized confinement. Performing ultrafast optical metrology of phonon transport and electron phonon coupling at cryogenic temperatures requires that the sample be placed in a cryostat with optical access. In addition, experiments involving high lateral spatial resolution require microscope focusing optics. Traditional cryostats preclude the use of microscope objectives because thermal expansion of the sample stage exceeds the depth

of focus resulting in temperature-induced defocusing. To overcome this difficulty, we purchased a specially designed cryostat with a freely floating sample stage (Fig. 1). Incorporating the new cryostat into our current system involved reconfiguring the focusing and scanning apparatus (Fig. 1). To test the stability of this new cryostat, we measured Brillouin oscillations in a Gallium Arsenide (GaAs) sample. The oscillations at three different temperatures are shown in Figure 1. The rate of decay of the thermal background increases with decreasing temperature, indicating an increase in the thermal conductivity with decreasing temperature. The measurements taken at cryogenic temperatures did not require refocusing, demonstrating the thermomechanical stability of the new cryostat.

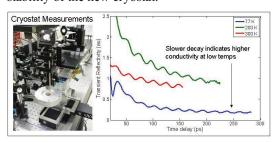


FIGURE 1. Left: Cryostat for optical microscopy. Right: Brillouin oscillations and thermal decay with temperature as a parameter.

Generation and Detection of Picosecond Surface Acoustic Waves: The samples for this experiment were provided via collaboration with the Center for Integrated NanoTechnologies (CINT), a Nanoscience User Facility located iointly at Sandia and Los Alamos National Laboratories. This collaboration provided a nocost access to the CINT growth and processing facilities and low-cost availability of speciallyprepared samples. Absorption gratings consisting of metal bars on a GaAs substrate were fabricated using electron beam lithography and metal lift-off within the clean room facilities at CINT. The bar height, h, was approximately 30 nm and the periodicity, p, was 75 nm. The metallization duty cycle was approximately 70%. A SEM image of the metallic grating is shown in the inset in Figure 2.



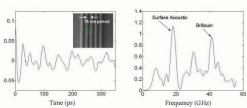


FIGURE 2. Left: Change in probe reflectivity associated with surface acoustic phonon pulse. Inset: SEM image of metallic grating. Right: Amplitude spectrum clearly showing the presence of the surface acoustic phonon pulse and the Brillouin oscillations.

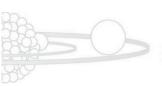
We performed picosecond ultrasonic measurements to investigate the generation and detection characteristics of the gratings. The pump and probe pulse trains are derived from the same femtosecond Ti:sapphire laser (Coherent Mira 900) with  $\sim\!100$  fs pulse duration and a 76-MHz repetition rate. The 800-nm output was used for the probe beam while the second harmonic at 400 nm was used for the pump beam. The pump beam was modulated at 1 MHz to facilitate lock detection. The pump and probe beams were combined using a dichroic beam splitter to allow both beams to pass through the same 20x microscope objective producing a 5- $\mu$ m spot at the sample surface.

The left side of Figure 2 shows the change in probe reflectivity produced by the propagating surface acoustic phonon tone burst. To highlight

the phonon contribution to the signal, we have subtracted the thermal and electronic background. The Fourier amplitude spectrum is shown on the right side of Figure 2. There are two peaks in the amplitude spectrum. The peak at ~18 GHz corresponds to the surface acoustic phonon pulse and the one at ~42 GHz corresponds to the Brillouin oscillations. The next step is to perform photoluminescence measurements on grating samples with buried quantum wells. We will seek to leverage existing, externally funded programs within our group to support this activity.

#### Benefits to DOE

The work, conducted in Fiscal Year 2010, which involved controlling materials' properties at the level of the electron, squarely addresses one of the five grand challenges posed by the Basic Energy Sciences (BES) Advisory Committee. The capability developed under this LDRD project has helped position the Laser Based Materials Characterization Group to expand its current BES footprint. We are in the process of developing two whitepapers to be submitted to BES. Both will involve elements of low-temperature energy carrier transport and interactions.



### A New Paradigm for Robust Catalysts

Helen Farrell

Testing encapsulated Pt nanoparticles to be used under high-temperature conditions BS109

The objective of this project was to prepare and test encapsulated Pt nanoparticles to be used under high-temperature conditions. The intent was to minimize evaporation and sintering of the catalyst to optimize its lifetime.

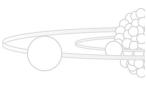
#### Project Summary

Difficulty was encountered in preparing the encapsulated Pt nanoparticles. At the end of the fiscal year, only one sample had been tested with encouraging, though not conclusive, results.

#### Benefits to DOE

From the beginning, we recognized this as a high-risk endeavor. If it had been successful, in addition to contributing to our basic understanding of nanoparticle catalyst aging, it would have extended the utility of such catalysts to higher temperatures and more extreme conditions. At best, this could reopen the possibility of using thermochemical cycles to produce hydrogen from water employing next generation nuclear reactors as heat sources.





#### **Particle-discrete Element Model Simulation**

Paul Meakin

Using experimental methods to provide a new way of simulating the coupling between fluid generation, fluid flow, and deformation/fracturing

This project builds on experience with discrete element models for material deformation and fracturing developed by the PI at the DuPont Company and the University of Oslo, and with particle models for single-phase and multi-phase fluid flow in confined systems developed at INL. X-ray tomography experiments performed in cooperation with the University of Oslo and the Statoil ASA at the European Synchrotron Radiation Facility are being used to motivate and guide the computer modeling work. To our knowledge, discrete element models have not previously been combined with discrete element models for this type of application. If successful, this work will provide a new way of simulating the coupling between fluid generation, fluid flow, and deformation/fracturing, which could be applied to a variety of processes that are important to conventional oil and gas exploration and unconventional oil and gas production. The ultimate goal is to develop computer models that can simulate in detail the behavior observed in the experiments.

#### Project Summary

The overall objective of this project is to develop a better understanding of how the generation of fluids in extremely impermeable rocks is coupled with deformation, fracturing, and fluid flow using numerical and experimental methods. The development of innovative models that will enable us to simulate the coupling between fluid generation in extremely impermeable rocks, deformation, fracturing, and fluid flow is also an important objective. Most of the research has been performed in

collaboration with the University of Oslo Center for the Physics of Geological Processes (a Norwegian Center of Excellence).

We have focused on x-ray tomography experiments and the analysis of x-ray tomography experiments. This is challenging because of the large three-dimensional (3-D) images (up to 2040 × 2048 × 2048). We found that fracturing is concentrated along bedding planes—presumably those with the highest kerogen content. Small cracks nucleate, grow, and coalesce until they occupy the entire bedding plane. A simple computer model based in invasion percolation concepts was developed to explain these observations.

#### Benefits to DOE

Some of the work has helped develop smoother particle dynamics and dissipative particle dynamics codes, but robust and reliable codes are not yet available.

We have also performed some preliminary correlation analysis to determine the strain field in the two-dimensional cut through the 3-D electron density map. In future experiments, we will attempt to obtain the full 3-D strain field in the same manner and determine its relationship with how and where fracturing occurs. A paper is being prepared for publication in *Reviews of Geophysics*.

The work is highly relevant to the production of oil from oil shale and basin modeling in support of oil exploration.

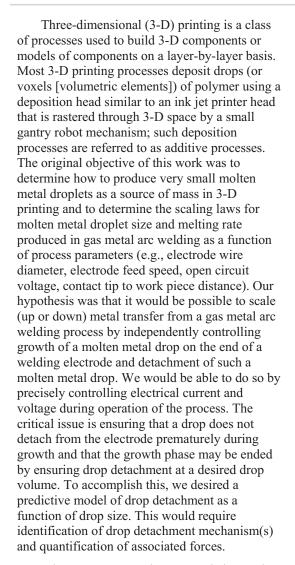


## **Scaling of Welding Processes**

Herschel Smartt

Exploring deposition of metals in a manner suitable for rapid prototyping or production of relatively large metal components

FF106



It became apparent that we needed a much greater mass deposition rate than would be provided by small diameter gas metal arc welding drops. This lead to a decision: we needed to use a different deposition process. This is contradictory to the conventional approach of achieving fine-scale dimensional resolution in the deposited material by use of small voxels. To obtain fine-scale dimensional resolution, we have combined a subtractive process with the additive process. Specifically, we are using friction stir welding (FSW) to deposit metal and computer numerically controlled (CNC) milling to remove excess metal. By doing so, we are extending conventional 3-D printing to allow 3-

D printing of large metal components such as aluminum engine blocks.

**Project Summary** 

We examined our hypothesis from the standpoint of our present understanding of the physics of drop growth and detachment in gas metal arc welding. Drop growth is directly controlled by resistance heating of the electrode by electrical current flowing in the welding electrode. The electrode is a series element in the secondary or output circuit driven by the welding power supply. The power supply functions as a constant voltage source providing nominal direct current, albeit current regulated by a pulsedwidth modulator operating at a nominal frequency of about 20 MHz. Current flow from the constant voltage source is controlled by the local electrical resistance of the electrode and welding arc. The electrode is driven into the arc by a constant speed wire feeder. Melting of the electrode controls arc length; arc and electrode resistance are controlled by the combined lengths of the electrode and arc in a self-regulating manner. This part of the process is well understood, as are the various modes by which drops are transferred from the electrode to the work piece. This process has been studied extensively for decades for a range of electrode wire diameters and drop sizes of practical interest for welding. However, our interest is for transfer of drops much smaller than are normally used. In prior scoping studies many years ago, we determined that there is a minimum drop size of about 1-mm in diameter below which we were not able to detach drops. In this work, we wanted to develop a fundamental understanding of the forces controlling small diameter drop transfer, formulate a predictive model of small-size drop detachment, and demonstrate an ability to employ such a model to control drop detachment.

We acquired various pieces of welding equipment, much of it from the recently completed Yucca Mountain Project, and used this equipment to build an experimental welding apparatus with precise computer control of the process parameters (voltage and thus current, electrode speed, and low frequency pulsed-width modulator parameters) also incorporating suitable data acquisition. We tested this



equipment and began experimental studies to identify the best starting parameters for our work. We also began formulation of a predictive model; the combination of these activities was complementary. We wanted to ensure that our selection of process control parameters and data acquisition capabilities properly supported our experimental needs.

To this end we considered that drop detachment involves the following: a drop is attached to the electrode by surface tension; there is a liquid neck between the drop and the electrode. Opposing this attachment force is gravity and aerodynamic drag associated with the flow of arc plasma past the drop in a downward direction. This plasma flow is driven by the divergence of electrical current flowing in the arc. The liquid neck between the drop and the electrode is subject to pinch-off by the electrical current, which passes through the neck into the top of the arc. This pinching of the liquid neck changes the magnitude of the attaching force to some degree. Drop detachment will occur when the sum of the detaching forces exceed the attachment force. Our intent was to predict these forces for different electrode diameters and choose low-frequency pulsed-width modulator control parameters to control a detachment event. However, late in the first project year, it had become apparent that the mass deposition rate we would obtain would not allow us to economically produce the large-sized components we were targeting.

During this second project year, we changed from gas metal arc welding to FSW. The FSW process was originally developed for autogenous welding to join plates of metal by butt welding of one edge to an adjacent edge, or by welding of overlapping edges. We are using the process to join one plate to another plate placed on top of the first plate, thus producing a stack of plates analogous to a stack of playing cards. In combination, we are using CNC machining to remove excess metal from the stack, thus producing a final engineering component. This combination of processes allows us to achieve a high mass deposition rate, precise final dimensions, and fine surface finish. We have fabricated bars of aluminum and hollow test pieces using this combination. The work has

allowed us to identify certain critical processing steps necessary to produce usable components.

The above photographs (Figs. 1, 2, and 3 [next page]) show, respectively, a stack of aluminum plates that have been joined by FSW. The stack was sectioned by making transverse saw cuts to provide coupons for metallographic examination and machining. One section was CNC machined to remove excess (non-welded) metal and is shown in Figure 2 marked A and B on its ends. Figure 3 (next page) shows a metallographically polished and etched transverse section of the welded stack prior to CNC machining. Other geometries were produced, including a coupon containing a hollow cavity. Results of these fabrication experiments will be exploited during the third year of the project to develop related technologies.



FIGURE 1. Stack of aluminum plates joined by friction stir welding.



FIGURE 2. Stack of aluminum plates section for examination. A solid bar of aluminum is marked  $A \leftarrow B$  in the photograph.

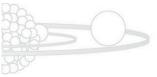




FIGURE 3. Cross-section of the stack of aluminum plates joined by friction stir welding shown prior to machining the solid bar from the center of the above stack.

#### Benefits to DOE

The goal of this work is to address net shape fabrication, specifically seeking means of fabricating metal components. Our long-range programmatic goal is to develop a means of fabricating large metal components that would ordinarily require the investment of significant infrastructure funds and the high-energy costs of a foundry or forging plant. Specifically, we want to be able to fabricate components such as engine blocks, cylinder heads, and crankshafts in a stand-alone "cell." Conceptually, one may think of a very large 3-D printer. The cell will incorporate robotics, CNC machining, and one or more precision metal deposition processes.

The conventional basis for 3-D printing is that precise final dimensions of the printed object may be obtained by employing very small voxels. Two issues using this approach are that mass deposition rates are typically very small; this results in small production rates unsuitable

for most manufacturing applications. It also results in a component surface finish representative of the voxel size. Finer surface finish is obtained by decreasing voxel size, resulting in an even smaller production rate. In our approach, combining additive and subtractive processes, mass deposition rate and surface finish are independent. The project has allowed us to experimentally explore the balance between production rates and product quality, select an optimal technology, and identify and develop technical solutions to certain issues, which will allow us to move the combined process to a point where industrial partnerships and cost-shared direct funding are realistic.

Specifically, we are targeting 3-D printing of large components (weighing hundreds of pounds). These components normally are produced by casting processes, which are done in foundries that require large capital investments and have large energy footprints. This work seeks to provide U.S. industry a means of producing such objects without a foundry, thus achieving significant cost and energy savings.

We have the experimental capability to fabricate certain geometrical shapes necessary to make the combined process attractive to industry. Project funding enabled us to hire a new Ph.D.-level mechanical engineer with expertise in internal combustion engines and energy efficiency. This new expertise will significantly increase our credibility to industrial partners and make our industrial research more relevant to Department of Energy-Office of Energy Efficiency and Renewable Energy program needs.

Idaho National Laboratory

124

## Effect of Glycosylation on the Activity and Stability of Bacterial Enzymes

William A. Apel

The goal of this project is to assess the effects of glycosylation on the activity and stability of bacterial enzymes

FF108

Initial work began early summer 2010 and centered on expressing a gene that we had previously studied in considerable detail and consequently had a strong understanding of its relative stability and activity profiles when expressed by its natural host organism. In this effort we attempted to express this gene in multiple laboratory expression systems but did not have acceptable expression in any of them. This gene is a multiple subunit protein, and we believe that the expression systems employed could not successfully assemble the subunits into the complete protein. As a result, we switched our efforts to a single subunit extremophilic enzyme. We had the gene for this enzyme synthesized based on its original, native sequence with the addition of a his-tag region to facilitate protein separation.

#### Technical Objectives

The goal of this project is to test the hypothesis that bacterial enzyme stability and activity profiles may be favorably affected through post-translation modifications such as glycosylation. This is being done by expressing genes from extremophilic bacteria in post-translationally modifying and non-post-translationally modifying expression systems. Through this effort our objective is to lay the groundwork for better understanding the effects of post-translational modifications on the stability and activity of bacterial enzymes.

#### Results and Accomplishments

We have successfully transformed our target non-post-translationally modifying host and have expressed the protein and separated it using the his-tag with a metal affinity column. Zymograms that allow screening for the target activity were developed. Data generated from those zymograms have confirmed that expression of our synthetic gene in the non-post-translationally modified expression system has been successful, and we are producing protein with high levels of the anticipated enzymatic activity. Despite the somewhat shaky start due to our inability to express the original target protein in our laboratory expression systems, diversion to the alternate protein has been a successful strategy and we are back on track to meet the goals of this laboratory-directed research and development (LDRD) project within budget and within the three-year time frame.

#### Benefit to DOE

This work is directed towards increasing the stability and activity of enzymes that are useful for generating biofuels and value-added products. As such, this work directly supports the Department of Energy's (DOE's) energy resource and environmental quality missions while also expanding its science base.





# **Energy Security Initiative**





### Generation and Expulsion of Hydrocarbons from Oil Shale

Carl D. Palmer, Earl D. Mattson, Hai Huang

Working to understand the geochemical factors that affect the quantity and quality of potential shale oil resources

EI107

A potential solution to meeting some of the increasing demand for liquid fuels is development of unconventional fossil resources. Within the U.S., shale-oil resources (estimated to be 3.8 trillion barrels) are a substantial portion of this unconventional resource. Better understanding of processes controlling the generation and expulsion of shale oil can help energy companies develop more efficient extraction processes and improve the Nation's ability to meet future energy demands. We have been focusing on geochemical factors that affect the quantity and quality of the generated shale oil and on the physical mechanisms governing the expulsion and the transport of product to an extraction well.

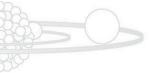
#### Project Summary

- 1. We summarized and analyzed thermal transport properties (thermal conductivity, heat capacity, thermal diffusivity) of oil shale. A three parameter model of thermal conductivity as a function of temperature and grade was obtained. The model fits literature data very well. Structurecomposition models suggest thermal conductivity behaves as a high-conductivity material (minerals) embedded in a continuous low-conductivity matrix (kerogen). This result is consistent with the effective Debye temperature of oil shale determined from our thermal conductivity model. Heat capacity is determined from a three component system composed of minerals, kerogen, and coke/char.
- 2. We designed and built a uniaxial stress device for use in our PARR retorting vessels. Testing on artificially fractured oil shale sample showed a three order of magnitude decrease in the fracture permeability with retorting.
- 3. We designed and tested a permeameter that allows for continuous measurement of oil shale air permeability and changes in strain under uni-axial stress. In tests up to 250°C, a step increase in temperature, resulted in an initial thermal expansion of the sample followed by consolidation. A walk-in fume hood was acquired and installed so that experiments can be run to temperatures

- where pyrolysis is occurring. The apparatus allows us to measure the permeability and strain over the temperature range of ambient to 400°C and loads from 0 to 11,000 lb (about 6000 psi stress with a typical sample).
- 4. We have designed and constructed a new retorting system that allows us to better simulate *in-situ* retorting conditions. The new system is open but with a back-pressure that can be set. As pressures exceed the set back-pressure, the gases bleed via a stainless-steel tube to a condenser which is kept at sub-ambient temperature. The non-condensable fraction is collected in a tedlar bag for subsequent analysis.
- We have developed pore-scale models of insitu oil shale retorting. We have continued to develop a computer code that couples fluid flow, heat transport, and rock deformation/fracturing process during in*situ* retorting process. This code supports our study of the evolutions of rock permeability and porosity due to rock deformation and fracturing induced by thermal expansion and fluid pressure buildup and diffusion, a critical factor for understanding fluid generation, oil expulsion, and residual oil content in spent oil shale. We successfully developed a rock mechanistic model based on a discrete element method (DEM). We coupled this DEM model with a fluid-flow and heat conduction models. We can use this code to simulate the *in-situ* generation of oil and gas inside the rock, the consequent buildup of fluid pressures that change the effective stress, the resulting rock deformation and (once the critical strain is exceeded) fracture generation/propagation.

Eight presentations were given at professional meetings while an additional five presentations were given at universities and other research institutes. In addition, work from this project indirectly contributed to another 11 presentations at professional meetings on water resources and environmental impact of oil shale development.



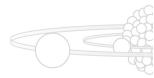




This project directly addresses a key element of DOE's strategic plan to promote "America's Energy Security through reliable, clean, and affordable energy." The project helped to strengthen INL's leadership role in the development of unconventional energy resource recovery as an important element of the energy security goal by building signature strength around "subsurface phenomenology and implications, including fate and transport, water management, in-situ conversion, and

environmental and regulatory implications associated with unconventional extraction." Such expertise will advance scientific and technical knowledge that will help the U.S. develop domestic energy supplies, such as oil shales. Collaborations with other water-focused projects will help to achieve the environmentally responsible development of these energy resources. Linkage to nuclear energy (e.g., as a heat source) will provide a strategy for conversion of energy to forms that correspond to existing and projected infrastructure needs of the

U.S.



### **Nuclear Pathways to Energy Security**

Craig Cooper

Building secure, affordable energy systems that operate within the carbon and water constraints imposed by the regional and global environment

The purpose of this work is to develop a better understanding of how to build secure, affordable energy systems that operate within the carbon and water constraints imposed by the regional and global environment. The world is at a crossroads. We do not have the resources for 6.5 billion people to consume energy and dispose of waste in the environment as we have for much of the past century, and the problem will only get worse as the global population grows to a projected 9 billion by 2050. We have to change our approach to energy and build fundamentally new kinds of systems whose operating parameters and environmental impacts are largely unknown today. Furthermore, these systems have to be optimized to accommodate factors other than cost and security of supply. They must also be optimized to account for carbon emissions, water demand, "other waste" emissions, resource availability, land use impacts, and public perception. This project seeks to advance our knowledge and capabilities in the development of affordable, clean, and secure energy systems by conducting systems design studies and analyses of the carbon and water impacts of key energy systems, with a focus on identifying ways nuclear power can mitigate those impacts while also improving U.S. energy security. As such, transportation fuels are of particular interest.

There are many analyses proposing various ways to improve the security of U.S. energy supplies and/or cost-effectively reduce U.S. carbon emissions. Few studies agree, none of them use the same basis for assessing capabilities, and none currently factor in the impact of these systems on water resources. This work will quantify the water impacts of energy systems in a rigorous manner and then use this quantification as a basis for contrasting the carbon and water impacts of various strategies for improving U.S. energy security. We anticipate that this work will further demonstrate that nuclear-driven energy systems can costeffectively provide a secure, low-carbon, watersmart source of energy for all market sectors.

Project Summary

This project uses systems modeling techniques to address two nested problems. First, we seek to improve our understanding of the

complex linkages between energy and environmental systems by demonstrating how innovative new "sustainable energy systems" can be designed on the basis of environmental constraints on greenhouse gas emissions and water availability. Second, within the context of this overarching goal, we will also determine whether nuclear energy can provide costeffective options for producing secure, lowcarbon transportation fuels from North American feedstocks between 2010 and 2030. The technical objectives of this project have been repeatedly modified over its lifetime, as (1) Idaho National Laboratory (INL) management has specifically requested mid-project changes in scope to reflect changing lab priorities, and (2) significant amounts of the funding originally dedicated to this project has been shifted towards other projects in response to changes in INL strategic priorities. Consequently, the objectives are presented here in terms of the hypotheses investigated and the methods used to demonstrate them in papers currently being written and reviewed.

Hypothesis #1: We can create a better understanding of the relationship between energy and environmental systems by building systems models that start from environmental constraints and "work backwards" to design new energy systems that are more sustainable. This hypothesis is tested by comparing how different options for greenhouse gas mitigation compare when prioritized in terms of (1) water use, (2) cost, and (3) difference in net energy consumption. The associated objective is to produce a paper that evaluates the observed differences and highlights options that have the greatest synergy.

Hypothesis #2: Nuclear energy can provide new options for producing clean, secure transportation fuels from North American feedstocks. This hypothesis is tested by evaluating how different scenarios for nuclear deployment in the U.S. impact various measures of energy security and comparing the result to the energy security impacts of baseline projections by the Energy Information Administration (EIA). The associated objective is to produce a paper that evaluates the energy security potential of nuclear energy deployment.





<u>Hypothesis #1</u>: We can create a better understanding of the relationship between energy and environmental systems by building systems models that start from environmental constraints and "work backwards" to design new energy systems that are more sustainable.

- 1. Year 1 (2008)— We identified a major gap in our understanding of water limitations and defined the water limit as being "when the lifecycle water cost of cleaning and treating water is greater than the amount of water needed to support the commercial process." We also began building a model that can test that concept both for an individual technology and for a combination of technologies drawing water from a hydrologic basin.
- 2. Year 2 (2009)— We progressed in development of lifecycle water model (though it is still incomplete) and developed data to support contention that including water as a constraint (in addition to carbon and cost) yields different energy system designs than would occur if only carbon and cost were considered. The same holds true for reliability of energy delivery. Environmental limitations force energy systems to operate within a complex set of constraints that cannot be accurately evaluated in isolation or from a purely economic standpoint (e.g., levelized cost of energy).
- 3. Year 3 (2010) An unplanned 80% reduction in funds in Fiscal Year 2010 forced a simpler approach to proving this hypothesis than originally planned. This reduction has also delayed publication of the associated manuscript. We demonstrated this hypothesis by calculating the global average cost of various greenhouse gas reduction options (from data gathered through a combination of systems models and literature searches) and demonstrating that prioritizing options based on water yields different results than prioritizing options based on cost or change in total energy (e.g., size of shift).

Outcome. Presentation at national conference (Geological Society of America annual meeting) and manuscript titled "Sustainability and Energy Development: Influences of Water Resource Limitations on Options for Greenhouse Gas Emissions Reductions," which explores the impacts of climate mitigation options on water resources. The final draft is currently in internal review with coauthors, prior to submittal for publication.

<u>Hypothesis #2</u>: Nuclear energy can provide new options for producing clean, secure transportation fuels from North American feedstocks.

- 1. **Year 1 (2008)** For the first time, we described how nuclear energy can be used to transition our transportation energy system from today's carbon-intensive economy to a future low-carbon economy using incremental change to existing infrastructure and vehicle technology. We incorporated this vision into a Department of Energy (DOE) strategic analysis for combating climate change.
- Year 2 (2009)— We quantified some of the carbon savings from nuclear, and begun to investigate the water, reliability, and economic impacts. Results semiquantitatively indicate that building nuclear energy to meet peak electricity demand and then diverting "excess energy" at sub-peak times to the production of hydrogen and/or hydrocarbons provides a way to increase energy security while reducing greenhouse gas emissions and facilitating reliable deployment of intermittent generation at a water cost that is likely to be acceptable. Regional limits on water availability may constrain where this option could be deployed and the extent to which water pretreatment is needed.
- 3. **Year 3 (2010)** As with Hypothesis #1, an unplanned 80% reductions in funds forced a change in how this hypothesis is addressed and delayed publication of associated work. Hypothesis #2 shifted towards



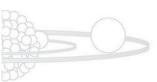


- demonstrating that nuclear energy can improve U.S. energy security and demonstrates it by evaluating several scenarios of nuclear expansion in terms of their qualitative and/or semi-quantitative impact on energy security measures. This concept is explored in the second paper, discussed previously.
- Outcome. Draft manuscript titled "Implications of Expanded Nuclear Power on Energy Security." Paper explores how deployment of nuclear energy can impact various measures of energy security from a U.S. perspective. Manuscript is currently an interim draft.

### Benefit to DOE

- Energy Security— "Promoting America's energy security through reliable, clean, and affordable energy."
  - Helped quantify the water resource impacts to energy development globally and in the Western Energy Corridor.
  - Pending publications provide insight into how various options for low-carbon energy deployment impact U.S. energy security goals and global energy sustainability (environmental, economic, and social impacts).
  - Helped the Climate Change Technology Program (CCTP) to expand its ability to conduct gap analysis and constraints analysis for deployment of low-carbon technologies by providing an enhanced understanding the water impacts of energy development.
  - o Provided information that INL management has used to help DOE-Office of Nuclear Energy (NE) develop a new research and development (R&D) program in Nuclear Energy Integration. This program demonstrates how integration of nuclear technologies with other energy technologies in "hybrid systems" can help mitigate climate change while also improving overall energy and environmental security.

- o Provided information that INL management has used to develop climate-water and energy-water portfolios within the INL Strategic Plan for water R&D. The purpose of this INL initiative is to conduct water-associated R&D to support the mission objectives of the Environmental Protection Agency, the Department of the Interior, the National Oceanic and Atmospheric Administration, and DOE.
- Scientific Discovery and Innovation— "Strengthening U.S. scientific discovery, economic competitiveness, and improving quality of life through innovations in science and technology."
  - Presentations given and manuscripts currently in review for publication (either peer-reviewed or INL report; peer-reviewed is preferred) provide insights into which technologies are best suited to address large-scale challenges in U.S. economic competitiveness and quality of life.
  - New technologies have not been created, but there is a better scientific and/or technical understanding of how currently available (and emerging) technologies impact the U.S. with respect to the science mission goals described above.



## Altering Wettability by Chemical Amendments to Improve Gas Production from Tight Sands

Eric Robertson

New laboratory measurement technique characterizes vast tight gas sands

The original hypothesis was that the near-wellbore region in strongly water-wet, tight gas sandstones could be treated with a chemical to reduce the water-wetness, thus improving gas deliverability. To accomplish this, tests would be performed to: (1) select an appropriate chemical additive to completion fluids, (2) measure rock properties, (3) measure wettability changes, (4) measure relativity changes, and (5) develop a field testing plan.

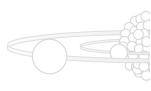
### Project Summary

An appropriate surfactant-based chemical was selected for permanently altering wettability based on capillary rise experiments. Key core parameters needed for the imbibition and relative permeability tests were measured. New methodologies were developed to measure

permeability in very tight rock (<1 micro darcy  $[1 \times 10^{-16} \text{ m}^2]$ ). Personnel were trained and new equipment was developed to measure spontaneous imbibition, which is a new method to quantify wetting states, as well as to measure gas/water relative permeability at reservoir pressure and temperature.

