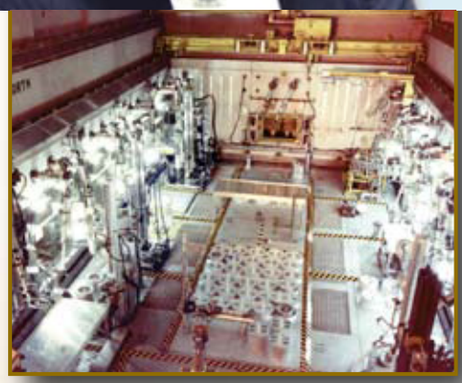
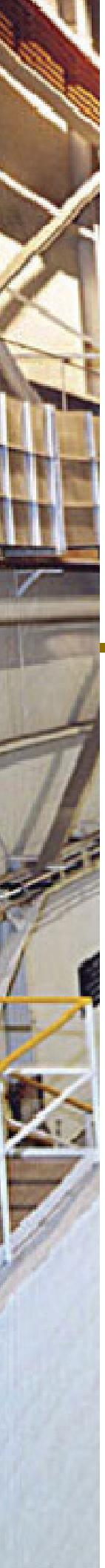
# ATR
## National Scientific User Facility



# 2009 Annual Report



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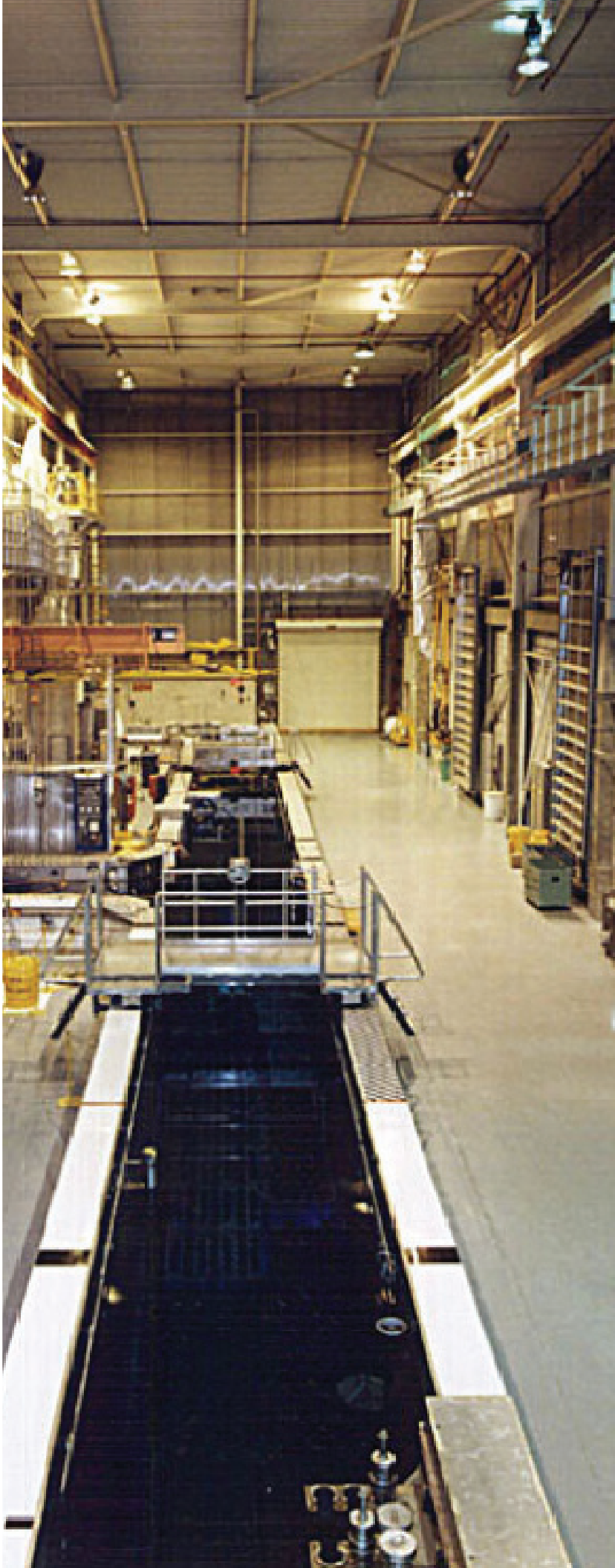

# Advanced Test Reactor National Scientific User Facility (ATR NSUF) FY 2009 Annual Report

Todd Allen, Scientific Director
Mary Catherine Thelen, Editor

November 2010

ATR National Scientific User Facility
995 University Boulevard
Idaho Falls, ID 83401-3550

For the most up-to-date information, visit the ATR-NSUF website at http://www.atrnsuf.inl.gov
A copy of this report is available in pdf format at http://www.atrnsuf.inl.gov

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*Dr. David Hill, Deputy Laboratory Director for Science and Technology, Idaho National Laboratory*

# Welcome from Idaho National Laboratory

As the national nuclear laboratory for the U.S. Department of Energy Office of Nuclear Energy (DOE-NE), Idaho National Laboratory (INL) has a significant responsibility to both lead and support nuclear energy research efforts. INL's responsibilities include coordinating and participating in research activities as well as providing its nuclear energy research infrastructure as a shared resource for the global nuclear energy enterprise.

