Post Irradiation Capabilities at the Idaho National Laboratory

J.L. Schulthess[a], K. E. Rosenberg[b]

Abstract. The U.S. Department of Energy (DOE), Office of Nuclear Energy (NE) oversees the efforts to ensure nuclear energy remains a viable option for the United States. A significant portion of these efforts are related to post-irradiation examinations (PIE) of highly activated fuel and materials that are subject to the extreme environment inside a nuclear reactor. As the lead national laboratory for nuclear energy, Idaho National Laboratory (INL) has a rich history, experience, workforce, and capabilities for performing PIE. However, new advances in tools and techniques for performing PIE now enable understanding the performance of fuels and materials at the nano-scale and smaller level. Examination at this level is critical since this is the scale at which irradiation damage occurs. The INL is on course to adopt advanced tools and techniques to develop a comprehensive nuclear fuels and materials characterization capability that is unique in the world. Because INL has extensive PIE capabilities currently in place, a strong foundation exist to build upon as new capabilities are implemented and work load increases. In the recent past, INL has adopted significant capability to perform advanced PIE characterization. Looking forward, INL is planning for the addition of two facilities that will be built to meet the stringent demands of advanced tools and techniques for highly activated fuels and materials characterization. Dubbed the Irradiated Materials Characterization Laboratory (IMCL) and Advanced Post Irradiation Examination capability, these facilities are next generation PIE laboratories designed to perform the PIE work that cannot be performed in current DOE facilities. In addition to physical capabilities, INL has recently added two significant contributors to the Advanced Test Reactor-National Scientific User Facility (ATR-NSUF), Oak Ridge National Laboratory and University of California Berkeley.

1. INTRODUCTION [1–3]

The U.S. Department of Energy (DOE) Office of Nuclear Energy (NE) oversees the research, development, and demonstration activities that will ensure nuclear energy remains a viable energy option for the United States. Fuel and material development through fabrication, irradiation, and characterization play a significant role in accomplishing the research needed to support nuclear energy. All fuel and material development requires the understanding of irradiation effects on the fuel performance and relies on irradiation experiments ranging from tests aimed at targeted scientific questions to integral effects under representative and prototypic conditions.

The DOE recently emphasized a solution-driven, goal-oriented, science-based approach to nuclear energy development. Nuclear power systems and materials were initially developed during the latter half of the 20th century and greatly facilitated by the United States' ability and willingness to conduct large-scale experiments. Fiftytwo research and test reactors were constructed at what is now Idaho National Laboratory (INL), another 14 at Oak Ridge National Laboratory (ORNL), and a few more at other national laboratory sites. During this time, the United States embraced a regulatory process that relied, and still relies, heavily on the use of experiments to confirm the ultimate safety of nuclear power systems and materials.

[a] Idaho National Laboratory (INL), USA
[b] Idaho National Laboratory (INL), USA

Building on the scientific advances of the last two decades, our understanding of fundamental nuclear science, improvements in computational platforms, and other tools can now enable technological advancements with less reliance on large-scale experimentation.

Advanced tools and techniques enable the science-based approach to emphasize an understanding of the behavior of fuels and materials in a nuclear reactor irradiation environment at the nano-scale and smaller. Understanding of nuclear fuel and material performance in the nuclear environment at this scale is critical to the development of the innovative fuels and materials required for tomorrow’s nuclear energy systems since this is the scale at which irradiation damage occurs. This of course is the driving need for establishing an advanced PIE capability at INL that can perform PIE on highly activated fuels and materials at ever increasing levels of sophistication.

2. GOALS [2]

Idaho National Laboratory has a vision to develop a consolidated PIE complex at the INL Materials and Fuels Complex (MFC), which is unique in the world in terms of comprehensive characterization and analytical capabilities applied to nuclear fuels and materials and efficient operations. In some cases, new capabilities beyond the current state-of-the-art need to be developed and implemented to perform detailed measurements. Such detailed measurements were not necessary in the more traditional empirically based approach that was used in previous fuel development and qualification programs.

