

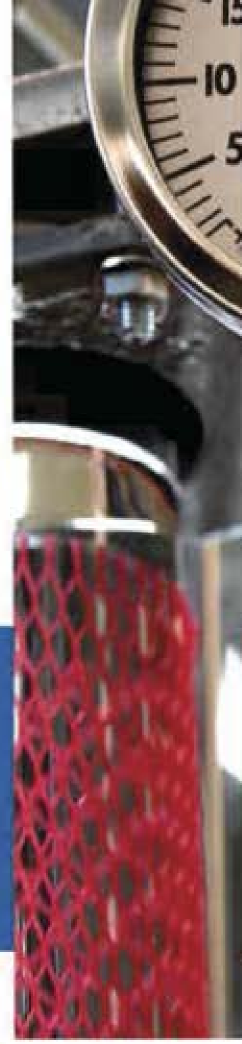


2016 | ANNUAL REPORT

Nuclear Science User Facilities

NSUF Nuclear Science User Facilities

Analytical Laboratory,
Materials & Fuels Complex (MFC),
Idaho National Laboratory (INL)

Nuclear Science User Facilities
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Disclaimer

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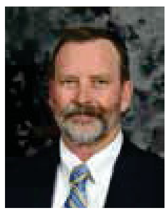

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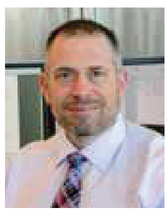

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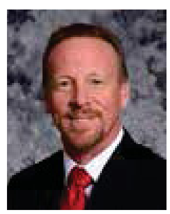

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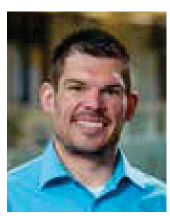

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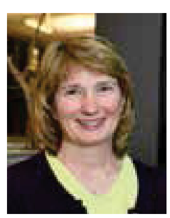

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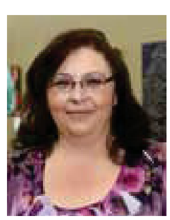

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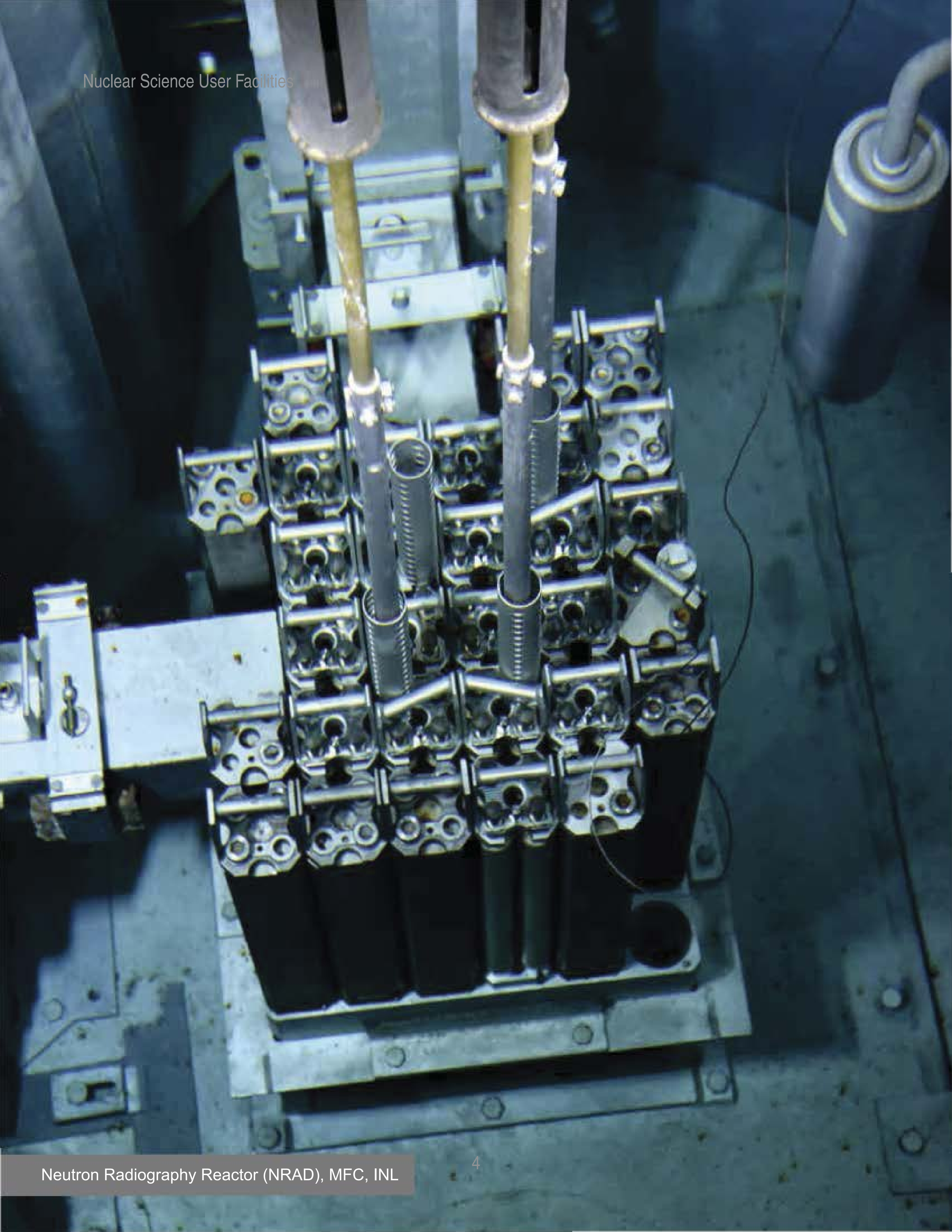

Neutron Radiography Reactor (NRAD), MFC, INL

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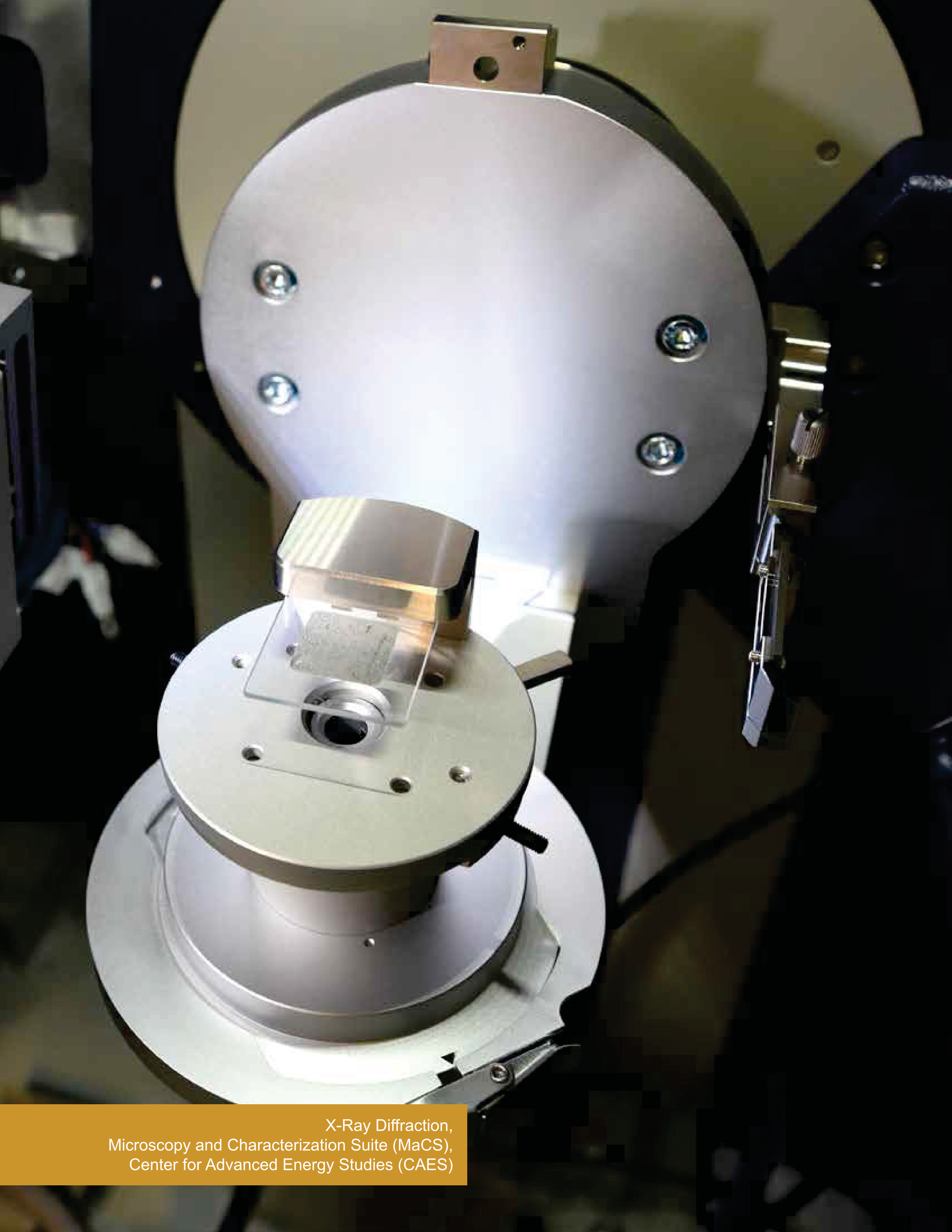

X-Ray Diffraction,
Microscopy and Characterization Suite (MaCS),
Center for Advanced Energy Studies (CAES)

FROM THE NSUF DIRECTOR

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The Nuclear Science User Facilities (NSUF) will celebrate our 10th anniversary in 2017. Since it was originally founded as the Advanced Test Reactor National Scientific User Facility (ATR-NSUF), the NSUF has grown into a truly national – and international – organization. Speaking of the ATR, this national research treasure will also be celebrating its 50th anniversary in July 2017. Established in 2007, the NSUF has focused on the goal of serving as a scientific and technical foundation for the U.S. Department of Energy's Office of Nuclear Energy (DOE-NE) as it advances nuclear power to meet our nation's energy, environmental, and national security needs.

The NSUF's shared history with ATR brings into focus the tremendous asset that ATR has provided to nuclear energy researchers and innovators. The NSUF has completed 13 ATR irradiations and currently has five ATR projects underway. NSUF researchers have published over 28 refereed journal papers to date based on research utilizing the ATR.

The NSUF team has also grown to support a research and development ecosystem to better enable high-impact nuclear energy research. We welcomed three new team members in 2016 who are profiled in this report:

- Kelly Cunningham, Nuclear Fuels & Materials Library Coordinator,
- Jonathan Kirkham, Scientific Support Professional, and
- John Coody, Project Scheduler.

With our added team members, we continued to enhance two important NSUF resources: the Nuclear Fuels and Materials Library (NFML) and the Nuclear Energy Infrastructure Database (NEID). Kelly Cunningham brought the NFML online as a web-based, searchable database of legacy irradiated nuclear fuels and materials available to the nuclear community for advanced scientific studies. Jonathan Kirkham, working with NSUF Capabilities Coordinator Brenden Heidrich, continued to build out the NEID database. In September 2016, it contained data from up to 127 institutions supporting about 465 facilities that house close to 1,000 instruments and equipment, including research and test reactors, conventional and advanced instrumentation as well as other related information from both U.S. and international institutions. Created in fiscal year (FY) 2015, the NEID is designed to be a tool for researchers and developers across federal government agencies, the national laboratory system, industry, universities, and international organizations.

In 2016, the NSUF made the NEID database available to the newly established Gateway for Accelerated Innovation in Nuclear (GAIN) initiative to facilitate GAIN industry users' identification of capabilities that can advance their technologies. The NSUF is pleased to engage with the GAIN initiative in its mission to provide industry innovators the technical and regulatory support they need to move new or advanced nuclear technology concepts and designs toward commercialization. The combined resources offered through the NSUF and the GAIN initiative provide an unprecedented diversity of nuclear energy researchers and innovators access to world-class capabilities, infrastructure, and expertise in all aspects of nuclear energy technologies.

2016 was a banner year for the NSUF in both the number of proposals received and the number and value of projects awarded. This is a clear indication of the worth that the nuclear energy research community finds in the NSUF, and bringing on John Coody has allowed the NSUF to continue to manage this increasing volume of projects effectively. In FY 2016, the NSUF awarded 39 projects from a pool of 75 applications from 24 institutions during the thrice-yearly Rapid Turnaround Experiments solicitations, a 30 percent increase in awarded proposals and a 60 percent increase in proposals received over FY 2015 respectively. The annual Consolidated Innovative Nuclear Research (CINR) Funding Opportunity Announcement (FOA) drew 67 pre-applications (a 116 percent increase) with final submissions of 32 full applications (an 88 percent increase) resulting in 12 awards (a 160 percent increase) totaling about $10 million in support (up from $4.2 million in FY 2015).

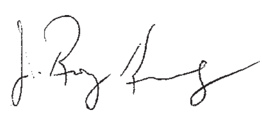
To expand user awareness of our capabilities, the NSUF maintains an active presence in research-related circles. We exhibited in FY 2016 at the American Nuclear Society (ANS) winter meeting, the Electric Power Research Institute (EPRI) International Light Water Reactor Materials Reliability Conference, the Materials Research Society (MRS) fall meeting, The Minerals, Metals and Materials Society (TMS) spring meeting, and the Nuclear Materials Conference (NuMat). Our impact becomes greater as traditionally non-nuclear focused researchers understand the opportunities posed in working in the nuclear field and how they can benefit from capabilities offered by the NSUF. In addition, the NSUF organized a session at the ANS winter meeting and, together with the NSUF Users Organization, will continue organizing NSUF conference sessions in the future to showcase the science we are producing.

Our researchers are achieving scientific prominence by publishing and documenting research results through peer-reviewed journals and presentations at conferences. We recorded the highest number of journal publications in NSUF history in 2016 with 55, which beat the previous high of 33 in 2015. Due to the nature of NSUF related research, which can require longer time duration to yield results from experiments that tend to involve greater expense and require access to specialized and unique facilities, we expect our publication record to continue to increase as we reap the benefits from earlier irradiation tests. Since the NSUF works to reduce barriers to research, the steady growth in publications is testimony to DOE-NE's commitment to advancing nuclear energy.

FY 2016 saw the NSUF expand its associations in the international arena. We continued our interactions with the UK's National Nuclear User Facility and finalized discussions with the SCK-CEN Belgian Nuclear Research Centre on a pilot project involving irradiations in the ATR and Belgium Reactor-2 (BR-2). This will be followed by post-irradiation examination (PIE) activities at the Laboratory for High and Medium Activity at the Belgian Nuclear Research Centre and at Idaho National Laboratory (INL) Materials and Fuels Complex (MFC), the Transient Reactor Test (TREAT) Facility, and High Temperature Test Laboratory (HTTL) in the United States. Other NSUF facilities may be brought in at a later time. We expect a Memorandum of Understanding to be signed between DOE-NE and the Belgian SCK-CEN in the early part of FY 2017.

Looking further ahead in 2017, we are working to provide our users access to additional capabilities, such as the National Synchrotron Light Source II (NSLS-II) at Brookhaven National Laboratory. Several other institutions have expressed interest in joining the NSUF Partner Facilities consortium and we will evaluate their applications in 2017. We will build on the productive outcomes from the FY 2016 Ion Beam Workshop by bringing together a committee to develop an Ion Beam Utilization Roadmap. We look forward to hosting a Thermal Hydraulics Workshop to identify potential research opportunities as part of an expanded NSUF scope. Finally, we will hold our inaugural NSUF Partner Facilities Working Group meeting in an effort to better engage our Partner Facilities in the activities of the NSUF.

Please take a few moments to learn more about all the important nuclear research facilitated by NSUF at Idaho National Laboratory and our diverse mix of affiliated partner institutions across the United States I thank the NSUF partners and users for their hard work to make the NSUF a successful strategic research organization.



Focused Ion Beam Confinement Box, Irradiated Materials Characterization Laboratory (IMCL), MFC, INL

Local Electrode Atom Probe (LEAP), MaCs, CAES

Q&A WITH TOM MILLER

Director
Office of Accelerated Innovation
in Nuclear Energy
U.S. Department of Energy

Q: *Can you tell us more about what you do at the Department of Energy Headquarters (U.S. DOE-HQ)?*

A: Currently, my role within the Office of Nuclear Energy is to direct and manage those research, development, and infrastructure programs that support the accelerated development of innovative nuclear technology. The Nuclear Science User Facilities (NSUF) is one of the key programs within my responsibilities.

Q: *What are some of your other responsibilities?*

A: Other key program responsibilities include the Nuclear Energy Enabling Technology (NEET) program sponsoring research in advanced modeling and simulation, advanced sensors and instrumentation, and advanced methods for manufacturing. In addition, I have responsibility for the university programs involving scholarships, fellowships, university reactor fuel, and the university research and development program.

Q: *How long have you been with DOE?*

A: I joined the U.S. Department of Energy in 1999.

Q: *What other areas have you worked with while at DOE?*

A: I have worked for the Office of Nuclear Energy throughout my tenure with DOE in a variety of programmatic roles supporting the deployment of new reactor technology. Most recently I served as the director for the Light Water Technology Office with responsibilities for the Small Modular Reactor Licensing Technical Support and Light Water Reactor Sustainability programs. Prior to that, I was responsible for the development and implementation of the Nuclear Power 2010 program, which supported the technology and licensing of the first new nuclear plants to be built in the United States in over 30 years.

Q: *What did you do prior to joining DOE-HQ?*

A: I worked in the commercial nuclear industry for 26 years prior to joining the DOE in a variety of roles and positions.

Q: *What's your educational background?*

A: Bachelor's degree in Nuclear Science from the State University of New York Maritime College

Q: *What got you interested in nuclear energy?*

A: During my sophomore year in college, I did a paper on the NS Savannah, the first commercial nuclear powered ship. As a maritime student, I was intrigued with the commercial applications of nuclear energy.

Q: *What brought you to NSUF oversight?*

A: Actually, it was the result of a job offer I couldn't refuse as part of a reorganization.

Q: *What do you like to do outside of the office (hobbies, volunteer activities, interests)?*

A: My wife (mostly) and I operate a horse farm. We board and take care of other people and our own horses. It is a lot of work, but we enjoy it. My wife is an avid rider and fox hunter.

Q: *The NSUF is celebrating its 10th anniversary in 2017. What do you see as the NSUF's greatest accomplishments?*

A: Establishing the Advanced Test Reactor – National Scientific User Facility, as it was originally known, was a significant success, but recognizing that the needs of the nuclear energy community extended beyond the original irradiation and post irradiation examination capabilities, and moving into the

NSUF consortium that exists today is one of its greatest achievements. The range of facility representation and capabilities in the NSUF consortium from universities, national laboratories, and even industry is very unique and quite impressive, encompassing a broad range of irradiated materials science. In late 2016, the NSUF established an international partnership agreement with the Belgium research reactor to further expand its research infrastructure capabilities. And new capabilities and facilities are being examined yearly, all in an effort to continuously support the nuclear stakeholder community and provide the necessary access to support the advancement of nuclear energy.

The NSUF also took on the challenge to develop and maintain the Nuclear Energy Infrastructure Database, which was no small feat, and was able to release this unique capability in FY 2016. We've received feedback from both users and facilities that the database does a great job identifying relevant nuclear related infrastructure. Building upon that success, the Nuclear Fuels and Materials Library will further assist researchers by identifying available irradiated specimens to examine at those available facilities.

Q: *Where do you see the NSUF's best opportunities?*

A: The NSUF's best opportunities are the access the program provides for researchers to unique nuclear irradiation and post-irradiation examination capabilities – not only to the physical instruments, but the knowledge, data, and expertise of the people operating these facilities and equipment. Being the link between all of those components will truly help the understanding and advancement of nuclear energy.

Q: *What are challenges facing the NSUF?*

A: The current challenge, and I think this is an agreement across the community, is the availability of neutrons. The NSUF originally began as a way to provide access to the Advanced Test Reactor, but space in the reactor has become limited in recent years. Limited space at the nation's top materials test reactor is, of course, reflective of the revitalization of nuclear energy, but it has also made the NSUF look at alternatives for meeting the community's demand. To help offset the demand for the ATR, the NSUF is partnered with a number of test reactors within the United States and has recently signed a Memorandum of Understanding with the Belgian Research Reactor in Belgium.

Q: *The Nuclear Fuels and Materials Library in FY 2016 evolved from spreadsheet data to an online database searchable by researchers. How do you foresee the NFML benefiting DOE-NE?*

A: I see this capability as one more component to the NSUF's access. We already discussed the NEID and this database will further assist the user community in accessing necessary irradiated materials that have a known pedigree. Those samples come from industry, national laboratories, previously funded NSUF projects, and samples that have been identified by the community for the NSUF to irradiate through a series of Sample Library (SAM) experiments. As to how it benefits DOE-NE, we're able to allocate more funding to more awards through the use of this database. That is a great advantage for everyone.

Q: *How is the NSUF viewed by DOE-NE?*

A: DOE-NE relies on the NSUF to make those connections to physical equipment, samples, researchers, knowledge, data, and other aspects within the nuclear energy research framework. Because of this, we look at the NSUF as the vital partner to the Gateway for Accelerated Innovation in Nuclear (GAIN) initiative to help the advancement of nuclear energy. The NSUF provides that lower level Technology Readiness Level (TRL) research that supports the greater industry.

Q: *If you look into your crystal ball, what changes do you see in store for the NSUF over the next ten years?*

A: The NSUF is currently focused on irradiated materials characterization, which will certainly provide the data needed by the advanced reactor community. Moving forward, it is important to look at what other research infrastructure is needed by the stakeholder community, whether this capability is available within the laboratory and university community, and whether DOE investment in this capability is necessary. The NSUF team has been doing this with a focus on irradiated materials and now is expanding to look at other nuclear science areas. As we move forward, the NSUF team will be looking to support the advancement of nuclear energy across a broad array of R&D areas, such as thermal hydraulics and other related advanced reactors areas.

Analytical Laboratory, MFC, INL

FOCUS ON NEW STAFF

Kelly Cunningham

Coordinator for the Nuclear Fuels and Materials Library

Nuclear Science User Facilities (NSUF) researchers access the Nuclear Fuels and Materials Library to locate irradiated material and fuel samples for further experimentation. Kelly Cunningham is charged with keeping the library information current, relevant to the NSUF mission, and available to researchers.

The Nuclear Fuels and Materials Library is owned by the U.S. Department of Energy Office of Nuclear Energy (DOE-NE) and curated by the NSUF. The Library is a collection of specialized information and nuclear fuel and material specimens from past and ongoing irradiation test campaigns, real-world components retrieved from decommissioned power reactors, and donations from other sources.

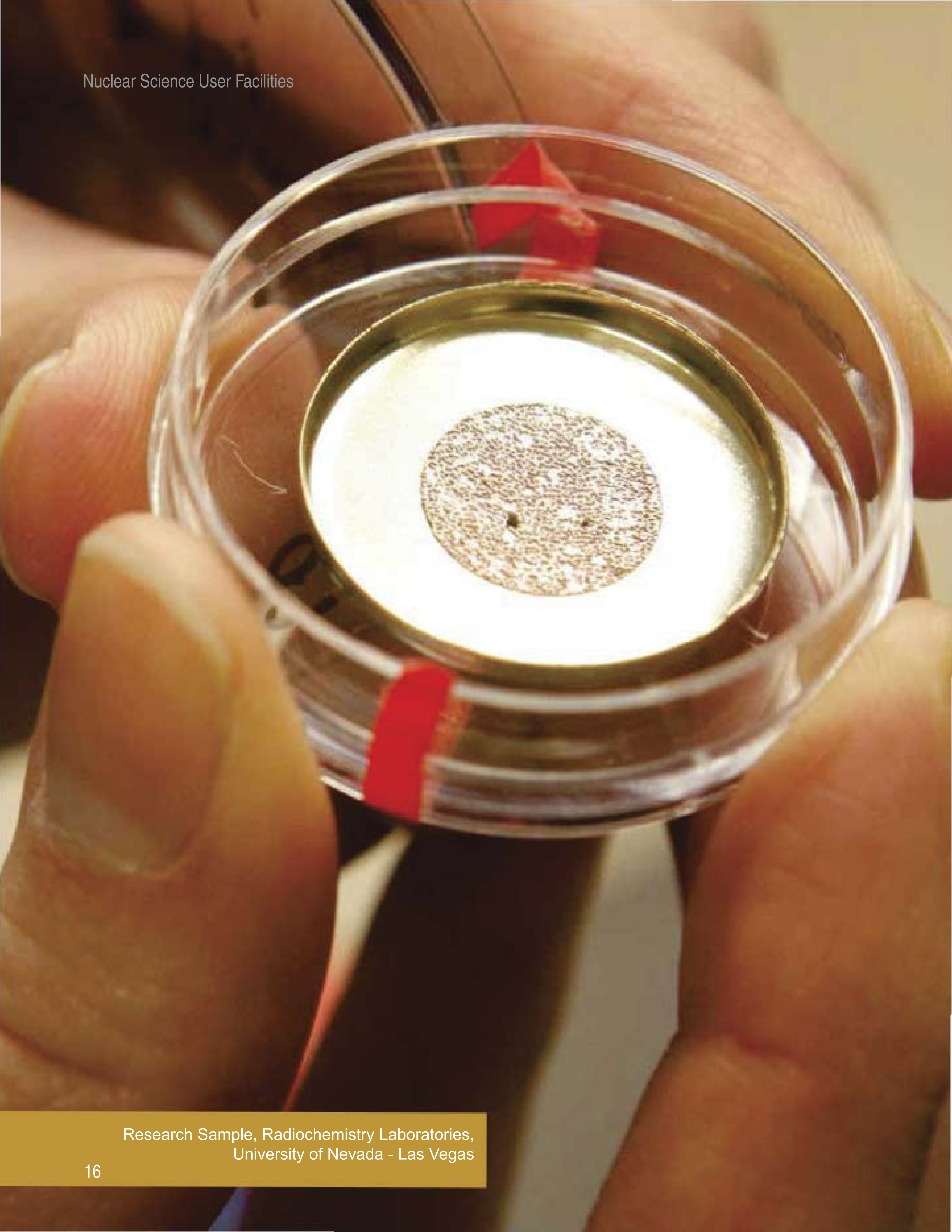
Cunningham's role when joining the NSUF was to transform the information contained in multiple spreadsheets into an online library to provide intuitive access to users. "Taking on a brand new project that had the potential to make researchers' lives easier was exciting," Cunningham said. "I like being the person who works with specialists to resolve any issues a researcher has when using the library."