At INL, our nuclear energy research capabilities are centered around the Advanced Test Reactor, a highly flexible materials test reactor that has successfully served the fuel and materials irradiation testing needs of the nation for decades. The Advanced Test Reactor is the only U.S. research reactor capable of providing large-volume, high-flux neutron irradiation in a prototypic reactor environment. In 2007, DOE designated the Advanced Test Reactor, in conjunction with INL's post-irradiation examination capabilities, a National Scientific User Facility. The designation was the first step in transforming the reactor's role to include collaborative research, which is needed to meet DOE-NE needs.

As the Advanced Test Reactor National Scientific User Facility program grows and expands, INL is committed to ongoing infrastructure upgrades and investments that will ensure that world-class researchers have access to truly world-class facilities.

In the past five years alone, INL has already demonstrated its commitment by investing more than $50 million in new research capabilities. These, as well as ongoing and future investments, will provide:

- Significant improvement in fabrication, characterization, testing and post-irradiation examination of nuclear fuels and materials
- Basic scientific understanding of fabrication processes and irradiation performance of fuels and materials at the microstructural level, which is needed to support development and deployment of high-performance fuels
- Improved ability to conduct research, develop and demonstrate advanced separation technologies, from an understanding of the fundamental science to integrated laboratory testing and planning for engineering-scale demonstration
- New reactor and fuel-cycle technologies that meet U.S. goals for improved economics, reduced waste intensity, improved proliferation-resistance and sustainability.

The research conducted through the Advanced Test Reactor National Scientific User Facility reflects INL's commitment to DOE-NE's mission — to meet the nation's nuclear energy research challenges by building on existing relationships, capabilities and underlying infrastructure.

*Dr. Todd Allen, ATR NSUF Scientific Director and professor of engineering physics at the University of Wisconsin-Madison*

## Welcome to ATR NSUF

The Advanced Test Reactor National Scientific User Facility (ATR NSUF) is a prototype for the laboratory of the future, where resources are shared among universities and national laboratories and collaborative research prepares a new generation of nuclear energy professionals. Research conducted through ATR NSUF supports DOE-NE in its efforts to advance research in nuclear technology, with specific focus on nuclear fuels as well as on materials to help extend the lifetime of structural components in nuclear systems.

ATR NSUF, in collaboration with affiliated partners, offers researchers no-cost access to world-class facilities with capabilities in reactor testing, post-irradiation examinations and beamline experiments. To facilitate this, the program has an open "rolling" solicitation for proposals with evaluations conducted twice each year and also accepts proposals for rapid turnaround experiments.

Since the first call for research in 2008, the program has funded 27 projects with approximately 81 participating researchers from 21 institutions. Fourteen of these projects are highlighted in this FY 2009 Annual Report. In some cases, the scientific results are still preliminary because research projects like these often require multiple years to complete. For example, some irradiated experiments are still awaiting post-irradiation examination. In others, post-irradiation examination has been completed but the research is awaiting analysis and publication. In all cases, however, the results will be published for use by other nuclear researchers.

Though we are still at a defining moment in this young program, we hope that our first annual report will help researchers understand how the program is growing and developing. We hope that it will help stimulate conversation and inspire proposals for new research or new capabilities that will support our nation's energy security needs.

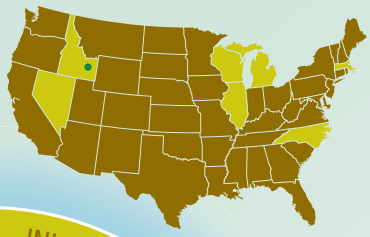
Even at this young stage in our program's growth, we have seen the strength of the ATR NSUF concept clearly demonstrated with ever increasing participation. In coming years, we anticipate our greatest strengths will be the breadth of our research achievements, publications and productivity toward meeting DOE-NE's research goals.

### Inside This Report

This inaugural report is a snapshot in time and therefore describes activities that occurred in FY 2009.

However, due to the late publication of this report, we decided some sections warranted a broader perspective of the program. For instance, the Call for Proposals section includes information on Rapid-Turnaround Experiments, which were not initiated until FY 2010. Also, the Users Week section provides a combined overview of all Users Weeks starting in FY 2008. Other examples can be found throughout the report.

We hope that including this additional information helps you see the tremendous growth the program has undergone since its inception.



capabilities at-a-glance

# Overview

## ATR NSUF — A New Model for Collaborative Research

ATR NSUF and its partner facilities represent a prototype laboratory for the future, a model that supports the DOE-NE mission by building collaborative research projects and sharing resources and capabilities. Through ATR NSUF, university researchers and their collaborators are building on current knowledge to better understand the complex behavior of materials in the radiation environment of a nuclear reactor.

The ATR NSUF is a distributed partnership with significant resources located at Idaho National Laboratory and complementary resources at partner institutions across the U.S. Within this distributed partnership are capabilities for performing reactor irradiations and post-irradiation examinations. These capabilities in conjunction with other national user facility partnerships provide a nation-wide infrastructure that allows the best ideas to be proven using the most advanced capabilities.

