

# **Advanced Post-Irradiation Examination Capability Technology Readiness Assessment**

May 2013

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**May 2013**

**Idaho National Laboratory  
Idaho Falls, Idaho 83415**

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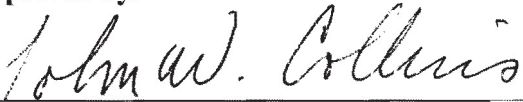


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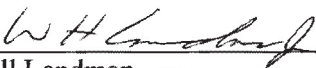
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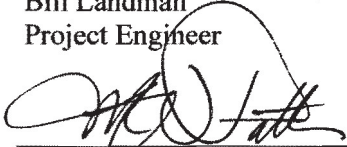
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## EXECUTIVE SUMMARY

This report describes the current status of the Advanced Post-Irradiation Examination Capability (APIEC) Project and the initial complement of post-irradiation examination (PIE) instruments proposed to advance the state-of-the-art for nuclear characterization. To facilitate this advancement, the project performed a technical readiness assessment for the instruments that are most likely to be used for the APIEC Project. This assessment assigned technology readiness levels (TRLs) for the current-state instrument and system readiness.

Technology maturation plans (TMPs) were developed for each of the PIE instruments in the initial complement to detail the next steps required to test the instruments and reduce the risk of installing immature technologies. An additional TMP was developed for the sample preparation equipment needed to prepare nuclear fuels and material specimens for advanced PIE. Technology development roadmaps were then generated to summarize the path forward to successful operation in a facility. The technology development roadmaps, current TRLs, and a brief description of each capability and instrument are contained herein. The nine TMPs are included as attachments to this report.

This assessment included 20 candidate instruments. These instruments ranged in technology readiness from instruments that have been successfully tested as integrated components (TRL-4) to instruments that have been demonstrated as a system in their final configuration. Many of these instruments are commercially available and have been demonstrated in industrial applications. However, some have not been demonstrated for personnel and equipment protection in the high-radiation environment anticipated for the APIEC Project. Hence, these instruments need to be demonstrated in dry inert atmosphere, radiation service (e.g., shielding or relocation of the detectors and printed circuit boards), remote sample handling, and, in some cases, remote control and maintenance of the instrument.

This report describes, qualitatively, the challenges facing each instrument and the path forward to overcome those challenges. As the initial complement of instruments anticipated for near-term APIEC Project application is finalized, more details will be added to the test plans to further quantify the path forward. Updates and additional details also will be added as the instruments advance through successive TRLs.





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## ACRONYMS

APIEC	Advanced Post-Irradiation Examination Capabilities
APT	atom probe tomography
CAES	Center for Advanced Energy Studies
DOD	U.S. Department of Defense
DSC	differential scanning calorimetry
EDS	energy dispersive spectroscopy
EMI	electromagnetic interference
EPMA	electron probe microanalyzer
FIB	focused ion beam
HT XRD	high-temperature x-ray diffraction
IMCL	Irradiated Materials Characterization Laboratory
INL	Idaho National Laboratory
ITU	Institute for Transuranium Elements
LEAP	local electrode atom probe
LFA	laser flash analyzer
MPM	mechanical properties microscope
$\mu$ XRD	micro x-ray diffraction
NASA	National Aeronautics and Space Administration
PAS	positron annihilation spectroscopy
PIE	post-irradiation examination
SEM	scanning electron microscopy
SIMS	secondary ion mass spectrometry
STA	simultaneous thermal analysis
STDM	scanning thermal diffusivity microscopy
TDRM	technology development roadmap
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
TMP	technology maturation plan
TRA	technology readiness assessment
TRL	technology readiness level

# Advanced Post-Irradiation Examination Capability Technology Readiness Assessment

## 1. INTRODUCTION

This document describes the current status of the instruments that will be used to advance the state-of-the-art for nuclear characterization in the Advanced Post-Irradiation Examination Capability (APIEC) Project. To facilitate this advancement, the APIEC Project performed a technical readiness assessment (TRA) for the candidate instruments likely to be used in an advanced post-irradiation examination (PIE) facility. This assessment resulted in assignment of technology readiness levels (TRLs) for the current state of each instrument. Technology development roadmaps (TDRMs) and technology maturation plans (TMPs) were developed for the initial complement of shielded instruments to document the path forward to successful operation in a PIE facility. This document contains the TDRMs, current TRLs, brief system design descriptions for the each capability and instrument, and the TMPs.

### 1.1 Purpose

The purpose of this document is to provide the APIEC Project with the current state of technology readiness for the candidate instruments. It defines a path forward to mature these instruments sufficiently to provide a high degree of confidence in their successful operation in an environment with vibration, electromagnetic interference (EMI), and high-radiation fields. Deployment in this environment will require personnel protection, component protection, and remote operation.

### 1.2 Technology Readiness Assessment

The TRA process was originated by the National Aeronautics and Space Administration (NASA) and the U.S. Department of Defense (DOD). It evaluates the deployment readiness of a technology, as well as its readiness to function in an integrated environment. This project uses the TRA process outlined in Department of Energy Guide 413.3-4, which is consistent with the NASA and DOD processes. This process (see Figure 1) is qualitative at first and was performed on the candidate instruments likely to be employed in the APIEC Project. This report documents the candidate instruments most likely to be used in an advanced PIE facility, their assigned TRLs, the TMPs required to systematically mature each technology, and the integrated TDRMs.

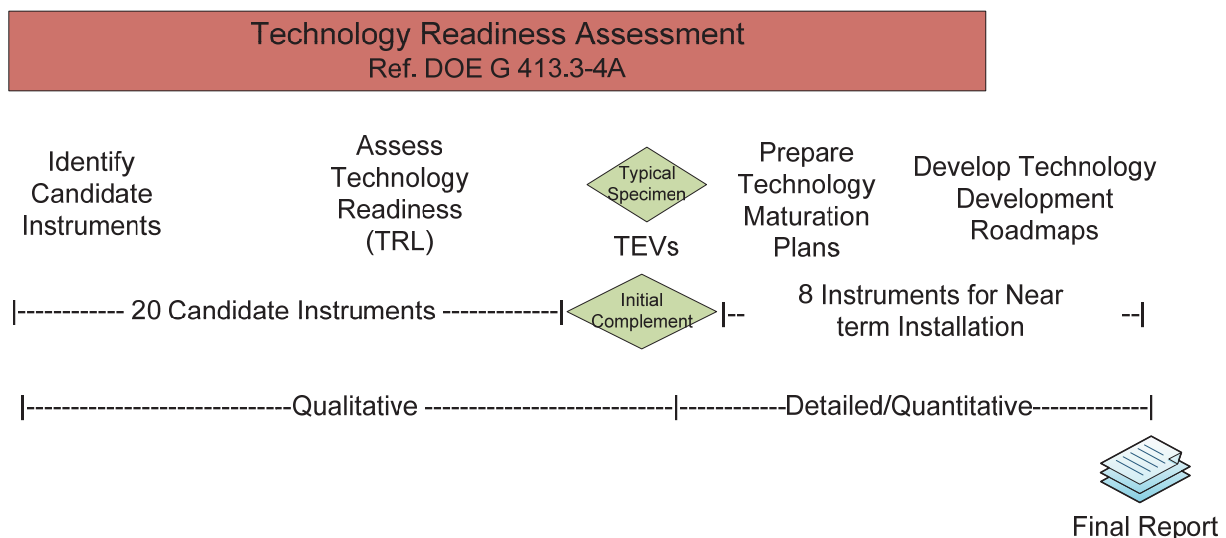


Figure 1. Technology readiness assessment process as applied to the Advanced Post-Irradiation Examination Capability Project.

The TRA is used to inform programmatic decisions concerning technology advancement, technology down-selects, task planning, risk analyses, task prioritization, and allocation of resources. To limit project technical risk, instruments will typically be advanced to TRL-7 (see Section 1.2.1) prior to insertion into the APIEC Project. Project management may decide to insert instruments of lower TRL and accept the attendant technical risk.

Objective evidence from demonstrations and past applications were compiled by subject matter experts for each candidate instrument. After reviewing the evidence, the project team used their expertise and knowledge to determine the validity of the evidence and assign the TRL ratings. The resulting TRL ratings become the TRL baseline for the APIEC Project. For the initial complement of instruments, TMPs were developed to document the demonstrations planned and the performance criteria that must be achieved to advance in technical maturity to the next TRL.

The purpose of this effort was to do the following:

- Establish the candidate instruments for the initial complement of PIE equipment
- Provide a common basis to compare the relative maturity of the candidate instruments
- Establish the baseline TRL ratings
- Establish performance criteria to achieve increasing TRL ratings.

As the instruments are tested in accordance with the TMPs and advance through the TRLs, the test plans and instrument performance will be re-evaluated and the TMPs revised if needed. Further detail will be added to define the performance required to achieve the next successive TRL.

### 1.2.1 Technology Readiness Level Definitions

TRLs are scales that represent the state of technological maturity. This project uses TRLs with a tailored scale of 1 to 9, as suggested in DOE Guide 413.3-4, and similar to those used by NASA and the U.S. DOD. TRL ratings indicate the maturity level of a given instrument and range from 1 (basic principle observed) through 9 (complete complement of instruments is fully operational) (see Figure 2).

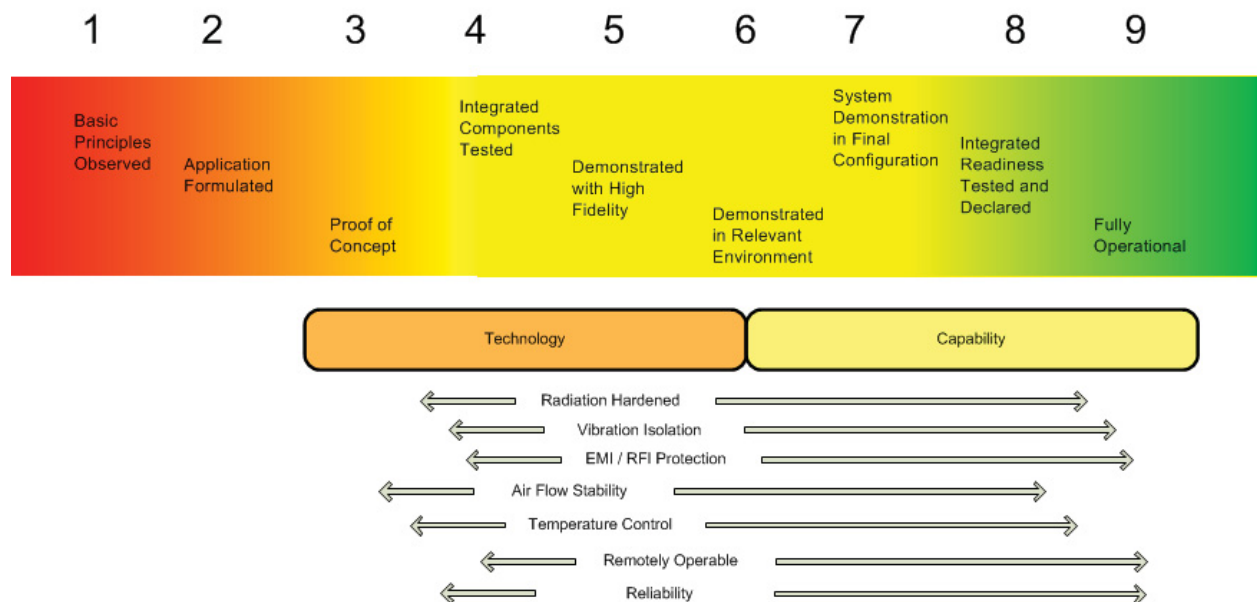


Figure 2. Advancing the technology – overcoming operational issues.

Detailed and abbreviated definitions for each TRL have been adapted specifically for the APIEC Project as suggested in DOE Guide 413.3-4. These adapted definitions are contained in Table 1. Terms used in assessing instrument readiness and their definitions are as follows:

- *EMI Shield* – The electromagnetic and radio frequency interference limitations established by the manufacturer.
- *Isothermal Operation* – The temperature variation limitations established by the manufacturer.
- *Radiation* – The radiation fields the instrument and detector will experience through its normal operation. Radiation includes alpha ( $\alpha$ ), beta ( $\beta$ ), and gamma ( $\gamma$ ) rays likely to be experienced by the detector. To limit instrument degradation due to radiation, the detector and sensitive electronics either will be shielded or located away from the source. For equipment protection, the radiation fields are expressed as “R/hr on contact.”
- *Relevant Environment* – The environment (e.g., temperature, vacuum, inert atmosphere, or flow rate) in which testing occurs becomes increasingly identical to the anticipated environment.
- *Reliability, Availability, Maintainability* – A measure of the instrument’s ability to operate without failure.
- *Remote Operation* – The ability to use the instrument with the operator shield from the high-radiation field or with the human interface removed from the high-radiation field.
- *Remote Sample Handling* – The ability to remotely (if required for human protection) prepare, transport, and insert the sample specimen into the instrument chamber, as required by the manufacturer. For human, protection, the radiation field is expressed as “R/hr at 1 meter.”
- *Vibration* – The vibration limitations established by the manufacturer.

Table 1. Technology readiness level definitions and abbreviations.

Rating Level	TRL Definition	TRL Abbreviated Definition
1	<b>Basic principles observed and reported</b> – Scientific research begins translation to applied research and development; lowest level of technology readiness. Examples might include paper studies of a technology’s basic properties.	Basic principles observed
2	<b>Technology concept and application formulated</b> – Invention begins. Once basic principles are observed, practical applications can be invented. Applications are speculative and there may be no proof or detailed analysis to support the assumptions. Examples are limited to analytic studies.	Application formulated
3	<b>Proof-of concept</b> – Active research and development is initiated. This includes analytical and laboratory studies to physically validate analytical predictions of separate elements of the technology. Examples include components that are not yet integrated or representative.	Proof of concept
4	<b>Basic technological components are integrated</b> – Basic technological components are integrated to establish that the pieces will work together in a laboratory environment.	Integrated components are tested
5	<b>Demonstrated with high reliability</b> – The basic technological components are integrated with reasonably realistic supporting elements and tested in an environment that does not yet include vibration, EMI, temperature, radiation, or remote operation. Examples include integration of components with reliable operation.	Demonstrated with high reliability

Rating Level	TRL Definition	TRL Abbreviated Definition
6	<b>Demonstrated in relevant environment</b> – Representative model or instrument system is tested in a relevant environment (including vibration, EMI, and temperature but in low or no beta/gamma radiation fields). Examples include testing an instrument in a simulated operational environment with high reliability.	Demonstrated in relevant (but low or non-radiation) environment
7	<b>Integrated system demonstrated in final configuration</b> – Actual instrument has been successfully operated in final configuration and in relevant environment (including radiation and remote operation). Represents a major step up from TRL 6, requiring demonstration of an actual system in a relevant operational environment with limited degradation due to radiation exposure.	System demonstration in final configuration
8	<b>Integrated readiness of the system is demonstrated</b> – Technology is proven to work. Actual technology completed and qualified through test and demonstration. Facility issues a declaration of readiness and readiness activity is successfully completed.	Integrated readiness tested and declared
9	<b>Fully operational</b> – Actual application of technology is in its final form. The full complement of instruments is proven through successful operation in the intended facility.	Fully operational

In the APIEC Project, instruments that are commercially available for non-nuclear applications are typically at TRL-5, because the requirements of the lower TRLs (i.e., proof of concept) are proven. Once the instrument has been proven in a relevant environment that includes vibration, EMI, and temperature, the instrument would be considered for TRL-6. To achieve TRL-7, the instrument must have been demonstrated in its final configuration in a relevant environment that includes anticipated radiation fields, sample specimen handling with needed personnel and equipment protection features, and remotely operable, as necessary. The TRL-7 further requires reliable operation with adequate availability and maintainability commensurate with that required in a production facility versus a laboratory setting. No instrument or group of instruments advances beyond TRL-7. The APIEC Project will achieve TRL-8 after completing final design, construction, and system operability testing. Once the facility declares readiness and the readiness activity is successfully completed, the facility and initial complement of instruments is granted TRL-8. TRL-9 is achieved by the facility after months of successful operation.

### 1.3 Technology Maturation Plans

To systematically mature the candidate instruments and increase their robustness in the anticipated operating environment, most of the instruments will need to undergo selected modifications. Modifications to these commercial off-the-shelf instruments include locating sensitive components out of the high-radiation fields; remotely locating rotating or vibration-producing components; constructing with suitable materials due to radiation fields, contamination, and dry inert atmospheres; shielding for radiation-sensitive materials and devices; integrating features in the design specific to remote installation, operation, and maintenance. Once modified, the instruments need to be tested to assure robust operability in the environment to which they will be subjected. The TMPs (attached to this report) provide the steps necessary to demonstrate operability, achieve TRL-7, and contribute to the facility receiving TRL-8. Typically, this involves instrument procurement actions, redesign, fabrication, and equipment qualification, which is divided into conceptual modifications to commercial off-the-shelf instruments (Idaho National Laboratory [INL] with vendor support); sole source or competitive bid instrument specification for the advanced PIE facility (INL with vendor support); and commercial off-the-shelf instrument redesign with vendor instrument fabrication (vendor):

- Phase I Equipment Qualification – Equipment checkout (may be done at vendor location):



- Mechanical Assembly – Assemble components and verify functional fit and intended function(s)
- Electrical Assembly – Assemble cabinet and components, including in-cell sensors and cabling; connect to hardware; and perform initial checks
- Control System Checkout – Software checkout, internal diagnostics, and IO control response checks.
- Phase II Equipment Qualification (Out-of-Cell Functional Testing) – Mockup testing by INL with vendor support (if applicable) could involve instrument and/or hot cell and/or confinement box modifications (may be performed at INL’s Center for Advanced Energy Studies [CAES], Irradiated Materials Characterization Laboratory [IMCL], or other suitable facility):
  - Cold Mockup Testing:
    - Installation and Assembly Testing – Verification of ability to transfer equipment into hot cell and remotely assemble (if appropriate) and verify instrument interfaces (fit/layout/clearances) with modular shielded cell and confinement box (inert atmosphere and alpha tight enclosure) as mocked up
    - Manual Remote Operations Testing – Verification of manipulator-assisted operations and operations utilizing in-cell handling systems (e.g., master-slave manipulators, electromechanical manipulator, and hoists), including operator viewing as mocked up
    - Automatic Operations Testing – Verification of automated machine sequencing (e.g., specimen autoloaders), motion profiles, data collection, and calibrated/acceptable results
    - Testing of Features Related to Equipment Maintenance – Verification of the ability to maintain equipment remotely, including component replacement, if applicable, as mocked up.
  - Hot Mockup Testing:
    - Operations Testing – Verification of equipment performance in the relevant radiation field as mocked up (e.g., with temporary shielding, master-slave manipulators, shield windows, and pass-throughs).
- Phase III (In-Cell System Operational and Acceptance Testing) – Final testing by INL in actual/real shielded enclosure located in the advanced PIE facility:
  - Cold Demonstration Testing:
    - Installation and Assembly Testing – Verification of ability to transfer equipment into hot cell and remotely assemble (if appropriate) and verify instrument interfaces (fit/layout/clearances) with modular shielded cell and confinement box (e.g., inert atmosphere and alpha tight enclosure)
    - Manual Remote Operations Testing – Verification of manipulator-assisted operations and operations utilizing in-cell handling systems (e.g., master-slave manipulators, electromechanical manipulator, and hoists), including operator viewing
    - Automatic Operations Testing – Verification of automated machine sequencing (e.g., specimen autoloaders), motion profiles, data collection, and calibrated/acceptable results
    - Testing of Features Related to Equipment Maintenance – Verification of the ability to maintain equipment remotely, including component replacement, if applicable.
  - Hot Demonstration Testing (after operational readiness review authorization to “go hot”):
    - Operations Testing – Verification of equipment performance in relevant radiation field.

## 1.4 Technology Development Roadmaps

In the APIEC Project, TDRMs provide the framework and structure required to systematically perform decision analysis, reduce risk, and mature technologies in a cost-effective and timely manner. The process includes instrument identification, TRA, TRL validation, technology maturation, and test plan development. The final product is a graphical representation of that process.

A TDRM documents the tasks needed to obtain information in key discriminating criteria to support technology down selection and the tasks and tests required to sufficiently mature the technology and enhance project performance. The set of TDRMs, along with their associated documentation, represents the path forward for this project to complete its mission. The mission is to design, build, and equip an advanced PIE laboratory capable of supporting the DOE Office of Nuclear Energy's science-based approach and research and development objectives, including:

- Develop technologies and other solutions that can improve the reliability, sustain the safety (e.g., accident tolerant fuels), 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.

This document contains the TDRMs that facilitate risk-informed decision making, the path forward, and technology down selection; manage uncertainty, technology qualification, and maturation while serving to coordinate engineering, research, and development; and mitigate the risk early in the project.

This process provides the following benefits to the APIEC Project:

- Identifies precise project objectives and focuses resources on critical technologies that are needed to meet those objectives
- Creates a consensus vision of APIEC Project needs based on capabilities required now and in the future
- Provides early identification of high-risk items and allows early focused attention to reduce cost overruns and schedule delays later in the project.
- Supports engineering and R&D priorities, schedule development, and assignment of resources
- Sets the vision for and drives the needed actions to down select technologies and designs
- Ensures technology readiness is demonstrated through testing, modeling, piloting, and prototyping
- Develops the test plans required to provide demonstrable evidence of the technology maturation required for qualification.

As the TDRM is fully executed, objective evidence will be provided to justify advancement along the TRL scale shown in Figure 2. In some cases, instrument operation in INL's IMCL demonstrates readiness for APIEC installation. In other cases, demonstration locations are unknown and will be determined as priorities dictate. As more is learned about the instrument's use in other radiation and non-radiation environments, both domestically and internationally, adequate justification may be obtained to advance the TRL without further demonstration. In all cases, objective evidence of the instrument's maturity associated with operation in a relevant environment, including vibration, EMI, and radiation issues, will be reviewed and accepted prior to the advancements.

## 2. ADVANCED POST-IRRADIATION EXAMINATION CAPABILITY

Advanced nuclear fuel development requires a detailed understanding of irradiation effects on nuclear fuel and material performance. Understanding the behavior of fuels and materials in a nuclear reactor irradiation environment is the limiting factor in improved nuclear plant safety, longevity, efficiency, and economics. To support the science-based development of advanced nuclear fuels and materials, including accident-tolerant fuels, the nation needs comprehensive, consolidated, state-of-the-art PIE capabilities for highly activated nuclear fuels and non-fuel materials. Providing the PIE instruments, sample preparation, facility, and support functions is the focus of the APIEC Project assessed in this document.

### 2.1 Initial Complement of Shielded Instruments

The following subsections provide an assessment of the technology readiness of the candidate instruments planned as the initial complement of shielded instruments for the new advanced PIE laboratory. These state-of-the-art materials science analytic tools represent the top eight priority capabilities (out of 24 originally considered) for deployment for advanced PIE. TEV-1722, “APEX Equipment List,” documents the process used to rank these instruments by importance based on stakeholder and researcher input.

It should be noted that the transmission electron microscope (TEM) and atom probe tomography (APT) microscope also are priority instruments. However, they require such small specimens that the associated dose rates are low and these two instruments are planned for installation in the new facility’s non-shielded instrument spaces. As such, their installation and subsequent use are not anticipated to be significantly different from typical laboratory applications of those instruments. A description of these instruments is provided in Section 2.2.

#### 2.1.1 Electron Probe Microanalysis

An electron probe microanalyzer (EPMA) is an analytical tool used to non-destructively determine the chemical composition of small volumes of solid materials. It works similarly to a Scanning electron microscope (SEM) in that the sample is bombarded with an electron beam, emitting x-rays at wavelengths characteristic of the elements being analyzed. This allows operators to quantify the elements present.<sup>[21]</sup> The concentrations of elements from boron to curium can be measured at levels as low as 100 parts per million.

##### 2.1.1.1 Instrument Design Description.

- Functions Performed – EPMA is used to do the following:
  - Characterize compositional homogeneity of as-fabricated, actinide-bearing transmutations fuels
  - Analyze fuel constituent migration in irradiated fuels
  - Quantify radial distributions of fission products such as rare earths
  - Characterize fracture surfaces of irradiated materials using electron imaging capability
  - Measure the localized micro-scale chemical composition of whole transverse cross-sections of irradiated fuels and materials
  - Perform imaging of these samples.<sup>[11]</sup>
- Existing Applications

EPMA is commonly used to analyze the chemical composition of metals, alloys, ceramics, and glasses, and is widely used for research, quality control, and failure analysis. It also is used by mineralogists and petrologists. Most rocks are aggregates of small mineral grains. This technique also is used for the study of extraterrestrial rocks (i.e., meteorites) and provides chemical data that are vital to understanding the evolution of the planets, asteroids, and comets.

### 2.1.1.2 Technology Readiness Level Status.

- Nuclear Applications

EPMA currently is in use at INL's Analytical Laboratory for fresh nuclear fuel analysis. Current shielded hot cell applications include Knolls Atomic Power Lab, Commissariat à l'énergie atomique Cadarache, SCK-CEN Belgium, Institute for Transuranium Elements (ITU), and Paul Scherrer Institute. At least one manufacturer makes an EPMA that is specifically designed for irradiated materials. To that end, the detectors and electronics have been shielded for specimens up to 3 Ci of Ce-137. These applications have demonstrated the use of shielded EPMA with remote operation for civilian nuclear power and post-irradiation analysis.<sup>[22,23]</sup>

- Readiness Rating in Relevant Environment

The industrial and nuclear applications incorporating EPMA to-date are sufficient to rate the instrument TRL-7 (Table 2).

Table 2. Electron probe microanalysis technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Electron Probe Microanalysis (EPMA)	7	7	7	7	7	7	7	7	7.0

**2.1.1.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Successful relocation of the INL's EPMA to IMCL (scheduled for FY 2014) and subsequent implementation of, and operation in, a shielded environment (scheduled for FY 2015)<sup>[48]</sup>
- Specification and procurement of a new EPMA specifically adapted for deployment in one of the APIEC facility's shielded cells including appropriate interfaces for the inert atmosphere glovebox, remote sample handling, and remote instrument operation.

## 2.1.2 Secondary Ion Mass Spectrometer

The secondary ion mass spectrometer (SIMS) is used to analyze the composition of solid surfaces and thin films by sputtering the surface of the specimen with a focused primary ion beam and collecting and analyzing ejected secondary ions. These secondary ions are measured with a mass spectrometer to determine the elemental, isotopic, or molecular composition of the specimen's surface.<sup>a</sup>

### 2.1.2.1 Instrument Design Description.

- Functions Performed – The SIMS is used to do the following:
  - Identify trace elements, organic molecules, and polymers on surfaces, with better than 1 part per million sensitivity for some molecules and part per billion sensitivity for some elements

<sup>a</sup> The NanoSIMS instrument from CAMECA was evaluated as a possible alternative to the SIMS for APIEC implementation. NanoSIMS is a very sensitive surface analysis technique capable of detecting elements present in the parts per billion range. NanoSIMS creates nanoscale maps of elemental composition, combining the high-mass resolution, isotopic identification, and subparts-per-million sensitivity of conventional SIMS with spatial resolution down to 50 nm, and can identify up to seven masses in parallel from the same small volume. In energy research, NanoSIMS can characterize the nanostructured materials with complex compositions that are increasingly important candidates for energy generation and storage.<sup>[24]</sup> However, the NanoSIMS instrument was not deemed mature enough to be included as a candidate for the APIEC facility's initial complement of equipment.

- Conduct surface chemical imaging with better than 200-nm resolution
- Provide extremely surface-sensitive information from the top 1 nm of a sample in static SIMS mode
- Show three-dimensional elemental/molecular distribution, with better than 50-nm depth resolution in depth profiling mode.
- Existing Applications  
Applications include the following:
  - Analysis of complex molecules and organics, including wool fibers, biomaterials, and drug delivery systems
  - Improvement of the performance of organic light-emitting diode displays and solar cells by characterizing surface and interfacial chemistry of organic layers and identifying contamination
  - Depth profiling of control release coatings of drugs on arterial stents used in heart surgery to measure drug distribution and migration
  - Investigation of how conditioners stick to hair and correlating with other physical properties, such as friction.

### 2.1.2.2 Technology Readiness Level Status.

- Nuclear Applications  
The shielded CAMECA IMS-6F double focusing SIMS was installed at Commissariat à l'énergie atomique Cadarache, France, in 1999. The same unit was acquired by ITU in 2003, which permits analysis of nuclear fuel samples with a gamma activity up to 75 GBq (2 Ci).<sup>[25,26]</sup>
- Readiness Rating in Relevant Environment  
The current readiness of a SIMS used for examining irradiated nuclear fuels and materials is TRL-7 (Table 3). This assessment assumes the use of a 7F SIMS similar to those used at Commissariat à l'énergie atomique Cadarache and ITU, in which high-radiation field applications have been demonstrated. Vibration and EMI issues also have been satisfactorily resolved.

Table 3. Secondary ion mass spectrometer technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Secondary Ion Mass Spectrometer (SIMS)	7	7	7	7	7	7	7	7	7.0

**2.1.2.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Integration with design activities to address vibration, EMI, and radiation challenges
- Understanding the currently high-radiation environment applications at existing facilities, and design and install the 7F SIMS in the APIEC Project.<sup>[3]</sup>

### 2.1.3 Simultaneous Thermal Analysis

Simultaneous thermal analysis (STA) combines a thermogravimetry analyzer (TGA) and differential scanning calorimetry (DSC) for successful routine application in advanced PIE of nuclear fuels and materials.

TGA is a type of testing that is performed on samples to determine changes in mass as a function of temperature or time in a controlled atmosphere. The principle uses for TGA are to study a material's thermal stability and composition. This analysis method is commonly coupled with a DSC or Differential Thermal Analysis instrument to provide a simultaneous thermal measurement curve containing all three data inputs.

DSC is a thermal analysis technique in which the difference in the amount of heat required to change the temperature of a sample and reference material at the same rate is measured as a function of temperature. Both the sample and reference are maintained at the same temperature throughout the experiment. The main applications of DSC are studying material properties (such as phase transformation melt temperatures, glass transitions, crystallization, product stability, oxidative stability, cure kinetics, and the associated reaction enthalpies). Additionally, DSC can be used to measure the heat capacity and calculate the specific heat of a wide variety of materials, including metals, alloys, ceramics, and composites.

### **2.1.3.1 Instrument Design Description.**

- Functions Performed

TGA is used to do the following:

- Determine mass change as temperatures vary
- Determine mass change as chemical reaction occurs under varying temperature or isothermal conditions.

DSC is used to do the following:

- Determine transition temperatures
- Determine heat of reaction at phase transitions
- Determine heat capacity of a material
- Determine kinetics of reactions.

- Existing Applications

The technique is widely used across a range of applications, both as a routine quality test and as a research tool. These applications include the following:

- *Thermal Conductivity* – Heat capacity determined from STA, combined with thermal diffusivity and density, gives thermal conductivity of a material.
- *Phase Diagram Determination* – STA is used to study the temperatures at which materials transition between phases. By varying the time and temperature profiles of STA measurements, data are collected that can be used to determine equilibrium phase diagrams and continuous cooling temperature diagrams.
- *Polymers* – STA is used widely for examining polymers for material properties. Melting points and glass transition temperatures are commonly measured properties for most polymers. The method also can show polymer degradation, stability temperatures, and curing processes.
- *Oxidative Stability* – STA is used to study the stability of a material to oxidation, which leads to determination of optimum storage conditions for a material or compound.

STA is widely used in materials research applications in a large number of industries (such as tools manufacturing, semiconductor technology, battery safety, food technology, explosives manufacturing, metallurgy, cosmetics, textiles, and energy companies, including petroleum and coal). Other applications include quality control, testing and catalysis, and explosives development.



### 2.1.3.2 Technology Readiness Level Status.

- Nuclear Applications

TGA is currently installed in the Analytical Laboratory and is used for  $\alpha$ -contaminated materials.

Current nuclear applications for DSC exist in INL's Analytical Laboratories A102 and B127 for fresh fuel  $\alpha$  materials and in the Fuels and Applied Sciences Building for highly enriched uranium and low-field  $\beta\gamma$  materials.

- Readiness Rating in Relevant Environment

The current readiness of STA used for examining irradiated nuclear fuels and materials is TRL-6 (Table 4) due to the need to demonstrate radiation-hardened operation.

Table 4. Simultaneous thermal analyzer technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Simultaneous Thermal Analyzer	6	7	7	7	7	7	7	6	6.9

**2.1.3.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Demonstrate alpha/inert atmosphere confinement enclosure design and interfaces as well as modifications for remote operability and maintainability in Analytical Laboratory B127 fresh fuel glovebox
- Work with manufacturer of STA to identify any additional radiation-sensitive components and approaches for radiation hardening<sup>b</sup>
- Procure and verify functionality, remote sample handling, and remote operability of the radiation-hardened instrument in IMCL (i.e., relevant laboratory environment) using a sealed radiation source external to the furnace (but near the instrument) instead of an irradiated specimen
- Relocate the radiation-hardened STA instrument from IMCL to the APIEC facility and perform testing in relevant environment including the use of irradiated specimens (i.e., hot testing).

### 2.1.4 Laser Flash Analyzer

The laser-flash analyzer (LFA) is a technique for measuring the thermal transport properties of thin-film coatings and bulk solid and liquid samples. In this technique, one face of a sample is heated by a short unfocused laser pulse, and an Infrared detector monitors the temperature rise of the opposite side of the sample.<sup>[41]</sup> In many cases, reliable measurement of thermal diffusivity can be obtained through the pulse laser flash technique. The LFA instrument can acquire data automatically from cryogenic temperatures up to 2500°C. The pulse LFA method is used in the study of microstructural factors affecting the thermal transport properties of materials.

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<sup>b</sup> The term "radiation hardening," as used in this report, is the act of making electronic or other sensitive components and systems resistant to, or otherwise protecting them from, damage or malfunctions caused by ionizing radiation (specifically alpha, beta, and gamma radiation) as expected to be encountered during examination or characterization of irradiated nuclear fuels and materials.

#### 2.1.4.1 Instrument Design Description.

- Functions Performed – LFA is used to do the following:
  - Identify microstructural factors affecting the thermal transport properties of materials such as thermal diffusivity
  - Quantify thermal conductivity as a function of temperature by utilizing the measured thermal diffusivity, density, and specific heat of a material.
- Existing Applications

Thermal diffusivity is the main transport property that is measured with the LFA method. The thermal conductivity of samples of ceramic, glass, metals, melts and liquids, powders, fibers, and multi-layer materials ranging from vacuum insulation panels to diamonds can be determined by taking the product of thermal diffusivity, density, and specific heat.

#### 2.1.4.2 Technology Readiness Level Status.

- Nuclear Applications

Currently, LFA is equipped with a furnace, which allows the thermal diffusivity to be measured over a range from 0.001 to 10 cm<sup>2</sup>/s and as a function of temperature between 0 to 1500°C. It is installed in the Analytical Laboratory for  $\alpha$  material and in the Fuels and Applied Sciences Building for low-field  $\beta\gamma$  materials.

- Readiness Rating in Relevant Environment

The current readiness of LFA used for examining irradiated nuclear fuels and materials is TRL-6 (Table 5) due to the need to demonstrate radiation-hardened, remote operability and remote sample handling.

Table 5. Laser flash analyzer technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Laser Flash Analyzer (LFA)	6	7	7	7	6	6	7	6	6.6

**2.1.4.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Demonstrate alpha/inert atmosphere confinement enclosure design and interfaces as well as modifications for remote operability and maintainability in Analytical Laboratory B127 fresh fuel glovebox
- Work with manufacturer of LFA to identify any additional radiation-sensitive components and approaches for radiation hardening
- Procure and verify functionality, remote sample handling, and remote operability of the radiation-hardened instrument in IMCL (i.e., relevant laboratory environment) except using a sealed radiation source external to the furnace (but near the instrument) instead of an irradiated specimen
- Relocate the radiation-hardened LFA instrument from IMCL to the APIEC facility and perform testing in relevant environment including the use of irradiated specimens (i.e., hot testing).



### 2.1.5 Nanoindenter

A nanoindenter (see Figure 3) is the main component for indentation hardness tests used in nanoindentation. Since the mid 1970s, nanoindentation has become the primary method for measuring and testing very small volumes of mechanical properties. Nanoindentation, also called *depth sensing indentation* or *instrumented indentation*, gained popularity with development of machines that could record small load and displacement with high accuracy and precision.<sup>[44,45]</sup> The load displacement data can be used to determine modulus of elasticity, hardness, yield strength, fracture toughness, scratch hardness, and wear properties.<sup>[46]</sup>

#### 2.1.5.1 Instrument Design Description.

- Functions Performed – The nanoindenter is used to do the following:
  - Measure and test mechanical properties at nano scale
  - Provide highly precise load displacement data needed to determine modulus of elasticity, hardness, yield strength, fracture toughness, scratch hardness, and wear properties
  - Yield relation of mechanical properties on a very small length scale.

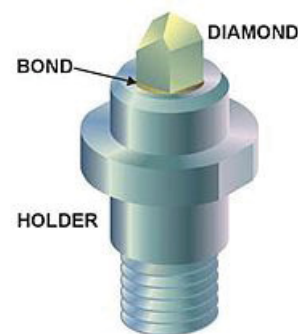


Figure 3. Nanoindenter.

- Existing Applications

Nanoindenters are made by several manufacturers, including Agilent Technologies, Oxford Instruments, and CSM Instruments. Applications of these instruments include semiconductor industry (e.g., thin films and wafers), hard coatings, composite materials, fibers, polymers, metals, and ceramics.

#### 2.1.5.2 Technology Readiness Level Status.

- Nuclear Applications

A nanoindenter (Hysitron Tribo Indenter [TI-950]) currently is in use at CAES at INL.

- Readiness Rating in Relevant Environment

The current readiness of the nanoindenter used for examining irradiated nuclear fuels and materials is TRL-6 (Table 6) due to the need to demonstrate remote operability and remote sample handling.

Table 6. Nanoindenter technology readiness level.<sup>c</sup>

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Nano-Indenter	7	7	7	7	6	6	7	6	6.7

**2.1.5.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through design and installation of the nanoindenter in the APIEC Project.<sup>[3]</sup>

<sup>c</sup> Subsequent data obtained during development of the Technology Maturation Plan for the nanoindenter indicates that the assessment of the radiation hardening attribute (i.e., TRL-7) may be overestimated primarily due to the electronic components that measure the indentation response. Additional development activities for radiation hardening will likely be required.

### 2.1.6 Focused Ion Beam

Focused ion beam (FIB) systems have been in commercial operation for approximately 20 years, primarily for large semiconductor manufacturers. FIB systems operate in a similar fashion to SEM except that, rather than a beam of electrons and as the name implies, FIB systems use a finely focused beam of ions (usually gallium). FIB can be operated at low-beam currents for imaging or high-beam currents for site-specific sputtering or milling.

#### 2.1.6.1 Instrument Design Description.

- Functions Performed – The FIB is used to do the following:
  - Imaging resolution at micro and nano scale (2.5 to 6 nm) when operated at low-beam currents
  - Machining resolution at micro and nano scale (10 to 15 nm) when operated at high-beam currents
  - Sample preparation for other imaging devices.
- Existing Applications

FIB is a technique used particularly in the semiconductor industry, materials science applications, and, increasingly, in the biological field for site-specific analysis, deposition, and ablation of materials.<sup>[1,2]</sup> FIB often is used in the semiconductor industry to patch or modify an existing semiconductor device. For example, in an integrated circuit, the gallium beam could be used to cut unwanted electrical connections or to deposit conductive material to make a connection. The high level of surface interaction is exploited in patterned doping of semiconductors. FIB also is used for maskless implantation.

#### 2.1.6.2 Technology Readiness Level Status.

- Nuclear Applications

FIB (see Figure 4 and Figure 5) has been demonstrated as a sample preparation instrument to allow detailed and effective examination of irradiated fuel under a TEM.<sup>[4]</sup> Current operation in the Electron Microscopy Laboratory has indicated degradation in the energy dispersive spectrometer (EDS) crystal and electronics from 8 years of operation in a non-radiation environment to approximately 3 years when operated in a radiation environment at about 8 R/hour  $\gamma$  on contact. Degradation of this magnitude is not experienced if using a wavelength dispersive spectrometer rather than an EDS, since the wavelength dispersive spectrometer detectors are not in direct exposure to the radiation.

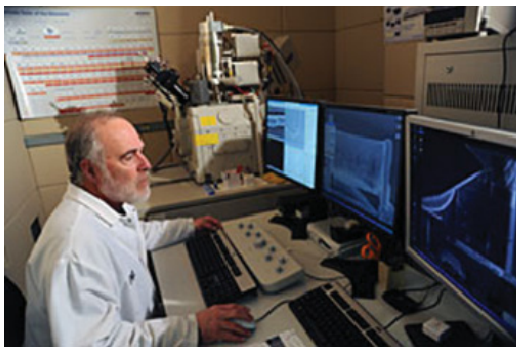


Figure 4. A focused ion beam, shown, enables close examination of irradiated fuel using a transmission electron microscope.

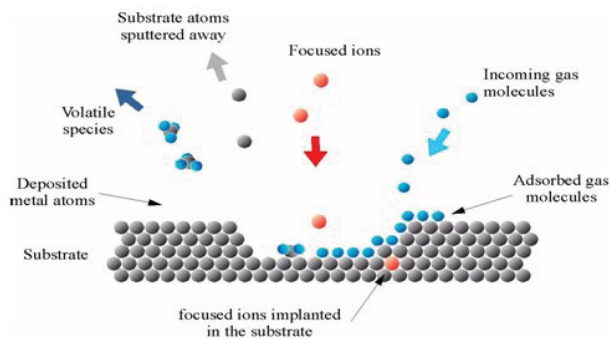


Figure 5. Focused ion beam technology now enables Idaho National Laboratory researchers to examine nuclear fuel samples that are just a few atoms thick.

- Readiness Rating in Relevant Environment

The industrial and nuclear applications that implement the FIB to-date are sufficient to rate the instrument at TRL-6 (Table 7). Initial operation in the Electron Microscopy Laboratory in a radiation environment has experienced electronic sensor degradation.

Table 7. Focused ion beam technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Focused Ion Beam (FIB)	6	7	7	7	6	6	7	6	6.6

**2.1.6.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Continue to collect operability and maintainability data for FIB in the Electron Microscopy Laboratory in associated radiation environment (i.e., up to 130 R/hour  $\alpha\beta$  and 8 R/hour  $\gamma$  on contact) to further understand the effects on EDS crystals and electronics. This experience also applies to the SEM, which uses similar EDS.
- Design and procure a new FIB (FY 2013) for installation in IMCL (scheduled for FY 2014) and subsequent implementation of shielded enclosure (scheduled for FY 2015).<sup>[48]</sup> Demonstrate remote operability and functionality in IMCL in a relevant radiation environment. It is anticipated that the IMCL FIB will be used for its sample preparation capability only and will not include the EDS and WDS analytical suite. Apply this learning to design and specification of the APIEC FIB.

## 2.1.7 Scanning Electron Microscope

SEM is a type of electron microscope that images a sample by scanning it with a beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample-producing signals that contain information about the sample's surface topography, composition, and other properties, such as electrical conductivity.

### 2.1.7.1 Instrument Design Description.

- Functions Performed – The SEM is used to do the following:

- Determine composition by EDS<sup>[10]</sup> and wavelength dispersive spectroscopy
- Identify phases using crystal structure information by electron backscatter diffraction.

- Existing Applications

The SEM has many applications across a multitude of industry sectors. It can produce extremely high magnification images (up to 200000x, see Figure 6) at high resolution up to 2 nm, combined with the ability to generate localized chemical information. This means SEM is a powerful and flexible tool for solving a wide range of product and processing problems for a diverse range of metals and materials.<sup>[10]</sup>

Typical applications include the following:

- Metals and materials identification
- Particle contamination identification
- Materials classification
- Product and process failure and defect analysis

- Examination of surface morphology (including stereo imaging)
- Analysis and identification of surface and airborne contamination
- Powder morphology, particle size, and analysis
- Welding and joining technology quality evaluation and failure investigation
- Paint and coating failure and delamination investigation
- Identification and elimination of corrosion and oxidation problems.

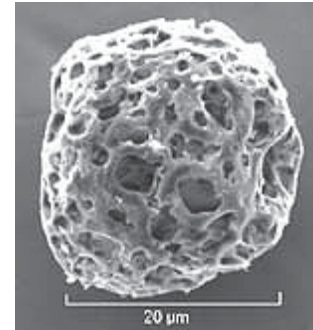


Figure 6. Extremely high magnification image produced by scanning electron microscope.

### 2.1.7.2 Technology Readiness Level Status.

- Nuclear Applications

SEM is used in fuel applications in studying fuel cladding chemical interaction. Elements from beryllium through curium are studied with full matrix correction.<sup>[11]</sup>

- Readiness Rating in Relevant Environment

The current readiness of a SEM used for examining irradiated nuclear fuels and materials is TRL-6 (Table 8) due to the need to demonstrate radiation hardness.

Table 8. Scanning electron microscope technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Scanning Electron Microscope (SEM)	6	7	7	7	7	7	7	6	6.9

**2.1.7.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Continue to collect operability and maintainability data for the SEMs located in Electron Microscopy Laboratory and the irradiation-assisted stress corrosion cracking hot cell to better understand the effects of cumulative radiation damage on these instruments and for the respective radiation fields.
- Relocate the SEM Model JSM-7600F from the Fuels and Applied Science Building to IMCL (scheduled for FY 2015) with subsequent implementation of a shielded enclosure (FY 2017) to allow use on high activity or hazardous materials.<sup>[48]</sup> Demonstrate remote operability and functionality in IMCL in a relevant radiation environment and monitor radiation degradation to the EDS crystal and other electronics. Apply this learning to the design and specification of the APIEC SEM.

## 2.1.8 Micro X-ray Diffraction

Micro x-ray diffraction ( $\mu$ XRD) is a structural analysis technique that allows for examination of very small sample areas. Like conventional XRD instrumentation,  $\mu$ XRD relies on the dual wave/particle nature of x-rays to obtain information about the structure of crystalline materials. Unlike conventional XRD, which has a typical spatial resolution ranging in diameter from several hundred micrometers up to several millimeters,  $\mu$ XRD uses x-ray optics to focus the excitation beam to a small spot on the sample surface so that small features on the sample can be analyzed<sup>[5]</sup> (see Figure 7).

### 2.1.8.1 Instrument Design Description.

- Functions Performed –  $\mu$ XRD is used to do the following:
  - Provide information on the structure of crystalline materials

- Provide spatial phase mapping of the crystalline structure.
- Existing Applications

Applications include characterization of crystalline inclusions within a material, regions of defect within a fabricated sample, reduction spots in ceramics, crystalline defects in glass, filler agglomeration in plastics, reduction spots in electro-ceramics, and layers deposited on a substrate. These applications are found in aerospace, automotive, glass, ceramics and refractories, healthcare, semiconductors, and electronics industries. Using these applications in industry demonstrates that vibration, EMI, and temperature stability issues can be overcome.<sup>[6,8]</sup>

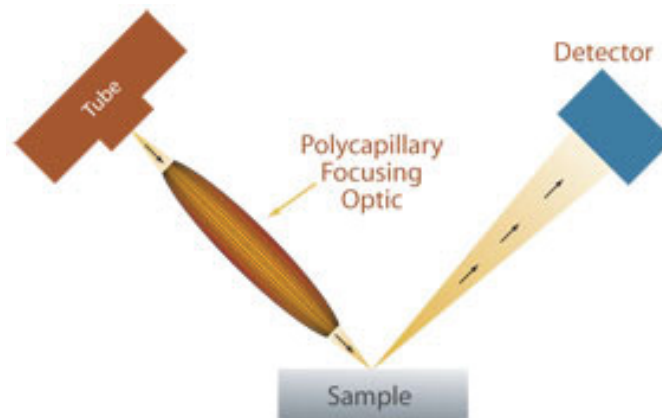


Figure 7. Micro x-ray diffraction operational concept.

### 2.1.8.2 Technology Readiness Level Status.

- Nuclear Application

$\mu$ XRD has been used in the nuclear industry in high radiological areas, which include nuclear waste disposal.<sup>[7]</sup>  $\mu$ XRD currently is operable in INL's Analytical Laboratory for fresh fuel applications with  $\alpha$  contamination and low  $\beta\gamma$  contamination.