### Benefits to DOE

Idaho National Laboratory (INL) and the U.S. Department of Energy (DOE) have benefitted directly through the development of new techniques, equipment, and personnel trained in working with very tight gas sandstones. INL can now work with industry and other research laboratories or universities on tight gas systems, an area of unconventional gas that is growing rapidly worldwide.



**Idaho National Laboratory** 

134

## **Adaptive Process Modeling of Hybrid Energy Systems**

Lee Shunn

Modeling, monitoring, and control of advanced energy processes

Next-generation hybrid energy systems will be an order of magnitude more complex than current energy systems. The additional complexity applies not only to the design of the system, but also to operations and controls. A major challenge to deploying hybrid energy systems will be to coordinate stable operation amongst all of the components without compromising performance or product quality. Unfortunately, the chemical processing industry has limited collective experience with these kinds of dynamic operations. Historically, chemical plants have been designed to run essentially at steady state and to avoid the kind of dynamic scenarios that are key to maximizing the utility of hybrid systems.

The objective of this project is to use physics-based modeling and lab-scale experiments to characterize the transient response of hybrid systems to disturbances and changes in operating conditions. The transient data collected in the experiments are used to validate the models and to provide guidance for model development. The computational models are used for prognostics in model-based control systems for hybrid energy processes. The process models offer a relatively spare description of the physics so that they can be solved tractably. Their predictive capabilities are extended using automated methods to periodically re-evaluate and update model parameters based on recent system measurements. Thus, simple models can be used for prognostics even as conditions within the process change over time.

### Project Summary

A laboratory-scale methanation experiment was constructed and operated during the first year of this project. That experiment helped identify pertinent research needs for hybrid synthetic fuel production and motivated investments in more sophisticated instrumentation for monitoring and control. This year, the project helped design and construct a liquid fuel experiment that will be operated to examine how step changes in temperature, pressure, and flow rates impact the production rate and quality of the synthetic fuel. As part of this project's contribution, a state-of-the-art Raman spectrometer is being installed in the

HYTEST synthetic fuels experiment to provide real-time, in situ measurements of the chemical composition at several locations within the system. The experimental data will be used for model validation and method development for adaptive modeling.

This year the project also developed software for dynamic simulations of chemical processes and energy systems. This software. written for the MATLAB Simulink environment, includes original transient models for packed bed catalytic reactors and membrane separations. Auxiliary models for process equipment such as compressors, valves, storage tanks, heaters, coolers, and their controllers have also been developed. The unit operations were combined in an overall process model that describes fuel synthesis from gasified coal. The resulting model offers a fully transient description of an advanced chemical process and will be used next year to demonstrate adaptive modeling concepts. The process model is currently being used by two additional laboratory-directed research and development projects as well as two university collaborations.

### Benefits to DOE

The potential to expand uses of nuclear energy is a prominent driver for the development of next-generation nuclear power plants. The hybrid energy systems at the core of this effort demonstrate the successful integration of nuclear energy research (production of hydrogen from high-temperature steam electrolysis) with processes that are familiar in conventional fossiland bio-energy systems (coal/biomass gasification and synthetic fuels production). These unconventional applications of nuclear energy advance renewable energy technologies and outline a pathway for using nuclear energy to produce high-quality transportation fuels. This project has helped identify and address technological gaps and research needs in the field of instrumentation, monitoring, and control of advanced hybrid energy systems. As this project continues, it will build new tools for adaptive modeling for process control, state assessment, and system prognostics in tightly coupled, interdependent reactor systems.





## Feedstock Processing & Energy Storage

Daniel M. Ginosar

Developing an INL process to convert gaseous feeds of hydrogen and carbon oxides to produce storable and transportable liquid fuels and chemicals
E1113

The objective of this project is to develop a process to work within the Idaho National Laboratory (INL) Hybrid Energy Systems Testing and Demonstration (HYTEST) framework to convert gaseous feeds of hydrogen and carbon oxides to produce storable and transportable liquid fuels and chemicals. Specifically, this research and development project is capitalizing on the researchers' innovative catalyst to develop a low-pressure, simple and efficient methanol synthesis process that could be integrated into the HYTEST platform. By taking advantage of the HYTEST concept of systems integration, fuels and chemicals could be produced efficiently and economically, reducing the country's dependence on imported fossil fuels and reduce the production of greenhouse gases.

### Project Summary

During the first fiscal year (FY) of the LDRD project, the research team built two syngas-to-liquid fuel experimental facilities. The first system was a laboratory bench-scale catalyst test system designed to work with up to 1 g of catalyst. The second system is the HYTEST Phase I laboratory-scale system designed to work

with up to 100 g of catalyst. The HYTEST Phase I system will have a water electrolysis system to generate hydrogen, a recycle compressor, and a semi-continuous liquid collection system. Tests using the HYTEST Phase I facility are expected to begin early in FY 2011.

In the laboratory bench scale catalyst test system, a methanol catalyst was tested and reaction rate data collected that will be used to guide tests in the HYTEST Phase I facility. Additional catalyst were synthesized and characterized that will enable a one-step reaction to convert syngas to dimethyl ether (DME). DME is a chemical that can be directly converted to liquid fuels and chemicals.

#### Benefits to DOE

This project directly benefits the Department of Energy (DOE) national security missions by developing technologies to produce alternative synthetic liquid fuels. This LDRD has built unique capabilities that increase INL's ability to compete for DOE-Fossil Energy and DOE-Energy Efficiency and Renewable Energy (EERE) syngas-to-fuels research funding.

## Mesocilicate Supported CO<sub>2</sub> Capture Material

Michael Benson

Developing new, more efficient  $CO_2$  capture agents to benefit energy resources, environmental quality, and lower the cost of electricity

EI114

The objective of this work was to synthesize and test solid CO2 capture materials based on substituted triazines tethered to a high surface area solid support. The currently used CO<sub>2</sub> capture agents are liquid amines, such as monoethanolamine. These agents have several drawbacks, such as corrosiveness, volatility, and cost. Due to the weak acid (CO2)/weak base (amine) interaction, allowing capture and subsequent release during regeneration, amines are still the best candidate for CO<sub>2</sub> capture. The materials proposed were based on substituted 1,3,5-triazine. The materials are solid—so volatility will not be an issue—noncorrosive, very versatile, and inexpensive; thus these materials will be an improvement in the state of the art. This work was broken down into four tasks: (1) synthesis of substituted triazines. (2) synthesis of zeolites and attachment of triazines, (3) CO<sub>2</sub> uptake/release characterization, and (4) construction of a working CO<sub>2</sub> capture device. A tether was attached to the substituted triazines, allowing the triazine to be attached to the mesosilicate support. In addition to synthesis, the material was tested for CO<sub>2</sub> uptake and release. A benchtop CO<sub>2</sub> separation apparatus was constructed.

Project Summary

An apparatus was constructed for measuring CO<sub>2</sub> uptake. This is a benchtop system, with a maximum sorbent capacity of roughly 50 g in a column. The flexibility built into the system

allows a variety of gases to be investigated. Currently only nitrogen and carbon dioxide are connected, but sorbent poisons, such as  $SO_x$ ,  $NO_x$ , and  $H_2S$ , could also be included. This system is an improvement from using thermogravimetric analysis (TGA) to measure  $CO_2$  uptake. In the TGA, only one gas can be used at a time, and the gas is passing over the sample, and not through it, as is possible with a column. A column in a temperature swing absorber setup is more realistic for industrial use.

Support materials have been generated using incipient wetness to attach the sorbent to the support. Initial loading has been low, but the results are promising. The most promising Idaho National Laboratory (INL) material (as a non-porous powder) captures 1.90 mol CO<sub>2</sub>/Kg sorbent. After loading onto an alumina support, capture increased to 1.99 mol CO<sub>2</sub>/Kg sorbent, even with low loading. As with any sorbent system, surface area is crucial. The exposure of more functional groups will increase the capacity. Experiments are ongoing to increase the loading onto the support.

Benefits to DOE

Part of the effort for cleaner energy production includes lowering CO<sub>2</sub> emissions. The development of new, more efficient CO<sub>2</sub> capture agents will benefit energy resources, environmental quality, and will lower the cost of electricity.



## **Membrane Separation System Research for the HYTEST Facility**

Frederick F. Stewart, Ph.D.

Chemical Separations research to enable Hybrid Energy Systems
EI115

The objective of this project is to develop membrane-based separations capability for hybrid energy systems (HES). This project has two main thrusts. First, membrane materials will be selected and characterized for their ability to address the separations requirements for various HES configurations. These separations include CO<sub>2</sub> from hydrogen, CO and CO<sub>2</sub> from light hydrocarbons, and dimethyl ether (DME) from CO, CO<sub>2</sub>, and H<sub>2</sub>. Second, the hardware necessary for a "bolt-on" membrane separator will be designed and constructed to handle gas flow rates of 0.25 ft<sup>3</sup>/min. To facilitate these two thrusts, previous laboratory-directed research and development (LDRD), Department of Energy (DOE), and industrial investments in Idaho National Laboratory (INL) will be used. Many promising materials have been developed in our labs and have been characterized at a fundamental level. This project will serve to advance materials from development to deployment. Activities to be performed will focus on the development of high surface area hollow fiber module designs from novel materials. Many materials that have been developed in our labs show higher performance than what is commercially available; however, the knowledge as to how to utilize these materials does not exist. Furthermore, the project will provide the necessary resourcing to adapt, but not limit, this technology to HYTEST, as many of the separations that will be addressed are of regional and national importance.

### Project Summary

During this initial year of the project, efforts were conducted to (1) developing a research needs plan for the inclusion of membrane systems into HES; (2) developing hardware for characterization of membrane modules against syn-gas at temperatures up to 400°C and pressures as high as 400 psi; and (3) selecting and characterizing suitable membrane materials for syn-gas separations. The needs document has

been submitted to the HES project manager. In this document, membrane capabilities were reviewed and clear technological gaps, which are being addressed in this LDRD project, were identified. To meet the separation challenges of HES, a new membrane test system was designed and constructed. This system is capable of meeting the physical and chemical requirements of the HES concepts.

The study of available technologies revealed that the hollow fiber geometry offered the most facile pathway for the development of high flux modules. The technological gap was the lack of knowledge concerning the permeability and selectivity of membranes towards DME. This gap is significant because in hollow fiber modules, a dense polymer layer is applied that provides gas selectivity. Without empirical knowledge about DME behavior, it was unknown which polymer would perform the desired separation. We have begun studies into characterizing polymer membrane performance against the analyte gases (CH<sub>4</sub>, H<sub>2</sub>, CO, CO<sub>2</sub>, and DME). We have determined that DME is a highly condensable gas, meaning that its permeability is driven by solubility. Separation of DME from lesser condensable gases, such as CH<sub>4</sub> and CO, appears to be practical. A manuscript is being prepared for submission to the Chemical Engineering Journal, a peerreviewed journal.

### Benefits to DOE

During this first year, we have determined that membranes are an enabling technology for the HES concept, thus supporting the energy resource and science thrusts within DOE. It is anticipated that in the out years of this project, specific separations capabilities will be designed and constructed to work in unison with other aspects of the HES processes, such as catalysis, to create practical new technologies to reduce the nation's dependence on imported energy.



## Electrochemical Reduction of CO<sub>2</sub> to CO and Hydrocarbon Fuels

Tedd Lister

Developing a cohesive system that could become a pilot-scale operation for the HYTEST facility E1118

This laboratory-directed research and development (LDRD) project, positioned in the carbon-to-chemicals thrust of Idaho National Laboratory's (INL) Science and Technology Directorate, involves developing the technology for electrochemical reduction of CO<sub>2</sub> to syn-gas or simple hydrocarbon products. It hopes to serve as a source of reduced feedstock for follow-on synthetic pathways to commodity chemicals. Electrochemical reduction of CO<sub>2</sub> is a technology that could integrate intermittent alternative energy sources into the electric grid by using excess energy to create a storable energy product. Electrochemical reduction of CO<sub>2</sub> has been demonstrated for many years and can generate high product yields of reduced carbon species using moderate conditions. The INL effort is focused on developing systems reported in lab-scale studies into working benchscale and eventually pilot-scale processes. The challenge is to perform the electrolysis with the minimum energy input and produce a useful product stream at high throughput. A postdoctoral candidate will help the effort. The eventual goal of this project is to develop a cohesive system that, if successful, could become a pilot-scale operation for the HYTEST facility.

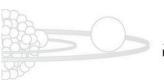
### Project Summary

Initial work has focused on learning more about the process using a bench-scale ambient pressure flow cell. To circumvent issues with CO<sub>2</sub> solubility in water, special gas diffusion electrodes employing a silver catalyst have been utilized. Electrolysis using potassium bicarbonate solutions have yielded syn-gas

mixtures ( $H_2 + CO$ ) while producing  $O_2$  from a potassium hydroxide at the anode. In particular, use of a silver-based cathode has produced high-quality syngas ( $CO + H_2$ ) streams with near 100% yield. In recent tests at INL, syngas streams have been produced with an electrical efficiency of up to 71%. The unique aspect of the INL work was the demonstration of significant reduction in cell power required when the cell electrolytes were heated to near the boiling point of water. The current system measures product gas flow from the cell and an online gas chromatogram to measure composition. To increase throughput, a move to a high-pressure cell design is anticipated.

### Benefits to DOE

This project has initiated INL's effort to develop electricity-based routes to production of hydrocarbon fuels and products from CO<sub>2</sub> and fits into a larger effort to develop technologies that use low-value energy to transform CO<sub>2</sub> into hydrocarbon products. This project addresses issues of integrating the vast renewable energy resources of the Western Inland Energy Corridor, which possesses many natural energy resources but has limited grid connections to transport electricity to larger population areas. Generation of hydrocarbon-based fuels during periods of low-cost power could act as a load level during periods of high power output or low customer usage. Thus this project could be a part of a group of technologies to reduce U.S. dependence on foreign oil imports. Early results were presented at an international conference and a paper describing novel results is in preparation.



## **Hybrid Systems Process Integration and Dynamics Studies**

Robert Cherry

Developing the dynamic models of hybrid energy systems to deliver improved operational efficiency, reduced CO<sub>2</sub> emissions, and reduced consumption of fossil resources

This work has only been going since December 2009—less than one year. The findings have followed the original project intent rather well. We have developed the dynamic models of several hybrid energy systems and with them have characterized the kinds of benefits that hybrid systems can deliver, such as improved operational efficiency, reduced CO<sub>2</sub> emissions, and reduced consumption of finite fossil energy resources.

### Project Summary

This year's work involved three major components. The first was an extensive (and ongoing) literature review to determine the consensus definition of national-level energy security and the types of processes proposed to deliver this. Diversity of energy supply options is the general approach to providing energy security. Many different combinations of energy supplies have been studied. Two areas in which Idaho National Laboratory (INL) is working apparently in advance of other groups are: (1) the use of nuclear power in conjunction with renewable, such as wind and solar, and (2) the use of dynamic energy conversion processes. which make variable ratios of synthetic fuels or electrical power in accordance with the immediate need for power that cannot be stored if not used.

The second aspect of the work has been to define several example hybrid systems and develop system dynamic models to predict their performance over a year's worth of operating conditions, driven by power demand or wind generation rates. We had originally expected that a single general purpose system dynamics model could be used for this and developed by a postdoc. Detailed consideration of the problems showed, however, that the models for each type of system would be rather different (because each problem would have its own calculation sequence based on what data was input or generated) and would not be especially difficult to custom-code each time. Consequently, each model was developed by the PI himself and run to generate the performance data required.

The third aspect of this year's work has been to prepare a manuscript on the results to date. Originally envisioned as one or more papers on the results of the models, it has instead developed as an extended overview paper of how large dynamic hybrid systems can provide national-level energy security, with the three model systems worked this year being used as illustrative examples. This will be submitted before yearend to a refereed journal, most likely Energy and Environmental Science.

As part of planning for the next year's work, we have identified an electrical power storage scheme that appears promising. A new Ph.D. mechanical engineer with background in energy systems is assisting with the preliminary analysis of that idea and, if warranted, will continue to develop it in more detail next year as part of the incorporation of energy storage ideas (such as the concept for geothermal storage of nuclear heat now being examined at the Massachusetts Institute of Technology [MIT] under funding from this laboratory-directed research and development [LDRD] project) into broader hybrid energy system ideas.

### Benefits to DOE

The work is just now reaching fruition, so it would be premature to claim benefits.

Nonetheless, the findings support more strongly than expected the idea that hybrid systems are of national value as an approach to energy security. The work has also identified a particular niche for INL in this area: the use of nuclear power in dynamic energy conversion systems to supply intermediate (beyond base load) power and to compensate for the intermittency of wind and solar power.

This work has deepened INL's capabilities in energy security analysis and in system dynamics modeling of energy conversion processes.



### Conversion of Light Hydrocarbons to Fuels and Chemicals

Lucia M. Petkovic, Harry W. Rollins

This project studies the direct reaction of light hydrocarbons to produce hydrogen and aromatics using molybdenum zeolite catalysts

EII16

The objective of this project is to study the conversion of light hydrocarbons to fuels and chemicals via catalytic dehydro-aromatization reactions. This project is aimed at obtaining the data necessary for integration into HYTEST, a complex of facilities and resources being built at the Idaho National Laboratory (INL) to support hybrid energy systems research and development. This research project started in FY-10 and is planned for 3 years. This first year was dedicated to obtaining the reaction rate data on a molybdenum-zeolite catalyst. Different temperatures, flowrates, and concentrations were studied and spent catalysts were characterized by surface area and coke oxidation to obtain an estimate of the optimum parameters for the process. Experiments during the second year will be aimed at analyzing the effect of heat and mass transfer on product selectivity, conversion, and catalyst deactivation. During the third year, a catalyst regeneration strategy will be selected for the future integration of this process into HYTEST Phase 2.

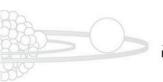
#### Project Summary

A series of catalyst characterization analyses and reaction rate measurements were performed on a molybdenum/zeolite catalyst synthesized by vaporization-deposition of molybdenum oxide. Methane and hydrogen partial pressure were varied and their effect on hydrogen and benzene

production was analyzed. The effect of reaction temperature was also studied. The results will be presented at the annual American Institute of Chemical Engineers Meeting in Salt Lake City, UT, November 11, 2010. This type of publication will contribute to highlight the research work done at INL.

### Benefits to DOE

This work directly supports the future success of HYTEST, a complex of facilities and resources being built at INL to support hybrid energy systems research and development. Development of hybrid energy systems helps attain the DOE's mission of working to assure clean, affordable, and dependable energy supplies for our nation. This project provides knowledge that may be utilized in the production of fuels and chemicals, starting from biomass, a carbon-neutral and renewable source. It also supports science and technology interests by increasing the knowledge about the behavior under different reaction conditions of heterogeneous catalysts. In addition, successful realization and testing of hybrid energy systems will reduce our country's reliance on imported fuels. This project will support those interests by providing crucial parameters for scaling up and analyzing a technology that has never been deployed beyond laboratory scale so far.





Idaho National Laboratory

142

# Nuclear Nonproliferation Initiative





### **Active Interrogation Die-Away Assay Development Program**

David L. Chichester, Augustine J. Caffrey, Edward H. Seabury

Development and demonstration of a prototype portable assay system capable of detecting the presence of shielded SNM

NN113

Today, only passive assay systems are readily available to detect the presence of special nuclear material (SNM) in field situations. Unfortunately, most of these instruments are based on low-energy gamma-ray spectroscopy and gross neutron detection. Therefore, they are ineffectual in reasonable time periods for detecting highly enriched uranium (HEU) hidden behind moderate-thickness shields (for the gamma-ray detector systems) or for detecting plutonium when hidden within legitimate shipments of spontaneous fission neutronemitting radioisotopes (for the neutron detector systems). Indeed, with less than 2 inches of lead, the passive gamma-ray signatures from HEU (not reprocessed) is very difficult to measure by these devices in reasonable assessment times.

This laboratory-directed research and development (LDRD) project seeks to develop and demonstrate a prototype portable assay system capable of detecting the presence of shielded SNM. We are working to develop a system exceeding recent Department of Homeland Security technology goals in this area. To achieve this, we are building upon previous research in active interrogation detection and assessment (AIDA) techniques at Idaho National Laboratory (INL) to detect, identify, and characterize SNM, and we are incorporating more recent INL research related to electronic neutron generators and advanced SNM detection techniques. Our work for this project incorporates simulation and modeling to investigate radiation transport issues effecting active interrogation die-away measurements and other AIDA techniques, as well as laboratory experiments to study instrumentation and engineering issues. Experiments for this project take place at the Active Neutron Interrogation Laboratory and at the Materials and Fuels Complex (MFC) Zero-Power Physics Reactor (ZPPR) facility. A key INL asset supporting these efforts is our accessibility to significant quantities of SNM at ZPPR including varying enrichment HEU samples and different grade plutonium samples.

The AIDA project is focused on developing a prototype portable system capable of detecting shielded SNM hidden inside moderate-sized objects. Specific project goals and milestones outlined in the original proposal that have been addressed in this, the third and final year of the project, include:

- Test ideas for the AIDA approach including delayed neutron, delayed gamma, and prompt/delayed neutron/gamma data fusion
- Select components for constructing a prototype instrument
- Design a final system concept.

### Summary and Conclusions

Active interrogation systems usually rely on a pulsed radiation source to induce nuclear reactions in a target area. We are particularly interested in distinguishing a pulsed neutron source from neutrons produced by fission, either prompt neutrons or delayed neutrons. Figure 1 shows an example of the response of a He-3 proportional counter to a 300-microsecond pulse

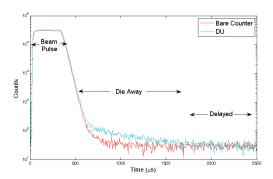


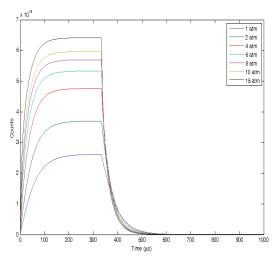
FIGURE 1. The time response of a moderated He-3 proportional counter to a neutron burst from a DT-ENG, in a bare configuration (without fissionable material present), and with depleted uranium (DU) present.

from a deuterium-tritium (DT) electronic neutron generator (ENG) as a function of time The counter reaches a plateau during the neutron pulse, and then the counts decay rapidly to a baseline value associated with cosmic-ray background-induced neutrons scattering about in the room. Detection of neutrons produced through fission of SNM may be considered to occur in three different time regimes. Counting of neutrons in excess of the generator pulse could occur during the beam pulse. Neutrons could also be counted in the die-away region or in the delayed region.



As part of this LDRD project, the MCNPX computer code has been used to perform simulations of the response of He-3 proportional counters to three different neutron sources: a deuterium-deuterium fusion source, a deuterium-tritium fusion source, and a Watt-spectrum source from U-235 fission. Various parameters of the proportional counters have been changed between simulations. These include the He-3 tube pressure, the amount of moderating material around the tube, and whether neutron poisons such as cadmium or boron are wrapped around the counter. In particular, we are interested in the

efficiency and time-response of the neutron counters used for detecting SNM. Figure 2 shows examples of some of the results of this modeling. Helium-3 is currently in short supply for radiation measurements applications. However, for many high-performance national security applications, it remains the material of choice. For special-purpose instrumentation, such as that needed for AIDA, He-3 will likely remain the detection material of choice for several more years until an equally sensitive detection medium is developed.



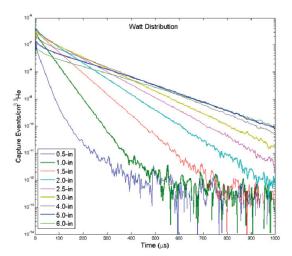


FIGURE 2. The effect of He-3 pressure on neutron sensitivity for a 333-microsecond DT interrogating pulse (left) and the inherent detector die-away time for varying thickness polyethylene moderators around the detector (right).

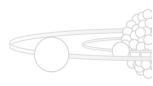
Last year the project focused primarily on investigating the neutron die-away signal from induced fission in fissionable material, caused by neutron reinterrogation of material during the slowing-down process. This year we have continued to evaluate our data from experimental campaigns at INL using fissionable material, primarily focusing on evaluating the delayedneutron signal originating from the decay of short-lived fission products. Figure 3 shows an example of the results of these efforts. Because of this work, a new empirical method has been developed to quantitatively express the detection confidence for active interrogation measurements. This year we have also examined the use of alternate neutron detector technologies for measuring active interrogation signatures,

including these neutron emissions as well as delayed gamma-ray emissions.

Publications resulting from this project are listed below.

Seabury, E. H. and Chichester, D. L., "Monte Carlo Simulations of the Response of Shielded SNM to a Pulsed Neutron Source," Amer. Inst. Phys. Conf Proc. (Submitted) (2010).

Seabury, E. H. and Chichester, D. L., "A Study of <sup>3</sup>He Detectors for Active Interrogation," IEEE Nucl. Sci. Symp. Conference Record, Orlando, Fla., Oct. 24–Nov. 1 (2009) 710–712.



Chichester, D. L. and Seabury, E. H., "Using Electronic Neutron Generators in Active Interrogation to Detect Shielded Fissionable Material," IEEE Trans. Nucl. Sci. 56 (2009) 441–447.

Chichester, D. L. and Seabury, E. H., "Active Neutron Interrogation to Detect Shielded Fissionable Material," Proc. International Topical Meeting on Nuclear Research Applications and Utilization of Accelerators, International Atomic Energy Agency, Vienna, Austria, May 4–8 (2009) SM/EN-02.

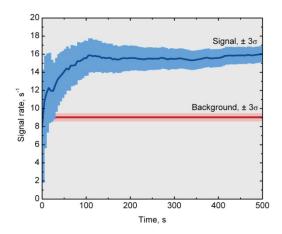


FIGURE 3. This plot demonstrates the buildup in intensity of the delayed neutron emission rate measured during the course of HEU irradiation using a deuterium-tritium electronic neutron generator. The solid lines show the background and signal neutron rates while shaded bands denote the 3-sigma uncertainty in these values.

### Benefit to DOE

This project serves as a foundation-building effort for developing the new research thrust area of portable SNM active interrogation screening and supports INL's nuclear nonproliferation initiative. Primarily, we are investigating the use of active interrogation die-away assay measurements to detect shielded SNM, an effort which both leverages and supports other efforts at the Laboratory related to SNM detection. Indirectly, this LDRD is also laying groundwork to support future work related to the testing and evaluation of active interrogation technology at INL using SNM. Looking towards project completion, we have begun long-term planning to develop industrial partnerships and to transition the work from LDRD funding to external support.



## Xe-135 Recovery from the Spontaneous Fission of Cf-252

Chris McGrath

Techniques for producing fission-product standards other than Xe-135 NN114

This project started as an attempt to develop a way to separate Xe-135 from Cf-252 fission products in order to produce a Xe-135 standard for national security customers. We were able to successfully find a direct funding source and were forced to find a new direction to comply with laboratory-directed research and development (LDRD) program prohibitions against mixing LDRD with direct funds. The direction we chose was to use the remaining time to develop techniques for producing fission-product standards other than Xe-135. Californium-252 would still be the source of fission products for the project.

### Project Summary

In Fiscal Year (FY) 2008, we designed and built a chamber to collect Xe-135 from Cf-252. In FY 2009 we were able to test this chamber and prove that the efficiency was such that this particular technique was possible. At this point we were able to obtain direct funding and deploy this technique. We then broadened the scope and started looking at a wide range of fission products that we could generate as fission product standards. We have performed group separations by oxidations state to prove the efficacy of this methodology. This has shown that producing fission-product standards using Cf-252 is possible and requires only a further refining of the techniques to produce specific isotope standards.

FY 2010 has been a difficult year for this research. For a different project, we have been attempting to purchase a five-microgram electroplated source of Cf-252. We had planned to leverage this source to produce the samples with which to test our production and separation techniques. For non-programmatic reasons, we have been unable to purchase this source, which has made the production of Cf-252 fission products very difficult. Because of this we have been unable to devote the resources to moving this project forward significantly in FY 2010. Significant resources have been devoted to building a model of fission-product production in a Cf-252 source and building the tools to estimate isotope availability under various production and decay conditions. This should allow us to finish the project quickly once the Cf-252 source has been received, and the estimation tools will allow us to "sell" the work to intended customers.

### Benefits to DOE

The ability to produce Xe-135 standards as well as other fission-product standards for an important national security customer is a significant contribution to non-proliferation and related activities. This ties in well with other standards-based activities at Idaho National Laboratory and provides significant efficiencies for our customers.

## Developing a Next-Generation, Risk-Informed Approach for Robust and Resilient Design Development

Trond Bjornard

Applying probabilistic risk assessment to nuclear reactor operations and safety has had a profound and positive impact

NN115

The Department of Energy (DOE) and other agencies' traditional approach to physical security is based on the concept of the design basis threat (DBT), and the technique used to assess a system's performance against the DBT is called vulnerability assessment (VA). VA is performed according to highly prescriptive methodology and assumptions. It assumes that the probability of attack is fixed (1.0) and does not consider consequences explicitly. Thus, VA considers only one of the three terms in the risk equation and ignores the other two. The system is either found to be adequately effective, or not.