In FY 2009, ATR NSUF partners included the INL and six universities:

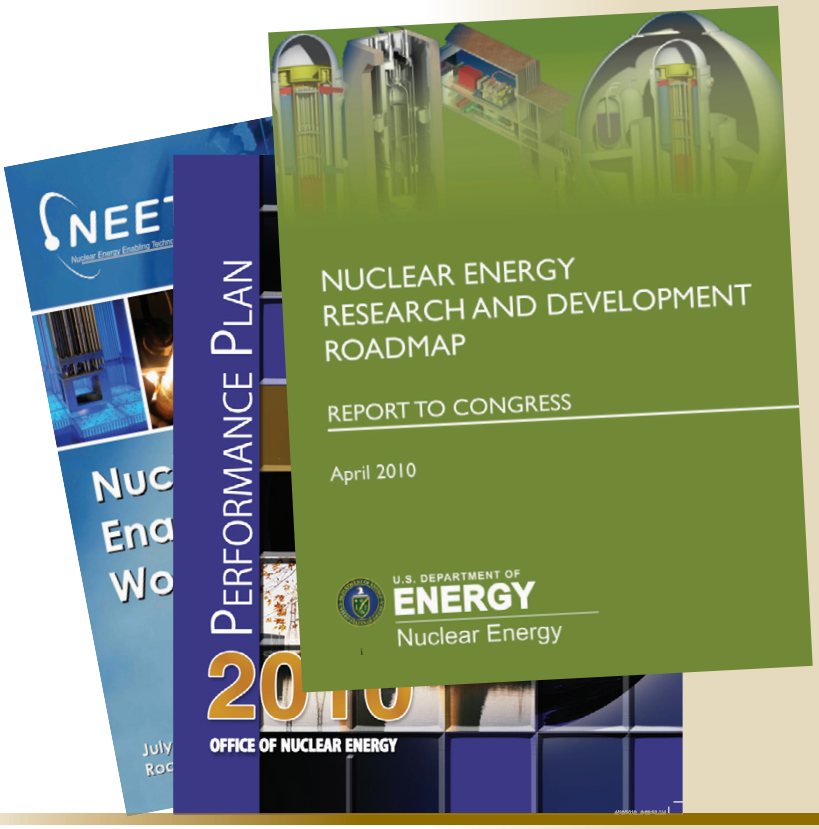
- Illinois Institute of Technology (IIT)
- Massachusetts Institute of Technology (MIT)
- North Carolina State University (NC State)
- University of Michigan (U–M)
- University of Nevada, Las Vegas (UNLV)
- University of Wisconsin–Madison (UW–M).

The number of partners continues to grow, offering researchers even broader opportunities and access to research capabilities in coming years.

*ATR NSUF offers university researchers and students a variety of opportunities for networking and collaborating, bringing the best minds together to research nuclear energy challenges.*

*Recent DOE-NE documents are useful guides for understanding the direction of nuclear energy research:*

- *Nuclear Energy Research and Development Roadmap: Report to Congress — a plan for research, development and demonstration activities that will ensure nuclear energy remains viable energy option for the U.S.*
- *Nuclear Energy Enabling Technologies (NEET) Workshop Report — a report of the recent meeting with stakeholders to obtain their input on the crosscutting technology needed to support NE roadmap objectives and to inform a possible solicitation for transformative, "out-of-the-box" solutions across the full range of nuclear energy technology issues*
- *2010 Performance Plan — a review of FY 2009 performance and FY 2010 objectives in critical program areas with an overview of NE programs, funding profile and designated role within the DOE Strategic Plan.*




## Supporting the DOE-NE Roadmap

DOE-NE organizes its research and development activities based on four main objectives that address challenges to expanding the use of nuclear power:

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

ATR NSUF provides access to many of DOE-NE's primary research assets, where researchers can gain access to government-owned test reactors, beamlines, a broad array of post-irradiation examination facilities, hot cells, specialty engineering facilities and small radiological laboratories. These are supplemented by university capabilities ranging from research reactors to materials science laboratories.

ATR NSUF research supports the DOE-NE roadmap in the following areas:

- Advanced structural materials
- Improved/advanced nuclear fuels
- Advanced instrumentation and controls
- Advanced computational modeling and simulation
- Stable waste forms.

**For more information**

Visit the DOE Office of Nuclear Energy webpage at **http://www.ne.doe.gov/**

The DOE-NE approach to meeting its objectives is described in the DOE-NE Research and Development Roadmap, available at **http://www.ne.doe.gov/pdfFiles/NuclearEnergy_Roadmap_Final.pdf**

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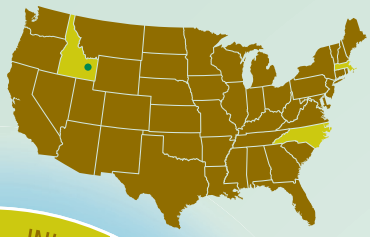