3. CHALLENGES [3,10]

Traditional PIE capabilities at U.S. DOE laboratories, universities, and in the private sector are widely distributed and lack the state-of-the-art capability necessary to meet the U.S. nuclear energy mission need. Current PIE capabilities will continue to be necessary to serve basic needs for fuel examination, material handling, and waste disposal but are limited in their ability to function on the micro, nano, and atomic scale. The DOE-NE mission will be difficult to achieve without establishing a suitable nuclear facility environment that can accommodate these capabilities for research on highly radioactive fuels and materials. In addition, DOE seeks to make these capabilities available to the broader nuclear energy research community as a user facility concept, similar to the INL Advanced Test Reactor-National Scientific User Facility (ATR-NSUF), to enable the United States to effectively harness the intellectual capital of the country to advance U.S. research and development (R&D) goals and objectives.

4. CURRENT CORE CAPABILITIES [2, 4, 9]

Idaho National Laboratory is the United States’ only DOE laboratory with a core mission of supporting the development of nuclear energy. INL has a long history of performing PIE to support various DOE programs and has an existing trained workforce, with considerable expertise in fuels and materials technology. This expertise forms a foundation to build upon as new capabilities are implemented and work load increases.

At INL, the core nuclear and radiological facilities needed to support PIE R&D capabilities already exist, e.g.:

(a) Hot Fuel Examination Facility (HFEF) with neutron radiography capability, a large inert environment hot cell facility with the ability to receive and process large material and fuel components,
(b) Analytical Laboratory (AL), focused on analysis of irradiated and radioactive materials

(c) Electron Microscopy Laboratory (EML), radiological facility containing optical, scanning, and analytical microscopes
(d) Fuels and Applied Science Building (FASB), used for fuel development, materials characterization and irradiated materials testing
(e) Carbon Characterization Laboratory (CCL), used to conduct both pre-irradiation and post-irradiation material property measurements of carbon/graphite materials.
(f) Center for Advanced Energy Studies (CAES), by design, the CAES research facility operates in the same manner as universities do; in the case of low risk radiological research, this approach provides a cost-effective, innovative, and productive environment for exploring fundamental science questions and executing basic research complementary to research at INL site facilities.

The majority of these facilities exist at MFC while CCL and CAES are located at the INL Research and Education Campus (REC). INL also has close proximity to irradiation facilities like ATR and the Transient Reactor Experiment and Test (TREAT) facility. INL's current capabilities are outlined in more specific detail in Tables 4.1–4.7. Recently many of these current capabilities, particularly the macroscopic examination capabilities, have undergone or been scheduled for refurbishment. Collectively, the combined capabilities at INL now provide the most comprehensive capability in the United States.

TABLE 4.1 NON-DESTRUCTIVE EXAMINATION CAPABILITIES

Capability	Description
Neutron radiography	250 kW TRIGA reactor, two beam tubes and two separate radiography stations.
Precision gamma scanning	Measures fission and activation-product activity distribution.
Dimensional inspection	Continuous contact profilometer. Measures diameter profiles.
Element/capsule bow and length examination	Measures distortion (bow) and length of fuel elements.
Visual exam	Dedicated workstation, and in-cell exam stage.
Eddy current examination	Measures material defects
High precision specific gravity measurements	Pycnometer and immersion scales

TABLE 4.2 SAMPLE PREPARATION CAPABILITIES

Preparation type	Preparation
Solid metallography	Sectioned and cut
	Mounted into metallographic bases
	ground and polished
Gas sampling	Laser puncture and gas collection

TABLE 4.3 CHEMICAL, ISOTOPE, AND RADIOLOGICAL ANALYSIS CAPABILITIES

Elemental / chemical mass concentration and isotopic analyses
Inductively coupled plasma mass spectrometry (ICP-MS) with dynamic reaction cell (DRC)
Inductively coupled plasma atomic emission spectroscopy (ICP-AES)
Carbon, oxygen, and nitrogen analysis
Atomic absorption analysis
Thermal ionization mass spectrometry (TIMS)
Gas mass analysis
Isotope mass separator
Gross and isotopic radiological analysis
Gross alpha/beta analysis
Gamma spectroscopy analysis
Alpha spectroscopy analysis
Micro-gamma analysis
Beta spectroscopy analysis

TABLE 4.4 MECHANICAL PROPERTY EXAMINATION CAPABILITIES

Capability	Equipment
Metallography	Leitz metallograph or Olympus IX70 optical microscope (up to 1500X) at EML
Microhardness testing	LECO AMH43 microhardness tester
Tensile testing	FASB or HFEF Instron tensile tester w/ furnace (1600 C)
Shear punch testing	FASB or HFEF Instron tensile tester w/ furnace (1600 C)