- Readiness Rating in Relevant Environment

The current readiness of  $\mu$ XRD used for examining irradiated nuclear fuels and materials is TRL-6 (Table 9) due to the need to demonstrate remote operation.

Table 9. Micro X-ray diffraction technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Micro X-Ray Diffraction (MXRD)	6	7	7	7	6	6	7	6	6.6

**2.1.8.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Demonstrate operability in Analytical Lab.
- Demonstrate operability in an IMCL shielded enclosure for use on high activity or hazard materials.

Table 10 provides a summary of the estimated TRL values for the initial complement of instruments and sample preparation equipment to be deployed in the APIEC facility's shielded cells.

Table 10. Technology readiness levels for initial complement of shielded instruments.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
<b>APIEC - Near Term Instruments</b>		6.5	7.0	7.0	7.0	6.5	6.5	7.0	6.0	6.8
<b>Chemical Analysis</b>		7.0	7.0	7.0	7.0	7.0	7.0	7.0	7	7.0
	Electron Probe Microanalysis (EPMA)	7	7	7	7	7	7	7	7	7.0
	Secondary Ion Mass Spectrometer (SIMS)	7	7	7	7	7	7	7	7	7.0
<b>Thermal Properties</b>		6.0	7.0	7.0	7.0	6.5	6.5	7.0	6	6.7
	Simultaneous Thermal Analyzer	6	7	7	7	7	7	7	6	6.9
	Laser Flash Analyzer (LFA)	6	7	7	7	6	6	7	6	6.6
<b>Mechanical Properties</b>		7.0	7.0	7.0	7.0	6.0	6.0	7.0	6	6.7
	Nano-Indenter	7	7	7	7	6	6	7	6	6.7
<b>Microstructural Analysis</b>		6.0	7.0	7.0	7.0	6.3	6.3	7.0	6	6.7
	Focused Ion Beam (FIB)	6	7	7	7	6	6	7	6	6.6
	Scanning Electron Microscope (SEM)	6	7	7	7	7	7	7	6	6.9
	Micro X-Ray Diffraction (MXRD)	6	7	7	7	6	6	7	6	6.6
<b>Sample Preparation Cell</b>		6.0	7.0	7.0	7.0	6.0	6.0	6.0	6.0	6.4

## 2.2 Other Instruments

### 2.2.1 Scanning Thermal Diffusivity Microscope

The scanning thermal diffusivity microscope (STDM) maps the local thermal diffusivity of a single exposed side of a sample. The probe in the STDM is sensitive to local variations of the thermal diffusivity on a micron scale. Thermal measurements at the micrometer scale are of both scientific and industrial interest, because they provide a means to understand how thermal gradients develop in materials undergoing thermal cycles.

#### 2.2.1.1 Instrument Design Description.

- Functions Performed – The STDM is used to do the following:
  - Map thermal diffusivity of a sample across an exposed surface
  - Quantify diffusivity changes with irradiation.
- Existing Applications

The STDM allows thermal measurements at the microscale. These measurements currently are limited to thermal diffusivity, which, when coupled with results from other instrumentation, allows other thermal properties (such as thermal conductivity and thermal emissivity) to be derived. The applications include the following:

- Ultra-large-scale integration lithography research and cellular diagnostics in biochemistry<sup>[25,28,29,30]</sup>
- Detecting parameters such as phase changes in metallic alloys and ceramics<sup>[31]</sup>
- Measuring material variations in semiconductor devices<sup>[32]</sup>
- Near-field photothermal microspectroscopy<sup>[34]</sup>
- Analyzing enthalpy in integrated circuits.<sup>[37]</sup>



### 2.2.1.2 Technology Readiness Level Status.

- Nuclear Applications

Current nuclear applications exist in INL's Analytical Laboratory for  $\alpha$  materials; a backup unit exists at the INL Research Center.

- Readiness Rating in Relevant Environment

The current readiness of STDM used for examining irradiated nuclear fuels and materials is TRL-6 (Table 11) due to the need to demonstrate remote operability and remote sample handling. Remote operability and remote sample handling are routine in the Analytical Laboratory Hot Cell.

Table 11. Scanning thermal diffusivity microscope technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Scanning Thermal Diffusivity Microscope (STDM)	6	7	7	7	7	7	7	6	6.9

**2.2.1.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Demonstrate remote operability in IMCL.<sup>[3]</sup>
- Design for APIEC Project installation.<sup>[3]</sup>

## 2.2.2 Thermal Arrest Melting Point Determination

The thermal arrest melting point technique is used for high melting point materials (such as ceramics and refractory metals). It uses an induction heating system to either directly or indirectly heat a capsule that is designed to meet the black-body condition past the melting point, while recording the temperature using an optical pyrometer. As the capsule (made of tungsten, with a melt temperature of about 3400°C) is heated and the material inside (e.g., UO<sub>2</sub>) starts to melt, there is a thermal lag in the temperature signal due to the endotherm associated with the phase transformation from solid to liquid.<sup>[42]</sup>

### 2.2.2.1 Instrument Design Description.

- Functions Performed

This instrument is used to determine the melting point and other phase transition temperatures.

- Existing Applications

Considerable effort has been placed on understanding the melting point behavior of UO<sub>2</sub>, (U,Pu)O<sub>2</sub> solid solutions and the effects of irradiation on the melting point followed by subsequent generation of fission products on these fuel materials. Starting in the early 1950s and ending in the mid 1970s, General Electric Company did much of the early work performed by the United States. After that time, the European Atomic Energy Community, the Japan Atomic Energy Agency, and the Korea Atomic Energy Research Institute continued to commit considerable resources to improving the knowledge base for fuel properties and characterization techniques, including melting point determination, allowing for future advances in nuclear fuel and reactor design. This continued commitment to fuels development provides these agencies with a much larger database of information on which to base designs and improvements in the nuclear fuel arena.<sup>[42,43]</sup>

### 2.2.2.2 Technology Readiness Level Status.

- Nuclear Applications

INL currently does not have thermal arrest melting point determination capability for active materials.

- Readiness Rating in Relevant Environment

The current readiness of thermal arrest melting point determination used for examining irradiated nuclear fuels and materials is TRL-5 (Table 12) due to the need to demonstrate remote operability and operability in an environment that overcomes vibration, radiation hardening, and EMI issues.

Table 12. Arrest melting point determination technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Arrest Melting Point Determination	5	6	6	6	5	5	6	5	5.6

**2.2.2.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Integrate with design activities to address vibration, EMI, and radiation challenges
- Design and install the thermal arrest melting point determination capability in the APIEC Project.<sup>[3]</sup>

### 2.2.3 Dilatometer

The dilatometer is an instrument that measures linear thermal expansion. This technique measures the dimension of a material under a negligible load as a function of temperature, while being subjected to a controlled temperature program. Consideration is given to the thermal expansion of the instrument components using a standard reference material as part of the data correction. Materials that can be measured using this technique are metals, alloys, ceramics, and polymers (all in the solid or semi-solid state).

#### 2.2.3.1 Instrument Design Description.

- Functions Performed – The dilatometer is used to do the following:
  - Determine linear thermal expansion as a function of temperature. These data are used to calculate the coefficient of thermal expansion as a function of temperature.
  - Determine volumetric thermal expansion as a function of temperature for use in calculating changes in density.
- Existing Applications

The dilatometer has found uses in a large number of industries (such as manufacturing, semiconductor technology, battery safety, food technology, metallurgy, cosmetics, textiles, and energy companies, including petroleum and coal). Others applications include quality control, testing and catalysis, and development of powder metallurgy techniques.

#### 2.2.3.2 Technology Readiness Level Status.

- Nuclear Applications

A dilatometer is currently installed at INL in the Fuels and Applied Sciences Building for highly enriched uranium and low-field  $\beta\gamma$  materials.

- Readiness Rating in Relevant Environment



The current readiness of a dilatometer used for examining irradiated nuclear fuels and materials is TRL-5 (Table 13) due to the need to demonstrate remote operability and operability in an environment that overcomes vibration and radiation-hardened environmental issues.

Table 13. Dilatometer technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Dilatometer	5	6	6	7	5	5	6	5	5.7

**2.2.3.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Demonstration in Analytical Laboratories for  $\alpha$  materials
- Demonstrate remote operation in the IMCL shielded enclosure<sup>[3]</sup>
- Design and install the dilatometer in the APIEC Project.<sup>[3]</sup>

## 2.2.4 Thermal Properties Microscope

A thermal properties microscope is a laser-based instrument that can directly measure thermal conductivity. The operation principle involves measurement of the thermal diffusivity and thermal effusivity. The thermal conductivity can be extracted from these two measurements. Direct measurement of the thermal conductivity is important because it alleviates the need to separately measure the specific heat. As a consequence, no waste issues are associated with small samples used for calorimetry measurements. The instrument is being designed so that it can be remotely operated and maintained, function in a radiation environment, and make measurements on nuclear fuel and other radioactive samples. Once operational, this instrument will provide micron-level thermal property information that is commensurate with microstructure heterogeneity in nuclear fuel.

### 2.2.4.1 Instrument Design Description.

- Functions Performed – The thermal properties microscope is used to do the following:
  - Directly measure thermal conductivity without having to measure specific heat
  - Measure thermal conductivity variations with micron-scale resolution
  - Measure thermal conductivity of metallic and ceramic nuclear fuels.
- Existing Applications

The individual techniques employed by the thermal properties microscope have been used in other applications, but have not been combined in the fashion envisioned with this microscope.

### 2.2.4.2 Technology Readiness Level Status.

- Nuclear Applications

The thermal properties microscope measurement technique has been demonstrated at INL and a conceptual instrument design has been initiated.

- Readiness Rating in Relevant Environment

The current readiness of a thermal properties microscope used for examining irradiated nuclear fuels and materials is TRL-4 (Table 14) due to the conceptual nature and the need to demonstrate remote operability, as well as robust operability, in an environment that includes vibration and EMI.

Table 14. Thermal properties microscope technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Thermal Properties Microscope	4	5	5	5	4	5	4	4	4.6

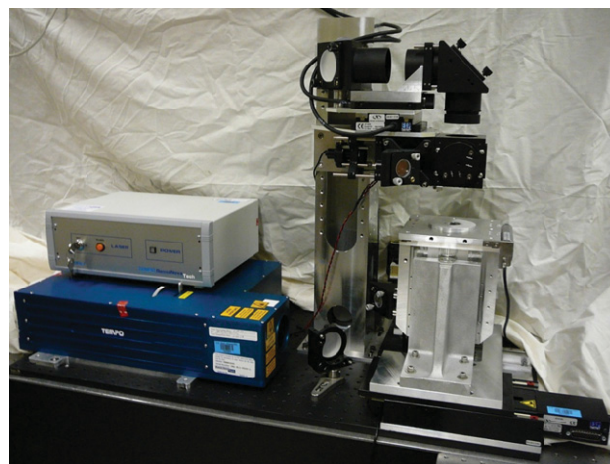
**2.2.4.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Finalize the conceptual design and fabricate a prototype for further testing and evaluation. The instrument will require optimization for functionality and compatibility with remote operation and remote sample handling.
- Integrate with design activities to address vibration, EMI, and radiation challenges.
- Demonstrate remote operability in a radiation environment at IMCL.
- Design and install the thermal properties microscope in the APIEC Project.<sup>[3]</sup>

## 2.2.5 Laser Resonant Ultrasound Spectroscopy – Mechanical Properties Microscope

INL has initiated mockup testing of a unique laser ultrasound instrument to measure the mechanical properties of nuclear fuel. The mechanical properties microscope (MPM), developed under the Fuel Cycle Research and Development program, is designed to operate in a radiation hot cell environment via remote control manipulation. The MPM will provide micron-level mechanical property information that is commensurate with microstructure heterogeneity. The spatial resolution of the MPM enables results to be tied directly with other electron/photon microstructural imaging technologies. This aspect is essential to understanding the role of microstructure in measuring mechanical and elastic properties of nuclear fuel. Development of the MPM connects closely with INL's larger PIE effort to provide new validation metrics for fundamental computational material science models. New insight provided by the MPM will help INL develop advanced nuclear fuels with improved reactor performance and safety margins.

A photograph of the MPM is shown in the upper pane of Figure 8. Ultrasonic generation is achieved by irradiating the sample of interest with a commercial pulsed laser. The absorbed optical energy is quickly converted into a steep thermal gradient. The sudden thermal expansion of the laser heated region causes ultrasonic



**Elastic property data on UMo fuel**

	Isotropic Ingot	Anisotropic rolled foil	Percent change
Young's Modulus (Gpa)	102	71	30
Shear Modulus	36	25	30

Figure 8. A photograph of the mechanical properties microscope, showing the sample stage and detection interferometer and preliminary data.

waves to propagate throughout the sample. Ultrasonic displacement is detected with a commercial interferometer. Preliminary data taken on a uranium molybdenum fuel surrogate is shown in the bottom pane of Figure 8. These data clearly show the influence of rolling texture on the elastic properties of this particular fuel surrogate.

### 2.2.5.1 Instrument Design Description.

- Functions Performed – The MPM is used to do the following:

- Measure elastic properties with 10-micron resolution
- Measure changes in elastic properties with temperature
- Quantify conductivity changes with irradiation
- Measure anelastic properties
- Measure coating thickness down the micron level.

- Existing Applications

Instruments based on laser ultrasonics are primarily used for materials characterization and for nondestructive examination. Examples include the following:

- *Thin film metrology* – Used to characterize films and film stacks with resolution exceeding that of ellipsometry.
- *Inspection of aircraft components* – Used by the aircraft industry to inspect composite airplane components.
- *Wall thickness* – Used to determine the wall thickness of seamless steel tubes while they are in production.

### 2.2.5.2 Technology Readiness Level Status.

- Nuclear Applications

Instruments based on laser ultrasonics are primarily used for fuel materials characterization and for nondestructive examination. Applications examples include the following:

- Measure texture in metallic fuel
- Measure porosity distribution in ceramic fuel
- Measure hydride formation in fuel cladding.

- Readiness Rating in Relevant Environment

The current readiness of an MPM used for examining irradiated nuclear fuels and materials is TRL-5 (Table 15) due to the need to demonstrate remote operability and robust operability in an environment with vibration and EMI.

Table 15. Mechanical properties microscope (laser resonant ultrasound spectroscopy) technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Mechanical Properties Microscope - Laser Resonant Ultrasonic Spectroscopy	5	5	5	5	5	5	5	5	5.0

**2.2.5.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Complete construction of a sample loading apparatus
- Design and complete construction of a sample heating stage
- Incorporate design improvements identified during mockup testing
- Integrate with design activities to address vibration, EMI, and radiation challenges
- Address radiation shielding issues
- Optimize for remote operation
- Demonstrate remote operability in a radiation environment at IMCL
- Design and install the MPM-laser resonant ultrasound spectroscopy capability in the APIEC Project.<sup>[3]</sup>

## **2.2.6 Transmission Electron Microscope**

TEMs have been used for years in non-nuclear materials development. In recent history, TEMs have been identified as extremely valuable in determining the structure of materials at the nano-scale. TEM transmits electrons through a sample, then captures an image of the electrons' interaction with the material that they were transmitted through. This transmission requires a very small sample size; therefore, it is expected that very little shielding will be required, although a glove box would be required to examine highly irradiated fuels and materials. TEMs are very sensitive to temperature gradients and vibration; therefore, the room/laboratory where a TEM is located must have the ability to control room temperature very accurately. For this assessment, the term TEM is used broadly and could include the field emission gun – scanning transmission electron microscope or the aberration-corrected TEM.

### **2.2.6.1 Instrument Design Description.**

- Functions Performed – The TEM is used to do the following:
  - Capture images of electrons' interaction with sample material
  - Understand the microstructure and lattice structure of the material
  - Validate micro-scale models for the “science-based” approach to nuclear fuel and materials development.
- Existing Applications

ITU operates a TEM laboratory that has the ability to examine highly irradiated nuclear fuels and materials using an integrated glove box, as discussed in the *International Workshop on Characterization and PIE Needs for Fundamental Understanding of Fuels Performance and Safety*.<sup>[9]</sup> Figure 9 shows an image of the ITU TEM. INL has demonstrated the capability to examine fuels and materials, but these samples are of low dose rate material. A shielded FIB is required to prepare a sample for TEM and INL currently does not have the ability to create a TEM sample from highly irradiated fuels.

### **2.2.6.2 Technology Readiness Level Status.**

- Nuclear Applications

TEM samples are so small that the sample size of highly irradiated fuels or materials will not impact the performance of the device. Operation in a glove box is planned for handling the samples. TEM is sensitive to temperature changes and vibration (e.g., mechanical, air flow, and acoustic). Design considerations need to be given to power routing; heating, ventilating, and air conditioning; and the placement of microscope support equipment.

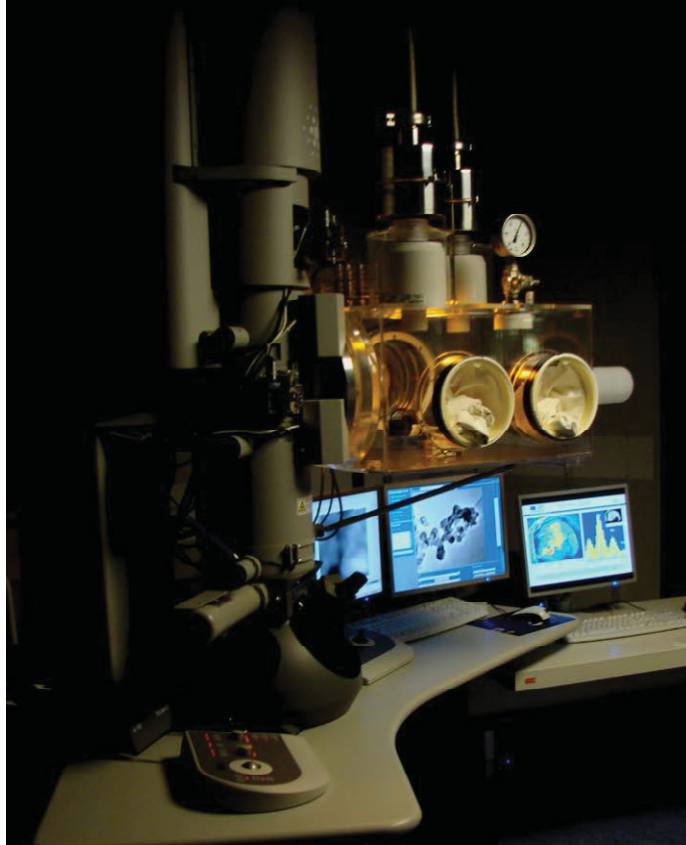


Figure 9. Institute for Transuranium Elements transmission electron microscope with glove box.

- Readiness Rating in Relevant Environment

The current readiness of a TEM used for examining highly irradiated nuclear fuels and materials is TRL-7 (Table 16) as a result of extensive industrial and nuclear applications. It should be noted that the aberration-corrected TEM is less mature, although its integration with design to ensure the 0.5°C/hour temperature stability requirement is achievable.

Table 16. Transmission electron microscope technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Transmission Electron Microscope (TEM)	7	7	7	7	7	7	7	7	7.0

**2.2.6.3 Maturation Path Forward.** This technology will be prepared for application in the APIEC Project through the following activities:

- Design and integrate a glove box with either a new or existing TEM at INL
- Move the 300-KeV TEM (Model: TF-30) from CAES to IMCL for irradiated fuel examination
- Demonstrate the ability to manufacture and handle TEM samples of small irradiated materials with a shielded FIB in IMCL.



## 2.2.7 Atom Probe Tomography

APT is a microscope that uses an electric field and needle-shaped sample tip to remove ions from the sample surface and identify them using time-of-flight spectroscopy. The atom probe will continue to emit ions from the surface until the sample is completely consumed. The sample can be reconstructed into a three-dimensional image, providing structural information of the sample at the nano-scale. Atom probes are very sensitive to temperature gradients and vibration; therefore, the room/laboratory where an APT is located must have the ability to control room temperature very accurately.

### 2.2.7.1 Instrument Design Description.

- Functions Performed – APT is used to do the following:
  - Provide atom-by-atom chemical and isotopic data in three dimensions
  - Determine precipitates, grain boundary segregation, and dopant distributions
  - Methodically destroy a sample of material and, with sensors, reconstruct the sample, providing nano-scale structural data
  - Provide data to validate models of fuels and materials at the micro-scale.

- Existing Applications

INL has a localized electron atom probe (LEAP; Imago LEAP 4000X HR) in the CAES facility that currently can perform APT on samples that it can make using an FIB. INL has demonstrated the capability to examine fuels and materials, but these samples are of low dose rate material. A shielded FIB is required to prepare a sample for LEAP and INL currently does not have a shielded FIB. Figure 10 shows LEAP in the INL CAES facility.

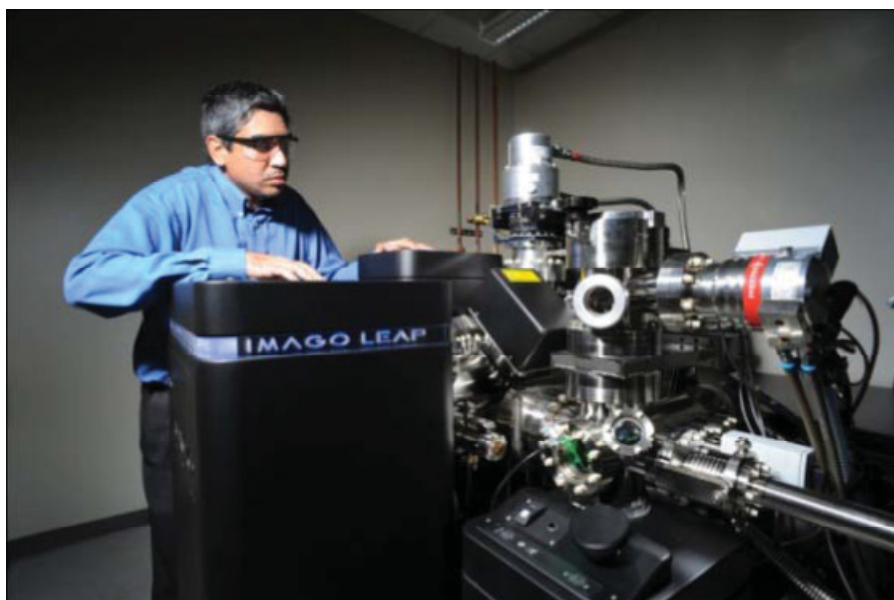


Figure 10. Localized electron atom probe in the Center for Advanced Energy Studies facility.

### 2.2.7.2 Technology Readiness Level Status.

- Nuclear Applications

LEAP samples are so small that the sample size of highly irradiated fuels or materials will not impact the performance of the device.

- Readiness Rating in Relevant Environment

The current readiness of an APT used for examining highly irradiated nuclear fuels and materials is TRL-6 (Table 17) due to the issues surrounding remote operation.

Table 17. Atomic probe tomography technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Atomic Probe Tomography (APT)	6	7	7	6	6	6	6	6	6.3

**2.2.7.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Develop the ability to manufacture LEAP samples of highly irradiated materials with a shielded FIB in IMCL
- Design and install APT in the APIEC Project.<sup>[3]</sup>

## 2.2.8 High-Temperature X-Ray Diffraction

The in situ, high-temperature x-ray diffraction (HT XRD) technique provides an effective and direct way to trace the phase structure during increasing and decreasing temperatures.

### 2.2.8.1 Instrument Design Description.

- Functions Performed

HT XRD is used to provide a micro-scale image of materials that need to be investigated in situ while the sample or specimen is undergoing high-temperature variations.

- Existing Applications

Applications include understanding material behavior during solid state, phase transitions, crystallite growth, and thermal expansion in the semiconductor industry (i.e., analyzing ZnO nanopowders) and chemical industry (coatings and catalysts).

### 2.2.8.2 Technology Readiness Level Status.

- Nuclear Applications

A HT XRD currently is located in INL's Analytical Laboratory and will be used for fresh fuel applications with  $\alpha$  contamination and low  $\beta\gamma$  contamination.

- Readiness Rating in Relevant Environment

The current readiness of HT XRD used for examining irradiated nuclear fuels and materials is TRL-5 (Table 18) due to the need to demonstrate remote operability and operability in an environment that overcomes vibration and EMI issues.

Table 18. High-temperature x-ray diffraction technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	High Temperature X-Ray Diffraction (HT XRD)	5	6	6	6	5	5	6	5	5.6

**2.2.8.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Achieve TRL-6 with the successful operation of the HT XRD in INL's Analytical Laboratory for fresh fuel applications
- Demonstrate operability in IMCL for use on high-activity or hazardous materials.

## 2.2.9 Knudsen Cell

A typical Knudsen cell contains a crucible (i.e., made of pyrolytic carbon boron nitride, quartz, tungsten, or graphite), heating filaments (often made of metal tantalum), water cooling system, heat shields, and an orifice shutter. In crystal growth, Knudsen effusion cells are often used as source evaporators for relatively low partial pressure elements (e.g., Ga, Al, Hg, and As). Tight temperature control of evaporating content is maintained and commonly used in molecular-beam epitaxy.

### 2.2.9.1 Instrument Design Description.

- Functions Performed

The Knudsen cell primarily is used to determine vapor pressures, but also can be used to identify quantities of materials in multi-component systems and to produce controlled deposition of thin films of materials.

- Existing Applications

Clemson University's Advanced Materials Research Laboratory<sup>[12]</sup> has addressed vibration (e.g., floor vibrations, acoustic noise, and thermal vibrations), air flow, and EMI issues through the design and installation process. The design includes the use of climate controls, foam-insulated compartments, isolated inertia floor slabs, air distribution diffusers, and Faraday cages.

### 2.2.9.2 Technology Readiness Level Status.

- Nuclear Applications

High-temperature Knudsen cells have been used in mass spectroscopic studies on lanthanum oxide and uranium oxide solid solutions to assess the volatility of rare earths present in irradiated nuclear fuel.<sup>[13]</sup> The European Commission Joint Research Centre ITU has performed a variety of experiments on irradiated fuel samples using a modified Knudsen cell apparatus.<sup>[14]</sup>

- Readiness Rating in Relevant Environment

The current readiness of a Knudsen cell used for examining irradiated nuclear fuels and materials is TRL-6 (Table 19) assuming the experience and design from the ITU instrument can be readily transferred.

Table 19. Knudsen cell technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Knudsen Cell	6	7	7	7	6	6	6	6	6.4

**2.2.9.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Integrate with design activities to address the challenges associated with radiation, remote operation, and the ability to remotely handle sample specimens



- Design and install the Knudsen cell in the APIEC Project.<sup>[3]</sup>

## 2.2.10 Photo Electron Spectroscopy

Photo electron spectroscopy is an analysis technique that can be used to analyze the surface chemistry (5 to 50Å depth) of a material in its as-is state or after some treatment (e.g., fracturing, cutting, ion beam etching to clean off some of the surface contamination, exposure to heat to study the changes due to heating, exposure to reactive gases or solutions, exposure to ion beam implant, or exposure to radiation) Photo electron spectroscopy uses either mono-energetic, soft x-rays, or lower energy photons in the ultraviolet spectrum to strike a sample material, causing photoelectrons to be ejected and enabling researchers to investigate the core energy levels of a material. With ultraviolet photo electron spectroscopy, the primary emphasis is on examining the valence electron levels rather than core levels. Auger electron spectroscopy can be used to study chemical and compositional properties of surfaces.

### 2.2.10.1 Instrument Design Description.

- Functions Performed – Photo electron spectroscopy is used to determine the following:
  - Elemental composition of the surface chemistry (top 1 to 10 nm)
  - Empirical formula of pure materials
  - Elements that contaminate a surface
  - Uniformity of elemental composition across the top surface (or line profiling or mapping)
  - Uniformity of elemental composition as a function of ion beam etching (or depth profiling)
  - Oxidation states of elements
  - Chemical binding energies of elements
  - Chemical and structural environments of elements within materials.

- Existing Applications

Photo electron spectroscopy is routinely used whenever elemental or chemical state analysis is needed at surfaces and interfaces.<sup>[46]</sup> It is used to analyze metal alloys, semiconductors, polymers, elements, catalysts, glasses, ceramics, paints, papers, inks, woods, plant parts, make-up, teeth, bones, medical implants, bio-materials, viscous oils, glues, ion-modified materials, and many others.

### 2.2.10.2 Technology Readiness Level Status.

- Nuclear Applications

During TRISO fuel development, photo electron spectroscopy was used to study the interaction between metal fission products and TRISO coating materials.<sup>[14]</sup>

- Readiness Rating in Relevant Environment

The current readiness of photo electron spectroscopy used for examining irradiated nuclear fuels and materials is TRL-5 (Table 20) due to the need to demonstrate radiation-hardened and remote operability.

Table 20. Photo electron technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Photo Electron	5	6	6	6	5	5	6	5	5.6

**2.2.10.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Integrate with design activities to address vibration, EMI, and radiation challenges
- Design and install photo electron spectroscopy in the APIEC Project.<sup>[3]</sup>

## 2.2.11 Raman Spectroscopy

Raman spectroscopy is a spectroscopic technique used to study vibrational, rotational, and other low-frequency modes in a system. It relies on inelastic scattering, or Raman scattering, of monochromatic light, usually from a laser in the visible, near infrared, or near ultraviolet range. The laser light interacts with molecular vibrations, phonons, or other excitations in the system, resulting in the energy of the laser photons being shifted up or down. The shift in energy gives information about the vibrational modes in the system. Infrared spectroscopy yields similar, but complementary, information.<sup>[17]</sup>

### 2.2.11.1 Instrument Design Description.

- Functions Performed – The Raman spectrometer is used to do the following:
  - Study chemical bonding changes in molecules
  - Measure stress distribution
  - Measure temperature
  - Measure thermal properties.
- Existing Applications

Raman spectroscopy is commonly used in chemistry, because vibrational information is specific to the chemical bonds and symmetry of molecules. Therefore, it provides a fingerprint by which the molecule or material can be identified. The fingerprint region of organic molecules is in the (wave number) range 500 to 2,000  $\text{cm}^{-1}$ . Another way the technique is used is to study changes in chemical bonding (such as when a change in composition occurs).

### 2.2.11.2 Technology Readiness Level Status.

- Nuclear Applications  
INL currently does not have a Raman spectrometer for radioactive materials. Raman spectroscopy has many nuclear materials applications. For instance, it can be used to monitor chemical composition and stress distribution of thin corrosion layers. The Raman signal also can be used as a thermometer to monitor thermal conductivity of materials. Because this is a laser-based characterization approach, it is well suited to make in-situ measurements in hostile environments (e.g., high temperature and high radiation).
- Readiness Rating in Relevant Environment  
The current readiness of Raman spectroscopy used for examining irradiated nuclear fuels and materials is TRL-6 (Table 21) due to the need to demonstrate remote operability and remote sample handling.

Table 21. Raman spectrometer technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Raman Spectrometer	6	7	7	7	6	6	7	6	6.6

**2.2.11.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Integrate with design activities to address vibration, EMI, and radiation challenges
- Design and install Raman spectroscopy in the APIEC Project.<sup>[3]</sup>

## **2.2.12 Positron Annihilation Spectrometer**

Positron annihilation spectroscopy is a non-destructive spectroscopy technique to study voids and defects in solids. The positron annihilation spectrometer (PAS) operates on the principle that a positron will annihilate through interaction with electrons. This annihilation releases gamma rays that can be detected; the time between emission of positrons from a radioactive source and detection of gamma rays due to annihilation corresponds to the lifetime of a positron. When positrons are injected into a solid body, they will interact in some manner with the electrons in that species. For solids containing free electrons (such as metals or semiconductors), the implanted positrons will annihilate very rapidly unless voids such as vacancy defects are present. If voids are available, positrons will reside in them and annihilate less rapidly than in the bulk of the material, on time scales up to about 1 n. For insulators (such as polymers or zeolites) implanted positrons interact with electrons and annihilate less rapidly, on time scales up to about 140 ns in a vacuum.

### **2.2.12.1 Instrument Design Description.**

- Functions Performed – The PAS is used to do the following:
  - Detect void fractions
  - Detect small-sized defects at low concentration that could not be detected by TEM or XRD
  - Probe both the bulk properties of matter and near-surface regions and layered structures.

- Existing Applications

PAS has demonstrated itself as a powerful tool of microstructural studies of condensed matter. Atomic-scale details of the microstructure-like (e.g., electronic structure and small-sized defects) can be investigated by PAS.

The main advantages of PAS can be summarized as follows:

- PAS can detect small-sized defects at low concentration, which cannot otherwise be investigated by traditional basic techniques (e.g., TEM or x-ray diffraction).
- PAS can probe both the bulk properties of matter and near-surface regions and layered structures.
- Reliable theoretical calculations of PAS parameters are nowadays feasible in metals and semiconductors. These may substantially simplify unique interpretation of data measured by PAS.
- PAS is a non-destructive technique. In many cases, PAS becomes not only a substantial complement of traditional methods (e.g., TEM or XRD), but is an equivalent and independent method of microstructural investigations of condensed matter.<sup>[20]</sup>

### **2.2.12.2 Technology Readiness Level Status.**

- Nuclear Applications

The Oak Ridge Linear Accelerator produced a pulsed positron beam to measure void sizes and distributions as a function of depth.

- Readiness Rating in Relevant Environment

The current readiness of PAS used for examining irradiated nuclear fuels and materials is TRL-5 (Table 22) due to the need to demonstrate radiation-hardened, remote operability.

Table 22. Positron annihilation spectroscopy technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Positron Annihilation Spectroscopy (PAS)	5	6	6	6	5	5	6	5	5.6

**2.2.12.3 Maturation Path Forward.** Further technology maturation is required and will be achieved through the following activities:

- Integrate with design activities to address vibration, EMI, and radiation challenges
- Design and install PAS in the APIEC Project.<sup>[3]</sup>

Table 23 provides a summary of the estimated TRL values for the other instruments that may be of interest for advanced PIE.

Table 23. Technology readiness levels for other instruments.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
<b>APIEC - Other Instruments</b>		5.3	6.0	6.0	6.0	5.5	5.5	5.9	4.0	5.7
<b>Thermal Properties</b>		5.3	6.3	6.3	6.7	5.7	5.7	6.3	5	6.0
	Scanning Thermal Diffusivity Microscope (STDM)	6	7	7	7	7	7	7	6	6.9
	Arrest Melting Point Determination	5	6	6	6	5	5	6	5	5.6
	Dilatometer	5	6	6	7	5	5	6	5	5.7
	Thermal Properties Microscope	4	5	5	5	4	5	4	4	4.6
<b>Mechanical Properties</b>		5.0	5.0	5.0	5.0	5.0	5.0	5.0	5	5.0
	Mechanical Properties Microscope - Laser Resonant Ultrasonic Spectroscopy	5	5	5	5	5	5	5	5	5.0
<b>Microstructural Analysis</b>		5.7	6.6	6.6	6.4	5.7	5.7	6.3	5	6.1
	Transmission Electron Microscope (TEM)	7	7	7	7	7	7	7	7	7.0
	Atomic Probe Tomography (APT)	6	7	7	6	6	6	6	6	6.3
	High Temperature X-Ray Diffraction (HT XRD)	5	6	6	6	5	5	6	5	5.6
	Knudsen Cell	6	7	7	7	6	6	6	6	6.4
	Photo Electron	5	6	6	6	5	5	6	5	5.6
	Raman Spectrometer	6	7	7	7	6	6	7	6	6.6
	Positron Annihilation Spectroscopy (PAS)	5	6	6	6	5	5	6	5	5.6

## 2.3 Sample Preparation Equipment

The advanced PIE equipment, identified in Sections 2.1 and 2.2, require specimens that are generated in a sample preparation area that will be part of the APIEC Project. This sample preparation area will have the ability to accept a bulk sample and create a specimen for examination with the equipment in that facility. There are three types of specimens that the APIEC Project equipment will examine:

(1) metallurgical [met] mounts, (2) non-met mounts, and (3) FIB samples. The met mount and non-met mount samples will be generated in the sample preparation area, while the FIB samples will be milled from a met-mounted specimen in the FIB shielded cell.

The sample preparation area for generating the met and non-met mounts currently is used routinely at INL and at many other facilities. The FIB samples are conditional to operating the FIB in a shielded enclosure for highly irradiated fuels and materials (see FIB discussion on TRL in Section 2.1.6.2 above);

otherwise, generating FIB specimens is routine. Table 24 identifies the type of specimen that the sample preparation area would need to provide the given instrument for examination.

Table 24. Type of specimens for the post-irradiation examination equipment.

Function	Instrument	Specimen Type
Microstructural Analysis	FIB	Met mount
	$\mu$ XRD	Met mount/non-met mount
	HT XRD	Non-met mount
	Aberration-corrected TEM	Met mount/FIB
	APT	Met mount/FIB
	SEM	Met mount
	PAS	Non-met mount
Chemical Analysis	EPMA	Met mount/FIB
	SIMS	Met mount
	Knudsen cell	Non-met mount
Thermal Analysis	STDM	Non-met mount
	DSC	Non-met mount
	Dilatometer	Non-met mount
	Laser flash diffusivity	Non-met mount
	Arrest melting point determination	Non-met mount
	TGA	Non-met mount
	Thermal properties microscope	Non-met mount
Spectroscopic Spectrometric Methods	Photo electron	Met mount
	Raman spectrometer	Non-met mount
Mechanical Analysis	Nanoindenter	Met mount/non-met mount/FIB
	MPM/ laser resonant ultrasound spectroscopy	Non-met mount
Sample Preparation	Electric discharge machining	Non-met mount

### 2.3.1 Technology Readiness Level Status

**2.3.1.1 Readiness Rating in Relevant Environment.** The industrial and nuclear applications using a sample preparation cell to create met mounts and non-met mount specimens are technically mature. However, using the sample preparation cell to create FIB specimens requires operating the FIB in a shielded enclosure for highly irradiated fuels and materials. Because the FIB is rated as TRL-6, the sample preparation cell using this instrument also will rate as TRL-6.

### 3. SUMMARY AND CONCLUSIONS

A TRA was performed that generated TRLs for the current state of each instrument in the initial complement of PIE equipment and the sample preparation equipment of the APIEC Project (as shown in Table 25).

TMPs were developed to define the modifications of each instrument, the tests to be performed, and the test results needed to deploy the equipment in the APIEC Project facility. The TMPs will be updated and further detailed as the tests are performed and as the instruments progress through successive TRLs. TDRMs were generated to document the path forward to successful operation in a facility. The TDRMs summarize the TMPs for the project. As each task shown on the TDRM is completed, demonstrable evidence of the technology maturation is provided as required for advancement to increasing technology readiness levels. The TDRM for each of the instruments illustrates the tasks needed to further mature the technology and demonstrate it as operational in vibration, EMI, relevant temperature, radiation, and remote environments (see Figures 11 and 12). A notional timeline is included to illustrate the technology development activities as they relate to project phases.

Table 25. Advanced post-irradiation capabilities technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
<b>APIEC - Near Term Instruments</b>		6.5	7.0	7.0	7.0	6.5	6.5	7.0	6.0	6.8
<b>Chemical Analysis</b>		7.0	7.0	7.0	7.0	7.0	7.0	7.0	7	7.0
	Electron Probe Microanalysis (EPMA)	7	7	7	7	7	7	7	7	7.0
	Secondary Ion Mass Spectrometer (SIMS)	7	7	7	7	7	7	7	7	7.0
<b>Thermal Properties</b>		6.0	7.0	7.0	7.0	6.5	6.5	7.0	6	6.7
	Simultaneous Thermal Analyzer	6	7	7	7	7	7	7	6	6.9
	Laser Flash Analyzer (LFA)	6	7	7	7	6	6	7	6	6.6
<b>Mechanical Properties</b>		7.0	7.0	7.0	7.0	6.0	6.0	7.0	6	6.7
	Nano-Indenter	7	7	7	7	6	6	7	6	6.7
<b>Microstructural Analysis</b>		6.0	7.0	7.0	7.0	6.3	6.3	7.0	6	6.7
	Focused Ion Beam (FIB)	6	7	7	7	6	6	7	6	6.6
	Scanning Electron Microscope (SEM)	6	7	7	7	7	7	7	6	6.9
	Micro X-Ray Diffraction (MXRD)	6	7	7	7	6	6	7	6	6.6
<b>Sample Preparation Cell</b>		6.0	7.0	7.0	7.0	6.0	6.0	6.0	6.0	6.4
<b>APIEC - Other Instruments</b>		5.3	6.0	6.0	6.0	5.5	5.5	5.9	4.0	5.7
<b>Thermal Properties</b>		5.3	6.3	6.3	6.7	5.7	5.7	6.3	5	6.0
	Scanning Thermal Diffusivity Microscope (STDM)	6	7	7	7	7	7	7	6	6.9
	Arrest Melting Point Determination	5	6	6	6	5	5	6	5	5.6
	Dilatometer	5	6	6	7	5	5	6	5	5.7
	Thermal Properties Microscope	4	5	5	5	4	5	4	4	4.6
<b>Mechanical Properties</b>		5.0	5.0	5.0	5.0	5.0	5.0	5.0	5	5.0
	Mechanical Properties Microscope - Laser Resonant Ultrasonic Spectroscopy	5	5	5	5	5	5	5	5	5.0
<b>Microstructural Analysis</b>		5.7	6.6	6.6	6.4	5.7	5.7	6.3	5	6.1
	Transmission Electron Microscope (TEM)	7	7	7	7	7	7	7	7	7.0
	Atomic Probe Tomography (APT)	6	7	7	6	6	6	6	6	6.3
	High Temperature X-Ray Diffraction (HT XRD)	5	6	6	6	5	5	6	5	5.6
	Knudsen Cell	6	7	7	7	6	6	6	6	6.4
	Photo Electron	5	6	6	6	5	5	6	5	5.6
	Raman Spectrometer	6	7	7	7	6	6	7	6	6.6
	Positron Annihilation Spectroscopy (PAS)	5	6	6	6	5	5	6	5	5.6



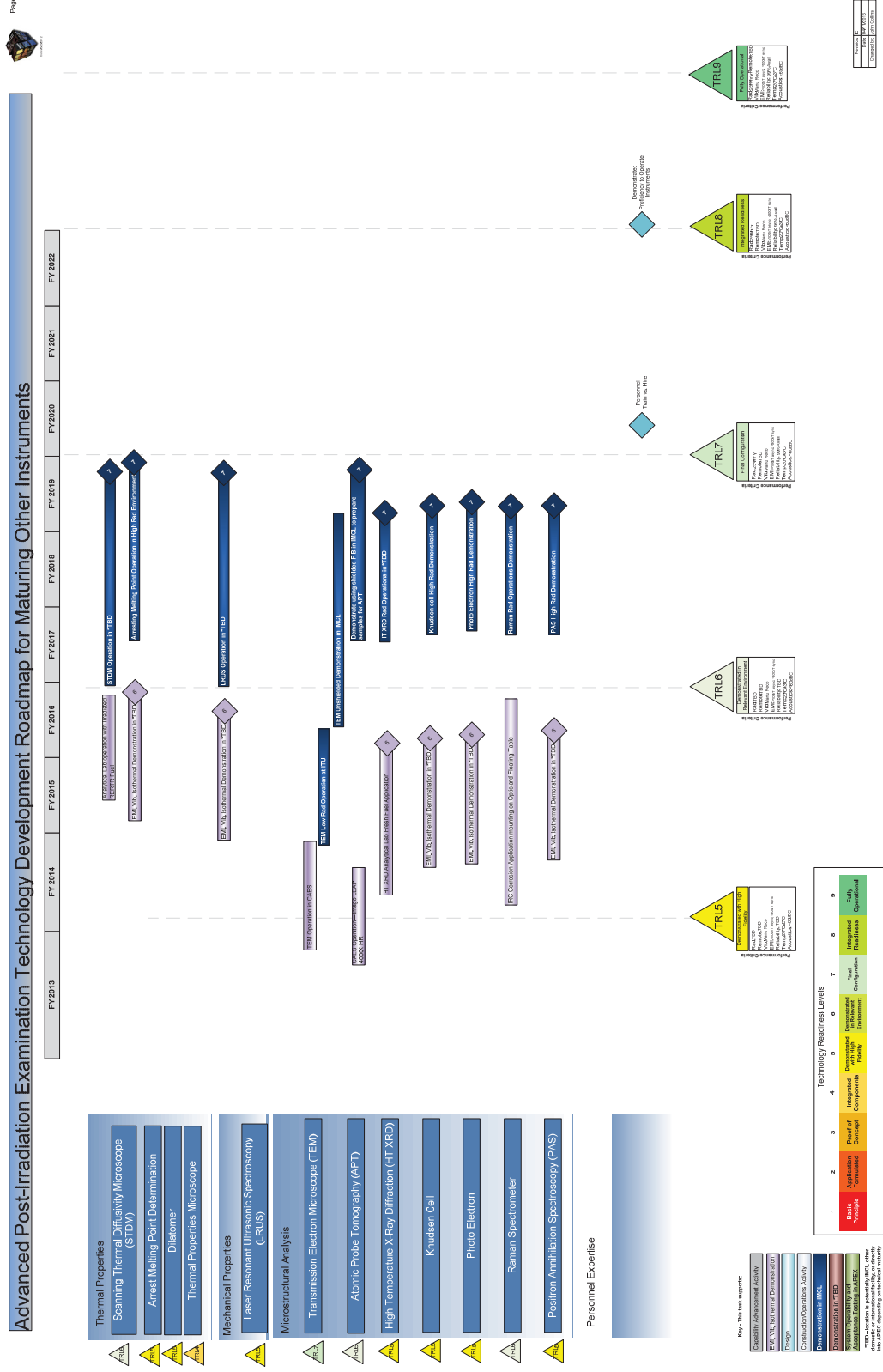


Figure 12. Advanced Post-Irradiation Examination Capabilities Project technology development roadmap for maturing other instruments.



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## 5. ATTACHMENTS

The following TMPs are included as attachments to this APIEC Project TRA report:

- Electron Probe Microanalyzer, PLN-4462
- Secondary Ion Mass Spectrometer, PLN 4458
- Simultaneous Thermal Analyzer, PLN-4456
- Laser Flash Analyzer, PLN-4459
- Nanoindenter, PLN-4463
- Focused Ion Beam, PLN-4461
- Scanning Electron Microscope, PLN-4460
- Micro X-ray Diffraction, PLN-4457
- Sample Preparation Equipment, PLN-4455.

## **Technology Maturation Plan**

# **Advanced Post-Irradiation Examination Capabilities Project Electron Probe Microanalyzer**



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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available electron probe microanalyzer (EPMA) for advanced post-irradiation examination (PIE) of nuclear fuels and materials. This effort is a part of the Advanced Post-Irradiation Examination Capabilities Project to design, construct, and deploy the Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

For the purpose of this plan, a CAMECA SX-100R shielded EPMA will be assumed. Other EPMA manufacturers exist; however, the only EPMA manufacturer that produces a radiation-hardened version is CAMECA. A JEOL EPMA would have to be customized for radioactive specimens. The CAMECA instrument appears to be the most technologically mature for deployment in the APEX facility.

The shielded SX-100R can handle and analyze irradiated nuclear fuels emitting gamma radiations up to 3 curies at 0.75 MeV (0.66MeV using cesium). It also is equipped with shielded wavelength-dispersive spectroscopy (WDS) and a shielded sample stage to ensure safe sample manipulation and to preserve analytical performance. It is believed that the SX-100R can be customized, as needed, for remote operation in the APEX facility. A newer model CAMECA EPMA (SXFive) has the capability to accommodate irradiated nuclear fuels emitting gamma radiations up to 3 curies at 1 MeV. The shielded SXFive has the same footprint, utilities, and layout as the SX-100R, but improved electronics and capability:

- The instrument (i.e., column, spectrometers, and sample stage) are installed in a “hot” cell (i.e., lead or concrete shielded room).
- The instrument will be remote-controlled (e.g., stage, column, or diaphragms) with electronics and a computer located outside the “hot” cell environment.
- Telemanipulators will be used to insert and mount the radioactive samples. For example, in the Institute for Transuranium Elements Karlsruhe-Germany configuration, the analysis chamber is vented and the stage is moved to a position directly accessible with ball-manipulators at each sample exchange. In the LECA/STAR Commissariat à l’Energie

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Atomique Cadarache-France design, samples are introduced via an airlock system located in a glove box for alpha confinement.