capabilities at-a-glance

# Reactor Capabilities

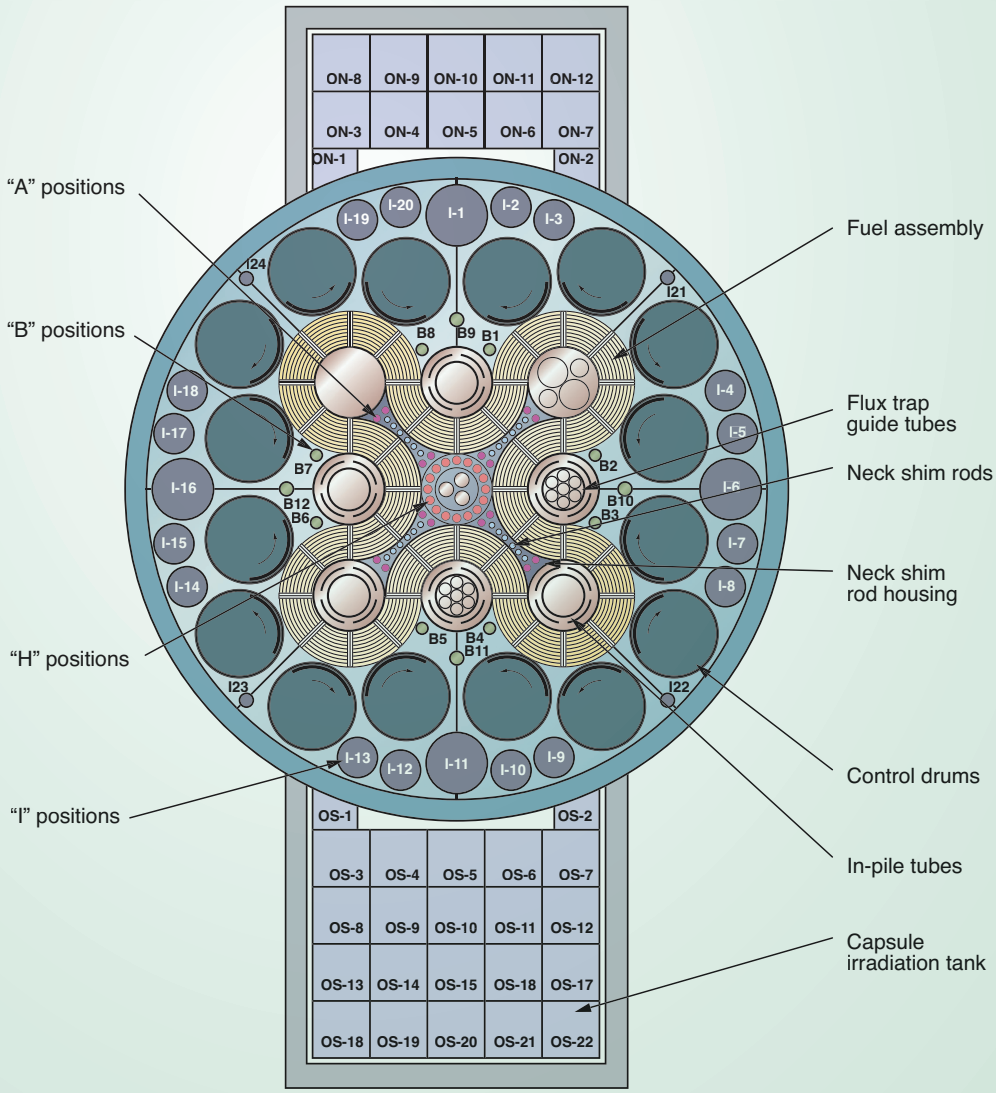
The ATR NSUF offers researchers access to the Advanced Test Reactor, which is located at the Reactor Technology Complex on the INL site.

The Advanced Test Reactor has been operating continuously since 1967, when it primarily served the U.S. Navy in the development and refinement of nuclear propulsion systems. In recent years, the reactor has been used for a wider variety of government and privately sponsored research and now serves a range of research and isotope production customers.

In FY 2009, the ATR NSUF also offered access to reactors at two university partner facilities: the Massachusetts Institute of Technology Reactor, which is the second largest research reactor at a U.S. university, and the PULSTAR reactor at North Carolina State University.

*The Advanced Test Reactor is essentially the nucleus of the ATR NSUF. The reactor's design exploits a unique serpentine core configuration that offers a large number of test positions (see the cross-section of the reactor core at right).*

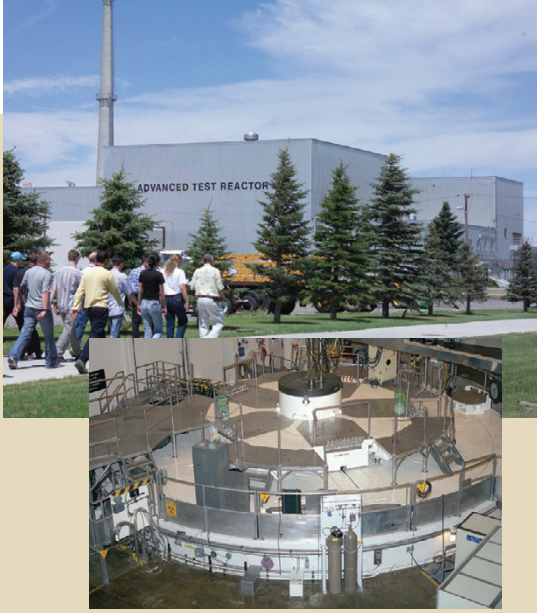
*Neutron flux in the Advanced Test Reactor varies from position to position and along the vertical length of the test position. It also varies with the power level in the lobe(s) closest to the irradiation position.*



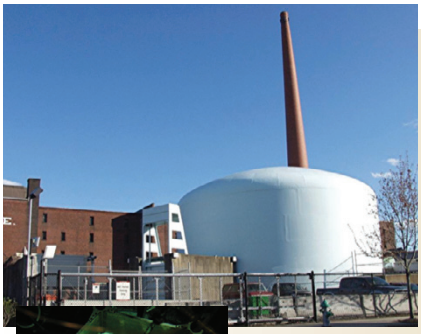
## Idaho National Laboratory: Advanced Test Reactor

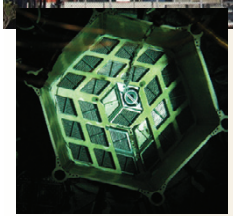
The Advanced Test Reactor is a water-cooled, high-flux test reactor, with a unique serpentine design that provides large power variations among its flux traps. The beryllium control cylinders contain hafnium plates, which can be rotated toward and away from the core, and hafnium shim rods, which withdraw vertically and can be individually inserted or withdrawn for minor power adjustments. The reactor's curved fuel arrangement places fuel closer on all sides of the flux trap positions than is possible in a rectangular grid. The reactor has nine of these high-intensity neutron flux traps and 68 additional irradiation positions inside the reactor core reflector tank, each of which can contain multiple experiments. There is a hydraulic shuttle irradiation system, which allows experiments to be inserted and removed during reactor operation, and pressurized water reactor (PWR) loops, which enable tests to be performed at prototypical PWR operating conditions.