Cunningham is used to tackling new challenges. Upon earning her degree in radiographic science, she worked for a few years as a radiographic and MRI technologist in health sciences. She joined Battelle Energy Alliance, LLC in 2004 and initially worked at the Materials and Fuels Complex as a Health Physics Technician. Over time, she worked in a variety of laboratory mission areas, including National & Homeland Security, before joining the NSUF.

"The best part of my time with Idaho National Laboratory (INL) has been the people," Cunningham said. "Working with really supportive people on interesting and important projects keeps me engaged."

The original materials database contained approximately 3,500 specimens, including legacy materials, volunteered materials, and NSUF project specimens. Most specimens had been neutron irradiated; some were proton irradiated. The spreadsheets were eventually consolidated and migrated into an online database with the help of an NSUF dedicated information management specialist.

Transmission Electronic Microscope (TEM),
Materials Science & Technology Laboratory,
Pacific Northwest National Laboratory (PNNL) - Courtesy of PNNL



Research Sample, Radiochemistry Laboratories,
University of Nevada - Las Vegas

"The biggest challenge we have is that the information has been provided by different researchers that don't use the same names for the same materials," Cunningham explained. "Developing a consistent nomenclature will streamline searches and continue the evolution of developing the database into a true Library."

The NSUF's goal is to have the Library continue to expand and showcase the available range of samples and experimental data. This goal means that Cunningham will continue to work with national labs, universities, and industry to collect valuable materials into a single, public repository that serves the nuclear community and DOE-NE's mission.

"The NSUF Nuclear Fuels and Materials Library will continue to be a valuable research portal to support scientific outcomes," said Cunningham. "I enjoy hearing the Principal Investigators present findings based on experiments done with Library materials, and I'm always interested in hearing feedback and suggestions on how to improve the Library."

Read more about the NSUF Nuclear Fuels and Materials Library on page 38.

"Developing a consistent nomenclature will streamline searches and continue the evolution of developing the database into a true Library."

Jonathan Kirkham

Keeper of the Nuclear Energy Infrastructure Database (NEID)

How the Nuclear Science User Facilities (NSUF) interacts with the United States nuclear industry and research community depends to a great extent on the information that's available to them. As the scientific support specialist responsible for the Nuclear Energy Infrastructure Database (NEID), Jonathan Kirkham plays a critical role on the NSUF staff.

The purpose of the NEID is to provide information on all capabilities and facilities related to nuclear energy, including institutions, facilities, and instrumentation. This gives members of the nuclear research community the means to search for capabilities on current or future projects. For the U.S. Department of Energy Office of Nuclear Energy, it provides aid for infrastructure investment decisions. For the NSUF itself, it allows staff members to search for the best and most cost effective capabilities available. The NEID provides support for the Gap Analysis Report, Nuclear Energy Request for Information (RFI), and Nuclear Energy Infrastructure Funding Opportunity Announcements (FOAs).

For Kirkham, the challenge is keeping the information complete and up to date, scouring reference sources for little known or overlooked resources (i.e., "Trying to find a lot of stuff that's not published in anything."). An Idaho Falls native who graduated from the University of Idaho with a bachelor's in physics, Kirkham was working as a research assistant and consultant in Boise before joining the NSUF staff in October 2015.

Making the jump from physics to materials science has meant learning about procedures and equipment. Making use of the environment at the Center for Advanced Energy Studies and its affiliation with four universities, Kirkham is pursuing a master's in nuclear engineering.

In 2016, one of his projects was to update the NEID's online presence. In accomplishing this, he has managed to give it:

- A new look, feel, and navigation properties
- Editing capabilities for institution officials and facility owners
- New coordinate mapping through Google Maps
- User profiles interconnected throughout all NSUF databases
- User sign-up through the main NSUF webpage
- The ability to save a list of facilities or instruments for quicker access

The NEID webpage can be accessed through NSUF's main webpage or at nsuf-infrastructure.inl.gov.

As of September 2016, NEID had data from 127 institutions, 465 facilities, and 963 instruments. The majority of institutional data – 79.5 percent — came from universities (49), government (30) and industry (22) in the United States. The remainder came from corresponding international sources.

Irradiation Assisted Stress Corrosion Cracking Facility,
Energy Innovation Laboratory (EIL), INL

Irradiated Materials Characterization Laboratory (IMCL), MFC, INL

John Coody

NSUF scheduler at home in Project Controls

As scheduler for the Nuclear Science User Facilities (NSUF), John Coody is used to keeping a lot of balls in the air.

His job involves working with researchers and experiment managers, looking at a scoping statement, and figuring out when to schedule design, fabrication, assembly, irradiation, post-irradiation examination, and disposition of materials. But Project Control is something that comes naturally, perhaps even by birth. His father, also John Coody, was a project controller for EG&G Idaho and Lockheed Martin Idaho.

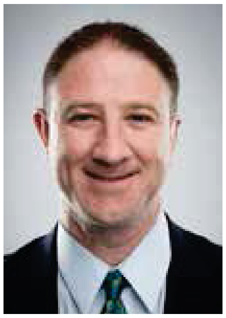
When Coody came to work for the NSUF in May 2016, it was after 20 years of working project controls for U.S. Department of Energy (DOE) contractors at Los Alamos National Laboratory, Hanford, Savannah River, and Idaho National Engineering and Environmental Laboratory. In the private sector, he worked for Motorola and at the Donald C. Cook Nuclear Generating Station in southwest Michigan.

Coody said his experience at D.C. Cook gave him valuable insight into one of the NSUF's key missions, the life cycle extension of nuclear power plants. "It's important now because all the plants are aging," he said. "The newest nuclear plants were built in the late '70s or early '80s and back then they were only licensed for 30 years." (D.C. Cook's two units, which went online in 1975 and 1978, have been relicensed by the Nuclear Regulatory Commission through 2034 and 2037.)

Coody said what he likes best about working for the NSUF is the variety. Since coming, he has worked on projects with GE Hitachi, Idaho State University, Boise State University, and Colorado School of Mines. With the number of Consolidated Innovative Nuclear Research (CINR) projects and Rapid Turnaround Experiments (RTEs) rising sharply (from 2015 to 2016, CINR full project applications went from 17 to 32 and RTE project applications rose from 47 to 75), the pace of work shows no sign of slowing down.

"We're doing something different all the time," Coody said.

RESEARCHER PROFILES



Matt Swenson

The NSUF enables researcher journey from Boise State University student to University of Idaho Assistant Professor

For a textbook case of how the Nuclear Science User Facilities (NSUF) is supposed to work, one might not need look further than Matthew Swenson, who defended his doctoral dissertation in May 2017 and is headed to the University of Idaho in the fall to be an assistant professor in mechanical engineering.

"The NSUF has been the complete enabler of all my research," he said.

After graduating from Oregon State University, Swenson spent 14 years in the heavy equipment industry as an engineering manager and platform leader. "All along I knew I was interested in graduate studies and obtaining an advanced degree," he said.

When he enrolled in Boise State University's (BSU) materials science and engineering program in 2013, he found a mentor in Dr. Janelle Wharry, whose focus was on irradiation experiments and introduced him to nuclear materials.

BSU is a member of the consortium that forms the Center for Advanced Energy Studies, where the NSUF has its offices. Over three years, Swenson estimated he made the 570 mile round-trip between Boise and Idaho Falls between 25 and 30 times.

Swenson joined Wharry's group at BSU in June 2013. One of the first things she had him do was attend the NSUF Users Week in Idaho Falls. "I think, in hindsight, it was a really good thing for him," she said. "He was exposed to all the research that was going on in the nuclear materials world and got to meet other students and learn what they were doing."

Not only did it give him a full picture of the research, "It gave me an orientation on how the process works, how proposals were to be written," Swenson said.

His research has focused on radiation resistance of iron based steel alloys containing nanoparticles, used as cladding and structural components in nuclear reactors, optimizing the microstructures to improve their durability under irradiation. More particularly, he has been developing lab experiments that use charged particles to emulate what is happening in nuclear reactors. Experiments with charged particles can be done in days rather than months, and the materials are not radioactive at the end of the experiment.

Matt Swenson, MaCS, CAES

“We’re hoping to have a calculation model that can be used as a predictive tool for folks developing alloy materials for reactors,” he said.

For the necessary comparisons, Swenson has used data collected on materials exposed to radiation in Idaho National Laboratory’s (INL) Advanced Test Reactor, the NSUF’s original asset and still one of its most important.

Swenson was awarded his first project, entitled “Understanding the Effects of Irradiation Dose Rate and Particle Type in Ferritic/Martensitic Alloys” in 2015. The award provided him with access to materials characterization equipment at the NSUF, where he compared the effects of charged particle irradiation to neutron irradiation on the microstructural evolution of advanced F/M alloys HT9 and HCM12A.

He made use of the Microscopy and Characterization Suite (MaCS) at the Center for Advanced Energy Studies (CAES), with instruments such as the focused ion beam (FIB), transmission electron microscope (TEM), and Localized Electron Atom Probe (LEAP). “It works really well to make a sample at CAES, do analysis onsite at CAES with the tools at MaCS, then take all the data home,” he said. “Without the resources at CAES, I probably would have picked a different school than Boise State University.”

His involvement in the NSUF has led to a seat as the student member on the NSUF Users Organization’s Executive Committee, which serves as an advocacy group for the NSUF’s experimental activities and provides a communication channel among users of the NSUF. “The networking opportunity really makes a big difference,” he said.

Wharry, now at Purdue University, views the NSUF as essential to Swenson’s success. “I don’t think I can give enough credit to how important it was as a resource to him,” she said. “The rapid turnaround projects gave him a lot of experience to help him become proficient in microscopy.”

They also provided a learning curve for proposal writing – a skill all graduate students need to learn. “Writing his own proposals, I think that was a really big step for him,” she said.

Wharry said the NSUF is a resource she plans to continue to use with her students. One of her Purdue students, Keyou Mao, had plans to spend more than a month at CAES in summer 2017. “It’s an incredible resource,” she said. “I’ve seen this model that has worked really well for Matt and me.”

“It works really well to make a sample at CAES, do analysis onsite at CAES with the tools at MaCS, then take all the data home. Without the resources at CAES, I probably would have picked a different school than Boise State University.”

Yong Yang

Users Organization: Building continuity to secure the NSUF's future

In the evolution of any organization, institutional memory and continuity are key to long-term success. As the Nuclear Science User Facilities (NSUF) approaches its tenth anniversary, some of the people who were "present at the creation" in support roles have become the leaders.

Now an associate professor of nuclear engineering at the University of Florida, Dr. Yong Yang was a postdoctoral fellow of Dr. Todd Allen at the University of Wisconsin in 2008 when he participated in the Advanced Test Reactor (ATR) NSUF pilot experiment.

After 40 years of almost exclusive use by the U.S. Navy, the U.S. Department of Energy in 2007 had designated ATR and associated post-irradiation examination facilities as a National Scientific User Facility, allowing broader access to nuclear energy researchers.

"It gave universities access to a lot of equipment and opened up partnership opportunities," said Allen, the first ATR NSUF director. As a member of Allen's team, Yang oversaw the assembly of capsules for irradiation in ATR, labeling them and making sure they got to Idaho National Laboratory safely and on time.

"He deserves a lot of credit for the work he did on the first experiment, getting it done," Allen said.

As a graduate student, Yang had studied metal matrix composite materials for car engines. His interest in nuclear energy started after graduation. "If you look at the energy industry, nuclear is the future," he said. His research focus shifted to radiation damage occurring in light water reactor core structural components.

Yang was one of the 68 people – students, university faculty, and industry representatives – to attend the first weeklong ATR NSUF summer sessions, which eventually became known as Users Week. It featured presentations by 19 technical experts and covered topics such as irradiation damage mechanisms, degradation of reactor materials, light water reactor and gas reactor fuels, and non-destructive evaluation.

"I saw a lot of great technical presentations," Yang said. "It covered a whole spectrum of new, innovative studies." In 2009, Yang's proposal for post-irradiation examination of ceramics for stability in advanced fuel applications was one of four NSUF experiments to be selected. Other projects have included:

- characterization on Bor-60 neutron irradiated austenitic stainless steels and cast stainless steel,
- characterization of neutron irradiated NF709 stainless steel using atom probe tomography,
- evaluation of ferrite decomposition in irradiated and aged duplex cast stainless steels,
- low temperature Fe-ion irradiation of 15-15Ti steel in different thermo-mechanical states, and
- synergistic effects of thermal aging and neutron irradiation in 304L welds.

Yong Yang, INL Research Center

Transmission Electron Microscope, MaCS, CAES

In 2016, Yang became chairman of the NSUF Users Organization, which was set up in 2010 to provide a formal and clear channel for the exchange of information, advice, and best practices between investigators and NSUF management. In this position, he has turned his attention to what can be done to increase membership and promote engagement.

At the next Users Week in 2018, Yang said he hopes to see the focus expanded beyond materials, capturing a bigger audience. "I think this should be a central piece for nuclear R&D," he said.

Some objectives to bring this about include more scientific and technical presentations to balance the education and training lectures, reaching out to the nuclear industry and recruiting more of its leaders to speak. Live conferencing has also been suggested as a way to reach out to partner facilities.

The NSUF Users Organization is also teaming up with the American Nuclear Society, sponsoring and presenting at its student conferences, and encouraging current members to help with recruiting through social media applications such as Facebook and LinkedIn.

Allen said Yang is a great example of the type of professional the NSUF hopes to develop. "He's a smart scientist, good at generating business, and a good mentor," he said.

The same year he became the NSUF Users Organization chairman, Yang received his tenure and promotion to associate professor at University of Florida in 2016. In March 2017, the university's training reactor received its license renewal from the Nuclear Regulatory Commission, allowing the training reactor to remain in operation until March 2037.

For more information on the NSUF Users Organization, please visit the NSUF website at https://nsuf.inl.gov.

"[The ATR National Scientific User Facility] gave universities access to a lot of equipment and opened up partnership opportunities,"

— Dr. Todd Allen, the first ATR NSUF director

PROGRAM OVERVIEW

The NSUF: A Model for Collaboration

The Nuclear Science User Facilities (NSUF) and its partner facilities represent a prototype laboratory that utilizes a distributed partnership, with each facility bringing exceptional capabilities to the relationship including reactors, beamlines, state-of-the-art instruments, hot cells, and equally important, expertise. These capabilities and people together create a nationwide infrastructure that allows the best ideas to be tested using the most advanced capabilities. Through the NSUF, researchers and their collaborators are building on current knowledge to better understand the complex behavior of materials and fuels in a nuclear reactor.

The NSUF's partnership program in 2016 included eight universities, one research and education consortium, three national laboratories, and one industry partner. The avenues opened through these partnerships facilitate cooperative research across the country, matching people with capabilities and students with mentors. The NSUF in 2016 included Idaho National Laboratory (INL) and the following institutions:

- Argonne National Laboratory
- Center for Advanced Energy Studies (a research and education consortium between Boise State University, INL, Idaho State University, University of Idaho, and University of Wyoming)
- Illinois Institute of Technology
- Massachusetts Institute of Technology
- North Carolina State University
- Oak Ridge National Laboratory
- Pacific Northwest National Laboratory
- Purdue University
- University of California, Berkeley
- University of Michigan
- University of Nevada – Las Vegas
- University of Wisconsin
- Westinghouse Materials Center of Excellence.

This report contains details on new capabilities brought into the NSUF in 2016, the evidence of the increasing research facilitated by the NSUF, and new tools and activities to propel nuclear research and development forward to meet energy reliability and security needs. Learn about the NSUF Nuclear Fuels and Materials Library and the Nuclear Energy Infrastructure Database, and how the robust NSUF website continues to evolve to support new tools. Read about how the NSUF supports U.S. Department of Energy (DOE) missions through efforts like the 2016 NSUF Ion Beam Options Workshop, which led to a prioritization of ion beam facilities according to their ability to support U.S. Department of Energy Office of Nuclear Energy (DOE-NE) missions.

NSUF Research Supports DOE-NE Missions

As referenced in the 2010 DOE Nuclear Energy Research and Development Roadmap, DOE-NE organizes its research and development activities based on four main objectives that address challenges to expanding the use of nuclear power:

- Develop technologies and other solutions that can improve the reliability, sustain the safety, and extend the life of current reactors.
- Develop improvements in the affordability of new reactors to enable nuclear energy to help meet the administration's energy security and climate change goals.
- Develop sustainable nuclear fuel cycles.
- Understand and minimize the risks of nuclear proliferation and terrorism.

The NSUF research consortium addresses the first three of these objectives. The NSUF does not conduct classified work and all NSUF work is non-proprietary and intended for open literature publication, so applications to the NSUF research consortium have addressed only the first three of these objectives. Most of the research contained in this report looks at either understanding the mechanisms of radiation damage to materials and fuels or looks at materials and fuels for the next generation of reactors.

Take time to read through this report and familiarize yourself with the many opportunities and resources offered by the NSUF. For specific information on DOE missions, go to http://www.energy.gov/ne/mission. To learn more about proposing a research project, visit the NSUF website: http://nsuf.inl.gov.

A TREMENDOUS SUCCESS
CINR FOA

Dan Ogden
Deputy Director

As stated in the Consolidated Innovative Nuclear Research (CINR) Funding Opportunity Announcement (FOA), the U.S. Department of Energy Office of Nuclear Energy (DOE-NE) conducts crosscutting nuclear energy research and development (R&D) and associated infrastructure support activities to develop innovative technologies that offer the promise of dramatically improved performance for advanced reactors and fuel cycle concepts while also maximizing the impact of DOE resources. DOE-NE funds research activities through both competitive and direct mechanisms, as required to best meet the Department's needs.

To support DOE-NE in maximizing its nuclear energy R&D portfolio, the NSUF in fiscal year (FY) 2015 transitioned from its historical call for proposals to the CINR FOA. The CINR FOA offers NSUF users a mechanism to request R&D funding along with NSUF access in a single proposal. The single proposal process eliminated user concerns regarding fragmented and leveraged proposals that many times resulted in partial awards, i.e., NSUF access without R&D funds – or worse, no awards.

The initial offering in FY 2015 drew a fair response with 31 pre-applications. Interest more than doubled in FY 2016. After the dust settled from the kick-off webinar and letter-of-intent submittals, 67 pre-applications were submitted for the NSUF related work scopes. About half of the pre-applications were invited to submit full applications and, in the end, 12 awards were made via the CINR FOA. The total access value of the NSUF awards was approximately $10 million and R&D funds associated with the access totaled $4 million.

The next CINR FOA cycle was initiated at the close of FY 2016 and the number of pre-applications more than tripled, with 109 received. The NSUF is hoping to increase our award budget in FY 2017 – so stay tuned for the results!

For information regarding the CINR FOAs, please visit the NSUF website at https://nsuf.inl.gov.

FY 2016 CINR Awards

Title	PI	Institution	NSUF Utilization
Effects of High Dose on Laser Welded, Irradiated AISI 304SS	Janelle Wharry	Purdue University	INL Materials and Fuels Complex Westinghouse, CAES Microscopy and Characterization Suite
Feasibility of Combined Ion-Neutron Irradiation for Accessing High Dose Levels	Zhijie Jiao	University of Michigan	ORNL Low Activation Materials Development and Analysis Laboratory, Michigan Ion Beam Laboratory
Fission Product Transport in TRISO Fuel	Fei Gao	University of Michigan	Michigan Ion Beam Laboratory at the University of Michigan
Irradiation Performance Testing of Specimens Produced by Commercially Available Additive Manufacturing Techniques	Jeffrey King	Colorado School of Mines	INL Advanced Test Reactor, INL Materials and Fuels Complex, CAES Microscopy and Characterization Suite
Radiation Enhanced Diffusion of Ag, Ag-Pd, Eu and Sr in Neutron Irradiated PyC/SiC Diffusion Couples	Tyler Gerczak	Oak Ridge National Laboratory	Michigan Ion Beam Laboratory, ORNL High Flux Isotope Reactor
Radial Heat Flux – Irradiation Synergism in SiC ATF Cladding	Yutai Katoh	Oak Ridge National Laboratory	ORNL High Flux Isotope Reactor, ORNL Low Activation Materials Development and Analysis Laboratory
Enhancing Irradiation Tolerance of Steels via Nanostructuring by Innovative Manufacturing Techniques	Haiming Wen	Idaho State University	INL Advanced Test Reactor, INL Materials and Fuels Complex, CAES Microscopy and Characterization Suite
Understand the phase transformation of thermally aged and neutron irradiated duplex stainless steels used in LWRs	Yong Yang	University of Florida	Illinois Institute of Technology – MRCAT beamline at the Advanced Photon Source
Irradiation Testing of LWR Additively Manufactured Materials	Ronald Horn	GE Hitachi Nuclear Energy	INL Advanced Test Reactor, INL Materials and Fuels Complex, CAES Microscopy and Characterization Suite
Effect of Gamma Irradiation on the Microstructure and Mechanical Properties of Nano-modified Concrete	Florence Sanchez	Vanderbilt University	ORNL Gamma Irradiation Facility
Correlative Atom Probe and Electron Microscopy Study of Radiation Induced Segregation at Low and High Angle Grain Boundaries in Steels	Phil Edmondson	Oak Ridge National Laboratory	ORNL Low Activation Materials Development and Analysis Laboratory
Role of minor alloying elements on long range ordering in Ni-Cr alloys	Julie Tucker	Oregon State University	Wisconsin Tandem Accelerator Ion Beam

RTE/BEAMLINE
DEMAND INCREASES

Jeff Benson
Program Administrator

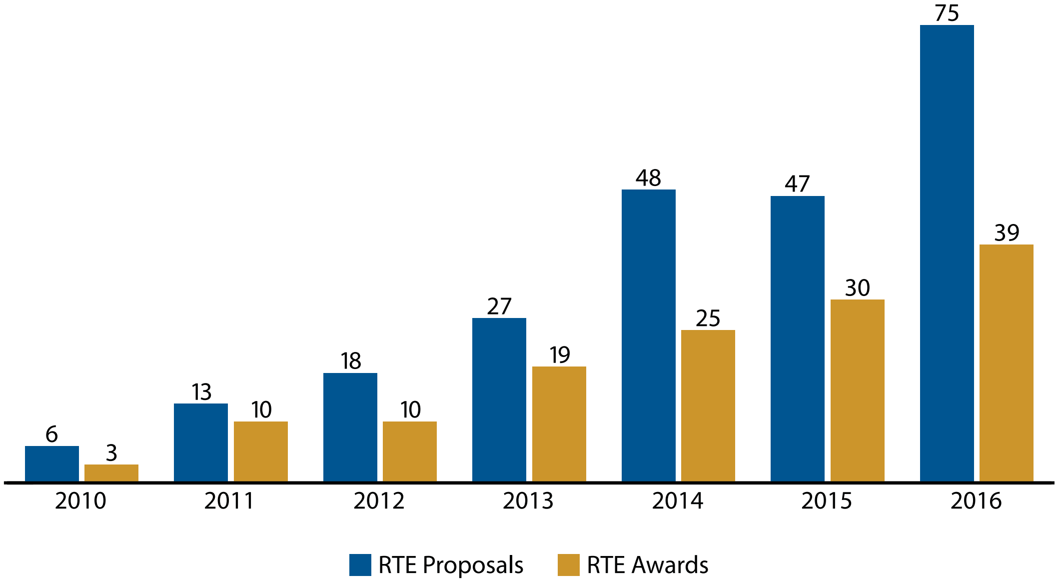
RTE Overview

The Nuclear Science User Facilities (NSUF) Rapid Turnaround Experiment (RTE) and Beamline awards provide an avenue to examine irradiation effects on nuclear fuels and materials. Once started, experiments typically are completed in two weeks or less. These awards allow researchers to utilize an NSUF facility to examine fuels or materials of interest for their research area.

RTE proposals typically request post irradiation examination requiring use of instruments (focused ion beam [FIB], transmission electron microscope [TEM], scanning electron microscope [SEM], etc.), synchrotron irradiation, ion beam irradiation, or high performance computing. Proposals are reviewed and awarded three times per year. Proposals are reviewed, and scored for technical merit, relevancy, feasibility, and then prioritized by total score.

2016 RTE Program Growth

The NSUF RTE program has grown significantly since its inception in 2010 and continued to grow in



fiscal year (FY) 2016. The chart illustrates the growth of the proposals and awards by year.

In FY 2016, the NSUF received 75 proposals from 24 different institutions. There were 28 more proposals received than from the prior year. From the 75 proposals, the NSUF selected 39 proposals for award to 18 different institutions. The number of RTE awards in 2016 represents a 30 percent increase from 2015.