- All WDS analyzers and detectors are shielded to prevent the background caused by the gamma radiations.
- The secondary electron detector is oriented to avoid gamma ray perturbation.
- The automation system controls all the parameters of the microprobe and offers features for quantitative analysis, x-ray mapping, line profile acquisition, and data processing.

### 1.3 Instrument Description

An EPMA is a microbeam instrument primarily used for in situ non-destructive chemical analysis of minute solid samples (Figures 1 and 2). EPMA also is informally called an electron microprobe or just probe. It is similar to a scanning electron microscope; however, where the scanning electron microscope is optimized for image collection, the EPMA is optimized for chemical composition analysis. The primary importance of an EPMA is the ability to acquire precise, quantitative elemental analyses at very small “spot” sizes (as little as 1 to 2 microns), primarily by WDS.

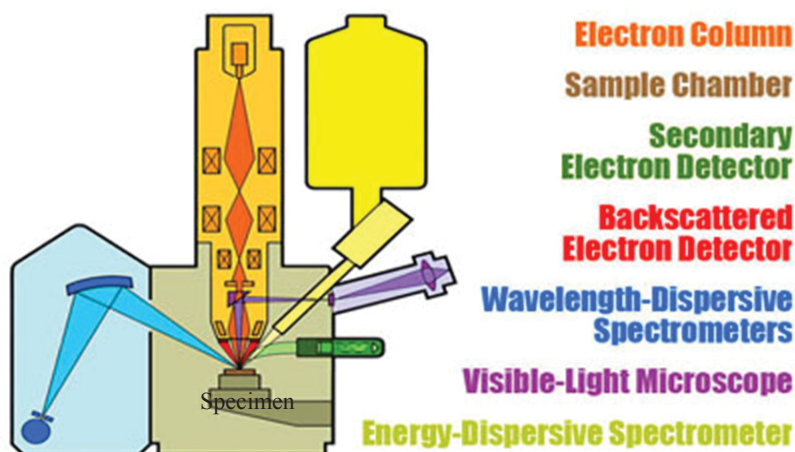


Figure 1. Typical configuration.



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EXAMINATION CAPABILITIES  
PROJECT ELECTRON PROBE  
MICROANALYZER**

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Figure 2. Typical size.

Specifically, low-energy electrons are produced from a tungsten, LaB6, or field emission electron source and accelerated by a positively biased anode plate from 3 to 30 thousand electron volts (keV). The anode plate has a central aperture, and electrons that pass through it are collimated and focused by a series of magnetic lenses and apertures. The resulting electron beam (approximately from 5 nm to 10 micrometer diameter) may be rastered across the sample or used in spot mode to excite various effects from the sample. Among these effects are: phonon excitation (heat), cathodoluminescence (visible light fluorescence), continuum x-ray radiation (bremsstrahlung), characteristic x-ray radiation, secondary electrons (plasmon production), backscattered electron production, and Auger electron production.

The characteristic x-rays are used for chemical analysis. Specific x-ray wavelengths are selected and counted, either by WDS or energy dispersive x-ray spectroscopy. WDS utilizes Bragg diffraction from crystals to select x-ray wavelengths of interest and direct them to gas-flow or sealed proportional detectors. In contrast, energy dispersive x-ray spectroscopy uses a solid state semiconductor detector to accumulate x-rays of all wavelengths produced from the sample. While energy dispersive x-ray spectroscopy yields more information and typically requires a much shorter counting time, WDS is the more precise technique because of its superior x-ray peak resolution and lower detection limits.

Chemical composition is determined by comparing the intensities of characteristic x-rays from the sample material, with intensities from known composition.

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standards. Counts from the sample must be corrected for matrix effects (i.e., absorption and secondary fluorescence) to yield quantitative chemical compositions. The resulting chemical information is gathered in textural context. Variations in chemical composition within a material (zoning), such as a mineral grain or metal, can be readily determined.

Volume from which chemical information is gathered (i.e., volume of x-ray generation) depends on the material being examined and the energy of the electron beam, but it is on the order of 0.3 to 3 cubic micrometers.

Unlike a scanning electron microscope, which can give images of three-dimensional objects, EPMA analysis of solid materials requires preparation of flat, polished sections. A brief protocol is provided as follows:

1. Nearly any solid material can be analyzed. In most cases, samples are prepared as met-mounts.
2. The most critical step prior to analysis is giving the sample a fine polish so surface imperfections do not interfere with electron-sample interactions. This is particularly important for samples containing minerals with different hardnesses; polishing should yield a flat surface of uniform smoothness.
3. Most silicate minerals and nuclear fuels are electrical insulators. Directing an electron beam at the sample can lead to electrical charging of the sample, which must be dissipated. Prior to analysis, samples are typically coated with a thin film of a conducting material (e.g., carbon, gold, and aluminum are most common) by means of evaporative deposition. Once samples are placed in a holder, the coated sample surface must be put in electrical contact with the holder (typically done with a conductive paint or tape). The choice of coating depends on the type of analysis to be done; for example, most EPMA chemical analysis is done on samples coated by C, which is thin and light enough that interference with the electron beam and emitted x-rays is minimal.
4. Samples are loaded into the sample chamber via a vacuum interlock and mounted on the sample stage. The sample chamber is then pumped to obtain a high vacuum.
5. To begin a microprobe session, suitable analytical conditions must be selected (such as accelerating voltage and electron beam current) and the electron beam must be properly focused. If quantitative analyses are planned, the instrument first must be standardized for the elements desired.

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#### 1.4 Current Technical Maturity

The Advanced Post-Irradiation Examination Capabilities Project uses a technology readiness level (TRL) maturity scale of 1 to 9 where 1 is just observed phenomena and 9 represents full deployment with operating experience. EPMA technology is currently at TRL-7 for this application in the APEX facility as defined below (see Table 1):

**“System demonstrated in final configuration** – Actual instrument has been successfully operated in final configuration and in relevant environment (including radiation and remote operation). This represents a major step up from TRL-6, requiring demonstration of an actual system in a relevant operational environment with limited degradation due to radiation exposure.”

Table 1. Electron probe microanalyzer technology readiness by attribute.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Electron Probe Microanalysis (EPMA)	7	7	7	7	7	7	7	7	7.0

Because the SX-100R has been deployed internationally for irradiated fuels in a hot cell “relevant environment,” this plan will describe the actions necessary to install such a probe into the APEX facility. This detail will be sufficient to estimate the cost and schedule to achieve this deployment. TRL-8 represents the following:

**“Integrated readiness of the system is demonstrated** – Technology is proven to work. Actual technology completed and qualified through test and demonstration. Facility issues a declaration of readiness and readiness activity is successfully completed.”

The plan addresses the technology maturation activities necessary to advance the EPMA at a TRL-7 through the necessary demonstrations, including associated test descriptions, conditions, and configurations as identified in Section 3, to support the APEX facility in achieving TRL-8. Verification of EPMA and shielding performance to achieve TRL-8 is expected to be demonstrated in the APEX facility using sealed radiological sources.

INL currently has a CAMECA SX 100R, but whether this instrument can be used depends on when the APEX facility can be beneficially occupied. It is possible that if the current instrument must be dismantled prior to moving it, it will be too contaminated to do so. The vendor must plan and supervise movement of the EPMA. If the instrument must be disassembled, the vendor must conduct that

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work. In addition, if the move involves changes in elevation, then appropriate tools must be available. For example, to remove the EPMA from the Analytical Laboratory will require a forklift to move it to ground level and back up onto a truck. If the EPMA is to be moved to a non-ground level floor in the APEX facility, an elevator with a door opening of 6 ft x 6 ft and a capacity of 3,000 lb will be required.

It may be desirable, but not mandatory, to mockup the EPMA in the Irradiated Materials Characterization Laboratory (IMCL) to optimize robotic controls and shielding placement. For this purpose, the existing EPMA could be re-located or a new EPMA initially staged in IMCL and then moved by the vendor to the APEX facility.

This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 through processing of the first production specimens). While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test plans and procedures, work orders, or laboratory instructions that will be used to direct the actual work.

The end user organizations for the matured and deployed EPMA are expected to be MFC Operations and Nuclear Science and Technology.

The specimens to be examined and processed in the APEX facility are expected to have much higher activities than those allowed for the unshielded Analytical Laboratory EPMA. This level is currently assumed to be 600 R/hour (i.e., three TRISO fuel particles at 200 R/hour each). An initial target value for the shielded instruments in the APEX facility is to provide the capability to examine specimens measuring up to 3 Ci of activity at 1 MeV gamma radiation. Because the SX 100R is shielded to 3Ci (Cs-137), and because it can only hold one sample at a time, it is reasonable to build the hot cell walls to effectively shield at least ten such samples (one would be in the instrument, nine would be in the queue waiting for analysis, and, during sample change, all could be in the glove box attached to the instrument).

The SX-100R does not include a vibration isolation system, but a third party isolation platform is assumed to be a part of installation.

The vendor will specify acceptable electro-magnetic interference (EMI) criteria when the instrument is specified, and may visit the site to measure the EMI field to determine whether the location is suitable or requires mitigation. Excessive EMI can cause artifacts in images, and, in fact, is a problem in the instrument's current location.

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The instrument should be procured with an uninterrupted power supply that can last 30 minutes. The uninterrupted power supply is located outside the hot cell; therefore, it is not exposed to radiation.

The instrument produces quite a bit of heat; other operators have had significant problems with the heat not being effectively dissipated in a hot cell. If the temperature exceeds 76°F for any length of time, the instrument will shut itself down. This includes the area outside the hot cell where the electronics rack will be located. Hot cell and electronics cabinet temperature could be constantly monitored to assist in troubleshooting maintenance issues.

There needs to be enough room around the column base to allow worker access to components beneath the column. In addition, room for a stair ladder or lab stool is required so that work in the top of the column can be accomplished. When the stage is being worked on, it is removed from the column. Several feet (i.e., 3 to 4 ft) between the front of the column and the hot cell wall are required to accommodate this. Maintenance is typically performed every 6 months during operation.

Insulating samples need to be coated with a conductive coating prior to analysis. The sample coater should be located in an inert gas box. This may be attached to the sample entry port of the instrument or, more ideally, in a separate sample preparation area. The coater itself will have issues operating in an inert gas environment. No instrument components will exist in an inert gas environment.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

## 3. MATURATION TEST PLANS

### 3.1 General Information

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

#### 3.1.1 Assumptions

- This plan uses a CAMECA SX-100R shielded EPMA instrument as a reference baseline to identify and define the necessary technology maturation activities.

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- It is assumed that the following accessories and add-on analysis components are needed to support its role in irradiated nuclear fuel and material characterization:
  - Vibration isolation platform
  - Sample coater
  - Inert glove box on EPMA sample introduction port
  - Uninterrupted power supply
  - Instrument chiller
  - Air compressor or building compressed air that is greater than 85 psi
  - Additional software (e.g., StrataGEM or Probe for EPMA)
  - A direct connection to ground (not a building ground but an actual “in the ground” ground for electricity).
- The shielded EPMA will be a special order unit that has been modified or built to meet INL specifications for service in a high-radiation environment. Specifically, these modifications may include, but are not limited to, the following:
  - Substitution of standard wiring, vacuum seals, and surface finishes/ coatings with ones that are compatible with high-radiation fields and, where applicable, a radiologically contaminated environment (including considerations for decontamination solutions that are anticipated to be used).
  - Extended (non-standard) vacuum chamber door rails for improved access into the vacuum chamber.
- Special customization may require additional technology maturation activities if verification of compliance is not performed by the instrument manufacturer.
- The EPMA from the Analytical Laboratory will be moved to ICML and installed in a shielded enclosure. Information from this



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deployment will be available to support design of the APEX facility's shielded enclosure for the new EPMA.

### **3.1.2 Prerequisites**

- INL and/or MFC personnel have developed a detailed specification for the EPMA.
- A special order EPMA, modified as necessary for high-radiation environments, has been procured, delivered, configured, and calibrated to support technology maturation testing and development activities.
- INL and/or MFC engineering personnel have worked with the instrument manufacturer to verify that radiation-sensitive and/or vulnerable components are physically separated from the main instrument/vacuum chamber or require add-on shielding to improve radiation hardening. Design and performance verification of such modifications may either be done by the manufacturer prior to shipping or by INL personnel after receipt of the instrument.

### **3.1.3 Identified Hazards and Safety Concerns**

The primary hazard associated with EPMA maturation activities involves working with highly radioactive sealed sources that pose a risk for unanticipated worker exposures. Other hazards include, but are not limited to, electric shocks, hoisting and rigging, rotating equipment, compressed gases, and confined spaces with the potential for oxygen deficient atmospheres.

### **3.1.4 Work Controls, Permits, and Caution/Danger Tags**

All work performed pursuant to this plan will be conducted using company standard work procedures, including work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## **3.2 Plan for Technology Readiness Level 7 to Technology Readiness Level 8**

For TRL-8 to be achieved by the APEX facility, the integrated readiness of the EPMA must be demonstrated and qualified within the finished APEX facility, including its final interfaces to the modular shielded enclosure, the inert confinement enclosure, and associated utilities provided by the facility.

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Specifically, this means that the EPMA and associated processes have been proven to work (through system operability and acceptance testing), facility representatives have issued a declaration of readiness, and readiness activities have been successfully completed. This readiness activity (typically an operational readiness review by the U.S. Department of Energy) will be performed on a facility basis and not for any one specific PIE instrument.

### **3.2.1 Technology Maturation Testing Approach**

The TRL-7 to TRL-8 maturation activities for the EPMA will be performed in conjunction with the readiness activity for the APEX facility. The facility readiness activity confirms that management has brought the facility to a state of readiness to commence program work. Once management concludes that readiness has been achieved, this state of readiness is independently verified by the contractor and confirmed by the U.S. Department of Energy. Only then will the APEX facility (a Hazard Category 2 nuclear facility) be authorized to “go hot” and perform programmatic PIE work.

The EPMA portion of the overall facility readiness activity will involve performance of a system operability and acceptance (SO&A) test for the instrument, modular shielded cell, and associated support systems.

### **3.2.2 Technology Maturation Testing Objectives**

The objective of the TRL-7 to TRL-8 maturation activities is to successfully demonstrate the integrated EPMA instrument for an advanced PIE application, including the adequacy of the following:

- Instrument radiation-hardening measures and interfaces with the modular shielded enclosure and the inert atmosphere confinement enclosure
- Acoustical noise and vibration control measures appropriate for the facility and location
- EMI protection measures appropriate for the facility and location
- Remote sample handling process, including associated methods, tools, and aids
- Remote instrument operation as deployed within the modular shielded enclosure



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- Any instrument modifications made to improve reliability, availability, and maintainability for its application for advanced PIE.

### 3.2.3 Technology Maturation Testing Description

The EPMA SO&A test will demonstrate correct system/process operation and verify that the installed instrument and associated modifications function as designed and in accordance with designated acceptance criteria and design inputs.

The SO&A will constitute an end-to-end test of the advanced PIE process for examining specimens, including their ingress into and egress from the modular shielded enclosure. A nonradioactive sample will be used for this transition test in the presence of an approximate 25-R/hour sealed gamma source.

After successfully demonstrating specimen examination, routine field maintenance (e.g., predictive/preventive maintenance actions and modular component replacements) and decontamination activities will be demonstrated.

### 3.2.4 Maturation Testing Conditions

Performance metrics (such as spectrometer reproducibility), crystal performance (signal to background), and image resolution are established at the factory before the instrument is shipped to the customer and checked again after installation. These parameters need to be periodically checked by the customer to evaluate maintenance needs; however, it should be noted that these parameters can degrade over time whether or not the instrument is in a radiation field:

- Specimens: metallurgical mounts (met-mounts) examined in the presence of a sealed radiography source measuring about 25 R/hr  $\gamma$
- Biological shielding: as provided by the APEX facility modular shielded enclosure
- Sealed radiological source: about 25 R/hr  $\gamma$
- Temperature:  $19^{\circ} \leq T^{\circ} \leq 23^{\circ}\text{C}$ , with fluctuation less than  $1^{\circ}\text{C}/\text{hour}$

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- Relative humidity: less than 75%, with fluctuation less than 10%/hour
- Spurious electromagnetic field (after mitigation, if necessary, for measured APEX facility location conditions):
  - Less than 3 mG peak-peak ( $\leq 3 \cdot 10^{-7}$  Tesla peak-peak) for 50 or 60-Hz frequencies and their harmonics
  - Less than 0.1 mG peak-peak ( $\leq 1 \cdot 10^{-8}$  Tesla peak-peak) for other frequencies
- Vibration: less than  $5 \times 10^{-4}$  m/s<sup>2</sup> peak to peak, for all spectral components
- Displacement: less than 3  $\mu$ m peak to peak for all frequencies  $\leq 2$  Hz
- Vibration: less than or equal to 3  $\mu$ m peak to peak below 2 Hz; and less than or equal  $5 \times 10^{-4}$  m/s<sup>2</sup> peak-peak above 2 Hz
- Power at 230 V ( $\pm 5\%$  with fluctuation less than 5%/10 sec.
- Pulses: maximum less than 260 V/10 ms), microbreak rms value greater than 190 V/10 ms
- Frequency: 57 to 63 Hz
- Power consumption: 5.0 KVA
- Instrument air, compressed: 6 to 8 bar (clean, dry, and oil-free)
- Ultra high purity nitrogen (N48) at 0 to 1 bar; all gases should be able to be controlled without entering the hot cell
- Ultra high purity P-10 (10% methane and 90% argon) as a fill gas for proportional counters of ionizing radiation at 2.5 to 5 bar
- Cooling water is circulated through a chiller and the medium of exchange can be air or water; the former puts excess heat into the room that must be mitigated and the chiller should be outside of the hot cell

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- Cryogenics: Some analyses require chilling a cold finger and provisions must be provided to accommodate this
- Vacuum: The primary pump can be either oil or oil-free; the vacuum pump should be located outside the hot cell.

The primary issue with these utilities is ease of operation and maintenance. This will be facilitated by keeping these outside the hot cell, but close enough to the instrument that they operate correctly.

### 3.2.5 Examination Testing Configuration

The SO&A testing configuration will be the actual EPMA instrument installed in APEX facility in its final configuration with the modular shielded enclosure, inert atmosphere confinement enclosure, and support systems and utilities. Final operating procedures, remote handling tools/aids, and maintenance procedures also will be used.

Some elements are volatile under the electron beam (e.g., Na, Cs, and Am). If these elements are radioactive, they will be volatilized as radioactive vapors and will condense on the walls and detectors in the sample chamber. Personnel servicing the chamber will need to take apart the column while under respiratory protection and determine the extent of contamination before proceeding.

### 3.2.6 Required Data

Record data proximal and external to vacuum chamber versus time for the duration of examination to ensure the parameter can be adequately controlled:

- Temperature and humidity
- Acoustic noise readings
- Vibration (e.g., accelerometer) readings
- EMI readings.

### 3.2.7 Maturation Testing Location

The APEX facility located within MFC at INL.

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### 3.2.8 Data Requirements

Data requirements include results of temperature/humidity, acoustic noise, vibration, and EMI data recordings.

### 3.2.9 Maturation Testing Evaluation Criteria

The first evaluation criterion for SO&A testing will be successful demonstration of the EPMA examination.

Second, all routine maintenance and decontamination activities must be demonstrated to be feasible, safe, and effective.

The SO&A test will be repeated, if necessary and after appropriate corrective actions, until acceptance criteria are satisfied.

### 3.2.10 Maturation Testing Deliverables

The primary deliverable from this maturation activity will be a completed SO&A test plan report with signatures of duly authorized personnel indicating acceptance.

### 3.2.11 Resource and Duration Estimates

Although the EPMA is available for purchase as a shielded instrument, there are many advantages to pre-staging the instrument in IMCL. These include demonstrating access for remote operation and maintenance; providing advance operator training; and establishing a maintenance baseline for needed spares and frequency of service.

- Lead-times for instrument and estimate of cost
  - 18 to 24 months to establish instrument-specific requirements, design, fabricate, test, deliver, setup, and verify installation
  - Currently, shielded EPMA's cost \$3.9 million (2013 \$US)
- Pre-staging EPMA in IMCL for remote operation
  - Designers (electrical/mechanical)
  - Manufacturer's representative (support subcontract)
  - Remote engineering

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- EPMA researcher/technician
- Craft support (machinists, laborers, pipefitters, or electronics technicians)
- Radiological engineering and radiological control support

Table 1. Time estimates for the Advanced Post-Irradiation Examination Capabilities Project.

Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Investigate Decon Methods								5			5		50	20	80
Obtain Instrument Information	15												10		25
Develop Conceptual Design of Instrument	80	80					10	10	5		5	40	10	10	250
Write Instrument Specification	40						10	10	5	5	5	40	10	10	135
Bid and Award Instrument	10						40	5		5		10	5		75
Procure Instrument	5						80						5		90
Develop Installation Design	80	80						5		5	5		5		180
Design and Procure Remotization Equipment	40						20	5	5	5		10	5	5	95
Configure Shielding	40		80	80	80	40		5	10	5	10				350
Install Instrument	40		40	40		40			5		5		5	5	180

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Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
(vendor support included in purchase)															
Cold Remote-Handling Demonstration	20	10	10	10		10		10	10	5		10	40	80	215
Hot Sample Remote-Handling Demonstration	20	5	5	5				20	10	5	20	10	20	80	200
Authorize Routine Operations	10							10	10		20		10	40	100
Relocate to Advanced PIE Capabilities Project (TBD)	40	20	40	40		40		10	10	10	10		20		240
Contract with Vendor to Relocate (add \$100k)	10						40					10	10		70
Total	450	195	175	175	80	130	200	95	70	45	85	130	205	250	2,285

#### 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.*

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**5. REFERENCES**

PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

**6. APPENDIXES**

None.

## **Technology Maturation Plan**

# **Advanced Post-Irradiation Examination Capabilities Project Secondary Ion Mass Spectrometer**



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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available secondary ion mass spectrometer (SIMS) for advanced post-irradiation examination (PIE) of nuclear fuels and materials. This effort is a part of the Advanced Post-Irradiation Examination Capabilities (APIEC) Project to design, construct, and deploy the Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

For the purpose of this plan, a CAMECA IMS 7fR shielded SIMS will be assumed. No other manufacturers of dynamic shielded SIMS are known.

The shielded IMS 7fR can handle and analyze irradiated nuclear fuels emitting gamma radiations up to 3 curies at 0.75 MeV. It is believed that the IMS 7fR can be customized, as needed, for remote operation in the APEX facility:

- The instrument (ion source column and sample stage) is installed in a “hot” cell (lead or concrete shielded room).
- The instrument will be remote-controlled (e.g., stage or source), with electronics and a computer located outside the “hot” cell environment.
- Telemanipulators will be used to insert and mount the radioactive samples.
- Detectors are shielded by a location outside the “hot cell” to prevent the background caused by gamma radiations.
- The automation system controls all parameters of the SIMS and offers features for quantitative analysis, line profile acquisition, and data processing.

### 1.3 Instrument Description

SIMS instruments are used to analyze the composition of sample surfaces and thin films by sputtering the surface of the specimen with a focused primary ion beam and collecting and analyzing emitted secondary ions from the sample surface. These secondary ions are measured with a mass spectrometer to determine the elemental, isotopic, or molecular composition of the surface analytes. Shielded SIMS instruments are applicable for use with radioactive samples, with specific

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models having submicron spatial resolutions or high mass resolution capable of analyzing actinides and fission product elements with high sensitivity for very low-concentration analytes. By their very nature, imaging SIMS instruments provide high resolution and detect all elements of the periodic table (specific models) (e.g., the CAMECA Model IMS 7f-GEO has upper mass limits around 240 amu).

The project initially considered a nano-SIMS instrument that provides a sensitive surface analysis instrument capable of high lateral resolution down to 50 nm and the simultaneous identification of up to seven masses using the multicollector system. Unfortunately, the nano-SIMS is not adapted to high-radiation environments and is not capable of the high mass resolution ( $M/\Delta M > 10,000$ ) required to eliminate mass interference when analyzing actinides and fission product elements.

A single SIMS instrument is planned for the APEX facility. CAMECA manufactures a shielded IMS 7fR that is specifically developed for analysis of highly radioactive and irradiated nuclear materials. The IMS 7fR SIMS instrument combines extreme sensitivity, high mass resolution, high dynamic range and precise measurement of isotopic ratios, although it is limited to 0.25 micron lateral resolution in the direct ion mapping (microscope) mode and 0.5 micron lateral resolution in the scanning ion image (microprobe) mode. However, using post-processing with available analysis software, the IMS 7fR is capable of up to 200-nanometer resolution (this is dependent on the target element and matrix). The IMS 7fR is a magnetic sector SIMS, having one electron multiplier detector capable of detecting a single mass range in the microprobe mode; however, magnet-controlled peak jumping on a single multiplier is achievable, allowing the instrument to cycle quickly between target masses. This allows for multiple elements to be collected within a single analysis. In the direct ion mapping (microscope) mode, the IMS 7fR acquires images up to a thousand times faster than with any scanning ion microprobe (all pixels are acquired in parallel). Additionally, simultaneous isotope collection of all isotopes and all masses is achieved, although at reduced mass resolution.

The previous generation's shielded imaging SIMS (IMS 6fR) has been successfully installed at both Commissariat à l'énergie atomique Cadarache, France and the Institute for Transuranium Elements in Germany. The IMS 7fR is delivered with shielding capable of reducing a 1-MeV, 400-rem/hr source to a less than 0.5-mrem/hour dose rate. It also is provided with an alpha-tight glove box complete with a sample transfer connection method. Some modifications may be necessary in order to utilize the proposed rabbit transfer system with the IMS 7fR.

It is anticipated that the IMS 7fR will use compressed nitrogen to both vent the chamber vacuum system and to operate the pneumatic components. The

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pneumatics system requires a 5 to 7-bar (72.5 to 101.5-psi) supply, while the vent system requires a maximum of 1 bar (14.5 psi) nitrogen with N48 purity (99.998% pure). An N48 purity oxygen supply is required for the 100-liter duoplasmatron primary ion source provided with the instrument.

Analysis of radioactive samples requires the protection of the detector instrument and the operator against radiation during sample transfer and analysis. Also, the sputtering of a micro-volume of the analyzed sample in the instrument chamber during the analysis requires the knowledge of the contamination produced and the possibility to easily decontaminate the exposed parts.

A pneumatic transfer cask allows safe transportation of the radioactive sample to the IMS 7fR cell. The sample is then introduced in a glove box via an alpha-tight door. It is opened with a manipulator and the sample is placed in the entry air lock. Instrument venting and sample transfers into the specimen chamber are fully automated.

The SIMS includes complete computer control of instrument tuning and operation. The alignment of sources, ion gun, primary and secondary optical system, and channel plate are performed via a dedicated keyboard, including full remote control of slits and diaphragms.

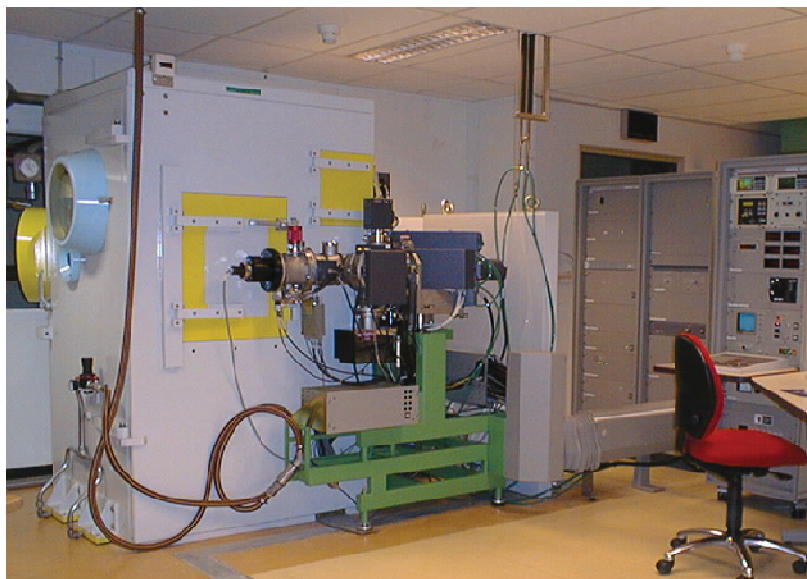


Figure 1. IMS 6fR installation in Commissariat à l'énergie atomique Cadarache (France).

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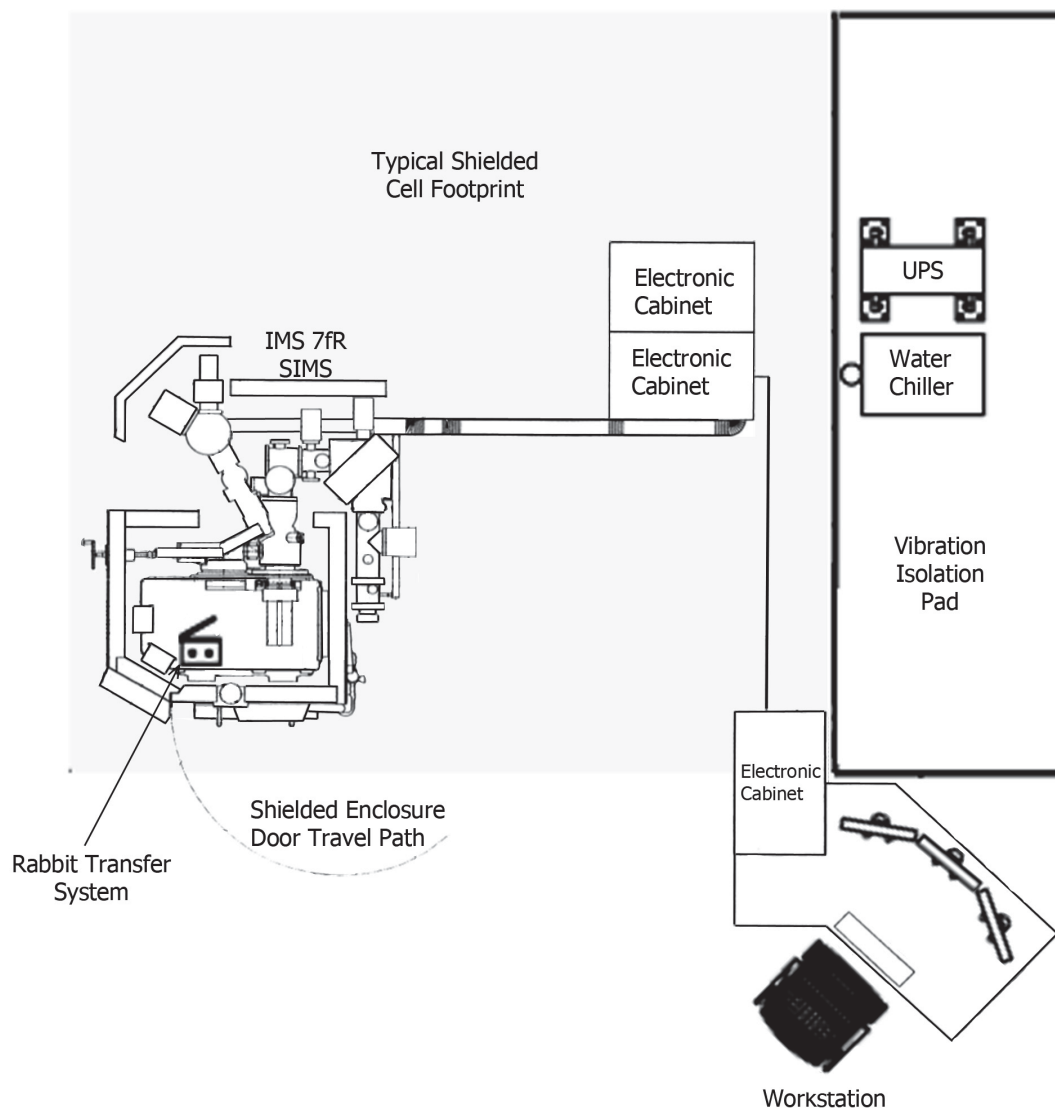


Figure 2. IMS 7fR, shielded secondary ion mass spectrometer conceptual layout.

A SIMS's analysis of solid materials requires preparation of flat, polished sections. A brief protocol is provided as follows:

1. Nearly any solid material can be analyzed. Samples must be less than 25.4 mm in diameter and 12.7 mm in thickness. Sample preparation needs to provide relatively flat sample surfaces. Mechanical polishing of the

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samples is appropriate; however, planar or flat samples can be mounted directly for SIMS analysis.

2. In most cases, samples are prepared as met-mounts. For SIMS, the sample surface must be highly polished (about 1 micrometer).
3. Most silicate minerals and nuclear fuels are electrical insulators. Directing an ion beam at the sample can lead to electrical charging of the sample, which must be dissipated. The IMS 7fR utilizes unique charge compensation for eliminating charging effects on insulating samples. The normal incident electron gun available on the IMS 7fR uses an electron gun to provide continuous feedback to remove charge buildup on the sample surface without flooding the sample with excess electrons.
4. Samples are loaded into the sample chamber via a vacuum interlock and mounted on the sample stage. The sample chamber is then pumped to obtain a high vacuum.
5. To begin a SIMS analysis, suitable analytical conditions must be selected (such as accelerating voltage and ion beam current) and the ion beam must be properly focused. If quantitative analyses are planned, the instrument first must be standardized for the elements desired.
6. Different types of sample holders can be introduced in the specimen sample chamber. Three standard sample holders are delivered with the instrument. These sample holders are as follows:
  - One hole of 20-mm diameter for loading one sample with a diameter greater than 22 mm and up to 25.4 mm (1 in.)
  - Four windows of 6 x 6 mm<sup>2</sup> size, for loading four samples maximum. Sample size must be greater than 6 x 6 mm<sup>2</sup> and up to about 8 x 8 mm<sup>2</sup>, typically.
  - Seven windows (four windows of 6 x 6 mm<sup>2</sup>, two windows of 2 x 7.5 mm<sup>2</sup>, and one window of size 2 x 2 mm<sup>2</sup>) for loading up to four samples with size greater than 6 x 6 mm<sup>2</sup>, as well as smaller samples with size greater than 2 x 2 mm<sup>2</sup>.

For all sample holders, the sample thickness must be less than or equal to 12.7 mm.



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7. High-resolution color charge-coupled device camera, with optical zoom system, provides a sample surface image while being in the analysis position.

#### 1.4 Current Technical Maturity

The Advanced PIE Capabilities Project uses a technology readiness level (TRL) maturity scale of 1 to 9, where 1 is just observed phenomena and 9 represents full deployment with operating experience. SIMS technology currently is at TRL-7 as described below (see Table 1):

**“System demonstrated in final configuration** – Actual instrument has been successfully operated in final configuration and in relevant environment (including radiation and remote operation). This represents a major step up from TRL-6, requiring demonstration of an actual system in a relevant operational environment with limited degradation due to radiation exposure.”

Table 1. Secondary ion mass spectrometer technology maturity by attributes.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Secondary Ion Mass Spectrometer (SIMS)	7	7	7	7	7	7	7	7	7.0

Because the IMS 7f has been deployed internationally for irradiated fuels in a hot cell “relevant environment,” this plan will describe the actions necessary to install it into the APEX facility. This detail will be sufficient to estimate the cost and schedule to achieve this deployment. TRL-8 represents the following:

**“Integrated readiness of the system is demonstrated** – Technology is proven to work. Actual technology is completed and qualified through test and demonstration. Facility issues a declaration of readiness and readiness activity is successfully completed.”

The plan addresses the technology maturation activities necessary to transition the SIMS from TRL-7 to TRL-8, including associated test descriptions, conditions, and configurations as identified in Section 3. Verification of the SIMS and shielding performance to achieve TRL-8 is expected to be demonstrated in the APEX facility using sealed radiological sources.

It may be desirable, but not mandatory, to mockup the SIMS in the Irradiated Materials Characterization Laboratory (IMCL) to optimize robotic controls and shielding placement. To accomplish this, the new SIMS initially would be setup in IMCL and then subsequently moved by the vendor to the APEX facility.



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The SIMS will be delivered and set up by the vendor, including all initial calibration. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 through the processing of the first production specimens). While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test plans and procedures, work orders, or laboratory instructions that will be used to direct the actual work.

The end user organizations for the matured and deployed SIMS are expected to be MFC Operations and Nuclear Science and Technology.

The specimens to be examined and processed in the APEX facility are expected to have high radiation levels. This level is currently assumed to be 600 R/hour (i.e., three TRISO fuel particles at 200-R/hour each). An initial target value for the shielded instruments in the APEX facility is to provide the capability to examine specimens measuring up to 3 Ci of activity at 1-MeV gamma radiation.

The vendor will specify acceptable electro-magnetic interference (EMI) criteria when the instrument is specified, and may visit the site to measure the EMI field to determine whether the location is suitable or requires mitigation.

The instrument should be procured with an uninterrupted power supply that can last 30 minutes. The uninterrupted power supply is located outside the hot cell; therefore, it is not exposed to radiation. The instrument produces heat and will require cooling in the hot cell environment.

There needs to be enough room around the instrument to allow worker access to maintain the components. In addition, room for a stair ladder or laboratory stool is required so that work in the top of the column can be accomplished. Several feet (3 to 4 ft) between the front of the SIMS and the hot cell wall are required. Maintenance typically is performed every 3 to 6 months during operation.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

## 3. MATURATION TEST PLANS

### 3.1 General Information

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

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### 3.1.1 Assumptions

1. This plan uses a CAMECA ISM 7f shielded SIMS instrument as a reference baseline to identify and define the necessary technology maturation activities.
2. The shielded SIMS will be a special order unit that has been modified or built to meet INL specifications for service in a high-radiation environment. Specifically, these modifications may include, but are not limited to, substitution of standard wiring, vacuum seals, and surface finishes/coatings with ones that are compatible with high-radiation fields and, where applicable, a radiologically contaminated environment (including considerations for decontamination solutions that are anticipated to be used).
3. SIMS is delivered with a set of spare parts and a set of eight reference samples for calibration.
4. Special customization may require additional technology maturation activities if verification of compliance is not performed by the instrument manufacturer.
5. The SIMS is assumed to be initially deployed in the APEX facility. However, if cost and schedule allow, the instrument may be pre-staged in IMCL to allow mockup of shielding and to demonstrate remote operations within INL protocols.

### 3.1.2 System Accessories and Add-Ons

The following system enhancements are available to customize the SIMS. For the purpose of the conceptual design cost estimate, it is assumed that the most capable instrument will be specified.

- High brightness duoplasmatron source producing  $O_2^+$ ,  $O^-$ ,  $Ar^+$  ions.
- High brightness cesium microbeam source.
- Normal incidence electron beam colinear with the secondary ion beam. Electron accelerating voltage adjustable up to -10 kV.
- Oxygen flooding attachment directs a stream of oxygen onto the analyzed area to increase the positive secondary ion yield.

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- Extraction system for the duoplasmatron source of the IMS 7f (DUO-ACCEL/DECEL). It makes possible to run analyses with  $O_2^+$  primary ions at impact energies as low as 500 eV.
- Isolation valve of the cesium microbeam source.
- X and Y sample stage with eucentric rotation capability (RS10).
- Resistive anode encoder ion imaging detector.
- Z-axis manual movement for the sample stage (Z-MOTION).  
*Note: not compatible with the RS10 accessory.*
- Post-acceleration for the electron multiplier. Allows operators to apply post acceleration to the secondary ions reaching the first dynode of the electron multiplier.
- One dry pump for the fore vacuum line of turbomolecular pumps (DRYPUMP1).
- Turbomolecular pump for the mass spectrometer (TURBOSPECTRO). Replacing the two ion pumps (in the spectrometer and the detection system) by two turbomolecular pumps offers an optimized pumping speed in the mass spectrometer, allowing it to maintain high-level performance of abundance sensitivity while using the oxygen flooding.
- Ultra fast airlock system for a fast sample introduction (ULTRA FAST AIRLOCK) speeds up sample holder exchange operation.
- Storage chamber for up to six sample holders (STORAGE CHAMBER). *Note: not compatible with the ultra fast airlock accessory.*
- Automated particle measurement (APM SOFTWARE Licence) significantly strengthens the current SIMS capabilities in terms of fast screening of large numbers of particles to select out particles of a specific element and to give an estimate of the isotopic composition and the location of each particle.
- Checkerboard software (CHECKERBOARD SOFTWARE License) allows the operator to greatly improve the data quality in some depth profile applications by optimizing both the gate

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size and position after the initial measurement has been performed.

- Instrument control from an operator room (DESK CONTROL DUPLICATION). The IMS 7f is delivered in standard with an operator desk control consisting of one PC computer with its mouse and keyboard, three screens and one CAMECA designed keypad; this desk control is installed nearby the tool. When the lab is split in two parts (one room for the instrument and one room for the operator control), the desk control duplication accessory must be added to the tool configuration for optimized operation comfort.
- Additional WinCurve software (WinCurve SOFTWARE License) is designed for curve processing and can read raw and processed data files from CAMECA PC and SUN automation.
- Additional WinImage Standard offline software (STANDARD WinImage SOFTWARE License) is available for external (offline) image processing of data acquired under Microsoft or Unix CAMECA acquisition software.

### **3.1.3 Prerequisites**

- INL and/or MFC personnel will develop a detailed specification for the SIMS, working with CAMECA technical support staff.
- A special order SIMS, modified as necessary for high-radiation environments, will be procured, delivered, configured, and calibrated to support technology maturation testing and development activities.
- INL and/or MFC engineering personnel will work with the instrument manufacturer to verify any other radiation-sensitive and/or vulnerable components that should be physically separated from the main instrument/vacuum chamber or that should be protected through installation of add-on shielding to improve radiation hardening. Design and performance verification of such modifications may either be done by the manufacturer prior to shipping or by INL personnel after receipt of the instrument.

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### 3.1.4 Identified Hazards and Safety Concerns

The primary hazard associated with SIMS maturation activities involves working with highly radioactive sealed sources that pose a risk for unanticipated worker exposures. Other hazards include, but are not limited to, electric shocks, hoisting and rigging, rotating equipment, compressed gases, and confined spaces with the potential for oxygen deficient atmospheres.

### 3.1.5 Work Controls, Permits, and Caution/Danger Tags

All work performed pursuant to this plan will be conducted using company standard work procedures, including work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## 3.2 Plan for Technology Readiness Level 7 to Technology Readiness Level 8 Transition

For TRL 8 to be achieved, the integrated readiness of the system must be demonstrated and qualified within the finished APEX facility, including its final interfaces to the modular shielded enclosure, the inert confinement enclosure, and associated utilities provided by the facility. Specifically, this means that the SIMS and associated processes have been proven to work, facility representatives have issued a declaration of readiness, and the readiness activities have been successfully completed. This readiness activity (typically an operational readiness review by the U.S. Department of Energy) will be performed on a facility basis and not for this specific PIE instrument.

### 3.2.1 Technology Maturation Testing Approach

The TRL-7 to TRL-8 maturation activities for the SIMS will be performed in conjunction with the readiness activity for the APEX facility. The facility readiness activity confirms that management has brought the facility to a state of readiness to commence program work. Once management concludes that readiness has been achieved, this state of readiness is independently verified by the contractor and confirmed by the U.S. Department of Energy. Only then will the APEX facility (a Hazard Category 2 nuclear facility) be authorized to “go hot” and perform programmatic PIE work.

The SIMS portion of the overall facility readiness activity will involve the performance of a system operability and acceptance (SO&A) test for the instrument, modular shielded cell, and associated support systems.

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### 3.2.2 Technology Maturation Testing Objectives

The objective of the TRL-7 to TRL-8 maturation activities is to successfully demonstrate, during the facility readiness activity, the integrated SIMS instrument for an advanced PIE application, including the adequacy of the following:

- Instrument radiation-hardening measures and interfaces with the modular shielded enclosure and the inert atmosphere confinement enclosure
- Acoustical noise and vibration control measures appropriate for the facility and location
- EMI protection measures appropriate for the facility and location
- Remote sample handling process, including associated methods, tools, and aids
- Remote instrument operation as deployed within the modular shielded enclosure
- Any instrument modifications made to improve reliability, availability, and maintainability for its application for advanced PIE.

### 3.2.3 Technology Maturation Testing Description

The SIMS SO&A test will demonstrate correct system/process operation and verify that the installed instrument and associated modifications function as designed and in accordance with designated acceptance criteria and design inputs.

The SO&A will constitute an end-to-end test of the advanced PIE process for examining specimens, including ingress and egress from the modular shielded enclosure. A nonradioactive sample will be used for this transition test in the presence of an approximate 25-R/hour sealed gamma source.

After successfully demonstrating specimen examination, routine field maintenance (e.g., predictive/preventive maintenance actions and modular component replacements) and decontamination activities will be demonstrated.

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### 3.2.4 Maturation Testing Conditions

Performance metrics (such as spectrometer reproducibility), detector performance (signal to background), and image resolution are established at the factory before the instrument is shipped to the customer and are checked again after installation. These parameters need to be periodically checked by the customer to evaluate maintenance needs; however, it should be noted that these parameters can degrade over time whether or not the instrument is in a radiation field.

- Specimens: metallurgical mounts (met-mounts) examined in the presence of a sealed radiography source measuring about 25 R/hour  $\gamma$
- Biological shielding: as provided by the APEX facility modular shielded enclosure
- Sealed radiological source: about 25R/hour  $\gamma$
- Temperature:  $19^{\circ} < t^{\circ} < 23^{\circ} \text{ C}$ , with fluctuation less than  $1^{\circ}\text{C/hour}$
- Relative humidity:  $30\% < \text{RH} < 75\%$ , with fluctuation less than  $10\%/\text{hour}$
- Spurious electromagnetic field (after mitigation, if necessary, for measured APEX facility location conditions): less than  $3 \cdot 10^{-7}$  Tesla peak to peak
- Vibration:

Vibrations(1-Hz bandwidth)	Less than 5 $\mu\text{m}$ peak to peak	Frequency range 0.4 to 2.5 Hz
	Less than $10^{-3} \text{ m.s}^{-2}$ peak to peak	Frequency range 2.5 to 20 Hz
	Less than $6 \cdot 10^{-6} \text{ m/s}$ peak to peak	Frequency range 20 to 200 Hz

- Static electricity: Output resistance between ground charges should be between 0.1 and 2,000 Mohms.
- Power: 230 V ( $\pm 5\%$ )
- Frequency: 47 to 63 Hz



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- Power consumption: 9.4 kW
- Instrument air, compressed: 5 to 7 bar
- Ultra high purity nitrogen: a 10 m<sup>3</sup> bottle will ensure operation for approximately 1 year; the bottle must be equipped with a pressure relief valve (so that the downstream pressure is adjustable from 0 to 1 bar), and a fitting for incorporating an 8-mm internal diameter vinyl hose
- Ultra high purity oxygen (minimum purity of the gas used is 99.998% - N48): a 2.5-L bottle of oxygen compressed at 200 bars is supplied with the equipment: these will ensure operation for approximately 3 years and all gases should be able to be controlled without entering the hot cell
- Cooling water is circulated through a chiller with the medium of exchange being air or water; the former puts excess heat into the room that must be mitigated and the chiller should be outside of the hot cell.

The primary issue with these utilities is the ease of operation and maintenance. This will be facilitated by keeping these outside the hot cell, but close enough to the instrument that they operate correctly.

### 3.2.5 Examination Testing Configuration

The SO&A testing configuration will be the actual SIMS instrument installed in the APEX facility in its final configuration with the modular shielded enclosure, inert atmosphere confinement enclosure, and support systems and utilities. Final operating procedures, remote-handling tools/aids, and maintenance procedures also will be used.

Some elements are volatile under the ion beam (e.g., Na, Cs, and Am). If these elements are radioactive, they will be volatilized as radioactive vapors and can condense on the walls and detectors in the sample chamber. Personnel servicing the chamber will need to take apart the column while under respiratory protection and determine the extent of contamination before proceeding.



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### 3.2.6 Required Data

Record data proximal and external to vacuum chamber versus time for the duration of the examination to ensure the parameters can be adequately controlled:

- Temperature and humidity
- Vibration (e.g., accelerometer) readings
- EMI readings.

### 3.2.7 Maturation Testing Location

The APEX facility located within MFC at INL.