**More information:** https://secure.inl.gov/atrproposal/documents/ATRUsersGuide.pdf




*(top) Advanced Test Reactor facility at the INL Site in Idaho (below) Aerial view of the Advanced Test Reactor head*





*(above) MIT reactor facility in Cambridge, Mass. (left) Annular fuel rig in the MIT reactor core*

## University Partner: Massachusetts Institute of Technology (MIT) Reactor

The MIT reactor is a 5 MW tank-type research reactor. It has three positions available for in-core fuel and materials experiments over a wide range of conditions. Water loops at pressurized water reactor/boiling water reactor (PWR/BWR) conditions, high-temperature gas reactor environments at temperatures up to 1400°C and fuel tests at light water reactor (LWR) temperatures have been operated and custom conditions can also be provided. A variety of instrumentation and support facilities are available. Fast and thermal neutron fluxes are up to $10^{14}$ and $5x10^{14}$ n/cm$^2$–s. The MITR has received approval from the Nuclear Regulatory Commission for a power increase to 6 MW which will enhance the neutron fluxes by 20%.

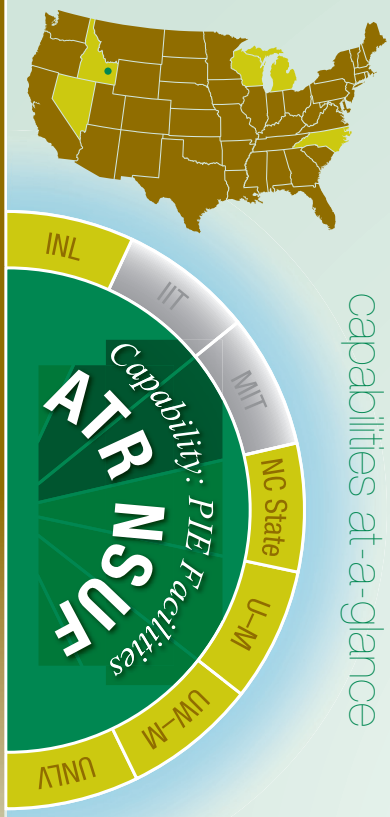
**More information:** https://secure.inl.gov/atrproposal/documents/MITR_UserGuide.pdf

## University Partner: North Carolina State University (NC State) PULSTAR Reactor

The PULSTAR reactor is a 1 MW pool-type nuclear research reactor located in NC State's Burlington Engineering Laboratories. The reactor, one of two PULSTAR reactors built and the only one still in operation, uses 4% enriched, pin-type fuel consisting of uranium dioxide pellets in zircaloy cladding. The fuel provides response characteristics that are very similar to commercial light water power reactors. These characteristics allow teaching experiments to measure moderator temperature and power reactivity coefficients including doppler feedback. In 2007, the PULSTAR reactor produced the most intense low-energy positron beam with the highest positron rate of any comparable facility worldwide.

**More information:** https://secure.inl.gov/atrproposal/documents/PULSTARReactor.pdf

*(above) PULSTAR reactor facility on the NC State North Campus in Raleigh, N.C. (right) View downward toward the PULSTAR reactor pool*

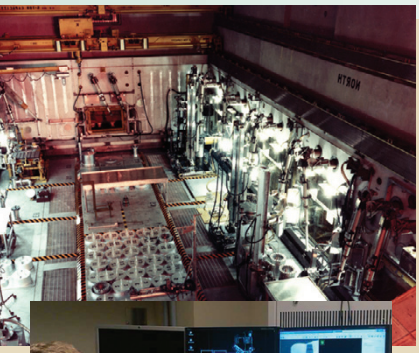



capabilities at-a-glance

# Post-Irradiation Examination Capabilities

The ATR NSUF offers researchers access to a broad range of post-irradiation examination facilities.

In FY 2009, the ATR NSUF program offered researchers access to capabilities at INL's Materials and Fuels Complex as well as at four university partner facilities. These include the Nuclear Services Laboratories at North Carolina State University; the Irradiated Materials Complex at University of Michigan; the Harry Reid Center Radiochemistry Laboratories at University of Nevada, Las Vegas; and the Characterization Laboratory for Irradiated Materials at University of Wisconsin-Madison.



*(above top) Hot Fuel Examination Facility, located at the Materials and Fuels Complex at DOE's INL Site in Idaho (below) A dual-beam Focused Ion Beam at the Center for Advanced Energy Studies (CAES)*

## INL: Hot Fuel Examination Facility, Analytical Laboratory, Electron Microscopy Laboratory

The Hot Fuel Examination Facility is a large alpha-gamma hot cell facility dedicated to remote examination of highly irradiated fuel and structural materials. Its capabilities include nondestructive examination, such as dimensional measurements and visual examination; and destructive examination, such as mechanical testing and metallographic/ceramographic characterization. The facility also offers a 250 $kW_{th}$ Training Research Isotope General Atomics (TRIGA) reactor used for neutron radiography to examine internal features of fuel elements and assemblies.