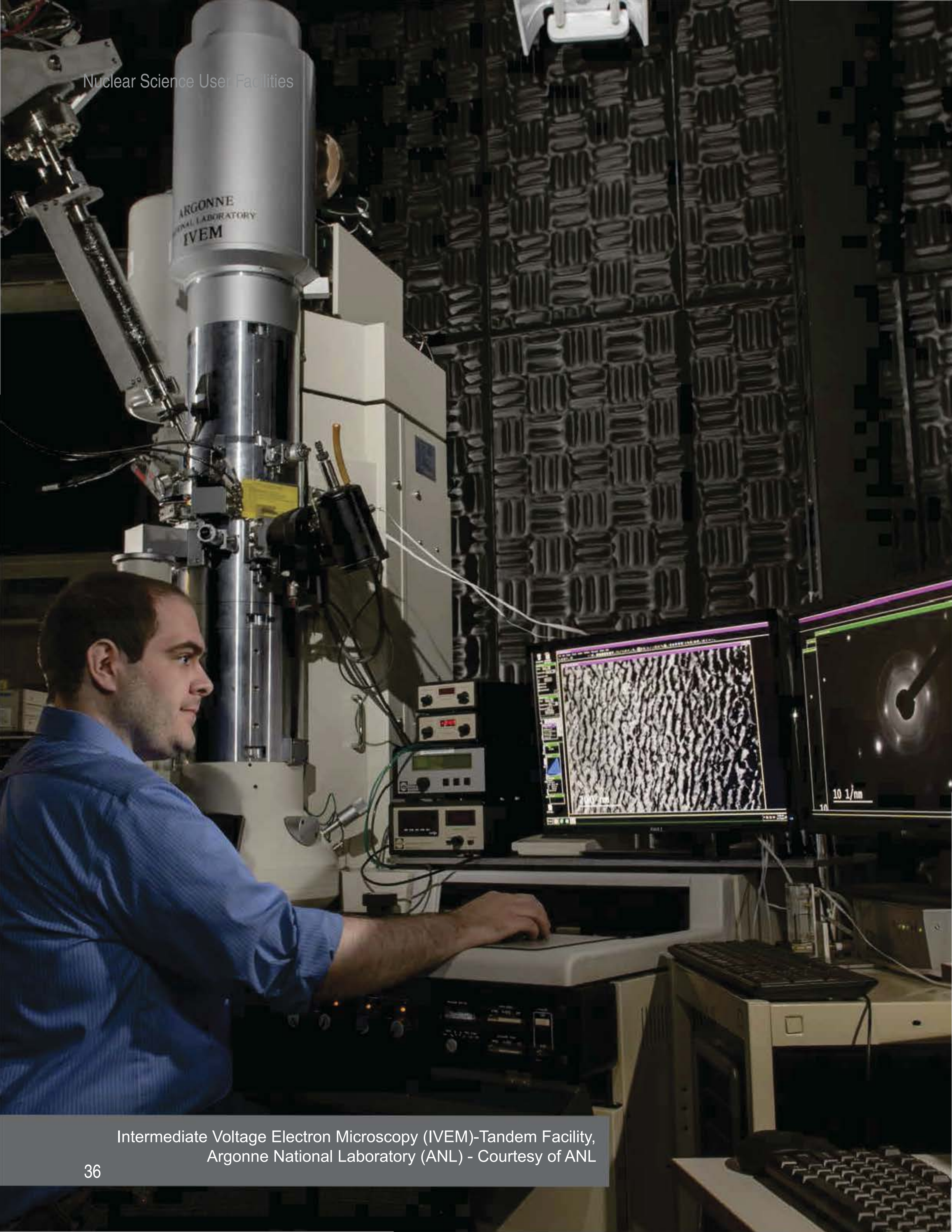
The RTE program also saw significant growth in the use of partner facilities. The 2016 RTE awards requested access to eight of the thirteen NSUF partner facilities to perform experiments. The number of NSUF partners requested for RTE awards in 2016 represents a 38 percent increase from 2015.

Summary

In 2016, the NSUF has seen a significant increase in the number of proposals and demand for access to partner facilities. As a result of the demand – and with improved budgets – the NSUF RTE program has been successful in increasing the number of awards for researchers (at no cost to the researcher) to world-class capabilities to facilitate the advancement of nuclear science and technology. Driving the increase in RTE/Beamline Awards is strong interest in the field of research and the addition of a new partner facility, the IVEM-Tandem Facility, in FY 2016.

Please visit the NSUF website at https://nsuf.inl.gov for up-to-date information on RTE and Beamline awards.

"Driving the increase in RTE/Beamline Awards is strong interest in the field of research and the addition of a new partner facility, the IVEM-Tandem Facility, in FY 2016."



Intermediate Voltage Electron Microscopy (IVEM)-Tandem Facility, Argonne National Laboratory (ANL) - Courtesy of ANL

NEW NSUF PARTNER FACILITY
IVEM-TANDEM

The Nuclear Science User Facilities (NSUF) welcomed in fiscal year (FY) 2016 a new partner facility, the Intermediate Voltage Electron Microscopy (IVEM)-Tandem Facility at Argonne National Laboratory. The IVEM-Tandem Facility combines ion beam irradiation capability with in-situ characterization using a transmission electron microscope. A total dose of 100 dpa can be achieved in about a day.

The IVEM is one of two similar facilities in the United States and is one of only five in the world. It has the unique ability to image the changes in microstructure and defect formation during irradiation at high magnification. The IVEM interfaces with an ion beamline incident from above at 30 degrees to the electron beam, allowing in-situ irradiations during observation under controlled sample and diffracting conditions.

Capabilities include continuous recording to provide real-time observation of defect formation and evolution during irradiation and well controlled experimental conditions (constant specimen orientation and area, specimen temperature, ion type, ion energy, dose rate, dose, and applied strain). A radiological facility, the Irradiated Materials Laboratory (IML), located in the same building as the IVEM-Tandem, can receive, handle, prepare, and store radioactive samples.

For more information on the IVEM-Tandem Facility, visit the NSUF website at https://nsuf.inl.gov or the Argonne National Laboratory's facility page at http://www.ne.anl.gov/ivem/.

NUCLEAR FUELS and MATERIAL ONLINE LIBRARY

Kelly Cunningham
Nuclear Fuels and Materials Library Coordinator

The Nuclear Science User Facilities (NSUF) Nuclear Fuels and Materials Library went online in 2016 to establish another pathway for research. The library is a collection of specialized information, nuclear fuel and material specimens from past and ongoing irradiation test campaigns, real-world components retrieved from decommissioned power reactors, and donations from other sources. It includes irradiated and unirradiated samples in a wide range of material types, from steel samples irradiated in fast reactors to ceramic materials irradiated in the Advanced Test Reactor.

The Nuclear Fuels and Materials Library is owned by the U.S. Department of Energy's Office of Nuclear Energy (DOE-NE) and curated by the NSUF. Many of the samples are from previous U.S. Department of Energy (DOE) funded material and fuel development programs. Researchers can propose to analyze these samples in a post-irradiation examination (PIE) only experiment. Samples from the library may be used for proposals for open calls and rapid turnaround experiments.

As the NSUF program continues to grow, so will easily accessible NSUF research tools like the Nuclear Fuels and Materials Library. Expanding the Library's inventory means reaching back to ensure that information and materials are properly archived. The NSUF has prioritized:

- documenting "tribal knowledge" (long stored legacy material information, locations,pedigrees, etc.),
- making materials inventories accessible to researchers, and
- including archived materials to validate past and current analyses.

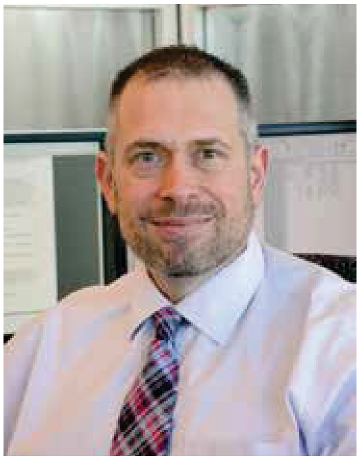
The library has grown to approximately 6,000 specimens, up from approximately 3,500 at its inception. Continued collaboration within the nuclear community will expand the inventory by adding materials in line with DOE-NE's current mission and future NE research needs. DOE-NE periodically issues Requests for Information to supplement the library's offering by asking researchers to provide information on:

- existing nuclear energy research materials and specimens that can, potentially, be added to the Nuclear Fuels and Materials Library, and
- future needs for nuclear energy related material to support ongoing nuclear energy challenges as well as future research advancements in nuclear energy.

The library can be accessed via the NSUF's website at https://nsuf.inl.gov or directly via the Nuclear Energy Infrastructure Database at https://nsuf-infrastructure.inl.gov/browse/materials.

Series of sample holders in a Containment Box, Hot Fuels Examination Facility (HFEF), MFC, INL

ION BEAM CAPABILITIES
NSUF AT WORK



Brenden Heidrich
Capabilities Scientist

When U.S. Department of Energy Office of Nuclear Energy (DOE-NE) leaders needed expert input on how to prioritize the importance of domestic ion beam irradiation capabilities, they called the NSUF – and NSUF ion beam research capabilities experts answered the call.

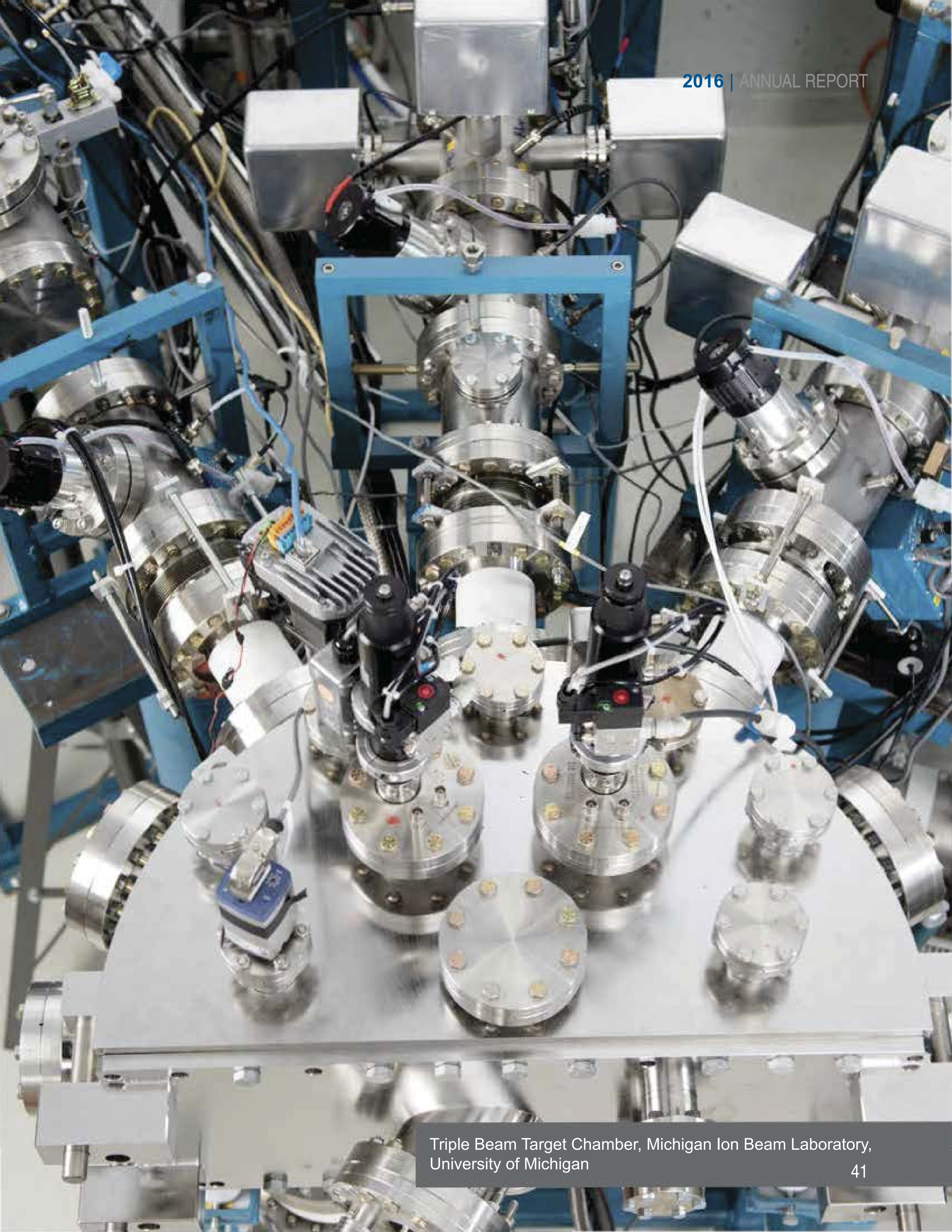
The NSUF, at the request of DOE-NE, held an Ion Beam Workshop in March 2016 in Idaho Falls, Idaho. The workshop was organized at the direction of DOE-NE to develop a set of recommendations for prioritizing domestic ion beam irradiation capabilities available to researchers. Domestic ion beam capabilities are focused on the support of nuclear energy research and development, both of which are key to DOE-NE's missions.

DOE-NE intends to use the input provided by the workshop when deciding how to best use NSUF ion beam capabilities, instruments, and facilities. A cross section of the ion beam community participated in the workshop.

Thirty-three members of the ion beam community attended the invitation-only workshop. Attendees represented 15 operating and proposed ion beam facilities, six DOE-NE research and development (R&D) programs, the Electric Power Research Institute, and the chairs of the NSUF Users Organization and the NSUF Science Review Board. Three members of the sponsoring agency, the Office of Science and Technology Innovation (DOE-NE-4), also attended the workshop.

Consensus Criteria

The workshop participants developed by consensus a list of ten criteria against which to compare the various ion beam facilities. The criteria are tied directly to ion beam capabilities that support DOE-NE's mission and thus will help DOE-NE understand the priorities for future federal decisions.



Triple Beam Target Chamber, Michigan Ion Beam Laboratory, University of Michigan

Michigan Ion Beam Laboratory, University of Michigan

COMBINED CRITERIA

C1 Viability for the capability to extend our understanding toward accurately simulating nuclear irradiation conditions (neutrons or fission fragments)

C2 Ability of the facility to provide a variety of ion irradiations (ion types, energies, multiple beams, etc.)

C3 Ability of the facility to provide a variety of well-controlled target environments and conditions

C4 Ability of the facility to collect and analyze properties of materials and/or perform microstructural characterization data on-site

C5 Ability of the facility to collect and analyze properties of materials and/or perform microstructural characterization data in-situ

C6 Current or potential productivity of the facility (e.g., fewer experiments but high-impact experiments or high volume sample throughput)

C7 Unique capabilities of the facility, including any new technology that the capability has to close technological gaps

C8 Ability of the facility to handle radioactive materials (structural materials and/or fuels) in the beams and elsewhere on-site

C9 Ability of the facility to produce quality-level data that can support licensing as well as verification and validation of modeling and simulation

C10 Ability of the facility to produce results that meet the needs of the DOE-Office of Nuclear Energy (including cross-cutting programs) and the nuclear energy industry

Participants from ion beam users and DOE-NE R&D programs first presented information on capabilities and needs to begin the consensus building process. The ion beam facility representatives then shared their respective presentations. Following this informative discussion, workshop participants buckled down and assessed each ion beam facility against the 10 criteria.

Workshop participants recognized early on that ion beam facilities have individual focus areas and objectives and thus may have significantly different designs. Since form follows function, the differing objectives were delineated by participants.

Ion Beam Facility Categories

Workshop participants reviewed and categorized 15 ion beam facilities.

Proposed Ion Beam Capabilities

Four facilities were proposed to be built in the future to provide expanded ion beam capabilities, including combining ion beam irradiation capability with in-situ characterization with an X-ray source:

- Argonne National Laboratory (ANL) – Extreme Materials Beam Line (XMAT)
- Brookhaven National Laboratory (BNL) – Ion X-Ray Beam (IXB)
- BNL – Ion Irradiation Facilities and Capabilities at the BNL Accelerator Complex – BLIP BLAIRR
- Massachusetts Institute of Technology (MIT) – MIT Nuclear Materials Laboratory

Multipurpose Ion Beam Capabilities

Workshop participants recognized that three facilities had primary focuses outside of materials irradiations supporting DOE-NE's key mission areas:

- The Center for Materials under Extreme Environment Facility at Purdue University focuses on surface science of materials and utilizes much lower energy ions than the others.
- The Edwards Accelerator Laboratory at The Ohio University is primarily engaged in nuclear data measurement and not in the irradiation effects on materials.
- The Idaho Accelerator Laboratory at Idaho State University is a multipurpose facility that supports a wide variety of research endeavors.

These three facilities should not be judged in the same manner as the others.

DOE-NE R&D Ion Beam Capabilities

Beyond this, the remaining eight currently operating facilities all provide vital support to nuclear materials researchers. The individual capabilities of these eight facilities differ based on their particular missions. Three facilities have – or will have soon – in-situ characterization capabilities that combine ion irradiation with a transmission electron microscope (IVEM, I3TEM, and MIBL (pending)).

Eight currently operating ion beam facilities that either already support DOE-NE R&D needs through the NSUF or could eventually support needs through the NSUF:

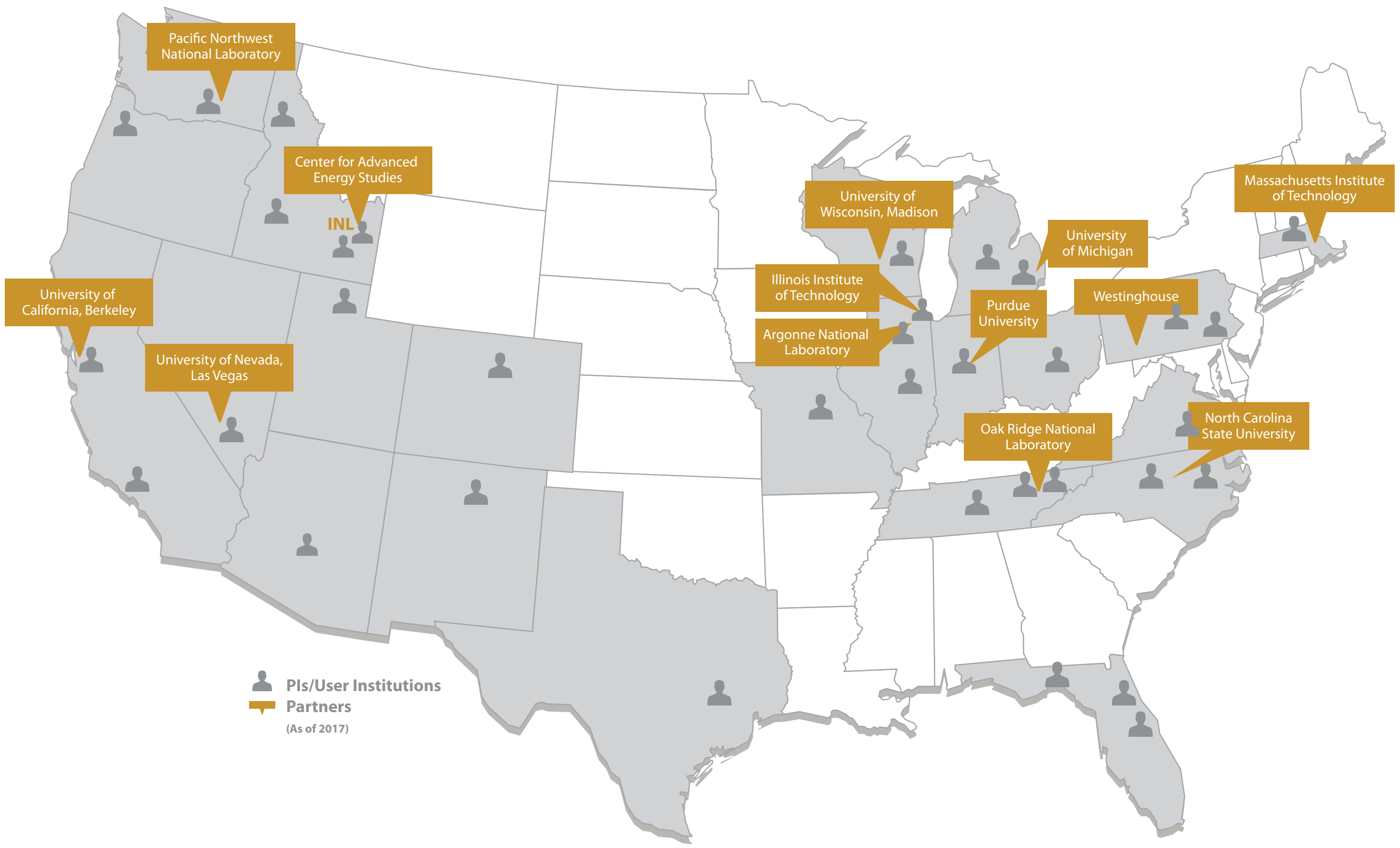
- ANL – Intermediate Voltage Electron Microscope (IVEM)
- Los Alamos National Laboratory – Ion Beam Materials Laboratory
- Lawrence Livermore National Laboratory – Center for Accelerator Mass Spectrometry
- Sandia National Laboratories – In-Situ Ion Irradiation Transmission Electron Microscope (I3TEM)
- Texas A&M University – Ion Beam Laboratory
- University of Michigan – Michigan Ion Beam Laboratory (MIBL)
- University of Tennessee – Ion Beam Materials Laboratory
- University of Wisconsin – Ion Beam Laboratory.

The NSUF will proceed in facilitating DOE-NE's effective use of ion beam capabilities by 1) producing an Ion Beam Utilization Roadmap and 2) evaluating additional ion beam capabilities through the NSUF Partner Facility application process from institutions interested in joining the consortium.

For the former, the NSUF will assemble a voluntary team of ion beam experts whose mandate will be to prepare a report describing current and potential future contributions of ion beam technologies to address the technical and regulatory challenges of the nuclear energy community for the advancement and implementation of nuclear energy technologies that are part of the mission of DOE-NE. The report should establish recommendations and their impacts for DOE-NE and its programs to use at their discretion in establishing future directives and priorities.

For the latter, the NSUF will review applications under the criteria of whether the capability is unique with respect to what is already part of the NSUF or if the capability is in such high demand that additional capability is needed in the NSUF in fulfilling the mission of DOE-NE. Site visits and capability inspections will be part of these evaluations.

DISTRIBUTED PARTNERSHIPS

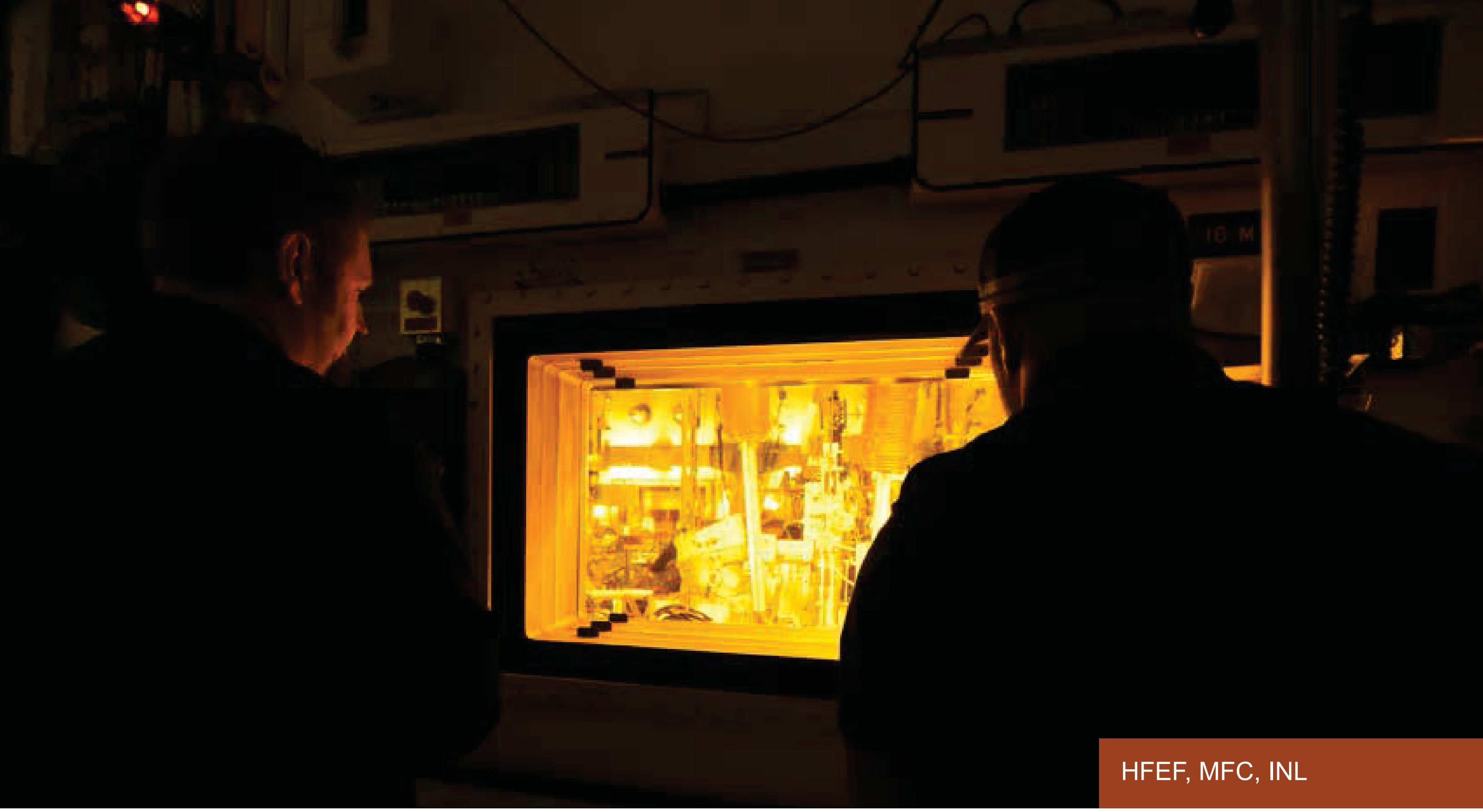


Partner Facilities

Berkeley University of California · Illinois Institute of Technology · Massachusetts Institute of Technology · Westinghouse · Pacific Northwest National Laboratory

Wisconsin University of Wisconsin–Madison · Oak Ridge National Laboratory · UNLV · NC State University · Purdue University · University of Michigan

Argonne National Laboratory · INL Idaho National Laboratory · CAES Center for Advanced Energy Studies



HFEF, MFC, INL

User Institutions

Arizona
Arizona State University

California
University of California, Berkeley
University of California, Santa Barbara

Colorado
Colorado School of Mines

Florida
Florida State University
University of Central Florida
University of Florida

Idaho
Boise State University
Idaho National Laboratory
Idaho State University
University of Idaho

Illinois
Argonne National Laboratory
Illinois Institute of Technology
University of Illinois

Massachusetts
Massachusetts Institute of Technology

Michigan
Michigan State University
University of Michigan

Missouri
University of Missouri

Nevada
University of Nevada – Las Vegas

New Mexico
Los Alamos National Laboratory

North Carolina
GE Hitachi Nuclear Energy
North Carolina State University

Ohio
The Ohio State University

Oregon
Oregon State University

Pennsylvania
Drexel University

Tennessee
Oak Ridge National Laboratory
University of Tennessee
Vanderbilt University

Texas
Texas A&M University

Utah
Utah State University

Virginia
Virginia Commonwealth University

Washington
Pacific Northwest National Laboratory

Wisconsin
University of Wisconsin

Australia
Australian Nuclear Science and Technology Organization

United Kingdom
Oxford University
University of Liverpool
University of Manchester

NRAD Reactor Pool, MFC, INL

NSUF AWARDED PROJECTS

This section contains reports on projects awarded through the NSUF and completed in FY 2016.