Project Summary

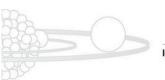
The project hypothesizes that risk-informed techniques from probabilistic risk assessment (PRA) and safety can be applied usefully to the problem of nuclear security and ameliorate some of the shortcomings of the DBT approach. In a risk-informed (R-I) approach, (1) a much broader variety of attack scenarios, threats, and consequences may be considered, and (2) varying frequencies of attack may be considered. This is widely reported as a very difficult problem deriving from the difficulty of predicting adversary behavior. One challenge in the project is to determine if a viable, quantitative approach can be developed. This will be considered in terms of both VA and riskmanagement decisions.

In Fiscal Year 2010, the project objective was to show the use of an R-I approach to a demonstration problem. A report will be issued in October summarizing results of that analysis and comparing them to the DBT results.

### Benefits to DOE

During the first year of the project, potential elements of an R-I physical protection methodologies were developed, and during the second year these elements were exercised against an example problem (hypothetical facility, so as to stay in the unclassified domain). Conclusions drawn and recommendations for continuing work will be reported; most likely this will include more detailed exploration of certain critical elements of the methodology.

Conclusions about the viability of employing risk-informed techniques to the analysis of security performance will be immensely useful to the now-emerging, serious interest by the Nuclear Regulatory Commission to explore the benefits of risk-informed security regulation. It can maintain or enhance security effectiveness while reducing costs. R-I approaches would also be extremely useful in informing the Security by Design process, specifically risk-management aspects.



### **Neutron Spectrometer Development**

David L. Chichester, Edward H. Seabury, Jim Johnson, Scott Watson

Development of high-resolution fast-neutron spectrometry capabilities at Idaho National Laboratory
(INL) exploiting the kinematic behavior of the <sup>3</sup>He(n,p)<sup>3</sup>H reaction

NN116

Research in the area of radiation instrumentation for nuclear nonproliferation and counterproliferation primarily focuses on gamma-ray spectrometers and gross thermalneutron counting detectors; together these present a useful suite of tools for passive screening and active interrogation. One area that has received comparatively less attention has been instrumentation for neutron spectrometry. Neutron spectra contain valuable information about the source material from which they are emitted. For example,  $(\Box,n)$  neutron spectra tend to be highly structured; this structure can provide insight into the material absorbing the alpha particles and generating the neutrons (e.g., Be, B, O, F, etc.). Spontaneous and induced fission produce a smoother continuum of neutron energies that is distinctive from  $(\Box,n)$  spectra. For both types of sources the presence of intervening materials that scatter neutrons can be investigated using fast-neutron spectrometry by assessing the perturbation of the source neutron spectra. For example, neutron spectrometry may be used as a tool for nonproliferation transparency and verification measurements by providing data to determine if plutonium in an inspection object is in a metallic or an oxide form.

One method to determine the energy distribution of a fast-neutron radiation field is to use a <sup>3</sup>He ionization chamber and measure the amplitude of signal pulses from the device. In these detectors neutrons are absorbed by <sup>3</sup>He in the  ${}^{3}\text{He}(n,p){}^{3}\text{H}$  reaction, the resulting proton and tritium atom then proceed to ionize the gas in the ionization chamber. An electronic signal is registered as these electrons and ions are collected. Neutron capture in <sup>3</sup>He is exothermic with a Q value of 0.764 MeV; the amplitude of signal pulses resulting from neutron capture corresponds to the sum of this Q value plus the incident neutron's energy. If proper care is taken to eliminate spurious noise signals and to amplify the small current pulses without adding noise, a neutron energy resolution of better then 20 keV can be achieved using this signal. Specialized <sup>3</sup>He ionization chamber designs have been developed to achieve this level of performance and are described in the literature; these instruments are occasionally referred to as Cuttler-Shalev (C-S) spectrometers, after two

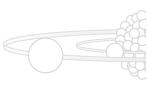
early researchers in the field. More sophisticated detectors have been designed that incorporate <sup>3</sup>He between opposing surface barrier detectors, using solid-state electronics to record the coincident energy deposition from protons and tritium atoms directly, but they are generally limited to small sizes.

This laboratory-directed research and development (LDRD) project is directed towards the study and development of high-resolution fast-neutron spectrometry capabilities at Idaho National Laboratory (INL) exploiting the kinematic behavior of the <sup>3</sup>He(n,p)<sup>3</sup>H reaction. The ultimate goal of this project is to develop techniques for performing fast-neutron detection and spectrometry in the 0.05 - 5 MeV range. The primary method for performing these measurements is to use the C-S style detector. Here, we report on work to evaluate a C-S style neutron spectrometer and to measure the transmission spectrum of a <sup>252</sup>Cf neutron source through several different materials including water, liquid nitrogen, graphite, and magnesium. To support this analysis, Monte Carlo simulations have been used to model our experimental approach and to predict the measured results.

### Project Summary

The Neutron Spectrometer Development Project is studying the technology and application of fast-neutron spectrometry for detecting, identifying, and characterizing fissionable material for nuclear nonproliferation and counterproliferation, and for nuclear forensics. Important project goals and milestones outlined in the original proposal for this LDRD include the following:

- Examine the leading-edge signal phenomenon in <sup>3</sup>He gas proportional counters to develop a baseline understanding
- Assemble bench-top equipment for high-speed signal digitization and digital signal processing for proportional counters
- Develop computational methods for analyzing and using leading-edge pulse shape discrimination from <sup>3</sup>He proportional counters



• Assemble a test fixture for testing <sup>3</sup>He proportional counters.

Originally, one task for this project included the construction of a new <sup>3</sup>He-based neutron detector. However, recent shortages in the worldwide availability of <sup>3</sup>He for research applications has made it difficult to achieve these goals. As a result, the project has focused on studies of a commercial C-S fast neutron spectrometer together with the development of new software algorithms for fast digital pulse shape analysis (dPSA) for analyzing its waveform data stream.

This year we have significantly advanced our fast neutron spectrometry capabilities. In particular, an important measurement campaign took place from November to February, coupled with a series of software development tasks to improve our fast dPSA algorithm library. Initial testing was performed using a deuterium-deuterium (DD) electronic neutron generator (ENG) to produce a monoenergetic neutron field. Following this measurement the neutron generator was replaced with a <sup>252</sup>Cf spontaneous fission neutron source. Data was collected to measure the unaltered neutron spectrum from this source, including room scattering, for 50.5 hours. The C-S spectrometer is a low-efficiency instrument; long counting times are often needed to achieve good results. Data was then taken to measure the neutron spectrum transmitted through different shield materials. The detector used for these experiments was the FNS-100 Fast Neutron Spectrometer, a commercial instrument made by made by Bubble Technologies, Inc. (Ontario, Canada). The detector is functionally equivalent to the FNS-100 previously manufactured by Seforad-Applied Radiation Ltd. (Emek Hayaeden, Israel), a gridded ionization chamber. Data was collected for these experiments using an Acqiris DC282 high-speed waveform digitizer and stored on high-capacity portable hard drives for post processing. Post processing was carried out on a computer using algorithms developed using National Instrument's LabView programming language. Taking advantage of the full capabilities for complete waveform analysis using dPSA, improvements upon prior work were developed, which included the application

of additional filters to remove noise and to discriminate pile-up events in the waveform data.

Simulations were developed to model the experiments, using the MCNP5 radiation transport code. Relatively simple models were initially explored using a Watt-spectrum point source to represent the <sup>252</sup>Cf and a cylinder of <sup>3</sup>He and methane to represent the sensitive volume of the spectrometer. However, it was quickly apparent that a higher-fidelity representation would be needed in order to accurately predict the high-resolution neutron spectra capable of being generated in the experiments. Further models were developed that used a previously created representation of the building—including the floor, walls, and ceiling structures—and that included more detailed representations of the californium source, the source support stand, and the attenuator supporting stand.

Examples of the measured and simulated neutron transmission spectra are shown in Figure 1 (next page). Some discrepancies have been identified between the simulations and the measurements, including a slight difference in the low-energy region: less than 0.75 MeV for the water and graphite, and to a lesser extent for the magnesium transmission measurements. A notable discrepancy between simulation and measurement was also observed for the graphite transmission experiments in the 3.2 to 4.5 MeV range. Further work will be needed to fully understand these differences.

#### Benefits to DOE

Fast-neutron spectrometry may be used as a complimentary analytical method to support nuclear nonproliferation and nuclear safeguards measurements. For example, accurate determination of the high-energy neutron spectrum from an unknown neutron source can be used to identify the target material in alpha-particle-based radioisotope neutron sources. Different low-Z materials produce different characteristic neutron spectra upon alpha-particle irradiation, with different overall energy ranges and very specific energy spectra, and neutron spectrometry may be used to distinguish between these target materials. In some cases neutron spectrometry may be used to



identify the alpha-emitting radioisotope; for example, different actinides emit different energy alpha particles, and in beryllium there is a neutron spectrum dependence on the energy of the incident alpha particles. Neutron spectrometry may also provide forensic-level evidence concerning the mean particle size of the

152

subcomponents in  ${}^9\mathrm{Be}(\square,n)$ -type neutron sources. For safeguards applications, fastneutron spectrometry can be used as a tool for discriminating between metallic and oxide forms of plutonium, as either a direct method or a supporting method in conjunction with gammaray spectrometry.

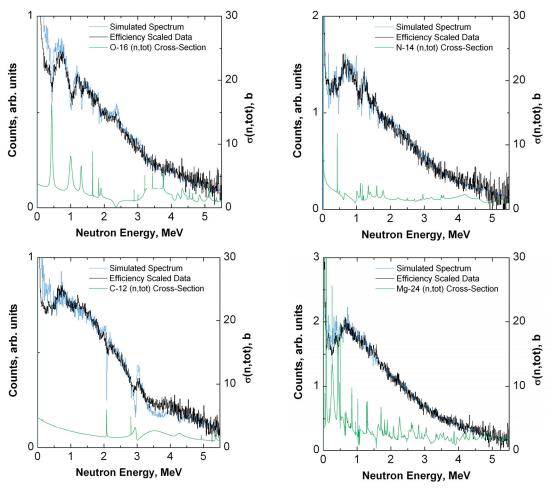


FIGURE 1. Comparisons of the measured neutron transmission spectra and efficiency-scaled simulations for water (upper left), liquid nitrogen (upper right), graphite (lower left), and magnesium (lower right). For reference, these plots also include the principal isotopic neutron scattering cross section for each case.

### **Nuclear Material Detection Using Neutron Time-of-Flight**

Mathew T. Kinlaw, Scott J. Thompson (INL), Alan W. Hunt (Idaho Accelerator Center) The efficacy of utilizing traditional time-of-flight methods for detecting the presence of nuclear material within an adverse, active inspection environment was examined NN117

This laboratory-directed research and development project represents a collaborative effort between the Idaho National Laboratory (INL) and Idaho State University's Idaho Accelerator Center (IAC), focusing on utilizing prompt neutrons, detected and characterized via a time-of-flight (ToF) technique, for possible inclusion into a larger active-inspection methodology. The overarching interest in developing prompt radiation-based techniques is motivated by the potential to generate substantially higher signal rates (100-1000 times greater than delayed neutrons), thus providing several advantages, including decreased inspection times, increased sensitivities, and lower minimum detectable limits. Further, prompt neutrons are emitted with an average energy of  $\sim 1-2$  MeV, which is two to four times larger than the average delayed neutron energy (400–500 keV), leading to greater target shielding penetration and a possible increase in standoff detection distances.

### Project Summary

Previous project tasks focused on assessing two specific neutron detection methods' (neutron ToF and pulse shape discrimination/pulse height analysis) abilities to effectively perform in highbackground, active-inspection environments. Prior work completed at the IAC has demonstrated nToF's effectiveness at performing similar measurements in a low-background, isolated environment [1,2]. However, many possible end-use applications of such technology, including field-deployable, outdoor, and/or standoff detection scenarios, are expected to occur within minimally controlled, highbackground radiation environments. Hence, Fiscal Year 2009 (FY-09) experiments demonstrated the capability to detect the presence of nuclear material, using high-energy prompt fission neutrons, by performing measurements immediately adjacent to the Linac and in the presence of the copious radiation generated by the beam-bending components of the Linac. As a secondary task, measurements were performed in an outdoor active inspection environment, utilizing a transportable Linac originally designed for standoff detection applications. While this Linac has successfully demonstrated its ability to generate photofission

reactions with nuclear materials at increased standoff distances, the specific characteristics of the inspection beam generated with this Linac have yet to be adequately defined. This, in conjunction with the level of energy resolution achieved with previous nToF measurements, directed FY-10 efforts towards employing the nToF methodology as a tool for further characterizing the bremsstrahlung inspection beam generated by an electron Linac. Further, as the current trends in standoff detection requirements continue to extend expected inspection beam energies to increasingly higher levels, remaining FY-10 efforts provided an initial examination of the high-energy prompt neutron signature resulting from inspection beam energies up to 40 MeV.

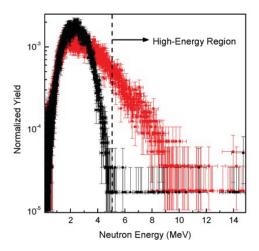
FY-10 technical objectives were defined as follows:

- Develop a method for characterizing bremsstrahlung photon spectra resulting from an electron Linac
- Perform bremsstrahlung characterization measurements with an existing, wellcharacterized Linac for bremsstrahlung endpoint energies from 12–20 MeV.
- Examine and compare a basic high-energy prompt neutron signature from a variety of nuclear and non-nuclear materials with bremsstrahlung endpoint energies up to 40 MeV.

By limiting the maximum energy of the photon inspection beam, a high-energy region can be defined wherein a statistically significant number of detected neutrons provides a signature for the presence of nuclear material. An example is presented in the top plot of Figure 1 (next page), where the photon beam energy was maintained below 12 MeV. Conservation of momentum dictates the maximum energy with which a neutron can be emitted from the NATPb target is ~5.3 MeV. In contrast, fission is induced in the <sup>238</sup>U target, producing prompt neutrons with energies in excess of 10 MeV. While this method base has been utilized and demonstrated previously (Ref. 1,2), the primary tasking in the first year of this project focused on demonstrating similar results were achievable



outside an isolated, low-radiation background environment. Hence, the data presented in Figure were collected immediately adjacent to the Linac, in the presence of an extremely intense photon and neutron background.



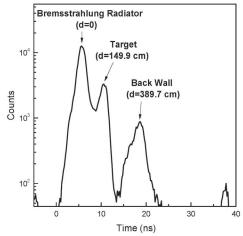


FIGURE 1. Top plot: Prompt neutron energy spectra from  $^{238}U$  ( $\blacksquare$ ) and  $^{NAT}Pb$  ( $\blacksquare$ ), irradiated with a 12-MeV endpoint energy bremsstrahlung beam. The vertical error bars represent  $1\sigma$  statistical uncertainties and the horizontal error bars represent a 2 ns timing uncertainty. Bottom plot: Based on the detection time of the Compton-scattered photons, the locations of the in-beam components can be determined.

During the course of the aforementioned efforts, an additional capability was observed regarding source photons that are Compton-scattered from the target of interest. These photons are typically monitored to determine the precise time at which the fission reactions and/or photonuclear reactions occurred. However, these

scattered photons can also provide information pertaining to the mass densities and localizations of undetermined targets of interest through the implementation of simple spectroscopic and range-finding analysis techniques. As the bottom plot in Figure 1 demonstrates, scattered photon arrival times will be grouped in spectroscopic peaks, each of which represents a beam-scattering target. The centroid of these peaks can used to determine the position for each of these scattering targets by exploiting the invariable speed of the bremsstrahlung photons and the fact that scattering interaction times are effectively instantaneous. The data presented were collected with detectors positioned at a single location, limiting this analysis to one dimension. However, by utilizing multiple detector locations and examining the differences in the timing of the detected events between detectors, individual targets can be localized. Further, photons will inherently interact with high-Z targets with greater probability than with materials composed of low-Z materials, enabling the Compton-scattered photons to serve as an enhancing addition to the high-energy neutron region technique.

More recently, nToF measurements were employed in an effort to more fully characterize the inspection beam. While it is quite common for Linac assemblies to incorporate electron-bending magnets that can be utilized to determine the final energy of the accelerated electrons, the distribution of photons that are actually interacting with the target is either assumed, based upon theoretical calculations, or modeled with computer codes. However, in the absence of an a priori knowledge of the electron beam energy, it remains possible to determine the bremsstrahlung photon energy distributions via nToF. Figure 2 presents the results of these measurements. A deuterium target was irradiated with various endpoint energy bremsstrahlung, and the resulting neutron emission energy spectra were collected and analyzed using the nToF method. Deuterium is somewhat unique in that the photoneutron emission energy directly corresponds to the incident energy of the reaction-inducing photon:  $E_n = (E_v - 2.2245)$ MeV)/2. Hence, the resulting neutron energy spectra, in combination with the previously determined energy-dependent detector efficiency

and ENDF-B/VII cross sections, were extrapolated to provide the initial photon inspection beam energy spectra incident on the target.

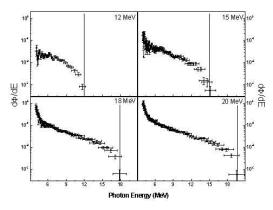


FIGURE 2. Bremsstrahlung photon energy spectra generated with the IAC Santa Barbara Linac, using a 4.2-g·cm² tungsten radiator. Vertical error bars are  $1\sigma$  statistical uncertainty, while the horizontal bars represent a ~2.4 ns timing uncertainty. Dashed vertical lines represent the electron beam energy, corresponding to the expected bremsstrahlung endpoint energy.

As the current interests in standoff material detection continue, increasingly higher inspection-beam energies are being proposed. Hence, to conclude FY-10 research, a preliminary set of data was collected with bremsstrahlung beam endpoint energies up to 40 MeV. Time-of-flight data were again used to calculate neutron energy spectra from various nuclear and non-nuclear materials. The results for three target materials, <sup>238</sup>U (•), Be (•), and Pb (■), are presented in Figure 3. With incident photon energies at this level, neutrons emitted via  $(\gamma,n)$  reactions will carry much higher energies than those originating from fission. Further, the  $(\gamma,n)$  cross-sections for  $^{238}$ U and Pb are quite comparable, leading to emission spectra that appear very similar. Of particular interest are the definitive shape differences between the <sup>238</sup>U (and Pb) and Be spectra. These differences are likely attributable to the stark dissimilarities of the respective cross-sections and reaction thresholds; however, continued investigation is warranted.

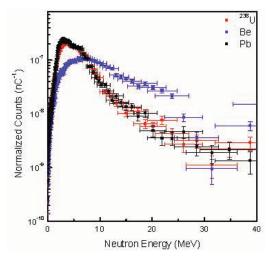


FIGURE 3. Energy spectra for prompt neutrons emitted from  $^{238}U$  ( $\blacksquare$ ), Be ( $\blacksquare$ ), and  $^{nat}Pb$  ( $\blacksquare$ ) targets, following irradiation with 40-MeV endpoint energy bremsstrahlung. Vertical error bars are  $1\sigma$  statistical uncertainty, while the horizontal bars represent a ~2.4 ns timing uncertainty The data are normalized such that the area under each curve is unity.

### Benefits to DOE

This work readily supports the Department of Energy national security mission of developing technologies to detect the presence and/or proliferation of nuclear materials. By furthering the understanding of how characteristic signatures from fissionable isotopes are emitted and how best to utilize and integrate these signatures, this effort is aimed at enhancing the nation's ability to detect and interdict the illegal trafficking or diversion of nuclear materials.



## **On-line Monitoring of Actinide Concentrations for Advanced Aqueous Separation Processes**

Shelly Li

**Develop an online actinide-monitoring sensor that can be applied to aqueous reprocessing systems**NN118

The main goal of this project is to design and develop an online actinide-monitoring sensor that can be applied to aqueous reprocessing systems. Solid-state ionic sensors, utilizing ionic conducting electrolytes, have been developed for decades. The advantages of these types of devices are simplicity, reliability, and high in-situ sensitivity. Even though the basic theory for the potentiometric sensor is well known, the electrolyte materials for actinidemonitoring sensor applications are not currently available. The criteria in selecting actinide sensor materials for the aqueous separation process are: (1) high room-temperature trivalent ionic conductivity (ideally, room-temperature trivalent ionic conductivity >10<sup>-6</sup> S-cm<sup>-1</sup> [siemens per cm]), (2) good corrosion resistance in acidic aqueous solution, (3) good radiation damage resistance, and (4) quick response to concentration changes in the solution. Once the solid electrolyte materials are identified and become available, design of the sensor assembly and performance evaluation will follow.

#### Project Summary

The specific objectives of this laboratory-directed research and development (LDRD) project are:

- Develop a strategy for a real-time actinide monitoring technique for aqueous-based separation processes
- Down select electrolyte material for further development
- Synthesize and characterize surrogate trivalent ion sensor materials
- Design sensor assembly and evaluate performance.

The first year's work focused on developing a strategy for actinide sensor development. Two groups of ionic conducting materials were selected for more detailed study: (1) PVC-based polymer and (2) NASICON-based ceramic. Cerium was selected as a surrogate for actinides.

PVC-based polymer electrolyte materials are known to exhibit adequate low-temperature conductivity and reasonable selectivity. Extensive computer simulation was carried out at Boston University to determine the mechanism limiting the selectivity of polymer electrolyte materials. Density functional theory (DFT) was applied to compute the binding energies between the following ions and a single polymer ionophore: Na<sup>+</sup>, Ag<sup>+</sup>, Tl<sup>+</sup>, Mg<sup>2+</sup>, Co<sup>2+</sup>, Pb<sup>2+</sup>, Zn<sup>2+</sup>, Cu<sup>2+</sup>, Ni<sup>2+</sup>, Cd<sup>2+</sup>, Sr<sup>2+</sup>, Hg<sup>2+</sup>, La<sup>3+</sup>, and Ce<sup>3+</sup>. The results indicate that the binding energy can be a critical parameter to control the ion selectivity.

In the area of inorganic membranes, NASICON-structured oxides were studied extensively. (Ce<sub>0.2</sub>Zr<sub>0.8</sub>)<sub>20/19</sub>Nb(PO<sub>4</sub>)<sub>3</sub> was downselected as the composition for solid electrolyte due to its high trivalent ion conductivity. Extensive effort was devoted to synthesis of the material. The main tasks carried out included: (1) improving density by adding a sintering agent, (2) reducing secondary phases and (3) optimizing electrode/electrolyte interface. High density, nearly single phase material has been obtained by improved fabrication techniques. By reducing electrolyte/electrode interface resistance, the room temperature conductivity of  $(Ce_{0.2}Zr_{0.8})_{20/19}Nb(PO_4)_3$  measured by AC impedance spectroscopy has met the conductivity requirement for the sensor applications (>10<sup>-6</sup> S-cm<sup>-1</sup>). Efforts are underway to eliminate all secondary phases and address corrosion and selectivity issues. Design of a sensor assembly and evaluation of its performance will start in Fiscal Year 2011.

### Benefits to DOE

The main obstacles to the expansion of nuclear energy involve proliferation and safeguard issues. Sensor-based actinide monitoring can enhance nuclear safeguard transparency and efficiency. Successful deployment of this technology will significantly reduce proliferation risks and material control and accountability concerns.



## **Development of a Safeguards Approach for Pyroprocessing**

Richard Metcalf, Robert Bean, Clint Baker, Phillip Casey Durst, David Gerts, Thomas Johnson, Shelley Li, Mark Schanfein, Michael Simpson, Charles Solbrig The development of a standard approach to the safeguards of pyroprocessing facilities is a vital step in the adoption and development of this technology NN119

Large-scale investment in pyroprocessing facilities will only become acceptable if comprehensive safeguards are established to meet the International Atomic Energy Agency's (IAEA) standards for detecting the diversion of significant quantities of nuclear material in a timely manner. Without these safeguards, proliferation concerns will impede the commercial adoption of pyroprocessing and limit the development and expansion of fast reactor technology.

The primary objectives of this laboratorydirected research and development (LDRD) project are to determine the validity of existing safeguards approaches for pyroprocessing facilities and exploit the unique attributes of the pyroprocess to identify new safeguards methods. Idaho National Laboratory's (INL) development of these pyroprocessing safeguards will constitute a new sub-field in reprocessing safeguards and therefore will bring INL recognition as the preeminent experts in international safeguards applied to pyroprocessing facilities. INL's unique pyroprocessing facilities create an ideal opportunity to take the lead on this topic. Further objectives of this project include:

- Continuing the advancement of the diversion pathway analysis, including mitigation actions
- Investigating proposed safeguards methods, depending upon the Pu/Cm ratio in the fuel
- Investigating and developing capability to perform safeguards measurements within the hot cell
- Investigating application of goals-driven safeguards to pyroprocessing facilities
- Designing and fabricating the digital cloud chamber and the further development of the inverse spectroscopy algorithm

- Conducting advanced laboratory analysis of the Curium content of selected fuel samples from INL's pyroprocessing facility
- Investigating the installation of IAEA safeguards equipment at INL's Fuel Conditioning Facility (FCF).

### Project Summary

A component-by-component diversion pathway analysis has been performed; the results have provided insight on the mitigation needs and equipment development needed for a valid safeguards approach. The effort to develop an inhot-cell detection capability led to the digital cloud chamber, and more importantly, the significant potential scientific breakthrough of the inverse spectroscopy algorithm, including the ability to identify energy *and spatial location* of gamma-ray-emitting sources with a single, noncomplex, stationary radiation detector system. A solid cost estimate of equipment installation at FCF has been developed to guide proposals and cost allocations to use FCF as a test bed.

### Benefits to DOE

The Department of Energy (via NNSA/NA-24) is committed to supporting IAEA and the international safeguards regime. This project has helped INL gain prominence in pyroprocessing safeguards, and will continue to allow the INL to provide expertise to assist the IAEA's development of an accepted standard approach.

The digital cloud chamber has potential application to the measurement and safeguarding of stored spent nuclear fuel. It may be able to measure the plutonium content of the spent fuel to a precision of a few percent, an impossible feat with existing NDA systems.

The inverse spectroscopy algorithm that was developed represents a significant breakthrough. It may be possible to determine the energy *and location* of a radiation source using one noncomplex detector system.



## Radionuclide Collection-Detection Device for Remote Monitoring of Tc-99 as a Proliferation Indicator

T. J. Tranter, M. R. Greenhalgh

Preparing and characterizing scintillating extractive resins for the quantification of Tc-99 NN120

The research objective is to produce the experimental data necessary for the development of an integrated radioisotope-collection-detection device capable of concentrating and detecting specific beta or alpha-emitting radioisotopes in situ and transmitting the data to offsite locations via telemetry and satellite link. The device will enable the target radioisotope to be concentrated inside the actual detector, providing very high counting efficiencies and the ability to passively collect the radionuclide while the device is in sleep mode. Specifically, the work under this proposal will be focused on a device for concentrating and measuring the fissionproduced radionuclide technetium-99 (<sup>99</sup>Tc), which can be used as a key identifier of nuclear fuel reprocessing to recover <sup>239</sup>Pu or uranium enrichment to produce highly enriched <sup>235</sup>U. The ability to accurately detect and quantify low levels of <sup>99</sup>Tc is of primary importance in determining, monitoring, and assessing declared or undeclared nuclear proliferation activities.

### Results and Accomplishments

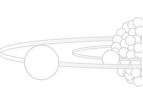
Technetium selective ion-exchange resins have been prepared on three different polystyrene support materials with varying surface areas (900, 750 and 500  $\text{m}^2/\text{g}$ ) by impregnating the support material with a quaternary amine extractant. The amount of extractant added to the support material was varied to test the extraction capacities of each support material. The surface areas of the newly prepared resins were measured to monitor the surface area effect of the resin preparation. Batch contacts were performed with the developed ionexchange resins with technetium-99m (99mTc). technetium-99 (99Tc), and rhenium (Re) in southeastern Idaho well water with a pH  $\approx$  8. The data from these experiments has been used to create isotherms of quantity absorbed versus the quantity remaining in the aqueous phase. The isotherms were also compared to the commercially available Eichrom TEVA resin.

Scintillating ion-exchange resins were prepared with extractant, various scintillating fluors, and wavelength shifters and evaluated for detection efficiency and extraction capacity. Extraction capacities of the scintillating resins were compared with the observed uptake capacities of the non-scintillating resins. The data indicates the detection efficiency of <sup>99</sup>Tc in the prepared resins ranged from 50–70%, and the extraction capacity was only mildly affected by the additional loading with scintillating fluors and wavelength shifters.

In 2010, a new surface area analyzer was acquired that has been used to characterize the newly prepared resins, adding a new capability to the department that can be used in materials analysis. Expertise and capability has been gained in development of scintillating ion-exchange resins that can be used to concentrate and detect radionuclides and will strengthen Idaho National Laboratory's reputation. Two summer interns were also funded as a result of this project.

#### Benefits to DOE

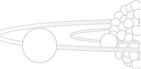
The proposed system could provide a rapid and more cost effective alternative for *in situ* radionuclide measurements of contaminated groundwater at the various DOE sites throughout the U.S. This work would fill a niche in the proliferation detection area that has not been previously exploited to its fullest potential. The capability gained from this work would add a needed tool for the detection of undeclared activities as well as the monitoring of declared activities.