### 3.2.8 Data Requirements

Data requirements include the results of the temperature/humidity, acoustic noise, vibration, and EMI data recordings.

### 3.2.9 Maturation Testing Evaluation Criteria

The first evaluation criterion for the SO&A test will be successful demonstration of the SIMS examination.

Second, all routine maintenance and decontamination activities must be demonstrated to be feasible, safe, and effective.

The SO&A test will be repeated, if necessary and after appropriate corrective actions, until acceptance criteria are satisfied.

### 3.2.10 Maturation Testing Deliverables

The primary deliverable from this maturation activity will be a completed SO&A test plan report with signatures of duly authorized personnel indicating acceptance.

### 3.2.11 Resource and Duration Estimates

Although the SIMS is available for purchase as a shielded instrument, there are many advantages to pre-staging the instrument in IMCL, including demonstrating access for remote operation and maintenance; providing advance operator training; and establishing a maintenance baseline for needed spares and frequency of service.

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- Lead-times for instrument and estimate of cost
  - 18 to 24 months to establish instrument-specific requirements, design, fabricate, test, deliver, setup, and verify installation
  - Currently, a shielded SIMS instruments cost is \$7.4 million (2013 \$U.S.)
- Pre-staging SIMS in IMCL for remote operation:
  - Designers (electrical/mechanical)
  - Manufacturer's representative (support subcontract)
  - Remote engineering
  - SIMS researcher/technician
  - Craft support (e.g., machinists, laborers, pipefitters, or electronics technicians)
  - Radiological engineering and radiological control support

Table 1: Time Estimates for project.

Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Investigate Decon Methods								5			5		50	20	80
Obtain Instrument Information	15												10		25
Develop Conceptual Design of Instrument	80	80					10	10	5		5	40	10	10	250

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Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Write Instrument Specification	40						10	10	5	5	5	40	10	10	135
Bid and Award Instrument	10						40	5		5		10	5		75
Procure Instrument	5						80						5		90
Develop Installation Design	80	80						5		5	5		5		180
Design and Procure Remotization Equipment	40						20	5	5	5		10	5	5	95
Configure Shielding	40		80	80	80	140		5	10	5	10				450
Install Instrument (vendor support included in purchase)	40		40	40		40			5		5		5	5	180
Cold Remote-Handling Demonstration	20	10	10	10		10		10	10	5		10	40	80	215
Hot Sample Remote-Handling Demonstration	20	5	5	5				20	10	5	20	10	20	80	200
Authorize Routine Operations	10							10	10		20		10	40	100
Relocate to Advanced PIE Capabilities Project (TBD)	40	20	40	40		40		10	10	10	10		20		240

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Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Contract with Vendor to Relocate ( add \$150k)	10						40					10	10		70
Total	450	195	175	175	80	230	200	95	70	45	85	130	205	250	2385

#### 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.*

#### 5. REFERENCES

Bremier, S., R. Hasnaoui, S. Portier, O. Bildstein, and C. T. Walker, 2006, "Installation of a Shielded SIMS for the Analysis of Irradiated Nuclear Fuels," *Microchimica Acta*, 155, 113–120.

PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

Portier, S., S. Bremier, and C. T. Walker, 2007, "Secondary Ion Mass Spectrometry of Irradiated Nuclear Fuel and Cladding: An Overview," *Mass Spectrometry*, 263, 113–126.

Rasser, B., L. Desgranges, and B. Pasquet, 2002, "A New Shielded SIMS Instrument for Analysis of Highly Radioactive Materials," *Applied Surface Science*, 203-204, 673–678.

#### 6. APPENDIXES

None.

## **Technology Maturation Plan**

# **Advanced Post-Irradiation Examination Capabilities Project Simultaneous Thermal Analyzer**



The INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance.

Manual: APIEC[illegible]

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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available simultaneous thermal analyzer (STA) that combines a thermogravimetry analyzer (TGA) and a Differential scanning calorimeter (DSC) for successful routine application in advanced post-irradiation examination (PIE) of nuclear fuels and materials within the planned Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

This plan applies to an STA system that includes the functions of a TGA and DSC. Alternatively, this plan also would apply to a DSC system that does not include the TGA capability. The unit requirements and maturation activities for both systems are the same and, therefore, are interchangeable. The final system will be determined prior to the execution of this plan, based on further definition of future programmatic needs. For the sake of simplicity, this plan will continue to discuss the STA system. The unit has a furnace, vacuum system and chamber, power, and control consoles. The plan addresses the technology maturation activities necessary to transition the STA from a technology readiness level (TRL)-6 (minimum) to a TRL-8,<sup>1</sup> including associated test descriptions, conditions, and configurations as identified in Section 3. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 through processing of the first production specimens).

This plan applies to Advanced PIE Capabilities Project personnel and other MFC or INL personnel identified to support the STA technology maturation tests and activities. While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test procedures, work orders, or laboratory instructions that will be used to direct the actual work.

The end user organizations for the matured and deployed STA are expected to be MFC operations and Nuclear Science and Technology.

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<sup>1</sup>Refer to *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, INL/EXT-12-27849, for a description of TRL levels.

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### 1.3 Instrument Description

The STA instrument is a thermal instrument that combines the DSC and TGA functions in the same instrument. Combination of the two instruments allows for a single sample to be generated and tested, which provides both sample preparation and characterization efficiencies. Figure 1 shows a cross section of the instrument and the major components of the STA.

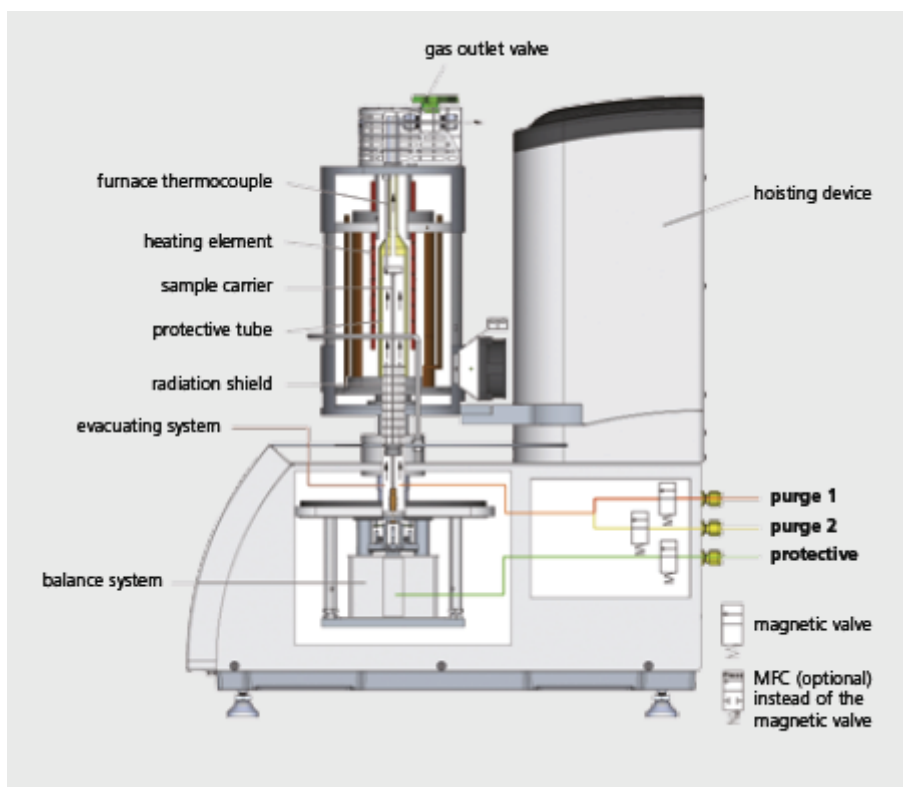


Figure 1. Simultaneous thermal analyzer 449 F3 instrument components.

The DSC is a thermal analysis technique in which the difference in the amount of heat required to change the temperature of a sample and reference material at the same rate is measured as a function of temperature or time. Both the sample and reference are maintained at the same temperature throughout the experiment. The DSC can be used to measure phase transformation temperatures; enthalpies of formation; heat capacity and calculated specific heat of a wide variety of materials, including metals, alloys, and ceramics; and composites depending on the selection of the furnace.

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TGA is a technique performed on samples to determine changes in mass as a function of temperature or time in a controlled atmosphere. The principle uses for TGA are to study a material's thermal stability and decomposition.

The STA can perform the following functions:

- Determine transition temperatures
- Determine heats of reaction at phase transitions
- Determine the heat capacity of a material
- Determine kinetics of reactions
- Determine mass change as temperatures vary
- Determine mass change as chemical reaction occurs under varying temperature or isothermal conditions.

#### 1.4 Current Technical Maturity

INL currently has an STA in MFC-768, Water Chemistry Laboratory, as well as DSCs and TGAs throughout the complex. Currently, these instruments are being used either on a bench top or in a glove box, which does not allow the examination of highly irradiated samples. Therefore, demonstration in a high-radiation environment is necessary to advance the instrument maturity for successful application in the APEX facility. The STA currently is assessed to be at a TRL-6 for this APEX application as explained in the *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment* report and depicted in Figure 2.

Table 1. Technology readiness level for the simultaneous thermal analyzer.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Simultaneous Thermal Analyzer	6	7	7	7	7	7	7	6	6.9

#### 1.5 Program Links

The following programs are stakeholders in the successful maturation and deployment of the STA in a shielded enclosure:

Light Water Reactor Sustainability – this program seeks to create economic and environmental benefits for existing nuclear power plants through improvements in

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reliability, availability, productivity, component life, safety, and security. Development of the scientific basis to allow evolution of fuels and understanding and predicting long-term degradation behavior and operational limits of materials relies on detailed PIE of reactor fuels and materials at the nano-scale.

Fuel Cycle Research and Development – this program has the responsibility of developing advanced fuel technologies using a goal-oriented, science-based approach. This approach requires a detailed thermal properties understanding of a broad spectrum of nuclear fuels and cladding materials.

Advanced Reactor Concepts – this program seeks to leverage the latest materials and fuels technologies to develop advanced reactor designs having improved efficiency and improved economics. The behavior of these materials and fuels in various reactor environments must be fully understood at the micro/nano-scale to support effective simulations and designs.

National Defense and Security – the National Nuclear Security Administration Naval Reactors program and other national security missions (e.g., nuclear non-proliferation) can benefit from nano-scale examination and characterization of nuclear materials and fuels in achieving their program objectives.

Nuclear Energy University Programs – this program plays a key role in helping the U.S. Department of Energy accomplish its mission of leading the nation's investment in development and exploration of advanced nuclear science and technology. It promotes nuclear energy as a resource capable of meeting the nation's energy, environmental, and national security needs by resolving technical, cost, safety, security, and proliferation resistance through research, development, and demonstration. Advanced PIE capabilities at APEX will provide student learning opportunities and help develop the nation's next generation of nuclear scientists and engineers.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

## 3. MATURATION TEST PLANS

### 3.1 General Information

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

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### 3.1.1 Assumptions

1. This plan uses an NETZSCH STA 449 F3 Jupiter instrument as a reference baseline to identify and define the necessary technology maturation activities. It is assumed that other STAs are available on the market and, having comparable capabilities and accessories, would require substantially similar activities to successfully deploy in an advanced PIE application.
2. The STA will perform the TGA and DSC measurements in a satisfactory level with the potential modifications that are proposed.
3. The STA procured is a special order unit that has been modified or built to meet INL and manufacturer agreed-upon specifications (TBD) for service in a high-radiation environment. Specifically, these modification may include, but are not limited to, the following:
  - Substitution of standard wiring, vacuum seals, furnace elements, and surface finishes/coatings with ones that are compatible with high-radiation fields and, where applicable, a radiologically contaminated environment (including considerations for decontamination solutions that are anticipated to be used).
  - After instrument delivery, the manufacturer will work with INL engineering and use a cold mockup cell to develop sample handling tools, fixtures, and aids for instrument deployment in a hot cell.

**NOTE:** *Additional technology maturation activities will need to be added to address these modifications if they are not available from and performed by the instrument manufacturer. Activities to substitute such items, after delivery, with ones that are compatible for use in a radiation environment are not included in this plan.*

### 3.1.2 Prerequisites

1. A special order STA, modified for compatibility with high-radiation environments, has been procured and is available to support technology maturation testing and development activities.

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2. INL and/or MFC engineering personnel have worked with the instrument manufacturer to identify radiation-sensitive and/or vulnerable components that can be removed and physically separated from the main instrument. Design and performance of such modifications may either be done by the manufacturer prior to shipment or by INL personnel after receipt of the instrument.

### 3.1.3 Identified Hazards and Safety Concerns

The primary hazard associated with STA maturation activities involves working with highly radioactive sources, which pose a risk for unanticipated worker exposures. Other hazards include, but are not limited to, radiological contamination, electric shocks, hoisting and rigging, rotating equipment, compressed gases, and confined spaces with the potential for oxygen deficient atmospheres.

### 3.1.4 Work Controls, Permits, and Caution/Danger Tags

All work performed pursuant to this plan will be conducted using company standard work procedures for work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## 3.2 Plan for Technology Readiness Level-6 to Technology Readiness Level-7 Transition

At TRL-7, the integrated system should be demonstrated in its final configuration and successfully operated in its relevant environment (including radiation and remote operation). This transition represents a major step up from TRL-6, requiring demonstration of a STA in a relevant operational environment with limited degradation due to radiation exposure.

### 3.2.1 Technology Maturation Testing Approach

To progress the STA from TRL-6 to TRL-7, the team will first work with NETZSCH to modify the instrument to operate in a high-radiation environment and by remote means. The team will leverage the current development activities that are integrating an STA to operate in a glove box in the Analytical Laboratory. The team will verify instrument

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functionality after radiation hardening<sup>2</sup> of the procured instrument. Approaches for ensuring an adequate instrument environment (e.g., temperature, humidity, vibration, and electromagnetic interference [EMI]) will be investigated to ensure the instrument works properly. A known sample will be tested to verify the unit is operating according to the design requirements. Remote sample handling and remote operation also will be investigated using a mockup cell.

Once the instrument has been verified as adapted to a hot cell environment, it will be relocated and reinstalled in the APEX facility, where it will be integrated into the actual radiological shielding (i.e., walls, windows, and transfer ports). Final testing of the confinement enclosure interface, remote sample handling, and remote operation will be accomplished and final the instrument functionality will tested in a relevant environment.

Optional scope for component lifetime estimation and radiation field envelope determination also is identified in the event that resources (time and funding) are available after completion of other maturation tasks and testing.

### 3.2.2 Technology Maturation Testing Objectives

1. Verify STA functionality and acceptable performance after:
  - (a) removal and physical separation of any radiation-sensitive components from the main instrument, and (b) installation of shielding in strategic locations to protect remaining radiation-sensitive components not addressed in (a).
2. Investigate, through mockup testing, various approaches for integrating the STA into an inert atmosphere confinement enclosure with glove ports with argon purge capability.
3. Investigate the feasibility of and verify through mockup testing various approaches for remote specimen handling, beginning with specimen arrival in the rabbit (mockup) and including various STA carrier configurations, specimen loading into and unloading from the sample carrier, and using the automatic sample changer as an option.

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<sup>2</sup>Radiation hardening measures may include removal and relocation of radiation-sensitive components and/or installation of strategically located shielding materials to protect radiation-sensitive components that cannot be relocated.



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4. Evaluate instrument performance in a relevant environment (i.e., temperature, humidity, vibration, and EMI) and investigate, through mockup testing, the feasibility and adequacy of proposed measures/approaches for providing environmental mitigation or control.
5. Investigate the feasibility of and verify through mockup testing various remote operation and maintenance approaches or methods for the STA components as deployed. This includes, but is not limited to, normal remote instrument operations such as power on, check out/diagnostics, methods for remote sample loading (including automatic sample changer if applicable), balance and hoisting device operation instrument and/or support equipment adjustments, instrument and process monitoring, and power off. Maintenance approaches and methods investigated will include, but are not limited to, remote sample loading, automatic sample changer, balance, hoisting device, and serviceability or modularization improvements implemented to facilitate replacement of components having shortened life spans due to high-radiation fields.
6. Verify radiation-hardened STA functionality and acceptable performance for a specimen in a relevant radiation field of about 25 R/hour  $\gamma$  on contact.
7. Verify acceptability of the final selected serviceability or modularization methods implemented to facilitate replacement of components having shortened life spans due to high-radiation fields in conjunction with final clearances and telemanipulator, shield wall, transfer port, and window configurations/positions.

### 3.2.3 Other Optional Maturation Testing Scope

1. Perform individual component testing of non-radiation hardened components of the main instrument (i.e., that cannot be relocated or adequately shielded) to determine expected life spans of the components as a function of radiation field intensity ( $\alpha$ ,  $\beta$ , and/or  $\gamma$ ). This information will be useful in setting limits, if appropriate, for the specimens to be examined/processed and for establishing associated maintenance and spares strategies that are economically supportable.



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### 3.2.4 Technology Maturation Testing Description

The maturation activities and testing provide an opportunity to verify the STA, as modified for high-radiation service, within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility. First, this test will verify that instrument modifications have not degraded functionality or performance to unacceptable levels.

Second, the mockup enclosure (i.e., with simulated shielding panels, windows, and transfer ports and with actual telemanipulators) will be used to investigate various instrument layouts, cell configurations, and associated interfaces with the inert confinement enclosure. The mockup easily will be reconfigured to support quick setup of alternative configurations for team review and evaluation. The use of actual telemanipulators will provide realism during process development testing and demonstration.

Third, process development testing will be performed to identify effective methods, equipment, and special tooling to facilitate handling, loading, and unloading of specimens.

Fourth, testing of the modified STA in the Irradiated Materials Characterization Laboratory or other like facility, within a full-scale, but temporary, shielded cell enclosure similar to that to be used in the APEX facility with a known non-irradiated sample, will be tested to verify the unit is operating according to the design requirements with a sealed radiation source present in the cell. The enclosure will be configured and instrumented to verify and validate the final approaches, methods, and layout (positioning) chosen based on earlier results. This includes any final mitigation measures implemented to correct the environmental constraints.

Fifth, the STA will be moved and integrated into the APEX facility for final installation and testing with a representative radiation sample (i.e., minimum of 25 R/hour  $\gamma$ ). The test also verifies integration of the final confinement enclosure and associated instrument/enclosure interfaces; final process equipment, tools, and aids; remote sample handling procedures; and remote instrument operation and maintenance activities under a radiological environment. An investigation of maintenance, servicing, and replacement of modularized components having shortened life spans also will be investigated.

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### 3.2.5 Maturation Testing Conditions

- Specimens: typical STA sample, nonradioactive (e.g., steel or sapphire samples) with known thermal properties
- Temperature: stable temperature  $\pm 2.5^{\circ}\text{C}$  for 1 hour prior to starting exam
- Relative humidity: TBD%
- Acoustics: TBD
- Vibration: TBD
- Available utilities:
  - Power: 220 V; 20 amp; single-phase; standard 120 V
  - Process gases: ultra high purity argon
  - Instrument air, compressed: 10 to 20 psi.

### 3.2.6 Maturation Testing Configuration

Work with NETZSCH to produce an STA that can be integrated into a glove box and hot cell.

There will be a mockup instrument enclosure (full scale, reconfigurable) for cold testing with actual telemanipulators (make/model TBD) but simulated (a) shielding walls, (b) shielded windows, and (c) shielded transfer port(s).

The STA will be tested in a radiation-hardened configuration and instrumented for obtaining environmental conditions such that measurements can be performed to verify the STA is working properly in a glove box and hot cell configuration. This may include temperature, humidity, acoustic noise, vibration, and EMI measurements.

There will be a shielded instrument enclosure for hot testing with actual telemanipulators (make/model TBD) and real (a) biological shielding blocks/panels, (b) shielded windows, and (c) transfer port(s). Shielding shall be adequate to provide personnel protection (i.e., less than 0.5 mrem/hour at 30 cm) for all radiological sources used during testing under this plan.

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The STA will be tested in a radiation-hardened configuration and instrumented for radiation field, temperature, acoustic noise, vibration and EMI measurements. Radiological monitoring instruments and controls also shall be in place for supporting personnel protection.

### 3.2.7 Required Data

- Environmental readings as appropriate (e.g., temperature/humidity plots versus time)
- STA readings from a known material to verify the unit is working as designed
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup)
- STA readings from a known radioactive sample to verify the unit is working as designed in a radiation field
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup).

### 3.2.8 Maturation Testing Location

Maturation activities and testing will be accomplished in the Irradiated Materials Characterization Laboratory or a comparable laboratory setting capable of achieving the conditions listed in Section 3.2.4 for the STA.

Prior to the activities that require the STA to test a highly irradiated sample in the furnace, the STA will be relocated to the APEX facility for the remaining testing being performed in the final configurations.

### 3.2.9 Data Requirements

Data quality (e.g., accuracy and precision) will be sufficient for the intended application and associated pass/fail decisions to be made. Specific data quality requirements are TBD and will be documented in detailed test plans, laboratory instructions, or other work control documents that are used to perform the maturation and testing activities.

Documentation of collected data and generated evaluations, assessments, and other results shall be retained as project records. Handling of

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documents shall be in accordance with PLN-4156, “Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project.”

### 3.2.10 Maturation Testing Evaluation Criteria

The following criteria apply for determining whether the maturation and testing activities can be considered successful in transitioning the STA from TRL-6 to TRL-7:

- Radiation hardening measures for the STA will be evaluated through proper instrument functioning and confirmed through testing on known materials. Determination of a maximum advisable radiation field and component longevity in relevant radiation fields will be done either as optional scope (if sufficient time and resources are available) or after turnover to operations.
- Acceptance of final instrument layout and shielded cell configuration (including the inert confinement enclosure) will be determined based on inputs from instrument technicians, facility operations, and maintenance personnel.
- Mitigation measures for ambient environmental conditions (i.e., other than radiation levels and including temperature, humidity, acoustic noise, vibration, and EMI levels) shall be evaluated against manufacturer’s recommendations for proper instrument operation and confirmed through testing on known irradiated material.

### 3.2.11 Maturation Testing Deliverables

A maturation testing report will be issued to document the results of mockup testing. The report will include any unresolved issues or recommended changes that need to be addressed in future TRL maturation activities.

### 3.2.12 Resource and Duration Estimates

Lead-times for instrument and estimate of cost:

- 12 to 18 months for NETZSCH to develop and deliver with modifications to be integrated into the hot cell

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- An STA was purchased for integration to the glovebox for about \$200K; it is assumed that it will be about 30% more to purchase the instrument for a hot cell.

STA modifications (for radiation hardening and remote operation):

- Designers (electrical/mechanical)
- Manufacturer's representative (support subcontract)
- Remote engineering
- STA researcher/technician
- Craft support (e.g., machinists, laborers, pipefitters, or electronics technicians).

Mockup:

- Designers (electrical/mechanical)
- Remote engineering
- STA researcher/technician
- Radiological engineering and radiological control support
- Craft support (e.g., machinists, carpenters, laborers, pipefitters, and electricians).

### 3.3 Plan for Technology Readiness Level-7 to Technology Readiness Level-8 Transition

In order to achieve TRL-8, the operational readiness of the integrated system must be qualified through test and demonstration and the facility must issue a declaration of readiness (see Table 2). No new tests or demonstrations will be performed to transition from TRL-7 to TRL-8. Rather, the same tests and demonstrations used to transition from TRL-6 to TRL-7 will be repeated as required as part of the facility operational readiness review. The tests and demonstrations need not be as detailed or extensive. The primary purpose is to demonstrate acceptable operability in the final operating environment (i.e., in a hot cell in the APEX facility).

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Table 2. Time estimates for project.

Description	Engineering	Draft	Mechanical	Electrical	Crafts	Procurement	System Engineer	Safety	Quality Assurance	Review	Principal Investigator	Operator	Total
Instrument selected	0												0
obtain instrument info	14												14
Develop conceptual design of instrument	120	120					4	4	4	8			260
write instrument specification	40					4	4	4	4	8			64
bid and award instrument	4					40	4	4	4				56
procure instrument	0					80							80
develop installation design	80	80											160
procure remotization equip	40					10	4	4	4	4			66
assemble mockup	40		80	80	80		8	4					292
install instrument	40		80	80	80		8	4					292
cold remote handling demonstration	10						10	10			40	40	110
disassemble cold test	10		40	40	40		10						140
reassemble in hot cell	40	40					20				10	60	170
hot demonstration	10						10	10			10	40	80
disassemble hot test	10		20	20	20		10	10			10	60	160
install in APEX	80	80	40	40	40		10	10		8	10		318
demonstrate in APEX	10						10	10			10	40	80
	548	320	260	260	260	134	112	74	16	28	90	240	2342

#### 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.*

#### 5. REFERENCES

NETZSCH, "Simultaneous Thermal Analysis," MFC News, < [http://www.netzsch-thermal-analysis.com/uploads/tx\\_nxnetzschmedia/files/STA\\_449\\_F3\\_E\\_0912.pdf](http://www.netzsch-thermal-analysis.com/uploads/tx_nxnetzschmedia/files/STA_449_F3_E_0912.pdf) >.

PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

#### 6. APPENDIXES

None.

## **Technology Maturation Plan**

# **Advanced Post- Irradiation Examination Capabilities Project Laser-Flash Analyzer**



The INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance.

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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available laser flash analysis (LFA) instrument that can be used to measure the thermal conductivity of a material for successful routine application in advanced post-irradiation examination (PIE) of nuclear fuels and materials within the planned Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

This plan applies to a NETZSCH LFA 427 system, which has been selected as an assumed baseline instrument for purposes of technology maturation planning. The unit has a furnace, laser system, infrared sensors, and software. The plan addresses the technology maturation activities necessary to transition the LFA from a technology readiness level (TRL)-6 (minimum) to a TRL-8,<sup>1</sup> including associated test descriptions, conditions, and configurations as identified in Section 3. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 through processing of the first production specimens).

This plan applies to Advanced PIE Capabilities Project personnel and other MFC or INL personnel identified to support the LFA technology maturation tests and activities. While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test procedures, work orders, or laboratory instructions that will be used to direct the actual work.

The end user organizations for the matured and deployed LFA are expected to be MFC Operations and Nuclear Science and Technology.

### 1.3 Instrument Description

The LFA instrument is a thermal instrument that measures the thermal diffusivity of a specimen. These data are used in conjunction with specific heat and density as a function of temperature to calculate the thermal conductivity of a material.

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<sup>1</sup>Refer to *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, INL/EXT-12-27849, for a description of TRL levels.

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The sample is mounted on a carrier system, which is located within a furnace. After the sample reaches a predetermined temperature, a burst of energy emanating from a pulsed laser is absorbed on the front face of the sample, resulting in homogeneous heating. The relative temperature increase on the rear face of the sample is then measured as a function of time by a liquid-nitrogen-cooled infrared detector. Thermal diffusivity is computed by the software using these time/relative temperature increase data. Figure 1 shows the major subsystems of the LFA.

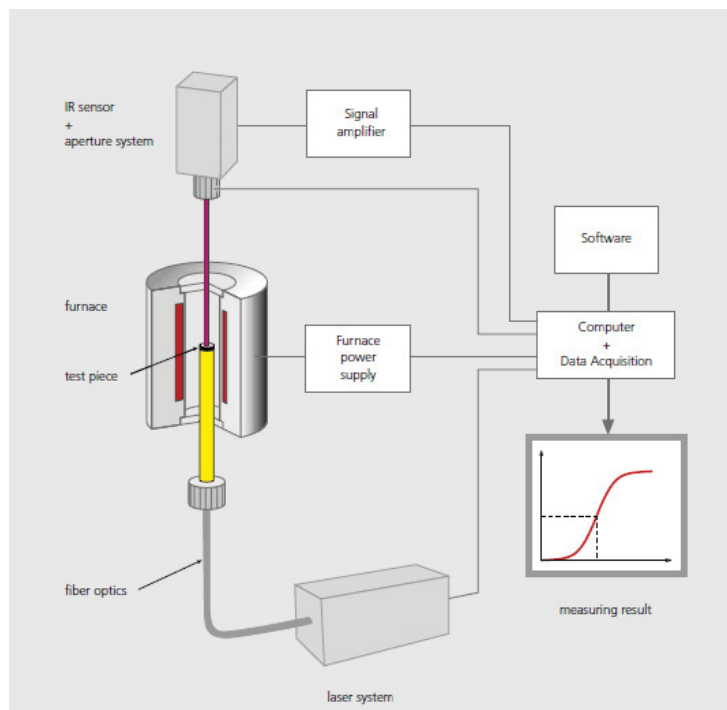


Figure 1. Laser flash analysis system block diagram.

The LFA 427 consists of the following four essential components (see Figure 2):

- Measuring unit with furnace, sample carrier, and infrared detector
- Controller for measuring unit
- Laser system connected via fiber optics
- Data acquisition system and computer.

The vertically constructed measuring unit is the primary component of the system. The laser system is connected with the measurement part by a sheeted glass fiber. The beam emerges from the outlet of the fiber optics. A recipient block is located above the laser optics. The tube-shaped sample holder and its adjusting device are

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mounted on this block. A toggle quick seal permits a vacuum/gas-tight coupling between the recipient block and the furnace. The furnace system is raised and lowered by means of a motor-driven hoist.

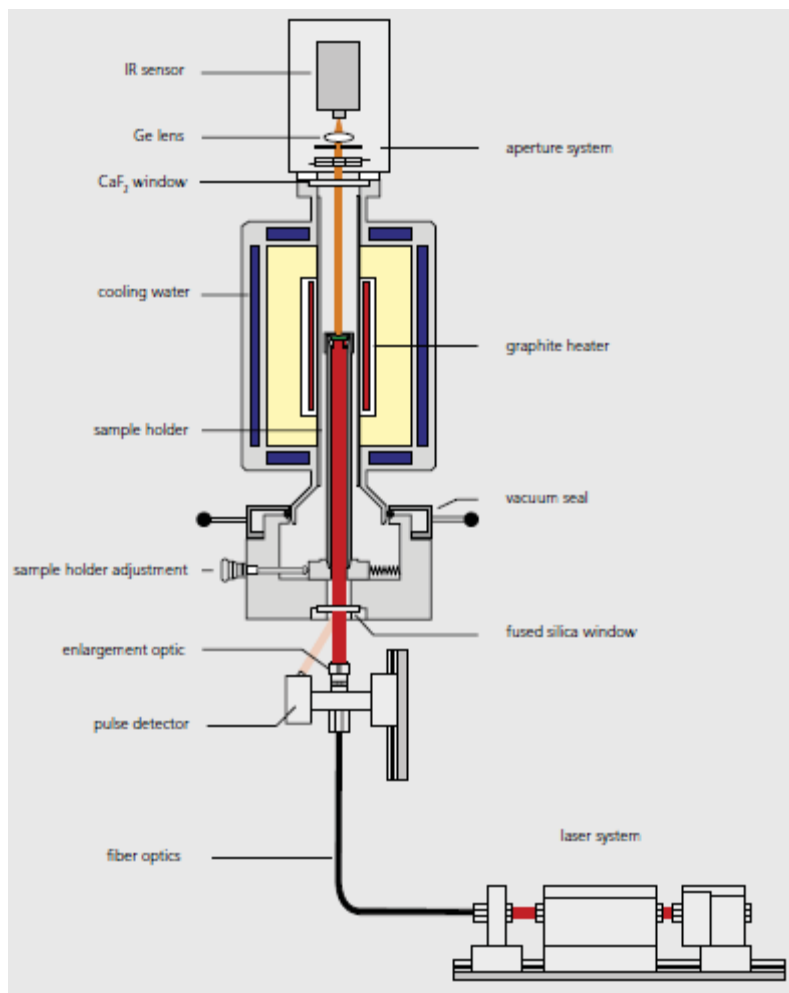


Figure 2. Essential components of laser flash analysis.

#### 1.4 Current Technology Maturity

INL currently has an LFA that has been developed for the fresh fuels glove box in MFC-752, Analytical Laboratory. INL has worked with NETZSCH to breakout the components of the LFA that are sensitive to radiation. Figure 3 shows an LFA that has been modified for glove box installation. The components that could be removed have been removed from the furnace, where the specimen will be loaded and tested.

The LFA currently is assessed to be at a TRL-6 for this application in the APEX facility (depicted in Table 1).

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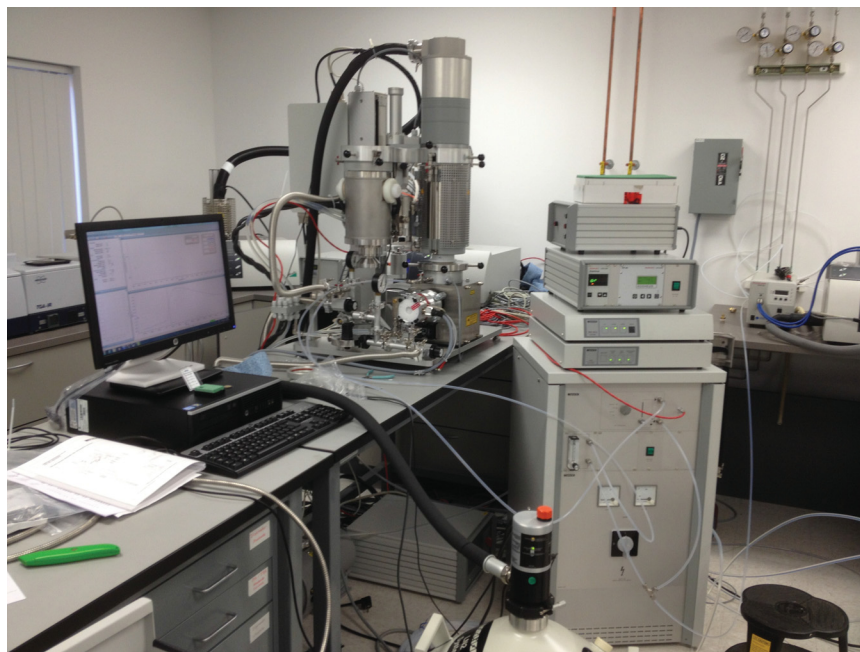


Figure 3. Laser flash analysis with critical components separated.

Table 1. Laser flash analysis technology readiness.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Laser Flash Analyzer (LFA)	6	7	7	7	6	6	7	6	6.6

## 1.5 Program Links

The following programs are stakeholders in successful maturation and deployment of the LFA in a shielded enclosure:

Light Water Reactor Sustainability – this program seeks to create economic and environmental benefits for existing nuclear power plants through improvements in reliability, availability, productivity, component life, safety, and security. Development of the scientific basis to allow the evolution of fuels and understanding and predicting long-term degradation behavior and operational limits of materials relies on detailed PIE of reactor fuels and materials at the nano-scale.

Fuel Cycle Research and Development – this program has the responsibility of developing advanced fuel technologies using a goal-oriented, science-based approach. This approach requires a detailed thermal properties understanding of a broad spectrum of nuclear fuels and cladding materials.

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Advanced Reactor Concepts – this program seeks to leverage the latest materials and fuels technologies to develop advanced reactor designs having improved efficiency and improved economics. The behavior of these materials and fuels in various reactor environments must be fully understood at the micro/nano-scale to support effective simulations and designs.

National Defense and Security – the National Nuclear Security Administration Naval Reactors program and other national security missions (e.g., nuclear non-proliferation) can benefit from nano-scale examination and characterization of nuclear materials and fuels in achieving their program objectives.

Nuclear Energy University Programs – this program plays a key role in helping the U.S. Department of Energy accomplish its mission of leading the nation's investment in the development and exploration of advanced nuclear science and technology. It promotes nuclear energy as a resource capable of meeting the nation's energy, environmental, and national security needs by resolving technical, cost, safety, security, and proliferation resistance through research, development, and demonstration. Advanced PIE capabilities at the APEX facility will provide student learning opportunities and help develop the nation's next generation of nuclear scientists and engineers.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

## 3. MATURATION TEST PLANS

### 3.1 General Information

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

#### 3.1.1 Assumptions

1. This plan uses a NETZSCH LFA 427 instrument as a reference baseline to identify and define the necessary technology maturation activities. It is assumed that other LFAs are available on the market and, having comparable capabilities and accessories, would require substantially similar activities to successfully deploy in an advanced PIE application.

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2. The LFA will perform the intended measurements in a satisfactory level with the potential modifications that are proposed.
3. The procured LFA is a special order unit that has been modified or built to meet INL and manufacturer agreed-upon specifications (TBD) for service in a high-radiation environment. After instrument delivery, the manufacturer will work with INL engineering and using a cold mockup cell to develop sample handling tools, fixtures, and aids for instrument deployment in a hot cell.

### 3.1.2 Prerequisites

1. A special order LFA, modified for compatibility with high-radiation environments, has been procured and is available to support technology maturation testing and development activities.
2. INL and/or MFC engineering personnel have worked with the instrument manufacturer to identify radiation-sensitive and/or vulnerable components that can be removed and physically separated from the main instrument. Design and performance of such modifications may either be done by the manufacturer prior to shipping or by INL personnel after receipt of the instrument.

### 3.1.3 Identified Hazards and Safety Concerns

The primary hazard associated with LFA maturation activities involves working with highly radioactive sources, which pose a risk for unanticipated worker exposures. Other hazards include, but are not limited to, radiological contamination, electric shocks, hoisting and rigging, rotating equipment, compressed gases, cryogenic liquid handling, and confined spaces with the potential for oxygen-deficient atmospheres.

### 3.1.4 Work Controls, Permits, and Caution/Danger Tags

All work performed pursuant to this plan will be conducted using company standard work procedures for work planning and control, radiological work permits, confined space work permits, and lockout/tagout.



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### 3.2 Plan for Technology Readiness Level-6 to Technology Readiness Level-7 Transition

At TRL-7, the integrated system should be demonstrated in its final configuration and successfully operated in its relevant environment (including radiation and remote operation). This transition represents a major step up from TRL-6, requiring demonstration of a LFA in a relevant operational environment with limited degradation due to radiation exposure.

#### 3.2.1 Technology Maturation Testing Approach

To progress the LFA from TRL-6 to TRL-7, the team will first work with NETZSCH to modify the instrument to operate in a high-radiation environment by remote means. The team will leverage the current development activities that are integrating an LFA to operate in a glove box in the Analytical Laboratory. The team will verify instrument functionality after radiation hardening<sup>2</sup> of the procured instrument. Approaches for ensuring an adequate instrument environment (e.g., temperature, humidity, vibration, and electromagnetic interference [EMI]) will be investigated to ensure the instrument works properly. A known sample will be tested to verify the unit is operating according to the design requirements. Remote sample handling and remote operation also will be investigated during this stage.

Once the instrument has been verified as adapted to a hot cell environment, it will be relocated and reinstalled in the APEX facility, where it will be integrated into the actual radiological shielding (i.e., walls, windows, and transfer ports). Final testing of the confinement enclosure interface, remote sample handling, and remote operation will be accomplished and final instrument functionality will be tested in a representative environment.

Optional scope for component lifetime estimation and radiation field envelope determination also is identified in the event that resources (e.g., time and funding) are available after completion of other maturation tasks and testing.

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<sup>2</sup>Radiation hardening measures may include removal and relocation of radiation-sensitive components and/or the installation of strategically located shielding materials to protect radiation-sensitive components that cannot be relocated.

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### 3.2.2 Technology Maturation Testing Objectives

1. Verify LFA functionality and acceptable performance after (a) removal and physical separation of any radiation-sensitive components from the main instrument, and (b) installation of shielding in strategic locations to protect remaining radiation-sensitive components not addressed in (a).
2. Investigate through mockup testing various approaches for integrating the LFA into an inert atmosphere confinement enclosure with glove ports with an Argon purge capability, leveraging the work that currently is being done with the fresh fuels glove box in the Analytical Laboratory.
3. Investigate the feasibility of and verify through mockup testing various approaches for remote specimen handling, beginning with specimen arrival in rabbit (mockup) and including various LFA carrier configurations and specimen loading and unloading of the sample carrier.
4. Evaluate instrument performance in a representative environment (i.e., temperature, humidity, vibration, and EMI) and investigate through mockup testing, the feasibility and adequacy of proposed measures/approaches for providing environmental mitigation or control.
5. Investigate the feasibility of and verify through mockup testing various remote operation and maintenance approaches or methods for the LFA components as deployed. This includes, but is not limited to, normal remote instrument operations such as power on, check out/diagnostics, methods for remote sample loading (including an automatic sample changer, if applicable), balance and hoisting device operation instrument and/or support equipment adjustments, instrument and process monitoring, and power off. Maintenance approaches and methods investigated will include, but are not limited to, remote sample loading, hoisting device, filling the liquid nitrogen dewar before and during instrument operation, and serviceability or modularization improvements implemented to facilitate replacement of components having shortened life spans due to high-radiation fields.
6. Verify radiation-hardened LFA functionality and acceptable performance for a specimen in a representative radiation field of about 25 R/hour  $\gamma$  on contact.

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7. Verify acceptability of the final selected serviceability or modularization methods implemented to facilitate replacement of components having shortened life spans due to high-radiation fields in conjunction with final clearances and telemanipulator, shield wall, transfer port, and window configurations/positions.

Other optional maturation testing scope:

1. Perform individual component testing of non-radiation hardened components of the main instrument (i.e., that cannot be relocated or adequately shielded) to determine expected life spans of the components as a function of radiation field intensity ( $\alpha$ ,  $\beta$ , and/or  $\gamma$ ). This information will be useful in setting limits, if appropriate, for the specimens to be examined/processed and for establishing associated maintenance and spares strategies that are economically supportable.

### 3.2.3 Technology Maturation Testing Description

The maturation activities and testing provide an opportunity to verify the LFA, as modified for high-radiation service, within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility. First, this test will verify that instrument modifications have not degraded functionality or performance to unacceptable levels.

Second, the mockup enclosure (i.e., with simulated shielding panels, windows, transfer ports, and actual telemanipulators) will be used to investigate various instrument layouts, cell configurations, and associated interfaces with the inert confinement enclosure. The mockup easily will be reconfigured to support quick setup of alternative configurations for team review and evaluation. The use of actual telemanipulators will provide realism during process development testing and demonstration.

Third, process development testing will be performed to identify effective methods, equipment, and special tooling to facilitate handling, loading, and unloading of specimens.

Fourth, test the modified LFA in the Irradiated Materials Characterization Laboratory or other like facility, within a full-scale, shielded cell enclosure similar to that which will be used in the APEX facility with a known non-irradiated sample. It will be tested to verify the unit is operating according to the design requirements with a sealed radiation source present in the cell.

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Fifth, the LFA will be moved and integrated into the APEX facility for final installation and testing with a representative radiation sample (i.e., minimum of 25 R/hour  $\gamma$ ). The test also verifies integration of the final confinement enclosure and associated instrument/enclosure interfaces; final process equipment, tools, and aids; remote sample handling procedures; and remote instrument operation and maintenance activities under a radiological environment. An investigation of maintenance, servicing, and replacement of modularized components having shortened life spans also will be investigated.

### 3.2.4 Maturation Testing Conditions

- Specimens: typical LFA sample, nonradioactive (e.g., graphite samples) with known thermal properties.
- Temperature: stable temperature  $\pm 2.5^{\circ}\text{C}$  for 1 hour prior to starting exam
- Relative humidity: TBD%
- Acoustics: TBD
- Vibration: TBD
- Available utilities:
  - Power: 230 V; 20 and 30 amp; single-phase; standard 120 V
  - Process gases: ultra high purity argon
  - Instrument air, compressed: TBD.

### 3.2.5 Maturation Testing Configuration

Work with NETZSCH to produce an LFA that can be integrated into a glove box and hot cell.

There will be a mockup instrument enclosure (full-scale, reconfigurable) for cold testing with actual telemanipulators (make/model TBD) but simulated (a) shielding walls, (b) shielded windows, and (c) shielded transfer port(s).

The LFA will be tested in its radiation-hardened configuration and instrumented for obtaining environmental conditions such that

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measurements can be performed to verify the LFA is working properly in a glove box and hot cell configuration. This may include temperature, humidity, acoustic noise, vibration, and EMI measurements.

There will be a shielded instrument enclosure for hot testing with actual telemanipulators (make/model TBD) and real (a) biological shielding blocks/panels, (b) shielded windows, and (c) transfer port(s). Shielding shall be adequate to provide personnel protection (i.e., less than 0.5 mrem/hour at 30 cm) for all radiological sources used during testing under this plan.

The LFA will be tested in its radiation-hardened configuration and instrumented for radiation field, temperature, acoustic noise, vibration and EMI measurements. Radiological monitoring instruments and controls also shall be in place for supporting personnel protection.

### 3.2.6 Required Data

- Environmental readings as appropriate (e.g., temperature/humidity plots versus time)
- LFA readings from a known material to verify the unit is working as designed
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup)
- LFA readings from a known radioactive sample to verify the unit is working as designed in a radiation field
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup).

### 3.2.7 Maturation Testing Location

Maturation activities and testing will be accomplished in the Irradiated Materials Characterization Laboratory or a comparable laboratory setting capable of achieving the conditions listed in Section 3.2.4 for the LFA.

Prior to activities that require the LFA to test a highly irradiated sample in the furnace, the LFA will be relocated to the APEX facility so the remaining testing can be performed in the final configurations.

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### 3.2.8 Data Requirements

Data quality (e.g., accuracy and precision) will be sufficient for the intended application and associated pass/fail decisions to be made. Specific data quality requirements are TBD and will be documented in detailed test plans, laboratory instructions, or other work control documents that are used to perform the maturation and testing activities.

Documentation of collected data and generated evaluations, assessments, and other results shall be retained as project records. Handling of documents shall be in accordance with PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

### 3.2.9 Maturation Testing Evaluation Criteria

The following criteria apply for determining whether the maturation and testing activities can be considered successful in transitioning the LFA from TRL-6 to TRL-7:

- Radiation-hardening measures for the LFA will be evaluated through proper instrument functioning and confirmed through testing on known materials. Determination of a maximum advisable radiation field and component longevity in representative radiation fields will be done either as optional scope (if sufficient time and resources are available) or after turnover to operations.
- Acceptance of final instrument layout and shielded cell configuration (including the inert confinement enclosure) will be determined based on inputs from instrument technicians, facility operations, and maintenance personnel.
- Mitigation measures for ambient environmental conditions (i.e., other than radiation levels and including temperature, humidity, acoustic noise, vibration, and EMI levels) shall be evaluated against manufacturer's recommendations for proper instrument operation and confirmed through testing on known irradiated material.

### 3.2.10 Maturation Testing Deliverables

A maturation testing report will be issued to document the results of mockup testing. The report will include any unresolved issues or

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recommended changes that need to be addressed in future TRL maturation activities.

### 3.2.11 Resource and Duration Estimates

#### Lead-times for instrument and estimate of cost:

- 12 to 18 months for NETZSCH to develop and deliver modifications to be integrated into the hot cell
- An LFA was purchased for integration into the glove box for about \$500K; it is assumed that it will be about 30% more to purchase the instrument for a hot cell.

#### LFA modifications (for radiation hardening and remote operation):

- Designers (electrical/mechanical)
- Manufacturer's representative (support subcontract)
- Remote engineering
- LFA researcher/technician
- Craft support (e.g., machinists, laborers, pipefitters, and electronics technicians).

#### Mockup

- Designers (electrical/mechanical)
- Remote engineering
- LFA researcher/technician
- Radiological engineering and radiological control support
- Craft support (e.g., machinists, carpenters, laborers, pipefitters, and electricians).

### 3.3 Plan for Technology Readiness Level-7 to Technology Readiness Level-8 Transition

In order to achieve TRL-8, the operational readiness of the integrated system must be qualified through test and demonstration and the facility must issue a declaration of readiness (Table 2). No new tests or demonstrations will be



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performed to transition from TRL-7 to TRL-8. Rather, the same tests and demonstrations used to transition from TRL-6 to TRL-7 will be repeated as required as part of the facility operational readiness review. The tests and demonstrations need not be as detailed or extensive. The primary purpose is to demonstrate acceptable operability in the final operating environment (i.e., in a hot cell in the APEX facility).

Table 2. Time estimates for the project.