Flashback: NSUF History

An Advanced Test Reactor National Scientific User Facility Program could serve a key role in reestablishing the U.S. scientific and nuclear industrial base that is needed for building a new generation of nuclear power plants in the United States and in ensuring the safety of the existing nuclear power plants. Establishing the Advanced Test Reactor as a National Scientific User Facility will help reassert U.S. leadership in nuclear science and technology and would expand the scientific foundation for fuels and materials to improve nuclear energy systems. It would also align with the President's American Competitive Initiative and strengthen nuclear engineering education in the United States.

– U.S. Department of Energy Memo: Designation of INL ATR as a National Scientific User Facility

April 13, 2007

Characterization of CANDU Core Internals via Small Scale Mechanical Testing

Peter Hosemann – University of California, Berkeley – peterh@berkeley.edu

The application of the Focused Ion Beam (FIB) to extract micro-scale samples of in-service components with pre-selected microstructures (i.e., individual grain boundaries) is a powerful technique that allows for insight into real-time mechanical testing observation of failure mechanisms, and the FIB research conducted at the MFC and the University of California, Berkeley moves this method to the forefront of the nuclear scientific community.

CANada Deuterium Uranium (CANDU) Inconel X-750 spacers incur higher doses and He/dpa ratios than light water reactor (LWR) materials. They separate hot pressure tubes from cold calandria tubes, preventing low-pressure moderator from contacting hot pressure tubes. Segments at 330°C exhibit lower strength and ductility than 200°C segments. Lower temperature parts support fuel channels, preventing contact between hot and cold tubes. Weakening and embrittlement occur after 2/3 of the tubes' expected lifetime. Inspected springs exhibited intergranular failure upon handling. Quantification of changes in failure stresses as compared to virgin materials is needed. Embrittlement occurs in Ni alloys with irradiation temperatures > 500°C, but there are few instances of low temperature effects. Failure mechanisms and degradation rates are essential for long term prediction of mechanical failure through appropriate models. Micro-scale testing will provide insight into the temperature dependence of embrittlement at operating temperatures that can then be applied to components subject to He embrittlement in all reactors.

Project Description

The objective of this research was to obtain quantitative mechanical properties of Inconel X-750 CANDU core components at two different irradiation temperatures and doses. A new in-situ scanning electron microscope (SEM) small-scale mechanical test, namely lift-out three-point bending, was developed and executed on active CANDU core components. Given the difficulties in manufacturing and testing multiple specimens to obtain good statistics from active bulk spring coils, the

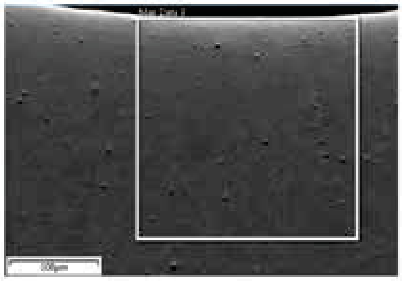
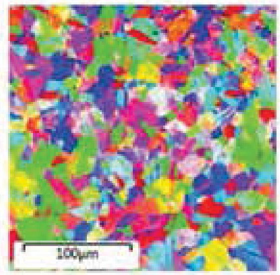
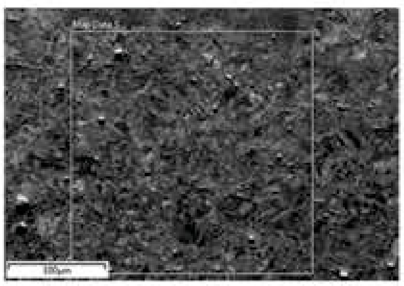
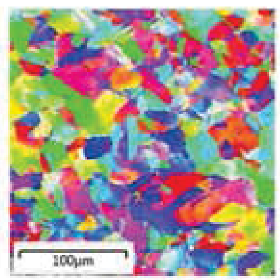
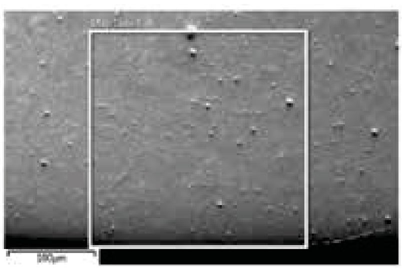
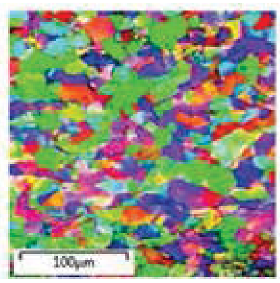
Inconel X-750 Spring Location	e^- Image	EBSD Orientation Map	$d_{avg\text{-}radial}$	$d_{avg\text{-}tangential}$
Inner Edge			8.4 ± 0.9	8.9 ± 1.0
Center			8.7 ± 0.9	10.4 ± 1.0
Outer Edge			6.9 ± 0.9	8.1 ± 1.0

Figure 1. Electron Backscatter Diffraction (EBSD) scans of 250 µm x 250 µm squares of the inner edge, center, and outer edge regions of an Inconel X-750 flat spring. EBSD orientation map grain analysis produces an average overall grain size of 8.5 ± 2.9 µm and indicates grain elongation in the tangential direction.

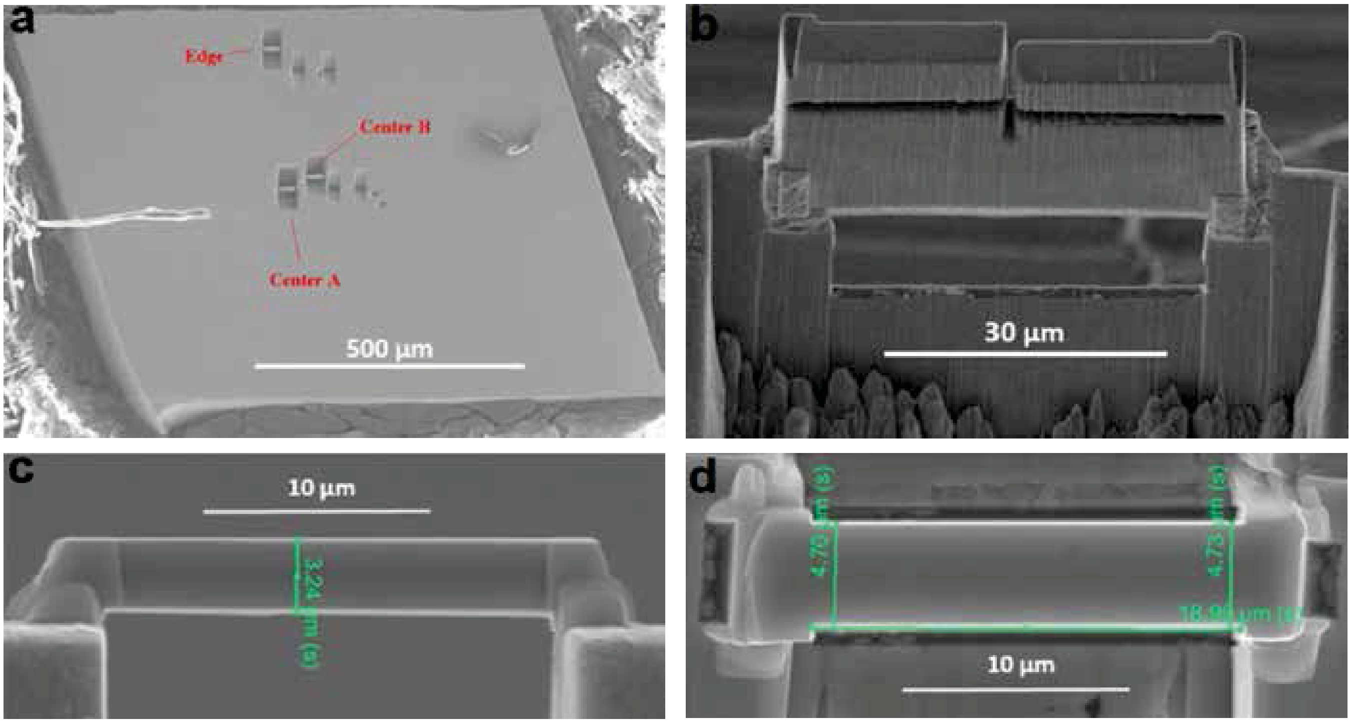


Figure 2. (a) cross-section of an Inconel X-750 spring showing focused ion beam (FIB) milled foils of material taken from both edge and center regions (b) resulting bending beam structures cut into the large lift-out foil which has already been removed from the bulk spring (c) side view of a finished three-point bend specimen (d) top view of a completed three-point bend specimen.

reproduceability of testing that provides quantitative yield-strength values and a real-time look at deformation processes makes this an exciting technique. This specific project was to conduct micro-scale mechanical testing on annulus spacers irradiated at 180 and 300°C in service for approximately 14 and 19 equivalent full-power years to quantify differences in mechanical properties. This research supports efforts to make the micro-scale investigation and testing of nuclear core structural components more reproduceable, quantitative, and cost-effective by minimizing the use of hot cells and developing new in-situ small-scale mechanical testing techniques. Because these CANDU core components are nickel superalloys, they incur more pronounced radiation damage effects (increased displacement damage and helium and hydrogen content) and can be used as a predictive model for future effects that will occur in LWR structural materials.

Accomplishments

Large foils of active Inconel X-750 spacers were fabricated on the focused ion beam at the Materials Fuels Complex (MFC) in Idaho under the direction of Colin Judge and Cameron Howard, and shipped to the University of California Berkeley nuclear materials laboratory. This research was conducted primarily by Cameron Howard and facilitated in large part by staff at MFC, including microscopist James W. Madden, staff scientist Brandon D. Miller, and project director Collin J. Knight. These foils were subsequently sectioned into individual specimens and an in-situ lift-out three-point bending test technique was developed

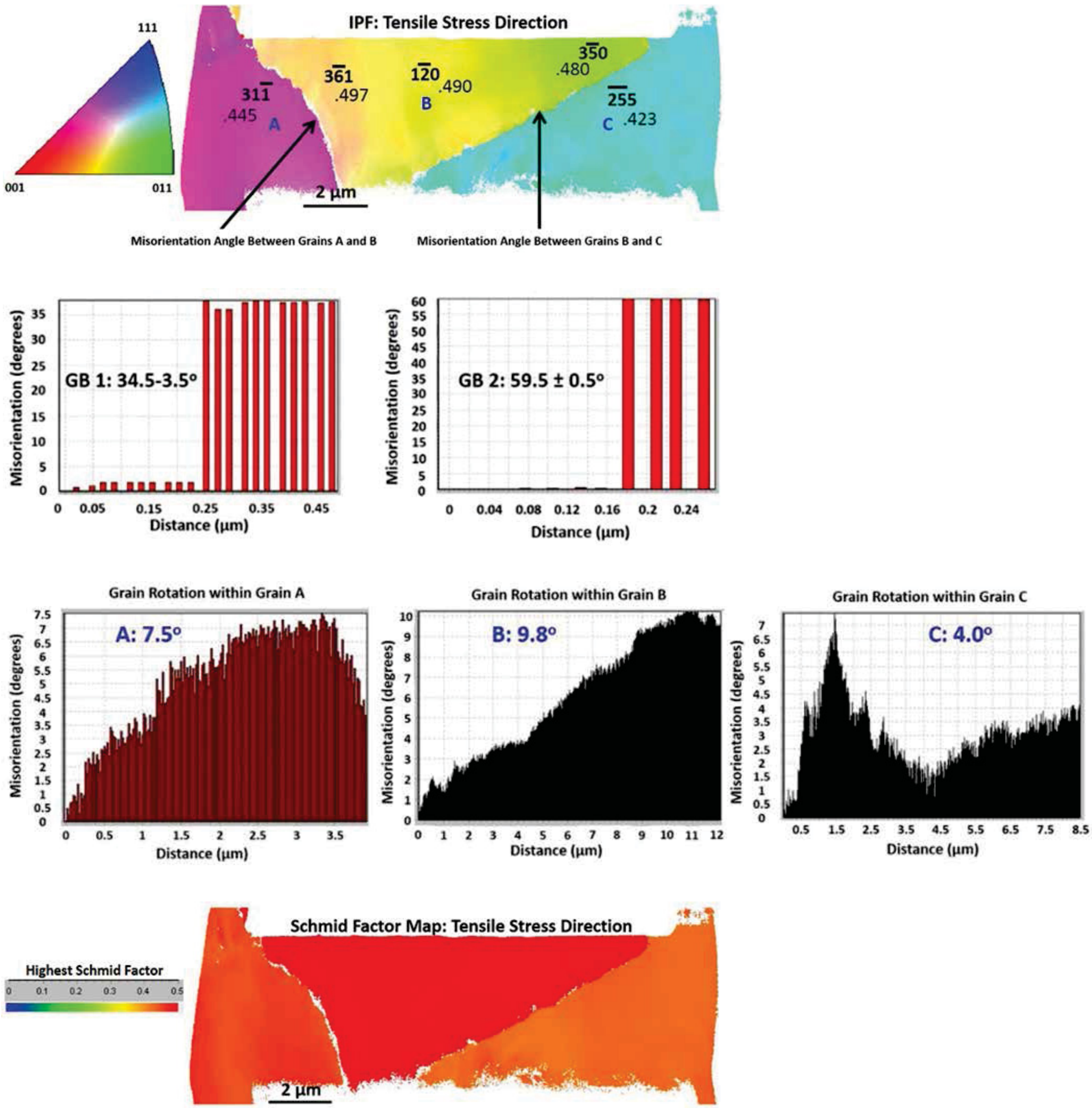


Figure 3. Pre-test Electron Backscattered Diffraction (EBSD) on the top surface of a representative three-point bend specimen including an Inverse Pole Figure (IPF) grain orientation map in the tensile stress direction on the outer fiber of the specimen, hkl orientation of individual grains, misorientation angles between two grains, grain rotation within each grain, and Schmid factor map indicating the likelihood of deformation slip along the most preferred slip system within each grain.

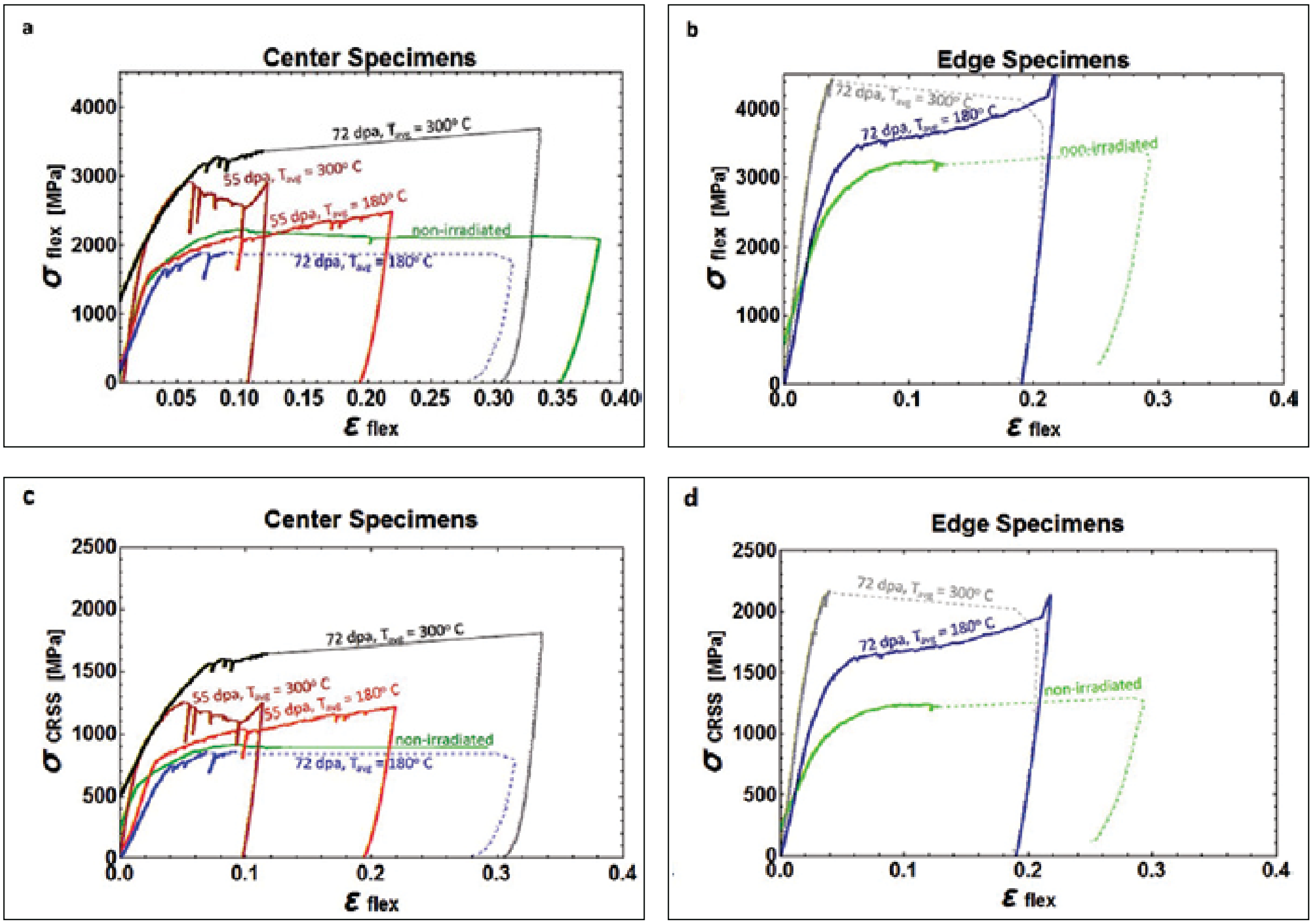


Figure 4. Representative flexural stress-strain curves calculated for the midpoint at the outer fiber of the (a) center and (b) edge specimens according to three-point bend theory for each irradiation condition. Loading curves plotted in terms of critical resolved shear stress (CRSS) can be seen for the center specimens in (c) and edge specimens in (d).

and applied by Cameron Howard and Steven Scott Parker to components irradiated at 180 and 300°C in service for approximately 14 and 19 equivalent-full-power years as well as to non-irradiated control specimens.

This work was performed in the FEI Quanta Dual Beam SEM/FIB in the University of California, Berkeley nuclear materials laboratory. In addition, investigations of pre-existing cold-work effects created during spring coil production and how these may change over the course of in-service time and irradiation temperature, as suggested by Malcolm Griffiths and Steven Xu, were performed for samples of all irradiation conditions by extracting and testing material from both the edge and center regions of the spacers.

Future Activities

A manuscript for the Journal of Nuclear Materials has been drafted with the intention of submission and publication in the near future on the novel in-situ three-point bending technique and the results found for these core components. Newly developed push-to-pull tensile testing to assess the grain boundary strengths of these components remains ongoing and will serve as the remainder of the Ph. D. thesis for Cameron Howard.

Publications

[1.] C. Howard, et al., "Mechanical Characterization of In Service Inconel X-750 Annulus Spacers," 146th Annual Meeting of the Minerals, Metals, and Materials Society, February 2017.

**See additional publications from other years in the Media Library on the NSUF website.*

Access to the MFC facility has provided unprecedented capabilities for large-scale work on in-service nuclear structural materials

— Cameron Howard, Ph.D. Researcher, Department of Nuclear Engineering, Nuclear Materials Group, University of California, Berkeley

Distributed Partnership at a Glance

NSUF and Partners	Facilities and Capabilities
Idaho National Laboratory	Electron Microscopy Laboratory
University of California, Berkeley	Nuclear Materials Laboratory
Collaborators	
Canadian Nuclear Laboratories	Colin Judge (Collaborator)
Kinectrics Inc.	Steven Xu (Collaborator)
Queen's University	Malcolm Griffiths (Collaborator)
University of California, Berkeley	Peter Hosemann (principal investigator), Cameron Howard (Co-Principal Investigator), Steven Scott Parker (Collaborator)

Atom Probe Characterization of Phase Separation During Age Hardening of a U-6wt.%Nb Alloy

Clarissa Yablinsky – Los Alamos National Laboratory – rizz@lanl.gov

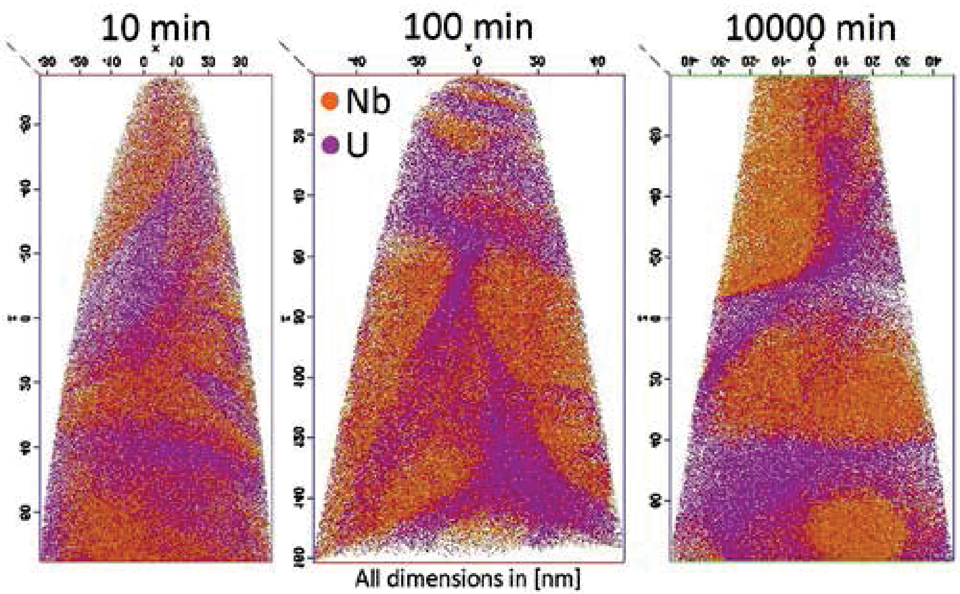


Figure 1. Tip reconstructions for aging times of 10 minutes at 500°C, 100 minutes and 500°C, and 10000 minutes at 500°C. The non-lamellar precipitate structures all contain interconnected alpha-U precipitates, which coarsen with age, of varying widths and orientations.

Understanding decomposition mechanisms in metallic fuel will help with performance prediction over the lifetime of a reactor.

Project Description

The technical objective of this study was to probe an aged material using an instrument capable of giving information on structure and chemistry. The two-phase microstructure of the U-6Nb alloy undergoes aging decomposition during heat treatments at temperatures used in reactors and is a simple alloy to probe phase transformations, kinetics, and age-hardening mechanisms. Metallic uranium fuel will be necessary in advanced reactors to meet the fissile-atom density requirements of reactors while maintaining low enrichment that policy demands. Understanding decomposition mechanisms and underlying kinetics within the operating temperature regime is necessary for performance prediction for reliability, safety, and life extension for new reactors.

Accomplishments

Clarissa Yablinsky was able to accomplish the microscopy and atom probe for one of the four samples proposed, rounding out a set of 3 aging conditions: 10 min at 500°C (completed this fiscal year [FY]), 100 min at 500°C, and 10000 minutes at 500°C (completed in previous year outside of Nuclear Science User Facilities [NSUF]). General trends were observed with aging: the Nb segregation to the matrix was greater with increased aging and the U precipitate increased in concentration as the Nb segregated. This increase in partitioning relates to the energy released during the decomposition; thus, the same trend is seen for the extent of partitioning, delta C, where delta C increases with aging time. The Center for Advanced Energy Studies (CAES) staff was instrumental in

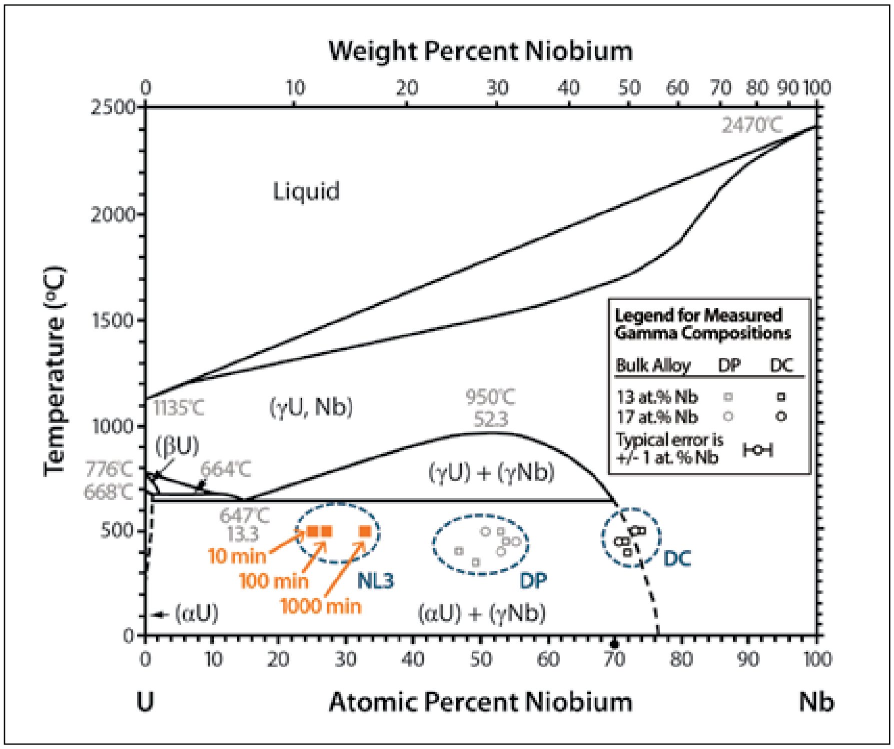


Figure 2. The data added at 500°C in the NL3 regime is presented with orange squares. The evolution in Nb partitioning throughtout the aging, moving from an NL3 decomposition mechanism, to the DP, and finally to DC, shows the chemical evolution of the metallic fuel with time.

performing the microscopy due to their radiological training and careful approach to sample handling.