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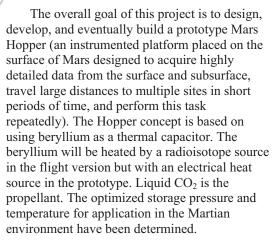


160

### **Design and Testing of a Mars Hopper**

Jim Werner

Designing, developing, and building a prototype vehicle to explore the surface of Mars in greater detail IN101



### Project Summary

A point design of the Hopper has been completed. To complete this project, a team was formed from Idaho National Laboratory (INL), the Center for Space Nuclear Research (CSNR), and three universities—Utah State, Oregon State, and the University of Idaho. The major issues that have to be resolved are thermal isolation of the beryllium core, heat transfer from the core to the propellant, choice of insulator materials, design of the core with respect to flow channel size and number, and testing and validation of the material behavior during the thermal cycle. In addition, assessment of the benefits of the Hopper concept to space exploration missions is needed to attract participation by NASA HQ and NASA centers.

Computational models for the thermal isolation and the heat transfer issues have been built in both STAR-CCM and COMSOL codes. In addition, the CO<sub>2</sub> liquefication system has been designed. Finally, beryllium rods coated in Hastelloy have been acquired. More specifically, the following tasks have been accomplished: In the CSNR Summer Fellows program, the fellows assessed the feasibility of a Hopper on the moon Europa, designed a Mars sample return mission enabled by the Mars Hopper, and designed a 10 kWe pulsed power system for application in Low Earth Orbit:

 Thermal isolation modeling – Steady-state temperature profiles of the core with various insulators has been accomplished; a STAR-CCM model has been built that includes the

- core, the pressure vessel, the CO<sub>2</sub> storage tank, and the Martian atmosphere.
- 2. Heat transfer models Time-dependent heat transfer calculations for CO<sub>2</sub> at various entrance pressures have been completed using Matlab, STAR-CCM, and COMSOL.
- 3. Power conversion systems Various power conversion cycles have been assessed for use with the Hopper and the masses estimated.
- CO<sub>2</sub> liquefaction system design A cryocooler based system that uses 250 W has been designed that can provide sufficient CO<sub>2</sub> in a 7-day interval.
- Beryllium rods Beryllium rods with two different cross sections have been purchased.
- Heat source development An electrically heated tungsten rod heat source has been designed and discussions with the NASA Marshall Space Flight Center initiated.
- 7. LENS production of tungsten Samples of tungsten powder have been processed in the LENS system; densities and material properties do not appear suitable.

### Benefits to DOE

This research has enhanced the capabilities of INL and the CSNR to computationally model heat transfer in turbulent flow conditions and to use complex, multi-physics codes to model coupled systems. In addition, the test rig will add to the infrastructure of the CAES laboratories. Because of university participation, the region has benefited in having roughly twelve students design subsystems for the Mars Hopper. Nationally, the Mars Hopper concept could revolutionize planetary exploration and could provide 100 times the science per launch, i.e. greatly reduce the cost. Finally, the DOE benefits from the visibility of developing innovative concepts for space exploration. Expertise in effectively using multi-physics codes enables lab researchers to work on a number of different national programs. For example, the modeling of structural changes resulting from heat transfer is applicable to ground-based nuclear systems. In addition, the ability to fabricate tungsten parts using the Spark Plasma Sintering furnace is still a rare capability in the U.S.



## In-situ Characterization of an Oxide Film for Prediction of Stress Corrosion Cracking Susceptibility

Sebastien Teysseyre

Developing an apparatus to perform multiple spectroscopy tasks IN102

This project involves the development of an apparatus that will allow the simultaneous performance of in-situ Raman spectroscopy, and electrochemical and laser acoustics measurements on a sample exposed to a boiling water reactor/pressurized water reactor (BWR/PWR) environment. This equipment will allow the study of the growth of the oxide layer in BWR/PWR environment, its reaction to changes in the water chemistry, and its behavior under stress.

It is believed that changes in the oxide layer composition are linked to stress corrosion cracking initiation, and that the efficiency of mitigation techniques and the long-term influence of a temporary loss of water chemistry control can be determined by in situ characterization of such changes.

### Project Summary

This project started about 4 months ago. The first tasks consisted of designing the water loop and chemistry control, reception of the Raman spectrometers, and training of the personnel.

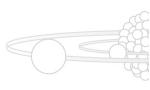
Two Raman spectrometers, a micro-Raman LabRam HR and a macro Raman spectrometer iHR550 from Horiba, and one laser (Verdi V2) were received and installed. The various optics and instruments have been installed on an optical table in IRC-B3 and the equipment has been tested. Oxide powders (Fe<sub>3</sub>O<sub>4</sub>, NiCrO<sub>4</sub>, NiFe<sub>2</sub>O<sub>4</sub>), relevant for future in situ analyzes, were used to acquire data and analyze spectra, which allowed personnel training and preparation of a database for analysis of complex oxide layers. Similar work was performed on the pre-oxidized sample made of X750.

The design of the water loop is complete and the parts that we could afford on the Fiscal Year 2010 (FY-10) budget were purchased. Although the autoclave design is not complete (it is a FY-11 task), the sapphire window to be mounted on the autoclave has been selected, purchased, and is being tested as it is necessary to know the spectrum generated when the laser goes through the sapphire window.

### Benefits to DOE

Stress corrosion cracking and irradiation stress corrosion cracking are phenomena that challenge the integrity on our nuclear power plants (energy resources) and the long term storage of nuclear waste (environmental quality). Therefore, they challenge the capability of the U.S.A. to extend the lifetime of the current fleet, impose down times that increase the cost of energy production, raise concern for the design of the next generation of nuclear reactors and our ability to achieve energy independence.

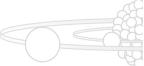
This work, and the equipment developed, will help in the study of stress corrosion cracking mitigation techniques and predict the deleterious effect of chemistry changes in the operation of a nuclear power plant. It will also permit an increase in fundamental knowledge about a phenomena present in many industries.



Center for Advanced Energy Studies







164

## **Investigation of Public Discourse Methods in Energy Policy Decision-making**

Steven J Piet, Jeffrey Joe, INL; Carole Nemnich, Mike Louis, BSU; Ann Hunter, ISU; Troy Hall, Patrick Wilson, UI

Reducing project risk by encouraging the public to engage in public and social issues

The team published *The Effects of Public* Discourse in Energy Policy Decision-Making (INL/EXT-10-19546) in September 2010 as the final report of a three-year effort to test different public discourse methods in the subject area of energy policy decision-making. We analyzed 504 mail-in surveys and 75 meeting participants in deliberation groups on the Boise State University campus for their (a) reflective preference (spontaneous) answers), (b) financial support, and (c) evaluations of eight attributes for five energy options: (1) energy conservation and efficiency, (2) fossil fuels, (3) nuclear energy, (4) hydropower, and (5) renewable energy. All meeting participants saw a diverse, seven-person energy expert panel. Some participants attended deliberation discussion sessions; some received a 35-page briefing document that included pros and cons of the different energy options. Of those who received the briefing paper, 90% viewed it positively, meaning our multi-discipline and multiviewpoint approach to preparing the briefing paper achieved credibility among those with different energy option preferences.

Project Summary

"With public sentiment nothing can fail; without it nothing can succeed."

- Abraham Lincoln

Many projects have failed because of strong public opposition, including natural gas and coal power plants proposed in Idaho over the past several years. This Idaho National Laboratory (INL), Boise State University, Idaho State University, and University of Idaho joint project has aimed to reduce project risk through encouraging the public to engage in more critical thought and be more actively involved in public or social issues.

Early in a project, project managers and decision-makers can talk with no one, pro and con stakeholder groups, or members of the public. Experience has shown that talking with no one outside of the project incurs high risk because opposition stakeholders have many means to stop most (if not all) energy projects. Talking with organized stakeholder groups

provides some risk reduction from mutual learning, but organized groups tend not to change positions except under conditions of a negotiated settlement. Achieving a negotiated settlement may be impossible. Furthermore, opposition often arises outside pre-existing groups.

Standard public polling provides some information but does not reveal underlying motivations, intensity of attitudes, etc. Improved methods are needed that probe deeper into stakeholder (organized groups and members of the public) values and beliefs (sometimes called heuristics) to increase the potential for change of opinions and/or out-of-box solutions. The term "heuristics" refers to the mental short-cuts, underlying beliefs, and paradigms that everyone uses to filter and interpret information, to interpret what is around us, and to guide our actions and decisions.

### Results and Accomplishments

Compared with the average Idahoan, due to self-selection versus the commercially prepared, demographically balanced mailing list, respondents were predominantly male (78% vs. 49%), formally educated (50% with four-year degree versus 16% of Idahoans in general), and more engaged than the average Idaho resident (96% claimed to have voted in an election in the past two years versus 61% that voted in the 2008 general election). Respondents were older, mostly Caucasian, and had lived in the state longer than the average Idahoan.

The percentage of all respondents with reflective preference for (positive scores on a scale of -5 to +5) each energy option was 92% for renewables, 92% conservation, 87% hydropower, 69% nuclear, and 47% fossil fuels. The distribution of answers for nuclear and fossil fuels showed polarization, with "strongly support" and "strongly oppose" as the two most common answers (+5 and -5 on the 11-point scale). All of the energy options had positive mean reflective preference (>0 on the 11-point scale), although that of fossil fuels was quite low (0.06).

Turning from reflective preference to financial support, we asked respondents and participants how they thought a power company

should allocate a hypothetical \$100 to buy energy to meet electricity demand. People did not want to depend on only a single energy option. Only about 3% would give all \$100 to a single energy option. Few respondents would put more than 50% of the \$100 to a single energy option. "Few" means 10% for renewable energy, 8% for nuclear energy, 7% for hydropower, 2% for conservation and efficiency, and 0% for fossil fuels. A significant fraction (but still a minority) would give no financial support to fossil fuels (48%) or nuclear energy (34%).

No one provided financial support for an option he or she opposed. On the opposite side of the scale, it is striking that people appear to require a very strong preference (a +5 on the -5 to +5 scale) before they wanted to provide more than average (>\$20) financial support as indicated by the consistent fraction of +5 preference with >\$20 financial support.

Responders tended to give the average \$20 support to energy options they gave a +4 support.

Respondents differentiated between the eight energy attributes fit each energy option.

For both renewable energy and energy conservation and efficiency, the attribute that respondents evaluated as best fitting these two energy options was "safety and security." In contrast, the attribute least matching these energy options was "cost." That is, cost was not typically viewed as a positive attribute of these two energy options.

For hydropower, the best-matched energy attribute was "safety and security" and the least-matched was "responsiveness and adaptability." In contrast, one of the seven energy panelists argued that the key disadvantage of hydropower was "impact to the environment."

For nuclear energy, the best-matched energy attribute was "reliable and predictable" and the least-matched was "cost." Surprisingly, it wasn't "impact to the environment" (e.g., waste) nor "safety and security."

For fossil fuels, the best-matched energy attribute was "reliable and predictable" and the least-matched was "impact to environment." Based on written comments on the surveys and

the follow-up telephone interviews eight months later, the latter is often associated, at least in part, with climate change.

We hypothesized that reflective preference resulted from a "formative preference" that people (generally unconsciously) create by their weighted evaluations of the attributes among energy options. We found that indeed reflective preference was positively correlated with formative preference. Reflective preference was also correlated with financial support of energy options, as noted above. That is, mail-in respondents and deliberation participants were generally internally self-consistent in their simple reflective preference, allocation of utility bill, and detailed assessments of energy options.

We found several other things of note. For example, 38% want energy policy decisions made by citizens. The rest chose business and industries (22%), government agencies (22%), elected officials (16%), and advocacy groups (4%).

Those who chose to participate in the deliberative sessions on the Boise State campus were slightly more knowledgeable than the average mail-in survey respondent. We asked, "Of the electricity that Idaho produces, the majority comes from what one source?" Of mail-in respondents, 86% answered correctly (hydropower) compared to 97% of those who attended the deliberative event. (This rose to 99% after the event.) We also asked, "Within its borders, Idaho has abundant resources of which of the following?" Of mail-in respondents, 68% answered correctly (hydropower) compared to 93% of those who attended the deliberative event. (This rose to 99% after the event.)

Those who attended the event increased in knowledge. We asked "Of the electricity that Idaho consumes, the majority comes from what one source?" The correct answer is fossil fuels, the source of most of the electricity we import from out of state; Idaho imports one-half to two-thirds of its electricity. The percent answering correctly rose from 17% pre-test to 53% post-test.

We asked participants to evaluate the seven energy panelists. The participants tended to rank each of the energy experts about the same



whether the question was credibility, trustworthiness, knowledge, and likability. For example, they did not like those they viewed as less credible. Evaluations of panelists differed both systematically (i.e., some were viewed more positively than others independent of energy preference) and by participants' energy preference. Statistically, the trend was that participants rated their regard for a panelist based on the extent to which that panelist agreed with the participants' prior positions.

Although knowledge was gained by those participating in the event, reflective preference, formative preference, and financial support all changed very little, independent of the discourse treatment. Our analysis indicates that people had strong mental models or heuristics before the deliberation event and were not given sufficient reason to change during the event.

There is no quick fix. Neither neutral briefing documents nor a balanced panel of experts substantially changed the views of the public in any direction. Opinions tend to change slowly.

More involved discourse methods (e.g., Deliberative Polling) can improve insights on public values and preferences. However, it can be difficult to get a representative sample. Those that participate tend to have existing opinions.

If one doubts the need to go beyond past methods, there are a number of anecdotal and more formally studied examples of when the government, or some other powerful entity, chose to use a Decide-Announce-Defend (DAD) strategy in their public engagement, only to have it backfire on the powerful entity.

- Department of Energy (DOE) decision to build a waste incinerator at INL, defeated by Keep Yellowstone Nuclear Free.
- DOE's effort in recent years to consolidate all Pu238 radioisotope production and power systems at INL.
- Two failed efforts to build natural gas electricity plants in northern Idaho, defeated via preventing access to water for cooling by a coalition of environmental groups, farmers, and labor unions.

- A failed effort to build a coal-fired electricity plant in southern Idaho.
- Executive summary of a report from Pacific Northwest National Laboratory, PNL-14200, "An Evaluation of DOE-EM Public Participation Programs."
- The most famous analysis of the use of DAD at Yucca Mountain, is "Uncertainty Underground: Yucca Mountain and the Nation's High-Level Nuclear Waste" by A. M. Macfarlane and R. C. Ewing. A review of the book states, "She [Macfarlane] also emphasizes that the process of selection of Yucca Mountain as a proposed site is an extreme example of the 'decide, announce, defend' strategy that has largely been rejected by the European nations and she comments favourably on the consensus building approaches that have been developed, e.g. in Sweden and Germany."
- "Talking Policy: An examination of public dialogue in science and technology policy" by Steven Wooding, Amanda Scoggins, Pernilla Lundin, Tom Ling for the RAND corporation.
- "Managing and leading: 52 lessons learned for engineers" by S. G. Walesh reads: "The traditional decide-announce-defend (DAD) approach is no longer appropriate. The progressive and inclusive public owns project (POP) view is more likely."
- Chapter 5 of "Communication and public participation in environmental decision making" by Stephen P. Depoe, John W. Delicath, Marie-France Aepli Elsenbeer.
- Brent spar, start with: http://en.wikipedia.org/wiki/Brent\_spar.

### Benefits to DOE

Public perceptions and values are integral to national policy prescriptions. In the quest to increase the nation's energy supplies, modernize energy infrastructure, and ensure optimal uses of energy resources, while meeting environmental and international constraints, solutions that are (understood and) acceptable to the public have a higher likelihood of success. Methods of public discourse that will allow scientists to engage and





inform the public about the positive and negative aspects of different energy generating technologies in moving toward energy independence are imperative in framing the energy policy debate. The methods will improve our ability to measure the level of agreement, how opinions shift, and possibly how deeply held the positions are.



## **Development of Lignocellulosic Ethanol Production Potential** in Idaho

Greg Bala
Solutions for high feedstock cost for biofuels production
CA116

A compelling need exists for new sources of clean, inexpensive, and renewable energy to replace/augment petroleum-based energy systems. One promising route of exploration includes discovery and development of new bioenergy strategies to enhance the efficiency of biomass conversion to chemicals and fuels and improve our ability to harness biofuel-producing capabilities of biological catalysts. Such bioenergy strategies can significantly expand our current régime of energy-producing technologies and therefore may play a significant role in transitioning our petroleum-dependent energy system to one based on clean, carbon-neutral renewable energy sources.

Advancements in biofuels research and technology can provide huge economic benefits for the State of Idaho. Idaho's agricultural activities result in large amounts of potential feedstocks for biofuel production—feedstocks that are often viewed as waste to the producer (i.e., corn stover, potato processing waste, sugar beet processing waste, etc.). Exploiting such "waste" feedstocks via the coupling of new biotechnologies and biofuels production facilities can create a new industry base for Idaho.

#### Project Summary

Our goal is to use expertise and collaboration among scientists at Idaho National Laboratory (INL), Boise State University (BSU), Idaho State University (ISU), and the University of Idaho (UI) to provide solutions for the problem of high feedstock cost for biofuels production. Novel feedstocks available within the State of Idaho (e.g., lignocellulosic) agricultural wastes or products may allow ethanol producers in the state and region to increase production capacity and improve profitability.

Importantly, this project forms the foundation for the focus area of Idaho materials as it strives to widen the applicable envelope of operations for ethanol production within the state. In addition, it fully supports the strategic goals of the Center for Advanced Energy Studies (CAES) in that it provides an existing, tangible connection between state research institutions and CAES that is already focused and engaged in bioenergy research.

In brief, ISU and BSU continue to evaluate biological catalysts and mechanisms for integrating catalysts into reactor platforms for optimum performance while UI is focused on reactor design and operations. Collectively, we now focus on the biological transformation of high-volume, low-value wastes to precursor compounds useful for biofuels production. We are currently evaluating waste effluents from the dairy industry such that we directly complement the proposed effort of the CAES Bioenergy Center. The new Bioenergy Center project proposes to utilize anaerobic digestion and associated technologies to convert dairy waste to value-added components including energy. This laboratory-directed research and development (LDRD) project integrates a microbiological evaluation of the conversion processes needed for entry into the proposed technology platforms.

### Benefits to DOE

In terms of building on accomplishments from last year, we have greatly extended previous work by further characterization of microbial isolates and their enzymes, and by initiating reactor studies using the same base materials that yielded the isolates DSX5 and DSX6. In addition, we have refined and optimized reactor conditions to improve encapsulated saccharification rates. Furthermore, last year's work to successfully immobilize Trichoderma reesei has allowed us to begin developing novel reactor designs and investigating the utility of calcium alginate hydro-gel in long-term reactor conditions. We have also leveraged funds and findings to build important capabilities at UI to evaluate stabilized enzymes and measure sugars produced by hydrolyzing material from lignocellulosic biomass, and we have developed techniques for conventional strong acid hydrolysis of biomass so that we will be able to compare this technique to the encapsulated enzyme approach.

A 20-liter pilot plant has been designed that will use the encapsulated enzyme beads to convert biomass to simple sugars which can then be fermented to ethanol, and we expect fabrication to begin during the last month of Fiscal Year 2010.



With respect to progress against the hypothesis of the original proposal, we have continued effort to optimize the calcium alginate matrix and have developed encapsulation and reactor conditions that result in approximately equal cellulytic activity observed in the unencapsulated treatments. While this result doesn't reflect a significant increase in enzyme production rate and activity over the unencapsulated organism, this work has resulted in generating an organism/encapsulation matrix platform that may be more amenable to operating and maintaining a meso-scale saccrification reactor. The encapsulation of *T. reesei* in alginate allows for simple and rapid separation of the fungal biomass from the enzyme-containing reactor supernatant. This property of the encapsulated whole organism catalyst simplifies reactor design and operation by reducing viscosity of the enzyme-producing reactor and minimizing loss of active fungal biomass between reactor runs (e.g., enzyme production batches).

We have been able to recycle encapsulated T. reesei for several weeks by simply stopping agitation of the reactor and allowing the encapsulated organism to separate from the enzyme-containing supernatant by settling to the bottom of the flask, a process that takes a few seconds. After removal of the supernatant new minimal media was added to the encapsulated organism and the incubation continued. Subsequent incubations of the recycled encapsulated biomass produced increasingly higher levels of filter paper activity for up to three rounds of incubation. After three rounds of incubation, the levels of biomass in the culture supernatant approach that of the unencapsulated treatment. However, it is important to point out that our biomass measure is a total protein assay (i.e., Bradford assay) and that the encapsulated treatment supernatant still appeared to clear rapidly when the reactor agitation was stopped, unlike the unencapsualted treatment. Therefore, it is possible that the high levels of "biomass" in the encapsulated treatment after three rounds of incubation could be primarily extracellular protein and not intact fungal biomass. Additional measures of fungal biomass are underway to confirm if the high protein levels in these treatments reflect high levels of fungal hyphae or if they indicate a protein-rich supernatant. Together these results demonstrate that the encapsulation strategy is an easy way to separate the enzyme supernatant from the organism for use in downstream applications.

## Results and Accomplishments

New capabilities and resources that have been generated as a result of this project include a culture collection of biocatalysts available to regional researchers. This project has also enabled continuing collaboration and funding in with the biofuels industry. Significant progress has also been made in methods development, particularly within the area of proteomics. The proteomics work resulted in a Pacific Northwest National Laboratory (PNNL)-Environmental Monitoring System Laboratory (EMSL) user agreement, and part of the proposed work includes proteomic methods development (this is important as the equipment for this work is very specialized and expensive). Thus, we have access to high-power analytical systems for proteomic analysis of biocatalyst proteins based on the capabilities built in this project.

Further, we have optimized conditions for small bench-scale cellulase production. This information can be used to help design novel larger volume continuous flow reactors. These physical and environmental reactor modulations will not only be applicable to current strains of cellulose-producing organisms but will still be relevant as the field of genetics continues to provide industry with new and improved strains of lignocelluloses-degrading microorganisms.

Additional equipment now located at the universities includes a high-performance liquid chromatography (HPLC)-based analytical technique for measuring sugars in hydrolyzed products from lignocellulosic biomass, as well as a compliment of processing equipment that will be utilized in reactor fabrication. Techniques include a technique for pretreating biomass with a ball mill and for using strong acid hydrolysis to produce sugars in a manner that can be used as a baseline for our on-going enzyme work.

This research has provided numerous benefits including: (1) a long-term collaborative relationship between researchers at UI, BSU, ISU and INL; (2) expertise and research capacity



at the three state universities in a field of interest to INL, the region, the nation, and DOE; (3) education for a Ph.D. student at UI and several graduate and undergraduate students at BSU and ISU; and (4) scientific data for the feasibility of encapsulated enzymes as a tool for conversion of lignoceullulosic biomass to ethanol. Specifically, tangible benefits include:

- Magnuson and ISU are continuing collaboration with VerdureTech Inc., a company based in Pocatello, in investigating large-scale conversion of animal waste to methane. This collaboration is a natural and very beneficial spinoff from the present CAES project. Goals include characterization of microbial communities in treatment ponds, and isolation of phototrophs involved in carbon processing.
- Members of the CAES team (Hamilton/Bala/Feris) were recently approached by a venture capital firm from the Boise area to aid in scoping a technology evaluation for a process that is intended to augment current waste water treatment plants for efficiency of waste treatment and potentially provide a useable energy stream. Components of this research team have been actively engaged in the evaluation. The outcome is a collaborative project driven by attempts to secure private funding. Technical execution includes the state universities (BSU/UI). This provides a tangible example of others recognizing the CAES research team as an important technical resource.
- Reactor platforms developed at UI will be valuable research and teaching tools capable of collaborative integration with other research areas (e.g., the CAES Bioenergy Center). The reactor is expected to be modular and therefore able to exhibit the flexibility required for analysis of other feedstocks or processes.

• BSU is investigating the possibility of pursuing Intellectual Property disclosure based on encapsulation of *T. reesei* as a novel approach for on-site catalyst generation for lignocellulose hydrolysis.

### Benefit to DOE

- This work supports the following topical DOE Energy Mission statements:
  - o To advance the national, economic, and energy security of the United States
  - To promote scientific and technological innovation in support of economic and energy security.
- The work directly supports the DOE Strategic Goal for Energy Efficiency and Renewable Energy (EERE): "To protect our national and economic security by promoting a diverse supply of reliable. affordable and environmentally sound energy." This is implemented by the following EERE strategic goals: (1) "Dramatically reduce or even end dependence on foreign oil" and (2) "Create the new, domestic bioindustry." Supporting goals of the Biomass Program include the topical areas or feedstocks, sugar platforms, products, and integrated biorefineries. As a composite, these topical areas all address the production of fuels and materials, albeit through different pathways or intermediates.



## **Optimization of Ceramic Waste Forms Used for Electrochemical Processing of Spent Nuclear Fuel**

Michael Simpson

Progress has been made towards understanding how to minimize salt waste volume CA117

This project aims to develop technology for minimizing the volume of high-level nuclear waste generated from pyroprocessing of spent fuel. The approach being investigated includes zone-freeze refining of salt to recycle the majority of useful LiCl-KCl back to the electrorefiners. Zone freezing concentrates fission products into approximately 20% of the original salt volume. To then immobilize the concentrated salt phase into a waste form, sorption of that salt into zeolite-A is being studied. Ultimately, it is expected that the volume of high-level waste can be reduced by a factor of 5 or more compared to current technology.

## Project Summary

The overall strategy of this project is to use experiments with zone freezing and salt sorption to develop models for these two processes. Those models can then be used to design and optimize an integrated salt treatment/waste fabrication process to be coupled with spent fuel electrorefining. Experimental and modeling progress has been made in the first year of the project. A zone freezing model has been derived from fundamental transport phenomena equations and numerically solved using a finite differences algorithm. A zone freezing furnace for testing the process with small (<100 grams) samples of salt has been procured, installed, and subjected to preliminary testing. Temperature

profiles in the salt have been measured and compared to the model calculations. In parallel, salt-zeolite sorption has been studied with single chloride salts at gram-scale to test the ability of the zeolite to absorb chloride salts with minimal or no LiCl-KCl present. Initial results were very promising. High levels of chloride salt sorption were measured with most of the salts. Only barium chloride appears to resist sorption into the zeolite. A diffusion-based sorption model has also been derived and compared with salt-zeolite ion exchange kinetics data obtained in a different project. A close fit between experiment and model was observed, indicating that the salt sorption is likely a diffusion-limited process. With experimental systems established for both zone freezing and salt sorption, the second year will include extensive testing to validate and refine the models.

### Benefits to DOE

This project benefits the Department of Energy's energy security mission by supporting the closing of the nuclear fuel cycle, potentially making nuclear energy sustainable with minimal environmental impact. If successful, the project will lead to development of improved spent fuel pyroprocessing technology that generates significantly less waste than current systems. Such a development would be expected to lower processing costs and minimize or even eliminate the need for a long-term repository for spent fuel.

## **Prediction and Monitoring of CO<sub>2</sub> Behavior in Deep Reactive Geologic Formations**

Travis McLing, Robert Podgorney (INL), Jerry Fairley (UI), Kasper van Wijk (BSU) Developing the methodology needed for determining site suitability for basalt CO<sub>2</sub> sequestration CA119

Deep saline aquifers or exhausted oil and gas reservoirs are often touted as the only viable targets for geologic sequestration of anthropogenic CO<sub>2</sub>. This is largely due to their pervasiveness and general proximity to carbon generation sources. However, these targets have significant drawbacks, most notably the generally low potential/slow kinetics for mineralogical trapping, issues and concerns over the need to produce brines from the formations to allow for CO<sub>2</sub> injection, and the potential to either cause real or perceived harm to overlying drinking water aquifers.

Reactive reservoir rocks are also pervasive throughout the world but have received considerably less attention. The most prolific of these reservoirs are comprised of basalts or other mafic rocks. They not only have the capacity for  $CO_2$  storage under hydrodynamic trapping and dissolution processes but also have significant mineralization potential, with a much more reasonable timeframe compared to most traditional brine-hosted formations.

#### Project Summary

A number of tasks are required to collect the data and develop the methodology needed for determining site suitability for basalt CO<sub>2</sub> sequestration. This research is aimed at addressing a number of the highest priority gaps in understanding: (1) site characterization methods; (2) monitoring, measurement, and verification (MMV) approaches; and (3) integration/application of these methodologies in numerical simulation models. Teams from the University of Idaho (UI), Boise State University (BSU), and Idaho National Laboratory (INL)

will explore and further develop methodologies for estimating permeability distributions in highly heterogeneous fractured rocks; focus on developing and collecting the data needed for accurate MMV of  $\rm CO_2$  injections; and concentrate efforts on numerical simulations of  $\rm CO_2$  injections into a number of geologic reservoir types. In addition, INL will also spend considerable effort developing relationships and finding avenues for applying the results of this research to other target mafic rock reservoirs.

## Benefits to DOE

The problems of characterizing fracture distributions in fractured rock domains, obtaining representative data on fracture networks, and identifying fractures in digitally acquired datasets are all relevant to a wide range of scientific and engineering disciplines, including civil engineering, geotechnical engineering, geothermal/petroleum reservoir engineering, remote sensing, structural geology, hydrogeology, environmental geology/engineering, and planetary geology. The work completed to date for this LDRD project constitutes a significant step forward relating to the characterization of fractured rock masses. As such, the nation, the Department of Energy (DOE), and the professionals working in the aforementioned disciplines may all benefit from this research. In addition, the practical applications of this work to carbon sequestration, hydrogeology, and geothermal reservoir management all have economic and intellectual benefits for INL and Idaho, as well as the U.S.A. and DOE.