TABLE 4.5 THERMAL PROPERTY EXAMINATION CAPABILITIES

Capability
Thermal diffusivity (laser flash method and scanning diffusivity analysis)
Differential scanning calorimitry (DSC)
High temperature accident testing of HTGR fuel

TABLE 4.6 MICROSTRUCTURE PROPERTY ANALYSIS CAPABILITIES

Capability	Equipment
Scanning transmission electron microscope (STEM)	JEOL 2010, 200 kV, 2,000 X to 1,500,000 X, equipped with energy dispersive X-ray spectrometer
Scanning electron microscope (SEM) with energy dispersive (EDS) and wavelength dispersive X-ray spectrometers (WDS) and electron back scatter diffraction detector (EBSD)	JEOL JSM 7000F FEG SEM, up to 30 kV and 600,000 X. Zeiss DSM 960a SEM, up to 30 kV LEO 1455 VP SEM, up to 30 kV, operates at higher pressures
Dual beam focused ion beam (FIB)	FEI Quanta 3D FEG, up to 1,280,000X, ion source enables site specific sectioning of materials for 3D analysis or high resolution TEM characterization
Shielded electron microprobe	Capable of analyzing elements from Be through Cm with full matrix correction, including fission gases on samples
X-ray diffractometer	Micro diffractometer performs micro-scale phase identification, small-sample powder diffraction and texture determination

TABLE 4.7 RECENT CAPABILITY ADDITIONS

Capability	Equipment
Nano indentation	CAES - Hysitron Tribo Indenter (TI-950) yields relation of mechanical properties on a very small length scale
Dual beam focused ion beam (FIB)	CAES - FEI Quanta 3D Field Effect Gun enables site specific micro sampling for use in TEM characterization
Field effect gun-scanning transmission electron microscope (FEG-STEM)	CAES - Tecnai TF30 Field Effect Gun Super Twin (STwin) with scanning transmission electron microscope, 300kV provides better penetration of heavy element nuclear fuels and better resolution with ferritic/martensitic materials
Local electrode atom probe (LEAP)	CAES – Imago LEAP 4000X HR, creates atom-by-atom maps by extracting atoms from a needle-shaped sample tip then using time-of-flight spectroscopy to determine the specific atom type. 3D reconstruction of up to hundreds of millions of atoms
Scanning electron microscope (SEM)	CAES – JEOL JSM-6610LV/TMP SEM with EDX and EBSD systems,
Scanning thermal diffusivity microscope (STDM)	AL – Shielded in cell thermal properties measurements

TABLE 4.7 RECENT CAPABILITY ADDITIONS (cont.)

Electron probe micro analyzer (EPMA)	AL – Elemental redistribution, coupled with FIB gives atomic scale elemental redistribution analysis
Carbon oxygen nitrogen analyzer	AL – LECO – provides elemental analysis

6. FUTURE CAPABILITIES [3, 5–7, 9–10]

Idaho National Laboratory hosts a large variety of PIE capabilities and is upgrading and adding more. For example, INL currently has plans to stand up an irradiation assisted stress corrosion cracking rig (IASCC) in FASB to provide valid crack growth data on unirradiated benchmark material specimens. INL also plans to stand up a hot cell electron discharge machine (EDM) for use in sample preparation.

Feedback from potential users was recently solicited via the U.S. National PIE Workshop to ensure any new capability is optimized for customer needs. The workshop helped identify many needs that are currently unfilled as well as capabilities that otherwise don't meet current needs due to capacity, configuration, etc. The top four needs identified in the workshop are as follows:

(a) Instrumentation that supports analysis of materials at the nano-scale to support increasing knowledge of mechanisms that causes material degradation at the nano-scale. As such, an advanced PIE facility designed and constructed to support analysis on highly activated fuels and materials that incorporates strict environmental controls (vibration, electro-magnetic interference, temperature, etc.) and can also support environmental testing of materials (e.g., high temperature testing) with sample storage and preparation that is adjacent to other irradiation and PIE capabilities to support the advanced characterization tools is critical.

(b) Coupling the advanced characterization capabilities with advanced modeling so that better and more predictive models can be developed, thus reducing the need for extensive empirical integral effects testing.