Future Activities

Because of shipping issues, we were unable to finish the other three samples in FY 2015. After the complete set is finished, we will do further analysis of the kinetics and structure and begin writing a paper that incorporates the results with previous hardening and decomposition mechanism data.

The CAES MaCS facility is well run and was an excellent place to do microscopy and atom probe for my uranium project.

— Clarissa Yablinsky, Scientist

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Los Alamos National Laboratory	Clarissa Yablinsky (principal investigator), Robert Hackenberg (collaborator), Amy Clarke (collaborator)

Si-Ni-Mn Clustering in Irradiated Fe-9Cr Oxide Dispersion Strengthend Alloy

Janelle P. Wharry – Purdue University – jwharry@purdue.edu

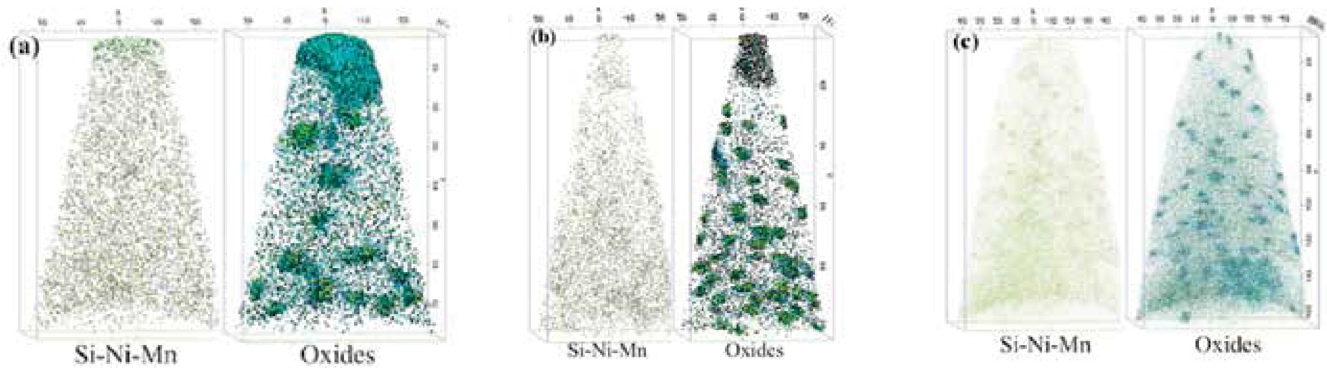


Figure 1. Atom probe tomography (APT) reconstructed tips showing Si-Ni-Mn clustering on oxide nanoparticles in model Fe-9Cr irradiated with (a) 5 MeV Fe++, 100 dpa, 400°C, (b) 2 MeV protons, 3 dpa, 500°C, and (c) neutrons in ATR, 3 dpa, 500°C.

Oxide dispersion strengthened (ODS) alloys are nanofeatured materials, having grains of diameter ~250 nm and containing a dispersion of oxide nanoparticles. The fine grains and oxide nanoparticles provide the alloy with high strength at elevated temperatures and dimensional stability under irradiation, making ODS alloys leading candidates for structural components in fusion and advanced fission reactors. Most post-irradiation evaluation (PIE) experiments of ODS alloys have focused on evaluating the stability of the oxide nanoparticles and several researchers have observed instability of these nanoparticles under irradiation at moderate temperatures. However, there have been no specific investigations into the clustering of Si, Ni, and Mn in ODS alloys. These minor elements have been found to cluster readily in other b.c.c. Fe-Cr alloys, including commercial ferritic/martensitic (F/M) steels. Since ODS steels are based upon the b.c.c. Fe-Cr alloy system, it is unsurprising that Si, Ni, and Mn would also cluster in ODS alloys.

Project Description

The objective of this project is to understand the role of Si, Ni, and Mn clustering in irradiated ODS alloys. Specifically, we test the hypothesis that there is a co-evolution of Si-Ni-Mn rich clusters (which nucleate under irradiation) and pre-exisitng oxide nanoclusters under irradiation. In this project, we first conducted proton and Fe2+ self-ion irradiations to several doses between 1 and 100 displacements per atom (dpa) at 500°C on a model Fe-9Cr ODS alloy. We also access a 3 dpa, 500°C neutron irradiated specimen of the same alloy heat through the NSUF Nuclear Fuels and Materials Library. We carried out microstructure characterization by atom probe tomography (APT) for the nanoscale cluster analysis, along with complementary transmission electron microscopy (TEM) to characterize dislocations, loops, carbide precipitates, and voids. All of these experiments were conducted at the Microscopy and Characterization Suite (MaCS) at the Center for Advanced Energy Studies (CAES).

This project will contribute to the scientific community in numerous ways. First, this project will help us gain a deeper understanding of the mechanisms of Si-Ni-Mn clustering in b.c.c. Fe-Cr alloys. More specifically, this project will help us understand the role of Si-Ni-Mn clustering in the irradiation stability of oxide nanoparticles in ODS steels. Stability of these oxide nanoparticles is critical to the long-term material integrity of ODS alloys in cladding and structural components in fission and fusion reactors. Second, this project presents a rare and unique opportunity to compare neutron, proton, and self-ion irradiation effects in identical heats of a model Fe-9Cr ODS alloy at identical irradiation temperatures and doses. This comparison offers great opportunity to help us understand fundamental differences in the creation of irradiation damage between neutrons, protons, and self-ions. Third, results of this project will be relevant to all ODS and other nanostructured alloys based on the b.c.c Fe-Cr matrix, which are of growing interest to the U.S. Department of Energy Office of Nuclear Energy due to their enhanced radiation resistance. Last, this project will enable a graduate student to utilize state-of-the-art ion beam irradiation techniques toward developing his thesis.

Accomplishments

The atomic level resolution of APT with Interactive Analysis and Visualization Software (IVAS) cluster analysis enabled both the characterization of the irradiation induced dissolution of oxide nanoclusters and the irradiation induced formation of Si-, Ni-, and Mn-rich clusters. The three-dimensional reconstructions of the neutron-, proton-, and self-ion-irradiated specimens all exhibit clustering of Ti, O, and Y atoms along with TiO, YO, FeO, and CrO compounds at coincident locations in the matrix. There is also visual evidence of clustering among the Si, Mn, and Ni atoms at the same locations. Multiple localized electron atom probe (LEAP) tips were analyzed from each of the as-received and irradiated specimens. Representative atom distribution maps, as created by APT analysis and 3D reconstruction, are shown in the attached figures. Quantitative chemical analysis, enabled by the APT IVAS software, confirms the visual evidence of Si, Mn, and Ni clustering. For example, the fraction of all detected Si, Mn, and Ni atoms that are found in clusters (i.e. percent clustered) increases with irradiation. In addition, the in-cluster composition of Si, Mn, and Ni increase with all types of irradiation, while the matrix composition of Si decreases, further affirming that these species cluster during irradiation.

Oxide nanoparticles co-evolve with Si, Mn, and Ni radiation induced segregation in ODS alloys.

The clustering of Si, Mn, and Ni is a notable irradiation induced chemistry change. The formation of similar clusters containing Si, Mn, and Ni is often reported in F/M steels such as T91, HCM12, and HT9 and in austenitic stainless steels, but rarely in ODS steels. Several cluster formation mechanisms have been proposed:

- Si, Mn, and Ni form oxide phases such as SiO_2, Mn_3O_4, or NiO and nucleate in conjunction with the existing oxide phases.
- Si, Mn, and Ni form silicides such as Mn_4Si_7 or Ni_3Si_2 and nucleate as their own precipitates, independent of the existing oxides.
- Si, Mn, and Ni diffuse towards sinks (such as oxides) due to radiation induced segregation (RIS).

The first possible mechanism must consider the enthalpy of formation of the Fe, Cr, Y, Ti, Si, Mn, and Ni oxides relevant for 9-Cr ODS. All of these elements have a high affinity for oxygen, with the Si and Mn having enthalpy of oxide formation on the same order as that of Fe, Cr, and Ti. Yet because Si, Mn, and Ni oxides or clusters are not present in the as-received condition, their clustering is irradiation induced. In addition, the oxide formation mechanism does not explain why Si, Mn, and Ni also cluster upon irradiation in other F/M and austenitic stainless steels that do not contain pre-existing oxide compounds. Therefore, this is not likely to be the primary mechanism of Si, Mn, and Ni clustering.

The second possibility considered is that the Mn and Ni form silicide compounds and precipitate out of solution. Although all of these silicides have a negative enthalpy of formation, these enthalpies are less favorable than those of the oxides considered in the first possible mechanism. Furthermore, if silicide formation were the primary mechanism for Si, Mn, and Ni clustering in ODS and F/M steels, it would be expected that Mo would also cluster in other F/M steels due to the negative Mo-silicide formation enthalpy, yet this has not been observed in irradiated F/M alloys. Thus, silicide formation is not likely the primary mechanism of Si, Mn, and Ni clustering in ODS and F/M steels

Finally, the possibility of RIS is considered as the driving mechanism for Si, Mn, and Ni clustering. Si, Mn, and Ni are found in higher concentrations within the oxides following irradiation. These same species are known to segregate toward grain boundaries in commercial F/M and austenitic stainless steels. Thus, the RIS mechanisms reported in literature appear to be consistent with the solute enrichments at oxides observed in this study, suggesting that the clustering of Si, Mn, and Ni is the likely result of RIS toward existing point-defect sinks within the matrix, such as oxide nanoclusters.

This work was performed primarily by Matthew Swenson and Corey Dolph, under the research advisorship of Janelle Wharry. At CAES, the students were assisted by Yaqiao Wu, Joanna Taylor, Alyssa Bateman, and Jatuporn

Burns. At the Michigan Ion Beam Laboratory at the University of Michigan, the students were assisted by Ovidiu Toader , Fabian Naab, and the graduate students employed there.

Future Activities

Our future work involves developing a rate theory model to predict the irradiation evolution of oxide and Si-Mn-Ni nanoclusters. This model will consider the RIS mechanism as the driver of Si, Mn, and Ni clustering on existing oxides, and will predict the long-term co-evolution of the Si-Mn-Ni-enriched oxide clusters.

Publications

[1.] M. J. Swenson and J. P. Wharry. "Standardizing atom probe cluster analysis methods: voltage and reconstruction considerations." Submitted manuscript.

[2.] J. P. Wharry, M. J. Swenson, and K. H. Yano. "A review of the irradiation evolution of dispersed oxide nanoparticles in b.c.c. Fe-Cr alloys: current understanding and future directions." Submitted manuscript.

[3.] M. J. Swenson and J. P. Wharry. "Collected data set size considerations for atom probe cluster analysis." *Microscopy & Microanalysis* 22.S3 (2016) 690.

[4.] C. K. Dolph, D. J. DaSilva*, M. J. Swenson, and J. P. Wharry. "Plastic zone size for nanoindentation of irradiated Fe-9wt% Cr ODS alloy." *Journal of Nuclear Materials* 481 (2016) 33. *undergraduate

[5.] M. J. Swenson, C. K. Dolph, and J. P. Wharry. "The effects of oxide evolution on mechanical properties in irradiated 9wt% Cr ODS alloy." *Journal of Nuclear Materials* 479 (2016) 426.

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Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor Facility
University of Michigan	Michigan Ion Beam Laboratory
Collaborators	
Boise State University	Janelle Wharry (principal investigator), Matthew Swenson (collaborator), Corey Dolph (collaborator)

Microstructural and Nanomechanical Characterization of a Lanthana-Bearing Nanostructured Ferritic Steel Irradiated with High Dose Iron Ions

Indrajit Charit – University of Idaho – icharit@uidaho.edu

Development of the new 14LMT alloy with excellent radiation resistant property may pave the way for using high performance fuel cladding and structural materials for advanced nuclear reactors.

Advanced reactors need high performance materials to serve under harsh service conditions such as higher temperature, higher radiation doses, and extremely corrosive environment. Nanostructured ferritic steels (NFS) are such a class of materials, produced via mechanical alloying (MA) of the elemental (or pre-alloyed) metallic powder, typically incorporating nanosized yttria powder, followed by a traditional consolidation process such as hot extrusion or hot isostatic pressing (HIP). They are potential materials for advanced fuel cladding and structural materials applications. The performance of NFS is largely determined by ultra-high-number density of nano-sized oxide particles, which are dispersed throughout the microstructure and stable at high temperatures. A new NFS, known as 14LMT (Fe-14Cr-1Ti-0.3Mo-0.5La2O3, wt.%), was recently developed by the principal investigator's group and his project collaborators. The 14LMT alloy uses lanthana in place of traditionally used Y_2O_3. Spark plasma sintering (SPS) was used to consolidate the mechanically alloyed powder.

Project Description

The project was geared toward investigating 14LMT samples exposed to higher self-ion irradiation doses (up to 400 dpa) and understanding the effects of radiation damage doses. No irradiation task was included in the scope of this Rapid Turnaround Experiment (RTE) project because the ion irradiation work has already been performed at the Texas A&M Ion Beam Laboratory led by Professor Lin Shao. The ion irradiation parameters used are summarized as follows. Ion type: Fe2+ ion; ion energy: 4 MeV; irradiation mode: defocused; dose: 100, 200, 300, and 400 dpa; dose rate: around 10−4 dpa/second; irradiation temperatures: 748 K. Furthermore, the initial microstructure of the 14LMT alloy has been thoroughly characterized by transmission electron microscopy (TEM) and atom probe tomography (APT) in CAES MaCS.

For the post-irradiation examination portion of the project, four samples, each irradiated at 748 K, were examined. The irradiated specimens are studied for microstructural characteristics and mechanical properties.

Task 1. Preparation of APT and TEM specimens by focused ion beam (FIB) at MaCS.

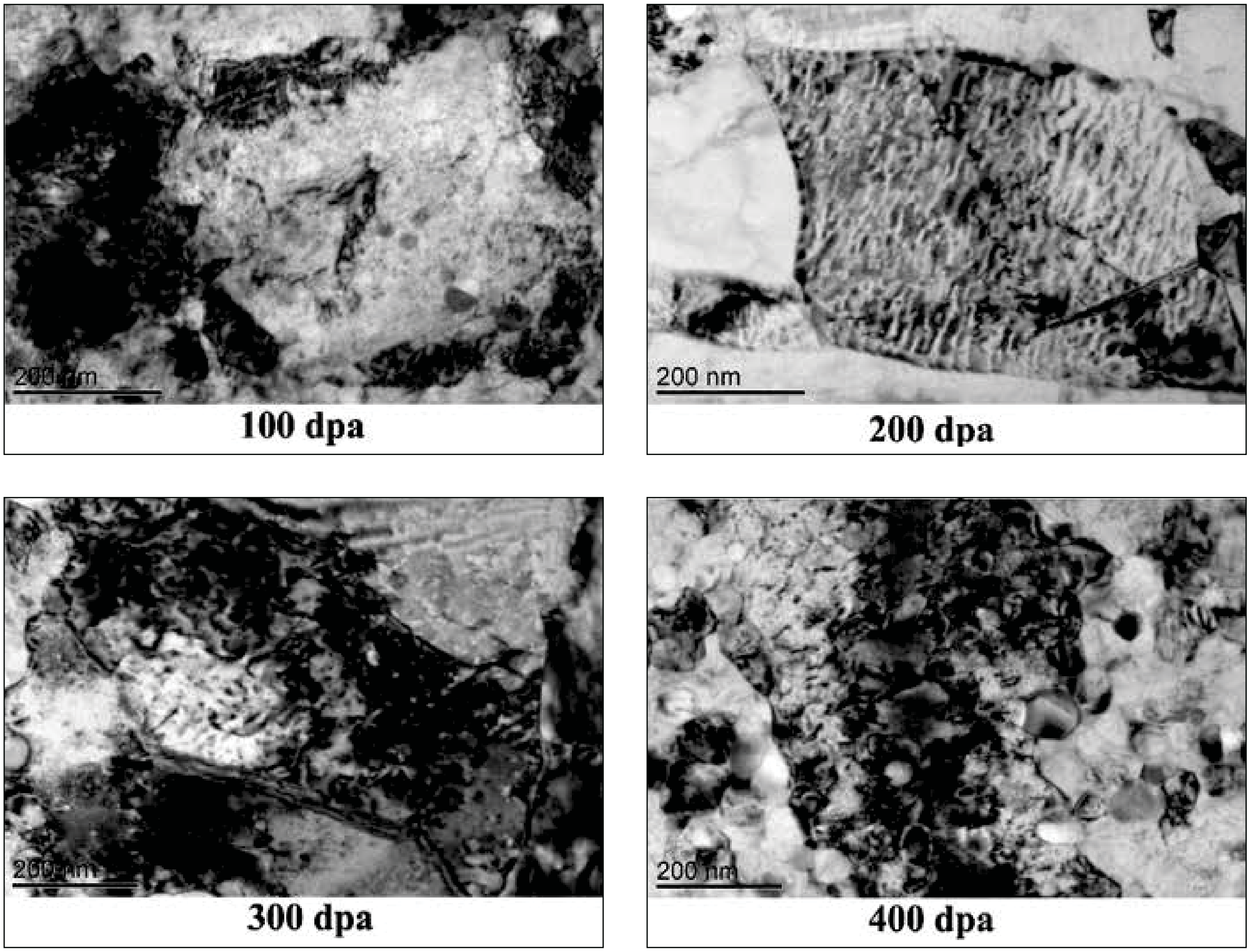

Figure 1. Transmission electron microscopy images of 14LMT alloy irradiated by ferrous ions to different ion-irradiation doses at 748 K.

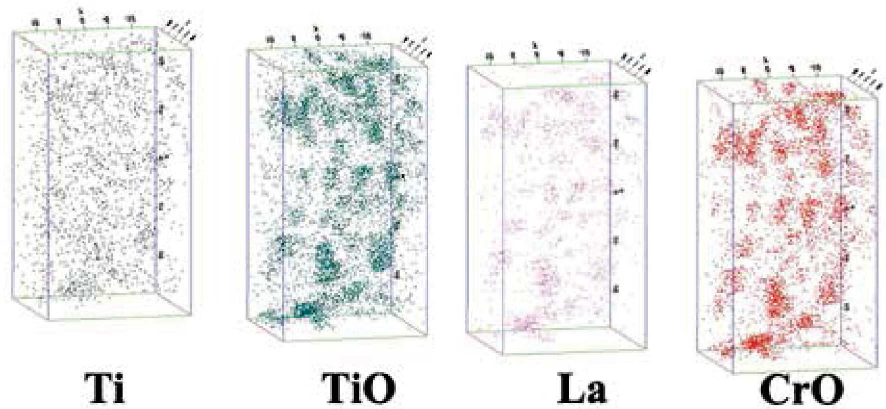

Figure 2. *Three-dimensional reconstructed atom probe tomography maps of 14LMT alloy specimen irradiated by ferrous ions to 300 dpa at 748 K.*

Task 2. Microstructural characterization of the prepared specimens. TEM studies were carried out on the prepared TEM specimens using the Tecnai TF30-FEG S-Twin STEM to study the ion irradiated microstructure with particular attention to both the irradiation induced damage formation and the morphology and chemical analysis of the nanometric particles. The APT studies, conducted using the Cameca LEAP 4000X HR, provided information on any change in their size, number density, and composition as a function of dpa and irradiation temperature.

Task 3. Nano-indentation testing. Heavy ion irradiation led to only about a micron thick surface damage layer. Hence, the nano-indentation technique served as an effective method to characterize mechanical property, such as elastic modulus and hardness of the ion irradiated damage layer. A Hysitron TI-950 Tribo-indenter available in MaCS was used for these experiments.

Accomplishments

The goal of the project was to examine the microstructure and mechanical properties of the 14LMT alloy irradiated with different radiation damage doses (100–400 dpa). Ion irradiation experiments on the 14LMT specimens were carried out by Dr. Lin Shao and his graduate student, Lloyd M. Price, at the Texas A&M University. Jatuporn Burns helped with the preparation of TEM and APT specimens using the FIB available at CAES-MaCS. Dr. Yaqiao Wu helped the graduate students (Somayeh Pasebani and Ankan Guria) perform the TEM experiments and APT analyses of the irradiated specimens of 14MLT. This research was primarily conducted by graduate students Somayeh Pasebani and Ankan Guria. Joanna Taylor was instrumental in facilitating our access to MaCS.

Future Activities

All the experimental activities have been completed. Some data analyses are still in progress.

Publications

[1.] S. Pasebani, I. Charit, Y. Wu, J. Burns, K. N. Allahar, D. P. Butt, J. I. Cole, and S. F. Alsagabi, "Lanthana-Bearing Nanostructured Ferritic Steels via Spark Plasma Sintering," *Journal of Nuclear Materials* 470 (2016), pp. 297–306.

[2.] S. Pasebani, I. Charit, D. P. Butt, J. I. Cole, Y. Q. Wu, J. Burns, "Sintering Behavior of Lanthana-Bearing Nanostructured Ferritic Steel Consolidated via Spark Plasma Sintering," *Advanced Engineering Materials* 18.2 (2016), pp. 324–332.

[3.] S. Pasebani*, A. Guria*, J. Burns, Y. Wu, I. Charit, D. P. Butt, J. L. Cole, L. Shao and L. Price, "Microstructural and Nanomechanical Characteristics of an Ion-Irradiated Lanthana-Bearing Nanostructured Ferritic Steel," Accelerated Materials Evaluation for Nuclear Application Utilizing Test Reactors, Ion Beam Facilities and Integrated Modeling, *TMS Annual Meeting, held Feb. 14–18, 2016 at Nashville, TN.*

**See additional publications from other years in the Media Library on the NSUF website.*

It was a wonderful opportunity for us to use the advanced tools/techniques available at MaCS to achieve our project objectives.

— Indrajit Charit, Associate Professor, Department of Chemical and Materials Engineering, University of Idaho

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Boise State University	Jatuporn Burns (co-principal investigator), Yaqiao Wu (collaborator)
Texas A&M University	Lin Shao (co-principal investigator)
University of Idaho	Indrajit Charit (principal investigator), Ankan Guria (team member - student), Somayeh Pasebani (team member - student)

Irradiation Effects on Structure and Properties of LWR Concrete

Chris Wetteland – University of Tennessee – cjw@utk.edu

Figure 1. Optical microscope image at 50, 100, and 150× magnification shows discoloration and cracking in the nanoconcrete surface. This flux used in this irradiation was 5 × 10^{13} protons/cm^2 and resulted in significant volatilization from the surface.

Accelerated aging experiments in concrete may offer insight into the longevity of this crucial reactor building material.

The long-term stability and performance of Portland cement concrete in nuclear power plants is of concern as little operational or experimental data exist to aid regulators in extending operating licenses. More complete knowledge of performance in radiation environments will determine concrete's role in setting the upper limits for lifetime extensions. In the awarded project, concrete, aggregate, and paste samples were irradiated with energetic protons to simulate radiation damage. Volatile loss during irradiation was monitored with a mass spectrometer to determine the ideal conditions to irradiate the concrete and concrete components. Samples were examined with optical and electron microsope post irradiation to determine the degree of surface damage. Collected results were used to determine possible radiation induced degradation in the current fleet, as well as aid in developing radiation tolerant concrete recipes for future plants and storage containers. The experiments further establish considerations for accelerated irradiation studies of concrete, which may be required due to the hydrous nature of the material.

Project Description

The technical objectives of the research are to understand the radiation tolerance of concrete and how to perform more-detailed accelerated-irradiation experiments in the future. Most accelerated-aging experiments using accelerators require the target materials to be stable in a vacuum environment; this is true for nearly all metal and ceramic materials. However, due to the hydrous nature of concrete, it requires extensive pump-down times prior to performing any irradiations. The hydrous nature further complicates experiments because using the most modest of beam fluxes can result in vacuum pressure changes two orders of magnitude and greater. Changes

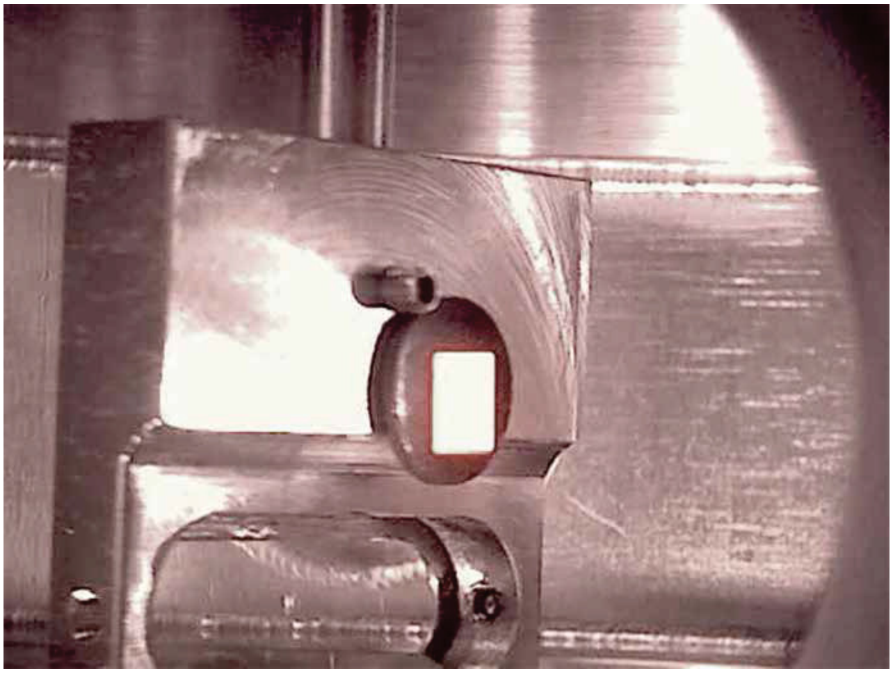

Figure 2. Irradiation of a limestone disk using 2 MeV protons. Limestone (calcite) exhibits a strong luminescence in the visible spectrum during proton irradiation.

on this magnitude can cause damage to internal accelerator and vacuum components and trigger safety valves during an experiment. Because of these restraints, experiments were conducted to determine the optimal experimental conditions, including sample mounting, vacuum requirements, and beam currents, to perform accelerated aging experiments.