## **Fabrication of Advanced ODS Alloys using Field Assisted Sintering**

James Cole, Mitchell Meyer Larry Zirker (INL); Indrajit Charit, Darryl Butt, Somayeh Pasebani, Nathan Jerred (U of I); Kerry Allahar, Brian Jaques (BSU)

Using Spark Plasma Sintering to eliminate hot extrusion and not processing for ODS alloys

CA118

The project fosters active collaboration between the Idaho National Laboratory (INL) and two Idaho universities (Boise State University [BSU] and University of Idaho [UI]) through the Center for Advanced Energy Studies (CAES). Oxide dispersion strengthened (ODS) steels have promise as future fuel cladding materials for advanced fast reactors due to their excellent high temperature strength, creep resistance, and radiation damage resistance. They are generally made via high-energy ball milling of constituent powders followed by other complex processing techniques. The purpose of this research has been to use the spark plasma sintering (SPS) process as a step for eliminating hot extrusion and hot pressing as an intermediate processing step. Weldability of such ODS alloys will also be investigated using the pressure resistance welding (PRW) technique.

### **Project Summary**

During the first year of the project, progress has been made in several areas. The required metallic and ceramic powders have been procured from the vendors. BSU is leading the processing of ODS steels from pre-alloyed powders. Currently, spark plasma sintering parameters (sintering temperature, time, and ramp rates) are being optimized on a pre-alloyed powder (Fe-16Cr-3Mo, wt%). Scanning electron microscopy (SEM) study of such samples shows that dense alloys can be produced using SPS. Based on these initial results, an experimental matrix is being created for the pre-alloyed powders by incorporating Y<sub>2</sub>O<sub>3</sub>: sintering temperatures in the range of 625–1100°C, dwell

times from 5–30 minutes, and ramp rates of 50 and 100°C/min. UI is working on developing ODS alloys using elemental constituent powders. SEM and x-ray diffraction (XRD) study have been completed on the initial powders as a quality control measure. Mechanical alloving of elemental powders has been carried out for 5 and 10 hours of high-energy ball milling. Preliminary XRD data show evidence of alloving (allov composition: Fe-14Cr-0.9Ti-0.3Mo-0.25La<sub>2</sub>O<sub>3</sub>, wt%). However, higher time durations (24 and 48 hours) of ball milling are being pursued. SPS will be applied as a processing route to consolidate the powders into the bulk alloys. Furthermore, INL and UI are working on PRW experiments at CAES to demonstrate the feasibility of welding currently available ODS alloys. ODS alloys produced during the project will be welded in future.

## Benefits to DOE

This research work has far reaching scientific and technical implications. The research using the field-activated sintering for ODS steels is a novel one, and INL with its university partners are uniquely positioned to lead this effort. This research is going to develop appropriate processing-structure-property correlations. This research is also facilitating development of state-of-the-art research capabilities and technical expertise at CAES, engaging two Idaho universities, and creating opportunities for graduate students and other research personnel in their educational/training activities.

ional Laboratory

## **Small Specimen Test Techniques for Evaluating Radiation- Induced Changes in Mechanical Properties**

Bulent Sencer

Developing improved small-sample testing techniques to train the next generation of radiation materials scientists

CA120

This project develops improved small-sample testing techniques and uses them to provide an outstanding opportunity to train the next generation of radiation materials scientists. Development of a flexible test station for testing of small specimens in the Center for Advanced Energy Studies (CAES) Materials Laboratory will allow a broader spectrum of university faculty and students to conduct scientific and engineering studies on the physical and mechanical properties of nuclear material without having to use a hot cell.

The project will focus initially on two mechanical property techniques that are exceptionally well suited for testing of very small specimens either in a fume hood or behind light shielding: the horizontal tensile testing and shear punch techniques pioneered at various national laboratories. Although very useful, further development is required to ensure that these techniques can provide reliable engineering data. A test station will be designed to incorporate tensile and shear punch equipment and include provisions to perform other nondestructive (electro-resistive and ultrasonic) test equipment to study void swelling, transmutation, and other radiation-induced property changes of small specimens and staging for examination by electron microscopy before and after testing. Provisions for installation of a commercial hardness tester will also be made. Future additions to the test station could include mechanical testing equipment, the primary candidate technique being the Charpy/Dynamic fracture toughness test.

This proposal team's seasoned national laboratory researchers with university students

and faculty will design and build equipment and conduct experiments. With national lab and consultant guidance, the university faculty and students will design the two test fixtures and test station, incorporating the best design features of previous systems; conduct the necessary feasibility and safety studies; build the shielded facility and the test machines; and validate the improved performance of the equipment. This proposal relies on graduate students to perform the research and provides an excellent opportunity to link CAES research with the ATR National Scientific User Facility.

## Project Summary

The principal investigator purchased the equipment, instron tester, high-temperature grips, furnace, two anvils, Linear Variable Differential Transformer (LVDT) and drop tower. This equipment is now in the CAES Materials Lab; it will be installed and graduate students will be trained in the third week of September. In addition, a micro density measurement cell was manufactured during the summer. This is an addition to the small sample testing techniques.

## Benefits to DOE

This project supports life extension of the current fleet of light water reactors and development of new materials for the next generation of reactors. At this time, it is critical to provide opportunities and training for a new generation of radiation materials scientists to enable continued use of nuclear energy in the U.S. as a clean, reliable energy source.



## **Design and Operation of Two-Stage Manure Anaerobic Digestion Process**

Erin Searcy

Design and study two novel processes for maximizing recovery and conversion of Idaho dairy manure into energy products

CA121

In Idaho, approximately 520,000 activelyproducing dairy cows produce ~3M dry ton of manure annually, creating nutrient management challenges not addressed by existing technology. This research seeks to develop an economically and environmentally viable, sustainable process that can be employed to upcycle dairy manure. Our objective is to design and study two novel processes for maximizing recovery and conversion of Idaho dairy manure into energy products. The first process will maximize conversion of pre-fermented manure to methane (CH<sub>4</sub>), while the second sequesters Anaerobic Digestion (AD) emissions (CO<sub>2</sub>, nitrogen [N], phosphorus [P]) to produce lipid-rich algae that can be recovered and converted to biofuels. A life cycle analysis (LCA) and economic study will be conducted to characterize the system. A 2-stage fermentation and AD test apparatus has been assembled to test system impact on CH<sub>4</sub> production from manure. The supernatant will be separated from the solids, and the effectiveness of nutrient removal using encapsulated algae (which may limit shear stress and increase lipid production) will be studied.

Project Summary

<u>Task 1</u>: Evaluate the 2-stage Anaerobic Digestion Process under Steady State Conditions.

Our preliminary data is highly encouraging for ultimate process success. In brief, the biogas production rate in our two-stage system (AD2) has exceeded the conventional single stage system (AD1). AD1 has recently been producing approximately 22 L of biogas per day, as compared to 31 L from AD2. The biogas in both ADs is approximately 50% CH<sub>4</sub>, 50% CO<sub>2</sub>. We are still collecting and processing data such as total solids, volatile solids, and organic acid concentrations so that we can perform a mass balance analysis on carbon for each system.

<u>Task 2</u>: Syntrophic Interactions involving fungi during AD of Fermented Dairy Manure.

We identified possible polymerase chain reaction (PCR) primers that can be used to specifically amplify anaerobic fungal DNA from dairy manure, and have applied commercial DNA extraction kits to extract DNA from manure. We are presently optimizing the

extraction protocol to produce higher DNA yields with consistent quality. We have evaluated one PCR primer pair on manure DNA extracts and detected PCR products within the expected size range. We are optimizing the PCR conditions and evaluating other possible PCR primer candidates. Based on these results, cow manure may be used as a source of anaerobic fungi, which we can then enrich. The next step is to optimize the process.

<u>Task 3</u>: Produce Biofuels Phototrophically from AD Effluents.

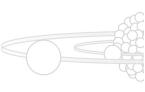
Optimal system requirements appropriate for algal growth using AD effluent and for CO<sub>2</sub> sequestration were compiled from literature. Using these parameters, a 10L photobioreactor was designed and constructed and initial testing started. B. braunii performance in the 10L reactor is being measured and is currently comparable to that observed in smaller scale systems. On-going experiments are monitoring algal growth rates, nutrient removal, and lipid production in small-scale systems fed with differing concentrations of AD effluent provided by the Coats Lab (University of Idaho [UI]). Future work will involve optimization of CO<sub>2</sub> sequestration, nutrient removal, and lipid production in the 10L reactor when fed AD effluent and artificial AD biogas. The ultimate goal is design, operation, and optimization of a system that can be amended directly to an operating AD process.

Task 4: Life Cycle Analysis (LCA).

A preliminary model has been developed for AD of dairy manure using SuperPro Designer. The data for the model available from literature have been added to the components and additional information required has been identified.

Benefits to DOE

The proposed research turns a regional problematic by-product (animal waste) into a resource for energy generation. The project will demonstrate a novel application of technology to enhance energy production from animal waste while concurrently building strategic partnerships between Idaho National Laboratory (INL), Boise State University, Idaho State



University, UI, and agricultural utilities and applicable equipment vendors. The project expands INL's experience in clean energy and LCA, and reduces the public and environmental nuisance caused by the waste. If the 2-stage system proves effective in increasing gas production and nutrient treatment, there would be many possible sources of future funding including the Department of Energy, United States Department of Agriculture, Verdure, and the dairy industry. Our very preliminary data has been highly encouraging.



## **Transmission Site Suitability Decision Support Technology**

Jeffrey Joe (INL); David Solan, Michael Louis, (BSU), Juliet Carlisle, Daniel Ames, Robert Beazer (ISU)

A prototype GIS-based decision support tool that can facilitate electricity transmission line route selections

CA122

New electricity transmission lines must be built in Idaho and across the western region to meet increasing demand. Developers currently submit proposals for transmission line corridors that are least-cost based on technical and geospatial considerations. Initial proposals miss the mark or create severe public opposition due to companies' acknowledged lack of understanding regarding public opinion and participation in siting decisions. The goal of this project is to develop a prototype geographic information system (GIS)-based decision support tool that can be used to facilitate electricity transmission line route selections. It will assess proposed alternatives and/or perform least-cost path analyses to identify feasible and potentially optimal transmission corridors by integrating geospatial data with socio-political constraints. and provide a visualization of the results. The socio-political constraints will be collected through a survey that identifies and measures levels of opposition, and this information will be fed into a newly constructed GIS-based tool. The methodology and the tool will be demonstrated by analyzing potential routes for a future transmission development project.

### Project Summary

Per the approved Fiscal Year 2010 (FY-10) statement of work for this laboratory-directed research and development (LDRD) project, our main goals this year were to develop detailed objectives and questions, and then formulate a detailed research plan and draft questions/items for the survey. In the process of trying to accomplish these goals, the research team had many discussions about what the objectives and research questions should be. Team members

had many different perspectives and ideas. Over time, however, each team member's understanding evolved and the team came to consensus on what some of the key research questions are and how the hypotheses will be tested. The research team has met their FY-10 deliverable goals by producing the draft research plan and draft survey questions for this project. These documents are available for review upon request.

## Benefits to DOE

The strategic value of this research project is that the resulting GIS tool will aid electrical infrastructure siting decisions by providing cost savings to public and private entities, minimizing impacts to the environment, minimizing the risk of project failure due to technical and sociopolitical factors, and strengthening our national security through enhancing our energy infrastructure. The results of the project will also provide insights into needs and prioritization of future siting decisions in other domains.

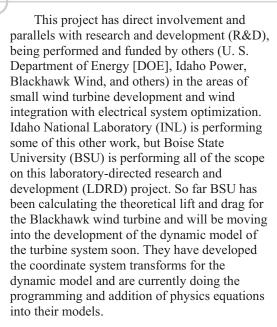
This research has also benefitted the Idaho National Laboratory (INL) and state universities by providing funding to INL staff and university faculty and staff as they collaborate on this research and development project. This research has increased the ability of the four main institutions to collaborate effectively. Furthermore, as an Environmental Policy Institute (EPI) energy-policy-focused LDRD, this research has and will continue to improve EPI and Center for Advanced Energy Studies' eminence as entities that have a considerable amount of subject matter expertise in the area of energy policy.

Idaho National Laboratory

## **CAES Vertical Axis Wind Turbine**

Kurt Myers

Developing the capability for a vertical axis turbine component for use in industry CA123



On the Computational Fluid Dynamics (CFD) research, progress has been good in the short time involved. BSU has already started modifying their CFD code and GPU processing programming to input our wind data and produce some preliminary model results for testing. The code is using our wind data and is producing meaningful results in regards to wind speed and direction for the intermediate coordinates chosen by the research group for modeling. The code will continue to be refined with changes to the physics equations; testing will proceed on the speed of the modeling, and refinement and validation of the results.

## Project Summary

We anticipate that both BSU model development projects will have results by the end of the year that we can compare with the other parallel research and models. The parallel research projects have made significant progress, implementing 14 weather data stations with Idaho Power along one of their transmission corridors, completing the first phases of CFD modeling and integration into the Idaho Power database and transmission models, and working with Blackhawk to get their communication links going and grid integration started for their wind turbine.

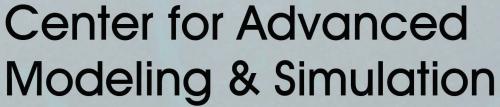
#### Benefits to DOE

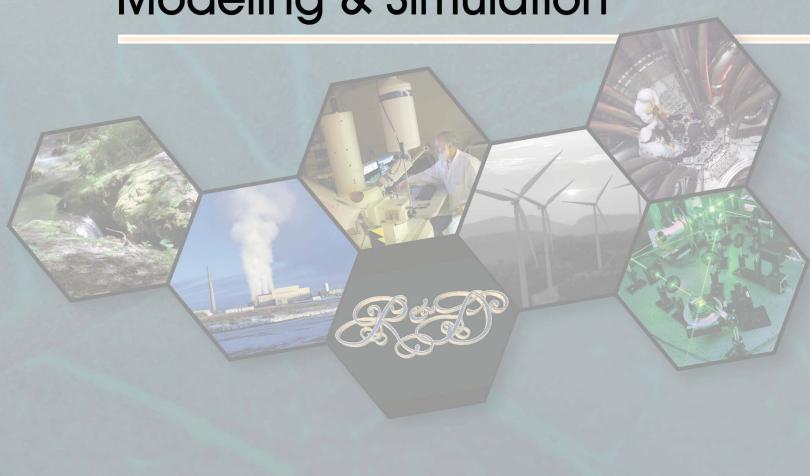
The vertical axis turbine component will ultimately benefit INL, BSU, and DOE in that capability will be developed to support development of new small wind turbines and add to the large scale deployment possibilities of wind energy around the nation. The CFD component is of major interest to utilities, INL, and DOE in that it is supporting research that may allow more dynamic use of existing power transmission systems, which can increase existing capacities and allow time for or deferring the development of new transmission lines. It may also allow for improved use of transmission for wind energy development around the country. All of these developments can help reduce the environmental impacts of power generation and increase the nation's energy security.













## **Study of Preconditioning Techniques for Krylov Solvers Applied to Hybrid Neutron Transport Calculations**

Cristian Rabiti

Study and testing of global preconditioners for the solution of the transport equation discretized by coexisting mixed algorithms

ΓM109

The objective of this work is to develop preconditioners for use in conjunction with Krylov methods to perform neutronics analyses using different angular discretizations in different reactor regions. We refer to calculations in which multiple discretizations are used as hybrid calculations. Examples of different angular discretizations are the even-parity discrete-ordinates equations, the sphericalharmonics equations, and the method of characteristics. Our plan for the first year is to demonstrate effective local and global preconditioners for a mixed discreteordinates/diffusion model in one dimension (1-D). Our plan for the next two years will be to generalize our 1-D approach to 2-D, combining two high-order angular discretizations. Our local preconditioners will be based upon block-Jacobi iteration schemes that are commonly referred to as response matrix methods. Our initial global preconditioner will be based on a diffusion approximation. Very little research has previously been done relating to preconditioned Krylov methods for hybrid calculations. This is the first time an overall global iteration approach will be tested. Even with large improvements in computing power, the challenge of performing a high-fidelity neutronics simulation of a full reactor seems unrealistic for several years to come. On the other hand, strong local heterogeneities arising from control rods, transmutation targets, and reflectors pose challenges with respect to the simulation accuracy that can be optimally met with a combination of different transport discretizations in a single calculation. If successful, this research will enable us to tailor computational discretizations to the needs of high-fidelity simulations and to maximize efficiency.

Texas A&M University (TAMU), under a subcontract funded by this laboratory-directed research and development (LDRD) project, will develop preconditioners for hybrid Sn-diffusion in 1-D and 2-D and for hybrid Sn-Pn in 2-D. The resulting algorithms will be implemented in an Idaho National Laboratory (INL) neutronics code, and the lessons learned in developing these preconditioners will impact the development of more general preconditioners developed within the INL code.

Project Summary

During our first year, we investigated and contrasted several strategies for solving hybrid Sn/diffusion equations in 1-D slab geometry. The slab domain is divided into two contiguous subdomains. In the Sn domain, the Sn approximation is used, and in the diffusion domain, the diffusion approximation is used. The two solutions are coupled at the interface between the two regions via boundary conditions. Note that the boundary conditions are interdependent in that both the Sn interface angular fluxes entering the Sn subdomain and the interface diffusion net current depend upon the same two interface quantities: the interface diffusion scalar flux and the Sn half-range current entering the diffusion subdomain. Given standard Sn and diffusion discretizations and solution techniques, one simple strategy for solving the coupled sets of equations is to independently solve the Sn and diffusion equations in their respective domains while lagging or iterating upon these two interface quantities. The problem with this approach is that fully solving each set of equations can be expensive, and an arbitrarily large number of iterations can be necessary in the limit as scattering dominates absorption in both the Sn and diffusion regions. However, it is important to recognize that one can "wrap" this iteration process with a Krylov method. When this is done, there are only two unknowns in the Krylov system: the interface diffusion scalar flux and the Sn half-range current entering the diffusion subdomain. This means that with an error-minimizing method such as GMRES, only two Krylov iterations (and thus two independent Sn and diffusion solves) are required for convergence regardless of the problem characteristics. Using a code written for this project, we have been able to computationally demonstrate this behavior. The main point is that if there are only a few subdomains in the overall domain with a vector of interface iterates of relatively low dimension relative to the dimensional of the vector of total unknowns in the problem, an iteration strategy based upon independent solutions in each subdomain may be viable and even highly efficient if wrapped in a Krylov method.



An alternative to the above approach is to iterate on both the equations within the subdomains and the interface unknowns simultaneously. This is a more obvious approach that has largely been taken in the past. The problem with this approach is that even when the entire iteration process is "wrapped" in a Krylov method, convergence may be quite slow. Preconditioning is generally required, and this can take two forms. The first is to apply standard preconditioners independently within each subdomain. This is better than no preconditioning at all, but convergence may still be quite slow because these local preconditioners are not "aware" of the coupling between subdomains. A better strategy is to use both local preconditioners and global preconditioners where the global preconditioners are low-cost but indeed aware of the coupling between subdomains. We have developed such a strategy for the coupled Sn/diffusion system based upon local Sn preconditioning using standard sweeps in conjunction with a global diffusion solve in both the Sn and diffusion regions. The Krylov

vector of unknowns consists of all of the Sn scalar fluxes and the interface diffusion scalar flux. In 1-D there is no reason to iterate on the diffusion equation because it is tridiagonal. This will not be the case in multi-dimensions and the Krylov vector will then contain all of the diffusion scalar fluxes, as well.

### Benefits to DOE

The overall goal of this project is to achieve a high-performance scheme for the integration of different algorithms for the solution of the transport equation. Success will eventually enhance accuracy and speed for nuclear reactor simulation. The approach under investigation, if it leads to a positive outcome, will be implemented in the reactor analysis suite of tools under development at INL (PHISICS). It should enhance INL's capability to perform fast and accurate neutronic core simulation. Moreover, this approach pioneers a new philosophy to solve computationally intensive problems that could be used in other applications of interest for the simulation of nuclear reactors.

## Fickian and Thermal Diffusion in Nuclear Materials from Linear Response Theory and Multiscale Simulations

Paul Millet (INL); Jacob Eapen (NCSU/INL)

Developing a theoretical framework and appropriate numerical implementation for thermal diffusion TM110

The central objective of this proposal is to develop new statistical-mechanical models and long-time atomistic simulation methods to determine complex diffusive properties in nuclear materials. Specifically, the proposal will develop state-of-the-art methods to compute Fickian and thermal diffusivities in alloy systems (U-Mo) and UO<sub>2</sub> under different ambient and irradiation conditions.

Project Summary

The theoretical framework and an appropriate numerical implementation for thermal diffusion are being developed. INL has a range of research facilities dedicated to materials testing, analysis, manufacture, and modeling. The laboratory's Advanced Test Reactor and hot cell examination facilities allow for materials irradiation testing, critical tools for accelerated tests to assess fuel behavior and radiation damage. The laboratory also hosts a number of controlled furnaces for precision hightemperature testing of materials. Materials analysis facilities include a wide range of capabilities, including high-luminosity neutron scattering, surface tomography, orientation imaging microscopy, and mechanical testing, enabling materials examination from the atomic to the macroscopic scale. The laboratory can develop materials as well, with deposition, bonding, extrusion, and rolling equipment. Highthroughput computational facilities allow for materials modeling.

Benefits to DOE

The project is embedded in the Materials and Nuclear Fuels Signature program. The Materials and Nuclear Fuel Science Signature features a number of growing research capabilities throughout the laboratory. At the heart of the signature are five areas of focus that together build materials prognostics expertise:

- Synthesis and processing of new and improved materials and nuclear fuels
- Characterizing materials microstructure, from the atomic to the macroscale
- Materials testing in representative environments that match temperature and corrosion conditions
- Real-time online material sensing and performance measurement
- Multi-scale computational models relating structure to properties.

Combined, these developing specialties will enable the laboratory to design, characterize, optimize, predict, and monitor materials performance in demanding environments.

INL hosts a multi-disciplinary team of more than one hundred scientists and engineers that specialize in materials performance research. Areas of expertise span from post-irradiation examination of nuclear fuel to molecular dynamics simulation. The signature serves as a focus for cross-disciplinary collaboration.



## **Error Estimation for Stochastic Uncertainty Quantification**

Michael Pernice

Studying the impacts of errors on uncertainty analysis and characterizing circumstances where these errors significantly affect uncertainty quantification

TM111

Uncertainty quantification (UQ) provides a decision-support framework that helps establish performance margins for complex systems whose behavior is studied via modeling and simulation. UO is generally deployed in a nonintrusive manner. Treating an application code as a black box creates a risk that UQ could be inappropriately applied to unverified software. Numerical errors could contaminate uncertainty analysis, and are often misinterpreted as perturbations to the physics. One common source of error is the use of time integration methods that either simplifies the physics through linearization or treats different physics separately. The objectives of this project are to study the impacts of such errors on uncertainty analysis and to characterize circumstances where these errors significantly affect UO. The purpose is to increase the reliability of UQ when it is used on numerically unreliable application codes.

## Project Summary

This is a late-start project that began in the second half of Fiscal Year 2010. The initial focus has been the composition of verified software for selected reaction-diffusion problems, implementation of several widely used time integration methods, and use of DAKOTA to study how uncertainty analysis is impacted by use of these methods. Time integration methods that were selected include fully implicit backward differentiation methods (embodied in the CVODE software package), semi-implicit methods based on lagging of data and linearization strategies, and operator-split methods that separately treat each physics

component. The fully-implicit methods serve as the "gold standard" against which the other methods are compared. The semi-implicit methods introduce linearization errors that are difficult to characterize; the operator-split methods introduce time splitting errors that lead to reduction of accuracy and possibly stability problems. Initial simulations clearly demonstrate the difference in results produced by the methods. Preliminary uncertainty analysis indicates the potential for misinterpreting solution properties such as boundedness and monotonicity. Finally, capabilities for performing a posteriori error analysis in the second year have been initiated.

### Benefits to DOE

The outcome of this project can benefit a number of DOE programs. The National Nuclear Security Administration labs make extensive use of UO in their efforts to assess the safety, reliability, performance, and effectiveness of the nuclear weapons stockpile in the absence of nuclear testing. The Fuel Cycle Research and Development program in the Office of Nuclear Energy is seeking to develop and qualify new fuel forms that are suitable for fast reactors. Since they are attempting to accomplish this in an accelerated time frame, opportunities for experimentation are limited, and the program will rely heavily on modeling and simulation. UQ is expected to play a critical role in guiding experimental efforts and validating the numerical models. Also in the Office of Nuclear Energy, UQ is an important element of the Nuclear Energy Advanced Modeling and Simulation program.

## **Advanced Visualization using Immersive Environments**

Patrick O'Leary

Designing an immersive interface toolkit that takes advantage of Apple's iPad as an input and feedback device

TM112

Immersive environments have demonstrated to be useful tools to support and enhance scientific and engineering visualization workflows. However, broad adoption of immersive environments in the day-to-day scientific and engineering workflow is limited by the lack of differentiation between desktop environment user interfaces and the immersive environment immersive interfaces; and the limited control, navigation, and interaction offered by currently available input and feedback devices for immersive interfaces. To overcome these problems, we will perform two integrated activities. First, we will expose, in-depth, immersive interfaces for enhanced visualization, exploration, and analysis of both scientific and engineering workflows. Second, we will design an immersive interface toolkit that takes advantage of Apple's newly released iPad as an input and feedback device.

Through this system, it will be possible to change visualization parameters, navigate within the environment, annotate discoveries, and visually analyze presented data. Instead of all user interactions performed as immersive interfaces, we will develop a natural division of work. User interactions predisposed to clicking or typing buttons, sliders, text input, and menus may migrate to Apple's iPad user interface, whereas actions that involve grabbing, touching, moving, probing, or measuring in three-space should be assigned to the immersive interface.

## Project Summary

The proposed activity for this laboratorydirected research and development (LDRD) project has three distinct phases: (1) the preliminary technical analysis and development of a new input and feedback device for immersive environments; (2) the proof-ofprinciple experimental analysis based on separating immersive (three-dimensional) interfaces from user (two-dimensional or desktop) interfaces to demonstrate enhanced usability in immersive environments by utilizing the new input and feedback device; and (3) innovative research and development (new data structures and algorithms) in scientific visualization, utilizing immersive environments on a variety of strategic Idaho National Laboratory (INL) and U. S. Department of

Energy Office of Nuclear Energy, Science, and Technology (DOE-NE) operational and scientific data. Finally, the focus of this work is not on inconsequential advances in computer graphics or computer gaming, but rather on innovative and novel approaches in scientific visualization, utilizing an immersive environment of relevant and important data at INL, DOE-NE, and beyond.

Immersive ParaView software is running on immersive systems in the CAES Advanced Visualization Laboratory. Since Apple's iPad has a completely different Operating System than most of our developed code, we decided to take an intermediate step to jumpstart the process and to demonstrate feasibility. Interaction with the system is via a client HP TouchSmart laptop connected wirelessly to the rendering server. To date, the laptop works with Immersive ParaView on the Minicave, with additional work to be completed on the four-sided and six-sided CAVEs. The tracking for both the four-sided and six-sided CAVEs requires calibration to nonstandard configurations. This is exactly the additional work that was required to get Patrick O'Leary's (INL) and Oliver Kreylos' (University of California Davis) applications running in these facilities using Vrui (a virtual reality user interface toolkit).

A Vrui version of Immersive ParaView has been completed, but not tested. Testing should occur during the month of October. This is important because 99% of our work utilizes the Vrui framework for application development and deployment.

Finally, skeleton client/server architecture has been developed to build a more robust to thin-client devises such as tablets and iPads. The current system allows for turning on and off various features in an immersive environment remotely using a tablet. To fully implement these devices, we need to add a more robust object-oriented abstraction for universal use. Currently, each feature requires a web of if/then/else statements to be parsed. We would like to modify this to be handled by an xml schema, which leaves the implementer to describe the functionality with the schema and produce a callback routine.





## Benefits to DOE

We completed the basic prototyping steps in the short three months of this LDRD project. A large number, given the short period of time, of disseminations and proposals were developed, and the work is being more than well received by our intellectual community, Idaho National Laboratory staff, peer DOE national laboratories, and our (INL's) collaborating university partners.

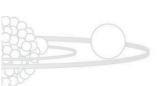
# Distinctive Signatures







# INSTRUMENTATION, CONTROL AND INTELLIGENT SYSTEMS DISTINCTIVE SIGNATURE





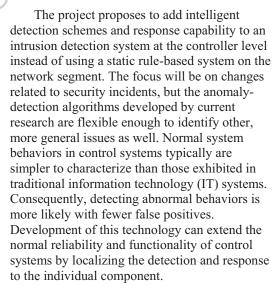


## **Resilient Control System Network Agents**

Denis Vollmer

Intelligent detection schemes and response capability for an intrusion detection system at the controller level

IC106



### Project Summary

Five goals for year three included:

- Refine development of the software prototype implemented in Years 1 and 2 to include machine intelligence algorithms
- Integrate prototype with SNORT IDS or a functional equivalent
- Implement prototype and IDS on vendor/Idaho National Laboratory (INL)-supplied hardware

- Interface with hardware vendor on technical communication issues
- Prepare documentation and summarize results for a conference paper.

## Results and Accomplishments

All of the above goals were accomplished. A working prototype hardware platform (Tofino) utilizing the developed algorithms was successfully implemented. One research paper was published and a second is being developed for publication. Overall the project will have produced six published papers and a working prototype.