(c) Accessibility and infrastructure that support sharing of data and creating administrative systems that facilitate collaborations with entities that are subject to different constraints than national laboratories. This need includes the physical infrastructure to perform PIE and the non-physical needs of creating systems that are flexible to deal with entities (i.e., national laboratory, industry, university) and projects (e.g., experiment, project and reactor timeline vs. career scale) that have different time cycles and support the development, hiring, and retention of nuclear material R&D talent.

(d) Pursued development of in-situ techniques, analysis, and instrumentation that supports real-time data acquisition for deformation mechanics, damage development, and other time resolved measurements. In-situ characterization shows the development of material characteristics with time compared to traditional static PIE work.

The reinvigoration of nuclear fuels and materials research is bringing new and different tools to PIE and nuclear and radioactive materials characterization. These new tools, and the research materials examined with them, require unique, reconfigurable, accessible, modularized support facilities that are not presently available at INL. To support the INL's customers, the CAES will house many of these new instruments that focus on nano-scale and atomic-level characterization, where examinations can be completed using micrograms or nanograms of irradiated specimens prepared at the MFC. As new capability is created by the scientific community, the CAES will be the entry point for bringing new analysis technologies to the INL.

The rapid evolution of analytical electron microscopes and the advent of high-performance computer interfaces with instruments were not envisioned when many of the existing facilities were constructed at INL. A new laboratory operational model will promote and support continual implementation of state-of-the-art tools and technologies.

To meet the needs of the future, INL is standing up new capabilities that have the sophistication and refinement to house next generation PIE characterization equipment. The near term capability is the Irradiated Materials Characterization Laboratory (IMCL); the subsequent longer term facility will be an Advanced Post-Irradiation Examination capability.

6.1 Irradiated Materials Characterization Laboratory (IMCL)

The IMCL (see Fig. 6.1) will be the first facility of its type in the United States designed specifically for advanced instrumentation and equipment. The IMCL will contain space for installation of instruments and equipment within shielding structures that can be redesigned and refitted whenever necessary. The IMCL will also have mechanical systems that tightly control temperature, electrical and magnetic noise, and vibration to the standards required for advanced analytical equipment. Although some of the advanced characterization equipment is already in use in other industries, IMCL will be unique in the United States because the equipment will be housed in a nuclear facility and dedicated to the examination of irradiated fuels and materials.

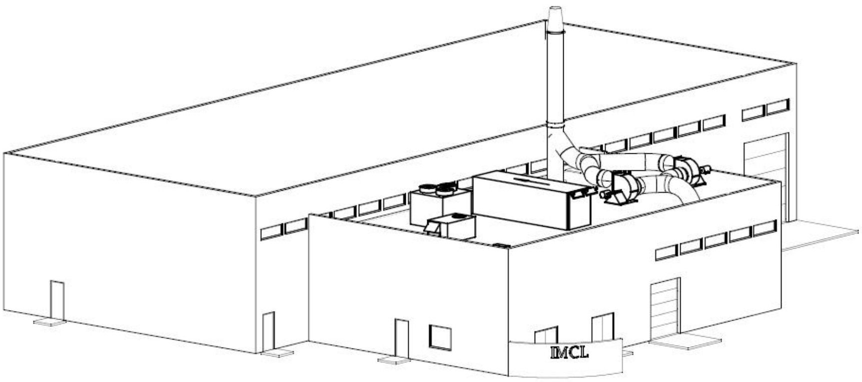

FIG. 6.1. Irradiated Materials Characterization Laboratory.

Designed as a multipurpose facility suitable for many different missions over its projected 40-year life, the IMCL will have as its first mission the task of housing modern, state-of-the-art PIE instrumentation. The IMCL will be used to routinely perform micro- and nano-scale characterization of material specimens and irradiated fuel samples in the mass range of tens of grams down to micro-grams. The facility will also be designed to allow easy routine maintenance of the instruments. The initial suite of equipment planned for installation into IMCL includes:

(a) Electron probe micro analyzer (EPMA)
(b) Focused ion beam (FIB)
(c) Micro-X-ray diffractometer (MXRD)
(d) Field effect gun-scanning transmission electron microscope (FEG-STEM)
(e) Mechanical testing
(f) Sample preparation

6.2 Advanced Post-Irradiation Examination Facility

Although the IMCL represents a significant advancement over current U.S. nuclear energy R&D capabilities, DOE has approved the mission to significantly expand the PIE and full-spectrum nuclear research capabilities of the United States through various alternatives, primary among them is a new multi-program facility called the Advanced Post-Irradiation Examination capability (see Fig. 6.2). This capability will handle considerably more sample and examination equipment than current facilities, both in types and quantities. The individual equipment and capabilities added will be based on an evaluation of need benefit and cost. As the project matures and the anticipated facility is built, some of the capability demonstrated in IMCL will transition to the new facility, freeing up IMCL to take on a key role of developing and deploying the next generation of "state-of-the-art" examination equipment. This would be consistent with the useful lifetime of such research equipment and would provide the newer facility with demonstrated state-of-the-art instrumentation.