As the U.S. reactor fleet ages and receives extensions for operating outside the intended lifespan, it is critical to understand how the concrete structural components respond to long-term radiation exposure. The research in this Nuclear Science User Facilities (NSUF) sponsored study is the first step in qualifying concrete to perform beyond its intended lifetime. As future researchers examine radiation effects in concrete and its individual components (aggregate and cement), these preliminary experiments will be vital in designing accelerated aging studies. Furthermore, these types of studies will aid in the materials selection for future reactors. This is particularly true in the selection of aggregate products in order to limit potential radiation accelerated alkali–silica reaction (ASR).

Accomplishments

Concrete irradiations are extremely difficult. The difficulties arise in limiting damage to the concrete arising from conditions that are outside the normal equilibrium conditions in which the structure resides, e.g., atmospheric pressure.

The series of irradiation experiments we performed on concrete and its aggregates are some of the first of their kind. This is the first step in performing accelerated aging studies in this complex materials system.

— Chris Wetteland, Lecturer, University of Tennessee

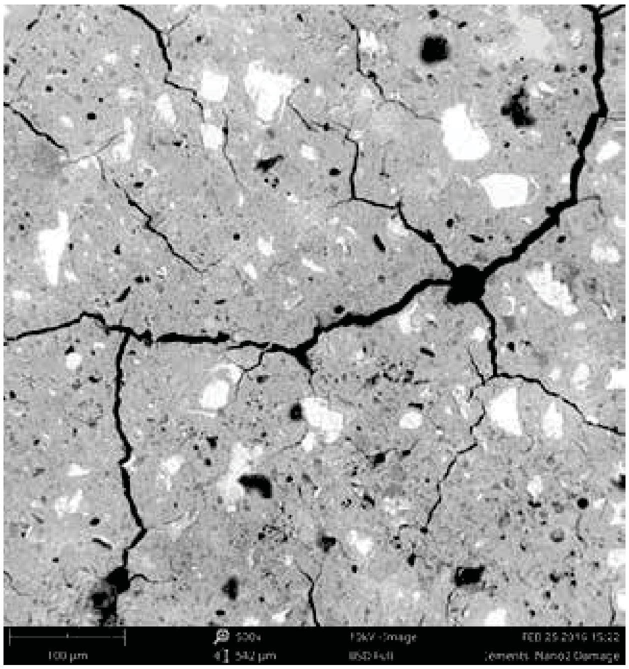

Figure 3. SEM image of irradiated nanocement showing degree of subsurface cracking.

A major finding of this study is that future experiments should be conducted in a differentially pumped vacuum station or through a foil (barrier) that would allow the concrete to be irradiated at pressures closer to ambient. This may eliminate water degassing in a high-vacuum environment that may affect the results prior to the irradiation.

A second major finding is that concrete does experience significant volatilization of hydrous components during the irradiation. The volatilization may be exacerbated by the vacuum environment. The hydrous bonds are also more sensitive to ionizing radiation as compared to metal-metal or metal-oxygen bonds. This is analogous to damage resulting from electronic stopping in ceramics and other insulating materials. In order to limit volatilization in any cement system tested, the particle flux should be kept below 1×10^{12} p/cm^2-sec. This flux will limit significant volatilization from the cement. Fluxes greater than this can result in macroscopic failure as seen in the figures below. Using such low fluxes does make it difficult to (i) identify the irradiation area on the target and (ii) perform radiations that simulate long term exposure.

Future experiments should use these parameters as starting points for performing irradiations.
The investigators would like to acknowledge the outstanding support they received from Mr. Kim Kriewaldt at the University of Wisconsin's Ion Beam Laboratory. The experiments required many on-the-fly changes to the experimental end station and sample mounting configurations. Mr. Kriewaldt was extremely resourceful in accommodating and supplying fixtures to make the investigation successful.

A challenge for future experiments will be performing irradiations and identifying the regions to be irradiated; this may be on the order of a 0.05 mm^2 area after the irradiation. The irradiation zone should be identified via scribing or some other mechanism so that post-irradiation characterization can properly identify low fluence irradiations. Not having x and y control of the sample stage limited the ability to perform irradiations without opening the

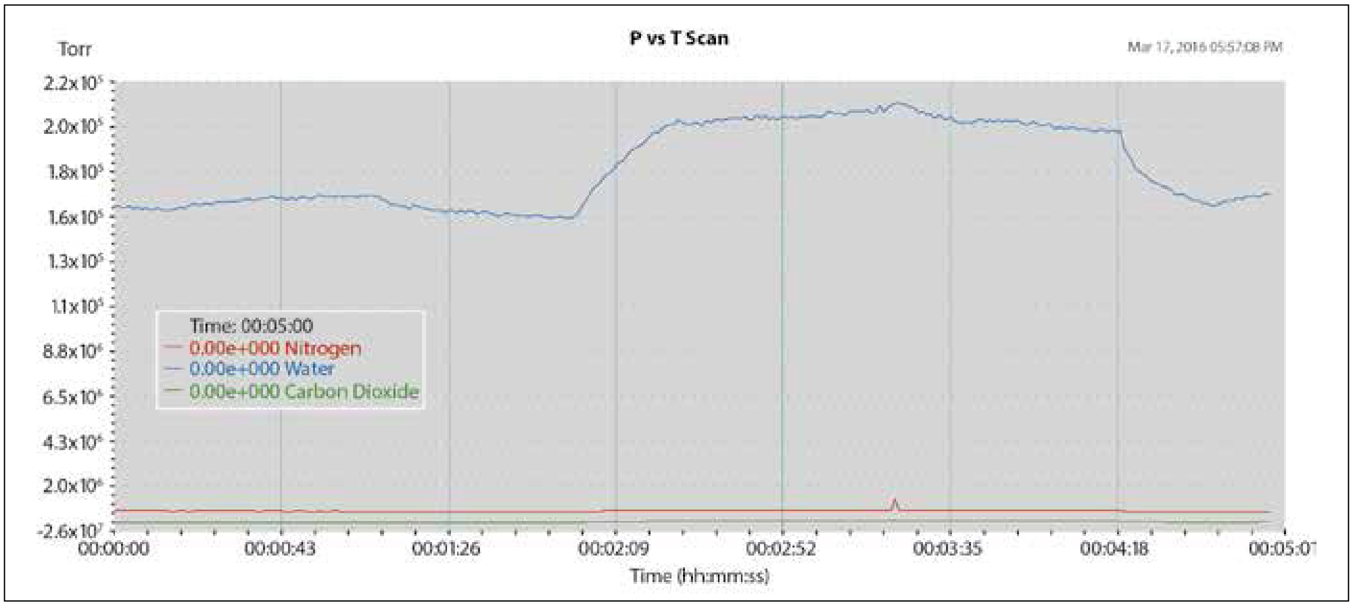

Figure 4. Mass Spectrum pre, during, and post irradiation using a Stanford Research Systems Residual gas analyzer. Irradiation begins at ~2 minutes and stops at ~4 minutes. An increase in the water signal is observed during the irradiation. Beam current used is ~80 nA.

chamber to air. This, and not being able to identify the irradiated area, prevented extensive mechanical properties measurements. Cursory micro-hardness measurements (Vickers) indicate that the irradiated areas may be harder than the native microstructure.

Future Activities

Future goals are to expand the capabilities of the chamber so that multiple irradiations can be performed on a single target. Further investigation should examine changes in the mechanical properties.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
University of Wisconsin	Tandem Accelerator Ion Beam
Collaborators	
University of Illinois	John Popovics (co-principal investigator), Paramita Mondal (co-principal investigator)
University of Tennessee	Chris Wetteland (co-principal investigator), Kurt Sickafus (co-principal investigator)
University of Wisconsin	Jeff Terry (co-principal investigator)
Oak Ridge National Laboratory	Kevin Field (co-principal investigator)

An Atom Probe Tomography Investigation of the Response of Oxide-dispersion Nanoclusters to Non-similar Friction Stir Welds

Chad Parish – Oak Ridge National Laboratory – parishcm@ornl.gov

This work provides basic science information on the ability to weld high performance radiation tolerant alloys for advanced nuclear systems.

This work examined friction stir welds that had been subjected to He or heavy-ion irradiation. Nanostructured ferritic alloys (NFAs) are strong candidates for Generation IV fission systems, but welding of NFAs is an immature technology. We used atom probe to study the effect of irradiation upon the nanostructures of the weld region.

Project Description

Our goal was to determine by irradiation whether friction stir welding (FSW)-processed NFA was grossly similar or different from irradiation of as-fabricated NFAs. This is important because, if the weld process should reduce the overall radiation tolerance of the material, then NFAs might not be usable in advanced Gen IV and fast reactor systems. NFAs show strong potential for application to the core structures in fast reactors or other advanced nuclear energy systems. However, any structure will require welding and joining, and welding of NFAs is not well developed. Traditional fusion (liquid state) welding is unusable because the oxide nanostructures in the NFA simply coarsen in the weld pool and the material ceases to be an NFA. As has been previously demonstrated, both by our group and other groups, FSW of NFAs can produce a structure that retains the nanostructured features, specifically the nano-oxide clusters. However, it is not presently known whether these nano-oxides retain the same radiation tolerance that makes as-fabricated NFAs attractive from an engineering point of view. Therefore, we chose to investigate the question of the radiation response of the nano-oxides in an NFA FSW. We performed heavy ion and helium ion irradiations on an NFA FSW zone at fission-relevant temperatures. This project involved using atom probe at the Center for Advanced Energy Studies (CAES) to investigate the irradiated zones.

Accomplishments

This work first accomplished the production of the FSW structure and irradiation using heavy ions and helium ions, both at Oak Ridge National Laboratory (ORNL). The co-principal investigator, Phil Edmondson, then carried the ion-irradiated samples to CAES and used the focused ion beam and atom probe to examine the different ion irradiated conditions. We then transferred the data to ORNL, where data analysis and manuscript preparation is underway.

Future Activities

Future activities involve finalizing the data analysis, preparing a manuscript, and submitting the manuscript to a journal for peer reviewed publication.

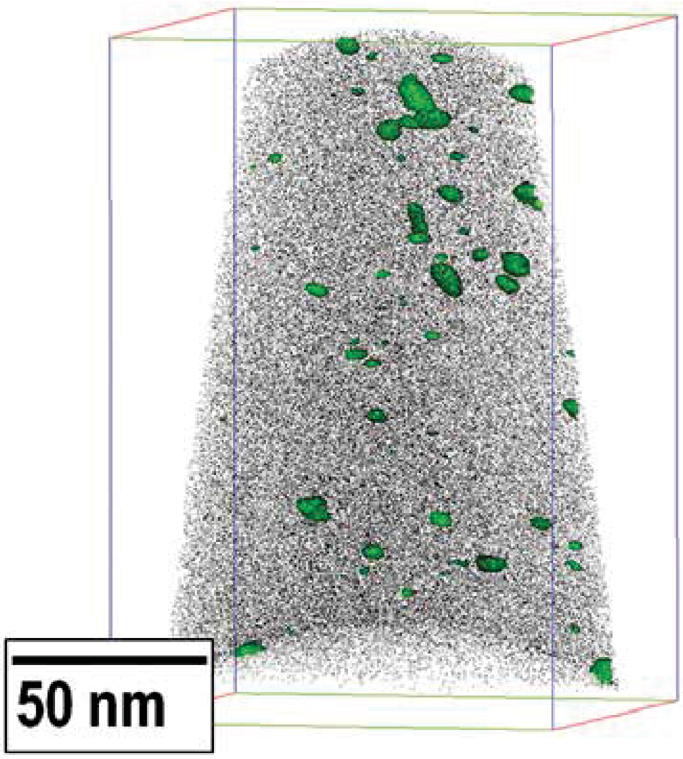

Figure 1. Atom probe map showing nanoclusters (green) in ion irradiated friction welded NFA 14YW.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Idaho National Laboratory	Joanna Taylor (principal investigator)
Oak Ridge National Laboratory	Chad Parish (principal investigator), Phil Edmondson (co-principal investigator)

Characterization on the Bor-60 Neutron Irradiated Austenitic Stainless Steels and Cast Stainless Steel

Yong Yang – University of Florida – yongyang@ufl.edu

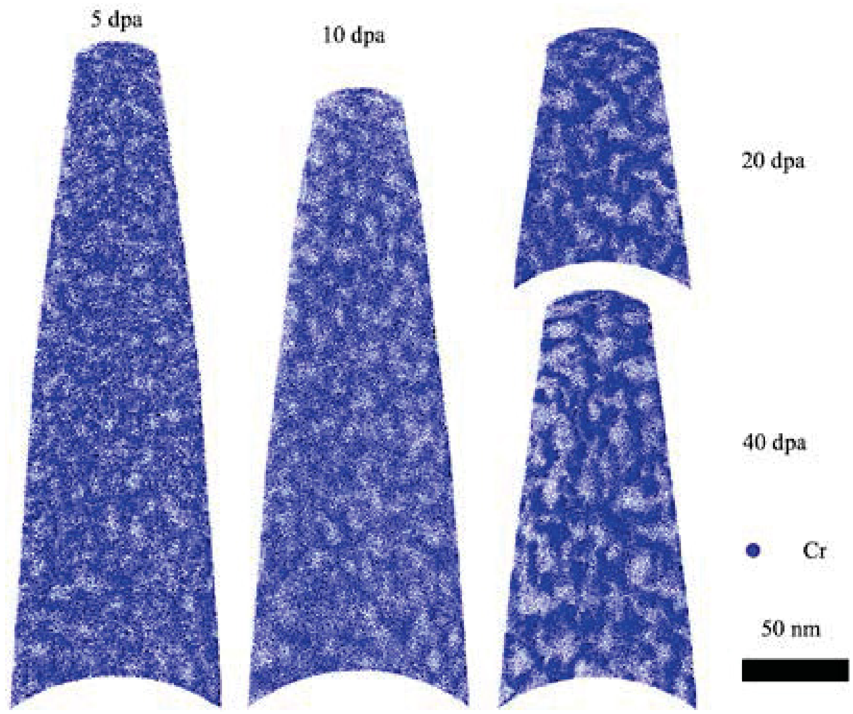

Figure 1. Spinodal decomposition in the ferrite phases of CF3 Cast Stainless Steel irradiated to 5, 10, 20 and 40 dpa, respectively.

The work was planned to systemically characterize the 316 SA and 304 SA stainless steels and CF-3 cast stainless steels irradiated to 45 dpa at 320°C in the Bor-60 reactor. Both transmission electron microscopy and atom probe tomography were used to study the irradiated induced nano-sized features, including dislocation, phase separation, and precipitates. The output from the execution of proposed study fills the existing knowledge gap by providing a more systematic study, at an atomic level, on neutron irradiation effects in light water reactor (LWR) internal structural materials at LWR relevant conditions. By correlating the microstructure study with selected mechanical tests, this proposed research provides a fundamental understanding of the response of austenitic stainless steels, cast stainless steel (welds) during the designed 40-year reactor life, and, more relevantly, during the extended life of 60 years and beyond.

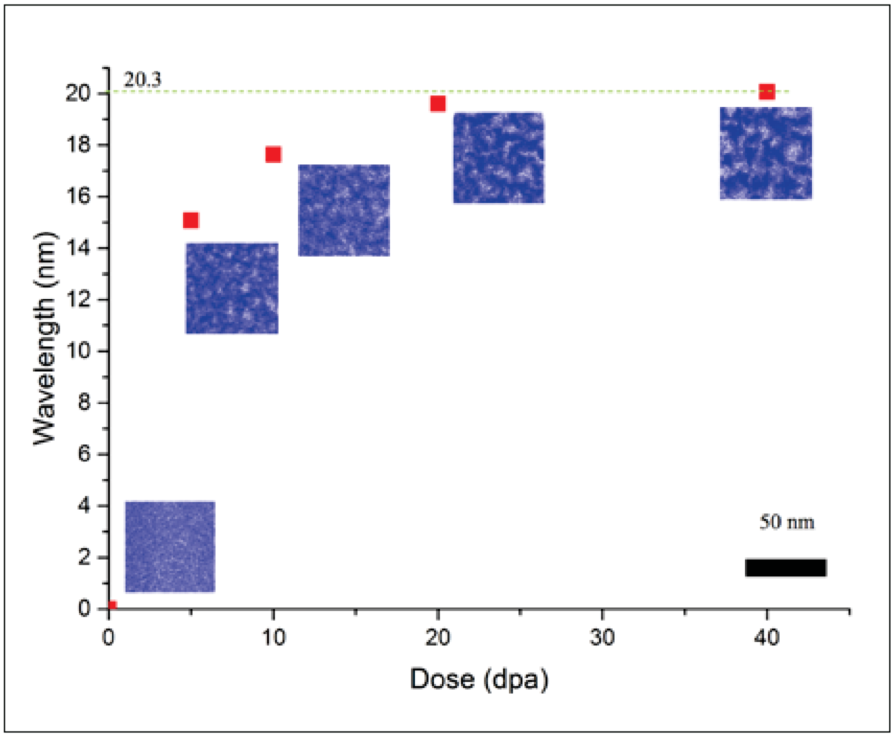

Figure 2. Spinodal decomposition Cr fluctuation wavelength vs. irradiation dose.

Project Description

The proposed study aims to provide a more fundamental and advanced knowledge of the irradiation response in austenitic stainless steel and cast stainless steel at LWR end-of-life (EOL) relevant conditions. To extend the service lifetime of LWRs beyond 60 years, the integrity of reactor components and the performance of structural materials must be evaluated adequately. The anticipated neutron dose on LWR internal structural material can be 50 dpa or above. The accurate assessment and prediction of materials performance under anticipated operating conditions are of particular importance for ensuring the safe operation of nuclear power plants, particularly at such high anticipated dose. As nuclear power plants age and neutron fluence increases, detrimental effects resulting from radiation damage and related degradation of the structural integrity of core internal components have become an increasingly important regulatory concern for the reactor license renewal. The proposed research is well aligned with the U.S. Department of Energy Office of Nuclear Energy's (DOE-NE) Sustainability of LWRs program and evaluating the materials' integrity is a key need for considering the life extension for the existing LWRs.

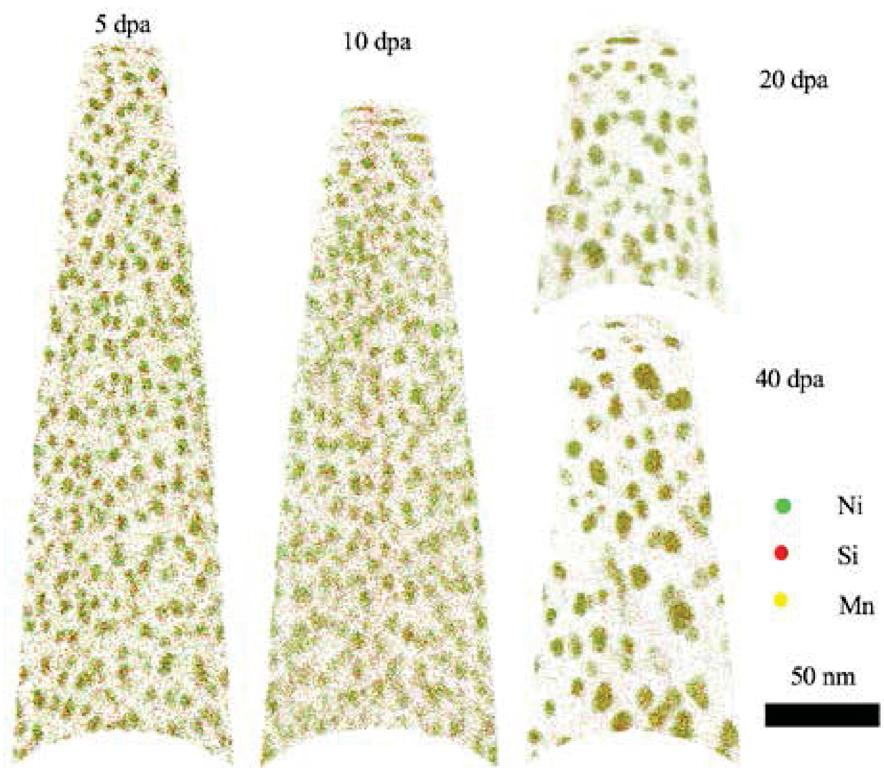

Figure 3. Ni-Si-Mn clusters in the irradiated ferrite phase.

Overall, this project is the first reported study on the ferrite phase in a duplex stainless steel ever irradiated to such a high neutron irradiation dose. It shows that the microstructural changes may saturate at much higher dose than normally expected: 5 dpa at a LWR relevant condition.

Accomplishments

Microstructural evolutions including spinodal decomposition (SD) (Figure 1) and G-phase precipitation were quantified using the atom probe tomography for the ferrite phase in a duplex structure cast stainless steel irradiated to 5, 10, 20, and 40 dpa, respectively. The SD wavelength was calculated using the radial distribution function (RDF) analysis and the amplitude was obtained from Cr-frequency distribution. Both the SD wavelength and amplitude increases follow a logarithmic curve and begin to saturate after 20 dpa (Figure 2).

For the G-phase precipitates, a decrease in number density and an increase in mean size were observed with the increase of neutron irradiation dose from 5 to 40 dpa (Figure 3). Overall, this project is the first reported study on the ferrite phase in a duplex stainless steel ever irradiated to such a high neutron irradiation dose and it shows that the microstructural changes may saturate at a much higher dose than normally expected: 5 dpa at a LWR relevant condition.

Future Activities

The project is now completed.

Atom Probe Tomography characterization really provides an unprecedented insight into the chemical evolution of materials under irradiation.

—Yong Yang, Associate Professor, Nuclear Engineering Program, University of Florida

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Argonne National Laboratory	Yiren Chen (co-principal investigator),
University of Florida	Yong Yang (principal investigator), Zhangbo Li (collaborator)

STEM/EELS Study of Fission Product Transport in Neutron Irradiated TRISO Fuel Particles

Haiming Wen – Idaho State University – haiming.wen@inl.gov

Understanding of fission products in irradiated TRISO fuel particles will facilitate design and fabrication of TRISO particles with improved performance and expedite the development and deployment of high-temperature gas reactors.

Tristructural isotropic (TRISO) coated fuel particles are designed for use as nuclear-fuel particles in high temperature nuclear reactors. They are composed of a uranium oxide (UO_2) or uranium oxy carbide (UCO) fuel kernel protected by a series of ceramic coating layers that retain fission products. The ceramic TRISO coatings are a finely tuned fission product containment system, which consist of, in the order of increased distance from the fuel kernel, a porous carbon buffer layer, an inner pyrolytic carbon (IPyC) layer, a SiC layer, and an outer PyC (OPyC) layer. The SiC layer is the primary fission product barrier of the TRISO particle and serves as the principal structural layer as well. Release of certain metallic fission products, e.g., Ag and Pd, through intact TRISO coatings has been evident for decades around the world, as well as in the recent Advanced Gas Reactor (AGR)-1 experiment at Idaho National Laboratory. The release of 110mAg is a potential worker safety concern due to plate out on the cooler metallic parts of the helium pressure boundary. This safety concern highlights the importance of identifying the metallic fission product transport mechanisms of 110mAg through the TRISO coating layers.

Project Description

The objectives of this project are to study the distribution, composition, and structure of fission products in neutron irradiated SiC layer of the TRISO particles, using electron microscopy including scanning transmission electron microscopy (STEM), energy dispersive x-ray spectroscopy (EDS), electron energy loss spectroscopy (EELS), high-resolution transmission electron microscopy (HRTEM), and precession electron diffraction (PED), in an effort to enhance the understanding of distribution, composition, structure, and transport mechanisms of fission products. In the past 40 years, numerous studies, including reactor experiments, out-of-pile experiments, and simulations, have been performed to investigate the

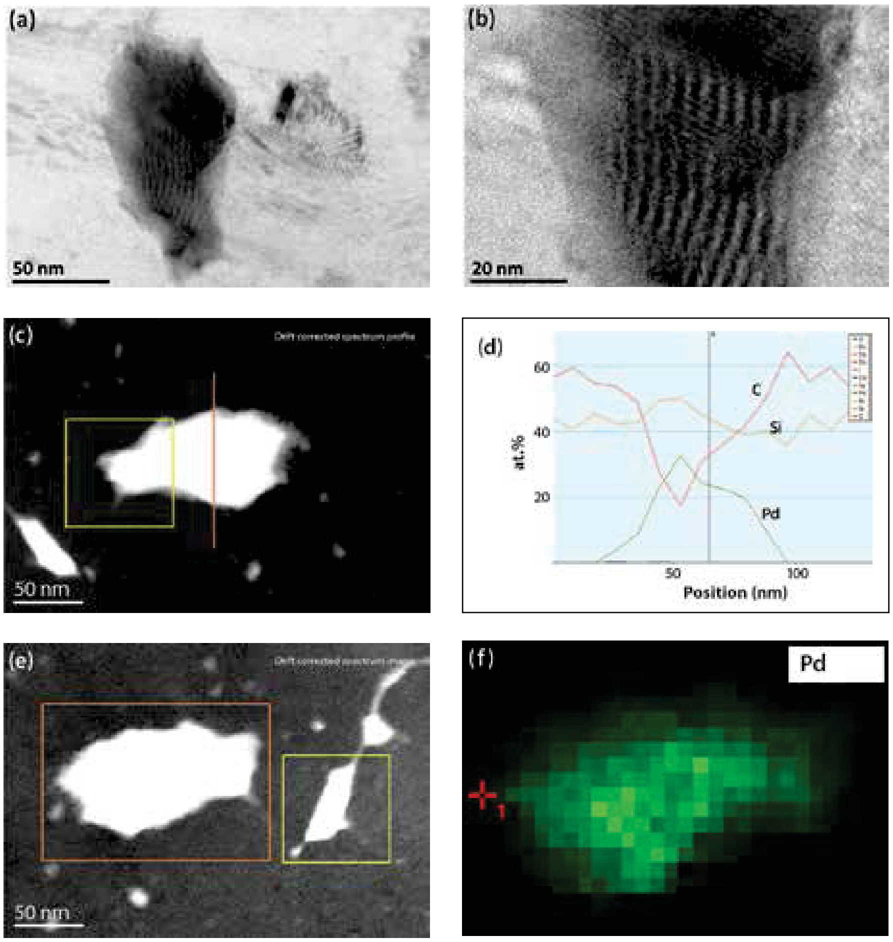

Figure 1. (a) (b) TEM images of a fission product precipitate; (c) STEM image of the same fission-product precipitate where the line indicates EDS line scan and the square represents a reference area for drift correction during EDS line scan; (d) EDS line scan results corresponding to (c); (e) STEM image of the same fission-product precipitate where the orange rectangle indicates EDS mapping area and the yellow square represents a reference area for drift correction during EDS mapping; (f) Pd map corresponding to EDS mapping area in (e).