Description	Engineering	Draft	Mechanical	Electrical	Crafts	Procurement	System Engineer	Safety	Quality Assurance	Review	Principal Investigator	Operator	Total
Instrument selected	0												0
obtain instrument info	14												14
Develop conceptual design of instrument	120	120					4	4	4	8			260
write instrument specification	40					4	4	4	4	8			64
bid and award instrument	4					40	4	4	4				56
procure instrument	0					80							80
develop installation design	80	80											160
procure remotization equip	40					10	4	4	4	4			66
assemble mockup	40		80	80	80		8	4					292
install instrument	40		80	80	80		8	4					292
cold remote handling demonstration	10						10	10			40	40	110
disassemble cold test	10		40	40	40		10						140
reassemble in hot cell	40	40					20				10	60	170
hot demonstration	10						10	10			10	40	80
disassemble hot test	10		20	20	20		10	10			10	60	160
install in APEX	80	80	40	40	40		10	10		8	10		318
demonstrate in APEX	10						10	10			10	40	80
	548	320	260	260	260	134	112	74	16	28	90	240	2342

#### 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.

#### 5. REFERENCES

NETZSCH, "Laser Flash Analysis", [http://www.netzsch-thermal-analysis.com/uploads/tx\\_nxnetzschmedia/files/LFA\\_427\\_E\\_0213.pdf](http://www.netzsch-thermal-analysis.com/uploads/tx_nxnetzschmedia/files/LFA_427_E_0213.pdf).



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PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

**6. APPENDIXES**

None.

## **Technology Maturation Plan**

# **Advanced Post- Irradiation Examination Capabilities Project Nanoindenter**



The INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance.

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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available nanoindenter for successful routine application in advanced post-irradiation examination (PIE) of nuclear fuels and materials within the planned Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

This plan applies to a nanoindenter system and addresses the technology maturation activities to transition the nanoindenter from technology readiness level (TRL)-6/7 (average) to a TRL-8,<sup>[1]</sup> including associated test descriptions, conditions, and configurations as identified in Section 3. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 by the processing of the first production specimens).

This plan applies to Advanced PIE Capabilities Project personnel and other MFC or INL personnel identified to support the nanoindenter technology maturation tests and activities. While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test procedures, work orders, or laboratory instructions that will be used to direct the actual work.

The end user organization for the matured and deployed nanoindenter is Nuclear Science and Technology, with support from MFC Operations.

### 1.3 Instrument Description

A nanoindenter (see Figure 1) is the main component for indentation hardness tests. Since the mid-1970s, nanoindentation has become the primary method for measuring and testing mechanical properties at nano-scale. Nanoindentation (also called depth sensing indentation or instrumented indentation) gained popularity with development of machines that could record small load and displacement with high accuracy and precision.<sup>[2,3]</sup> The load displacement data can be used to determine modulus of elasticity, hardness, yield strength, fracture toughness, scratch hardness, and wear properties.<sup>[4]</sup>

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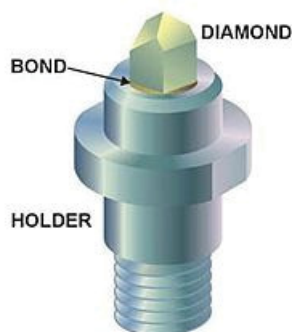


Figure 1. Nanoindenter.

#### 1.4 Advanced Post-Irradiation Examination Application

For advanced PIE, the nanoindenter would support the following needs:

- TBD

#### 1.5 Current Technology Maturation

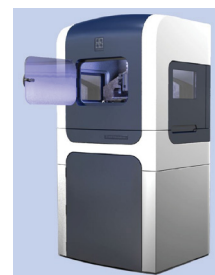
Nanoindenters currently are made by several manufacturers, including Hysitron, Agilent Technologies, Oxford Instruments, and CSM Instruments (Figure 2). Applications of these instruments include semiconductor industry (e.g., thin films and wafers), hard coatings, composite materials, fibers, polymers, metals, and ceramics.



Agilent Technologies  
Nanoindenter



CSM Instruments  
Nanoindenter



Hysitron TI 950  
TriboIndenter

Figure 2. Various nanoindentation testers.

A Hysitron TriboIndenter (TI-950) currently is in use at the Center for Advanced Energy Studies at INL. The Center for Advanced Energy Studies' nanoindenter is expected to start accepting radioactive specimens, including irradiated steels (no fuels yet) in April 2013. The radiation levels will be limited to the Center for Advanced Energy Studies limits.

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The current readiness of a nanoindenter used for examining irradiated nuclear fuels and materials is a technology readiness level (TRL) of 6 due to the need to demonstrate remote operation in a radiation field (see Table 1). (The TRL was assessed to be an average of 6.7, with a minimum of 6.)

Table 1. Nanoindenter technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	*Nano-Indenter	7	7	7	7	6	6	7	6	6.7

Remote specimen handling challenges will include appropriate sample preparation prior to insertion into the nanoindenter. The required sample surface will be prepared with a Vibromet 2® or an electrolytic polisher to produce an extremely smooth surface finish. This surface needs to be protected and cannot be scratched prior to measurements. Once the sample is placed in the system, the alignment is all conducted remotely via computer control.

The nanoindenter system will consist of the instrument, load tester tips, stable platform (e.g., a granite base), controls (computer and/or keypad), cables, and a low magnification microscope to focus the specimen and conduct alignment. If an optional temperature-controlled stage is used, water cooling may be necessary.

The existing Hysitron TriboIndenter system can be and is operated remotely, in the sense that the instrumentation and movement is controlled by either a wired remote keypad or by a computer. However, it is anticipated that remote handling is still going to be a challenge due to some of the fine alignment adjustments necessary for sample analysis. For example, with the existing system, indenter heads must be changed to establish the desired indentation size on the sample (i.e., low load versus high load). Special tools and/or jigs may have to be designed to allow exchange of the load tips with either a telemanipulator or with gloves through a glove port.

It may be that activities such as change out of load tips can be accomplished before the sample is emplaced or after it is removed; therefore, remote operations for such change outs is not necessary. They could, perhaps, be performed through a glove port or by directly accessing the instrumentation if it is either not contaminated or can be decontaminated. Or, on rare occasion, they could be performed by personnel wearing appropriate personal protective equipment.

The current nanoindenter system and set up is not optimized for high dose specimens. The main challenge with the current system is that the electronics are very close to the specimens.

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The University of California Berkeley Nuclear Materials Laboratory has implemented a nanoindentation tester within an environmental enclosure that allows for examining specimens needing an inert atmosphere. CSM Instruments also offers an optional vacuum or environmental control enclosure for its nanoindentation tester.

## 1.6 Program Links

All of the following programs will be stakeholders in the successful technology maturation of the nanoindenter for the APEX facility:

Light Water Reactor Sustainability – this program seeks to create economic and environmental benefits for existing nuclear power plants through improvements in reliability, availability, productivity, component life, safety, and security. Development of the scientific basis to allow evolution of fuels and understanding and predicting long-term degradation behavior and operational limits of materials relies on detailed PIE of reactor fuels and materials at the nano-scale.

Fuel Cycle Research and Development – this program has the responsibility of developing advanced fuel technologies using a goal-oriented, science-based approach. This approach requires a detailed mechanical properties understanding of a broad spectrum of nuclear fuels and cladding materials at the nano-scale.

Advanced Reactor Concepts – this program seeks to leverage the latest materials and fuels technologies to develop advanced reactor designs having improved efficiency and improved economics. The behavior of these materials and fuels in various reactor environments must be fully understood at the micro/nano-scale to support effective simulations and designs.

National Defense and Security – the National Nuclear Security Administration Naval Reactors program and other national security missions (e.g., nuclear non-proliferation) can benefit from nano-scale examination and characterization of nuclear materials and fuels in achieving their program objectives.

Nuclear Energy University Programs – this program plays a key role in helping the U.S. Department of Energy accomplish its mission of leading the nation's investment in development and exploration of advanced nuclear science and technology. It promotes nuclear energy as a resource capable of meeting the nation's energy, environmental, and national security needs by resolving technical, cost, safety, security, and proliferation resistance through research, development, and demonstration. Advanced PIE capabilities at the APEX facility will provide student learning opportunities and help develop the nation's next generation of nuclear scientists and engineers. It also will support the Advanced Test Reactor's National Scientific User Facility and potentially a large range of



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cooperative research and development agreements and work for others within the commercial nuclear industry.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

## 3. MATURATION TEST PLANS

### 3.1 General Information

Further technology maturation is required and will be achieved through the following activities:

- Moving the existing nanoindenter, currently located at the Analytical Laboratory, to the Irradiated Materials Characterization Laboratory
- Performing tests to bring the TRL to a maturity level of 7
- Procuring a new nanoindenter that will, at a minimum, meet the same capabilities and performance specifications as the existing instrument
- Installing the new nanoindenter in the APEX facility
- Performing system checkouts to ensure the new nanoindenter performs as specified
- Performing a system operability test to demonstrate that the nanoindenter supports the APEX facility TRL-8.

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

#### 3.1.1 Assumptions

- The instrument manufacturer knows what components of their system are sensitive to the expected kinds and levels of radiation.

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- It is possible to keep radiation-sensitive components out of high-radiation areas or provide adequate shielding without causing unacceptable degradation to instrument performance.

### 3.1.2 Prerequisites

- A special order nanoindenter, modified for compatibility with high-radiation environments, has been procured and is available to support technology maturation testing and development activities.
- INL engineering has worked with the instrument manufacturer to identify radiation-sensitive and/or vulnerable components that can be removed and separated from the main instrument or that must be protected through installation of add-on shielding to improve radiation hardening of the nanoindenter. Design and performance of such modifications may either be done by the manufacturer or by INL personnel after receipt of the instrument. Procurement requirements must take this into consideration. To the extent possible, all modifications should be done by the vendor before final acceptance, because they are likely to degrade performance unless done by an expert with in-depth knowledge and experience with design of the original system.

### 3.1.3 Identified Hazards and Safety Concerns

The primary hazard associated with nanoindenter maturation activities involves working with highly radiological sources, which pose a risk for unanticipated worker exposures. Other hazards include radiological contamination, electric shocks, hoisting and rigging, rotating equipment, compressed gases, and confined spaces with the potential for oxygen-deficient atmospheres.

### 3.1.4 Work Controls, Permits, and Caution/Danger Tags

All work performed pursuant to this plan will be conducted using company standard work procedures for work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## 3.2 Plan for Technology Readiness Level-6 to Technology Readiness Level-7 Transition

In order to achieve TRL-7, the actual instrument must have been successfully operated in its final configuration and in a relevant environment (including

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radiation and remote operation), with limited degradation due to radiation exposure.

### **3.2.1 Radiation Hardening and Performance in High-Radiation Fields**

The existing nanoindenter is not completely designed for radiation hardness and has been used to examine radioactive samples, including irradiated metals, but not irradiated fuels (which are much more radioactive). The components that would be vulnerable are the electronics that measure the indentation response (i.e., potential interference with measurements and potential long-term damage), the low magnification microscope lens (i.e., radiation damage causes darkening and opacity), and the indentation tips (i.e., alteration of mechanical properties via radiation damage).

A two-stage approach will be used to progress the nanoindenter from TRL-6 to TRL-7. The first stage uses a mockup enclosure (dimensionally accurate, but no shielding blocks/panels or shielding windows) and addresses verification of instrument functionality after component removal/separation and location-specific shielding to improve radiation hardening of the procured instrument. Approaches for confinement box integration/interfaces, remote sample handling, and remote operation will be investigated during this stage. The procurement specification for the existing nanoindenter addresses some of this and can provide useful insights. Vibration protection and electromagnetic interference (EMI) protection are not issues for the nanoindenter.

The second stage adds radiological shielding and finalizes the confinement box interface, remote sample handling, remote operation and maintenance, and allows instrument functionality to be tested in a relevant environment.

Optional scope also is identified in the event that resources (e.g., time and funding) are available after completion of other maturation tasks and testing.

#### **3.2.1.1 Maturation and Testing Objectives**

##### Stage I:

1. Verify nanoindenter functionality and acceptable performance after (a) removal and physical separation of any radiation-sensitive components from the main instrument; and (b) installation of shielding in strategic locations to protect remaining

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radiation-sensitive components not addressed in (a). Installation of such shielding may be non-trivial and, if done incorrectly, may degrade instrument performance in subtle ways. Nanoindenter functionality and acceptable performance will have to be verified with the shielding in place.

2. Verify through mockup testing the conceptual layout (e.g., fit, clearances, and functionality) of the instrument within the inert confinement enclosure and shielded cell, including window and telemanipulator locations.
3. Investigate the feasibility of and verify through mockup testing various approaches for remote specimen handling (beginning with specimen arrival in rabbit mockup and including various carrier configurations), specimen loading onto the sample stage, specimen removal from the nanoindenter, subsequent handling, packaging, and removal from the shielded enclosure, using the rabbit, or other pass-through mechanisms.
4. Evaluate instrument performance in relevant temperature, humidity, air flow, vibration (i.e., should be  $\leq$  ISO VC-C), acoustic noise (i.e., should be  $\leq$  75dB), and EMI environments. Investigate through mockup testing the feasibility and adequacy of proposed measures/approaches for providing temperature control, air flow, and vibration/noise and EMI protection. Extremely high or rapidly changing air flow could be an issue. Otherwise, none of these is likely to be an issue for a nanoindenter. Many metal and sodium-bonded fuel samples are air-sensitive and need to be handled in inert atmospheres. An inert atmosphere will be required; therefore, instrument procurement specifications must indicate this and the instrument will need to be tested in such an atmosphere.
5. Investigate the feasibility of and verify through mockup testing, various remote operation and maintenance approaches or methods for the nanoindenter as deployed. This includes, but is not

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limited to, normal instrument operations such as power on, check out/diagnostics, , instrument and/or support equipment adjustments, instrument monitoring, change-out of the indentation tip, and power off. Maintenance approaches and methods investigated will include, but are not limited to, improved decontamination methods and improved serviceability or modularization measures taken for components having shortened life spans due to high radiation fields.

Stage II:

1. Verify radiation-hardened nanoindenter functionality and acceptable performance for metallurgical-mounted specimens in a relevant radiation field (e.g., at or above 25 R/hr  $\gamma$  and 130 R/hr  $\beta$  on contact).
2. Verify remote operability, sample handling, and updated configuration (e.g., layout, clearances, and interfaces) for instrument as mocked up including temporary shielding, telemanipulators, inert confinement enclosure (e.g., prototype), and associated tools, aids, and procedures.
3. Verify final approaches or methods selected through Stage I investigations for temperature control, air flow, vibration protection, EMI protection, confinement box integration, tools/aids, remote sample handling, and remote operation and maintenance in conjunction with radiation fields, final clearances, and telemanipulator, shield wall, and window positions.
4. Verify acceptability of the selected decontamination method, serviceability and modularization measures for anticipated, shortened-life components under actual radiological conditions.

Other optional maturation scope:

1. Perform individual component testing of non-radiation hardened components of the main instrument (i.e., that cannot be relocated or adequately shielded) to determine expected life

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spans of the components as a function of radiation field intensity ( $\alpha$ ,  $\beta$ , and/or  $\gamma$ ). This information will be useful in setting limits, if appropriate, for the specimens to be examined/processed and for establishing associated maintenance and spares strategies that are economically supportable.

### 3.2.1.2 Maturation and Testing Description

#### Stage I:

The Stage I maturation activities and testing provide an opportunity to verify the nanoindenter, as modified for high-radiation service, within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility.

- First, this test will verify that instrument modifications have not degraded functionality or performance to unacceptable levels.
- Second, the mockup enclosure (i.e., simulated shielding panels, windows, and transfer ports) will be easily reconfigurable to allow various hot cell layouts, instrument/component placements, and inert confinement enclosure configurations to be investigated and evaluated. Actual telemanipulators will be employed to provide realism during process development testing. As with the existing nanoindenter system, it is assumed that a new nanoindenter can be easily operated remotely, in the sense that the instrumentation and movement is controlled by either a wired remote keypad or by a computer. Control equipment can and will be placed outside of the shielded cell mockup, and only necessary instruments will be placed inside. It will be necessary to demonstrate that change outs of small components (such as the indentation tips) and fine adjustments can be made either (1) by telemanipulator, perhaps with special tools and/or jigs; (2) through a glove port after the sample has been removed, again with special tools and/or jigs; or (3) by directly accessing instrument components, based on an assumption that there is no residual contamination after the sample has been removed.

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and the instruments have been decontaminated (if necessary). The design will have to take accessibility for the small component change outs. Also, it will be necessary to specify and test any extra-long cables as part of procurement to ensure that timing signals still work in spite of propagation delays in the cables.

- Third, process development testing will be performed to identify effective methods, equipment, and tools for handling, loading, and unloading specimens.

#### Stage II:

The Stage II maturation activities and testing provide an opportunity to verify the nanoindenter (as modified for high-radiation service) within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility and within a relevant radiation environment (e.g., at or above 25 R/hour  $\gamma$  and 130 R/hour  $\beta$  on contact). Actual shielding blocks/panels, windows, and transfer ports, albeit temporary, provide personnel protection and added realism for this test.

The mockup enclosure will be configured and instrumented to verify the final approaches, methods, and layout (positioning) chosen based on Stage I results. This includes any final mitigation measures implemented for temperature control, air flow, and vibration and EMI protection. The test also verifies integration of the final inert confinement enclosure and associated instrument/enclosure interfaces; final process equipment, tools, and aids; remote sample handling procedures; and remote instrument operation and maintenance activities under a radiological environment. Maintenance activities to be verified include decontamination and servicing/replacement of modularized components having shortened life spans under real, relevant radiological conditions.

### **3.2.1.3 Test Conditions**

#### Stage I:

- Non-radiological specimens (e.g., steel samples).



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- Air Environment – a mockup (not necessarily a working prototype) of the inert confinement enclosure will be used to evaluate the conceptual design for this component.
- Controlled temperature and humidity – the tests will be conducted under standard laboratory environmental operating conditions. Temperature swings can affect the performance of the nanoindenter system. (details TBD by SME)
- EMI source (variable frequency, variable intensity) – the nanoindenter is not believed to be susceptible to EMI; therefore, it is not expected to require EMI shielding. (SME to verify)
- Vibration source (variable frequency, variable amplitude) and acoustic noise – vibration stabilization is critical for this instrument to operate properly. Even ventilation blowing directly onto the instruments can cause sufficient vibration to affect the instrument. However, the existing instrument includes (as will, presumably, any newly procured instrument) a granite stabilizer and a dampening system. This should provide adequate vibration protection. Acoustic noise protection components will be present if deemed appropriate and applicable.

Stage II:

- Non-radiological specimens – the current limitation for the radiation level is set by the Radiological Work Permit at 1 Rem/hour ( $\gamma$  and  $\beta$ ) on contact. Test materials (known, nonradioactive) will be evaluated in the presence of sealed radioactive sources to produce the relevant radiation environment.
- Inert Environment – A mockup (preferably a working prototype) of the inert confinement enclosure will be used to evaluate the conceptual design for that component. The instrument components do not necessarily need to be in an inert environment; however, it may be advantageous to



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have the sample in an inert environment to prevent oxidation of the specimens (hence removing the influence that the oxidation layer may have on the nanoindentation results).

- Controlled temperature/humidity – the tests will be conducted under standard laboratory environmental operating conditions. Temperature swings can affect the performance of the nanoindenter system. (details TBD by SME)
- EMI source (variable frequency, variable intensity) – the nanoindenter is not believed to be susceptible to EMI; therefore, it is not expected to require EMI shielding. (SME to verify)
- Vibration source (variable frequency, variable amplitude) and acoustic noise – vibration stabilization is critical for this instrument to operate properly. Even ventilation blowing directly onto the instruments can cause sufficient vibration to affect the instrument. However, the existing instrument includes (as will, presumably, any newly procured instrument) a granite stabilizer and a dampening system. This should provide adequate vibration protection. Acoustic noise protection components will be present if deemed appropriate and applicable.

#### 3.2.1.4 Test Configuration

##### Stage I:

The Stage I test configuration will consist of a mockup instrument enclosure (full scale, reconfigurable) with actual telemanipulators (make/model TBD) and simulated (a) shielding walls, (b) shielded windows, and (c) shielded transfer port(s).

The nanoindenter will be deployed in a radiation-hardened configuration. Vendor data, analysis, and/or testing will determine which components are likely to be sensitive to radiation. Control components of the instrument will sit outside the shield enclosure. Potentially sensitive components that cannot be removed will have to be

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shielded/protected. Localized shielding is an option for the electronics, the microscope lens (when not in use), and the tip (when not in use), although practicality of the operation of the instrument may prevent shielding of the lens and tip. Also, according the subject matter expert, the shielding will require a more sophisticated and engineered approach than just throwing a piece of lead in the instrument.

Instrumentation for obtaining temperature, humidity, air flow, vibration, and noise measurements is required in order to demonstrate acceptable conditions for testing.

#### Stage II:

Stage II will consist of a shielded instrument enclosure (full scale, temporary) with actual telemanipulators (make/model TBD) and real (a) shielding blocks/panels, (b) shielded windows, and (c) transfer port(s). Shielding shall be adequate to provide personnel protection to less than or equal to 0.25 mrem/hour ( $\gamma$  and  $\beta$ ) for all radiological sources used during testing under this maturation plan.

Again, the nanoindenter will be deployed in a radiation-hardened configuration. No special instruments are required. Standards will be run in the presence of sealed radioactive sources to verify instrument functionality by determining whether measurements meet specifications for those standards.

Instrumentation for obtaining temperature, humidity, air flow, vibration, and noise measurements is required in order to demonstrate acceptable conditions for testing.

If an optional temperature-controlled stage is used, water cooling may be necessary.

Radiological control monitoring instruments shall be in place for ensuring personnel protection.

The indentation tip will become contaminated because it will need to directly indent into the sample. The tips will need to be replaced periodically, because over time they degrade from normal use. Other potential areas of contamination could include the platen (sample is mounted

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on this) and the base that the platen sits on if a sample were to fracture and a piece was removed during indentation.

### 3.2.1.5 Required Data

#### Stage I:

- Instrument alignment, repeatability, load/unload duration, quality of output, number of drops, and damage; the performance specification for the existing nanoindenter should provide insights to useful or necessary performance tests.
- Measured parameters for comparison with known standards.
- Environmental measurements (i.e., temperature, humidity, air flow, vibration, and noise).
- Written evaluation of the configuration (e.g., layout and clearances) by a subject matter expert and an operator/maintainer (qualitative; keyed to configuration managed mockup).

#### Stage II:

- Radiation measurements (i.e.,  $\alpha$ ,  $\beta$ , and  $\gamma$ ) of sealed sources on contact and at 30 cm.
- Radiation measurements (i.e.,  $\alpha$ ,  $\beta$ , and  $\gamma$ ) on contact at nanoindenter main instrument components that are thought to be radiation sensitive or vulnerable to gamma radiation and that could not be relocated external to the shielded cell walls, with any supplemental shielding in place.
- Typical calibrations will be monitored, including the tip to optic calibration, the air calibration, and the hardness/modulus response in a known material (keyed to the ID and/or description of the sealed radioactive source).
- Environmental measurements (i.e., temperature, humidity, air flow, vibration, and noise).

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- Written evaluation of the configuration by a subject matter expert and an operator/maintainer (qualitative; keyed to configuration managed mockup).

#### **3.2.1.6 Test Location**

Stage I and II maturation activities and testing will be accomplished in the Irradiated Materials Characterization Laboratory or comparable laboratory setting approved for radiological work. If the instrument is to be tested elsewhere and then moved to the APEX facility, then it will be essential to avoid any test samples that could cause radiological contamination to the instrument.

#### **3.2.1.7 Data Requirements**

Data quality (e.g., accuracy and precision) will be sufficient for the intended application and associated pass/fail decisions to be made. Tests will have to be repeated to check for instrument degradation. The number of tests and the wait time between tests are yet to be determined.

Documentation of collected data and generated evaluations, assessments, and other results shall be retained as project records. Handling of documents shall be in accordance with PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

#### **3.2.1.8 Maturation and Testing Evaluation Criteria**

The following criteria apply for determining whether the maturation and testing activities can be considered successful in transitioning the nanoindenter from TRL-6 to TRL-7:

- Radiation-hardening measures for the nanoindenter will be evaluated through proper instrument functioning and confirmed through characterization of a metallurgical-mounted specimen in the presence of a sealed radioactive source that creates relevant radiation levels of at least 25 R/hour ( $\gamma$  and  $\beta$ ) on contact. Determination of component longevity in relevant radiation fields (and the acceptability

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thereof) will be done after turnover to operations due to the length of time needed for such an assessment.

- Acceptance of final layout and instrument configuration.
- Mitigation measures for ambient environmental conditions (i.e., other than radiation levels and including temperature, air flow, vibration, and noise levels) shall be evaluated against the manufacturer's recommendations for proper instrument operation and confirmed through characterization of a non-radiological specimen.

#### **3.2.1.9 Test Deliverables**

A test report will be issued to document the results of Stage I and II mockup testing. The report will include any unresolved issues or recommended changes that need to be addressed in Future TRL maturation activities.

#### **3.2.2 Resource and Duration Estimates**

Table 2 summarizes the estimated labor hours.

### **3.3 Plan for Technology Readiness Level-7 to Technology Readiness Level-8 Transition**

In order to achieve TRL-8, the operational readiness of the integrated system must be qualified through test and demonstration and the facility must issue a declaration of readiness. No new tests or demonstrations will be performed to transition from TRL-7 to TRL-8. Rather, the same tests and demonstrations used to transition from TRL-6 to TRL-7 will be repeated as part of the facility operational readiness review. The tests and demonstrations need not be as detailed or extensive. The primary purpose is to demonstrate acceptable operability in the final operating environment (i.e., in a modular shielded cell in the APEX facility).

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Table 2. Nanoindenter technology readiness level.

Resource	Instrument Modifications	Stage I	Stage II	Totals
Engineering	178	260	110	548
Drafting	120	120	80	320
Mechanical Craft	0	200	60	260
Electrical Craft	0	200	60	260
Carpenters	0	120	0	120
Laborers	0	60	0	60
Procurement	124	10	0	134
Systems Engineering	12	60	40	112
Safety	12	22	40	74
Quality Assurance	12	4	0	16
Reviewers	16	4	8	28
Principle Investigator or SME	0	50	40	90
Operators	0	60	140	200
Totals	474	1,170	578	2,222

#### 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.*

#### 5. REFERENCES

*Advanced Post-Irradiation Examination Capability Technology Readiness Assessment*, INL/EXT-12-27849, December 2012.

Ki Won Kang et al. 2004, "Measurement of Melting Temperatures of UO<sub>2</sub>, (U,Gd)O<sub>2</sub> and (U,Er)O<sub>2</sub> Fuels," *Journal of the Korean Nuclear Society*, 36, 1, 104-111.

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## **6. APPENDIXES**

None.

## **Technology Maturation Plan**

# **Advanced Post- Irradiation Examination Capabilities Project Focused Ion Beam**



The INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance.



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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available focused ion beam (FIB) for successful routine application in advanced post-irradiation examination (PIE) of nuclear fuels and materials within the planned Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

This plan applies to a dual-beam (DB) FIB system (i.e., a FIB combined with a scanning electron microscope [SEM]) and includes components such as an electron beam column, ion (typically gallium) emitter column, motorized stage, detectors, image processor, vacuum system and chamber, power and control consoles, support equipment, and associated enclosures. The plan addresses the technology maturation activities necessary to transition the FIB from a technology readiness level (TRL)-6 (minimum) to a TRL-8,<sup>a</sup> including associated test descriptions, conditions, and configurations as identified in Section 3. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 through processing of the first production specimens).

This plan applies to the Advanced PIE Capabilities Project personnel and other MFC or INL personnel identified to support the DB FIB technology maturation tests and activities. While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test procedures, work orders, or laboratory instructions that will be used to direct the actual work.

Some portions of this plan also may apply to technology maturation activities for the SEM (e.g., the vacuum chamber and associated modifications). As such, changes in one plan should be reviewed for applicability and possible inclusion in the other.

The end user organizations for the matured and deployed DB FIB are expected to be MFC Operations and Nuclear Science and Technology.

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<sup>a</sup>Refer to *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, INL/EXT-12-27849, for a description of TRL levels.

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### 1.3 Instrument Description

FIB systems are considered microstructural analysis instruments and have been in commercial operation for approximately 20 years. The majority of this use has been applied in the semiconductor manufacturing industry to patch or correct defects in existing semiconductor devices. For example, in an integrated circuit, the gallium beam could be used to cut unwanted electrical connections or to deposit conductive material to make a needed connection. The high level of surface interaction is exploited in patterned doping of semiconductors and also can be used for maskless implantation.

FIB systems operate in a similar fashion to a SEM except that, rather than a beam of electrons and as the name implies, FIB systems use a finely focused beam of ions (usually gallium) that can be operated at low-beam currents for imaging or high-beam currents for site-specific sputtering or milling.

The FIB to be matured for deployment in the APEX facility will be used exclusively for this latter purpose. It will be a “dual beam” model, having both an ion emitter column for milling out transmission electron microscope (TEM) lamellae (i.e., thin, plate specimens) and atom probe microscopy/tomography (APM/APT) needles/tips, as well as an electron beam column for simultaneous imaging to support milling process control.

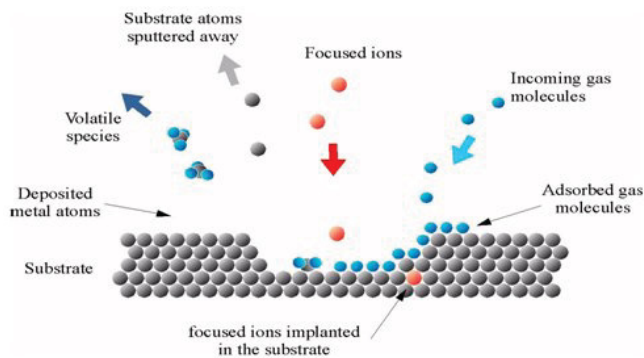
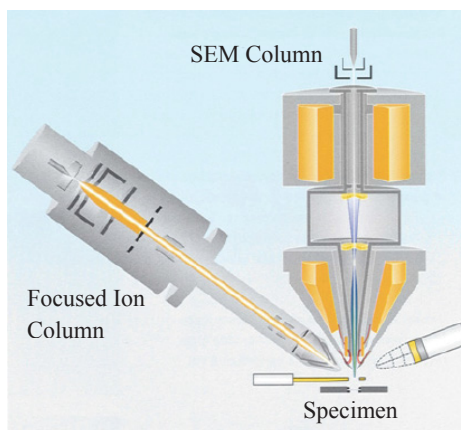


Figure 1. Dual beam configuration. Figure 2. Focused ion beam milling/sputtering.

### 1.4 Current Technology Maturity

The DB FIB has been demonstrated as a sample preparation instrument to allow detailed and effective examination of irradiated fuel under a TEM.<sup>[1]</sup> Current operation in the Electron Microscopy Laboratory (EML) has indicated no impairment of the SEM imaging or FIB milling functionality over 7 years of operation in a radiation environment up to about 25 R/hour  $\gamma$  on contact maximum

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(about 8 R/hour  $\gamma$  being typical) and up to about 130 R/hour  $\beta$  on contact. During this time, both irradiated fuels and materials have been examined and/or processed. However, some degradation has been observed in the effective life of certain electronics components (e.g., energy dispersive spectroscopy) from 8 years of operation in a non-radiation environment to approximately 3 years. A warranty contract is in place for the EML DB FIB, which covers replacement of failed components due to normal and PIE use.<sup>b</sup>

The specimens to be examined and processed in the APEX facility are expected to have much higher activities than those allowed for the unshielded EML FIB. An initial target value for the shielded instruments in the APEX facility is to provide the capability to examine specimens measuring up to 3 Ci of activity at 1 MeV gamma radiation.<sup>c</sup>

EML experience in preparing TEM lamellae from irradiated samples indicates that sputtered atoms (fuel and activated metals) will plate out and accumulate on the interior surface of the vacuum chamber over time. This contamination typically is not picked up on smears, but is removable with decontamination solutions. Periodic decontamination of the EML FIB's vacuum chamber has been performed to maintain optimum equipment performance and to keep radiation doses to instrument technicians as low as reasonably achievable.

A technology readiness assessment for the DB FIB has determined that the instrument rates a TRL-6 (Table 1), based on industrial and nuclear applications to date.

Table 1. Focused ion beam technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	*Focused Ion Beam (FIB)	6	7	7	7	6	6	7	6	6.6

## 1.5 Program Links

The following programs are stakeholders in the successful maturation and deployment of the DB FIB in a shielded enclosure:

<sup>b</sup>Vacuum system components that become suspect contaminated and require rebuild after failure are not covered by the warranty and must be purchased separately.

<sup>c</sup>While it is very desirable for the APEX facility to provide a full suite of shielded instruments capable of examining specimens containing this amount of activity, it should be noted that this target value may not be achievable for all of the PIE instruments to be deployed.

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Light Water Reactor Sustainability – this program seeks to create economic and environmental benefits for existing nuclear power plants through improvements in reliability, availability, productivity, component life, safety, and security. Development of the scientific basis to allow evolution of fuels and understanding and predicting long-term degradation behavior and operational limits of materials relies on detailed PIE of reactor fuels and materials at the nano-scale.

Fuel Cycle Research and Development – this program has the responsibility of developing advanced fuel technologies using a goal-oriented, science-based approach. This approach requires a micro-structural understanding of a broad spectrum of nuclear fuels and cladding materials.

Advanced Reactor Concepts – this program seeks to leverage the latest materials and fuels technologies to develop advanced reactor designs having improved efficiency and improved economics. The behavior of these materials and fuels in various reactor environments must be fully understood at the micro/nano-scale to support effective simulations and designs.

National Defense and Security – the National Nuclear Security Administration Naval Reactors program and other national security missions (e.g., nuclear non-proliferation) can benefit from nano-scale examination of nuclear materials and fuels in achieving their program objectives.

Nuclear Energy University Programs – this program plays a key role in helping the U.S. Department of Energy accomplish its mission of leading the nation's investment in development and exploration of advanced nuclear science and technology. It promotes nuclear energy as a resource capable of meeting the nation's energy, environmental, and national security needs by resolving technical, cost, safety, security, and proliferation resistance through research, development, and demonstration. Advanced PIE capabilities at the APEX facility will provide student learning opportunities and help develop the nation's next generation of nuclear scientists and engineers.

## **2. APPLICABLE DOCUMENTS**

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

“The Dual Beam FIB as a PIE Characterization Tool,” James I. Cole, 2008.

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### 3. MATURATION TEST PLANS

#### 3.1 General Information

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

##### 3.1.1 Assumptions

- This plan uses an FEI Quanta™ 3D DualBeam™ instrument as a reference baseline to identify and define the necessary technology maturation activities. It is assumed that other dual beam FIBs available on the market, having comparable capabilities and accessories, would require substantially similar activities to successfully deploy in an advanced PIE application.
- The DB FIB will be used exclusively for milling TEM lamellae and APM/APT needles.
- It is assumed that the following accessories and add-on analysis components are not present on the FIB and are not needed to support its role in nuclear fuel and material characterization. Other PIE instruments are assumed to be available within the APEX facility to provide these analysis capabilities:
  - Charge neutralization accessory (i.e., low-energy electron flood gun)
  - Energy dispersive spectroscopy – analysis component
  - Wavelength dispersive spectroscopy – analysis component
  - Electron backscatter diffraction – analysis component.

**NOTE:** *Additional technology maturation activities would need to be identified and defined if these analysis components were included on the DB FIB to be deployed in the APEX facility. Such activities are not included in this plan.*

- The procured DB FIB is a special order unit, which has been modified or built to meet INL and manufacturer agreed-upon specifications (TBD) for service in a high-radiation environment.



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Specifically, these modifications may include, but are not limited to, the following:

- Substitution of standard wiring, vacuum seals, and surface finishes/coatings with ones that are compatible with high-radiation fields and, where applicable, a radiologically contaminated environment (including considerations for decontamination solutions that are anticipated to be used)
- Extended (non-standard) vacuum chamber door rails for improved access into the vacuum chamber.

**NOTE:** *Additional technology maturation activities will need to be added to address these modifications if they are not available from and performed by the instrument manufacturer. Activities to substitute such items after delivery, with ones that are compatible for use in a radiation environment are not included in this plan.*

- A TEM and/or APM is assumed available for evaluating DB FIB performance relative to milled lamellae and needles, respectively.

### 3.1.2 Prerequisites

- A special order DB FIB, modified for compatibility with high-radiation environments, has been procured and is available to support technology maturation testing and development activities.
- INL and/or MFC engineering personnel have worked with the instrument manufacturer to identify radiation-sensitive and/or vulnerable components that can be removed and physically separated from the main instrument/vacuum chamber or that must be protected through installation of add-on shielding to improve radiation hardening of the DB FIB. Design and performance of such modifications may either be done by the manufacturer prior to shipping or by INL personnel after receipt of the instrument.

### 3.1.3 Identified Hazards and Safety Concerns

The primary hazard associated with FIB maturation activities involves working with highly radioactive sources, which pose a risk for unanticipated worker exposures. Other hazards include, but are not limited to, electric shocks, hoisting and rigging, rotating equipment,

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compressed gases, and confined spaces with the potential for oxygen-deficient atmospheres.

### **3.1.4 Work Controls, Permits, and Caution/Danger Tags**

All work performed pursuant to this plan will be conducted using company standard work procedures for work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## **3.2 Plan for Technology Readiness Level-6 to Technology Readiness Level-7 Transition**

To achieve a TRL rating of 7, the integrated system (including radiation-hardened DB FIB, inert confinement enclosure, shield walls and windows, transfer ports, and telemanipulators) needs to be successfully demonstrated in its final layout and in a relevant environment (including a high-radiation field and remote operation). This transition represents a major step up from a TRL-6, where a representative model of the system was tested in a relevant operational environment (including vibration, electromagnetic interference [EMI], and temperature, but without the high-radiation field).

### **3.2.1 Technology Maturation Testing Approach**

A two-stage maturation approach will be used to progress the DB FIB from TRL-6 to TRL-7. The first stage primarily will use a mockup enclosure that is dimensionally accurate (but no shielding blocks/panels or shielding windows) to verify instrument functionality after radiation hardening<sup>d</sup> of the procured instrument. Approaches for acoustic noise, vibration protection, EMI protection, confinement enclosure integration/interfaces, remote sample handling, and remote operation also will be investigated during this stage.

The second stage adds actual radiological shielding (i.e., walls, windows, and transfer ports) and finalizes the confinement enclosure interface, remote sample handling, and remote operation and allows the instrument functionality to be tested in a relevant environment.

Optional scope for component lifetime estimation and radiation field envelope determination also is identified in the event that resources (time

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<sup>d</sup>Radiation hardening measures may include removal and relocation of radiation-sensitive components and/or the installation of strategically located shielding materials to protect radiation-sensitive components that cannot be relocated.

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and funding) are available after completion of other maturation tasks and testing.

### 3.2.2 Technology Maturation Testing Objectives

#### Stage I:

1. Using the FIB in EML, investigate the feasibility of and verify improved vacuum chamber decontamination methods. The vacuum chamber of this instrument is already contaminated and requires periodic decontamination. New methods (e.g., strippable tape/coating) can be investigated with little or no impact to current operations.
2. Verify DB FIB functionality and acceptable performance after
  - (a) removal and physical separation of any radiation-sensitive components from the main instrument/vacuum chamber; and
  - (b) installation of shielding in strategic locations to protect remaining radiation-sensitive components not addressed in (a).
3. Investigate through mockup testing various approaches for integrating the inert atmosphere confinement enclosure onto the DB FIB and its interface with the DB FIB's vacuum chamber, as well as with the modular shielded enclosure, telemanipulators, and transfer ports.
4. Investigate the feasibility of and verify through mockup testing various approaches for remote specimen handling, beginning with specimen arrival in rabbit (mockup) and including various DB FIB carrier configurations, specimen loading into and unloading from the vacuum chamber, and, in particular, lamellae/needle (FIB-milled) specimen removal from the specimen carrier, subsequent handling, packaging, and removal from the shielded enclosure (as mocked up).
5. Evaluate instrument performance in relevant temperature, humidity, acoustic noise, vibration, and EMI environments and investigate through mockup testing the feasibility and adequacy of proposed measures/approaches for providing temperature and humidity mitigation or control and any noise, vibration, and EMI protection.
6. Investigate the feasibility of and verify through mockup testing various remote operation and maintenance approaches or methods for the DB FIB components as deployed. This includes,

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but is not limited to, normal remote instrument operations (such as power on, check out/diagnostics, establishing a conduction path for the specimen, instrument and/or support equipment adjustments, instrument and process monitoring, and power off). Maintenance approaches and methods investigated will include, but are not limited to, improved vacuum chamber decontamination methods, DB FIB demating from the inert confinement enclosure without compromising the enclosure's integrity or environment, and serviceability or modularization improvements implemented to facilitate replacement of components having shortened life spans due to high-radiation fields.

**NOTE:** *Maintenance activities do not necessarily require remote methods if the irradiated specimen can be removed from the shielded cell prior to their initiation. Maintenance activities on components within the vacuum chamber may require the use of a glove bag or separate glove box.*

Stage II:

1. Verify radiation-hardened DB FIB functionality and acceptable performance for milling lamella/needle specimens from metallurgical-mounted specimens in a relevant radiation field of about 25 R/hour  $\gamma$  on contact.
2. Verify final approaches or methods selected through Stage I investigations for temperature/humidity control, noise reduction, vibration protection, EMI protection, inert confinement enclosure integration, tools/aids, remote sample handling, and remote operation and maintenance in conjunction with radiation fields, final clearances, and telemanipulator, shield wall, transfer port, and window configurations/positions.
3. Verify acceptability of the final selected serviceability or modularization methods implemented to facilitate replacement of components having shortened life spans due to high-radiation fields in conjunction with final clearances and telemanipulator, shield wall, transfer port, and window configurations/positions.

Other optional maturation testing scope:

1. Assess the radiation-hardened DB FIB's functionality and milling performance in producing lamella/needle specimens from metallurgical-mounted specimens in the presence of a sealed

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radiological source containing up to 3 Ci of about 1 MeV gamma ( $\gamma$ ).

2. Perform individual component testing of non-radiation-hardened components of the main instrument (i.e., that cannot be relocated or adequately shielded) to determine expected life spans of the components as a function of radiation field intensity ( $\alpha$ ,  $\beta$ , and/or  $\gamma$ ). This information will be useful in setting limits, if appropriate, for the specimens to be examined/processed and for establishing associated maintenance and spares strategies that are economically supportable.

### 3.2.3 Technology Maturation Testing Description

#### Stage I:

The Stage I maturation activities and testing provide an opportunity to verify the DB FIB, as modified for high-radiation service, within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility. First, this test will verify that instrument modifications have not degraded functionality or performance to unacceptable levels.

Second, the mockup enclosure (i.e., with simulated shielding panels, windows, and transfer ports and with actual telemanipulators) will be used to investigate various instrument layouts, cell configurations, and associated interfaces with the inert confinement enclosure. The mockup will be easily reconfigured to support quick setup of alternative configurations for team review and evaluation. The use of actual telemanipulators will provide realism during process development testing and demonstration.

Third, process development testing will be performed to identify effective methods, equipment, and special tooling to facilitate handling, loading, and unloading of metallurgical-mounted specimens, as well as the handling of lamella and needle specimens after milling.

It is anticipated that several TEM lamellae and APM needles will be milled during Stage I from nonradioactive specimens. This will allow evaluation of operability and maintainability and instrument performance for various instrument/cell configurations under relevant temperature, humidity, noise, vibration, and EMI environments. Mitigation measures for identified, or anticipated, environmental conditions (i.e., those resulting in unacceptable DB FIB performance) also can be proposed, designed, installed, and tested, as applicable, in order to restore satisfactory instrument performance.

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### Stage II:

The Stage II maturation activities and testing provide an opportunity to verify the DB FIB, as modified for high-radiation service, within a full-scale, but temporary, shielded cell enclosure similar to that used in the APEX facility and within a relevant radiation environment (i.e., minimum of 25 R/hour  $\gamma$ ).<sup>e</sup> Actual shielding blocks/panels, windows, and transfer ports, albeit temporary, will be used to provide personnel protection and added realism for this test. During this test, lamella/needle specimens will be milled from nonradioactive surrogates, representing the irradiated material and fuel samples. The relevant radiation environment will be created by a sealed source placed into the DB FIB vacuum chamber and near the specimen to be milled.

The Stage II enclosure will be configured and instrumented to verify and validate the final approaches, methods, and layout (positioning) chosen based on Stage I results. This includes any final mitigation measures implemented to correct temperature/humidity levels, acoustic noise, vibration, and EMI problems. The test also verifies integration of the final confinement enclosure and associated instrument/enclosure interfaces; final process equipment, tools, and aids; remote sample handling procedures; and remote instrument operation and maintenance activities under a radiological environment. Maintenance activities to be verified include vacuum chamber decontamination<sup>f</sup> and servicing/replacement of modularized components having shortened life spans.

### **3.2.4 Maturation Testing Conditions**

#### Stage I:

- Specimens: met-mounted, nonradioactive (e.g., steel samples)
- Temperature: 20°C  $\pm$  3°C
- Relative humidity: less than 80%
- Stray electromagnetic field (after mitigation, if necessary, for known or anticipated APEX facility location conditions):

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<sup>e</sup>Optional maturation scope, if conducted, includes milling lamella/needle specimens in the presence of sealed radioactive sources containing up to 3 Ci at about 1 MeV gamma (e.g., a Cs-137/Ba-137m source).

<sup>f</sup>Simulated only because the vacuum chamber will not be radiologically contaminated at this point.



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- Less than 100 nT asynchronous
- Less than 300 nT synchronous
- Acoustics: less than 60 dBC
- Vibration: approximate limits<sup>g</sup> as shown in Figures 3 through 5 for one-third octave vibration spectrum.

**NOTE:** *A site-specific survey may be necessary if any of the limits in Figures 3 through 5 are exceeded.*

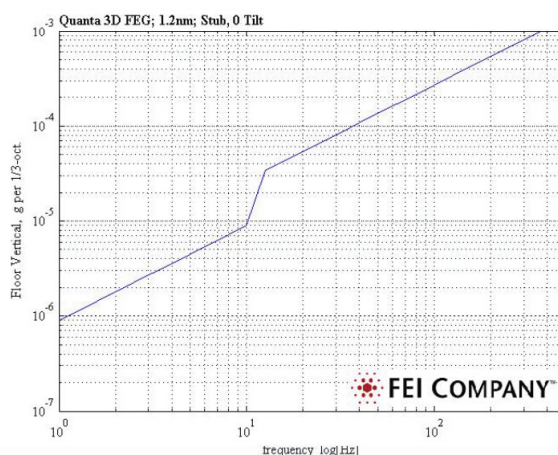


Figure 3. Floor vertical guideline.

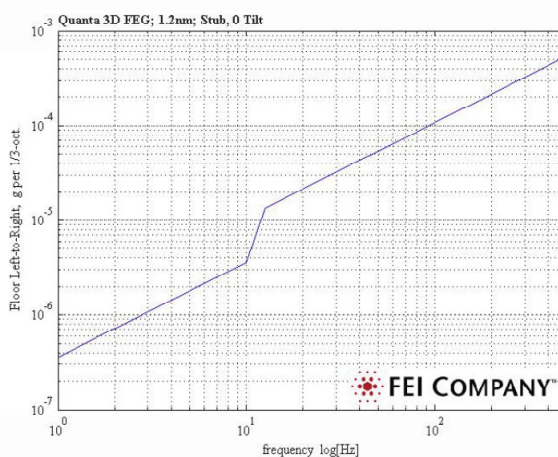


Figure 4. Floor left-to-right vibration guideline.

<sup>g</sup>Based on Quanta's three-dimensional FEG SEM vibration limits.

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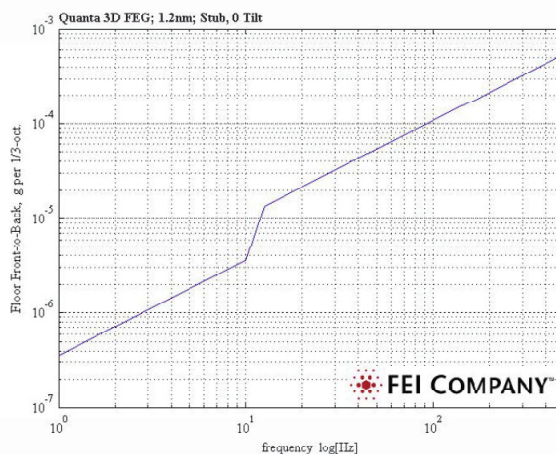


Figure 5. Floor front-to-back vibration guideline.