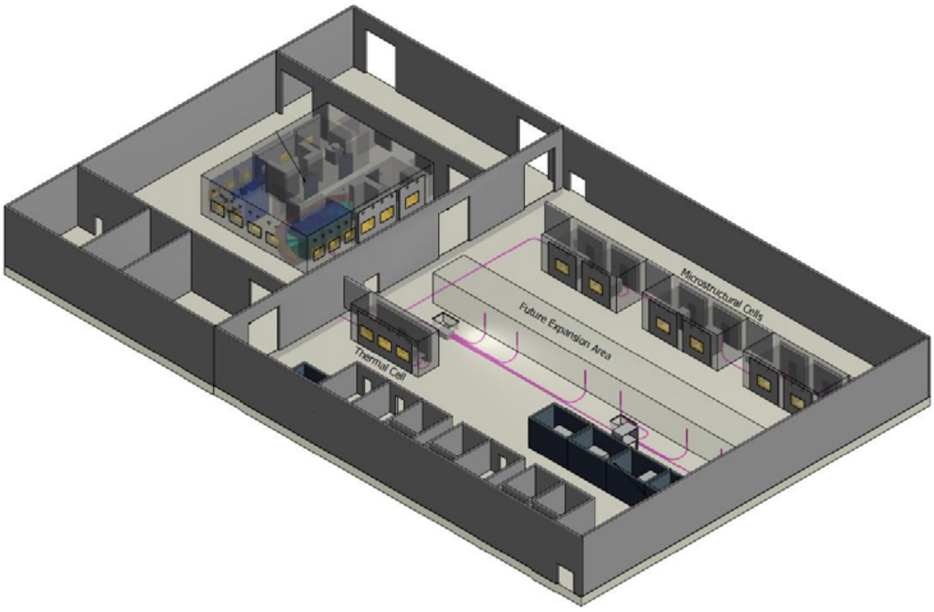

FIG. 6.2. Advanced post-irradiation examination.

The process for achieving advanced PIE capabilities in the United States, inclusive of standing up the Advanced Post Irradiation Examination capability, recently marked a major milestone with the approval of the Mission Need Statement in January 2011. The Advanced Post-Irradiation Examination facility alternative is anticipated to be a modular design to facilitate equipment specific shielding and flexibility for future equipment development, configuration alteration, and ease of replacement. The initial suite of equipment anticipated for installation includes:

(a) Cask receipt
(b) Comprehensive sample preparation
(c) Expanded mechanical testing
(d) Focused ion beam (FIB)
(e) High temperature X-ray diffractometer (HT-XRD)
(f) Local electrode atom probe (LEAP)
(g) Secondary ion mass spectrometer (SIMS)
(h) Full suite of thermal property characterization: laser flash thermal diffusimeter (LFD), STDM, differential scanning calorimeter (DSC), thermogravimetry (TGA)
(i) Knudsen cell
(j) Auger electron microprobe (AEM)
(k) Positron annihilation spectrometer (PAS)
(l) Advanced transmission electron microscope (TEM)
(m) Atomic force microscope (AFM)
(n) Laser resonant ultrasound spectrometer (LRUS)
(o) Nuclear magnetic resonance using Raman spectroscopy
(p) Anklylography

7. PARTNERSHIPS/ATR-NSUF [8]

In addition to increasing physical capabilities through equipment and facilities, INL is expanding its partnerships and collaborations. Recently the ART-NSUF added ORNL and the University of California Berkeley as facility partners. Such partnerships increase access to national irradiation and testing capabilities and provide greater flexibility to respond to user needs.