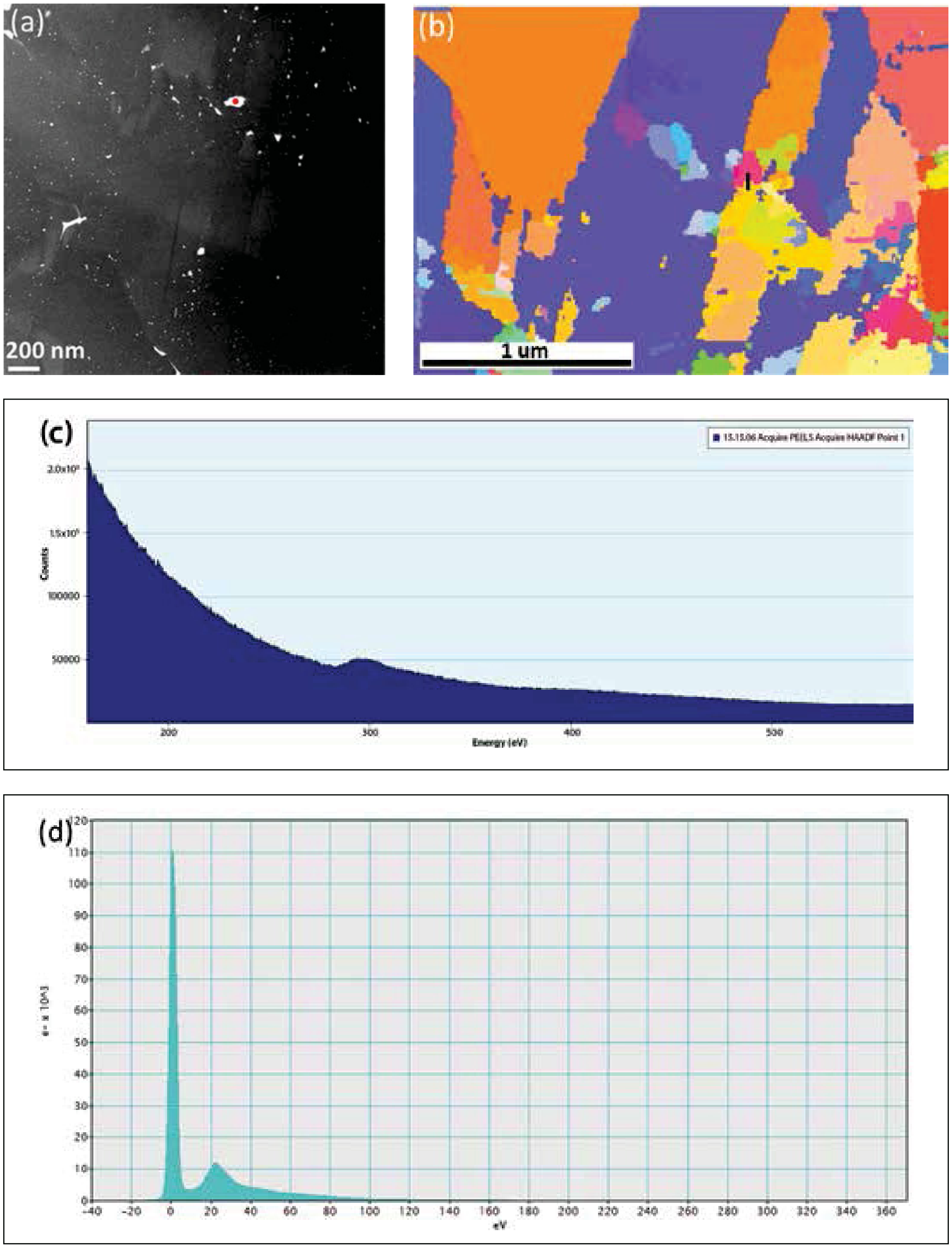

Figure 2. (a) STEM image; (b) precession electron diffraction derived ASTAR map, where the fission product precipitate highlighted in (a) with a red spot is located at a grain boundary indicated by a black line (the black line is drawn during ASTAR data analysis across the grain boundary so that the characteristics of the grain boundary are obtained); (c) EELS spectrum corresponding to the precipitate highlighted in (a); (d) zero-loss peak of the EELS spectrum corresponding to the precipitate highlighted in (a).

fission product transport behavior and mechanisms. Grain boundary (GB) diffusion, neutron induced diffusion, catalytic Pd assisted transport, and vapor-phase migration all have been suggested as possible governing transport mechanisms. The previous studies, however, have not been able to reproduce the transport behavior of fission products and the mechanisms remain poorly understood. This project is anticipated to contribute to an enhanced understanding of the mechanisms of fission product transport in the irradiated SiC layer of TRISO particles. Thorough understanding of fission product transport mechanisms in the SiC layer will improve fuel modeling and design, and better SiC layers and TRISO coated particles can be designed to enhance retention of fission products and reduce their release. Eventually, TRISO coated particles with improved performance can be fabricated and this advancement will expedite the qualifying and licensing processes for TRISO coated particles. Hence, this research is contributing to achieving the objectives of U.S. Department of Energy Office of Nuclear Energy (DOE-NE) to develop the next generation nuclear reactors, particularly high temperature gas reactors.

Accomplishments

The technical goals of the research are to determine the distribution, composition, and structure of fission products in the SiC layer of a TRISO particle, neutron-irradiated to 3.6×10^{21} n/cm^2 fast fluence at 1040°C, utilizing electron microscopy methods. STEM was used to investigate distribution of fission products; EDS in STEM was employed to obtain fission product composition; information on structure of fission products was acquired using HRTEM; PED derived ASTAR was executed to study characters of grain boundaries where fission products are located; attempts were made to measure charge states of precipitates using EELS. The experiments and data analyses were conducted by Haiming Wen, and the samples were from the Advanced Microscopy and Microanalysis Program that Isabella van Rooyen is in charge of under the Advanced Gas Reactor Program of DOE-NE. Both Wen and van Rooyen participated in the design of the project. Accomplishments were made toward the technical goals. In summary, a high density of nanoscale fission product precipitates was observed in the SiC layer close to the SiC-IPyC interface, most of which are rich in Pd, while Ag was not identified. Some Pd-rich precipitates contain U. Precipitates tend to have complex structure. Although a precipitate appears to have uniform contrast in STEM, it may exhibit non-uniform contrast in TEM/HRTEM, which may be caused by composition variations

The advanced electron microscopy techniques available at CAES provide great tools for studying structure and composition of fission products in neutron irradiated nuclear fuels.

— Haiming Wen, Research Assistant Professor

throughout the precipitate (as indicated by EDS), and by variations in crystal structure, orientation, or dislocation density among different parts of the precipitate (as suggested by HRTEM). ASTAR results indicate that the majority of precipitates are located at random high-angle grain boundaries. Significant challenges were encountered during EELS study and no useful EELS results were obtained. The challenges were ascribed to the fact that the TEM specimen was not thin enough. The TEM specimen was prepared previously in the Electron Microscopy Laboratory (EML) and, due to the significant radioactivity, it could not be further thinned at the Center for Advanced Energy Studies (CAES). In addition, it could not be shipped back to EML for further thinning due to the limit in time and budget for this Rapid Turnaround Experiment (RTE) project. Although the EELS study was not successful, the other data (STEM, EDS, TEM/HRTEM, and ASTAR) enabled significant accomplishments toward the technical goals, which rendered this project successful.

Figure 1 shows TEM and STEM images of a fission product precipitate and EDS line scan and mapping results. In TEM images (a) and (b), there is contrast variation throughout the precipitate, although the precipitate appears to have uniform contrast in STEM image (c). EDS line scan and mapping indicate that the precipitate is rich in Pd, however, there is variation in the Pd concentration throughout the precipitate. Note that Si and C concentrations are from the SiC matrix.

Figure 2 displays STEM, ASTAR, and EELS results. The ASTAR results indicate that the precipitate highlighted in (a) is located at a random high-angle grain boundary with a misorientation angle of 30 degrees. Note that this precipitate is the same with the one studied in Figure 1. Although it is clear from EDS results that this precipitate is Pd-rich, no peak corresponding to Pd is evident in the EELS spectrum (c); the only peak present in the EELS spectrum corresponds to C (that comes from the SiC matrix). From the zero-loss peak (d), the thickness of the precipitate is computed as 0.54 electron mean free path, which is too thick to get good EELS results. In order to obtain good EELS signals, the thickness needs to be on the order of 0.1 to 0.2 electron mean free path. Thickness measurements were conducted using EELS at many different locations and it was found that the TEM specimen is too thick for EELS study.

Figure 3 shows TEM, HRTEM, STEM, and EDS results on another fission product precipitate. From the TEM and HRTEM images, there is contrast variation across the precipitate and it appears that there are multiple layers in the precipitate in the thickness direction of the TEM specimen. From the HRTEM images, it appears that the parts with different contrast in the precipitate have the same crystal

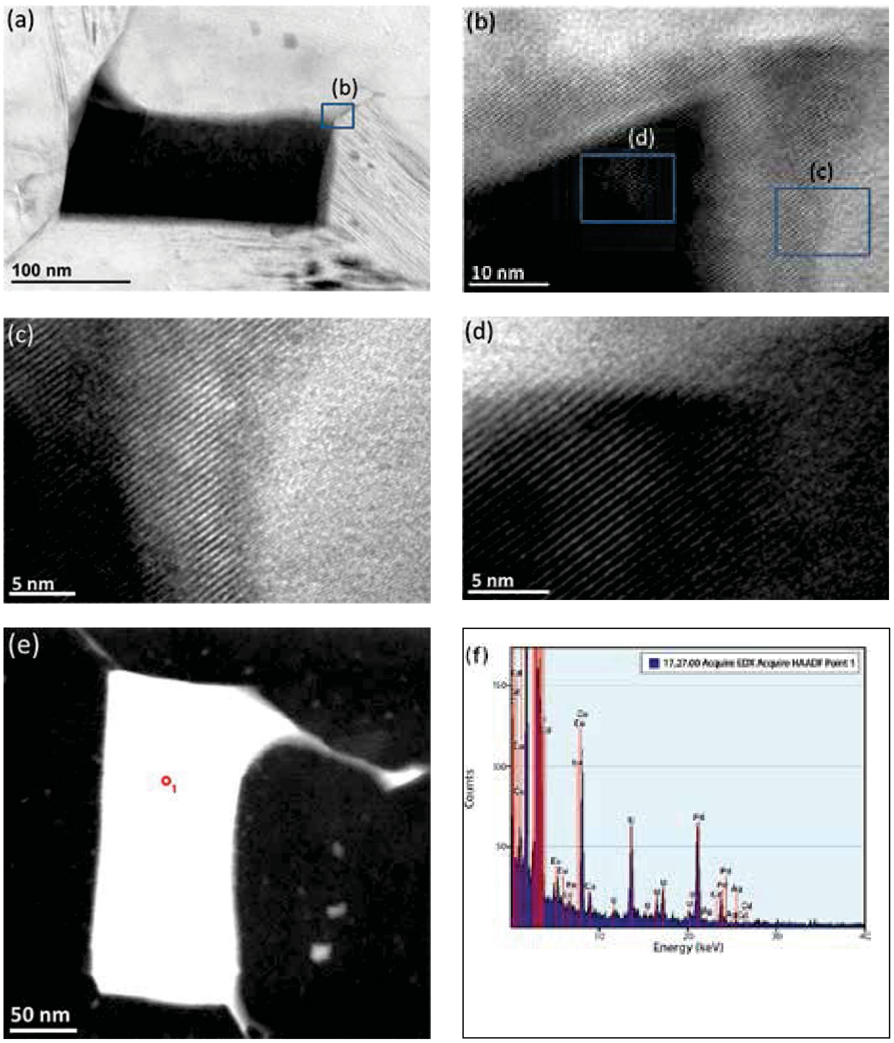

Figure 3. (a) TEM image of a fission-product precipitate; (b) (c) (d) high-resolution TEM images of the fission-product precipitate in (a), where the rectangular areas indicated in (b) are magnified in (c) and (d) respectively; (e) STEM image of the same fission-product precipitate; (f) EDS spectrum corresponding to the EDS point scan indicated in (e).

structure, however, they may have slightly different orientation and/or dislocation density. The contrast in the precipitate in the STEM image is uniform and EDS point-scan results indicate that this precipitate is rich in Pd, with a U concentration.

STEM, EDS, TEM, and HRTEM results on another fission product precipitate are displayed in Figure 4. In the STEM image, the precipitate has uniform contrast. The EDS results indicate that the precipitate is rich in Pd. Note that the Cu concentration is from the TEM grid. Multiple EDS point scans indicate that there are variations in Pd concentration across the precipitate. Similarly, in TEM and HRTEM images, there are contrast variations among different parts of the precipitate. From HRTEM images, the precipitate appears to have a face centered cubic structure and different parts of the precipitate have the same structure. The contrast variations may be caused by variations in Pd concentration and by slightly different orientation and/or dislocation density.

Future Activities

This project is now complete.

Publications

[1.] Haiming Wen, Isabella J. van Rooyen, "Advanced electron microscopy study of fission products in a TRISO coated particle neutron irradiated to 3.6×10^{21} n/cm^2 fast fluence at 1040°C," International Conference on Nuclear Materials 2017, June 25-26, Paris, France, accepted.

[2.] Haiming Wen, Isabella J. van Rooyen, "Distribution, composition and structure of fission products in a TRISO coated particle neutron irradiated to 3.6×10^{21} n/cm^2 fast fluence at 1040°C," journal paper in preparation

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Idaho National Laboratory	Isabella J van Rooyen (co-principal investigator)
Idaho State University	Haiming Wen (principal investigator)

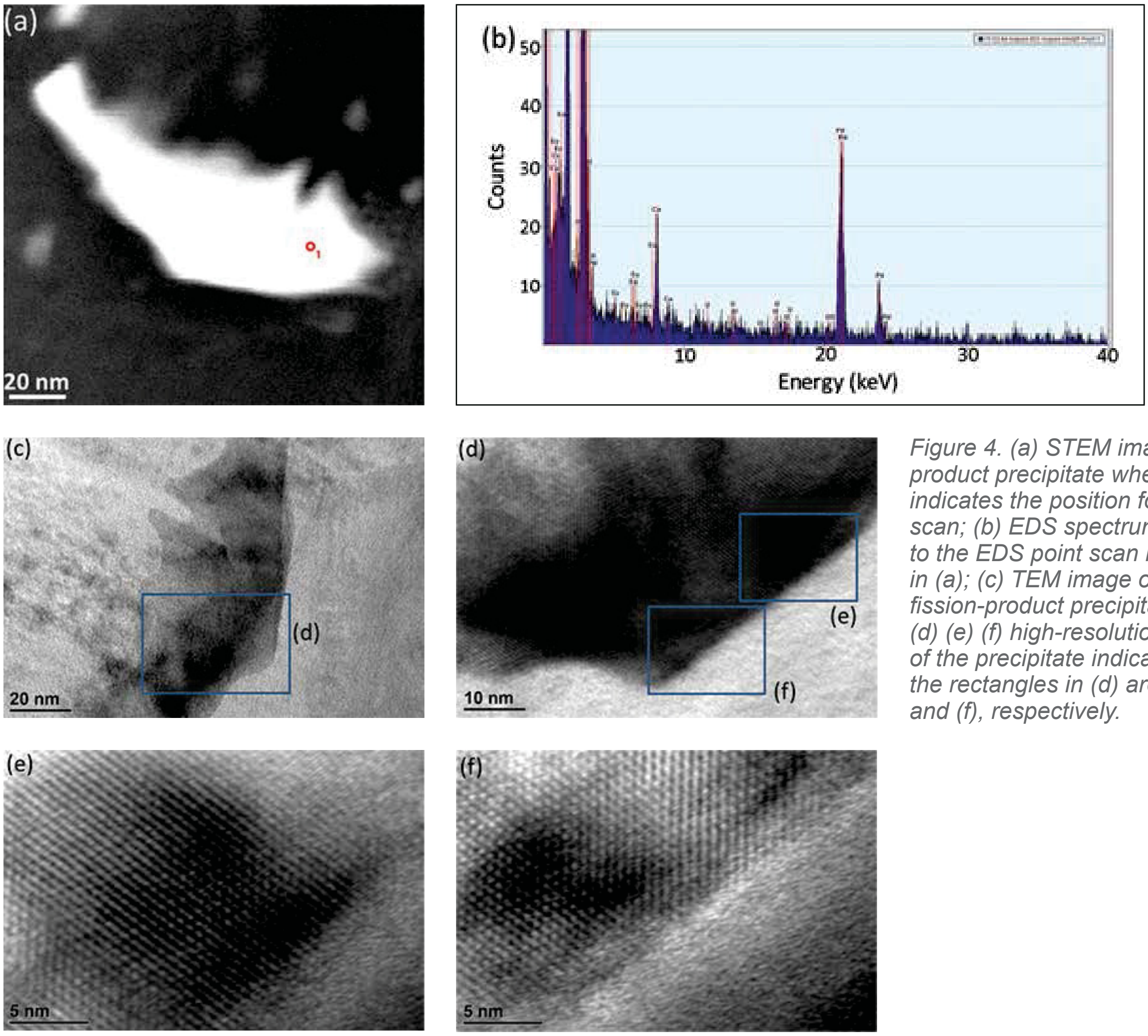

Figure 4. (a) STEM image of a fission-product precipitate where the red circle indicates the position for a EDS point scan; (b) EDS spectrum corresponding to the EDS point scan indicated in (a); (c) TEM image of the same fission-product precipitate shown in (a); (d) (e) (f) high-resolution TEM images of the precipitate indicated in (c), where the rectangles in (d) are magnified in (e) and (f), respectively.

Investigation of Dislocation Loop Hardening and Stability in Irradiated RPV Steels

Peter Wells – University of California, Santa Barbara – well7765@gmail.com

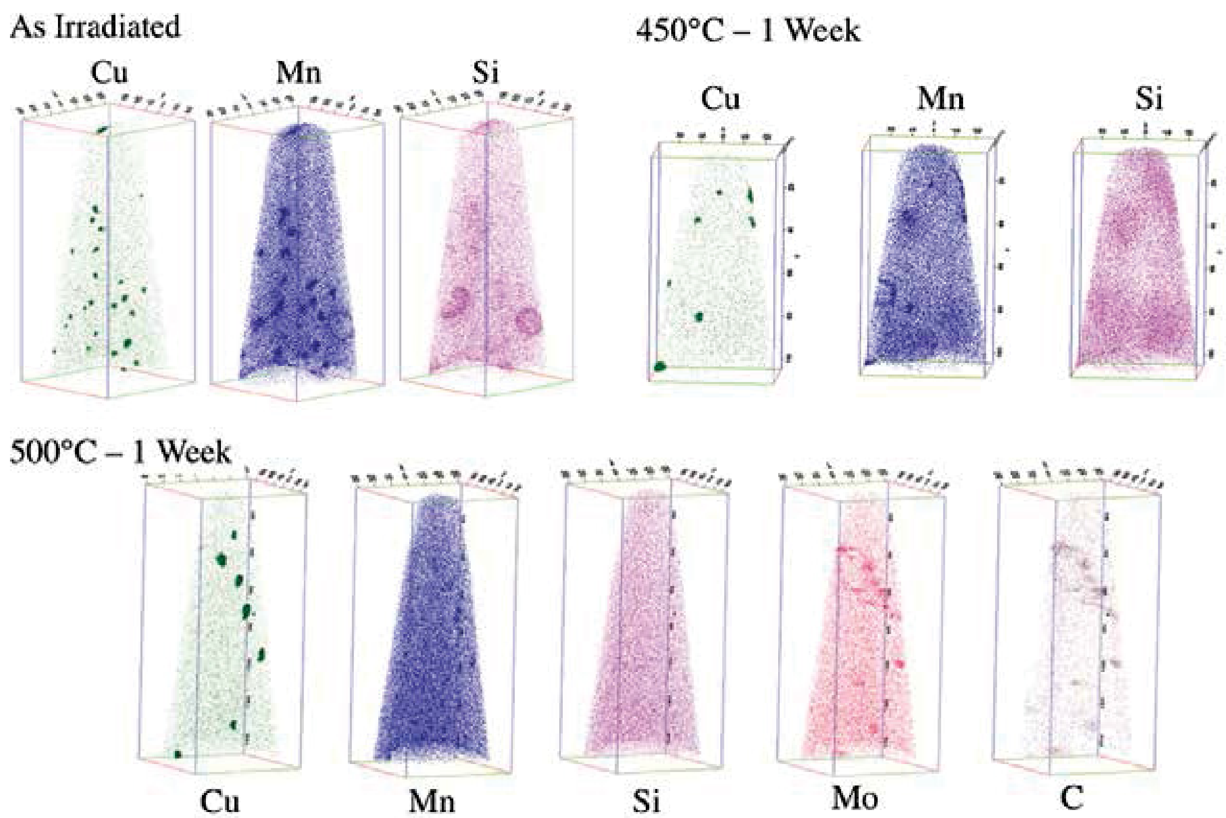

Figure 1. Atom probe maps from the Ni-free steel (LA) showing evolution of both CRP and dislocation loop enrichment under annealing.

One potential barrier to extending light water reactor (LWR) lifetimes to 80 years is embrittlement of their massive reactor pressure vessels (RPV). Embrittlement is primarily due to the formation of nm-scale precipitates as well as solute defect clusters, which cause hardening and a corresponding increase in the ductile-to-brittle transition temperature. Safely extending the lifetime of the US fleet of LWRs will require robust prediction models that not only include the effect of irradiation variables such as neutron flux, fluence, and operating temperature, but also alloy chemistry. This research aims to better understand the role of alloy Ni content in the formation and thermal stability of hardening features formed under irradiation.

The insight gained from atom probe tomography on this series of irradiated alloys will enable us to make better, physically based predictive models for extended life LWR operation.

Project Description

A recent Nuclear Science User Facilities (NSUF) funded irradiation in the Advanced Test Reactor included four reactor pressure vessel steels with the same nominal bulk Cu contents (Д0.4 wt.%) but varying Ni levels (0.01, 0.18, 0.84, and 1.25 wt.%) to isolate this element's effect on irradiation hardening. The samples were irradiated to 1.7 dpa at Д310°C. Microhardness on the irradiated samples showed that the steels with lower Ni contents (0.01-0.18) had much less hardening than those with higher Ni contents (0.84-1.25). Previous atom probe tomography (APT) studies of the higher Ni samples showed that the microstructure was dominated by a very high density of nm-scale Cu-Mn-Ni-Si precipitates with very few dislocation loops observed. In addition, post irradiation annealing studies showed that the hardening features in the lower Ni content steels were more stable than those in the higher Ni steels at temperatures up to 500°C. Thus, the purpose of this experiment was to use APT and transmission electron microscopy (TEM) to investigate the microstructure of both the as-irradiated and the irradiated and annealed low Ni alloys to better measure both the precipitates and the dislocation loops.

Accomplishments

Atom probe tomography and transmission electron microscopy were both completed on the two low Ni steels. The atom probe showed large dislocation loops (<d> Д10 nm) in all samples that were enriched predominantly in Si, but also in Mn and Ni, shown in Figure 1. In addition, the density of Cu-rich clusters in the low Ni alloys was significantly lower than in the higher Ni alloys that were previously studied. Line profiles through the loops in the lowest Ni content (0.01 wt.%) steel,

Annealing and atom probe tomography characterization of these RPV steels provides a quantitative assessment of the various hardening features formed under irradiation, supporting model development at extended life LWR fluences.

— Nathan Almirall, Graduate Student

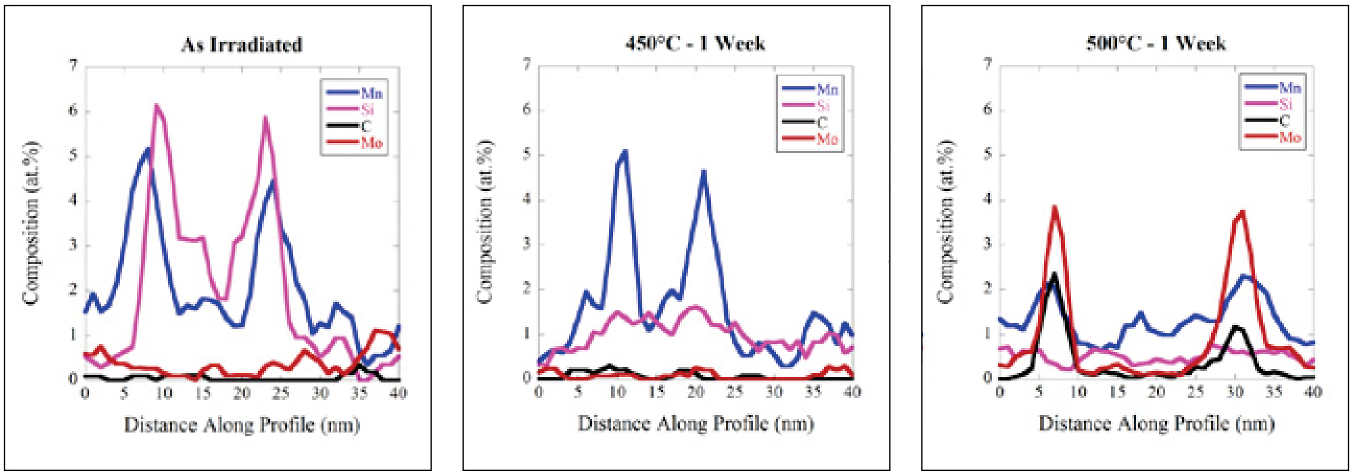

Figure 2. Line profiles through dislocation loops showing change in solute segregation around dislocation loops under annealing.

seen in Figure 2, show enrichment of Mn on the outer edges of the loop and Si enrichment on the inner edges. Sequential post irradiation annealing treatments for 1 week each were performed at temperatures between 350°C and 550°C in 25°C increments in order to track the changes in both the Cu-rich precipitates and also the solute segregation to dislocation loops. The precipitates in the 450°C and 500°C annealed samples, also shown in Figure 1, began coarsening but still had slight Mn enrichments. The Si was no longer enriched around the dislocation loops after the 450°C anneal, seen in both Figures 1 and 2, but some slight Mn segregation was still observed. After the 500°C anneal, the loops were only slightly enriched in Mn, but also significantly enriched in Mo and C. These results will be used to better calibrate hardening models and determine the hardening efficiency of dislocation loops and precipitates. In addition, they can help to better understand radiation induced segregation of various elements.