- Available utilities:
  - Power: voltage 230 V (+6%, -10%); frequency 60 Hz ( $\pm 1\%$ ); power consumption: 3.0 KVA (minimum available for basic microscope)
  - Process gases: nitrogen gas (purge gas and valve control)
  - Instrument air, compressed: 4 to 6 bar (clean, dry, and oil-free).

Stage II:

- Specimens: met-mounted, nonradioactive (e.g., steel samples) but milled in the presence of a sealed radiography source measuring about 25 R/hour  $\gamma$
- Sealed radiological sources: about 25R/hour  $\gamma$  (optional scope sealed sources [example activities; all at 1 MeV  $\gamma$ ]: 0.01 Ci, 0.1 Ci, 0.5 Ci, 1.0 Ci, 1.5 Ci, 2.0 Ci, 2.5 Ci, and 3 Ci).
- Temperature:  $20^{\circ}\text{C} \pm 3^{\circ}\text{C}$
- Relative humidity: less than 80%
- Stray electromagnetic field (after mitigation, if necessary, for known or anticipated APEX facility location conditions):
  - Less than 100 nT asynchronous



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- Less than 300 nT synchronous
- Acoustics: less than 60 dBC
- Vibration: see Stage I vibration guidelines
- Available utilities:
  - Power: voltage 230V (+6%, -10%); frequency 60 Hz ( $\pm 1\%$ ); power consumption: 3.0 KVA (minimum available for basic microscope)
  - Process gases: nitrogen gas (purge gas and valve control)
- Instrument air, compressed: 4 to 6 bar (clean, dry, and oil-free).

### 3.2.5 Maturation Testing Configuration

#### Stage I:

- Existing EML FIB, as currently installed, for investigating improved vacuum chamber decontamination methods.
- Mockup instrument enclosure (full scale, reconfigurable) with actual telemanipulators (make/model TBD) but simulated (a) shielding walls, (b) shielded windows, and (c) shielded transfer port(s).
- DB FIB deployed in radiation-hardened configuration and instrumented for obtaining temperature, humidity, acoustic noise, vibration, and EMI measurements.

#### Stage II:

Shielded instrument enclosure (full scale, temporary) with actual telemanipulators (make/model TBD) and real (a) biological shielding blocks/panels, (b) shielded windows, and (c) transfer port(s). Shielding shall be adequate to provide personnel protection (i.e., less than 0.5 mrem/hour at 30 cm) for all radiological sources used during testing under this plan.

DB FIB deployed in radiation-hardened configuration and instrumented for radiation field, temperature, acoustic noise, vibration, and EMI measurements. Radiological monitoring instruments and controls also shall be in place for supporting personnel protection.

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### 3.2.6 Required Data

#### Stage I:

- Temperature/humidity plots at DB FIB proximal and external to vacuum chamber versus time
- Acoustic noise readings at DB FIB proximal and external to vacuum chamber versus time
- Vibration (e.g., accelerometer) readings at DB FIB proximal and external to vacuum chamber versus time
- EMI readings at DB FIB proximal and external to vacuum chamber versus time
- TEM lamellae/APM needle specimen adequacy (qualitative; annotated with time of milling)
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup).

#### Stage II:

- Temperature/humidity plots at DB FIB proximal and external to vacuum chamber versus time (for the duration of milling test)
- Acoustic noise readings at DB FIB proximal and external to vacuum chamber versus time (for the duration of milling test)
- Vibration (e.g., accelerometer) readings at DB FIB proximal and external to vacuum chamber versus time (for the duration of milling test)
- EMI readings at DB FIB proximal and external to vacuum chamber versus time (for the duration of milling test)
- Radiation measurements (i.e.,  $\gamma$  only) external to the DB FIB vacuum chamber (keyed to a description of the sealed radiation source used)
- Radiation measurements (i.e.,  $\gamma$  only) at DB FIB key instrument components that are thought to be radiation-sensitive or vulnerable to gamma radiation and that could not be relocated

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external to the shielded cell walls, with any supplemental shielding in place (keyed to a description of the sealed radiation source used)

- TEM lamellae/APM needle specimen adequacy (qualitative; annotated with test sequence, date, and time of milling)
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup).

### **3.2.7 Maturation Testing Location**

Stage I maturation activities and testing will be accomplished in the Irradiated Materials Characterization Laboratory or a comparable laboratory setting capable of achieving the conditions listed in Section 3.2.4 for Stage I. Also, FIB vacuum chamber decontamination method improvement activities will take place in EML.

Stage II maturation activities and testing will be accomplished in the Irradiated Materials Characterization Laboratory or a comparable laboratory setting approved for radiological work. The selected location must be capable of achieving the conditions listed in Section 3.2.4 for Stage II.

### **3.2.8 Data Requirements**

Data quality (e.g., accuracy and precision) will be sufficient for the intended application and associated pass/fail decisions to be made. Specific data quality requirements are TBD and will be documented in detailed test plans, laboratory instructions, or other work control documents that are used to perform the maturation and testing activities.

Documentation of collected data and generated evaluations, assessments and other results shall be retained as project records. Handling of documents shall be in accordance with PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

### **3.2.9 Maturation Testing Evaluation Criteria**

The following criteria apply for determining whether the maturation and testing activities can be considered successful in transitioning the DB FIB from TRL-6 to TRL-7:

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- Radiation-hardening measures for the DB FIB will be evaluated through proper instrument functioning and confirmed through the production (milling) of acceptable lamellae/needle specimen from nonradioactive metallurgical-mounted specimen while in a relevant radiation environment (i.e., at a minimum of 25 R/hour  $\gamma$ ). Determination of a maximum advisable radiation field and component longevity in relevant radiation fields will be done either as optional scope (if sufficient time and resources are available) or after turnover to operations.
- Acceptance of final instrument layout and shielded cell configuration (including the inert confinement enclosure) will be determined based on inputs from instrument technicians, facility operations, and maintenance personnel.
- Mitigation measures for ambient environmental conditions (i.e., other than radiation levels and including temperature, humidity, acoustic noise, vibration, and EMI levels) shall be evaluated against manufacturer's recommendations for proper instrument operation and confirmed through the production (milling) of acceptable lamellae/needle specimen (non-radiological).

### 3.2.10 Maturation Testing Deliverables

A maturation testing report will be issued to document the results of Stage I and II mockup testing. The report will include any unresolved issues or recommended changes that need to be addressed in future TRL maturation activities.

### 3.2.11 Resource and Duration Estimates

Table 2 provides labor hour estimates for the resources expected to be required to perform activities leading up to and through Stage I and II maturation and testing.

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Table 2. Labor hour estimates for the resources associated with Stage I and II maturation and testing.

Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Instrument selected (milestone)															0
Investigate Vacuum chamber decon methods													60	20	80
Obtain instrument information	14														14
Develop conceptual design of instrument	120	120						4	4	4		8			260
Write instrument specification	40						4	4	4	4		8			64
Bid and award instrument	4						40	4	4	4					56
Procure instrument	0						80								80
Develop installation design	80	80													160
Procure remotization equipment	40						10	4	4	4		4			66
Assemble mockup	40		80	80	80	40		8	4						332
Install instrument	40		80	80				8	4						212
Cold remote handling demonstration	40	20	20	20				10	20	10			80	80	300
Disassemble cold test	10		40	40	40	20		10							160
Reassemble in hot cell mockup (temporary shielding)	40	40	80	80		80		20			20		10	60	430
Hot demonstration	10							10	10		20		10	40	100

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Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Disassemble hot test	10		20	20				10	10				10	60	140
Totals	488	260	320	320	120	140	134	92	64	26	40	20	170	260	2,454

### 3.3 Plan for Technology Readiness Level-7 to Technology Readiness Level-8 Transition

For TRL-8 to be achieved, the integrated readiness of the system must be demonstrated and qualified within the finished APEX facility, including its final interfaces to the modular shielded enclosure, the inert confinement enclosure, and associated utilities provided by the facility. Specifically, this means that the DB FIB and associated processes have been proven to work, facility representatives have issued a declaration of readiness, and the readiness activity has been successfully completed.

**NOTE:** *This readiness activity (e.g., an operational readiness review by the U.S. Department of Energy) will be performed on a facility basis and not for a single PIE instrument.*

#### 3.3.1 Technology Maturation Testing Approach

The TRL-7 to TRL-8 maturation activities for the DB FIB will be performed in conjunction with the readiness activity for the APEX facility. The facility readiness activity confirms that management has brought the facility to a state of readiness to commence program work. Once management concludes that readiness has been achieved, this state of readiness is independently verified by the contractor and confirmed by the U.S. Department of Energy. Only then will the APEX facility, a Hazard Category 2 nuclear facility, be authorized to “go hot” and perform programmatic PIE work.

The DB FIB portion of the overall facility readiness activity will involve the performance of a system operability and acceptance (SO&A) test for the instrument, modular shielded cell, and associated support systems.

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### 3.3.2 Technology Maturation Testing Objectives

The objective of the TRL-7 to TRL-8 maturation activities is to successfully demonstrate, during the facility readiness activity, the integrated DB FIB instrument for an advanced PIE application, including the adequacy of the following:

- Instrument radiation-hardening measures and interfaces with the modular shielded enclosure and the inert atmosphere confinement enclosure
- Acoustical noise and vibration control measures appropriate for the facility and location
- EMI protection measures appropriate for the facility and location
- Remote sample handling process, including associated methods, tools, and aids
- Remote instrument operation as deployed within the modular shielded enclosure
- Any instrument modifications made to improve reliability, availability, and maintainability for its application of advanced PIE.

### 3.3.3 Technology Maturation Testing Description

The DB FIB SO&A test will demonstrate correct system/process operation and verify that the installed instrument and associated modifications function as designed and in accordance with designated acceptance criteria and design inputs.

The SO&A will constitute an end-to-end test of the advanced PIE process for milling TEM and APM/APT specimens (from receipt of a metallurgical-mounted sample to removal of the milled lamellae/needles from the modular shielded enclosure). A nonradioactive sample will be used for this transition test, with FIB milling occurring in the presence of an approximate 25-R/hour sealed gamma source.

After completion of specimen milling tests, routine field maintenance (i.e., predictive/preventive maintenance actions and modular component replacements) and decontamination activities will be demonstrated.

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### 3.3.4 Maturation Testing Conditions

- Specimens: met-mounted, nonradioactive (e.g., steel samples), but milled in the presence of a sealed radiography source measuring about 25 R/hour  $\gamma$
- Biological shielding: as provided by the APEX facility modular shielded enclosure
- Sealed radiological sources: about 25R/hour  $\gamma$
- Temperature: 20°C  $\pm$ 3°C
- Relative Humidity: less than 80%
- Stray electromagnetic field (after mitigation, if necessary, for measured APEX facility location conditions):
  - Less than 100 nT asynchronous
  - Less than 300 nT synchronous
- Acoustics: less than 60 dBC
- Vibration: see TRL-6 to 7, Stage I vibration guidelines
- Available utilities:
  - Power: voltage 230 V (-6%, +10%); frequency 60 Hz ( $\pm$ 1%); power consumption: 3.0 KVA (minimum available for basic microscope)
  - Process gases: nitrogen gas (purge gas and valve control)
- Instrument air, compressed: 4 to 6 bar (clean, dry, and oil-free).

### 3.3.5 Maturation Testing Configuration

The SO&A testing configuration will be the actual DB FIB instrument installed in APEX facility in its final configuration, with the modular shielded enclosure, inert atmosphere confinement enclosure, and support systems and utilities. Final operating procedures, remote-handling tools/aids, and maintenance procedures also will be used.



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### 3.3.6 Required Data

- Temperature/humidity plots at DB FIB proximal and external to vacuum chamber versus time (for the duration of the milling test to ensure the temperature/humidity can be adequately controlled).
- Acoustic noise readings at DB FIB proximal and external to vacuum chamber versus time (for the duration of the milling test to ensure that noise reduction measures are adequate).
- Vibration (e.g., accelerometer) readings at DB FIB proximal and external to vacuum chamber versus time (for the duration of the milling test to ensure that vibration control measures are adequate).
- EMI readings at DB FIB proximal and external to vacuum chamber versus time (for the duration of the milling test to ensure that the stray electromagnetic fields are adequately mitigated).
- TEM lamellae/APM needle specimen adequacy (qualitative assessment by operator).
- Successful demonstration of maintenance and decontamination activities with operator/maintenance technician concurrence.

### 3.3.7 Maturation Testing Location

The APEX facility, which is located within MFC at INL.

### 3.3.8 Data Requirements

Data requirements include results of temperature/humidity, acoustic noise, vibration, and EMI data recordings.

### 3.3.9 Maturation Testing Evaluation Criteria

The first evaluation criterion for the SO&A will be successful demonstration of the DB FIB end-to-end lamella/needle milling process, yielding specimens adequate for TEM/APM examination, respectively.

Second, all routine maintenance and decontamination activities must be demonstrated to be feasible, safe, and effective.

The SO&A test will be repeated, if necessary, and after appropriate corrective actions, until acceptance criteria are satisfied.

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### 3.3.10 Maturation Testing Deliverables

The primary deliverable from this maturation activity will be a completed SO&A test plan report with signatures of duly authorized personnel indicating acceptance.

### 3.3.11 Resource and Duration Estimates

Table 3 provides labor hour estimates for the resources anticipated to be required to perform activities associated with the TRL-7 to TRL-8 transition.

Table 3. Labor hour estimates for the resources associated with the technology readiness level-7 to technology readiness level-8 transition.

Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Install in APEX	80	80	40	40				10	10			8	10		278
Demonstrate in APEX (SO&A Testing)	10							10	10		10		10	40	90
Totals	90	80	40	40	0	0	0	20	20	0	10	8	20	40	368

## 4. RECORDS

Records generated as a result of activities described in this maturation plan may include, for example, detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.*

## 5. REFERENCES

D. Keiser, "New FIB Sample Prep Technique Could Lead to Better Design for Nuclear Fuel," MFC News, October 4, 2011  
<[http://webfiles.inel.gov/webfiles/mfc/news/mfcnews\\_100411.pdf](http://webfiles.inel.gov/webfiles/mfc/news/mfcnews_100411.pdf)>.

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PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

**6. APPENDIXES**

None.

## **Technology Maturation Plan**

# **Advanced Post-Irradiation Examination Capabilities Project Scanning Electron Microscope**



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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available scanning electron microscope (SEM) for successful routine application in advanced post-irradiation examination (PIE) of nuclear fuels and materials within the planned Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

This plan applies to a SEM system, including components such as an electron beam column, motorized stage, detectors, image processor, vacuum system and chamber, satellite analytical tools (e.g., wavelength dispersive x-ray spectroscopy [WDS]<sup>a</sup>, energy dispersive x-ray spectroscopy [EDS]<sup>b</sup>, and electron backscatter diffraction [EBSD]<sup>c</sup>), power and control consoles, support equipment and

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<sup>a</sup>WDS is a method used to count the number of x-rays of a specific wavelength diffracted by a crystal. The wavelength of the impinging x-ray and the crystal's lattice spacing are related by Bragg's law and produce constructive interference if they fit the criteria of Bragg's law. Unlike the related technique of EDS, WDS reads (or counts) only the x-rays of a single wavelength at a time. WDS primarily is used in chemical analysis.

<sup>b</sup>EDS is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of x-ray excitation and a sample. Its characterization capabilities are due, in large part, to the fundamental principle that each element has a unique atomic structure, allowing a unique set of peaks on its x-ray spectrum. To stimulate the emission of characteristic x-rays from a specimen, a high-energy beam of charged particles (such as electrons) is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an x-ray. The number and energy of the x-rays emitted from a specimen can be detected and measured by the EDS. Because the energy of the x-rays is characteristic of the difference in energy between the two shells and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured. EDS produces a broad spectrum of wavelengths or energies simultaneously.

<sup>c</sup>EBSD is a microstructural-crystallographic technique used to examine the crystallographic orientation of many materials. EBSD can be used to index and identify the seven crystal systems; therefore, it is applied to investigations such as crystal orientation mapping, defect studies, phase identification, and grain boundary and morphology studies. EBSD can be conducted in a SEM that is equipped with an EBSD detector containing at least a phosphor screen, compact lens, and low-light charged-coupled device camera chip. When a flat/polished crystalline specimen is placed in the SEM chamber at a highly tilted angle (about 70° from horizontal) toward the diffraction camera, some of the electrons that enter the sample backscatter and may escape. The phosphor screen (located within the SEM's specimen chamber) is at an angle to intercept the escaping electrons (i.e., exiting the material at the Bragg condition related to the spacing of the periodic atomic lattice planes of the crystalline structure). Some electrons will collide and excite the phosphor causing it to fluoresce. The phosphor screen is



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associated enclosures. The plan addresses the technology maturation activities necessary to transition the SEM from a technology readiness level (TRL)-6 (minimum) to a TRL-8,<sup>d</sup> including associated test descriptions, conditions, and configurations as identified in Section 3. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 through the processing of the first production specimens).

This plan applies to the Advanced PIE Capabilities Project personnel and other MFC or INL personnel identified to support the SEM technology maturation activities and tests. While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test procedures, work orders, or laboratory instructions that will be used to direct the actual work.

Some portions of this plan also may apply to technology maturation activities for the dual-beam focused ion beam system (which includes a SEM) (e.g., the vacuum chamber and associated modifications). As such, changes in one plan should be reviewed for applicability and possible inclusion in the other.

The end user organizations for the matured and deployed SEM are expected to be MFC Operations and Nuclear Science and Technology.

### 1.3 Instrument Description

A SEM is a type of electron microscope that images a sample by scanning it with a beam of electrons in a raster scan pattern. The beam's electrons interact with electrons in the sample producing signals that contain information about the sample's surface topography, structure, composition, and other properties (such as electrical conductivity).

The types of signals produced by the interaction include secondary electrons, back-scattered electrons, and characteristic x-rays. Secondary electron detectors are standard in all SEMs and most SEMs typically include options for additional detectors. However, few SEM systems have detectors for all of the possible signals.

In the most common or standard detection mode (i.e., secondary electron imaging), SEMs can produce very high-resolution images of a sample surface,

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coupled to a compact lens, which focuses the image (diffraction pattern) from the phosphor screen onto the charged-coupled device camera.

<sup>d</sup>Refer to *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, INL/EXT-12-27849, for a description of TRL levels.

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revealing details less than 1 nm in size. Specimens can be observed in high vacuum or low vacuum. Environmental SEMs can observe specimens in wet conditions.

Magnification in a SEM is controlled by changing the size of the raster in relation to the size of the raster on the display device. In this manner, magnification of specimen images can be controlled over a very wide range (e.g., from 6 to 1000000X in the better, commercially available systems<sup>e</sup>).

SEMs were invented in the 1930s. Today, SEMs have broad application in many scientific, engineering, and industrial applications such as medicine, chemistry, energy research, life sciences, materials science, electronics, forensics, geosciences, and nanotechnology.

#### 1.4 Current Technology Maturity

SEMs have been demonstrated to be effective PIE instruments, allowing detailed and effective examination of irradiated nuclear fuels and materials. For example, SEMs are used in fuel applications in studying fuel/cladding chemical interactions and fission gas pore growth and distribution. In this application, elements from Be through Cm are studied with full matrix correction.<sup>[1]</sup> Many national laboratories and international nuclear research laboratories include SEMs for PIE and several of these systems are housed in shielded enclosures or hot cells.<sup>f</sup>

JEOL, one of several SEM manufacturers, has placed SEMs in hot cells with all electronics removed to the outside control room to avoid deterioration of the solid-state components. These instruments can be operated and maintained remotely through use of telemanipulators and through alpha-shield glove walls. JEOL also has implemented special tungsten shielding for detector preamps and for x-ray analyzers (EDS and WDS units) that allows samples with activities up to 3 Ci to be examined. In one case, a SEM chamber was attached to the back side of a glove box so the sample never left the protective atmosphere.<sup>g</sup>

Operations in the Electron Microscopy Laboratory, located within MFC at INL, have indicated no impairment of the SEM imaging functionality over 7 years of

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<sup>e</sup>This example magnification range is for a Quanta™ 650 Field Emission Gun (FEG) SEM made by the FEI Company.

<sup>f</sup>Knolls Atomic Power Laboratory in New York (state) [JEOL JSM-6490LV], Japan Atomic Energy Agency at the Oarai Research Center [JEOL JSM-7001F], Commissariat à l'énergie Atomique at the Actinide-based Materials Study Laboratory [Zeiss SUPRA 55/55VP], and Nuclear Research and Consultancy Group at Petten, Netherlands [JEOL JSM-6490] have all implemented SEMs in shielded cells in recent years (i.e., during or since 2010).

<sup>g</sup>Reference: <http://www.jeolusa.com/APPLICATIONS/Energy/tabid/733/Default.aspx#LiveTabsContent16965-lt>.

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operation in a radiation environment up to about 25 R/hour  $\gamma$  on contact (about 8 R/hour  $\gamma$  being typical) and up to about 130 R/hour  $\beta$  on contact. During this time, both irradiated fuels and materials have been examined. However, some degradation has been observed in the effective life of certain analysis components (e.g., the EDS detector) from 8 years of operation in a normal, non-radiation application to approximately 3 years in the described radiation fields. No special shielding has been implemented for the EDS unit on the Electron Microscopy Laboratory's SEM.

The specimens to be examined and processed in the APEX facility are expected to have much higher activities than those allowed for the unshielded Electron Microscopy Laboratory's SEM. An initial target value for the shielded instruments in the APEX facility is to provide the capability to examine specimens measuring up to 3 Ci of activity at 1 MeV gamma radiation or up to approximately 600 R/hour at 10 mm.<sup>h</sup> In addition to the high-radiation fields, which will necessitate remote instrument operation, the APEX facility's PIE application will involve maintaining an inert atmosphere for some samples and containment of alpha contamination.

A technology readiness assessment has been completed for SEM, which indicated that its readiness for examining irradiated fuels and materials is a TRL-6 (Table 1) due to the need to demonstrate radiation hardening of the instrument.

Table 1. Scanning electron microscope technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	Scanning Electron Microscope (SEM)	6	7	7	7	7	7	7	6	6.9

## 1.5 Program Links

The following programs are stakeholders in the successful maturation and deployment of the SEM in a shielded enclosure:

Light Water Reactor Sustainability – this program seeks to create economic and environmental benefits for existing nuclear power plants through improvements in reliability, availability, productivity, component life, safety, and security. Development of the scientific basis to allow evolution of fuels and understanding

<sup>h</sup>While it is very desirable for the APEX facility to provide a full suite of shielded instruments capable of examining specimens containing this amount of activity, it should be noted that this target value may not be achievable for all of the PIE instruments to be deployed.

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and predicting long-term degradation behavior and operational limits of materials relies on detailed PIE of reactor fuels and materials at the nano-scale.

Fuel Cycle Research and Development – this program has the responsibility of developing advanced fuel technologies using a goal-oriented, science-based approach. This approach requires a micro-structural understanding of a broad spectrum of nuclear fuels and cladding materials.

Advanced Reactor Concepts – this program seeks to leverage the latest materials and fuels technologies to develop advanced reactor designs having improved efficiency and improved economics. The behavior of these materials and fuels in various reactor environments must be fully understood at the micro/nano-scale to support effective simulations and designs.

National Defense and Security – the National Nuclear Security Administration Naval Reactors program and other national security missions (e.g., nuclear non-proliferation) can benefit from nano-scale examination of nuclear materials and fuels in achieving their program objectives.

Nuclear Energy University Programs – this program plays a key role in helping the U.S. Department of Energy accomplish its mission of leading the nation's investment in development and exploration of advanced nuclear science and technology. It promotes nuclear energy as a resource capable of meeting the nation's energy, environmental, and national security needs by resolving technical, cost, safety, security, and proliferation resistance through research, development, and demonstration. Advanced PIE capabilities at the APEX facility will provide student learning opportunities and help develop the nation's next generation of nuclear scientists and engineers.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

## 3. MATURATION TEST PLANS

### 3.1 General Information

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

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### 3.1.1 Assumptions

- This plan assumes that a SEM will be deployed in the shielded enclosure as depicted in Figures 1 through 4.
- This plan assumes the use of a field emission gun<sup>i</sup> SEM as the baseline instrument to support identification and definition of necessary technology maturation activities. Specifically, this plan references the JEOL JSM-7100F and the FEI Quanta<sup>TM</sup> 650 FEG SEM. It also is assumed that other comparable SEMs may be available on the market that would require substantially similar activities to successfully deploy in an advanced PIE application. The particular models referenced were selected because they are proven instruments.
  - The JEOL JSM-7100F SEM has already been deployed by the manufacturer for a customer who examines irradiated specimens (i.e., Japan Atomic Energy Agency Oarai Research Center). This work included radiation hardening of the instrument, modifications for remote operation, and use of an automated specimen loader. Radiation-hardened JEOL JSM-6490 SEMs also have been deployed within shielded enclosures and in conjunction with alpha confinement glove boxes (i.e., either mated to or inside the glove box). Selection of this SEM make/model for APEX would leverage JEOL's collective experience and significantly advance the starting point of these activities.
  - The FEI Quanta<sup>TM</sup> 650 FEG SEM is substantially similar to a Quanta<sup>TM</sup> three-dimensional, dual-beam focused ion beam (i.e., the make/model that is being referenced for defining associated technology maturation activities for that instrument) in that there is a significant overlap in the components used in their construction. Selection of this SEM make/model would leverage designs and lessons learned from the maturation of the dual beam focused ion beam, as well as have efficiencies due to the commonality of spare parts.

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<sup>i</sup>A field emission gun typically has years of operation before replacement as opposed to a few weeks for filament-type electron sources.

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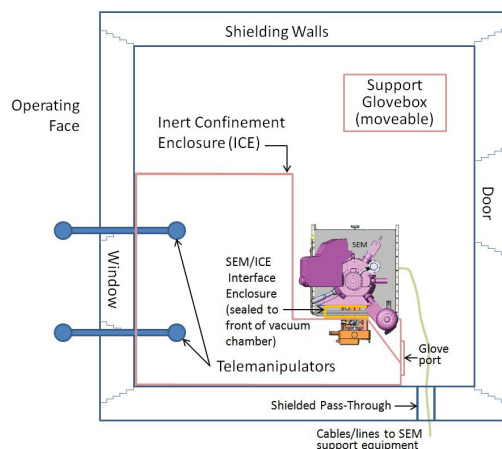


Figure 1. Scanning electron microscope deployment concept (cell arrangement).

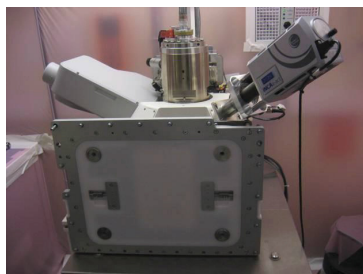


Figure 2. Scanning electron microscope side of inert confinement enclosure interface (demated).

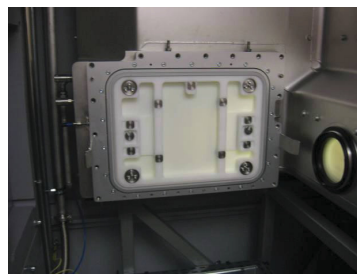


Figure 3. Inert confinement enclosure side of scanning electron microscope interface (demated).

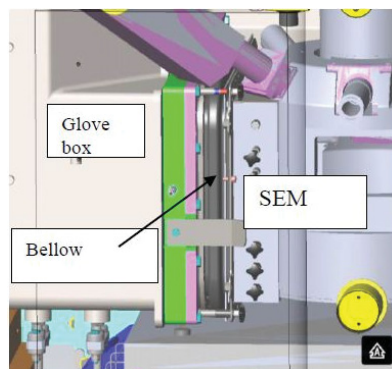


Figure 4. Scanning electron microscope to inert confinement enclosure interface (mated).<sup>j</sup>

<sup>j</sup>Figure represents a CEA patent (#11 60216). The use here is only to illustrate a feasible concept.



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- The SEM will be used exclusively for sample examinations (i.e., no ion column for focused ion beam milling). This examination capability will include detection and imaging of secondary electrons, back-scattered electrons, characteristic x-rays, and, possibly, light (cathodoluminescence) and transmission electrons. As such, it is assumed that the following accessories or third-party, add-on analysis components are present on the procured SEM and are needed to support its role in nuclear fuel and material characterization:
  - EDS – silicon drift detector type with Peltier (thermoelectric) cooling (i.e., no liquid nitrogen required)
  - WDS
  - EBSD
  - Cathodoluminescence – TBD optional examination
  - Transmission electron (STEM) – TBD optional examination
  - Other – TBD.
- The procured SEM is a special order unit that has been modified or built to meet INL and manufacturer agreed-upon specifications for service in a high-radiation environment. Specifically, these modifications will include, but are not limited to, the following:
  - Substitution of standard wiring, vacuum seals, and surface finishes/coatings with items that are compatible with high-radiation fields and, where applicable, a radiologically contaminated environment (including considerations for decontamination solutions that are anticipated to be used).
  - Relocation of certain electronics components (TBD) and controls from the SEM instrument and/or base to a location outside the shielded enclosure to enable remote operation and/or to avoid deterioration of the solid state components.
  - Modifications (TBD) to allow remote SEM loading and unloading using telemanipulators (e.g., autoloader).

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- Addition of special appliqué shielding for detector preamps and x-ray analyzers (i.e., EDS and WDS), as well as electronic components in the microscope base to prevent degradation of SEM performance and/or component operating life (see Figures 5 through 7).
- Modification or replacement of the SEM vibration protection measures (e.g., air table), if necessary, to account for microscope weight increases/redistribution, while continuing to meet SEM vibration limits/specifications.
- Modifications for implementing a leak-tight interface between the SEM vacuum chamber and the inert confinement enclosure, including provisions for vibration isolation (e.g., bellows) when mated and for maintaining confinement on both sides of the interface when demated.
- Extended (non-standard) vacuum chamber door rails, if applicable, for improved access into the vacuum chamber when mated to the inert confinement enclosure.

**NOTE:** *If these modifications are not available from or performed by the manufacturer, maturation activities will need to be added to this plan on a case-by-case basis and performed after SEM delivery.*

### 3.1.2 Prerequisites

- A special order SEM, modified for compatibility with high-radiation environments, has been procured and is available to support technology maturation testing and development activities.
- INL and/or MFC engineering personnel have worked with the instrument manufacturer to design an interface for connecting the SEM to the inert confinement enclosure that allows separation of the two for SEM maintenance while maintaining alpha/inert atmosphere enclosure integrity on both sides of the interface.<sup>k</sup> Fabrication and installation of this interface may either be done

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<sup>k</sup>Alternatively, in the case of the FEI Quanta™ 650 FEG SEM, the maturation activities for the Quanta™ three-dimensional, dual-beam FIB should be completed which include the design and fabrication of a substantially similar, if not identical, interface between the instrument and the inert confinement enclosure.



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by the manufacturer prior to shipping or by INL personnel after receipt of the instrument.

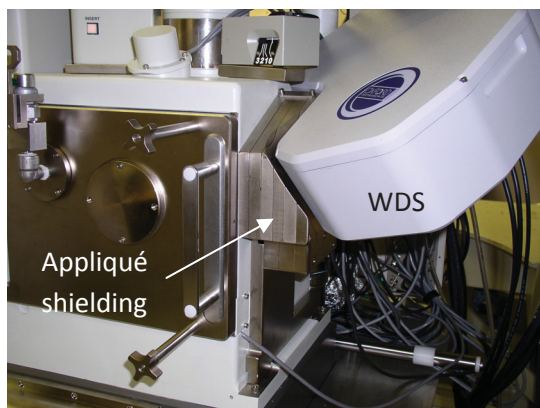


Figure 5. Example of appliqué shielding (tungsten) for wavelength dispersive x-ray spectroscopy analysis tool.

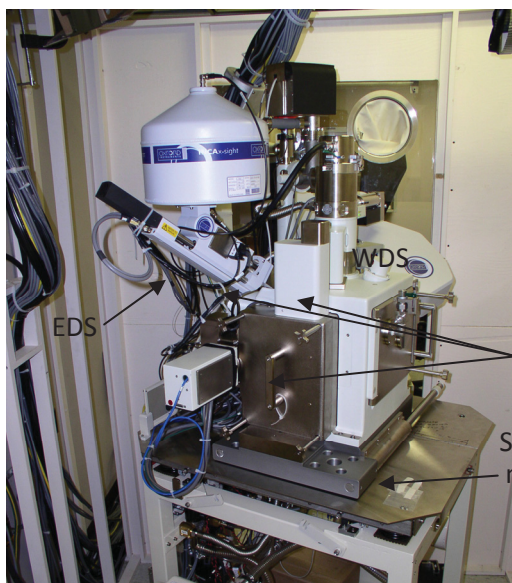


Figure 6. View of left side of radiation-hardened scanning electron microscope (JEOL JSM-6490).

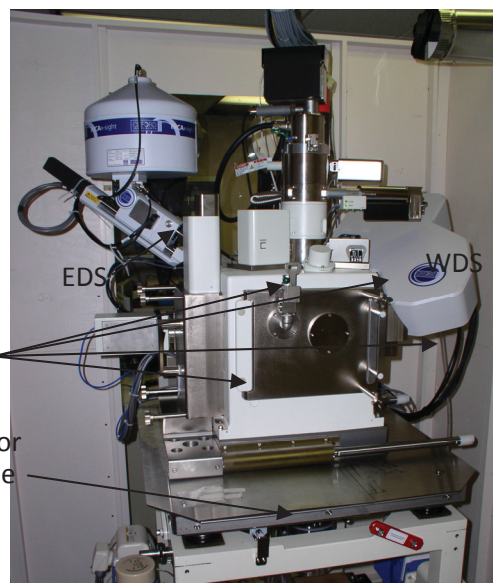


Figure 7. Front view of radiation-hardened scanning electron microscope (JEOL JSM-6490).

- The following items/utilities are available to support SEM operation and testing in the locations selected for performing the maturation activities:
  - Cooling water (e.g., for instrument chiller)

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- Instrument air (dry, oil free)
- Suspect exhaust ventilation system (including a high-efficiency particulate air filter canister near the tie-in to the SEM's vacuum pump outlet)
- Appropriate electrical power (conditioned, with backup/uninterruptible power supply)
- Adequate lighting
- Oxygen monitoring
- Fixed air-sample system
- Devices appropriate for the measurement and recording of data as identified in Sections 3.2.6 and 3.3.6.

### 3.1.3 Identified Hazards and Safety Concerns

The primary hazard associated with SEM maturation activities involves working with highly radioactive sources, which pose a risk for unanticipated worker exposures. Other hazards include, but are not limited to, electric shocks, hoisting and rigging, rotating equipment, compressed gases, and confined spaces with the potential for oxygen-deficient atmospheres.

### 3.1.4 Work Controls, Permits, and Caution/Danger Tags

All work performed pursuant to this plan will be conducted using INL standard work procedures for work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## 3.2 Plan for Technology Readiness Level-6 to Technology Readiness Level-7 Transition

To achieve a TRL-7 rating, the integrated system (i.e., including radiation-hardened SEM, inert confinement enclosure, shield walls and windows, transfer ports, and telemanipulators) needs to be successfully demonstrated in its final layout and in a relevant environment (including a high-radiation field and remote operation). This transition represents a major step up from a TRL-6, where a representative model of the system was tested in a relevant operational environment (including vibration, electromagnetic interference [EMI], and temperature, but without the high-radiation field).

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### 3.2.1 Technology Maturation Testing Approach

A two-stage maturation approach will be used to progress the SEM from TRL-6 to TRL-7. The first stage will primarily use a mockup enclosure that is dimensionally accurate (but no shielding blocks/panels or shielding windows) to verify instrument functionality after radiation hardening<sup>l</sup> of the procured instrument and the planned process steps for examining prepared samples. Approaches for integration of acoustic noise reduction measures, vibration protection, EMI protection, and confinement enclosure integration/interfaces also will be investigated and/or verified during this stage.

The second stage adds actual radiological shielding (i.e., walls, windows, and transfer ports) and finalizes the confinement enclosure interface, methods, and special tooling for remote sample handling and remote operation and allows the instrument functionality to be tested in the relevant radiation and operational environment.

Optional scope for component lifetime estimation and radiation field envelope determination also is identified in the event that resources (time and funding) are available after completion of other maturation tasks and testing.

### 3.2.2 Technology Maturation Testing Objectives

#### Stage I:

- Verify SEM functionality and acceptable performance after:
  - (a) removal and physical separation of any radiation-sensitive components from the main instrument/vacuum chamber; and
  - (b) installation of appliqué shielding in strategic locations to protect the remaining radiation-sensitive components not addressed in (a).<sup>m</sup>
- Verify through mockup testing the method for interfacing the radiation-hardened SEM microscope, including the installed analytical satellite components (at a minimum, EDS, WDS, and

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<sup>l</sup>Radiation hardening measures may include removal and relocation of radiation-sensitive components and/or the installation of strategically-located shielding materials to protect radiation-sensitive components that cannot be relocated. Radiation hardening of the SEM is assumed to have completed by the equipment manufacturer/vendor.

<sup>m</sup>This step of Stage I may be accomplished at the vendor's laboratory and instrument acceptability should be documented in a vendor test report and certification.

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EBSD) with a mockup of the inert atmosphere confinement enclosure (primarily at the SEM's vacuum chamber). Also, verify the inert atmosphere enclosure interfaces with the modular shielded enclosure, telemanipulators, and, if applicable, transfer ports.

- Verify through mockup testing the planned approach for remote specimen handling, beginning with specimen arrival in rabbit (mockup) and including unpackaging and placement in the planned SEM carrier configuration(s), specimen loading into and unloading from the vacuum chamber, specimen removal from the carrier, and, finally, subsequent specimen handling, including repackaging, storage in-cell, and removal from the shielded enclosure (as mocked up).
- Evaluate instrument performance in the relevant temperature, humidity, acoustic noise, vibration, and EMI environments provided by the selected mockup testing location<sup>n</sup> and investigate through additional mockup testing the feasibility and adequacy of any proposed mitigation measures/approaches that are anticipated to be needed.
- Verify through mockup testing planned remote operation and maintenance approaches or methods for the SEM components as deployed. This includes, but is not limited to, normal remote instrument operations (such as power on, check out/diagnostics, establishing a conduction path for the specimen, instrument and/or support equipment adjustments, instrument monitoring, and power off). Planned maintenance approaches and methods to be verified will include, but are not limited to, SEM demating from the inert confinement enclosure without compromising the enclosure's integrity or environment and serviceability or modularization improvements implemented to facilitate replacement of components having shortened life spans due to high-radiation fields.

**NOTE:** *Maintenance activities do not necessarily require remote methods if the irradiated specimen can be removed from the shielded cell prior to their initiation. Maintenance activities on components within the*

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<sup>n</sup>The environment of the selected mockup testing location should be representative of the planned APEX facility.

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*vacuum chamber may require the use of a glove bag or separate glove box.*

Stage II:

- Verify adequacy of the radiation-hardening modifications to the SEM and acceptable performance for specimen examination and analysis in a representative radiation field of about 25 R/hour  $\gamma$  on contact (sealed radiological source).
- Verify final approaches or methods selected through Stage I mockup investigations for temperature/humidity control, noise reduction, vibration protection, EMI protection, inert confinement enclosure integration (working prototype), tools/aids, remote sample handling, and remote SEM operation in conjunction with radiation fields, final clearances, and telemanipulator, shield wall, transfer port, and window configurations/positions.
- Verify acceptability of the final selected serviceability or modularization methods implemented to facilitate SEM maintenance and replacement of components having shortened life spans due to high-radiation fields in conjunction with final clearances and telemanipulator, shield wall, transfer port, and window configurations/positions.

Other optional maturation testing scope:

- Assess the radiation-hardened SEM's performance in examining and analyzing metallurgical-mounted specimens in the presence of a sealed radiological source containing up to 3 Ci of about 1 MeV gamma ( $\gamma$ ).
- Perform individual component testing of non-radiation-hardened components of the main instrument (i.e., that cannot be relocated or adequately shielded) to determine the expected life spans of the components as a function of radiation field intensity ( $\alpha$ ,  $\beta$ , and/or  $\gamma$ ). This information will be useful in setting limits, if appropriate, for the specimens to be examined/analyzed and for establishing associated maintenance and spares strategies that are economically supportable.



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### 3.2.3 Technology Maturation Testing Description

#### Stage I:

The Stage I maturation activities and testing provide an opportunity to verify the SEM, as modified for high-radiation service, within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility. First, this test will verify that SEM radiation-hardening modifications have not degraded the instrument's functionality or performance to unacceptable levels.

Second, the mockup shielded enclosure (i.e., with simulated shielding panels, windows, and transfer ports and with actual telemanipulators) will be used to verify the planned instrument layout, cell configuration, and associated interfaces with the inert confinement enclosure (also a dimensional mockup). The mockup shielded enclosure easily will be reconfigured to support quick setup of alternative configurations for team review and evaluation if any problems are identified. The use of actual telemanipulators will provide realism during process verification testing and demonstration.

Third, process verification testing will be performed to ensure that selected methods, equipment, and special tooling are effective and efficient relative to handling, loading, unloading, storing (in-cell), and removal of metallurgical-mounted specimens.

It is anticipated that several nonradioactive specimens will be examined and analyzed in the SEM during Stage I. This will provide multiple opportunities to demonstrate instrument operability, maintainability, and performance in the planned instrument/cell configuration and under relevant temperature, humidity, noise, vibration, and EMI conditions. Mitigation measures for identified, or anticipated, environmental problems (i.e., those resulting in unacceptable SEM performance) also can be proposed, designed, installed, and tested, as applicable, in order to restore satisfactory instrument performance.

#### Stage II:

The Stage II maturation activities and testing provide an opportunity to verify the SEM (as modified for high-radiation service) within a full-scale, but temporary, shielded cell enclosure similar to that which will be used in the APEX facility and within a representative radiation

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environment (i.e., minimum of 25 R/hr  $\gamma$ ).<sup>o</sup> Actual shielding blocks/panels, windows, and transfer ports, albeit temporary, will be used to provide personnel protection and added realism for this test. During this testing stage, nonradioactive specimens representing the irradiated material and fuel samples will be examined/analyzed. The representative radiation environment will be created by a sealed source placed into the SEM vacuum chamber and near the specimen to be examined.

The Stage II mockup testing will be configured and instrumented to verify integration of the inert confinement enclosure (i.e., functional prototype) and associated instrument/enclosure interfaces; final process methods, tools, and layout (positioning); remote sample handling procedures; and remote instrument operation and maintenance activities in the relevant radiological environment, including any adjustments identified based on Stage I results. These activities also will include verifying the performance of mitigation measures implemented to correct any identified, or anticipated, temperature/humidity, acoustic noise, vibration, and EMI problems. Maintenance activities to be verified include mating and demating the SEM from the inert confinement enclosure and servicing/replacement of modularized components having shortened life spans.

### 3.2.4 Maturation Testing Conditions

#### Stage I:

- Specimens: met-mounted, nonradioactive (e.g., steel samples)
- Temperature: 20°C  $\pm$  3°C
- Relative humidity: less than 80%
- Stray electromagnetic field (after mitigation, if necessary, for known or anticipated APEX facility location conditions):
  - Less than 100 nT asynchronous
  - Less than 300 nT synchronous

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<sup>o</sup>Optional maturation scope, if conducted, includes examining/analyzing non-radioactive specimens in the presence of sealed radiological sources containing up to 3 Ci at  $\sim$ 1 MeV gamma (e.g., a Cs-137/Ba-137m source).

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- Acoustics: less than 60 dBC
- Vibration: approximate limits<sup>p</sup> as shown in Figures 8 through 10 for 1/3 octave vibration spectrum:

**NOTE:** *A site-specific survey may be necessary if any of the limits in Figures 8 through 10 are exceeded.*

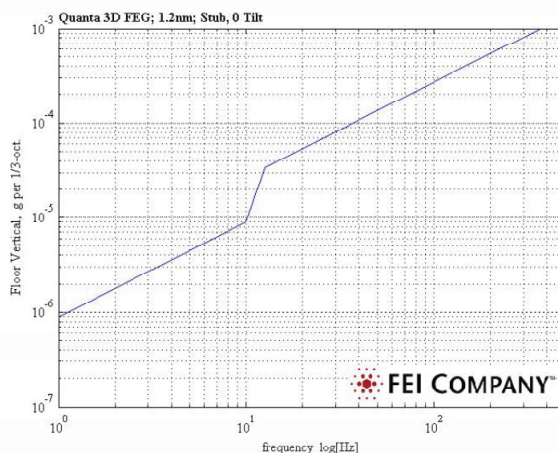


Figure 8. Floor vertical guideline.

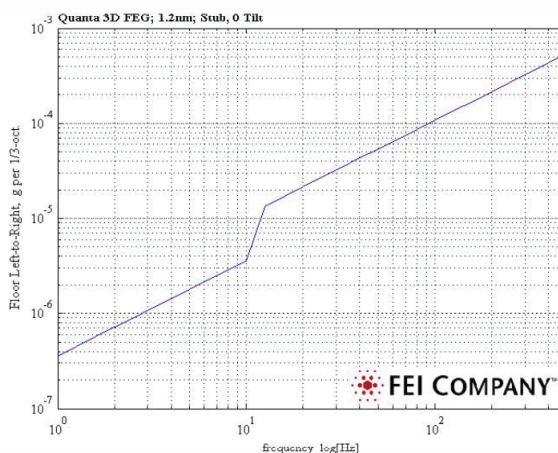


Figure 9. Floor left-to-right vibration guideline.

<sup>p</sup>Based on Quanta 3D FEG SEM vibration limits.



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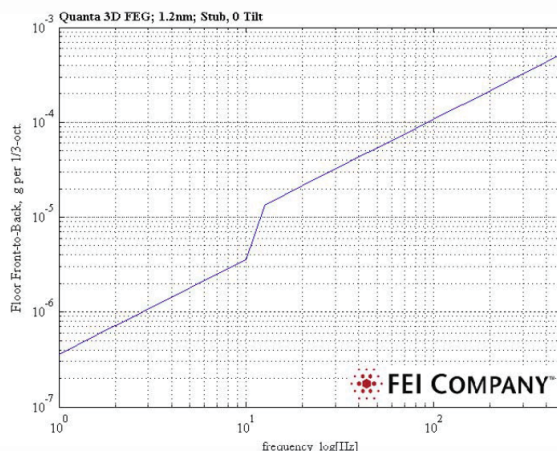


Figure 10. Floor front-to-back vibration guideline.

- Available utilities:
  - Power: voltage 230 V (+6%, -10%); frequency 60 Hz ( $\pm 1\%$ ); power consumption: 3.0 KVA (minimum available for basic microscope)
  - Process gases: nitrogen gas (purge gas and valve control); WDS tool requires P10 gas
  - Instrument air, compressed: 4 to 6 bar (clean, dry, and oil-free).

Stage II:

- Specimens: met-mounted, nonradioactive (e.g., steel samples), but examined/analyzed in the presence of a sealed radiography source measuring about 25 R/hour  $\gamma$  (minimum)
- Sealed radiological sources: about 25R/hour  $\gamma$  (optional scope sealed sources [example activities; all at 1 MeV  $\gamma$ ]: 0.01 Ci, 0.1 Ci, 0.5 Ci, 1.0 Ci, 1.5 Ci, 2.0 Ci, 2.5 Ci, and 3 Ci)
- Temperature:  $20^{\circ}\text{C} \pm 3^{\circ}\text{C}$
- Relative humidity: less than 80%
- Stray electromagnetic field (after mitigation, if necessary, for known or anticipated APEX facility location conditions):

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- Less than 100 nT asynchronous
- Less than 300 nT synchronous
- Acoustics: less than 60 dBC
- Vibration: See Stage I vibration guidelines
- Available utilities:
  - Power: voltage 230 V (+6%, -10%); frequency 60 Hz ( $\pm 1\%$ ); power consumption: 3.0 KVA (minimum available for basic microscope)
  - Process gases: nitrogen gas (purge gas and valve control); WDS requires P10 gas
- Instrument air, compressed: 4 to 6 bar (clean, dry, and oil-free).