### Benefits to DOE

The project produced a cyber-aware sensor for detecting control system network anomalies. The results of this project have been successfully used to support proposals for research efforts in securing our national energy systems. In addition, six published documents at Institute of Electrical and Electronics Engineers conferences have raised the visibility of the Laboratory's expertise in this area.



## **Integrated Control Systems Data Fusion**

Tim McJunkin, Shane Cherry, Miles McQueen, Dr. Milos Manic, Dr. Lee Shunn, Dr. David Gertman

Enhancing manager/operator response and increasing overall facility stability and efficiency IC107

Modern critical infrastructure control and security systems have the capability to provide facility mangers, operators, and security personnel with an abundance of monitoring data. It is critical for facilities to turn that data into information in a timely, actionable time frame. This Laboratory Directed Research and Development (LDRD) project is led by Idaho National Laboratory (INL) principal investigator, Timothy McJunkin, and includes contributions from INL researchers Miles McQueen, Lee Shunn, David Gertman, Ronald Boring, and Jason Wright. The team is also collaborating with University of Idaho (U of I) professor, Milos Manic, and his students, Kevin McCarty and Ondrej Linda.

During the first year of this project, a very simplified process model and threat scenario was created to aid in the development of the initial data fusion conceptual approach. Then, in its second year, these research efforts were built upon by adding complexity to the process as well as adding a more sophisticated cyber-event to further define and test the data fusion capability.

### Project Summary

By bringing in experts in process information, cyber-security, and computational intelligence, this year's project became multidisciplinary. Ultimately, each member of the research team is trying to capsulate a lot of data into a usable form. For example, human factor experts Ronald Boring and David Gertman take the data and try to determine the best way to present that information and test the human performance in the monitoring and control of the system. In addition, the U of I collaborators are focused on the computational intelligence side of

the research project in order to advance data mining tools for strategic optimization and tactically important early accurate detection of anomalies.

In addition, a transient model for a synthetic fuels production facility is being developed to run in MATLAB's Simulink computing environment. The finished model will simulate substitute natural gas (SNG) production using upgraded synthetic gas (syngas) from a coal- or biomass-fired gasification reactor. We will model the potential process impacts due to malware introduced into the simulated process control system and further develop data-fusion capability that will focus on determining "normal" process operating conditions versus those which may be the result of outside tampering, i.e., cyber threat, or random malfunction. Further research into developing data-fusion human-machine interface display capability was initiated.

### Benefits to DOE

This research directly benefits INL through the merging of vast amounts of data from numerous sources and the capability to draw actionable information together, which will be further exploited as the results are extended and published in the coming year. One way the research is currently being used is by collecting synthetic fuel model information, such as temperature, pressure, flow rate, and collecting data on the process. In addition, this type of research—gathering information so timely decisions can be made—could have helped with human error associated with plant systems or the Northeast U.S. blackout of 2003.

Idaho National Laboratory

## **Wireless Sensor Testing**

John Buttles

Design and analysis of full-scale WSN control systems in order to resolve system degradation and instability due to RF interference

Wireless sensor network (WSN) technologies have become a focus of development for instrumentation and control (I&C) applications in many industries. The promise of lower installation cost, while achieving reliable monitoring of process parameters at virtually any location when needed, has driven many existing instrumentation vendors as well as new startup companies to fill a need in wireless technology. These technologies are in their infancy and have not been fully tested for security, reliability, compatibility or cooperation. We hypothesize that radio frequency (RF) and/or cyber interference to a WSN can lead to destabilization of the control system by introducing latency or modifying data, which can undermine the control system design and amplify traditional concerns where impact is limited to the target of the interference. The result is a domino effect, that, while it may have originated as loss of an individual sensor's data or bad data, will lead to changes on a facility level that impact safety and/or production. Distributed control networks and complex network theory, though current topics of research, have not considered how multiple interference regimens can lead to latencies that create destabilization. Therefore, to address this knowledge gap, the proposed research will design and analyze full-scale WSN control systems that can be stressed by multiple interference types in order to develop approaches and solutions to resolve the expected system degradation and instability.

Project Summary

This laboratory-directed research and development (LDRD) project is divided into four tasks to be done over three years:

- Year 1: Define the state of the WSN market and prioritize WSN technology targets
- Year 2: Establish a research test area and set up multiple wireless sensor networks
- Year 3: Perform multiple experiments to measure cyber and RF interference effects on wireless sensor network communication; develop policies, best practices, and a monitoring device equipment specification to mitigate disruption of system data.

This was the second year of my LDRD, which builds on last year's findings that identified which dominant technologies are being used for WSNs. These technologies are 802.11 (WiFi) and 802.15.4 (Zigbee).

This year's main goal was to establish a research test area to perform my research experiments. This area has now been set up in the Center for Advanced Energy Studies (CAES) facility to take advantage of the building's structure, which provides an excellent industrial-like environment for RF signals and also has many existing wireless IT networks. Four WSNs, which are not being used by a different project, are being used for this LDRD. Because each system has its own operating system with different protocols and uses different network interfaces, a considerable amount of time was expended to set them up and make them operational.

Once the WSNs were established, the test infrastructure was set up to be able to communicate with all of the WSNs and monitor the RF signals simultaneously. This required writing custom interface software and installing commercial software, then configuring each one to operate on the test platform. This also required a significant amount of time, and I used several Idaho National Laboratory resources to assist in writing and installing the software. Due to budget constraints, some additional work to complete the test infrastructure remains, which will be done at the start of next year's work.

The work this year has been in line with my original LDRD schedule, and now that I have the research test area operational. I can follow the third-year work plan, which will include testing the effect of different types of interference on multiple WSNs simultaneously operating in a challenging RF environment.

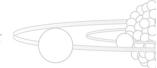
Benefits to DOE

This year, my research was published in the CAES newsletter, the FedTech magazine, and the *Homeland1* online newsletter. Also, many WSN vendors, user groups, university researchers, and standards groups are aware of my research, which will ultimately benefit INL, the Department of Energy, and others.





With the establishment of WSN research expertise, INL is now able to test different wireless sensor systems and have a better awareness of WSNs along with the problems they face when used in critical infrastructure industries. The work under this LDRD will expand our capabilities in this field and become a valuable asset of the National Supervisory Control and Data Acquisition (SCADA) testbed program at INL.



## **Anomaly Detection, Diagnosis, and Resilient Control in Complex Engineered Systems**

Craig Rieger, Humberto Garcia, INL; Vemlat Venkatasubramanian, Purdue University Designing and implementing intelligent, resilient control systems that will be robust to accidental and intentional abnormal events

IC109

One of the most important challenges for control system engineers is designing and implementing intelligent, resilient control systems that will be robust to accidental and intentional abnormal events and assist operators in making supervisory control decisions for such abnormal situations, start up and shut down, and in performing controller assessment and so on. Operators' failure to exercise the appropriate supervisory control decisions in abnormal conditions often have an adverse effect on product quality, process safety, occupational health, and environmental impact. The economic impact of such abnormal situations is enormous—about \$20 billion/year in losses in the petrochemical industries alone in the U.S. The cost is much more when one includes similar situations in other industries such as pharmaceutical, specialty chemicals, power, and desalination. Thus, there exists considerable incentive in developing intelligent, resilient control systems that can provide automated operator assistance for supervisory control in complex process plants.

The objective of this laboratory-directed research and development project is to develop a model-based hybrid framework for anomaly detection, diagnosis, and resilient control in complex process systems. This hybrid framework will integrate signed digraphs-based causal models of the process, dynamic trend models, statistical techniques for state awareness, and model-based supervisory control. Such an approach overcomes the shortcomings of the individual components and is a significant advancement over the state-of-the-art. This framework will be tested and evaluated on both dynamic simulations and experimental testbeds. The lessons from this research can be generalized for other such process systems.

### Project Summary

In Fiscal Year 2009, mathematical frameworks for Principal Component Analysis (PCA), Signed Digraphs (SDG), Qualitative Trend Analysis (QTA) and model-based Kalman filtering (MKF) were implemented. In FY-10, the developments for full Bayesian integration using these methods were continued, and initial tests were further extended on a CSTR reactor system as well as the Machine Condition

Monitoring (MCM) "big loop" system dynamic simulations. In addition, an extensive measurement campaign by means of automated experiments were carried out at Idaho National Laboratory (INL). Sensor bias, actuator faults, valve drift, valve stiction, and noise were simulated to test our framework with successful detection and diagnostic outcomes. A LabView program was written at Purdue to facilitate the testing of the MCM testbed for such faults. For the third year, we plan to further refine the theoretical methods as well as to conduct more experimental campaigns and simulations to develop a reconfigurable control framework for resilient control strategies.

Figure 1 shows the likelihood function evaluated for four faults in the CSTR testbed system, when the correct fault is identified (Fault #4, sensor bias) by the pronounced peak in the likelihood function.

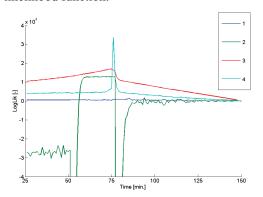


FIGURE 1. Kalman-based likelihoods for four faults (1: valve stiction, 2: valve stuck, 3: valve bias, 4: sensor bias).

To integrate the methods developed, a Bayesian framework is developed to simultaneously use all information that becomes available through the different methods. To this end, each method delivers a likelihood to observe the available data, Y, in each fault scenario in the library of faults, written as: L(Y|M,S), where M stands for the method and S for the evaluated scenario. Then, the likelihood for the data set for a given scenario is based on the integration over the four available methods:  $L(Y|S) = \sum L(Y|S,M)$ . L(S|M). Here, L(M) is the prior for each likelihood, conditional to the method used. Using these priors as weights in the integration scheme, one can account for the





fact that one method or the other is better at identifying certain types of faults.

Figure 2 shows the MCM big loop testbed for simulating a cooling loop for a nuclear reactor system.

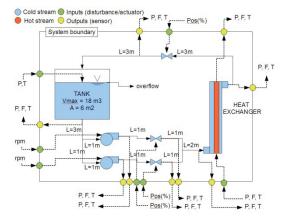


FIGURE 2. MCM big loop.

Our DAE model is sufficiently complex to simulate realistic challenges in fault detection and diagnosis for this testbed. Our first campaign aimed primarily at the identification of the big loop in the MCM test bed was executed in July 2010. During this three-day measurement campaign, online measurements were taken related to the hydraulics of the system. Online measurements included the position of the butterfly valve (BFV), pressures in the system, and the flow rate (FT). The gate valve position was noted down manually. Different faults were also induced to identify the system's behavior qualitatively and quantitatively in such conditions. Induced faults were introduced manually (e.g., leak flow, cavitation) or semiautomatically (BFV bias, drift, stiction and noise, FT bias, drift, stiction, and noise). Faults were introduced while the BFV position was controlled (inner loop in cascade controller). Different scenarios were created by switching the controller for the flow rate on or off (outer loop in cascade controller).

By means of the measurement campaign, an extensive data set has been produced including data from nominal and anomalous operational conditions. The nominal operation data will be used for full characterization of the system. Figure 3 shows a short time segment of the

nominal data in which the valve position was increased and decreased in steps to characterize its steady-state performance. In this experiment, the flow rate was not controlled. An initial result of this effort is the dynamic characterization of the relationship between the BFV and its discrete inputs (open/close digital channels). In Figure 3, the filtered values for the BFV measurement are also shown using the resulting model and the Kalman filter.

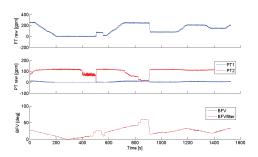
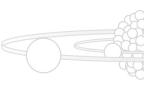


FIGURE 3. Charts for BFV, PT and FT variables.

In the third year, many of the developments will be extended to completion and tested extensively via simulation and in real-life pilot-scale testing. Further theoretical development is necessary in the context of Bayesian integration. This will enable the effective integration of heterogeneous results from possible monitoring and diagnosis modules. Simulated tests for the simulation benchmark tank model and for the cooling loop will be extended to have a proper view on the critical aspects of the methods. Real-life testing will be continued at INL facilities in a follow-up visit in which reconfigurable control will be tested in view of resilient control strategies. A tentative time table is set for November 2010.

## Benefits to DOE

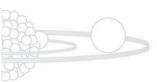
Given the above accomplishments, we are confident that the project is progressing well. The theoretical frameworks, dynamic simulations, and experimental campaigns are advancing the state-of-the-art in developing the next generation of highly automated resilient control systems that can provide greater stability and efficiency while achieving graceful degradation by anticipating failures. Such an advance is required for a wide range of



applications seen in energy, environment, and nuclear industries. The papers that have already come out of this project as well as the simulation environment are beneficial to core Department of Energy and International Conference on Information Systems objectives.

Benefits to DOE

This research provides fundamental tools and applied research for the resilient design of control system networks, and methods to ensure the stability, efficiency, safety and security of control systems and their corresponding applications. In critical applications, such as safety systems for existing and next generation nuclear plant designs (as well as other energy generation systems), concern over the resilience of digital control versus analog has received great attention. It is clear that the control system is paramount in providing not only continued operation, but also the mechanism to achieve efficiency in operation.



## **3D Spatial Representation in Support of Design Inspection and Verification**

Doug Few

Creating a facility information system to address data storage and spatial registration

There are two fundamental difficulties when investigating unfamiliar indoor locations: (1) sifting through facility information documentation in search of domain-relevant information (i.e., security, safety, or nuclear safeguard information); and (2) spatially correlating the relevant information to the facility physical design. The principal investigator (PI) proposes the creation of a facility information system (FIS) to address data storage and spatial registration of facility information. Operators of the FIS will interact with the information system through a three-dimensional (3D) digital replication of the facility.

### Project Summary

3D virtual environments (VE) can provide rich visual cues, but evidence suggests that visual information alone is not sufficient to adequately navigate a VE effectively. To overcome the shortcomings of navigating on visual cues alone, the PI will investigate positioning solutions for accurately tracking human movement in global positioning system (GPS)-denied areas and by using the obtained position information to auto-register the VE perspective to that of the human operator in the physical world.

In addition to investigating GPS-denied positioning, creating a useful system will require novel contributions in the following areas:

- Task analysis of system users roles and responsibilities
- Rapid VE modeling toolsets
- Position correlation from physical to virtual environment
- Human-computer interaction (HCI) research.

Successful deliveries of the proposed work will not only provide facility inspectors with a useful tool, but advances on the sub-component level will enhance Idaho National Laboratory's (INL's) core capabilities.

## Benefits to DOE

This INL laboratory-directed research and development (LDRD) project has benefitted the U. S. Department of Energy's (DOE) national security missions by developing a set of tools that help solve the nuclear non-proliferation DIV problem. During FY-10, researchers:

- Completed the human task analysis of a design information verification (DIV) inspector by soliciting input from a former International Atomic Energy Agency (IAEA) DIV inspector.
- 2. Designed a DIV database to contain information pertinent to the DIV task.
- 3. Updated the virtual model of INL's Fuel Conditioning Facility (FCF) to be spatially accurate.
- 4. Modeled INL's North Boulevard Annex (NBA) facility for a program testbed.
- 5. Coupled the inertial position tracking system to the virtual environment camera perspective.
- Presented and published a conference paper detailing progress to date at the Institute of Nuclear Materials Management (INMM) 51<sup>st</sup> Annual Meeting, July 11-15, 2010.
- 7. Brought on two interns to support the project:
  - Katya LeBlanc, a Human Factors specialist from the University of New Mexico
  - Vanessa Murray, a Chemical Engineering undergrad at Montana State University.

Idaho National Laboratory

## Resilient Condition Assessment Monitoring (ReCAM) System

Humberto E. Garcia, Wen-Chiao Lin, Miles McQueen, Jason Wright, Lee Shunn (INL); Seymon Meerkov, Heng Kuang, Pravallika Vinnakota, Naman Jhamaria (University of Michigan)

Implementing condition assessment monitoring systems that are both resilient and adaptive IC111

The major goals of this Idaho National Laboratory (INL)-led laboratory-directed research and development (LDRD) project were to:

- Develop an innovative architecture and its constituents for implementing condition assessment monitoring systems that are both resilient and adaptive
- Develop the notions of information quality (IQ) and assessment quality (AQ) that can be utilized on-line to reconfigure monitoring systems as needed based on assessed conditions of the monitored process
- Develop and demonstrate methodologies for the effective deployment of resilient monitoring systems that can accommodate natural and malicious perturbations to both the monitored process and its data generation and acquisition systems
- Develop and demonstrate resilient
  monitoring approaches that drive data needs
  according to possible time-varying AQ/IQ
  requirements, leading to the autonomous
  selection of sensors and a monitoring system
  that marshals data as AQ requirements
  change and that gracefully degrades
  (as opposed to collapsing) under
  perturbations, even under severe sensory
  loss.

### Project Summary

In summary, during FY-10, the researchers on this project:

- Developed overall functional and mathematical architectures of ReCAM.
- Defined and implemented the structure and information topology of the overall demonstration environment.
- Constructed code infrastructure for ReCAM.
- Developed an interpretation of data quality associated with sensor measurements to reconstruct signal probability distributions.
- Integrated data quality interpretation into belief network (BN)-based condition assessment using virtual evidence methods.

- Designed (local & global) rational controllers to achieve assessment entropy minimization and tracking.
- Developed software to implement components of a ReCAM prototype:
  - Established interconnections between Simulink, Matlab, and Netica
  - Established interconnections among ReCAM, sensors, disturbances, and monitored system
  - Developed software for plant model, sensor model, controller, and BN-based assessment.
- Demonstrated ReCAM within simplified models of monitored plants and obtained preliminary results.
- Developed computer infrastructure to accommodate modeling of threats.
- Developed operating prototypes for the Simplified Power Plant (PP) model (*in-progress*), the Hybrid Energy System (HES)-0 model (*in-progress*), and the HES-I model (*in-progress*).
- Completed a contract with the University of Michigan to accommodate a three-year contract for three graduate students.

## Benefit to DOE

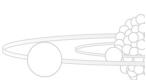
This research has already provided many benefits, such as the mathematical architecture and methods that were developed to introduce a unique approach for implementing monitoring systems that are both resilient and adaptive. Likewise, the developed simulations provide demonstrations of plant operations and on-line resilient condition assessments that are easily understood. INL has further enhanced its technical expertise in the development and demonstration of techniques for resilient process monitoring and plant condition assessment. As a result, INL should retain the top position for this particular area of resilient systems within research and development (R&D) institutions. This project also supported technical collaborations with top R&D institutions, particularly with the University of Michigan.



Future directions include the following tasks:

- Develop increasingly complex plant models and ReCAM-based algorithms for evaluating resilient monitoring performance
- Investigate alternatives for processing sensor data and sensor quality information
- Develop notions and metrics associated with resiliency and vulnerability

- Develop rational controllers that increase resiliency and decrease vulnerability
- Complete software development of ReCAM prototypes:
  - Accommodate increasingly complex plants and threat models
  - Implement developed rational controller algorithms
  - Implement sensor data processing algorithms.



## **Automated Differential Equation-Based Identification**

Lee Shunn

Developing general techniques for nonlinear system identification and automatic control IC112

System identification (sys-ID) relates to building mathematical models of dynamical systems based on observed input-output data. For the case of linear models, there are wellstructured theories, methodologies, and algorithms to construct the relationships. For complex nonlinear models, however, general techniques are not as established or robust. The main objective of this project is to develop and demonstrate a new class of algorithms for nonlinear system identification. The new technique is based on Perona et al.'s trajectory method (Nonlinear Dynamics, 23:13-33, 2000) for nonlinear model reconstruction. The objectives of the current project are to: (1) extend Perona's method to high-dimensional systems with forced dynamics, (2) demonstrate the ability of the method to reconstruct nonlinear systems in a variety of numerical and experimental examples, and (3) apply the method to problems of interest at Idaho National Laboratory (INL) including energy systems and electric vehicles.

#### Project Summary

Early in the year a MATLAB implementation of the sys-ID method was applied to data from simple partial-differential equations (PDEs). The MATLAB code was limited to a three-dimensional state space and could only be applied to unforced dynamical systems. Despite these limitations, the algorithm successfully estimated the values of the parameters in the PDE example problems under a variety of conditions.

Work was begun to write a new code that could be applied to higher-dimensional systems with external forcing. The only restriction on the dimension of the state space in the new code is the physical limits of the computer system. Work to include forcing terms in the code has begun and will likely be completed early next year. Additional limits of the current technique have motivated an exploration of genetic algorithms to discover the "best" basis functions for a given analysis.

Recognizing the value of "real world" demonstrations, construction on several validation experiments commenced. Several small-scale experiments have been constructed as test beds to validate the sys-ID method, including an inverted pendulum and submarine thrusters. If the current validation problems show promising results, work on more complicated systems will be started.

## Benefit to DOE

There are many engineering systems in which the full governing equations are not well understood, or in which parameter uncertainty limits their utility. Modeling such systems from first principles can be costly due to the expert knowledge and tuning required to produce satisfactory results. Automated methods that "reconstruct" the governing equations from observed input-output data can be a costeffective approach provided that the resulting models perform adequately. Many existing sys-ID techniques use linear approximations to represent the system or produce complicated expressions that fail to provide insight into the physics of the process. The current work attempts to directly reconstruct the nonlinear differential equations in the time domain, providing a state-space representation useful for standard control techniques and informative to analysts. If successful, this project will provide valuable contributions to applied system identification as well as dynamical systems theory and chaos. In addition, this work will enable the deployment of highly adaptable systems for prognostics and control, benefiting a variety of Department of Energy and Department of Defense application areas. Developments will be communicated through peer-reviewed journal articles and conference presentations, enhancing Idaho National Laboratory's technical standing in the instrumentation and controls community.

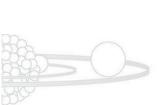






204

## MATERIALS FOR HARSH ENVIRONMENTS SIGNATURE





# **Development of a Small Sample Volume Mechanical Properties Testing Technique for Irradiated Fuels and Materials**

James I. Cole, Dennis D. Keiser, (INL); Dave Bahr (Washington State University) A mechanical testing system and methodology to rapidly assess the strength, stiffness, and, in some cases, toughness of materials after irradiation

AF105

The objective of this project is to design, fabricate, and test an instrument that will enable micromechanical testing through instrumented indentation in a footprint of a device that could fit into a glovebox enclosure and be used to assess strength, hardening, and fracture behavior of materials after exposure to irradiation. The end result will be a system delivered to Idaho National Laboratory (INL) from Washington State University (WSU) that can be used on site to provide relatively quick and low-cost screening of mechanical properties in irradiated materials.

### Project Summary

The instrumented indentation system has been constructed and calibrated. Control software for the system has been written, and the initial testing and refinement of system performance has been conducted.

Three materials that have typical ranges of hardness and strain hardening behavior were selected as simple test samples: steel, brass and aluminum. Indentations and tensile tests were performed on the materials (Fig. 1). From the equivalent indentation strain, and using the Tabor relationship between hardness and flow strength, indentation stress-strain data has also been generated.

### Benefits to DOE

Initial testing proves the system is working, the methodology for developing a quick and low-cost screening instrument for assessing strength and hardening is valid, and the system is reproducible.

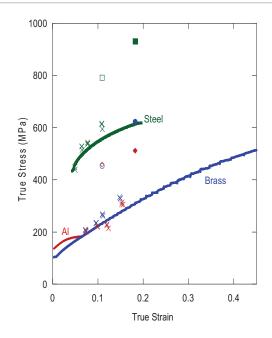
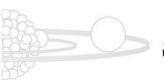


FIGURE 1. Stress-strain behavior for three representative materials (brass, steel, and aluminum). Solid lines are tensile tests, X's are from spherical indentation, and solid symbols from the 90°tip and open symbols from the 120°tip.

The work benefits Department of Energy (DOE) interests in terms of establishing new capabilities and expertise that enhance the laboratory's ability to conduct post-irradiation examination of a wide range of nuclear fuels and materials in support of both industry and DOE customers. Understanding fuels and materials changes under irradiation will be critical for current reactor technology sustainability and the development of advanced energy systems such as high-temperature gas and sodium fast reactors.



# Irradiation-Induced Evolution of Defects and Microstructures in Nanocrystalline BCC Mo

Paul Millet

Investigating the influence of irradiation-induced defects and defect clusters on the mechanical behavior of body-centered-cubic metals

AF106

The research objective of this laboratorydirected research and development (LDRD) project is to investigate the influence of irradiation-induced defects and defect clusters on the mechanical behavior of body-centered-cubic (bcc) metals using atomistic simulation techniques tightly coordinated with experimental characterization and validation. The material of interest is nanostructured molybdenum, which was chosen due to its bcc crystal structure at all temperatures and non-magnetic behavior. The project initiated at the beginning of Fiscal Year 2010, with a projected budget for three years. The vast majority of the budget (\$273k for 2010) goes toward supporting two postdoctoral associates (one at Idaho National Laboratory [INL] and one at North Carolina State University [NCSU]) to carry out the computational and experimental research. The computational approach involves molecular-dynamics simulations, coupled with advanced visualization techniques, to reveal the relevant atomic mechanisms of such interactions in carefully selected model microstructures. Concurrent experiments involving high-resolution electron microscopy of well-characterized nanocrystalline specimens will be performed for validation and further insight.

### Project Summary

We have made significant progress in this research during the last fiscal year, and we have certainly achieved this year's deliverables. For the computational investigation, the deliverable

as stated in the proposal is: "Initiation and development of atomistic simulations of plastic deformation behavior of unirradiated nc Mo. Closely compare observed mechanisms with experiments." Dr. Zhang (INL postdoc) and Dr. Millett (INL) have performed an extensive amount of molecular-dynamics simulations of deformation in bcc molybdenum columnar nanocrystalline structures. Various parameters have been systematically varied including the grain size, temperature, and strain rate. Comparatively little is known about the deformation processes in bcc metals relative to face-centered cubic metals, and these computational results have provided great insight. Most striking in our observations has been the predominance of deformation twinning within the nanoscale grains (Fig. 1). We are currently preparing a journal publication detailing the results.

In the experimental investigations, the deliverable as stated in the proposal is: "Process nc Mo samples and perform HREM study on the nc Mo." Dr. Cheng (NCSU postdoc) and Mr. Jian (NCSU Ph.D. candidate) have prepared samples of nanostructured Mo using high-pressure torsion (HPT) under different pressure (1, 2, and 4 GPa). Dr. Cheng has characterized the microstructure by TEM and HREM. The work has focused on the distribution of nanograins, the grain and subgrain boundaries, and the different defects including dislocations (type and interaction) and stacking faults (Fig. 2).

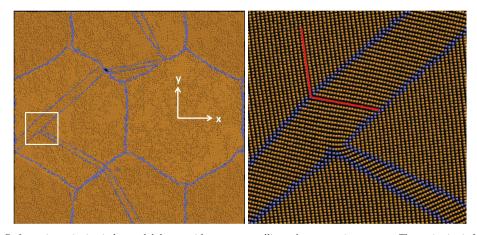
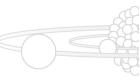


FIGURE 1. Deformation twinning in bcc molybdenum with a nanocrystalline columnar grain structure. The grain size is 36 nm and the temperature is 300 K. The sample has been strained in the y-direction by 3.6%.



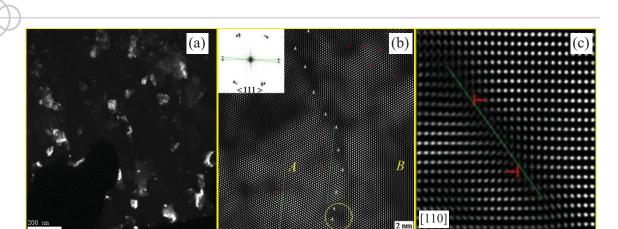


FIGURE 2. TEM and HREM investigations of nc Mo prepared by HPT. (a) Distribution of nanograins in the HPT sample with a pressure of 4 GP; (b) HREM investigation of a subgrain boundary with a misorientation angle of 5.6 degrees; (c) a partial dislocation induced by an interaction of screw dislocations.

### Benefits to DOE

This research project aims to fill in a knowledge gap pertaining to the underlying deformation mechanisms associated with bcc metals in nuclear reactor conditions. Bcc metals, such as ferritic steels and alloys, are used extensively as structural materials in reactor cores. We are developing an atomistic-based knowledge of their deformation behavior for varying conditions: with and without irradiation

damage, and with and without helium gas. This research is breaking new ground in this respect and will better enable other scientists and engineers to predict crack initiation and propagation, ductile-to-brittle transitions, and how irradiation and inert gases may accelerate these failure processes. Such a capability will improve the safety and performance of existing nuclear power plants, as well as allow improved material designs for future plants.



### Microstructural Evolution of UO2 and U under Irradiation

Maria Ann Okuniewski

Using PAS and TEM to elucidate the microstructural evolution in irradiated  $UO_2$  and U AF107

The objective of this research is to elucidate the microstructural evolution in irradiated UO<sub>2</sub> and U with positron annihilation spectroscopy (PAS) and transmission electron microscopy (TEM). The results from these tests will be used to understand the microstructural evolution in UO2 and U as a function of various irradiation parameters, such as dose, type of irradiation particle, and temperature. PAS and TEM will be utilized to analyze defects, determine defect energies, and diffusion lengths in UO<sub>2</sub> and U. Since PAS is relatively insensitive to defects greater than 1 nm, TEM will be utilized to qualify and quantify defects greater than 1 nm. UO<sub>2</sub> is of particular interest since it is a model oxide fuel for nuclear reactors (e.g., light water and fast reactors), while U is a model metallic fuel for fast reactors. The significance of this research is that it will provide unique data, lead to a better understanding of the impact of microstructure on the thermal and mechanical properties of fuels, and provide crucial input and validation data for multi-scale, multi-physics models. More specifically, this research will help to support the efforts of the Energy Frontier Research Center (Center for Materials Science of Nuclear Fuel) at Idaho National Laboratory, as well as modeling efforts being undertaken by Nuclear Energy Advanced Modeling and Simulation (NEAMS) program.