Future Activities

This work is part of a much larger project designed to create an RPV embrittlement prediction model for extended life fluences. These results have helped to guide the creation of this model, but future work will focus on generating a large database of high fluence embrittlement for various RPV alloys and irradiation conditions.

Publications

[1.] P. B. Wells, N. Almirall, G. R. Odette, T. Yamamoto, D. Gragg, H. Ke, D. Morgan, "Thermal Stability of Nanoscale Mn-Ni-Si Precipitates in Irradiated Reactor Pressure Vessel Steels," *TMS 2016 Annual Meeting, Nashville, TN, February 15-19, 2016.*

[2.] P. B. Wells, N. Almirall, G. R. Odette, T. Yamamoto, D. Gragg, H. Ke, D. Morgan, "Thermal stability of hardening features formed under high dose neutron irradiation," *International Group of Radiation Damage Mechanisms 19th Semiannual Meeting, Asheville, NC April 10-15, 2016.*

A publication of this study is planned.

Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor, Hot Fuel Examination Facility
Collaborators	
University of California, Santa Barbara	G. Robert Odette (co-principal investigator), Takuya Yamamoto (co-principal investigator), Peter Wells (co-principal investigator), Nathan Almirall (collaborator)

Microstructure Characterization of High-energy Xe Ion Irradiated U-Mo

Bei Ye – Argonne National Laboratory – bye@anl.gov

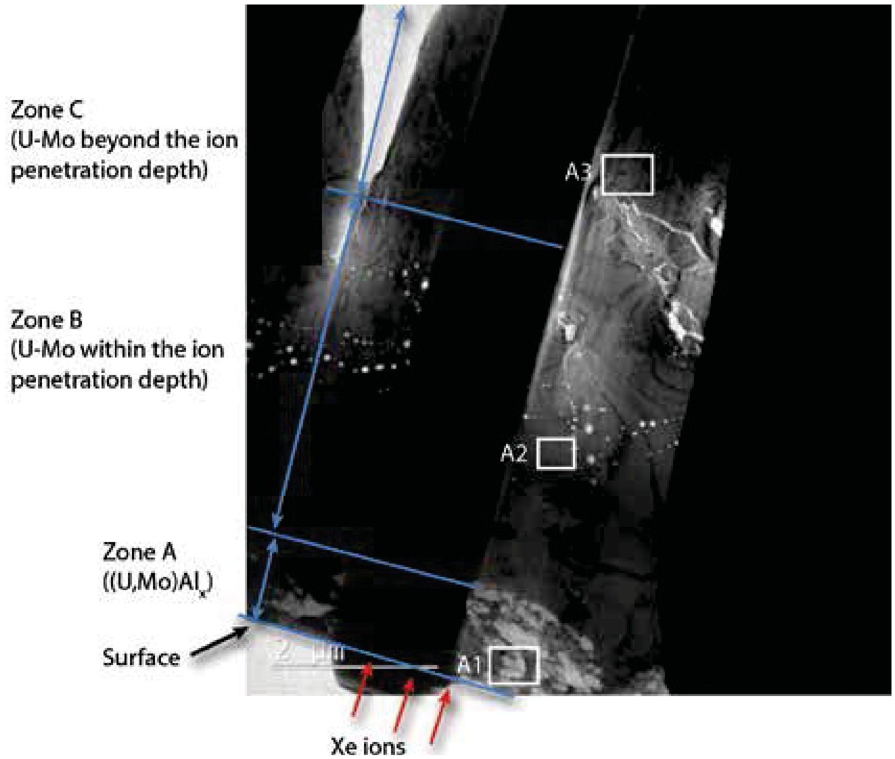

Figure 1. An overview of the TEM sample prepared from a U-Mo/Al sample irradiated with 84 MeV Xe ions to a dose of 2.9 × 10^{17} ions/cm^2.

The information obtained in this study provides new perspective on microstructural evolution in nuclear fuels under irradiation, for example, irradiation induced recrystallization.

In this project, U-Mo/Al dispersion fuel samples irradiated with high energy heavy ions were characterized with TEM to reveal ion modified microstructure in U-Mo. Qualitative and quantitative assessments of the microstructural evolution of U-Mo as a function of the ion penetration depth were obtained.

Project Description

U-Mo/Al dispersion fuels, developed for reducing uranium enrichment in research and test reactor fuels for non-proliferation purpose, underwent drastic microstructural changes during in-pile irradiations, including the excessive growth of (U,Mo)AlX interdiffusion layer and large fission gas filled pores at the interfaces of U-Mo fuel particles and the Al matrix.

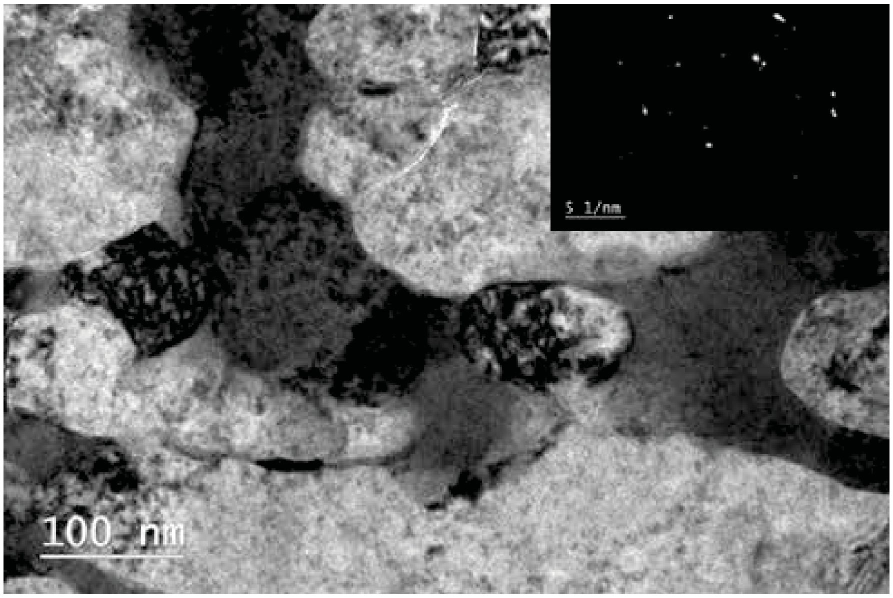

Figure 2. The magnified view of the "A1" area marked in Figure 1, showing the microstructure of the (U,Mo)Alx layer.

These microstructural changes degrade fuel thermal conductivity, increase fuel temperature, and eventually lead to excessive fuel swelling. In order to improve the fuel's irradiation performance, it is critical to adjust fuel fabrication parameters based on the correct understanding of the causes of the microstructural changes. Heavy ion irradiation was employed to screen fuel fabrication variables and to investigate separate effects of irradiation behavior of U-Mo/Al dispersion fuels. By comparing the post-irradiation examination (PIE) results of carefully selected samples, a qualitative assessment of the effects of dose, dose rate, U-Mo grain size, irradiation temperature, and Mo contents on U-Mo/Al irradiation behavior can be obtained. The characterization results of ion irradiated fuels will be used in designing the next irradiation test in reactor.

The characterization experiments at CAES revealed a lot of new information of the microstructure of irradiated U-Mo fuel.

— Bei Ye, Materials Scientist

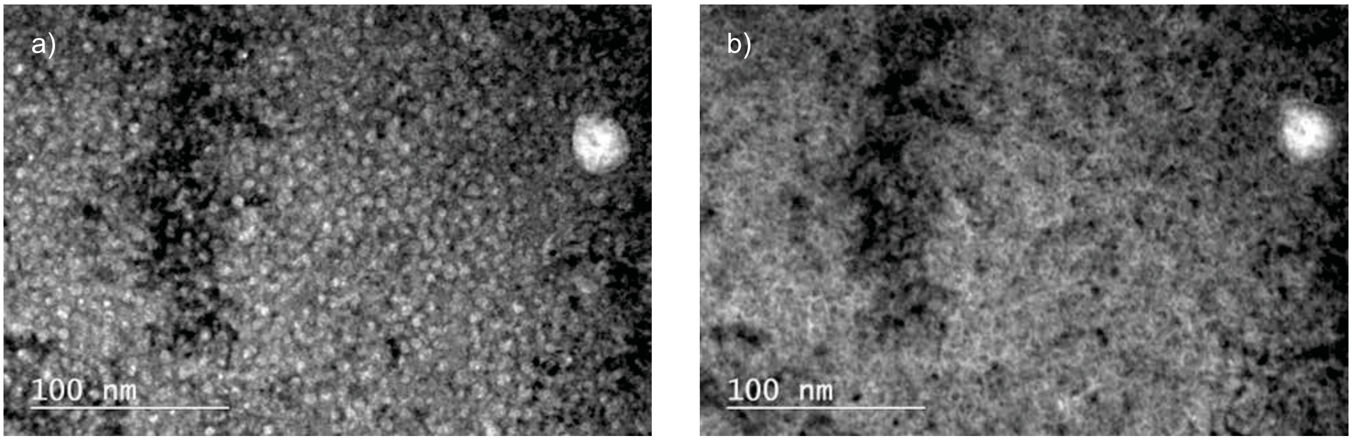

Figure 3. The magnified views of the "A2" area marked in Figure 1 taken at the (a) underfocus and (b) overfocus conditions, showing the high density nano-bubbles inside fuel grains.

Accomplishments

TEM specimens of irradiated U-Mo/Al dispersion fuel were fabricated using the FEI Quanta focused ion beam (FIB) and characterized at the Tecnai STEM at the MaCS at CAES. Several 20 × 20 × 10 μm^3 U-Mo cubes were lifted out using the FEI Quanta FIB for synchrotron irradiation characterization at the APS MRCAT Line at Argonne National Laboratory. Additional TEM specimens were prepared from un-irradiated U-Mo fuel using the FIB for in-situ irradiation study at the IVEM Facility at ANL. All FIB prepared samples were readied by the team with the great help of Jatu Burns. Joanna Taylor helped a great deal in shipping and handling the radioactive samples.

Future Activities

The TEM study performed in 2016 revealed the microstructure of heavy ion irradiated U-Mo at certain irradiation temperature and dose rate. We will expand the investigation to samples irradiated at different temperatures and dose rates.

Publications

[1.] B. Ye, L. Jamison, S. Bhattacharya, A. Yacout, "TEM Characterization of U-Mo Irradiated with High-Energy Xe Ions," Transaction of ANS Nuclear Society 114, (2016) 1285–1288.

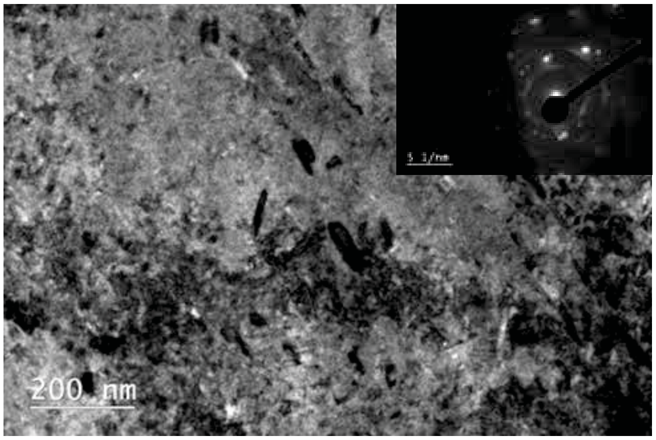

Figure 4. The magnified view of the "A3" area marked in Figure 1, showing second-phase precipitates forming beyond the Xe deposition range.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Argonne National Laboratory	Bei Ye (principal investigator), Laura Jamison (collaborator), Yinbin Miao (collaborator)

Effects of Carbon Addition on Solute Redistribution in Fe-9Cr Alloys Under Irradiation

Cheng Sun – Oak Ridge National Laboratory /Idaho National Laboratory – cheng.sun@inl.gov

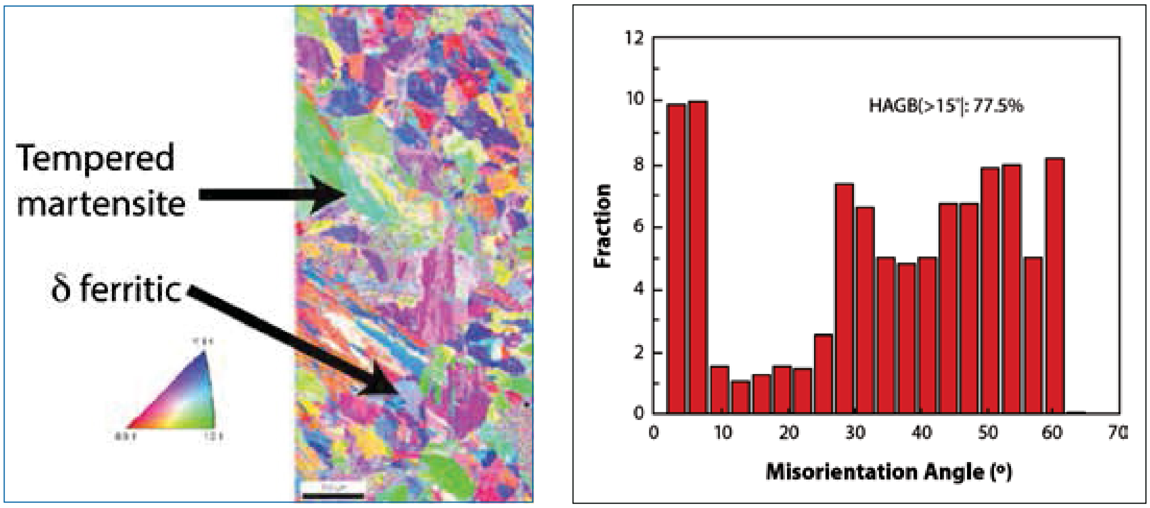

Figure 1. EBSD mapping showing the δ-ferrite phase in an equiaxed morphology and tempered martensite phase in a lath structure. The portion of high-angle grain boundaries in this Fe-9Cr alloy is 77.5%.

The designs of the next generation of nuclear reactors call for the development of materials that can withstand severe neutron irradiation over a wide range of temperatures. Fe-Cr-based alloys are leading candidates for cladding and core structure applications in reactors. Radiation induced solute redistribution results in the degradation of mechanical properties and structure integrity. This research focused on the microstructure and microchemistry characterization of high dose irradiated Fe-Cr alloy by advanced electron microscopy and atom probe tomography.

Project Description

Fe-9Cr alloys are leading candidates for cladding and core components in the next generation of nuclear reactors. Two issues that limit their lifetime at high doses are hardening at low temperatures and doses, and void swelling at high doses and intermediate temperatures. A fundamental understanding of the effects of carbon in these alloys will aid in overcoming these obstacles. Surveys of irradiation behavior of alloys by neutron and ion irradiation indicate that the nucleation and growth kinetics of radiation induced defects are strongly influenced by the minor elemental additions. The hypothesis is that carbon vacancies can trap small dislocation loops and prevent

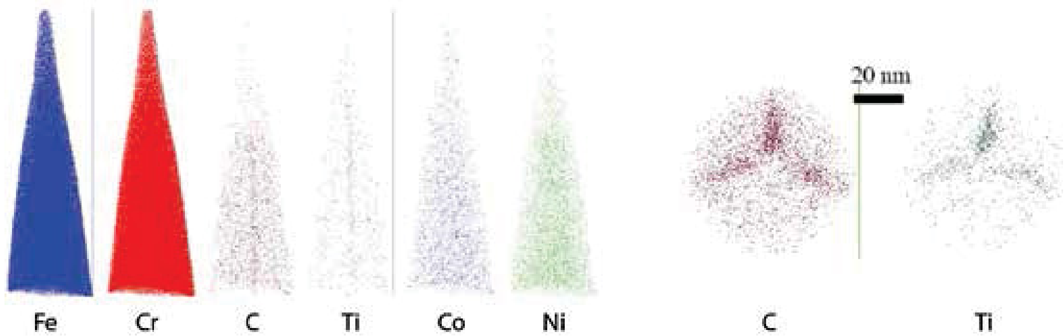

Figure 2. Atom probe tomography of tempered martensite in as-received Fe-9Cr alloy. Enrichment of carbon and titanium in the grain boundary were obtained.

A fundamental understanding of the effects of carbon on the solute redistribution in these alloys will help us design the chemical composition of Fe-Cr based alloys with improved radiation performance in nuclear reactors.

a migration towards sinks. At the same time, vacancies are inclined to diffuse to voids and promote the growth of voids at intermediate temperatures. The impact of carbon addition on the accumulation of defect clusters and solute redistribution in Fe-9Cr alloys during ion irradiation is not clear. This research provided fundamental knowledge on the materials degradation of materials in reactor environments and sheds light on the design of microstructure in radiation resistant materials for advanced nuclear reactors of many types, including light water cooled reactors.

Accomplishments

We have prepared Fe-9Cr with 0.02wt.% C samples for irradiation studies. High temperature and high dose irradiation experiments were performed in the Michigan Ion Beam Laboratory at the University of Michigan. By using electron back-scatter diffraction (EBSD), we identified two phases in the matrix: they are ĝ-ferrite and tempered martensite. Atom probe tomography at the Center for Advanced Energy Studies (CAES) was used to measure the differences in chemistry in both phases. The focused ion beam at the CAES was utilized to make samples for atom probe tomography (APT) analysis and TEM characterization. The results

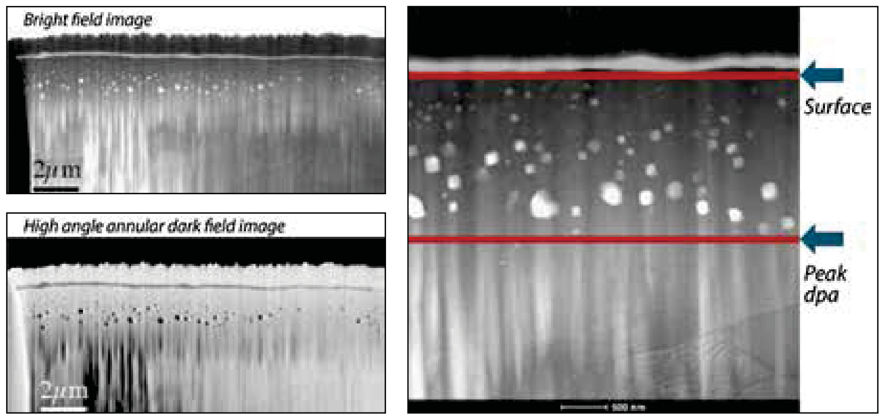

Figure 3. Bright-field and dark-field TEM micrographs of irradiated ĝ-ferrite in Fe-9Cr alloy. Irradiation-induced voids were formed in the damaged region.

indicate that carbon concentration in tempered martensite is lower than that in ĝ-ferrite phase and TEM micrographs of irradiated samples suggest that tempered martensite exhibits a stronger swelling resistance. This might be attributed to the lowered carbon concentration in tempered martensite and/or sink effects of lath boundaries. In addition, we performed in-situ irradiation experiments at the Intermediate Voltage Electron Microscopy (IVEM) Facility at Argonne National Laboratory to understand the influence of carbon addition on the growth kinetics of dislocation loops at 300°C. This research provided a comprehensive understanding on how carbon addition affects the solute redistribution of Fe-9Cr alloy under irradiation at different conditions.

We would like to acknowledge the following institute and individuals for the contribution.

MIBL, University of Michigan, Gary Was.

IVEM, Argonne National Laboratory, Mark Kirk.

SCK CEN, Belgian Nuclear Research Centre, L. Malerba and M. Konstantinovic.

Future Activities

A new proposal based on this research has been submitted to NSUF.

Publications

[1.] C. Sun, et al., "Void swelling of ĝ-ferrite and tempered martensite in a Fe-9Cr model alloy subjected to 8 MeV Fe ion irradiation," 2017 *Environmental Degradation of Materials in Nuclear Power Systems—Water Reactors Conference*, submitted.

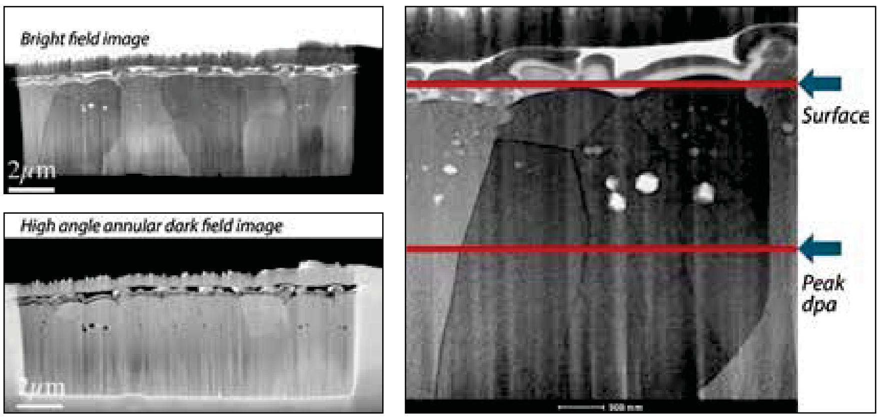

Figure 4. Bright-field and dark-field TEM micrographs of irradiated tempered martensite phase in Fe-9Cr alloy. Irradiation-induced voids were formed in the damaged region. The void density is much lower compared to that in irradiated ĝ-ferrite phase.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Argonne National Laboratory	Stuart Maloy (co-principal investigator)
Idaho National Laboratory	Assel Aitkaliyeva (co-principal investigator)

MaCS at CAES facility has provided opportunities to perform world-class research on nuclear materials.

— Cheng Sun, Postdoc

Analytical Laboratory, MFC, INL

NSUF LIST OF ACRONYMS

AGR .... Advanced Gas Reactor
ANL .... Argonne National Laboratory
ANS .... American Nuclear Society
APT .... Atom Probe Tomography
ASR .... Alkali-Silica Reaction
ATLAS .... Argonne Tandem Linac Accelerator
ATR .... Advanced Test Reactor
BNL .... Brookhaven National Laboratory
BR-2 .... Belgium Reactor-2
CAES .... Center for Advanced Energy Studies
CINR .... Consolidated Innovation in Nuclear Research
CRSS .... Critical Resolved Shear Stress
DOE .... U.S. Department of Energy
DOE-NE .... U.S. Department of Energy Office of Nuclear Energy
dpa .... Displacements Per Atom
EBSD .... Electron Backscatter Diffraction
EDS .... Energy Dispersive X-Ray Spectroscopy
EELS .... Electron Energy Loss Spectroscopy
EML .... Electron Microscopy Laboratory
EOL .... End-of-Life
EPRI .... Electric Power Research Institute
F/M .... Ferritic/Martensitic
FIB .... Focused Ion Beam
FOA .... Funding Opportunity Announcement
FSW .... Friction Stir Welding
FY .... Fiscal Year
GAIN .... Gateway for Accelerated Innovation in Nuclear

GB .... Grain Boundary
HFEF .... Hot Fuels Examination Facility
HIP .... Hot Isostatic Pressing
HQ .... Headquarters
HRTEM .... High-Resolution Transmission Electron Microscopy
HTTL .... High Temperature Test Laboratory
IFP .... Inverse Pole Figure
IML .... Irradiated Materials Laboratory
INL .... Idaho National Laboratory
IVAS .... Interactive Analysis and Visualization Software
IVEM .... Intermediate Voltage Electron Microscopy
IXB .... Ion X-Ray Beam
LEAP .... Localized Electron Atom Probe
LWR .... Light Water Reactor
MA .... Mechanical Alloying
MaCS .... Microscopy and Characterization Suite
MFC .... Materials and Fuels Complex
MIT .... Massachusetts Institute of Technology
MRCAT .... Materials Research Collaborative Access Team
MRS .... Materials Research Society
NEET .... Nuclear Energy Enabling Technology
NEID .... Nuclear Energy Infrastructure Database
NFA .... Nanostructured Ferritic Alloy
NFML .... Nuclear Fuels and Materials Library
NFS .... Nanostructured Ferritic Steels
NSLS-II .... National Synchrotron Light Source II
NSUF .... Nuclear Science User Facilities
NuMat .... Nuclear Materials Conference
ODS .... Oxide-Dispersion-Strengthened
ORNL .... Oak Ridge National Laboratory
PED .... Precession Electron Diffraction
PI .... Principal Investigator
PIE .... Post-Irradiation Examination

PNNL........ Pacific Northwest National Laboratory
RDF........ Radial Distribution Function
RFI........ Request for Information
RIS........ Radiation-Induced Segregation
RPV........ Reactor Pressure Vessel
RTE........ Rapid Turnaround Experiment
SAM........ Sample Library
SD........ Spinol Decomposition
SEM........ Scanning Electron Microscope
SPS........ Spark Plasma Sintering
STEM........ Scanning Transmission Electron Microscopy
TEM........ Transmission Electronic Microscope
TMS........ The Minerals, Metals and Materials Society
TREAT........ Transient Reactor Test
TRISO........ Tristructural Isotropic
TRL........ Technology Readiness Level
XMAT........ Extreme Materials Beam Line

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Irradiated Materials Characterization Laboratory (IMCL), MFC, INL

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