### 3.2.5 Maturation Testing Configuration

#### Stage I:

- Mockup shielded instrument enclosure (full-scale, reconfigurable) with actual telemanipulators (make/model TBD) but simulated (a) shielding walls, (b) shielded windows, and (c) shielded transfer port(s).
- SEM deployed in radiation-hardened configuration and instrumented for obtaining temperature, humidity, acoustic noise, vibration, and EMI measurements.

#### Stage II:

- Shielded instrument enclosure (full-scale, temporary) with actual telemanipulators (make/model TBD) and real (a) biological shielding blocks/panels, (b) shielded windows, and (c) transfer port(s). Shielding shall be adequate to provide personnel protection (i.e., less than 0.5 mrem/hour at 30 cm) for all radiological sources used during testing under this plan.
- SEM deployed in radiation-hardened configuration and instrumented for radiation field, temperature, acoustic noise, vibration, and EMI measurements. Radiation monitoring

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instruments and control measures also shall be in place for supporting personnel protection.

### 3.2.6 Required Data

#### Stage I:

- Temperature/humidity plots at SEM proximal and external to vacuum chamber versus time
- Acoustic noise readings at SEM proximal and external to vacuum chamber versus time
- Vibration (e.g., accelerometer) readings at SEM proximal and external to vacuum chamber versus time
- EMI readings at SEM proximal and external to vacuum chamber versus time
- SEM image adequacy (qualitative; annotated with sample ID and date/time of examination)
- EDS analysis results (for later comparison of results in radiation field; annotated with sample ID and date/time of examination)
- WDS analysis results (for later comparison of results in radiation field; annotated with sample ID and date/time of examination)
- EBSD analysis results (for later comparison of results in radiation field; annotated with sample ID and date/time of examination)
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup).

#### Stage II:

- Temperature/humidity plots at SEM proximal and external to vacuum chamber versus time (for the duration of examination/analysis)
- Acoustic noise readings at SEM proximal and external to vacuum chamber versus time (for the duration of examination/ analysis)

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- Vibration (e.g., accelerometer) readings at SEM proximal and external to vacuum chamber versus time (for the duration of examination/analysis)
- EMI readings at SEM proximal and external to vacuum chamber versus time (for the duration of examination/analysis)
- Radiation measurements (i.e.,  $\gamma$  only) external to the SEM vacuum chamber (keyed to a description/ID of the sealed radiation source used)
- Radiation measurements (i.e.,  $\gamma$  only) at SEM key instrument components that are thought to be radiation-sensitive or vulnerable to gamma radiation and that could not be relocated external to the shielded cell walls, with any appliqué shielding in place (keyed to a description of the sealed radiation source used)
- SEM image adequacy (qualitative; annotated with sample ID, sealed source ID, and date/time of examination)
- EDS analysis results (annotated with sample ID, sealed source ID, and date/time of analysis)
- WDS analysis results (annotated with sample ID, sealed source ID, and date/time of analysis)
- EBSD analysis results (annotated with sample ID, sealed source ID, and date/time of analysis)
- Operator/maintenance technician written evaluation of equipment configuration and associated processes (qualitative; keyed to configuration managed mockup).

### 3.2.7 Maturation Testing Location

Stage I maturation activities and testing will be accomplished in the Irradiated Materials Characterization Laboratory or a comparable laboratory setting capable of achieving the conditions listed in Section 3.2.4 for Stage I.

Stage II maturation activities and testing will be accomplished in the Irradiated Materials Characterization Laboratory or a comparable laboratory setting approved for radiological work. The selected location

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must be capable of achieving the conditions listed in Section 3.2.4 for Stage II.

### 3.2.8 Data Requirements

Data quality (e.g., accuracy and precision) will be sufficient for the intended application and associated pass/fail decisions to be made. Specific data quality requirements are TBD and will be documented in detailed test plans, laboratory instructions, or other work control documents that are used to perform the maturation and testing activities.

Documentation of collected data and generated evaluations, assessments, and other results shall be retained as project records. Handling of documents shall be in accordance with PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."<sup>[2]</sup>

### 3.2.9 Maturation Testing Evaluation Criteria

The following criteria apply for determining whether the maturation and testing activities can be considered successful in transitioning the SEM from TRL-6 to TRL-7:

- Radiation-hardening measures for the SEM will be evaluated through proper instrument functioning and confirmed through examination and analysis (i.e., EDS, WDS, and EBSD) of nonradioactive metallurgical-mounted specimen while in a representative radiation environment (i.e., at a minimum of 25 R/hour  $\gamma$ ). Determination of a maximum advisable radiation field and component longevity in representative radiation fields will be done either as optional scope (if sufficient time and resources are available) or after turnover to operations.
- Acceptance of final instrument layout and shielded cell configuration (including the inert confinement enclosure) will be determined based on inputs from instrument technicians, facility operations, and maintenance personnel.
- Mitigation measures for ambient environmental conditions (i.e., other than radiation levels and including temperature, humidity, acoustic noise, vibration, and EMI levels) shall be evaluated against manufacturer's recommendations for proper instrument operation and confirmed through the successful imaging and analysis of the examined specimens.

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### 3.2.10 Maturation Testing Deliverables

A maturation testing report will be issued to document the results of Stage I and II mockup testing. The report will include any unresolved issues or recommended changes that need to be addressed in future TRL maturation activities.

### 3.2.11 Resource and Duration Estimates

Table 2 provides labor hour estimates for the resources expected to be required to perform activities leading up to and through Stage I and II maturation and testing.

Table 2. Resource labor hour estimates for Stage I and II maturation and testing.

Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Instrument selected (milestone)															0
Obtain instrument information	14														14
Develop conceptual design of instrument	120	120						4	4	4		8			260
Write instrument specification	40						4	4	4	4		8			64
Bid and award instrument	4						40	4	4	4					56
Procure instrument	0						80								80
Develop installation design	80	80													160
Procure remotization equipment	40						10	4	4	4		4			66

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Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Assemble mockup	40		80	80	80	40		8	4						332
Install instrument	40		80	80				8	4						212
Cold remote handling demonstration	40	20	20	20				10	20	10			80	80	300
Disassemble cold test	10		40	40	40	20		10							160
Reassemble in hot cell mockup (temporary shielding)	40	40	80	80		80		20			20		10	60	430
Hot demonstration	10							10	10		20		10	40	100
Disassemble hot test	10		20	20				10	10				10	60	140
Totals	488	260	320	320	120	140	134	92	64	26	40	20	110	240	2,374

### 3.3 Plan for Technology Readiness Level-7 to Technology Readiness Level-8 Transition

For TRL-8 to be achieved, the integrated readiness of the system must be demonstrated and qualified within the finished APEX facility, including its final interfaces to the modular shielded enclosure, the inert confinement enclosure, and associated utilities provided by the facility. Specifically, this means that the SEM and associated processes have been proven to work, facility representatives have issued a declaration of readiness, and the readiness activity has been successfully completed.

**NOTE:** *This readiness activity (e.g., an operational readiness review by the U.S. Department of Energy) will be performed on a facility basis and not for a single PIE instrument.*

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### 3.3.1 Technology Maturation Testing Approach

The TRL-7 to TRL-8 maturation activities for the SEM will be performed in conjunction with the readiness activity for the APEX facility. The facility readiness activity confirms that management has brought the facility to a state of readiness to commence program work. Once management concludes that readiness has been achieved, this state of readiness is independently verified by the contractor and confirmed by the U.S. Department of Energy. Only then will the APEX facility, a Hazard Category 2 nuclear facility, be authorized to “go hot” and perform programmatic PIE work.

The SEM portion of the overall facility readiness activity will involve the performance of a system operability and acceptance (SO&A) test for the instrument, modular shielded cell, and associated support systems.

### 3.3.2 Technology Maturation Testing Objectives

The objective of the TRL-7 to TRL-8 maturation activities is to successfully demonstrate, during the facility readiness activity, the integrated SEM instrument for an advanced PIE application, including the adequacy of the following:

- Instrument radiation-hardening measures and interfaces with the modular shielded enclosure and the inert atmosphere confinement enclosure
- Acoustical noise and vibration control measures appropriate for the facility and location
- EMI protection measures appropriate for the facility and location
- Remote sample handling process, including associated methods, tools, and aids
- Remote instrument operation as deployed within the modular shielded enclosure
- Any instrument modifications made to improve reliability, availability, and maintainability for its application for advanced PIE.



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### 3.3.3 Technology Maturation Testing Description

The SEM SO&A test will demonstrate correct system/process operation and verify that the installed instrument and associated modifications function as designed and in accordance with designated acceptance criteria and design inputs.

The SO&A will constitute an end-to-end test of the advanced PIE process for examining/analyzing specimens, including receipt of a metallurgical-mounted specimen via the facility rabbit system, specimen handling, loading, examination/analysis, unloading, storing (in-cell), and return to the specimen storage area. A nonradioactive sample will be used for this transition test, which will occur in the presence of an approximate 25-R/hour sealed gamma source. The end-to-end test also will include demonstrating that selected methods, equipment, and special tooling effectively and efficiently support the process.

After completion of specimen examination/analysis, routine field maintenance (i.e., predictive/preventive maintenance actions and modular component replacements) and decontamination activities will be demonstrated.

### 3.3.4 Maturation Testing Conditions

- Specimens: met-mounted, nonradioactive (e.g., steel samples), but examined/analyzed in the presence of a sealed radiography source measuring about 25 R/hour  $\gamma$
- Biological shielding: as provided by the APEX facility modular shielded enclosure
- Sealed radiological source(s): about 25R/hour  $\gamma$
- Temperature: 20°C  $\pm$ 3°C
- Relative humidity: less than 80%
- Stray electromagnetic field (after mitigation, if necessary, for measured APEX facility location conditions):
  - Less than 100 nT asynchronous
  - Less than 300 nT synchronous

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- Acoustics: less than 60 dBC
- Vibration: see TRL-6 to 7, Stage I vibration guidelines
- Available utilities:
  - Power: voltage 230 V (+6%, -10%); frequency 60 Hz ( $\pm 1\%$ ); power consumption: 3.0 KVA (minimum available for basic microscope)
  - Process gases: nitrogen gas (purge gas and valve control); WDS (if installed) requires P10 gas
- Instrument air, compressed: 4 to 6 bar (clean, dry, and oil-free).

### 3.3.5 Maturation Testing Configuration

The SO&A testing configuration will be the actual SEM instrument installed in APEX facility in its final configuration with the modular shielded enclosure, inert atmosphere confinement enclosure, and support systems and utilities. Final operating procedures, remote handling tools/aids, and maintenance procedures also will be used.

### 3.3.6 Required Data

- Temperature/humidity plots at SEM proximal and external to vacuum chamber versus time (for the duration of examination/analysis to ensure the temperature/humidity can be adequately controlled).
- Acoustic noise readings at SEM proximal and external to vacuum chamber versus time (for the duration of examination/analysis to ensure that noise reduction measures are adequate).
- Vibration (e.g., accelerometer) readings at SEM proximal and external to vacuum chamber versus time (for the duration of examination/analysis to ensure that vibration control measures are adequate).
- EMI readings at SEM proximal and external to vacuum chamber versus time (for the duration of examination/analysis to ensure that the stray electromagnetic fields are adequately mitigated).
- SEM image adequacy (qualitative assessment by operator).

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- Successful demonstration of maintenance and decontamination activities with operator/maintenance technician concurrence.

### 3.3.7 Maturation Testing Location

The APEX facility located within MFC at INL.

### 3.3.8 Data Requirements

Data requirements include the results of temperature/humidity, acoustic noise, vibration, and EMI data recordings.

### 3.3.9 Maturation Testing Evaluation Criteria

The first evaluation criterion for the SO&A will be successful demonstration of the SEM end-to-end examination/analysis process, including clear/usable images at the SEM's maximum level of magnification.

Second, all routine maintenance and decontamination activities must be demonstrated to be feasible, safe, and effective.

The SO&A test will be repeated, if necessary and after appropriate corrective actions, until acceptance criteria are satisfied.

### 3.3.10 Maturation Testing Deliverables

The primary deliverable from this maturation activity will be a completed SO&A test plan report, with signatures of duly authorized personnel indicating test acceptance.

### 3.3.11 Resource and Duration Estimates

Table 3 provides labor hour estimates for the resources anticipated to be required to perform activities associated with the TRL-7 to TRL-8 transition.

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Table 3. Resource labor hour estimates for the technology readiness level-7 to technology readiness level-8 transition

Task	Engineering	Drafting	Electrical Craft Support	Mechanical Craft Support	Carpenter Craft Support	Laborer	Procurement	Systems Engineering	Safety	Quality Assurance	Radiological Engineering	Review	Principle Investigator	Operator	Total
Install in APEX	80	80	40	40				10	10			8	10		278
Demonstrate in APEX (SO&A Testing)	10							10	10		10		10	40	90
Totals	90	80	40	40	0	0	0	20	20	0	10	8	20	40	368

#### 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.*

#### 5. REFERENCES

[1] Schulthess, J. L., *Post-Irradiation Capabilities at the Idaho National Laboratory*, INL/EXT-11-22624, Idaho National Laboratory, July 2011.

[2] PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

#### 6. APPENDIXES

None.

## **Technology Maturation Plan**

# **Advanced Post-Irradiation Examination Capabilities Project Micro X-Ray Diffraction Examination**



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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing a commercially available micro x-ray diffraction ( $\mu$ XRD) examination instrument for successful routine application in advanced post-irradiation examination (PIE) of nuclear fuels and materials within the planned Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

This plan applies to a  $\mu$ XRD examination system. It addresses the technology maturation activities to transition the  $\mu$ XRD from technology readiness level (TRL)-6.6 (average) to TRL-8,<sup>[1]</sup> including associated test descriptions, conditions, and configurations as identified in Section 3. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 by the processing of the first production specimens).

This plan applies to Advanced PIE Capabilities Project personnel and other MFC or INL personnel identified to support the  $\mu$ XRD technology maturation tests and activities. While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test procedures, work orders, or laboratory instructions that will be used to direct the actual work.

The end user organization for the matured and deployed  $\mu$ XRD is Nuclear Science and Technology, with support from MFC Operations.

### 1.3 Instrument Description

$\mu$ XRD is an extension of X-ray diffraction (XRD).

XRD is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactured materials. A crystal lattice is a regular three-dimensional distribution (e.g., cubic or rhombic) of atoms in space. These are arranged so that they form a series of parallel planes separated from one another by a distance  $d$ , which varies according to the nature of the material. For any crystal, planes exist in a number of different orientations; each with its own specific  $d$ -spacing. When a monochromatic x-ray beam with wavelength  $\lambda$  is projected onto a crystalline



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material at an angle *theta*, diffraction occurs only when the distance traveled by the rays reflected from successive planes differs by a complete number *n* of wavelengths. By varying the angle *theta*, the Bragg's Law conditions are satisfied by different *d*-spacings in polycrystalline materials. Plotting the angular positions and intensities of the resultant diffracted peaks of radiation produces a pattern, which is characteristic of the sample. Where a mixture of different phases is present, the resultant diffractogram is formed by addition of the individual patterns. Based on the principle of x-ray diffraction, a wealth of structural, physical, and chemical information about the material investigated can be obtained. A host of application techniques for various material classes is available, each revealing its own specific details of the sample studied.<sup>[2]</sup>

$\mu$ XRD is a structural analysis technique that allows for the examination of very small sample areas and is used to provide information on the structure of crystalline materials. Like conventional XRD instrumentation,  $\mu$ XRD relies on the dual wave/particle nature of x-rays to obtain information about the structure of crystalline materials. Unlike conventional XRD, which has a typical spatial resolution ranging in diameter from several hundred micrometers up to several millimeters,  $\mu$ XRD uses x-ray optics to focus the excitation beam to a small spot on the sample surface so that small features on the sample can be analyzed<sup>[3]</sup> (see Figure 1). In newer instruments, the focusing optics may be replaced by point-focus tubes.

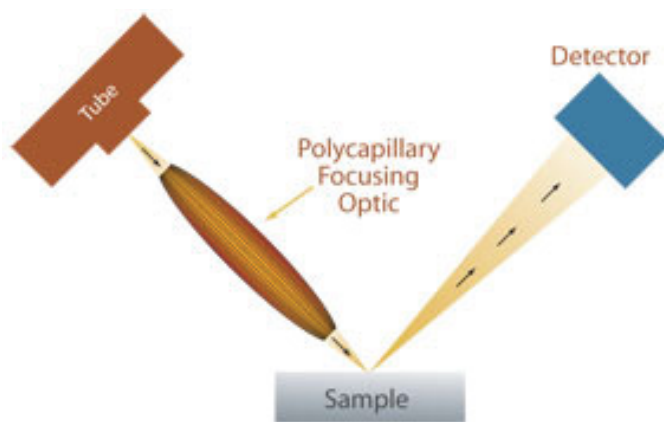


Figure 1. Micro x-ray diffraction operational concept.

#### 1.4 Advanced Post-Irradiation Examination Application

For advanced PIE, the  $\mu$ XRD would support the following needs:

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- Identify and quantify proportions of phases from small areas (few tens or hundreds of nm across) in as-fabricated and irradiated fuels and other nuclear materials. (Phase identifications are needed to use databases of thermodynamic and kinetic properties for modeling material behavior.)
- Obtain data from specific areas identified in the electron probe micro-analysis and the scanning electron microscope images, (e.g., precipitates and fuel-matrix reaction zones in dispersion fuels).
- Measure small changes in lattice parameters (e.g. to quantify the extent of radiation-induced swelling).
- Measure crystallographic preferred orientation (“texture”) (e.g. for studies of zirconium cladding failure mechanisms).

## 1.5 Current Technology Maturation

Applications include characterization of crystalline inclusions within a material, regions of defect within a fabricated sample, reduction spots in ceramics, crystalline defects in glass, filler agglomeration in plastics, reduction spots in electro-ceramics, and layers deposited on a substrate. These applications are found in aerospace, automotive, glass, ceramics and refractories, healthcare, semiconductors, and electronics industries. These applications have served to overcome vibration, electromagnetic interference (EMI), and temperature stabilities issues.<sup>[4,5]</sup>

$\mu$ XRD has been used in the nuclear industry in high-radiological areas, which include nuclear waste disposal.<sup>[6]</sup> A Bruker  $\mu$ XRD currently is operable in INL’s Analytical Laboratory for fresh fuel applications with  $\alpha$  contamination and low  $\beta\gamma$  contamination. This  $\mu$ XRD was originally purchased for eventual installation in the Irradiated Materials Characterization Laboratory and it is an older water-chilled system. Also, if this instrument is destined for relocation and use in the APEX facility, a new x-ray source (at a cost of about \$200K) would be needed in order to obtain true microfocus data. An upgrade to a new source would remove the need for the water chiller and many of the manual alignments, as well as provide significant enhancements to other capabilities. It would bring the existing XRD up to the capabilities of many new systems and make it considerably better than some.

Requirements need to be established before deciding between upgrading the existing instrument or procuring a new one. The existing Bruker  $\mu$ XRD requires a lot of adjustments to support collecting excellent data for a variety of different applications. If the only application is micro-diffraction, many of these

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adjustments will not be necessary. However, if the requirement is to collect high-precision lattice parameter determination from an irradiated metal (e.g., to measure swelling), then the  $\mu$ XRD will have to act like a powder diffractometer, which is a very different configuration from that used for micro-diffraction. And, if the requirement is to measure preferred orientation (important for understanding cladding failure during long-term storage), then yet another configuration may be required.

However, the current plan is to purchase a new state-of-the-art instrument, rather than refurbish the existing instrument. Newer systems are not likely to require the chilled water. PANalytical offers a microfocus x-ray tube that comes with a 19-in. rack mounted generator, but they have not yet integrated it into one of their XRD systems. In the future, they may be a candidate provider. This x-ray tube is “the same microsource that is being used by Rigaku on some of their SAXS instruments.”

Advanced PIE applications only work if the beam can be aimed at a specified spot. Unfortunately, not all microfocus XRDs can do this. At the time of the procurement of the existing Bruker (located in the Analytical Laboratory), Bruker and Seifert were the only diffractometer manufacturers in the world who claimed to do this. PANalytical and Rigaku could not. Therefore, Bruker and Seifert are the current likely candidates.

The  $\mu$ XRD will consist of a  $\mu$ XRD instrument combined with a video or other optical system for locating areas of interest and a five-axis Eulerian cradle. The cradle might be available as an add-on for some manufacturers, but needs to be incorporated in the initial procurement for others. The existing Bruker  $\mu$ XRD was procured (and tested) with 30-ft cables between the diffractometer and the control electronics. Two cabinets were purchased: one to go inside the high-radiation area to support the diffractometer, and one to be used outside as an electronics cabinet. Similar support equipment will be required with a new  $\mu$ XRD.

The existing Bruker  $\mu$ XRD typically uses metallurgical sample mounts, because those mounts also are used for samples prepared for the scanning electron microscope and focused ion beam instruments and it is convenient not to have to re-mount. It is anticipated that any future system also would use these same mounts. Although the  $\mu$ XRD technology can be used for examination of powders, there is no intent to examine free powders as part of the advanced PIE processes. If examined, free powders will be mounted in a high-quality encapsulated powder mount. Even if all adjustments could be done remotely, samples will still have to be loaded manually using remote methods.

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The current readiness of a  $\mu$ XRD used for examining irradiated nuclear fuels and materials is technology readiness level (TRL)-6 (minimum) due to the need to demonstrate remote operation in a radiation field (see Table 1). (The TRL was assessed to be an average of 6.6, with a minimum of 6.)

Table 1. Micro x-ray diffraction technology readiness level.

Advanced PIE		Radiation	Vibration	EMI Shield	Isothermal Operation	Remote Sample Handling	Remote Operation	RAM	Min TRL	Avg TRL
Function	Instrument									
	*Micro X-Ray Diffraction (MXRD)	6	7	7	7	6	6	7	6	6.6

The existing Bruker system can be and is operated remotely, in the sense that the instrumentation and movement is controlled by either a wired remote keypad or by a computer. However, it is anticipated that remote operation is still going to be a challenge due to some of the fine alignment adjustments necessary for sample analysis. For example, with the existing system, focusing elements must be changed to establish the desired beam size on the sample. These focusing elements are small and require attachment with small bolts. Special tools and/or jigs may have to be designed to allow attachment of such focusing elements with either a telemanipulator or with gloves through a glove port. Bruker provides a special alignment system called the Da Vinci System, which involves magnets and microchips. The focusing element is placed in close proximity to its attachment point and a magnet pulls it roughly into place. The microchips then perform the fine alignment. It is not known if the microchips are radiation hardened, but it is assumed they are not.

Some of these fine alignments might not be required if the \$200K upgrade to a point-focus tube is procured for the existing instrument or if a new instrument is appropriately specified.

It may be that activities such as change out of focusing elements can be accomplished before the sample is emplaced or after it is removed; therefore, remote operations for the finer installations and adjustments are not necessary. They could, perhaps, be performed through a glove port or by directly accessing the instrumentation if it is either not contaminated or can be decontaminated. Or, on rare occasion, they could be performed by personnel wearing appropriate personal protective equipment.

## 1.6 Program Links

All of the following programs will be stakeholders in the successful technology maturation of the  $\mu$ XRD for the APEX facility:

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Light Water Reactor Sustainability – this program seeks to create economic and environmental benefits for existing nuclear power plants through improvements in reliability, availability, productivity, component life, safety, and security. Development of the scientific basis to allow evolution of fuels and understanding and predicting long-term degradation behavior and operational limits of materials relies on detailed PIE of reactor fuels and materials at the nano-scale.

Fuel Cycle Research and Development – this program has the responsibility of developing advanced fuel technologies using a goal-oriented, science-based approach. This approach requires a micro-structural understanding of a broad spectrum of nuclear fuels and cladding materials.

Advanced Reactor Concepts – this program seeks to leverage the latest materials and fuels technologies to develop advanced reactor designs having improved efficiency and improved economics. The behavior of these materials and fuels in various reactor environments must be fully understood at the micro/nano-scale to support effective simulations and designs.

National Defense and Security – the National Nuclear Security Administration Naval Reactors program and other national security missions (e.g., nuclear non-proliferation) can benefit from nano-scale examination of nuclear materials and fuels in achieving their program objectives.

Nuclear Energy University Programs – this program plays a key role in helping DOE accomplish its mission of leading the nation's investment in the development and exploration of advanced nuclear science and technology. It promotes nuclear energy as a resource capable of meeting the nation's energy, environmental, and national security needs by resolving technical, cost, safety, security, and proliferation resistance through research, development, and demonstration. Advanced PIE capabilities at APEX will provide student learning opportunities and help develop the nation's next generation of nuclear scientists and engineers. It also will support the Advanced Test Reactor National Scientific User Facility and potentially a large range of cooperative research and development agreements and work for others within the commercial nuclear industry.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

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### 3. MATURATION TEST PLANS

#### 3.1 General Information

Further technology maturation is required and will be achieved through the following activities:

- Procure a new  $\mu$ XRD that will, at a minimum, meet the same capabilities and performance specifications as the existing instrument.<sup>[7,8]</sup>
- Perform mockup tests to bring the technology readiness to a maturity level of 7.
- Relocate and install the new  $\mu$ XRD in the APEX facility.
- Perform system checkouts to ensure the new  $\mu$ XRD performs as specified.
- Perform a system operability and acceptance test to demonstrate that the  $\mu$ XRD supports the APEX facility TRL of 8.

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

##### 3.1.1 Assumptions

- A new MXRD is procured for technology maturation and subsequent installation in the APEX facility that, at a minimum, meets the same capabilities and performance specifications as the AL Bruker MXRD instrument.
- The instrument manufacturer is willing and able to support INL engineers in identifying what components of their system are sensitive to the expected kinds of radiation.
- It is possible to keep radiation-sensitive components out of high-radiation areas or provide adequate shielding without causing unacceptable degradation to instrument performance.

##### 3.1.2 Prerequisites

- A special order  $\mu$ XRD, modified for compatibility with high-radiation environments, has been procured and is available



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to support technology maturation testing and development activities.

- INL engineering has worked with the instrument manufacturer to identify radiation-sensitive and/or vulnerable components that can be removed and separated from the main instrument or that must be protected through installation of add-on shielding to improve radiation hardening of the  $\mu$ XRD. Design and performance of such modifications may either be done by the manufacturer or by INL personnel after receipt of the instrument. Procurement requirements must take this into consideration. To the extent possible, all modifications should be done by the vendor before final acceptance, because they are likely to degrade performance unless done by an expert with in-depth knowledge and experience with the design of the original system.

### 3.1.3 Identified Hazards and Safety Concerns

The primary hazard associated with  $\mu$ XRD maturation activities involves working with highly radiological sources, which pose a risk for unanticipated worker exposures. Other hazards include radiological contamination, electric shocks, hoisting and rigging, rotating equipment, compressed gases, and confined spaces with the potential for oxygen-deficient atmospheres.

### 3.1.4 Work Controls, Permits, and Caution/Danger Tags

All work performed pursuant to this plan will be conducted using company standard work procedures for work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## 3.2 Plan for Technology Readiness Level-6 to Technology Readiness Level-7 Transition

In order to achieve TRL-7, the actual instrument must have been successfully operated in its final configuration and in a relevant environment (including radiation and remote operation), with limited degradation due to radiation exposure.

### 3.2.1 Radiation Hardening and Performance in High-Radiation Fields

The existing  $\mu$ XRD is designed for radiation hardness and has been used to examine radioactive samples, including irradiated metals, but not

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irradiated fuels (which are much more radioactive). Although hardened for radiation, at some point, the noise of the sample is expected to overcome the sample signal. In other words, the instrument sensitivity is assumed to be background limited. A new instrument is presumed to be similarly capable. This will be addressed in the procurement specification and through appropriate system design (e.g., filtering out all but the relevant x-ray energies).

A two-stage approach will be used to progress the  $\mu$ XRD from TRL-6 to TRL-7. The first stage uses a mockup enclosure (dimensionally accurate but no shielding blocks/panels or shielding windows) and addresses verification of instrument functionality after component removal/separation and addition of location-specific shielding to improve radiation hardening of the procured instrument. Approaches for confinement box integration/interfacing, remote sample handling, and remote operation will be investigated during this stage. The procurement specification for the existing  $\mu$ XRD addresses some of this and can provide useful insights<sup>[8]</sup>. Vibration protection and EMI protection are not issues for the  $\mu$ XRD.

The second stage adds radiological shielding and finalizes the confinement box interface, remote sample handling, and remote operation and allows the instrument functionality to be tested in a relevant environment.

Optional scope also is identified in the event that resources (time and funding) are available after completion of other maturation tasks and testing.

### 3.2.1.1 Maturation and Testing Objectives

#### Stage I:

- Verify  $\mu$ XRD functionality and acceptable performance after: (a) removal and physical separation of any radiation-sensitive components from the main instrument; and (b) installation of shielding in strategic locations to protect remaining radiation-sensitive components not addressed in (a). Installation of such shielding may be non-trivial and, if done incorrectly, may degrade instrument performance in subtle ways.  $\mu$ XRD functionality and



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acceptable performance will have to be verified with the shielding in place.

- Verify through mockup testing the conceptual layout (e.g., fit, clearances, and functionality) of the instrument in conjunction with the inert confinement enclosure and shielded cell, including window and telemanipulator locations.
- Investigate the feasibility of and verify through mockup testing various approaches for remote specimen handling (beginning with specimen arrival in rabbit mockup and including various carrier configurations), specimen loading onto the sample stage, specimen removal from the  $\mu$ XRD, subsequent handling, packaging, and removal from the shielded enclosure, using the rabbit or other pass-through mechanisms.
- Evaluate instrument performance in relevant temperature, humidity, air flow, vibration, noise, and EMI environments and investigate through mockup testing the feasibility and adequacy of proposed measures/approaches for providing any necessary mitigations. Extremely high or rapidly changing air flow could be an issue. Otherwise, none of these is likely to be an issue for the  $\mu$ XRD. Many metal and sodium-bonded fuel samples are air-sensitive and will need to be handled in inert atmospheres. An inert atmosphere will be required; therefore instrument procurement specifications must indicate this and the instrument will need to be tested in such an atmosphere.
- Investigate the feasibility of and verify through mockup testing various remote operation and maintenance approaches or methods for the  $\mu$ XRD as deployed. This includes, but is not limited to, normal instrument operations such as power on, check out/diagnostics, instrument and/or support equipment adjustments, instrument monitoring, change-out of focusing elements, and power off. Maintenance approaches and methods investigated

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will include, but are not limited to, improved decontamination methods and improved serviceability or modularization measures taken for components having shortened life spans due to high-radiation fields.

Stage II:

- Verify radiation-hardened  $\mu$ XRD functionality and acceptable performance for metallurgical-mounted specimens in a relevant radiation field (e.g., at or above 25 R/hour  $\gamma$  and 130 R/hour  $\beta$  on contact).
- Verify remote operability, sample handling, and updated configuration (e.g., layout, clearances, and interfaces) for instrument as mocked up, including temporary shielding, telemanipulators, inert confinement enclosure (e.g., prototype), and associated tools, aids, and procedures.
- Verify final approaches or methods selected through Stage I investigations for temperature control, air flow, vibration protection, EMI protection, confinement box integration, tools/aids, remote sample handling, and remote operation and maintenance in conjunction with radiation fields, final clearances, and telemanipulator, shield wall, and window positions.
- Verify acceptability of the selected decontamination method, serviceability, and modularization measures for anticipated, shortened-life components under actual radiological conditions.

Other optional maturation scope:

- Perform individual component testing of non-radiation hardened components of the main instrument (i.e., that cannot be relocated or adequately shielded) to determine expected life spans of the components as a function of radiation field intensity ( $\alpha$ ,  $\beta$ , and/or  $\gamma$ ). This information will be useful in setting limits, if appropriate, for the

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specimens to be examined/processed and for establishing associated maintenance and spares strategies that are economically supportable.

### 3.2.1.2 Maturation and Testing Description

#### Stage I:

The Stage I maturation activities and testing provide an opportunity to verify the  $\mu$ XRD (as modified for high-radiation service) within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility.

- First, this test will verify that instrument modifications have not degraded functionally or to unacceptable performance levels.
- Second, the mockup enclosure (i.e., simulated shielding panels, windows, and transfer ports) will be easily reconfigurable to allow various hot cell layouts, instrument/component placements, and inert confinement enclosure configurations to be investigated and evaluated. Actual telemanipulators will be employed to provide realism during process development testing. As with the existing Bruker system, it is assumed that a new  $\mu$ XRD easily can be operated remotely, in the sense that the instrumentation and stage movement is controlled by either a wired remote keypad or by a computer. Control equipment can and will be placed outside of the shielded cell mockup and only necessary instrument components will be placed inside. However, it will be necessary to demonstrate that change outs of small components (such as the focusing elements) and fine adjustments can be made (1) by telemanipulator, perhaps with special tools and/or jigs; (2) through a glove port, after the sample has been removed, again with special tools and/or jigs; or (3) by directly accessing instrument components, based on an assumption that there is no residual contamination after the sample has been removed and the instruments have been decontaminated (if necessary). The design will have

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to take into account the accessibility needs for the small component change outs. Also, it will be necessary to specify and test any extra-long cables as part of procurement to ensure that timing signals still work in spite of propagation delays in the cables.

- Third, process development testing will be performed to identify effective methods, equipment, and tools for handling, loading, and unloading specimens.

### Stage II:

The Stage II maturation activities and testing provide an opportunity to verify the  $\mu$ XRD (as modified for high-radiation service) within a full-scale mockup of the shielded cell enclosure to be used in the APEX facility and within a relevant radiation environment (e.g., at or above 25 R/hour  $\gamma$  and 130 R/hour  $\beta$  on contact). Actual shielding blocks/panels, windows, and transfer ports, albeit temporary, provide personnel protection and added realism for this test.

The mockup enclosure will be configured and instrumented to verify the final approaches, methods, and layout (positioning) chosen based on Stage I results. This includes any final mitigation measures implemented for temperature control, air flow, and vibration and EMI protection. The test also verifies integration of the final inert confinement enclosure and associated instrument/enclosure interfaces; final process equipment, tools, and aids; remote sample handling procedures; and remote instrument operation and maintenance activities under a radiological environment. Maintenance activities to be verified include decontamination and servicing/replacement of modularized components having shortened life spans under real, relevant radiological conditions.

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### 3.2.1.3 Test Conditions

#### Stage I:

- Non-radiological specimens (e.g., Cu or Ni, or even a NIST standard, such as alumina that is intended for testing diffractometer performance).
- Air environment – A mockup (not necessarily a working prototype) of the inert confinement enclosure will be used to evaluate the conceptual design for this component.
- Controlled temperature – The tests will be conducted under standard laboratory environmental operating conditions. The existing  $\mu$ XRD has to be shut down if the temperature exceeds 85°F (about 29°C). However, it is not anticipated that laboratory temperatures in the APEX facility would normally exceed this temperature. Even so, it currently is believed that the main cause of this temperature limit is the coiled power and signal cables stored within the lower cabinet. In the APEX facility, these cables would be uncoiled and extended to connect the instrumentation heads (emitters and detectors) within the shielded cell to the control electronics outside of the cell. Low temperatures do not affect the instrument. Humidity also is not an issue.
- EMI source (variable frequency, variable intensity) – The  $\mu$ XRD is not susceptible to EMI; therefore, it is not expected to require EMI shielding.
- Vibration source (variable frequency, variable amplitude) and acoustic noise – The only issue with vibration is maintaining the sample in place. Vibrations strong enough to cause flexing of the moving arms (e.g., from construction activities immediately outside the  $\mu$ XRD enclosure) could be an issue, but this kind of vibration is unlikely to occur during normal operating conditions. The  $\mu$ XRD itself is not susceptible to vibration; therefore, it does not require stringent vibration

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protection. Acoustic noise protection is not anticipated to be required.

Stage II:

- Non-radiological specimens – The current limitation for the radiation level is set by the Radiological Work Permit at 1 Rem/hour ( $\gamma$  and  $\beta$ ) on contact. Test materials (known properties, nonradioactive) will be evaluated in the presence of sealed radioactive sources to produce the relevant radiation environment. (One SME suggested that a NIST standard be used, such as alumina, which is intended for testing diffractometer performance. Another option might be to measure the outer surface of a sealed source if it is made of a suitable, known material.)
- Inert environment – A mockup (preferably a working prototype) of the inert confinement enclosure will be used to evaluate the conceptual design for that component. The instrument components do not necessarily need to be located within the inert environment; however, it may be advantageous to have the sample in an inert environment to prevent oxidation of the specimens (hence removing the influence that the oxidation layer may have on the  $\mu$ XRD results).
- Controlled temperature/humidity – The tests will be conducted under standard laboratory environmental operating conditions. The existing  $\mu$ XRD has to be shut down if the temperature exceeds 85°F (about 29°C). However, it is not anticipated that laboratory temperatures in the APEX facility would normally exceed this temperature. Even so, it currently is believed that the main cause of this temperature limit is the coiled power and signal cables stored within the lower cabinet. In the APEX facility, these cables would be uncoiled and extended to connect the instrumentation heads (emitters and detectors) within the shielded cell to the control electronics outside of

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the cell. Low temperatures do not affect the instrument. Humidity also is not an issue.

- EMI source (variable frequency, variable intensity) – The  $\mu$ XRD is not susceptible to EMI; therefore, it is not expected to require EMI shielding.
- Vibration source (variable frequency, variable amplitude) and noise – The only issue with vibration is maintaining the sample in place. Vibrations strong enough to cause flexing of the moving arms (e.g., from construction activities immediately outside the  $\mu$ XRD enclosure) could be an issue, but this kind of vibration is unlikely to occur during normal operating conditions. The  $\mu$ XRD itself is not susceptible to vibration; therefore, it does not require stringent vibration protection. Acoustic noise protection is not anticipated to be required.

#### 3.2.1.4 Test Configuration

##### Stage I:

The Stage I test configuration (see Figure 2) will consist of a mockup instrument enclosure (full-scale, reconfigurable) with actual telemanipulators (make/model TBD) and simulated (a) shielding walls, (b) shielded windows, and (c) shielded transfer port(s). The water chiller will likely not be required for newer  $\mu$ XRD systems, but is included in the diagram to reflect the existing system, which is installed at the Analytical Laboratory.

The  $\mu$ XRD will be deployed in a radiation-hardened configuration. Vendor data, analysis, and/or testing will determine which components are likely to be sensitive to radiation. To the extent possible, radiation-sensitive components will be removed and control components of the instrument will sit outside the shield enclosure. Potentially sensitive components that cannot be removed will have to be shielded/protected.

Instrumentation for obtaining temperature will be present in order to demonstrate acceptable conditions for testing.

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Humidity, air flow, vibration, noise, and EMI measurements are not expected to be required.

The existing  $\mu$ XRD is water chilled. However, it is assumed that a new, state-of-the-art instrument will not require chilled water.

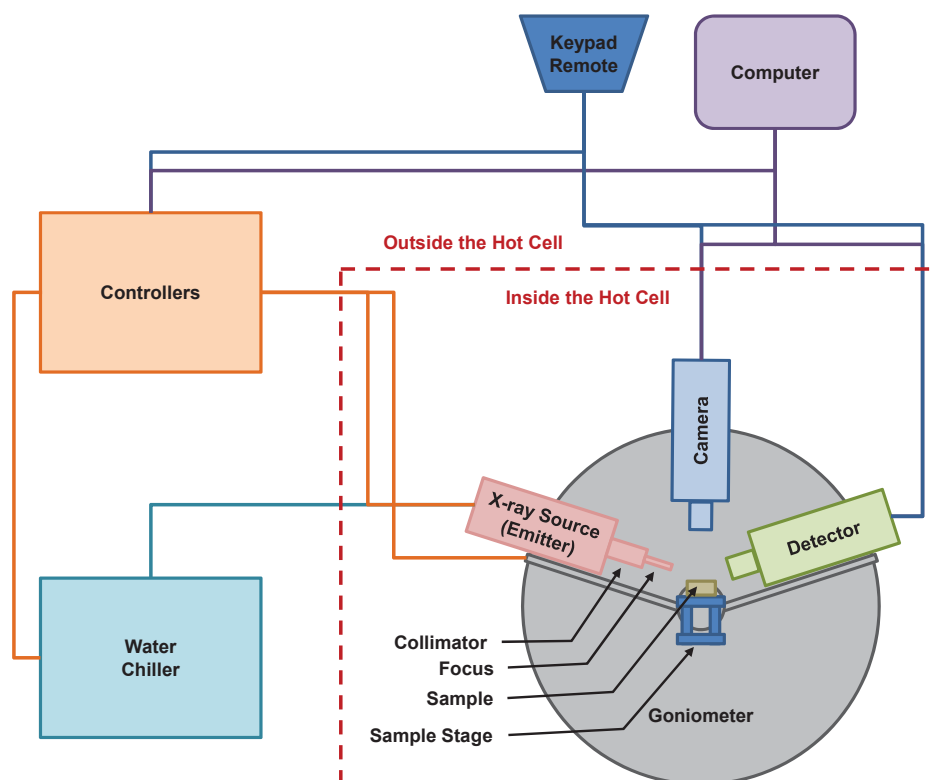


Figure 2. Micro x-ray diffraction component conceptual layout.

Stage II:

Stage II will consist of a shielded instrument enclosure (full-scale, temporary) with actual telemanipulators (make/model TBD) and real (a) shielding blocks/panels, (b) shielded windows, and (c) transfer port(s). Shielding shall be adequate to provide personnel protection to less than or equal to 0.25 mrem/hour ( $\gamma$  and  $\beta$ ) outside the shielded enclosure for all radiological sources used during testing under this maturation plan.



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Again, the  $\mu$ XRD will be deployed in a radiation-hardened configuration. No special instruments are required. Standards will be run to ensure that the measurements meet the specifications for those standards.

Instrumentation for obtaining temperature will be present in order to demonstrate acceptable conditions for testing. Humidity, air flow, vibration, noise, and EMI measurements are not expected to be required.

Again, it is assumed that a new, state-of-the-art instrument will not require chilled water.

Radiological control monitoring instruments shall be in place for ensuring personnel protection.

### 3.2.1.5 Required Data

#### Stage I:

- Instrument alignment, repeatability, load/unload duration, quality of output, number of accidental specimen drops, and damage. The performance specification for the existing  $\mu$ XRD should provide insights to useful or necessary performance tests.<sup>[8]</sup>
- Measured parameters for comparison with known standards.
- Environmental measurements (i.e., temperature).
- Written evaluation of the configuration (e.g., layout and clearances) by a subject matter expert and an operator/maintainer (qualitative; keyed to configuration managed mockup).

#### Stage II:

- Radiation measurements (i.e.,  $\alpha$ ,  $\beta$ , and  $\gamma$ ) of sealed radiation sources on contact and at 30 cm.
- Radiation measurements (i.e.,  $\alpha$ ,  $\beta$ , and  $\gamma$ ) on contact at  $\mu$ XRD main instrument components that are thought to be radiation sensitive or vulnerable to

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gamma radiation and that could not be relocated external to the shielded cell walls with any supplemental shielding in place.

- Measured parameters for comparison with known standards (keyed to the ID and/or description of the sealed radiation source).
- Environmental measurements (i.e., temperature).
- Written evaluation of the configuration by a subject matter expert and an operator/maintainer (qualitative; keyed to configuration managed mockup).

#### 3.2.1.6 Test Location

Stage I and II maturation activities and testing will be accomplished at the Irradiated Materials Characterization Laboratory or comparable laboratory setting approved for radiological work. If the APEX facility is available and ready before the  $\mu$ XRD, then the instrument could be installed in the APEX facility and tested there. If the instrument is to be tested elsewhere and then moved to the APEX facility, then it will be essential to avoid any test samples that could cause contamination to the instrument.

#### 3.2.1.7 Data Requirements

Data quality (e.g., accuracy and precision) will be sufficient for the intended application and associated pass/fail decisions to be made. Tests will have to be repeated to check for instrument degradation. The number of tests and the wait time between tests are yet to be determined.

Documentation of collected data and generated evaluations, assessments, and other results shall be retained as project records. Handling of documents shall be in accordance with PLN-4156.

#### 3.2.1.8 Maturation and Testing Evaluation Criteria

The following criteria apply for determining whether the maturation and testing activities can be considered

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successful in transitioning the  $\mu$ XRD from TRL-6.7 to TRL-7:

- Radiation-hardening measures for the  $\mu$ XRD will be evaluated through proper instrument functioning and confirmed through characterization of an irradiated metallurgical-mounted specimen having relevant radiation levels of 1 Rem/hour ( $\gamma$  and  $\beta$ ) on contact. Determination of component longevity in relevant radiation fields (and the acceptability thereof) will be done after turnover to operations due to the length of time needed for such an assessment.
- Acceptance of final layout and instrument configuration.
- Mitigation measures for ambient environmental conditions (i.e., other than radiation levels and includes temperature, air flow, vibration, and EMI levels) shall be evaluated against manufacturer's recommendations for proper instrument operation and confirmed through characterization of a non-radiological specimen.

### 3.2.1.9 Test Deliverables

A test report will be issued to document the results of Stage I and II mockup testing. The report will include any unresolved issues or recommended changes that need to be addressed in future TRL maturation activities.

### 3.2.2 Resource and Duration Estimates

Table 2 summarizes the estimated labor hours.

Table 2. Micro x-ray diffraction resource labor hours.

Resource	Instrument Modifications	Stage I	Stage II	Totals
Engineering	178	260	110	548
Drafting	120	120	80	320
Mechanical Craft	0	200	60	260
Electrical Craft	0	200	60	260

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Resource	Instrument Modifications	Stage I	Stage II	Totals
Carpenters	0	120	0	120
Laborers	0	60	0	60
Procurement	124	10	0	134
Systems Engineering	12	60	40	112
Safety	12	22	40	74
Quality Assurance	12	4	0	16
Reviewers	16	4	8	28
Principle Investigator or SME	0	50	40	90
Operators	0	60	140	200
Totals	474	1,170	578	2,222

### 3.3 Plan for Technology Readiness Level-7 to Technology Readiness Level-8 Transition

In order to achieve TRL-8, the operational readiness of the integrated system must be qualified through test and demonstration and the facility must issue a declaration of readiness. No new tests or demonstrations will be performed to transition from TRL-7 to TRL-8. Rather, the same tests and demonstrations used to transition from TRL-6 to TRL-7 will be repeated as part of the facility operational readiness review. The tests and demonstrations need not be as detailed or extensive. The primary purpose is to demonstrate acceptable operability in the final operating environment (i.e., in a hot cell in the APEX facility).

## 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.* <sup>[9]</sup>

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## **Technology Maturation Plan**

# **Advanced Post-Irradiation Examination Capabilities Project Sample Preparation Equipment**



The INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance.

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## 1. INTRODUCTION

### 1.1 Purpose

The purpose of this plan is to define the objectives, scope, requirements, and parameters for maturing commercially available sample preparation equipment for successful routine application in preparing nuclear fuels and materials specimens for advanced post-irradiation examination (PIE) within the planned Advanced Post-Irradiation EXamination (APEX) facility to be located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL).

### 1.2 Scope

This plan applies to multiple sample preparation equipment items planned for use within the alpha line and non-alpha line confinement areas (i.e., 1 through 4) within the APEX facility's sample preparation hot cells. These equipment items are listed and briefly described in Table 1 (in Section 1.3.1).<sup>a</sup>

This plan identifies and describes the technology maturation activities necessary to transition the sample preparation equipment from their current readiness level (i.e., either a technology readiness level [TRL]-6, at a minimum, or TRL-7) to a TRL-8.<sup>b</sup> This plan also defines the associated test conditions and configurations that are included in Section 3. This plan does not include maturation activities occurring after APEX facility turnover (e.g., transition from TRL-8 to TRL-9 through the preparation of the first irradiated production specimens).

This plan applies to Advanced PIE Capabilities Project personnel and other MFC or INL personnel identified to support the sample preparation equipment technology maturation activities and tests. While this plan includes general work identification, sequencing, and coordination information, it is not intended to take the place of detailed test procedures, work orders, or laboratory instructions that will be used to direct the actual work.

The end user organizations for the matured and deployed sample preparation equipment are expected to be MFC Operations and Nuclear Science and Technology.

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<sup>a</sup> The equipment items listed in Table 1 are assumed to be representative articles that have been referenced herein for planning purposes only. Other comparable equipment items that are available commercially would likely require substantially similar maturation activities. Actual equipment items to be implemented in the APEX facility may vary and may not be selected until preliminary design.