This first year of this LDRD has focused on three major areas: (1) acquiring and fabricating specimens, (2) accelerator operations, and (3) designing of a positron annihilation spectroscopy (PAS) system to analyze irradiated samples. Polycrystalline metallic alloy specimens (pure U, U-Zr and U-Mo) have been both acquired and fabricated, which will be utilized for the positron annihilation spectroscopy and transmission electron microscopy. A single crystal UO<sub>2</sub> fabrication system was designed by a collaborator and will be tested in FY-11 to fabricate specimens for this research. The accelerator that is located at the university collaborator's lab was upgraded to allow for implantation ions, current, and temperatures of interest to this project. A system was designed to utilize PAS to examine specimens that have been irradiated. The components of this system were received in FY-10.

#### Benefits to DOE

This research has benefited DOE's national security mission of science through the design of a unique positron annihilation system that has been specifically designed to analyze radioactive materials.

### Irradiation Testing and Molecular Modeling of Irradiation-Assisted Diffusion and Microstructural Evolution (FCCI)

Bulent Sencer

Measuring thermal- and radiation-enhanced activation energies
AF108

Irradiation of nuclear fuel in reactors results in fuel swelling, and production and transport of fission products to the cladding. The contact between fuel matrix fission products and fuel cladding will promote chemical and mechanical interactions with the cladding. The chemical compatibility between the fuel and cladding is imperative for safe operation of a reactor, and it is essential to limit the interdiffusion between the fuel and cladding. This study will investigate, with the use of ion irradiation created in an accelerator, the effects different irradiation types might have on fuel-cladding interactions with prototypic material systems that have been traditionally difficult to study. Modeling efforts will be supported with this data to demonstrate the combination of testing simulated systems and modeling. The proposed study will contribute to a better understanding of fuel cladding interaction by investigating model systems coupling with the fuel materials. Fuel cladding diffusion experiments will be conducted at temperatures of up to 600°C under proton, helium, and other heavy ion irradiation, if appropriate. The various diffusion couples will be analyzed by imaging, and phase analytical and chemical methods. We will use electron microscopy and other tools to investigate the interdiffusion kinetics, phase formation at the clad-fuel interface, diffusion coefficients, and the clad-fuel microstructural development.

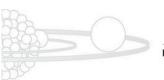
The main purpose of the research is to measure activation energies, thermal- and radiation-enhanced. We have about 40 diffusion couples (single and binary) for a single temperature. We will continue to prepare diffusion couples for thermal activation energies, followed by irradiation, to see if there is any enhancement in the interface.

Project Summary

University of Nevada, Las Vegas (UNLV) and Georgia Institute of Technology (GaTech) are the collaborators for this project. Contracts were set up with the universities. Needed materials were cast or bought by Idaho National Laboratory (INL) and shipped to the universities. UNLV employed two graduate students and GeTech employed one graduate student. UNLV started preparing diffusion couples for characterization: pure Fe, Mo, Zr, Ce, and Nd; alloys of Ce-50Nd, Fe-15Cr, and Fe-15Cr-15Ni; and diffusion couples (bonded) of Fe versus Zr and Mo versus Zr. These materials are being irradiated with protons to 5 dpa. GaTech is using models (current and new) for verification and validation.

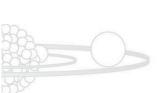
#### Benefits to DOE

Interdiffusion phenomena are very important to understanding the useful lifetime of nuclear reactor components and fuels. One of the goals of the Materials and Nuclear Fuels Science Signature activity is to determine measurable materials' changes associated with degradation in service that are indicative of incipient failure. For example, fast reactor fuels are often limited in operating characteristics or lifetime by interaction of the fuel or fission products with the fuel cladding materials. Detailed models for some of these effects, such as the fuel-cladding interactions, are just now being considered, and data is needed to test the models (verification and validation). It is also difficult and costly to construct in-reactor experiments to study these effects.





## SEPARATIONS & ACTINIDE SCIENCE SIGNATURE





# **Evaluation of Covalent Interactions in Actinide Coordination Compounds**

Dean R. Peterman

Studying the coordination and separations chemistry of INL dithiophosphinic acids compounds AS106

Complexing agents containing soft-base (e.g., N or S) donors, such as dithiophosphinic acids, have significant potential for the efficient separation of trivalent actinides, An(III), from lanthanides, Ln(III). A family of dithiophosphinic acids (DPAH) developed at Idaho National Laboratory (INL) has shown unprecedented selectivity for americium and curium over lanthanides in acidic solution (Fig. 1).

$$F_3C$$
 $CF_3$ 
 $F_3C$ 
 $CF_3$ 
 $F_3C$ 

FIGURE 1 Structures of bis-(3,5-bis(trifluoromethyl) phenyl)dithiophosphinic acid (1) and bis(o-trifluoromethylphenyl) dithiophosphinic acid (2).

#### Project Summary

We propose to study the coordination and separations chemistry of the INL DPAH compounds with U(III), Np(III), Pu(III), Am(III), and Cm(III). UV-vis, infrared, and Raman spectroscopies will be used to quantify the extent of covalency in the An(III)-DPAH coordination compounds. In addition, the thermodynamic features of the bonding and phase transfer reactions such as metal complex formation constants, phase partitioning equilibrium constants, and associated reaction enthalpies and entropies— $\Delta H^{o}$  and  $\Delta S^{o}$ —will be determined. An interesting paradox discovered for these ligands is that effective extraction only occurs in the polar diluents whereas no extraction or separation occurs in toluene and more traditional non-polar diluents.

Fiscal Year 2010 (FY-10) experiments were carried out using multinuclear nuclear magnetic resonance (NMR) techniques to understand the aggregation of the INL dithiophosphinic acids in various organic solvents. An understanding of the importance of aggregation in these ligand

systems is necessary in order to explain the selectivity of the solvent extraction system. Acid dissociation constants for a number of the dithiophosphinic acids have been determined. The acid dissociation constant can be associated to the Lewis basicity of the dithiophosphinic acid, which will directly impact aggregation, extraction mechanism, and selectivity.

Continuing collaboration with Prof. Dale Russell at Boise State University in FY-10 led to performing experiments with macroscopic amounts of americium and plutonium in the FY-09 installed inert atmosphere glove box. The experiments are part of developing the methodologies necessary for the electrochemical production of trivalent plutonium and uranium in aqueous solutions and organic media. These experiments were part of the developing methodology for the production of trivalent plutonium and uranium. These will be used along with trivalent americium to spectroscopically compare the dithiophosphinic acid complexes and the role of covalency in binding trivalent actinides.

### Benefits to DOE

By investigating potentially superior technology for the treatment of spent fuel using potentially superior extractants, this project:

- Addresses the need for improved nuclear waste clean-up processes to mitigate the risks and hazards posed by the legacy of nuclear weapons production and research
- Supports United States leadership in science and technology and promotes international nuclear safety and nonproliferation
- Furthers the research goals of the Office of Science by investigating potentially superior extractants in the processing of actinide containing streams
- 4. Advances the mission of the Advanced Fuel Cycle Initiative (AFCI) to close the nuclear fuel cycle.



# Rapid Detection of Plutonium, Neptunium and Technetium in Water Samples

Sarah Pepper

Design of a novel separations method based on cloud point extraction to facilitate the rapid separation and detection of radionuclides

AS105

The detection of anthropogenic radionuclides in the environment, such as plutonium and technetium, is essential for verifying whether nuclear materials and activities are non-clandestine. Due to their low concentrations, pre-concentration techniques must be employed prior to detection. Solvent extraction techniques are commonly used, but there is a move to replace them due to the hazards associated with the organic solvents used. Therefore, the objective of this project was to design a novel separations method based on cloud point extraction (CPE) to facilitate the rapid separation and detection of radionuclides in aqueous samples by coupling CPE to existing detection methods, such as radiometric or spectrometric detection methods. CPE utilizes water as the predominant solvent along with small amounts of inexpensive surfactants, such as Triton X-114. Trivalent lanthanides were studied in the presence of the ligands 8hydroxyquinoline (8-HQ) and ammonium pyrrolodine dithiocarbamate (APDC), which were chosen due to their extensive use for the separation of other trivalent metals via CPE. In addition, the separation of americium was investigated.

#### Project Summary

Literature on CPE implies that it would be a relatively simple technique to perform. However, experience has shown that the technique is neither simple nor straightforward. Much of the project has been spent trying to determine the optimum experimental conditions to achieve a reproducible separation of europium. With the ligand APDC, a pH study was performed to determine the optimum working pH, a pH of 5. Experiments were also performed to assess the effect of using buffers to control the pH, as was done in previous studies, and allowing the system to equilibrate prior to heating. It was determined that using buffers or equilibrating the system prior to heating led to a decrease in extraction efficiency, which was attributed to the degradation of the ligand. Degradation of APDC

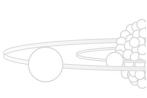
and its impact on CPE has not been previously reported, although it appears well known outside CPE. With the ligand 8-HQ, initial experiments focused on repeating a previous study looking at trivalent lanthanides. While this was successful, a precipitate was formed in all cases. Decreasing the ligand concentration removed the problem and similar experiments as with APDC were performed. The optimum solution pH was determined to be 7. The inclusion of buffers to control the pH led to a decrease in extraction, although not because of degradation. It was also determined that the process of extraction was slow, with maximum extraction occurring after 24 hours equilibration prior to heating. Additionally, using APDC, americium was separated from europium, and results indicated that the method was selective for americium over europium.

Overall, results suggest that APDC is not a suitable ligand due to its degradation, which leads to a complicated system that requires further study. 8-HQ is not suitable for the development of a rapid technique.

### Benefits to DOE

In assessing the effectiveness of the DOE's continuing mission to clean-up contaminated sites and disposing of nuclear waste, environmental monitoring is crucial. This research directly supports this mission by designing a detection method which would be capable of detecting low levels of radionuclides in water samples.

CPE was investigated as an alternative to classic solvent extraction techniques for the rapid pre-concentration and detection of anthropogenic radionuclides. However, it was determined that CPE is not currently a suitable replacement and demonstrates that a significant body of work needs to be performed to thoroughly understand the various experimental parameters that control the extraction-separation. Once the process is understood at a more fundamental level, CPE may turn out to be a viable alternative.



### Measuring Actinide Speciation in High pH Solutions

Gary S. Groenewold, Anthony D. Appelhans, Garold L. Gresham New approaches for measuring metal chemistry in concentrated solutions
AS107

Metal-ligand coordination complexes are chemical species that control transport and fate of the metals in environmental and industrial processes. While measurement of the metals is fairly straightforward, there is an on-going need for measuring chemical speciation of metals and organic ligands in solutions that contain both species. Chemical speciation refers to the array of chemical forms that an element can exist in and includes metal-ligand coordination complexes, various oxidation states, adsorbate species, and precipitates or particulates. In a natural or industrial environment, metals will normally be present as multiple species at the same time, a fact that complicates measurement. Speciation measurements become more challenging under aggressive chemical conditions, such as high ionic strength (I) or extreme pH values. These conditions, together with high radiation fields, can modify organic ligands used in industrial separations, which further diversifies the metal-ligand speciation as well as the distribution behavior.

The conventional approach for measuring metal-ligand speciation is UV-visible spectroscopy (UV-vis): the wavelength and intensity of absorption maxima can be correlated with the identity and quantity of given metalligand coordination complexes. UV-visible spectroscopy measurements are relatively easy to make; however, a drawback to this approach is that the absorption bands are broad, and so the specificity and dynamic range tend to be limited. Alternative vibrational spectroscopies, such as infrared or Raman spectroscopies, have great utility in selected media, but in general are limited by the background in solutions that are of highest interest. A more powerful technique for measuring speciation is X-ray absorption spectroscopy (XAS), which can exactly determine atomic positions within coordination complexes. However, XAS has difficulty differentiating multiple species present in the same sample, which makes data interpretation difficult, and it requires synchrotron radiation, making analysis of many types of samples difficult or impossible.

Electrospray ionization mass spectrometry (ESI-MS) has emerged as a powerful tool for measuring speciation in complex mixtures. In the

analysis of low-concentration solutions, ESI-MS data can be correlated with metal speciation, providing an effective means for measuring phenomena like hydrolysis, condensation, and coordination reactions: these are common processes that complicate metal speciation in solution. A current limitation is that application of ESI-MS to highly concentrated solutions has not been demonstrated. This would seem to be a relatively trivial extension, but most if not all techniques struggle to generate meaningful speciation data from matrices that have a high "chemical background." One of the hypotheses of this project was that ESI-MS would be capable of generating accurate metal speciation information from solutions having high ionic strength or extreme pH values.

A second potentially significant limitation of ESI-MS is that it does not tolerate solutions that significantly modify the properties of the spray solution. ESI functions by spraying an analyte solution through a high potential (e.g., 4 kV) capillary, forming charged droplets that evaporate, producing gaseous ionic solutes that are transferred via a skimmer cone and capillary to the high vacuum of a mass spectrometer (Fig. 1). Solutions that are highly concentrated can impede ion formation by interfering with the ability of the droplets to accommodate surface charge and to evaporate. In addition, concentrated solutions can corrode or plug the capillary. An appealing approach for circumventing these problems is to use desorption electrospray-MS, or DESI-MS.



FIGURE 1. Schematic diagram of ESI-MS.

DESI-MS functions by bombarding the analytical sample with electrosprayed droplets, which first wet the sample (if dry to start out with), and then "splash" analyte-containing droplets into the gas phase (Fig. 2, next page). Droplet evaporation completes the transit of the analyte ions into the gas phase, as in the case of conventional ESI-MS. Because the analyte

solution is not driven through the electrospray capillary, difficulty with plugging, deteriorated spray behavior, and modified speciation chemistry may be avoided.

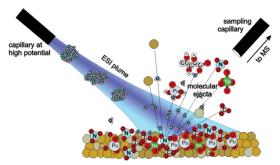


FIGURE 2. Schematic diagram of DESI-MS.

DESI-MS has been used principally for analysis of organic compounds and thus is expected to function well for characterization of organic ligands that form coordination complexes with metals. However DESI-MS has not been demonstrated for metal complexes, and a further uncertainty is its ability to measure trace levels of degradation products in concentrated ligand mixtures. The second hypothesis of this project was that DESI-MS would be able to effectively measure both metal coordination complexes and ligand degradation products.

#### Project Summary

The experimental plan for testing these hypotheses has involved two overarching tasks:

- Modification of an existing ESI-MS instrument to enable function in a DESI-MS mode
- Speciation measurement of selected metalligand systems, emphasizing identification of both coordination complexes and ligand-degradation products.

The project was initiated in Fiscal Year 2009 (FY-09), during which time the initial instrument modifications and speciation studies were performed. More in-depth, rigorous speciation studies were conducted in FY-10, with minor changes to the instrument modification.

I. <u>DESI Modification</u>. The electrospray ionization mass spectrometry was modified in FY-09 to conduct what is referred to as transmission mode DESI (TM-DESI) experiments. This involved orienting the spray capillary coaxial with the skimmer cone aperture and placing a mesh screen in between (Figs. 3 and 4). The screen functions to hold the analyte solution and the electrospray droplets are fired directly through it toward the sampling cone aperture. This approach simplifies geometric

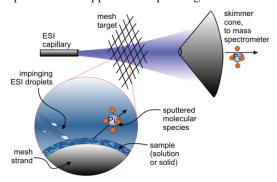


FIGURE 3. Schematic diagram of the TM-DESI process: droplets impacting and recoiling from a screen target.

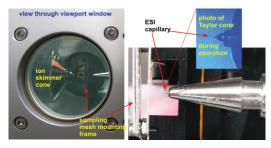
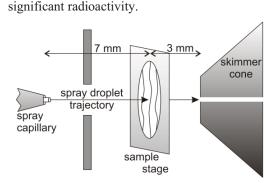


FIGURE 4. Photographs of the modified source region of the ESI instrument. Droplets are formed from a Taylor cone at the capillary tip during ESI.

orientation problems common to standard DESI-MS experiments. The instrument in this configuration was used to conduct characterizations of lanthanide and Group II nitrates, alkali carbonates, and alkylammonium salts. The drawback discovered for this approach was that the mesh screen would plug after a relatively small volume of electrospray fluid was delivered, which caused large variations in the number of ions entering the sampling cone. Beginning in FY-10, alternative experimental arrangements were sought to improve the stability of the MS ion signal.

The initial modification to the TM-DESI was to replace the mesh screen target with a single strand of a polymeric material, reasoning that at a microscopic scale, the basic interaction involved a droplet with a single fiber strand, as shown in the inset in Figure 3. This approach is depicted in Figure 5, and was accomplished by securing two or three fibers in the sample frame originally used to hold the mesh in place. The single fiber was loaded with a sample solution either by dipping it in solution or applying a microliter using a micropipette. Analysis showed facile production of alkali-crown ether complexes identical to those produced from the screen, only a much smaller analyte quantity was needed. This suggested that a nanoliter volume may be sufficient for generating a high-quality



mass spectrum, a finding that might have good

applicability for analysis of solutions having

FIGURE 5. Schematic diagram of the DESI source configured with a single-strand target.

The second modification involved installation of a capillary target between the ESI capillary and the skimmer cone (Fig. 6). The purpose of this experiment was to eliminate the need to first wet the sample and provide a constant flow of analyte solution, with the intent to stabilize the mass spectrometric signal. These expectations were realized in experiments involving metal-trialkyl phosphate, and crown-ether coordination complexes.

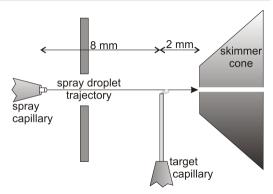


FIGURE 6. Schematic diagram of the DESI source configured with a target capillary.

**Speciation Measurements.** Initial testing of the TM-DESI instrument in FY-09 demonstrated production of coordination complexes, quaternary alkylammonium salts, and clusters composed of carbonate and ethylene diamine tetraacetic acid (EDTA). Results of lanthanum nitrate experiments are representative: singly and doubly charged ion pair complexes containing La<sup>3+</sup> and NO<sub>3</sub> were formed using the mesh screen target doped with dilute solutions of the salt. Equally encouraging were production of cation cluster ions from sodium bicarbonate solutions corresponding to [(NaHCO<sub>3</sub>)<sub>n</sub>M]<sup>+</sup>, where  $M = Na^{+}$  and  $Na_{3}CO_{3}^{+}$ . Bicarbonatecontaining species had not been previously generated, because they are not stable with respect to elimination of CO<sub>2</sub>. This suggests that the DESI approach is capable of measuring relatively fragile species in their intact state. EDTA clusters were also generated intact: this molecule is an aggressive complexing agent for metal cations, and gaseous species formed by DESI contained multiple alkali or alkaline earth cations depending on the salt that was used for generating the target solution. This result was significant because EDTA clusters are not readily formed in the gas phase.

Using the capillary target, experiments were conducted to demonstrate DESI-MS efficacy for generating coordination complexes. Drops of Ba<sup>2+</sup> and La<sup>3+</sup>, both containing 18-crown-6, were generated at the end of capillary target, and analyte ions were then splashed into the gas phase by the ESI droplets. Abundant coordination complexes containing the metal, nitrate and the ligand were generated (Fig. 7).



The studies were then extended to cerium(III) tri-*n*-butyl phosphate (TBP) mixtures: activation of the target capillary syringe pump produced coordination complexes having the general formula [Ce(NO<sub>3</sub>)<sub>n</sub>(TBP)<sub>m</sub>]<sup>(3-n)+</sup> at *m/z* 1328, 766, 668, 663, and 579 (Fig. 8a).<sup>2</sup> Turning the pump off resulted in disappearance of the coordination complexes (Fig. 8b), and produced a spectrum containing species containing only TBP and adventitious Na<sup>+</sup>. Restarting the pump for 1 second (Fig. 8c) reproduced the Ce coordination complexes in abundance, an observation that suggests that the technique will be capable of very good sensitivity.

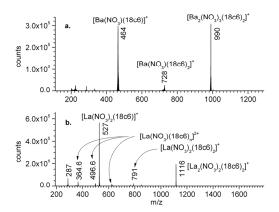


FIGURE 7. Positive ion capillary target-DESI mass spectrum of solutions containing 18-crown-6 and a. barium nitrate, and b. lanthanum nitrate.

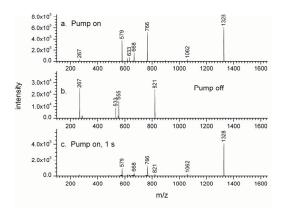


FIGURE 8. Positive ion capillary target DESI mass spectrum of an equimolar solution of Ce(III) and tri-n-butylphosphate.

#### Benefits to DOE

This research project has produced a new capability for Idaho National Laboratory (INL), enabling mass spectrometric analyses of metal speciation from solutions and solids that are at high ionic strength and or extreme pH. The research will enable INL to compete more effectively for programs in the areas of metal contamination, separations and renewable fuels (since the TM-DESI approach is also very likely applicable to rapid characterization of biological molecules). In the remaining years of this project, the path forward will be toward measurement of ligand radiolysis products, a technical problem that needs improved measurement technology.

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220

# **Isotope Ratio Measurements Methods for Direct Analysis of Samples**

Jill R. Scott (PI), Andrew J. Effenberger Jr., Timothy R. McJunkin Acquisition of isotope ratios with little or no sample preparation

AS108

The Nuclear Nonproliferation Initiative and the Separations and Actinide Science Distinctive Signature both have identified the need to develop advanced detection technologies for identifying nuclear signatures (i.e., Pu and U isotope ratios) from a variety of sample types. The current workhorse for determining isotope ratios is thermal ionization mass spectrometry (TIMS). Two of the limiting factors for application of TIMS are: (1) it requires extensive, time-consuming sample preparation and (2) its ionization efficiency is reported to be only 0.2 to 10%. Both of these factors contribute to its incompatibility with the analysis of small particles and heterogeneous environmental samples.

Therefore, we are developing the following two laser-desorption-based methods to analyze isotope ratios from heterogeneous samples with little or no sample preparation: (1) laser-induced breakdown spectroscopy (LIBS) and (2) laser desorption/ionization (LD) Fourier transform ion cyclotron resonance mass spectrometry (FTICR-MS). Both of these techniques will be coupled with our highly accurate laser positioning system associated with the laser-based optical and chemical imager (LOCI) that allows surfaces of heterogeneous samples to be mapped.

### Project Summary

During the first year (Fiscal Year 2009 [FY-09]), we focused on incorporating a new single-photon ionization source and improvements to cell design and electronics used for the LD-FTICR-MS. In Year 1, we also conducted a preliminary investigation on the best methods to improve LOCI's optical detection for detecting isotope ratios via LIBS. The keys to improvement required understanding the best vacuum or low-pressure environment conditions and how the high magnetic field effects the LIBS plume. To this end, we constructed a test

chamber to explore the vacuum/pressure conditions separate from the magnetic field effects. During FY-10, we have (1) conducted studies on the effect that different atmospheric conditions have on LIBS plume and spectra, especially vacuum and gases (e.g., Ar and He); (2) explored improved sensitivity using double pulsed LIBS at the various conditions, including the variations in experimental arrangements; and (3) designed and tested an optical detection methodology that should allow observation of high-resolution spectra for desired isotopes more economically and in a more compact form factor compared to the traditional, expensive 1-to-2 m long spectrographs. Results were presented at the International Atomic Energy Agency (IAEA) in Japan and LIBS 2010 and published in Andrew J. Effenberger, Jr. and Jill R. Scott, "Effect of Atmospheric Conditions on LIBS Spectra," Sensors 2010, 10, 4907–4925.

### Benefits to DOE

While the technologies being developed in this project are not yet fully realized, they have attracted interest from the Department of Energy (DOE) and other agencies (i.e., IAEA) because of the potential to eliminate the time consuming and costly sample preparation associated with isotope ratio analysis by thermal ionization mass spectrometry (TIMS). These new capabilities, coupled with the imaging capability of LOCI, would allow heterogeneous samples to be analyzed via the imaging capability of LOCI as opposed to the bulk analysis provided by TIMS. This type of capability would be useful for nonproliferation analyses as well as analyzing nuclear fuels to determine burn-up rates, which could lead to better, more efficient designs of nuclear fuel. Thus, this work can help DOE and the nation in the area of nuclear energy, both in the arena of national security and energy efficiency.



# **Investigation of Molten Bromide Salt Systems for Separation and Recovery of Actinides from Fission Products**

Tedd Lister, Steve Herrmann, Guy Fredrickson
Investigating the applicability of a bromide-based molten salt system to improve the separation and recovery of actinides from fission products in spent nuclear fuel

AS109

The objective of this proposed research is to investigate the applicability of a bromide-based molten salt system, such as LiBr-KBr or CsBr-LiBr-KBr, to improve the separation and recovery of actinides from fission products in spent nuclear fuel. The use of bromide-based salts may introduce three technical benefits: (1) a lower operating temperature, (2) improved separation of actinides from fission products, and (3) the potential to improve recovery of actinides by employing bromide electrolysis at the anode.

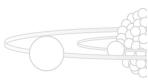
### Project Summary

The proposal outlined a strategy in which the initial task would involve planning and preparation for experimental work. This strategy has been out focus to date; experimental work has not been initiated, although experiments have been planned. A thorough literature review has been completed where both physical/chemical data for bromide salt systems were identified. Using this data, comparisons have been made with the established chloride salts systems in use. The lower melting point of bromide salts suggests that electrochemical studies can, in principle, be carried out at a lower operating temperature. However, lower operating temperatures have other disadvantages such as higher electrolyte drag out during electrorefining and lower throughput. Bromide salts are less oxidizing compared to their chloride counterparts, an added advantage in limiting the

number of electro-active species during a variety of electrochemical measurements. Bromide salts are less dense than chloride salts, which might help limit electrolyte loss by way of vaporization. The melting point of the actinide tri-bromides (UBr<sub>3</sub>, NPBr<sub>3</sub>, PuBr<sub>3</sub>, AmBr<sub>3</sub>, and CmBr<sub>3</sub>) is usually lower than the corresponding tri-chlorides, which clearly can be an advantage. One of the less-desirable properties of bromide salts is the increased cost over equivalent chloride salts.

#### Benefits to DOE

The proposed research will expand expertise in the electrochemical treatment of spent nuclear fuels and further establish Idaho National Laboratory as a world leader in innovative fuelcycle technology. This work supports a recently hired postdoctoral candidate and advancement of the spent nuclear fuel treatment and spent nuclear fuel cycle programs. Advances in separation of actinides could lead to development of proliferation-friendly technologies for peaceful recycling of spent nuclear fuels. By providing insight into the behavior of fission-product metals in electrochemical systems, this research directly supports efforts related to the reduction and minimization of radioactive wastes generated by the spent nuclear fuel treatment and spent nuclear fuel cycle programs.