The Analytical Laboratory is dedicated to analytical chemistry of irradiated and radioactive materials. It offers NIST-traceable chemical and isotopic analysis of irradiated fuel and material via wide range of spectrometric techniques, including inductively coupled plasma mass and optical emission spectrometry (ICP-MS and ICP-OES), a dynamic reaction cell for inductively coupled plasma-mass spectrometry (ICPMS-DRC) and thermal ionization mass spectrometry (TIMS).

The Electron Microscopy Laboratory (EML) is dedicated to materials characterization, primarily using transmission electron, scanning electron and optical microscopy. The EML also houses a dual-beam Focused Ion Beam (FIB) that allows examination and small-sample preparation of radioactive materials for further atom probe, transmission electron microscopy (TEM) and micro-mechanical testing. Part of the laboratory is dedicated to sample preparation, providing researchers with support, equipment, safety systems and procedures to prepare samples of diverse materials for analysis.

**More information:** https://secure.inl.gov/atrproposal/documents/PIECapabilitiesGuide.pdf

## University Partner: North Carolina State University (NC State) Nuclear Services Laboratories

Post-irradiation examination capabilities at NC State's Nuclear Services laboratories include neutron activation analysis, radiography and imaging capabilities and positron spectrometry.

**More information:** https://secure.inl.gov/atrproposal/documents/PULSTARReactor.pdf

*The Positronium Annihilation Lifetime Spectrometer, located in the PULSTAR reactor facility on the NC State North Campus in Raleigh, N.C.*

*Capabilities at the Irradiated Materials Complex on the U-M campus in Ann Arbor, Mich.*

## University Partner: University of Michigan (U-M) Irradiated Materials Complex

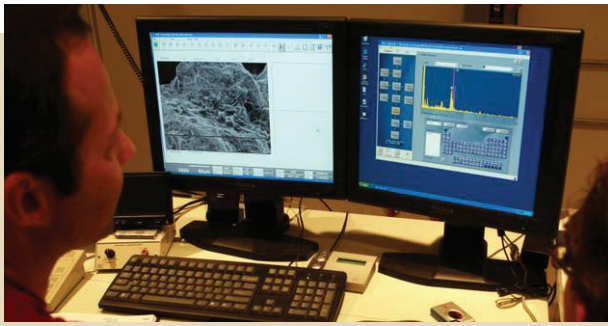
The Irradiated Materials Complex provides capabilities (laboratories and hot cells) for conducting high-temperature mechanical properties, and corrosion and stress corrosion cracking experiments on neutron irradiated materials in an aqueous environment, including supercritical water, and for characterizing the fracture surfaces after failure.

**More information:** https://secure.inl.gov/atrproposal/documents/UniversityofMichiganIMCandMIBLFacilities.pdf

## University Partner: University of Nevada, Las Vegas (UNLV) Harry Reid Center Radiochemistry Laboratories

Post-irradiation examination capabilities at the Radiochemistry Laboratories include metallographic microscopy, x-ray powder diffraction, Rietveld analysis, scanning electron and transmission electron microscopy, electron probe microanalysis and x-ray fluorescence spectrometry.

**More information:** https://secure.inl.gov/atrproposal/documents/UNLVPartnerFacilityUserGuide.pdf



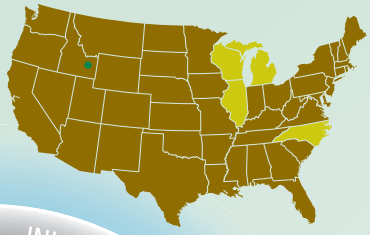
*Post-irradiation examination capabilities at the Harry Reid Center Radiochemistry Laboratories, located on the UNLV campus in Las Vegas, Nev.*

*A JEOL 200CX TEM equipped with EDS and scanning system, and an electropolisher and dimpler at the Characterization Laboratory for Irradiated Materials, located on the UW-M campus in Madison, Wis.*

## University Partner: University of Wisconsin–Madison (UW-M) Characterization Laboratory for Irradiated Materials

The Characterization Laboratory for Irradiated Materials offers post-irradiation examination capabilities including scanning electron and transmission electron microscopy on neutron-irradiated materials.

**More information:** https://secure.inl.gov/atrproposal/documents/UniversityofWisconsinCLIMGuide.pdf



capabilities at-a-glance

# Beamline Capabilities

The ATR NSUF offers researchers access to a broad range of facilities with beamlines, including accelerator facilities for radiation damage experiments, synchrotron radiation studies, neutron diffraction and imaging, as well as positron and neutron activation analysis.

In FY 2009, the ATR NSUF program offered researchers access to four university partner facilities. These include the Illinois Institute of Technology Materials Research Collaborative Access Team (MRCAT) beamline at Argonne's Advanced Photon Source; the PULSTAR reactor facility at North Carolina State University; the University of Michigan Ion Beam Laboratory; and the University of Wisconsin-Madison Tandem Accelerator Ion Beam.