The new partnerships will make ORNL's High Flux Isotope Reactor (HFIR) and its associated capabilities available to the ATR-NSUF users. The HFIR is a versatile, 85-megawatt isotope and test reactor that provides one of the highest steady-state neutron fluxes of any reactor in the world. Irradiation experiment facilities include a wide variety of test positions, a hydraulic shuttle, and the capability for multiple instrumented target positions. Target fabrication, hot cell facilities for the examination of nuclear fuels and irradiated materials, the Radiochemical Engineering Development Center, and a set of special radiological laboratories at ORNL will also join the partnership. HFIR is operated by DOE, Basic Energy Sciences.

University of California Berkeley will bring several capabilities for examining irradiated material samples. Its facilities include a nano-indentation system for nano- and microscale hardness testing at ambient and elevated temperature and inert environments, positron annihilation spectroscopy, and warm sample preparation.

8. CONCLUSIONS [3]

The DOE’s ability to build a technical foundation to sustain the long-term contribution of nuclear generation to meet U.S. energy needs will be significantly hampered without obtaining a fundamental understanding of irradiation behavior at the nano-scale and below. All significant advances in nuclear energy technology will rely on the development of improved performance of fuels and more durable materials. Development of improved fuels and materials relies on the fundamental understanding of the effects of irradiation, which in turn relies on a program that couples experimental investigation with modeling and simulation. Current PIE capability does not provide the information required to obtain the fundamental understanding to continue to advance nuclear technology as an economical and sustainable energy source.

For this reason, INL has embarked on a significant effort to expand its already extensive capability to perform PIE through the addition of physical capabilities to the addition of partnerships. These capabilities will be expanding with the installation of state-of-the-art nano- and atomic-scale characterization equipment and the addition of two new facilities, IMCL and Advanced Post Irradiation Examination Facility, which will follow a reconfigurable and flexible design plan to ensure compatibility for performing advanced PIE on highly activated fuels and materials along with flexibility for future capabilities.

The INL incorporates all the necessary elements for successfully performing PIE and meeting the research needs to enable the advancement of nuclear energy. It allows parties interested in the advancement of scientific knowledge access to the powerful and versatile irradiation capabilities of the ATR and provides them with the diverse equipment and methods needed to analyze their experiment after irradiation. The quality of the program will only improve with time as the INL adds equipment and facilities to what is already available.

REFERENCES

[1] UNITED STATES DEPARTMENT OF ENERGY OFFICE OF NUCLEAR ENERGY, Nuclear Energy Research and Development Roadmap, Report to Congress (2010). http://nuclear.energy.gov/pdfFiles/NuclearEnergy_Roadmap_Final.pdf

[2] IDAHO NATIONAL LABORATORY, Consolidated World-Class Post-Irradiation Examination Capabilities Strategic Plan, INL/EXT-09-16831, Idaho National Laboratory (2009).

[3] UNITED STATES DEPARTMENT OF ENERGY OFFICE OF NUCLEAR ENERGY, Mission Need Statement for Advanced Post-Irradiation Examination Capability: A Non-Major Systems Acquisition Project, U.S. Department of Energy (2011).

[4] CARMACK, J., Post-Irradiation Examination Capabilities at the Idaho National Laboratory, INL/MIS-11-21996, Idaho National Laboratory (2011).

[5] AUSTED, S., Conceptual Design Report for the Irradiated Materials Characterization Laboratory (IMCL), INL/EXT-10-17562, Idaho National Laboratory (2010).

[6] SCHULTHESS, J.L., DRAFT: National Post Irradiation Examination Workshop Report, INL/EXT-11-21922, Idaho National Laboratory (2011).

[7] LANDMAN, W.H., Jr., Preconceptual Design Report for Post Irradiation Examination Line Item building, INL/EXT-10-19923, Idaho National Laboratory (2010).

[8] IDAHO NATIONAL LABORATORY NEWS RELEASE, Two new partners join Advanced Test Reactor National Scientific User Facility (2011) https://inlportal.inl.gov/portal/server.pt?open=514&objID=1555&mode=2&featurestory=DA_574357.

[9] IDAHO NATIONAL LABORATORY, Ten-year Plan for Implementation of Advanced Post Irradiation Examination Capability at the Idaho National Laboratory (FY 2011 to FY 2020), INL/EXT-10-20022, Idaho National Laboratory (2010).

[10] ROSENBERG, K.E., ALLEN, T.R., HALEY, J.C., MEYER, M.D., National Science User Facility purpose and capabilities, Proceedings of HOTLAB 2010 Conference, Dimitrovgrad (2010) Session 5, Paper 34.