# Appendices





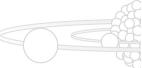
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224

# Appendix A — Authors







226

# Appendix A — Authors

| Project ID     | Principal Investigator  | Page #     | Project ID | Principal Investigator | Page # |
|----------------|-------------------------|------------|------------|------------------------|--------|
|                | A                       |            | ST141      | Henriksen, J.          | 109    |
| CT125          | Andaman M.O.            | 101        | NE161      | Herbst, R.             | 36     |
| ST135<br>FF108 | Anderson, M. O.         | 101<br>125 | NE157      | Herrmann, S.           | 34     |
| ST140          | Apel, W. A.<br>Apel, W. | 106        | BS108      | Hurley, D. H.          | 118    |
| ST140<br>ST145 | Apel, W.                | 114        | ST136      | Hurley, D. H.          | 103    |
| 51175          | B                       | 114        |            | J                      |        |
|                |                         |            | NE163      | Janney, D.             | 44     |
| CA116          | Bala, G.                | 169        | ST139      | Jeffers, R.            | 105    |
| EI114          | Benson, M.              | 137        | CA122      | Joe, J.                | 178    |
| ST132          | Benson, M. T.           | 95         |            |                        |        |
| NN115          | Bjornard, T.            | 149        |            | K                      |        |
| IC108          | Buttles, J.             | 195        | PH102      | Kelly, D.              | 79     |
|                | C                       |            | NN117      | Kinlaw, M.             | 153    |
| EI117          | Cherry, R.              | 140        | ST131      | Klaehn, J. R.          | 90     |
| NN113          | Chichester, D. L.       | 145        | NE158      | Knudson, D.            | 47     |
| NN116          | Chichester, D. L.       | 150        |            | L                      |        |
| AF105          | Cole, J.                | 207        |            |                        |        |
| CA118          | Cole, J.                | 174        | NS166      | Lacy, J.               | 68     |
| EI109          | Cooper, C.              | 131        | GB104      | Lee, B.                | 107    |
| 2110)          |                         |            | GB105      | Lee, B. D.             | 108    |
|                | D                       |            | ST133      | Lee, R.                | 99     |
| PH101          | Deaton, J.              | 77         | NU101      | Lessing, P.            | 41     |
| NE156          | Dinh, N.                | 7          | NN118      | Li, S.                 | 156    |
| NE156          | Dinh, N.                | 24         | BS110      | Lillo, T.              | 85     |
|                | Г                       |            | AS109      | Lister, T.             | 222    |
|                | F                       |            | EI118      | Lister, T.             | 139    |
| BS109          | Farrell, H.             | 120        |            | M                      |        |
| IC110          | Few, D.                 | 200        | PH103      | Marshall, D.           | 54     |
|                | G                       |            | ST143      | Mattson, E.            | 112    |
| EN102          | Gan, J.                 | 59         | NN114      | McGrath, C.            | 148    |
| IC111          | Gan, J.<br>Garcia, H.   | 201        | IC107      | McJunkin, T.           | 9      |
| NU100          | Gertman, D.             | 49         | IC107      | McJunkin, T.           | 194    |
| AF104          | Giglio, J.              | 39         | PH105      | McJunkin, T            | 115    |
| EI113          | Ginosar, D.             | 136        | CA119      | McLing, T.             | 173    |
| AS107          | Groenewold, G. S.       | 217        | NS169      | McQueen, M.            | 75     |
| AS10/          |                         | 21/        | FF105      | Meakin, P.             | 121    |
|                | Н                       |            | NN119      | Metcalf, R.            | 11     |
| NE159          | Hansen, G.              | 25         | NN119      | Metcalf, R.            | 157    |
| PH104          | Harris, B.              | 55         | AF106      | Millet, P.             | 208    |
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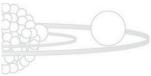


## Appendix A — Authors

| Project ID | Principal Investigator | Page # | Project ID | Principal Investigator  | Page # |
|------------|------------------------|--------|------------|-------------------------|--------|
| TM110      | Millet, P.             | 185    | CA120      | Sencer, B.              | 175    |
| NE147      | Mincher, B.            | 33     | EI112      | Shunn, L.               | 15     |
| NE160      | Mincher, B.            | 35     | EI112      | Shunn, L.               | 135    |
| NS168      | Moradi, H.             | 72     | IC112      | Shunn, L. (Tolle, Chip) | 203    |
| NE166      | Mousseau, V.           | 29     | CA117      | Simpson, M.             | 172    |
| CA123      | Myers, K.              | 179    | FF106      | Smartt, H. B.           | 122    |
|            | N                      |        | EI115      | Stewart, F. F.          | 138    |
|            |                        |        | ST130      | Stewart, F. F.          | 89     |
| ST142      | Newby, D.              | 111    | ST144      | Stewart, F. F.          | 17     |
|            | 0                      |        | ST144      | Stewart, F. F.          | 113    |
|            |                        |        | FF107      | Stone, M. L.            | 86     |
| AF107      | Okuniewski, M. A.      | 13     |            | T                       |        |
| AF107      | Okuniewski, M. A.      | 210    |            | 1                       |        |
| TM112      | O'Leary, P.            | 187    | NE165      | Tawfik, M.              | 53     |
|            | P                      |        | NE164      | Taylor, S. C.           | 51     |
| E1107      | D.I. C.D.              | 120    | IN102      | Teysseyre, S.           | 162    |
| EI107      | Palmer, C. D.          | 129    | NE146      | Tranter, T.             | 31     |
| EN101      | Payne, S.              | 58     | NN120      | Tranter, T.             | 158    |
| AS105      | Pepper, S. E           | 216    |            | V                       |        |
| TM111      | Pernice, M.            | 186    |            |                         |        |
| AS106      | Peterman, D. R.        | 215    | NS170      | Van Siclen, C.          | 76     |
| EI116      | Petkovic, L. M.        | 141    | IC106      | Vollmer, D.             | 193    |
| CA115      | Piet, S.               | 165    |            | W                       |        |
|            | R                      |        | D.1101     | XX7 I                   | 1.61   |
| NE153      | Rabiti, C.             | 21     | IN101      | Werner, J               | 161    |
| TM109      | Rabiti, C.             | 183    | FF104      | Wolf, D W               | 56     |
| BS107      | Rashkeev, S.           | 116    | ST134      | Wood, T. R.             | 96     |
| EN103      | Richins, W.            | 60     |            | Z                       |        |
| IC109      | Rieger, C.             | 197    | NE162      | Zalumalai D             | 37     |
| NS165      | Rieger, C.             | 65     |            | Zalupski, P.            |        |
| NE150      | Rempe, J.              | 45     | SM101      | Zagula, T.              | 83     |
| EI111      | Robertson, E. P.       | 134    |            |                         |        |
| GS101      | Robertson, E. P.       | 94     |            |                         |        |
| NS167      | Roybal, L.             | 70     |            |                         |        |
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| AS108      | Scott, J. R.           | 221    |            |                         |        |
| CA121      | Searcy, E.             | 176    |            |                         |        |
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| 111 100    | bollooi, D.            | 211    |            |                         |        |

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228





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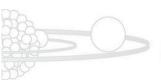
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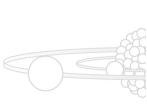
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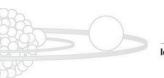
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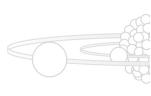
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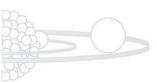
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| AF104   | PI<br>Hilton,<br>Bruce Allen | Title  Development of Advanced Burnup Measurement and Nuclear Forensics using Inductively Coupled Mass Spectroscopy (ICP-MS) Isotopics Analysis Techniques | S        | <u> </u> | <u> </u> | H  | S  | <u>Z</u><br>P | Z  | N<br>N | O   | 0  | <u>a</u> | .T |
| AF105   | Cole, James                  | Development of a<br>Small Sample Volume<br>Mechanical Properties<br>Testing Technique for<br>Irradiated Fuels and<br>Materials                             |          |          |          |    | S  | P             | S  |        |     |    |          |    |
| AF106   | Millet, Paul                 | Irradiation-Induced<br>Evolution of Defects<br>and Microstructures in<br>Nanocrystalline BCC<br>Mo   |          |          |          |    | S  | P             |    |        |     |    |          |    |
| AF107   | Okuniewski,<br>Maria         | Microstructural Evolution of UO2 and U Under Irradiation   |          |          |          |    | P  | S             |    |        |     |    |          |    |
| AF108   | Sencer,<br>Bulent            | Irradiation Testing and<br>Molecular Modeling of<br>Irradiation-Assisted<br>Diffusion and<br>Microstructural<br>Evolution (FCCI)                           |          |          |          |    |    | P             |    |        |     |    |          |    |
| AS105   | Pepper,<br>Sarah             | Rapid detection of plutonium, neptunium and technetium in water samples  | P        |          |          |    | S  | S             |    |        |     |    |          |    |
| AS106   | Peterman,<br>Dean            | Evaluation of Covalent<br>Interactions in Actinide<br>Coordination<br>Compounds  | S        |          | S        |    | S  | P             |    |        | S   |    |          |    |
| AS107   | Groenewold,<br>Gary Steven   | Measuring Actinide<br>Speciation in High pH<br>Solutions   |          |          | S        |    | P  | S             |    |        |     |    |          |    |
| AS108   | Scott, Jill R                | Isotope Ratio<br>Measurements Methods<br>for Direct Analysis of<br>Samples   | P        | S        | S        |    | S  | S             | S  |        |     |    |          | S  |
| AS109   | Lister, Tedd                 | Investigation of Molten<br>Bromide Salt Systems<br>for Separation and<br>Recovery of Actinides<br>from Fission Products                                    |          |          | S        |    |    | P             |    |        | S   |    |          |    |
| BS107   | Rashkeev,<br>Sergey          | Conducting perovskite materials for catalytic applications   |          | P        |          | S  | S  |               |    |        |     |    | S        |    |



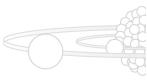
| Project<br>ID | PI                    | Title   | NNS<br>A | EE | EM | FE | SC | NE | NR | Z | OCR | OE | ESH | ГМ |
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| BS108         | Hurley,<br>David H    | Acoustic<br>Nanostructures  |          | S  |    | P  |    |    |    |   |     |    |     |    |
| BS109         | Farrell,<br>Helen     | A new Paradigm for<br>Robust Catalysts  |          | P  |    | S  | S  | S  |    |   |     |    |     |    |
| BS110         | Lillo, Tom            | Advanced Multi-Layer<br>Laminate Blast<br>Mitigation System   | P        |    |    |    | S  | S  |    |   |     | S  |     |    |
| CA115         | Piet, Steven<br>James | Investigation of Public<br>Discourse Methods in<br>Energy Policy<br>Decision-making   |          | S  | S  |    |    | P  |    |   | S   |    |     |    |
| CA116         | Bala, Greg            | Development of<br>Lignocellulosic Ethanol<br>Production Potential in<br>Idaho   |          | P  |    |    |    |    |    |   |     |    |     |    |
| CA117         | Simpson,<br>Michael   | Optimization of Ceramic Waste Forms Used for Electrochemical Processing of Spent Nuclear Fuel                                       |          |    |    |    |    | P  |    |   |     |    |     |    |
| CA118         | Cole, James           | Fabrication of<br>Advanced ODS Alloys<br>using Field Assisted<br>Sintering  |          |    |    |    | S  | P  |    |   |     |    |     |    |
| CA119         | McLing,<br>Travis     | Prediction and Monitoring of CO2 Behavior in Deep Reactive Geologic Formations  |          |    |    | P  |    |    |    |   |     | S  | S   |    |
| CA120         | Sencer,<br>Bulent     | Small specimen test<br>techniques for<br>evaluating radiation-<br>induced changes in<br>mechanical properties                       |          |    |    |    |    | P  |    |   |     |    |     |    |
| CA121         | Searcy, Erin          | Design and Operational<br>Improvements and<br>LCA in Anaerobic<br>Digestion of Fermented<br>Dairy Manure using a<br>2-Stage Process |          | P  | S  |    |    |    |    |   |     |    |     |    |
| CA122         | Joe, Jeffrey          | Transmission Site<br>Suitability Decision<br>Support Technology   |          | S  | S  |    | S  |    |    |   |     | P  |     |    |
| CA123         | Myers, Kurt           | CAES Vertical Axis<br>Wind Turbine  |          | P  |    |    |    |    |    |   |     |    |     |    |
| EI107         | Palmer, Carl<br>David | Generation and<br>Expulsion of<br>Hydrocarbons from Oil<br>Shale  |          |    |    | P  | S  |    |    |   |     |    |     |    |

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| Project<br>ID | PI                               | Title  | SNN 4 | EE | EM | FE | SC | NE | NR | Z | OCR<br>WM | OE | ESH | ГМ |
| EI109         | Cooper,<br>David Craig           | Pathways to Energy<br>Independence   |       | P  |    | S  | S  | S  |    |   |           | S  |     |    |
| EI111         | Robertson,<br>Eric<br>Partridge  | Altering Wettability by<br>Chemical Amendments<br>to Improve Gas<br>Production from Tight<br>Sands |       |    | S  | P  |    |    |    |   |           |    |     |    |
| EI112         | Shunn, Lee                       | Adaptive Process Modeling Using Parameter Estimation and Mechanism Sensitivity Analysis.           |       |    |    | S  |    | P  |    |   |           |    |     |    |
| EI113         | Ginosar,<br>Daniel               | Feedstock Processing and Energy Storage  |       | P  |    | S  |    | S  |    |   |           | S  |     |    |
| EI114         | Benson,<br>Michael               | Mesocilicate Supported CO2 Capture Material  |       | P  |    | S  |    |    |    |   |           |    |     |    |
| EI115         | Stewart,<br>Frederick<br>Forrest | Membrane Separation<br>System Research for<br>the HYTEST facility                                  |       | P  |    | S  |    |    |    |   |           |    |     |    |
| EI116         | Petkovic,<br>Lucia               | Conversion of light hydrocarbons to fuels and chemicals  |       | P  |    | S  | S  | S  |    |   |           | S  |     |    |
| EI117         | Cherry,<br>Robert                | Hybrid Systems process integration and dynamics studies  |       | S  |    | S  |    | P  |    |   |           |    |     |    |
| EI118         | Lister, Tedd                     | Electrochemical reduction of CO2 to CO and Hydrocarbon Fuels                                       |       | P  |    | S  |    |    |    |   |           | S  |     |    |
| EN101         | Payne,<br>Suzette                | Aleatory vs Epistemic<br>Uncertainty in Seismic<br>Hazard Analyses                                 | S     |    | S  |    |    | Р  | S  |   |           |    |     | S  |
| EN102         | Gan, Jian                        | In-Pile Temperature<br>Monitor and Control for<br>ATR  |       |    |    |    |    | P  |    |   |           |    |     |    |
| EN103         | Richins,<br>William              | Multi-Rate Shock<br>Physics Simulation of<br>Blast and Penetration<br>Events in Concrete           | S     |    |    |    | S  | P  |    |   | S         |    |     |    |
| FF104         | Wolf, Dieter<br>Wilhelm          | Integrated Mesoscale<br>Approach for the<br>Simulation of Nuclear-<br>Fuel Behavior                |       |    |    |    | P  | S  |    |   |           |    |     |    |
| FF105         | Meakin,<br>Paul                  | Integrated Mesoscale<br>Approach for the<br>Simulation of Nuclear-<br>Fuel Behavior                |       |    | S  | P  | S  |    |    |   |           |    |     |    |
| FF106         | Smart,<br>Herschel               | Scaling of Welding<br>Processes  |       | P  |    | S  | S  | S  |    |   |           |    |     |    |



| Project<br>ID | PI                              | Title   | NNS<br>A | EE | EM | FE | SC | NE | NR | Z | OCR<br>WM | OE | ESH | LM |
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| FF107         | Stone, Mark<br>L                | Enhanced Metal Ion<br>Analysis  | P        |    | S  |    | S  | S  |    |   |           |    | S   |    |
| FF108         | Apel,<br>William<br>Arnold      | Effect of glycosylation<br>on the activity and<br>stability of bacterial<br>enzymes   |          | S  |    |    | P  |    |    |   |           |    |     |    |
| GB104         | Lee, Brady<br>Douglas           | Metabolic Engineering<br>of Alicyclobacillus<br>acidocaldarius for<br>Lactic Acid Production<br>from Biomass Derived<br>Monosaccharides       |          | P  |    |    | S  |    |    |   |           |    |     |    |
| GB105         | Lee, Brady<br>Douglas           | Network Interaction In<br>the Thermoacidophile<br>Alicyclobacillus<br>acidocaldarius In<br>Response to Different<br>Complex Carbon<br>Sources |          | P  |    |    | S  |    |    |   |           |    |     |    |
| GS101         | Robertson,<br>Eric<br>Partridge | Reducing CBM Water<br>Discharge Volume  |          |    | S  | Р  |    |    |    |   |           |    |     |    |
| IC106         | Vollmer,<br>Denis Todd          | Resilient Control<br>System Network<br>Agents   | S        | S  |    |    |    | S  |    |   |           | P  |     |    |
| IC107         | Cherry,<br>Shane                | Integrated Control<br>System Data Fusion  |          |    |    |    | S  | S  |    |   |           | P  |     |    |
| IC108         | Buttles, John                   | Wireless Sensor<br>Testing  | S        |    | S  |    |    | P  |    |   | S         |    | S   |    |
| IC109         | Rieger,<br>Craig                | Anomaly Detection, Diagnosis, and Resilient Control in Complex Engineered Systems   |          | S  |    | S  | S  | P  |    |   |           | S  | S   |    |
| IC110         | Few, Doug                       | 3D Spatial<br>Representation in<br>Support of Design<br>Inspection and<br>Verification  | P        |    |    |    |    | S  |    |   |           |    |     |    |
| IC111         | Garcia,<br>Humberto             | Resilient Condition<br>Assessment Monitoring<br>(ReCAM) System  | S        | P  |    | S  | S  | S  |    |   |           | S  | S   |    |
| IC112         | Shunn, Lee                      | Automated Differential<br>Equation-Based<br>Identification  | S        | S  |    |    | P  | S  |    |   |           |    |     |    |
| IN101         | Werner, Jim                     | Design and Testing of a<br>Mars Hopper  |          |    |    |    | S  | P  |    |   |           |    |     |    |



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| Project<br>ID | PI                      | Title  | NNS A | EE | EM | FE | SC | NE | NR | Z | OCR<br>WM | OE | ESH | ГМ |
| IN102         | Teysseyre,<br>Sebastian | In situ characterization<br>of an oxide film for<br>prediction of stress<br>corrosion cracking<br>susceptibility   |       |    |    |    | S  | Р  |    |   |           |    |     |    |
| NE146         | Tranter,<br>Troy Joseph | Process Modeling of<br>Solvent Extraction<br>Separations for<br>Advanced Nuclear Fuel<br>Cycles  | S     |    | S  |    |    | P  |    |   | S         |    |     |    |
| NE147         | Mincher,<br>Bruce Jay   | Reactivity of radiolytically produced nitrogen oxide radicals toward aromatic compounds.   | S     |    |    |    | S  | P  |    |   |           |    |     |    |
| NE150         | Rempe, Joy<br>Lynn      | Viability Evaluations of<br>Linear Variable<br>Differential<br>Transformers (LVDTs)<br>and Capacitive Micro-<br>Machined Ultrasonic<br>Transducers (CMUTs)<br>for In-Pile<br>Instrumentation |       |    |    |    |    | S  | P  |   |           |    |     |    |
| NE153         | Rabiti,<br>Cristian     | Development of Reactor Physics Sensitivity Analysis, Uncertainty Quantification, and Data Assimilation Capability at INL for Validation Applications   |       |    |    |    |    | P  |    |   |           |    |     |    |
| NE156         | Dinh, Nam               | Next-Generation Code<br>for Nuclear System   |       |    |    |    |    | P  | S  |   |           |    |     |    |
| NE157         | Herrmann,<br>Steve      | Characterization of a Consolidated Electrochemical Technique for Separation and Recovery of Actinides from Fission Products in Oxide Fuels   |       |    |    |    |    | P  |    |   |           |    |     |    |
| NE158         | Knudson,<br>Darrell     | Instrumentation for inpile detection of thermal conductivity   |       |    |    |    |    | P  | S  |   |           |    |     |    |
| NE159         | Hansen,<br>Glen         | Multiscale Modeling<br>and Simulation of<br>Nuclear Fuel<br>Performance  |       |    |    |    | S  | P  |    |   |           |    |     |    |



| Project<br>ID | PI                        | Title   | NNS | EE | EM | FE | SC | NE | NR | NN | OCR<br>WM | OE | ESH | LM |
|---------------|---------------------------|---|-----|----|----|----|----|----|----|----|-----------|----|-----|----|
| NE160         | Mincher,<br>Bruce         | Neptunium redox<br>chemistry in irradiated<br>aqueous nitric acid   |     |    | S  |    |    | P  |    |    |           |    |     |    |
| NE161         | Herbst, Scott             | Reversible Gas Phase<br>Reactions for Recovery<br>of Graphite from<br>Recycled HTGR<br>TRISO Fuel                       |     | S  |    |    |    | P  |    |    |           |    |     |    |
| NE162         | Zalupski,<br>Peter        | Comprehensive Thermodynamic Models for Aqueous Partitioning of Actinides from Used Nuclear Fuel.                        | S   |    | S  |    | S  | P  |    |    | S         |    |     | S  |
| NE163         | Janney,<br>Dawn           | Spatially-correlated microstructural characterization: From centimeters to nanometers                                   | S   |    | S  |    | S  | P  |    |    | S         |    |     | S  |
| NE164         | Taylor,<br>Steven         | Ultrasonic Transducer<br>Sensors for In-Pile<br>Detection of<br>Dimensional changes                                     | S   |    |    |    |    | P  | S  |    |           |    |     |    |
| NE165         | Tawfik,<br>Magdy          | Smart grid impact on commercial nuclear plants  |     | S  |    |    |    | P  |    |    |           |    |     |    |
| NE166         | Mousseau,<br>Vince        | Nuclear Reactor Safety<br>Case  |     |    |    |    | S  | P  | S  |    |           |    |     |    |
| NN113         | Chichester,<br>David Lee  | Active Interrogation Die-away Assay Development Program   | P   |    |    |    |    | S  |    |    | S         |    |     |    |
| NN114         | Baker, John<br>David      | 135Xe recovery from<br>the spontaneous fission<br>of 252Cf  | P   |    |    |    |    |    |    |    |           |    |     |    |
| NN115         | Phillips,<br>Jerry Harold | Developing a Next<br>Generation, Risk-<br>Informed Approach for<br>Robust and Resilient<br>Design Development<br>(R2D2) | S   |    |    |    |    | P  |    |    |           |    |     |    |
| NN116         | Chichester,<br>David Lee  | NEUTRON<br>SPECTROMETER<br>DEVELOPMENT  | P   |    | S  |    | S  | S  | S  |    | S         |    | S   |    |
| NN117         | Kinlaw,<br>Matthew        | Nuclear Material<br>Detection Using<br>Neutron Time-of-Flight   | P   |    |    |    |    |    |    |    |           |    |     |    |

| Project<br>ID | PI                      | Title   | NNS<br>A | EE | EM | FE | SC | NE NE | NR. | Z | OCR | OE | ESH | LM |
|---------------|-------------------------|---|----------|----|----|----|----|-------|-----|---|-----|----|-----|----|
| NN118         | Jue, Jan-<br>Fong       | On-line Monitoring of<br>Actinide<br>Concentrations for<br>Advanced Aqueous<br>Separation Processes   | P        |    |    |    |    | S     |     |   |     |    |     |    |
| NN119         | Metcalf,<br>Richard     | Develop a Safeguards<br>Approach for INL<br>Pyroprocessing lines<br>for Demonstration to<br>IAEA  | P        |    |    |    |    | S     |     |   |     |    |     |    |
| NN120         | Tranter,<br>Troy Joseph | Radionuclide Collection-Detection Device for the in situ Remote Monitoring of 99Tc as a Proliferation Indicator   | P        |    | S  |    | S  | S     |     |   | S   |    | S   |    |
| NS165         | Rieger,<br>Craig        | Methodologies for the<br>Design, Analysis, and<br>Validation for<br>Operation of Complex<br>Resilient Networks  |          | S  |    | S  | S  | S     |     |   |     | P  | S   |    |
| NS166         | Lacy, Jeffrey           | Computational<br>Materials<br>Characterization  | P        |    |    |    |    |       |     |   |     |    |     |    |
| NS167         | Roybal, Lyle            | Next Generation<br>Control System "Smart<br>Grid" Simulation<br>Environment   |          | S  |    |    |    |       |     |   |     | P  |     |    |
| NS168         | Moradi,<br>Hussein      | Wireless<br>Communications R&D  | S        |    |    |    | S  | S     |     |   | P   |    |     |    |
| NS169         | McQueen,<br>Miles       | Cyber Security for Protection of Critical Infrastructure Expanding the Scientific and Engineering Bases   |          |    |    |    | S  | S     |     |   |     | P  |     |    |
| NS170         | Van Siclen,<br>Clint    | Object Reconstruction<br>Technique for use in<br>Radiography  | P        |    |    |    |    |       |     |   |     |    |     |    |
| NU100         | Gertman,<br>David Ira   | Human Performance Assessment for Technology Neutral Evaluation: Combining Virtual and Physical Testing for Design, Development and Review of Digital Control Systems and Interfaces | S        |    |    |    | S  | P     |     |   |     |    |     |    |



| Project<br>ID | PI                               | Title   | NNS | EE | EM | FE | SC | NE | NR | Z | OCR<br>WM | OE | ESH | LM |
|---------------|----------------------------------|---|-----|----|----|----|----|----|----|---|-----------|----|-----|----|
| NU101         | Lessing,<br>Paul Alan            | Advanced Ceramic<br>Nuclear Fuels   |     |    |    |    |    | Р  |    |   |           |    |     |    |
| PH101         | Deaton, Juan<br>Diego            | Cognitive Network Engine and Simulation Framework,Ph.D. Candidate Proposal for Juan Deaton                          | P   | S  |    |    |    |    |    |   |           | S  |     |    |
| PH102         | Kelly, Dana                      | Application of Dynamic Bayesian Networks to Systems with Ambient Intelligence                                       | S   |    |    |    |    | P  |    |   |           | S  |     |    |
| PH103         | Marshall,<br>Douglas             | Characterization of<br>Fluidized Beds via<br>Pressure-Fluctuation<br>Analysis                                       |     | S  |    | S  |    | P  |    |   |           |    |     |    |
| PH104         | Harris, Brian                    | Extraction of Fission Products in Room Temperature Ionic Liquids and sc-CO <sub>2</sub> with Study of Radiolysis    | S   |    | P  |    |    | S  |    |   |           |    |     |    |
| PH105         | McJunkin,<br>Timothy             | Advanced Adaptive Algorithms in Phased Array Ultrasonics for Materials Inspection                                   |     |    | S  | S  | S  | P  |    |   | S         |    |     |    |
| SM101         | Zagula,<br>Thomas                | Materials Research for Armor Development  | P   |    |    |    | S  | S  |    |   |           | S  |     |    |
| ST130         | Stewart,<br>Frederick<br>Forrest | Investigation of Low<br>Temperature<br>Performance in<br>Membrane Materials<br>and Processes for Gas<br>Separations |     | P  | S  | S  |    | S  |    |   |           |    | S   |    |
| ST131         | Klaehn, John<br>Ray              | High-Performance Polymer Membranes for High Temperature Gas Separations   |     |    | S  | P  |    | S  |    |   |           | S  |     |    |
| ST132         | Benson,<br>Michael<br>Timothy    | Triazine-Based CO2<br>Capture Agents  |     | S  |    | P  |    |    |    |   |           |    |     |    |
| ST133         | Hruska,<br>Ryan<br>Christopher   | Dynamic Impact Model<br>and Information System<br>to support<br>Unconventional Fuels<br>Development                 |     |    | S  | P  | S  |    |    |   |           |    |     | S  |
| ST134         | Wood,<br>Thomas<br>Ronald        | Near Field Impacts of<br>In-Situ Oil Shale<br>Development on Water<br>Quality-                                      |     | S  | S  | P  | S  |    |    |   |           |    | S   |    |

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| Project<br>ID | PI                               | Title  | NNS A | EE | EM | FE | SC | NE | NR | Z | OCR<br>WM | OE | ESH | ГM |
| ST135         | Anderson,<br>Matthew             | Advanced Remote<br>Sensing for Energy and<br>Environmental<br>Applications using<br>Unmanned Aerial<br>Vehicles          | S     |    | P  | S  | S  | S  |    |   |           |    |     | S  |
| ST136         | Hurley,<br>David                 | Addressing the<br>Spectrum of Nuclear<br>Related NDE Needs: A<br>hybrid laser ultrasonic<br>and eddy current<br>approach |       |    |    | S  |    | P  | S  |   |           |    |     |    |
| ST139         | Jeffers,<br>Robert               | Water and Energy<br>System<br>Interdependency<br>Modeling for<br>Multicriteria Decision<br>Analysis                      |       | P  | S  | S  | S  | S  |    |   |           | S  |     |    |
| ST140         | Apel,<br>William                 | Degradation and<br>Conversion of Lignin<br>using Extremophilic<br>Systems  |       | P  |    |    | S  |    |    |   |           |    |     |    |
| ST141         | Henriksen,<br>James              | Specific Biological<br>Responses to Nano<br>Metal Oxides   |       |    | S  |    | P  |    |    |   |           |    | S   |    |
| ST142         | Newby,<br>Deborah                | Development of Non-<br>Lethal Methods for<br>Enhanced Lipid<br>Recovery from<br>Microalgae                               |       | P  |    |    | S  |    |    |   |           |    |     |    |
| ST143         | Mattson,<br>Earl                 | Development of Thermally Generated In Situ Precipitation Barriers due to Subsurface Heat Injection                       |       |    | S  | S  | P  |    |    |   | S         |    |     |    |
| ST144         | Stewart,<br>Frederick<br>Forrest | Hybrid Osmosis Water<br>Purification Systems<br>Research   |       | P  | S  | S  |    | S  |    |   |           |    |     |    |
| ST145         | Apel,<br>William                 | Biological Fixation of<br>CO2 for Fuel and<br>Chemical Production<br>Using Acetogen<br>Intermediary<br>Metabolism        |       | P  |    |    | S  |    |    |   |           |    |     |    |
| TM109         | Rabiti,<br>Cristian              | Study of preconditioning techniques for Krylov solvers applied to hybrid neutron transport calculations                  |       |    |    |    |    | P  |    |   |           |    |     |    |



| Project<br>ID | PI                  | Title   | NNS | EE | EM | FE | SC | NE | NR | NN | OCR<br>WM | OE | ESH | LM |
|---------------|---------------------|---|-----|----|----|----|----|----|----|----|-----------|----|-----|----|
| TM110         | Millet, Paul        | Fickian and Thermal Diffusion in Nuclear Materials from Linear Response Theory and Multiscale Simulations |     |    |    |    | S  | P  |    |    | S         |    |     |    |
| TM111         | Pernice,<br>Michael | Error Estimation for stochastic uncertainty quantification  | S   |    | S  | S  | S  | P  |    |    |           |    |     |    |
| TM112         | O'Leary,<br>Patrick | Advanced Visualization using Immersive Environments   | S   | S  | S  | S  | S  | P  |    |    |           | S  |     |    |