*Aerial view of the Advanced Photon Source at Argonne National Laboratory, located in Argonne, Ill.*

**University Partner: Illinois Institute of Technology (IIT) Beamline at Argonne National Laboratory's Advanced Photon Source**

The Materials Research Collaborative Access Team (MRCAT) beamline offers a wide array of synchrotron radiation experiment capabilities, including x-ray diffraction, x-ray absorption, x-ray fluorescence and 5 µm spot size fluorescence microscopy.

**More information:** https://secure.inl.gov/atrproposal/documents/AdvancedPhotonSource.pdf

## University Partner: North Carolina State University (NC State) PULSTAR Reactor Facility

The PULSTAR reactor facility offers a selection of dedicated irradiation beam port facilities — neutron powder diffraction, neutron imaging, intense positron source and ultra-cold neutron source. An intense positron source has been developed to supply a high rate positron beam to two different positron/positronium annihilation lifetime spectrometers.

**More information:** https://secure.inl.gov/atrproposal/documents/PULSTARReactor.pdf

*Positron beam cave containing magnetic switchyards and transport solenoids, located in the PULSTAR reactor facility on the NC State North Campus in Raleigh, N.C.*

## University Partner: University of Michigan (U-M) Michigan Ion Beam Laboratory

The 1.7 MV Tandetron accelerator in the Michigan Ion Beam Laboratory offers controlled temperature proton irradiation capabilities with energies up to 3.4 MeV as well as heavy ion irradiation.

**More information:** https://secure.inl.gov/atrproposal/documents/UniversityofMichiganIMCandMIBLFacilities.pdf

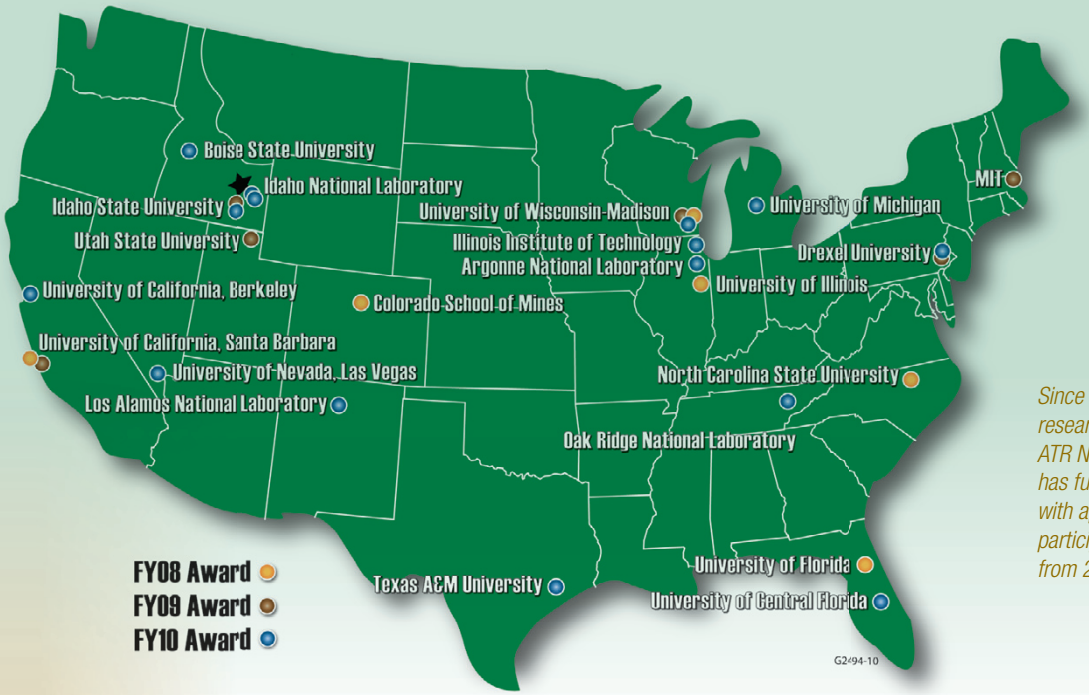
*Michigan Ion Beam Laboratory for Surface Modification and Analysis, located on the U-M campus in Ann Arbor, Mich.*

## University Partner: University of Wisconsin–Madison (UW-M) Tandem Accelerator Ion Beam

A 1.7 MV terminal voltage tandem ion accelerator (Model 5SDH-4, National Electrostatics Corporation Pelletron accelerator) installed at UW-M features dual ion sources for producing negative ions with a sputtering source or using a radio frequency (RF) plasma source. The analysis beamline is capable of elastic recoil detection and nuclear reaction analysis.

**More information:** https://secure.inl.gov/atrproposal/documents/UniversityofWisconsinCLIMGuide.pdf

*Tandem Ion Beam Accelerator, located on the UW-M campus in Madison, Wis.*




*Since the first call for research in 2008, the ATR NSUF program has funded 27 projects with approximately 81 participating researchers from 21 institutions.*

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# *ATR NSUF — Calls for Proposals*

The ATR NSUF mission is to provide nuclear energy researchers access to world-class capabilities to facilitate the advancement of nuclear science and technology. This mission is supported by providing cost-free access to state-of-the-art experimental irradiation testing and post-irradiation examination facilities as well as technical assistance in design and analysis of reactor experiments. Access is granted through a competitive proposal process.

ATR NSUF offers three research proposal options (described in more detail below) through a user-friendly online submittal system that helps prospective researchers develop, edit, review and submit their proposals. ATR NSUF staff is available to help any researcher who desires to submit a proposal.