<sup>b</sup> Refer to *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, INL/EXT-12-27849, for a description of TRL levels.

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### 1.3 Background

This section provides a brief description of the sample preparation equipment, the current status of development toward their application in supporting advanced PIE for the U.S. Department of Energy (DOE) and associated programmatic stakeholders in this endeavor.

#### 1.3.1 Instrument Description






Preparation of specimens for PIE requires an assortment of equipment; all of which must be located in a shielded hot cell and capable of remote operation, including loading and unloading using telemanipulators. This equipment will be used to produce specimens (i.e., typically metallurgical mounts, chunks/disks, and powders) for examination and/or analysis in the APEX facility's shielded instrument hot cells.

Table 1. Sample preparation equipment list (in no particular order).

Equipment Name (Quantity)	Sample Graphic	Vendor/ Model No.	Location(s)	Description/Comments
Benchtop CNC Milling Machines (2)		Tormach/ PCNC-770	CA 1, CA 4	<ul style="list-style-type: none"> <li>- Used to core/mill specimens from larger source material/fuel samples.</li> <li>- Milling machine is compact in size and meets unspecified sealed containment size envelope.</li> <li>- Modular assembly, cast iron construction, automatic tool changer compatible, and 4<sup>th</sup> axis compatible.</li> </ul>
High-Speed Rotary Wafering Saw (2)		Struers/ Accutom 5 (or Secotom-10 hot cell model)	CA 1, CA 4	<ul style="list-style-type: none"> <li>- Used to saw specimens from larger source material/fuel samples.</li> <li>- The Accutom 5 has a position accuracy of 5 micrometers, adjustable force limit, sample rotation or oscillation, variable speed cutting wheel, built in recirculation unit, and enclosed cutting chamber with safety switch.</li> <li>- Closed loop, recirculation, cooling fluid system required with filtration of particulates.</li> <li>- The Secotom-10 (hot cell modified) has approximately the same capacity as the Accutom-5 for consideration.</li> </ul>





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Equipment Name (Quantity)	Sample Graphic	Vendor/ Model No.	Location(s)	Description/Comments
Low-Speed Wafering Saw (2)		Struers/ Minitom Diamond Saw (hot cell)	CA 1, CA 4	<ul style="list-style-type: none"> <li>- Used to saw specimens from larger source material/fuel samples.</li> <li>- The Minitom is controlled from outside the cell with touchpad controls placed on the front plate of the control box.</li> <li>- Operation in the hot cell can be carried out using manipulators.</li> <li>- The cabinet is made of painted aluminum. The tank for cooling water is made of stainless steel. The main motor has been fastened with screws and mounted with cable connectors; it is possible to replace the main motor in the hot cell.</li> <li>- All electrical parts, except the motor, are placed outside the cell. Just a cable needs to be led into the cell.</li> <li>- The electrical parts are mounted in a control box with the normal touchpad front plate. This box is made for rack mounting or it can stand alone.</li> </ul>
Micro Crushing/ Grinding Mill (planetary ball mill) (1)		Frisch/ Pulverisette 7	CA 1	<ul style="list-style-type: none"> <li>- Used for producing powdered samples.</li> <li>- Need to know sample particle size.</li> <li>- Uses tungsten carbide or zirconium oxide media.</li> </ul>
Dimple Punch (2)		Gatan/ Model 659	CA 1, CA 4	<ul style="list-style-type: none"> <li>- Manual punch for generating discs for dimple grinder.</li> <li>- Additional vertical stand available.</li> </ul>
Scale (4)		Mettler Toledo/ XP2002S	CA 1, CA 2, CA 3, CA 4	<ul style="list-style-type: none"> <li>- Used for weighing source sample and specimens for process control and accountability.</li> <li>- Cutting edge weighing technology, repeatability, rugged construction, protection against dust and water, and multiple interface options.</li> <li>- Hands-free operation and wireless data transfer with Bluetooth supports remote weighing safely.</li> </ul>
Hot Plate (2)		Thermo- Scientific Thermolyne/ YO-03400-10	CA 1, CA 4	<ul style="list-style-type: none"> <li>- Used in miscellaneous applications.</li> <li>- Hot plates feature a sturdy stainless steel case that resists spills and corrosion and supports heavy loads.</li> <li>- The 6-1/4-in. x 6-1/4-in.' hot plates have a 20-lb maximum load capacity.</li> <li>- 100 to 700°F range.</li> <li>- Dial operation with indicator light.</li> </ul>



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Equipment Name (Quantity)	Sample Graphic	Vendor/ Model No.	Location(s)	Description/Comments
Ultrasonic Cleaner (4)		Cole-Parmer/ S-08895-00	CA 1, CA 2, CA 3, CA 4	<ul style="list-style-type: none"> <li>- Used for cleaning specimens.</li> <li>- 3/4-gallon stainless steel vessel.</li> <li>- Mechanical timer.</li> <li>- Dial operation.</li> </ul>
Vacuum Oven (2)		Lindberg (Blue M)/ V0914SA	CA 1, CA 4	<ul style="list-style-type: none"> <li>- Used for a range of applications, including drying, curing, outgassing, aging, process control, and other applications requiring elevated temperature in reduced atmospheres or vacuum/purge with nonflammable and inert atmospheres.</li> <li>- Digital electronic control, built-in over temperature protection, and a fully flexible vacuum/purge/release system.</li> <li>- Maximum temperature = 260°C</li> <li>- Vacuum capacity = 1 Torr</li> <li>- 18.6-L volume</li> <li>- Stainless steel cabinet with three shelves.</li> </ul>
Inverted Digital Microscope (3)		Leica/ DMI 5000-M	CA 2, CA 3, CA 4	<ul style="list-style-type: none"> <li>- Used in examining specimens during preparation processes.</li> <li>- Completely mechanized microscope with remote smart panel touch pad controls.</li> <li>- Digital camera system for viewing.</li> <li>- Electronic power box can be located remotely outside hot cell.</li> </ul>
Alpha Smear Station (with smear dispenser) (3)		Custom/ NA	CA 2, CA 3, CA 4	<ul style="list-style-type: none"> <li>- Used for assessing cleanliness of the confinement area and for radiological control.</li> <li>- Custom-designed smear dispenser for use with manipulators.</li> </ul>




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Equipment Name (Quantity)	Sample Graphic	Vendor/ Model No.	Location(s)	Description/Comments
Thermal Set Resin Moulder (hot moulder) (2)		Struers (hot cell)/ CitoPress-1 and Mounting Unit	CA 1, CA 4	<ul style="list-style-type: none"> <li>- Used to create met-mounted specimens.</li> <li>- Controllable from outside the hot cell with touchpad controls placed on the front plate of the control box.</li> <li>- Operation can be carried out in the cell using manipulators.</li> <li>- Standard unit has been prepared for various cooling options such as water recirculation or air cooling.</li> <li>- Relocatable by simply lifting the front end.</li> <li>- The cabinet has been designed in stainless steel and fastened with screws that are easy to remove. The cabinet appears to have an electropolished surface.</li> <li>- The tank for the hydraulic system also is of stainless steel.</li> <li>- The top closure elevator has not been installed.</li> <li>- Electrical elements are connected with plugs for individual removal. The electric parts are mounted in a control box with the normal touchpad front plate. This box is made for rack mounting or it can stand alone.</li> </ul>
Thermal Etching (1)		Carbolite/ RHF 15/3	CA 3	<ul style="list-style-type: none"> <li>- Used for etching of specimens prior to coating (if applicable).</li> <li>- Furnace design temperature 1200 to 1500°C. Model selected is rated to 1500°C, 4,500 watts.</li> <li>- Silicon carbide elements located on both side of chamber.</li> <li>- Vertical counter-balanced door, double-skinned construction allows air flow to cool outer case to conform with EN61010.</li> <li>- Chamber vent provides for process exhaust.</li> </ul>
Spray Coating Application Station (1)	Not Available	Custom/ NA	CA 3	<ul style="list-style-type: none"> <li>- Coats specimens for applicable examination methods.</li> <li>- Custom-designed enclosure with spray nozzle for robotic manipulator operation.</li> <li>- Enclosure contains spray, preventing inadvertent coating of neighboring equipment.</li> </ul>



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Equipment Name (Quantity)	Sample Graphic	Vendor/ Model No.	Location(s)	Description/Comments
Electro- Etching (1)		Struers/ LectroPol-5 Control Unit and Polisher	CA 3	<ul style="list-style-type: none"> <li>- Used for etching of specimens prior to coating (if applicable).</li> <li>- Scanning function for easy determination of parameters.</li> <li>- Advanced software with built-in safety functions if electrolyte temperature rises above a pre-defined temperature.</li> <li>- Control/power unit is separate from polishing unit with touch panel operation.</li> <li>- Database with methods for various materials. Up to 20 user-defined methods can be saved in the database.</li> <li>- Polishing unit electrolytic solutions are stored in easily exchangeable containers, which are inserted into the polishing unit.</li> <li>- Often used in conjunction with chemical etching.</li> </ul>
Sputter Coating System (1)		Hummer/ Hummer 6.2 Sputtering System	CA 3	<ul style="list-style-type: none"> <li>- Coats specimens for applicable examination methods.</li> <li>- Single cabinet design. Complete package with etch cathode, mechanical pump, and oil.</li> <li>- Noble metal targets (i.e., gold, silver, palladium, and platinum).</li> <li>- Dual-state, direct-drive rotary vane 1.4 cfm vacuum pump; 3,000-volt, 30-milliamperes power supply.</li> <li>- Chamber: 115-mm diameter x 125-mm height; Unit: 559 mm x 508 mm x 356 mm.</li> </ul>
Sputter Coating System (1)		Leica/ EM ACE600 (basic unit)	CA 3	<ul style="list-style-type: none"> <li>- Coats specimens for applicable examination methods.</li> <li>- One touch coating.</li> <li>- Small footprint.</li> <li>- Cryo upgrade.</li> <li>- Customized configurations.</li> <li>- Intuitive operation.</li> <li>- Fully automated.</li> </ul>

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Equipment Name (Quantity)	Sample Graphic	Vendor/ Model No.	Location(s)	Description/Comments
Surface Grinder/ Polisher (3)		Struers (hot cell)/ TegraPol-15 with TegraForce-1	CA 1, CA 2, CA 4	<ul style="list-style-type: none"> <li>- Used for accessing the area of interest in the met*mounted specimen and polishing the surface prior to examination.</li> <li>- Controllable from outside the cell with touchpad controls placed on the front plate of the control box. The data are shown on a display.</li> <li>- Operation in the hot cell can be carried out using manipulators.</li> <li>- Can be moved by lifting the front end where a handle is placed. Three lifting eyes facilitate relocation.</li> <li>- All electric parts, except the motor, are placed outside the cell. Only a cable, supplying power to the motor, needs to be led into the cell.</li> <li>- Pressure and rotation are controlled from outside the cell with touchpad controls placed on the front plate of the control box.</li> <li>- The release handle for lifting the specimen mover plate has been extended, thus it can be operated by manipulators. The specimen mover is supplied with a handle for easy lowering of the specimen mover plate using manipulators. The horizontal position of the specimen mover plate is fixed.</li> <li>- TegraForce-1 can be installed on and separated from TegraPol-15 using the manipulators.</li> </ul>
Gamma Counter (2)		Ludlum/ Eberline RO-7 (discontinued Ludlum 9-7 display unit – direct replacement	CA 3, CA 4	<ul style="list-style-type: none"> <li>- Measures specimen's gamma dose rates.</li> <li>- High-range radiation ion chamber 0 to 19.99 kR/hour range</li> <li>- Digital readout replacement for Eberline Model RO-7.</li> <li>- Separate battery compartment</li> <li>- Adapts to existing Eberline RO-7 detectors</li> <li>- No mechanical switch</li> <li>- Backlit LCD</li> <li>- Three available detector ranges: <ul style="list-style-type: none"> <li>- Low range 0.001 to 1.99 R/hour</li> <li>- Mid range 0.1 to 199.9 R/hour</li> <li>- High range 0.01 to 9.99 kR/hour</li> </ul> </li> </ul>

CA = confinement area

### 1.3.2 Status of Instrument Development – Advanced Post-Irradiation Examination

Currently, irradiated fuel, cladding, and structural materials are sectioned, mounted into metallographic (or “met”) mounts, ground,



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polished, and etched in the containment box located in the Hot Fuel Examination Facility (HFEF) main cell (i.e., at Window 2M). The HFEF is located within MFC at INL. The main cell is an inert atmosphere, argon cell with approximately 4-ft thick concrete shield walls. The containment box within the main cell is sealed to the cell wall and has its own argon atmosphere and atmosphere control system. The box is separated from the rest of the main cell to minimize the spread of alpha contamination to the cell and to keep etchant and cutting fluid vapors used in the preparation of metallographic samples from contaminating the argon atmosphere of the main cell. Samples prepared in the containment box are transferred to the Metallographic loading box via a pneumatic transfer system for visual inspection using a Leitz Metallograph.

Operations within the HFEF containment box have provided a great deal of experience on associated sample preparation equipment, including cut-off saws, grinder/polishers, met mounters, etching stations, and ultrasonic cleaners. This experience includes remote sample handling, loading/unloading, equipment operation, and maintenance, as well as equipment performance and reliability in a high-radiation field and inert atmosphere environment.

The specimens to be examined and processed in the APEX facility are expected to have dose rates similar to (or slightly lower) than those processed in the HFEF containment box. Implementation of equipment into the APEX facility sample preparation hot cells will be somewhat more straightforward than for the HFEF containment box because of the planned use of nitrogen gas rather than argon. A pure nitrogen atmosphere is more similar to air than argon—both in terms of dielectric and thermal conduction properties. Sample preparation equipment to be selected for APEX facility implementation will still need to be compatible with a dry, very low moisture environment.

Thus, elements of the APEX facility design (including specific sample preparation equipment items selected for installation) that remain to be matured include the following:

1. Equipment items requiring modification for remote loading/unloading and operation
2. Equipment items requiring modification for operating in a dry, nitrogen gas atmosphere

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3. Equipment items requiring modification to function well in a high-radiation environment<sup>c</sup>
4. Final sample preparation equipment layout for process verification, including integration with hot cell and confinement enclosure clearances, interior features, and installed equipment (e.g., telemanipulators, shielding windows, material handling equipment, pass-throughs, and waste ports).

Table 2 identifies the sample preparation equipment maturation and testing activities toward deployment in the APEX facility hot cells based on INL experience and/or apparent technology readiness.

Table 2. Sample preparation equipment maturation and testing activities.

Equipment Item	Vendor/Model No.	Modification and Testing for Remote Operation/Maintenance	Modification and Testing for Dry, Inert Atmosphere (nitrogen)	Modification and Testing for Radiation Dose Rate	Layout and Process Integration Testing	Remote Operation and Fixturing Needs
Benchtop CNC Milling Machines	Tormach/PCNC-770	Yes	Yes	Yes	Yes	Coolant system integration with wafering saws. Customization of coolant catch tray. Separation of all electronics from the main frame. Extension of all cables. Adaptation of auxiliary tools.  Fixture for coring bits and MSM interface required or automatic tool changer.
High-Speed Rotary Wafering Saw	Struers/Accutom-5 (or Secotom-10 hot cell model)	Yes, for Accutom-5  No, for Secotom-10	Yes, for both	Yes, for Accutom-5  No, for Secotom-10	Yes, for both	Accutom 5 would require working with Struers to design for hot cell use like other models that Struers makes. Models would include radiation-resistant materials of construction, surfaces easy to decontaminate, easy maintenance in hot cell, compact size to reduce radioactive waste, operation optimized for use with manipulators, and separate control panel/electronics for locating outside hot cell.  Different vices required for specific cuts. Space required for dies and space required for MSM loading of vice and clamping.

<sup>c</sup> Longevity for low-cost equipment items is less of a concern than proper functionality, operability, and process reliability in the high-radiation fields. However, longevity is still a concern from a radioactive waste generation standpoint.

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Equipment Item	Vendor/Model No.	Modification and Testing for Remote Operation/Maintenance	Modification and Testing for Dry, Inert Atmosphere (nitrogen)	Modification and Testing for Radiation Dose Rate	Layout and Process Integration Testing	Remote Operation and Fixturing Needs
Low-Speed Wafering Saw	Struers/ Minitom Diamond Saw (hot cell)	No	Yes	No	Yes	Comes designed for hot cell use: Includes radiation-resistant materials of construction, easy to decontaminate, easy maintenance in hot cell, compact size to reduce radioactive waste, operation optimized for use with manipulators. Control panel with electronics is separate for locating outside hot cell. Cooling system integration with CNC mill and additional wafer saw.  Different vices required for specific cuts. Space required for dies and space required for MSM loading of vice and clamping.
Micro Crushing/ Grinding Mill (planetary ball mill)	Frisch/ Pulverisette 7	Yes	Yes	Yes	Yes	Per vendor, this model would be the easiest to remotize. Control screen could be removed and relocated, software is more robust, and operations are simpler.  Fixture for loading material in grinding mill chamber and grinding media replacement required.
Dimple Punch	Gatan/ Model 659	No	No	No	Yes	Additional vertical stand potentially for easier use.
Scale	Mettler Toledo/ XP2002S	No	No	No (low cost)	Yes	Terminal is removable and is Blue Tooth printer capable. Need extension of ribbon cable.  Check weights staging area required with MSM interface.
Hot Plate	Thermo-Scientific Thermolyne/ YO-03400-10	No	No	No (low cost)	Yes	No remotization required.  No fixturing required.
Ultrasonic Cleaner	Cole-Parmer/ S-08895-00	No	No	No (low cost)	Yes	No remotization required.  Basket loading fixture needed for MSM interface.
Vacuum Oven	Lindberg (Blue M)/ V0914SA	Yes	Yes (heating element compatibility)	No	Yes	Oven controls, electronics, and vacuum pump will need to be relocated outside the hot cell. Vacuum pump exhaust will need to be filtered.  Crucible or tray is needed for the loading area.

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Equipment Item	Vendor/Model No.	Modification and Testing for Remote Operation/Maintenance	Modification and Testing for Dry, Inert Atmosphere (nitrogen)	Modification and Testing for Radiation Dose Rate	Layout and Process Integration Testing	Remote Operation and Fixturing Needs
Inverted Digital Microscope	Leica/ DMI 5000-M	Yes	No	No	Yes	Requires locating Leica STP6000 SmartTouch Panel outside hot cell, along with PC and screen for viewing camera display. Fixturing is TBD.
Alpha Smear Station (with smear dispenser)	Custom/ NA	No	No	No	Yes	None required.
Thermal Set Resin Mounter (hot mounter)	Struers (hot cell)/ CitoPress-1 and Mounting Unit	No	Yes	No	Yes	Comes designed for hot cell use: Includes radiation-resistant materials of construction, easy to decontaminate, easy maintenance in hot cell, compact size to reduce radioactive waste, operation optimized for use with manipulators. Control panel with electronics is separate for locating outside hot cell.  It is possible that a new fixture will be needed to provide the ability to move hot mounter around in cell.
Thermal Etching	Carbolite/ RHF 15/3	Yes	No	Yes	Yes	Unit's electronics and controls will need to be relocated outside of the hot cell. Fixturing is TBD.
Spray Coating Application Station	Custom/ NA	Yes (design verification and validation)	No	No	Yes	Custom unit built for remote application. Fixturing involves custom enclosure with spray nozzle attachment.
Electro-Etching	Struers/ LectroPol-5 Control Unit and Polisher	Yes	No	Yes	Yes	Power unit may need to be located outside hot cell. Anode and cathode can be located in hot cell with extended cables.  May require holding station and clamps for anode and cathode to sample.
Sputter Coating System	Hummer/ Hummer 6.2 Sputtering System	Yes	Yes	Yes	Yes	Unit's electronics, vacuum pump and controls will potentially need to be relocated outside hot cell. Vacuum pump exhaust will need to be filtered. Unit is complex and remotization may be difficult. Shielding the unit also may be required. Fixturing is TBD.

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Equipment Item	Vendor/Model No.	Modification and Testing for Remote Operation/Maintenance	Modification and Testing for Dry, Inert Atmosphere (nitrogen)	Modification and Testing for Radiation Dose Rate	Layout and Process Integration Testing	Remote Operation and Fixturing Needs
Sputter Coating System	Leica/EM ACE600 (basic unit)	Yes	Yes	Yes	Yes	Touch screen and electronics need to be relocated outside of the hot cell and materials of construction may need to be substituted for radiation resistance. Fixturing is not applicable.
Surface Grinder/Polisher	Struers (hot cell)/TegraPol-15 with TegraForce-1	Yes (without Tegradoser)  No (with Tegradoser-5 or 1)	Yes	No	Yes	Requires customization of cooling fluid system or the addition of the Tegradoser-5 (\$20,000) or TegraDoser-1 (\$1,000), which is hot cell ready.  It is possible that a new fixture will be needed to provide the ability to move grinder/polisher around in the hot cell.
Gamma Counter	Ludlum/Eberline RO-7 (discontinued Ludlum 9-7 display unit – direct replacement)	Yes	No	Yes	Yes	Requires custom shielding and stand for counting samples. Remotization of the control unit, perhaps located outside hot cell. Ionization chamber only in hot cell (see options for cable lengths up to 60 ft).  Ionization chamber to be located in hot cell wall with capability to replace. Table or stand required for placement of sample count.

CNC = computer numerically controlled

MSM = master-slave manipulator

TBD = to be determined

### 1.3.3 Program Links

The following programs are stakeholders in the successful maturation and deployment of the sample preparation equipment into the APEX facility's sample preparation hot cells:

Light Water Reactor Sustainability – this program seeks to create economic and environmental benefits for existing nuclear power plants through improvements in reliability, availability, productivity, component life, safety, and security. Development of the scientific basis to allow evolution of fuels and understanding and predicting long-term degradation behavior and operational limits of materials relies on detailed PIE of reactor fuels and materials at the nano-scale.

Fuel Cycle Research and Development – this program has the responsibility of developing advanced fuel technologies using a

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goal-oriented, science-based approach. This approach requires a micro-structural understanding of a broad spectrum of nuclear fuels and cladding materials.

Advanced Reactor Concepts – this program seeks to leverage the latest materials and fuels technologies to develop advanced reactor designs having improved efficiency and improved economics. The behavior of these materials and fuels in various reactor environments must be fully understood at the micro/nano-scale to support effective simulations and designs.

National Defense and Security – the National Nuclear Security Administration Naval Reactors program and other national security missions (e.g., nuclear non-proliferation) can benefit from nano-scale examination of nuclear materials and fuels in achieving their program objectives.

Nuclear Energy University Programs – this program plays a key role in helping DOE accomplish its mission of leading the nation's investment in development and exploration of advanced nuclear science and technology. It promotes nuclear energy as a resource capable of meeting the nation's energy, environmental, and national security needs by resolving technical, cost, safety, security, and proliferation resistance through research, development, and demonstration. Advanced PIE capabilities at the APEX facility will provide student learning opportunities and help develop the nation's next generation of nuclear scientists and engineers.

## 2. APPLICABLE DOCUMENTS

INL/EXT-12-27849, *Advanced Post-Irradiation Examination Capability Project Technology Readiness Assessment*, December 2012.

## 3. MATURATION TEST PLANS

### 3.1 General Information

The following subsections identify assumptions, prerequisites, anticipated safety hazards, and work controls associated with the technology maturation activities of this plan.

#### 3.1.1 Assumptions

- This plan assumes that the sample preparation equipment items (i.e., makes/models) identified in Table 1 will be the ones

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deployed in the APEX facility sample preparation hot cells (alpha and non-alpha lines).

**NOTE:** *It is likely that some of these items may be changed out during preliminary or final design. If this should occur, the change should be evaluated relative to the maturation activities defined in this plan and adjustments made as applicable.*

- It is assumed that, if available, hot cell compatible versions of these items are procured (i.e., as off-the-shelf or catalog items). Otherwise, it is assumed that standard items are purchased and modified after receipt, if needed and as practical, to meet INL specifications for service in a hot cell. Specifically, these modifications may include, but are not limited to, the following:
  - Substitution of standard components (e.g., wiring, seals, and electric motors) with ones that are compatible with high-radiation fields and the dry, nitrogen gas atmosphere.
  - Relocation of certain electronics components (TBD) and controls from the main unit to a location outside the hot cell to enable remote operation and/or to avoid deterioration of solid state components.
  - Modifications (TBD) to allow remote sample loading and unloading using telemanipulators.
- It is assumed that the alpha and non-alpha line sample preparation equipment all needs to be fully remote (including operation and preventive/corrective maintenance) from initial installation to its replacement at the end of useful life. This assumption is consistent with the equipment's use within a contaminated hot cell, where manned entries will not typically be made for maintenance.
- It is assumed that all equipment items will need to be compatible with a dry, nitrogen atmosphere.
- It is assumed that all equipment items essentially will be plug-and-play to facilitate replacement at the end of their useful life.
- It is assumed that the shielded sample preparation area (SSPA) has not been contaminated (at least on one side) and is available



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for use to support the Advanced PIE Capabilities Project sample preparation equipment maturation and testing activities.

### 3.1.2 Prerequisites

- The various sample preparation equipment items have been procured and are available to support technology maturation testing and development activities.
- The SSPA has been installed in the Irradiated Materials Characterization Laboratory (IMCL), including associated ventilation, power, and utilities.
- The following items/utilities are available to support sample preparation equipment operation and testing in the locations selected for performing maturation activities:
  - Cooling water
  - Appropriate electrical power
  - Adequate lighting
  - Nitrogen gas supply (e.g., bottles)
  - Oxygen monitoring
  - Telemanipulators (1 pair) – Central Research Laboratories, Model R
  - Devices appropriate for the measurement and recording of data as identified in Sections 3.2.6 and 3.3.6.

### 3.1.3 Identified Hazards and Safety Concerns

The primary hazard associated with sample preparation maturation activities involves working with highly radioactive sealed sources, which pose a risk for unanticipated worker exposures. Other hazards include electrical shocks, rotating equipment, flying debris, hot surfaces, compressed gases, chemical exposures, and, possibly, confined spaces, with the potential for oxygen-deficient atmospheres.



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### 3.1.4 Work Controls, Permits, and Caution/Danger Tags

All work performed pursuant to this plan will be conducted using INL standard work procedures for work planning and control, radiological work permits, confined space work permits, and lockout/tagout.

## 3.2 Plan for Technology Readiness Level 6 to Technology Readiness Level 7 Transition

To achieve a TRL-7 rating, the sample preparation equipment should be successfully demonstrated in their final configuration (i.e., verifying individual functionality after modifications if any) within an integrated layout (i.e., for overall process verification) and in a relevant environment (i.e., verifying operability in a high-radiation field and dry, nitrogen atmosphere environment). This transition represents a major step up from TRL-6, where representative models of system elements may have been tested in a relevant operational environment (including vibration, electromagnetic interference, and temperature, but without the high-radiation field). Some maturation activities, other than the overall layout and process integration testing, may be omitted for sample preparation equipment items that have already been sufficiently demonstrated for use in PIE work.

### 3.2.1 Technology Maturation Testing Approach

Largely due to the various starting points relative to the technology readiness for the sample preparation equipment items, the first stage of maturation testing will use a piecemeal approach to “fill in the gaps” for individual equipment items. Once all of the sample preparation equipment have been prepared for remote use and for a high-radiation and dry, nitrogen atmosphere environment, then all of the equipment will be tested in a second maturation stage (in an integrated mockup configuration) to verify overall process capability.

### 3.2.2 Technology Maturation Testing Objectives

#### Stage I:

- Determine (by analysis, similarity, and/or testing) whether equipment items, for which INL (or other organizations) have

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little or no hot cell experience, will operate normally in a relevant radiation environment.<sup>d</sup>

- Verify normal equipment operation and functionality after modifications, if any, for the following:
  - Enabling remote control, loading/unloading, maintenance, and/or replacement via telemanipulators and in-cell material handling equipment
  - Ensuring compatibility with the dry, nitrogen gas environment<sup>e</sup>
  - Improving radiation-hardening and ability for decontamination, including removal and physical separation of any radiation-sensitive components from the main unit.
- For individual equipment items with which INL (or other organizations) has little or no remote experience, verify remote loading, operability, unloading, and maintainability (including replacement at end of life) of these items using telemanipulators.

### Stage II:

Verify the adequacy of the planned layout and integration of all sample preparation equipment, by line (i.e., alpha and non-alpha, if applicable), for supporting the sample preparation process. This includes verifying this equipment in conjunction with associated sample preparation hot cell and confinement enclosure clearances, interior features, and other installed equipment (e.g., telemanipulators, shielding windows, hoists, pass-throughs, and waste ports).

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<sup>d</sup>This determination does not include assessing the impacts on the equipment item's lifespan in the relevant radiation environment.

<sup>e</sup>These modifications will be limited to either relocation of incompatible components outside the confinement enclosure or replacement of such components with versions that are compatible with the dry, nitrogen gas environment (e.g., replace standard electric motors with brushless versions).

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### 3.2.3 Technology Maturation Testing Description

#### Stage I Activities:

1. Modify equipment items not already set up or procured specifically for remote loading/unloading, operation, maintenance, and/or replacement in a hot cell using telemanipulators and in-cell material handling equipment. These may include the following (see Table 2 for necessary modifications):
  - Benchtop computer numerically controlled (CNC) milling machine
  - High-speed rotary wafering saw (if Accutom-5 is used)
  - Micro-crushing/grinding mill
  - Vacuum oven
  - Inverted digital microscope
  - Thermal etching system
  - Spray coating application system
  - Electro-etching/polishing system
  - Sputter coating systems (2)
  - Surface grinder/polisher systems
  - Gamma counter.
2. In a mockup or suitably equipped enclosure (e.g., MFC-765 window/telemanipulator mockup station, newly constructed mockup, or the SSPA), verify that the equipment items identified in Activity 1 can be successfully and efficiently loaded/unloaded, operated, and maintained via telemanipulators and that the equipment items operate and function normally after modification.

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3. Modify equipment items not already set up or procured specifically for the dry, nitrogen gas environment. These may include the following (see Table 2 for necessary modifications):
  - Benchtop CNC milling machine
  - High-speed rotary wafering saw
  - Low-speed wafering saw
  - Micro-crushing/grinding mill
  - Vacuum oven (ensure heating element compatibility)
  - Thermal set resin mounter (hot mounter)
  - Sputter coating systems (2)
  - Surface grinder/polisher systems.
4. Verify that equipment items identified in Activity 3 operate and function normally after modification.
5. In a shielded enclosure (e.g., the SSPA or similar approved hot cell/ shielded enclosure), verify, if deemed necessary,<sup>f</sup> that the equipment items listed as follows will operate normally in a relevant radiation environment (i.e., in the presence of a sealed radiation source):<sup>g</sup>
  - Analytical scale
  - Hot plate
  - Ultrasonic cleaner
  - Vacuum oven
  - Inverted digital microscope.

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<sup>f</sup>These equipment items are not expected to require radiation hardening based on previous experience or due to their relatively low cost (i.e., expendable).

<sup>g</sup>This determination does not include assessing the impacts on the equipment item's lifespan in the relevant radiation environment.

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6. Modify equipment items, if necessary, that are not already radiation-hardened or procured specifically for the high-radiation dose rate (and, if applicable, contaminated) environment. These are expected to include the following (see Table 2 for necessary modifications):
  - Benchtop CNC milling machine
  - High-speed rotary wafering saw (if Accutom-5 is used)
  - Micro-crushing/grinding mill
  - Thermal etching system
  - Electro-etching/polishing system
  - Sputter coating systems (2)
  - Gamma counter.
7. In a shielded enclosure (e.g., the SSPA or similar approved hot cell/ shielded enclosure), verify that equipment items listed in Activity 6 (above) will operate normally in a relevant radiation environment (i.e., in the presence of a sealed radiation source) after radiation hardening modifications have occurred.<sup>h</sup>

#### Stage II Activities:

1. For each sample preparation line (i.e., alpha and, if provided, non-alpha), construct a mockup for and perform integration testing of the sample preparation equipment layout (by window) in conjunction with hot cell and confinement area clearances, interfaces, features, transitions, and installed equipment.<sup>i</sup> For example, this mockup might be the sample preparation equipment arranged on connected, rolling tables with partitions/dividers to represent pass-throughs at confinement area boundaries.

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<sup>h</sup>This determination does not include assessing the impacts on the equipment item's lifespan in the relevant radiation environment.

<sup>i</sup>Installed equipment also includes non-powered tools (e.g., calipers and punches) used within the hot cell and cooling fluid recirculation and filtering systems that support some of the sample preparation equipment items.

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2. Using the mockup created in Activity 1, perform integrated testing to verify the remote sample preparation process flows (i.e., for each specimen type: met mount, chunk, etc.) and that prepared specimens of adequate quality can be produced. This testing will be performed for non-radioactive, surrogate samples only.
3. Using the mockup created in Activity 1, perform integrated testing to verify methods and any associated fixtures or special tooling to be used for remote maintenance and replacement of the sample preparation equipment items.

### 3.2.4 Maturation Testing Conditions

#### Stage I:

- Surrogate source material and fuel samples: non-radioactive (e.g., steel samples)
- Relevant radiation environment (i.e., as created by use of one or more sealed radiation sources) (see Stage I Activities 5 and 7, above)
- Shielded enclosure: SSPA, or equivalent (see Stage I Activities 5 and 7, above)
- Temperature: TBD
- Vibration: TBD
- Available utilities:
  - Power: voltage 115 V and 230 V; frequency 60 Hz; power consumption: TBD
  - Process gases: TBD (likely dry, nitrogen gas).

#### Stage II:

- Surrogate source material and fuel samples: non-radioactive (e.g., steel samples)
- Temperature: TBD

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- Vibration: TBD
- Available utilities:
  - Power: voltage 115 V and 230 V; frequency 60 Hz; power consumption: TBD
- Process gases: TBD (likely dry, nitrogen gas).

### 3.2.5 Maturation Testing Configuration

#### Stage I Configurations (by maturation activity):

1. Individual equipment items (listed as follows) as procured from the manufacturer/vendor to be modified for remote operation and maintenance:
  - Benchtop CNC milling machine
  - High-speed rotary wafering saw (if Accutom-5 is used)
  - Micro-crushing/grinding mill
  - Vacuum oven
  - Inverted digital microscope
  - Thermal etching system
  - Spray coating application system
  - Electro-etching/polishing system
  - Sputter coating systems (2)
  - Surface grinder/polisher systems
  - Gamma counter.
2. Individual equipment items (as listed and modified in Activity 1 for remote sample loading/unloading, operation, and maintenance) set up for testing (i.e., actual sample preparation activity on a non-radioactive surrogate material) one at a time at a location with comparable telemanipulators to be used in the APEX facility. Location may be the MFC-765, Fuel Conditioning

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Facility, window/telemanipulator high bay mockup station, a newly constructed mockup station, or the SSPA after installation in IMCL.

3. Individual equipment items (listed as follows) as procured from the manufacturer/vendor and, possibly, previously modified for remote operation and maintenance and to be modified for the dry, nitrogen atmosphere:
  - Benchtop CNC milling machine
  - High-speed rotary wafering saw
  - Low-speed wafering saw
  - Micro-crushing/grinding mill
  - Vacuum oven (ensure heating element compatibility)
  - Thermal set resin mounter (hot mounter)
  - Sputter coating systems (2)
  - Surface grinder/polisher systems.
4. Individual equipment items (as listed and modified in Activity 3 for a dry, nitrogen atmosphere) set up for testing (i.e., actual sample preparation activity on a non-radioactive surrogate material) one at a time at a suitable location. This activity does not need to be performed remotely because it is only intended to verify that the equipment still functions after modification. Location is TBD, but can be the instrument shop where the modifications take place.
5. Individual equipment items (listed as follows) as procured from the manufacturer/vendor to be verified, if desired, for normal operation and functionality in a relevant radiation environment (i.e., actual sample preparation activity on a non-radioactive surrogate material, but in the presence of a sealed radiation source). If deemed necessary, these items must be set up in a shielded enclosure (e.g., in the SSPA after installation in IMCL or in a similar approved hot cell) for individual testing and evaluation:



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- Analytical scale
  - Hot plate
  - Ultrasonic cleaner
  - Vacuum oven
  - Inverted digital microscope.
6. Individual equipment items (listed as follows) as procured from the manufacturer/vendor and, possibly, previously modified for remote operation and maintenance and a dry, nitrogen atmosphere that are to be modified, as appropriate, for the high-radiation/contamination environment (i.e., to be radiation hardened):
- Benchtop CNC milling machine
  - High-speed rotary wafering saw (if Accutom-5 is used)
  - Micro crushing/grinding mill
  - Thermal etching system
  - Electro-etching/polishing system
  - Sputter coating systems (2)
  - Gamma counter.
7. Individual equipment items (as listed and modified in Activity 6 for a high-radiation/contamination environment) set up for testing (i.e., actual sample preparation activity on a non-radioactive surrogate material) individually in an appropriate shielded enclosure (e.g., the SSPA after installation in IMCL or similar approved hot cell) to verify normal operation and functionality in a relevant radiation environment (i.e., in the presence of a sealed radiation source) after modification.

Stage II Configurations (by maturation activity):

- A separate mockup configuration will be provided for each sample preparation line (i.e., alpha and, if provided, non-alpha

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line). These configurations include a mockup of the hot cell working surface with the actual sample equipment installed in the planned arrangement. The mockup will include a pair of actual telemanipulators (preferably CRL Model R), working hoist(s), and physical representations for defining hot cell and confinement enclosure clearances, interior features, and other installed equipment (e.g., shielding windows, pass-throughs, pneumatic rabbit station, and waste ports) as planned for the APEX facility's sample preparation hot cells.

This mockup must be capable of demonstrating a workable equipment layout with appropriate clearances between equipment items, but also be capable of supporting other Stage II maturation testing activities. One concept is to install the sample preparation equipment arranged on connected, rolling tables with partitions/dividers representing the boundaries between adjacent confinement areas. When maturation tasks have been completed for one confinement area (i.e., table), the tables can then be translated relative to the mockup window/telemanipulators to allow maturation activities to continue for the adjacent confinement area (i.e., table).

- The same configuration used in Stage II Activity 1 will then be used to support integrated testing to verify the remote sample preparation process flows. This testing will be performed using non-radioactive, surrogate sample materials only to produce actual specimens from the surrogate material.
- The same configuration used in Stage II Activity 1 and 2 will then be used to support integrated testing to verify remote sample preparation equipment maintenance and replacement methods, including associated fixtures or special tooling to be used.

### 3.2.6 Required Data

#### Stage I:

- Temperature/humidity plots for tests/activities if this information is deemed applicable and appropriate.
- Vibration (e.g., accelerometer) readings for tests/activities if this information is deemed applicable and appropriate.

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- Prepared specimen adequacy (qualitative; annotated with sample ID and date/time of preparation). This data also should include radiation dose rate measurements for tests performed within a relevant radiation environment.
- Operator/technician written evaluation of equipment performance after modifications (qualitative; keyed to particular modification and evaluation, such as radiation-hardening modifications or modifications for remote operation and maintenance).

Stage II:

- Temperature/humidity plots for tests/activities if this information is deemed applicable and appropriate.
- Vibration (e.g., accelerometer) readings for tests/activities if this information is deemed applicable and appropriate.
- Operator/technician written evaluation of equipment arrangement (qualitative space allocation assessment; keyed to configuration managed mockup for each sample preparation line and confinement area).
- Operator/technician written evaluation of equipment arrangement relative to supporting sample preparation process flows (qualitative; keyed to configuration managed mockup for each sample preparation line and confinement area).
- Operator/maintenance technician written evaluation of equipment arrangement relative to supporting equipment maintenance and replacement (qualitative; keyed to configuration managed mockup for each sample preparation line and confinement area).

**3.2.7 Maturation Testing Location**

Possible locations for Stage I maturation activities include the following:

- SSPA after installation in IMCL – this shielded enclosure can support remote operability and maintainability assessments for individual equipment items, and equipment operability and functionality in a relevant radiation environment. Alternate shielded enclosure/hot cell locations are TBD.

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- MFC-765 (Fuel Conditioning Facility) high-bay mockup window and telemanipulator station; this station can support remote operability and maintainability assessments for individual equipment items.
- IMCL (open floor space) – this area could support remote operability and maintainability assessment if a new mockup window and telemanipulator station were constructed.
- MFC mechanical/electrical/instrumentation and control shops (various, TBD) – these areas might be used to support equipment modifications and for performing verification tests for normal equipment operation and functionality after modifications have been made.
- All locations should be capable of achieving the conditions listed in Section 3.2.4 for Stage I, as applicable. Tests involving sealed radiation sources will need to be performed in areas approved for that type of radiological work.

Stage II maturation activities and testing will be accomplished in IMCL or a comparable laboratory setting. Stage II activities do not require an area approved for radiological work. The selected location must be capable of achieving the conditions listed in Section 3.2.4 for Stage II.

### 3.2.8 Data Requirements

Data quality (e.g., accuracy and precision) will be sufficient for the intended application and associated pass/fail decisions to be made. Specific data quality requirements are TBD and will be documented in detailed test plans, laboratory instructions, or other work control documents that are used to perform the maturation and testing activities.

Documentation of collected data and generated evaluations, assessments, and other results shall be retained as project records. Handling of documents shall be in accordance with PLN-4156, “Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project.”<sup>[1]</sup>

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### 3.2.9 Maturation Testing Evaluation Criteria

The following criteria apply for determining whether the maturation and testing activities can be considered successful in transitioning the sample preparation equipment from their various starting points to a TRL-7:

- Radiation-hardening measures for the sample preparation equipment items will be evaluated through proper equipment functioning and, if applicable, confirmed through the examination and acceptance of the intermediate or final specimen product (i.e., non-radioactive surrogate material).
- Acceptance of final sample preparation equipment arrangement, associated hot cell/confinement area configuration, and sample preparation process flows will be determined based on inputs from PIE researchers/technicians, facility operations, and maintenance personnel.

### 3.2.10 Maturation Testing Deliverables

A maturation testing report will be issued to document the results of Stage I equipment modifications and verification testing and Stage II mockup testing. The report will include any unresolved issues or recommended changes that need to be addressed in future TRL maturation activities.

### 3.2.11 Resource and Duration Estimates

#### Stage I Activities:

- Design engineering (electrical/mechanical)
- Remote engineering
- Radiological engineering and radiological control support
- Sample preparation equipment subject matter experts (researchers/technicians)
- Craft support (e.g., machinists, carpenters, laborers, pipefitters, and electricians).

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Stage II Mockup:

- Design engineering (electrical/mechanical)
- Remote engineering
- Sample preparation equipment subject matter experts (researchers/technicians)
- Craft support (e.g., machinists, carpenters, laborers, pipefitters, and electricians).

### 3.3 Plan for Technology Readiness Level-7 to Technology Readiness Level-8 Transition

For TRL-8 to be achieved, the integrated readiness of the sample preparation equipment must be demonstrated and qualified within the finished APEX facility sample preparation hot cells, including final interfaces to the hot cell components, the confinement area enclosures, installed equipment (e.g., telemanipulators, windows, pass-throughs, and waste ports) and associated utilities provided by the facility. Specifically, this means that the sample preparation equipment and associated sample preparation methods/processes have been proven to work, facility representatives have issued a declaration of readiness, and the readiness activity has been successfully completed.

**NOTE:** *This readiness activity (e.g., an operational readiness review by DOE) will be performed on a facility basis and not for individual pieces of equipment.*

#### 3.3.1 Technology Maturation Testing Approach

The TRL-7 to TRL-8 maturation activities for the sample preparation equipment will be performed in conjunction with the readiness activity for the APEX facility. The facility readiness activity confirms that management has brought the facility to a state of readiness to commence program work. Once management concludes that readiness has been achieved, this state of readiness is independently verified by the contractor and confirmed by DOE. Only then will the APEX facility, a Hazard Category 2 nuclear facility, be authorized to “go hot” and perform programmatic PIE work.

The sample preparation portion of the overall facility readiness activity will involve the performance of a system operability and acceptance (SO&A) test for the equipment as installed in the sample preparation hot cells and supported by facility utility systems.

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### 3.3.2 Technology Maturation Testing Objectives

The objective of the TRL-7 to TRL-8 maturation activities is to successfully demonstrate, during the facility readiness activity, the integrated sample preparation system (i.e., hot cell lines and installed equipment) relative to its ability to support the advanced PIE capability, including the following:

- Equipment radiation-hardening measures and features to facilitate decontamination
- Equipment modifications for improving compatibility with the dry, nitrogen atmosphere of the sample preparation hot cells
- Remote sample preparation processing (comprehensive for sample preparation lines and specimen types), including source material handling, sample loading/unloading, remote equipment operation with any associated fixtures, tools, and aids
- Remote sample preparation equipment maintenance and equipment handling evolutions for replacement at the end of equipment life.

### 3.3.3 Technology Maturation Testing Description

The sample preparation equipment SO&A test will demonstrate proper system/process operation and verify that the installed equipment items and associated modifications function as intended and in accordance with designated acceptance criteria and design inputs.

The SO&A will constitute an end-to-end test of the advanced PIE sample preparation process, including receipt of sample source material through finished specimen loading into the facility rabbit system for transfer. This testing will apply to both the alpha and, if provided, non-alpha sample preparation lines. A nonradioactive source material sample will be used for this transition test, which will occur in the presence of an approximate 25-R/hour sealed gamma source. The end-to-end test also will include demonstrating that selected methods, equipment, and special tooling effectively and efficiently support the sample preparation process.

After completion of testing for sample preparation process adequacy, routine equipment maintenance (i.e., predictive/preventive maintenance

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actions), equipment item replacement, and decontamination activities will be demonstrated.

### 3.3.4 Maturation Testing Conditions

- Specimens: nonradioactive surrogate source material (e.g., steel samples), but examined/analyzed in the presence of a sealed radiography source measuring about 25 R/hour  $\gamma$
- Biological shielding: as provided by the APEX facility sample preparation hot cells
- Sealed radiological source(s): about 25R/hour  $\gamma$
- Temperature: TBD
- Relative humidity: TBD
- Vibration: TBD
- Available utilities:
  - Power: voltage 115 V and 230 V; frequency 60 Hz; power consumption: TBD
  - Process gases: dry, nitrogen gas (hot cell atmosphere).

### 3.3.5 Maturation Testing Configuration

The SO&A testing configuration will be the actual sample preparation equipment pieces installed in APEX facility sample preparation hot cells in their final configuration, confinement area enclosures, and support systems and utilities. Final operating procedures, remote handling tools/aids, and maintenance procedures also will be used/demonstrated.

### 3.3.6 Required Data

- Temperature/humidity plots if this information is deemed applicable and appropriate.
- Vibration (e.g., accelerometer) readings if this information is deemed applicable and appropriate.



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- Prepared specimen adequacy (qualitative assessment by PIE researcher/technician/operator).
- Successful demonstration of maintenance and decontamination activities with operator/maintenance technician concurrence.

### 3.3.7 Maturation Testing Location

The APEX facility located within MFC at INL.

### 3.3.8 Data Requirements

Data requirements include results of temperature/humidity and vibration data recordings if collected.

### 3.3.9 Maturation Testing Evaluation Criteria

The first evaluation criterion for the SO&A will be the successful demonstration of the end-to-end sample preparation process, including successful production of each type of specimen (and from each sample preparation line).

Second, all routine remote maintenance and decontamination activities must be demonstrated to be feasible, safe, and effective. This includes evolutions for changing out equipment items at the end of their useful life.

The SO&A test will be repeated, if necessary and after appropriate corrective actions, until acceptance criteria are satisfied.

### 3.3.10 Maturation Testing Deliverables

The primary deliverable from this maturation activity will be a completed SO&A test plan report, with signatures of duly authorized personnel indicating test acceptance.

### 3.3.11 Resource and Duration Estimates

SO&A test:

- Sample preparation subject matter experts (researchers/technicians)
- Radiological engineering and radiological control support

## Idaho National Laboratory

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#### 4. RECORDS

Records generated as a result of activities described in this maturation plan may include detailed test plans, test procedures, and test reports. Final versions, as well as any revisions, of these documents will be submitted to Document Control and made available through the INL Electronic Document Management System.

**NOTE:** *PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project," provides information on the management, retention, quality assurance, and/or destruction moratorium requirements for these records.*

#### 5. REFERENCES

[1] PLN-4156, "Records Management Plan for the Advanced Post-Irradiation Examination Capabilities Project."

#### 6. APPENDIXES

None.