Submitted proposals should be consistent with the DOE-NE mission and its programmatic interests. These include the Light Water Reactor Sustainability, Fuel Cycle Research and Development, Advanced Modeling and Simulation, Next Generation Nuclear Plant and the Generation IV Nuclear Energy Systems Initiative programs.

All proposals are subject to a peer-review process before selection. An accredited U.S. university or college must lead research proposals for irradiation/post-irradiation experiments. Collaborations with other national laboratories, federal agencies, non-U.S. universities and industries are encouraged. Any U.S.-based entities, including universities, national laboratories and industry can propose research that would utilize the Materials Research Collaborative Access Team (MRCAT) beamline at the Advanced Photon Source or would be conducted as a Rapid-Turnaround Experiment.

## Open Calls

### Irradiation, Post-Irradiation Examination, Critical Facility and Synchrotron Radiation Experiments

The ATR NSUF annually conducts two open calls for proposals: a fall call, which opens in the spring and closes in October, and a spring call, which opens in late

October and closes in late March or early April. Proposals are accepted for:

- Irradiation/post-irradiation examination of materials or fuels
- Post-irradiation examination of previously irradiated materials or fuels from the ATR NSUF sample library (described below)
- Research that requires the unique capabilities of the Advanced Photon Source through the MRCAT beamline, operated by the Illinois Institute of Technology.

All proposals submitted to the open calls undergo thorough reviews for feasibility, technical merit, relevance to DOE-NE missions and cost. The results are compiled and provided to a panel committee who performs a final review and ranks the proposals. The ranking is given to the ATR NSUF director. Awards are announced within two to three months of the call's closing date, generally in January and June. Awards allow users cost-free access to specific ATR NSUF and partner capabilities as determined by the program.

## Other Calls

### Rapid-Turnaround Experiments

Rapid-Turnaround Experiments are experiments that can be performed quickly — in two months or less — and include, but are not limited to, post-irradiation examination, ion beam irradiation and neutron scattering experiments. Proposals for Rapid-Turnaround Experiments are reviewed within a month of submittal and awarded based on the following rankings:

- High Priority — Proposal is awarded immediately upon review if funding is available
- Recommended — Proposal is placed in a queue from which awards are made approximately every other month if funding is available
- Not Recommended — Proposal is not awarded, but the project investigators are offered an opportunity to read the review comments and then resubmit.

### New-User Experiments

University or college researchers can gain experience in the intricacies of designing and conducting an in-reactor test by submitting a letter of interest through the Call for Proposals web page. A New-User Experiment is initiated when three to five universities have expressed an interest. Each experimenter will have an opportunity to work with a variety of INL staff to design a capsule to meet an experiment's needs. The project ends with the samples inserted into the reactor.

For additional information about all call types, visit the ATR NSUF online webpage for proposals at https://secure.inl.gov/atrproposal/Common/UserHome.aspx

## ATR NSUF Sample Library

In 2009, the ATR NSUF established a sample library as an additional pathway for research. The library contains irradiated and non-irradiated samples in a wide range of material types, from steel samples irradiated in fast reactors to ceramic materials irradiated in the Advanced Test Reactor. Many samples are from previous DOE-funded material and fuel development programs. University researchers can propose to analyze these samples in a post-irradiation examination (PIE)-only experiment. Samples from the library may be used for proposals for open calls and Rapid-Turnaround Experiments.

As the ATR NSUF program continues to grow, so will the sample library. To review an online list of available specimens, visit https://secure.inl.gov/atrproposal/Common/UserHome

**For more information**

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*Some of the participants at the FY 2009 Users Week gather for a group photo.*

# *ATR NSUF Users Week*

The annual ATR NSUF Users Week offers researchers five full days of workshops, tours, discussions and classes. The focus is on providing an understanding of key nuclear technology gaps, capabilities required for addressing those gaps, recent or emerging advances, and techniques for conducting reactor experiments and post-irradiation examination.

*An ATR NSUF Users Week participant looks into the Hot Fuel Examination Facility during a tour of INL's post-irradiation examination facilities at the Material and Fuels Complex.*

Users Week is not just a way to learn more about ATR NSUF, its capabilities and ongoing research, it is also a great opportunity to meet other students, scientists and engineers who are interested in responding to the ATR NSUF's call for proposals. Users Week supports ATR NSUF as a model for the laboratory of the future, where collaborative research and shared resources among universities and national laboratories will help prepare a new generation of nuclear energy professionals.

The week's events are free of charge for students, faculty and post-docs as well as researchers from industry and national laboratories who are interested in materials, fuels, post-irradiation examination and reactor-based technology development. In the three years since its inception, ATR NSUF Users Week has hosted 326 participants from 29 countries and 32 U.S. universities.

Scholarships to help defray travel, hotel and meal expenses are offered to university faculty and students on a competitive basis.

*"We want to encourage people to become nuclear engineers and scientists and get back to the high numbers of graduates we had in the 1970s. Without [a new generation of nuclear scientists], there will be no nuclear renaissance in the United States."*

— Dennis Miotla, DOE-Office of Nuclear Energy Deputy Assistant Secretary for Nuclear Power Deployment, speaking at the second annual ATR NSUF User's Week dinner

*Two researchers listen intently during a Users Week Research Forum.*

## What To Expect at Users Week

Users Week kicks off with an introductory workshop to ATR NSUF, which includes a description of current and upcoming research capabilities offered by INL and university partners, a briefing on the solicitation process and opportunities within the education program, and a welcome from DOE, usually delivered by an official from DOE headquarters.

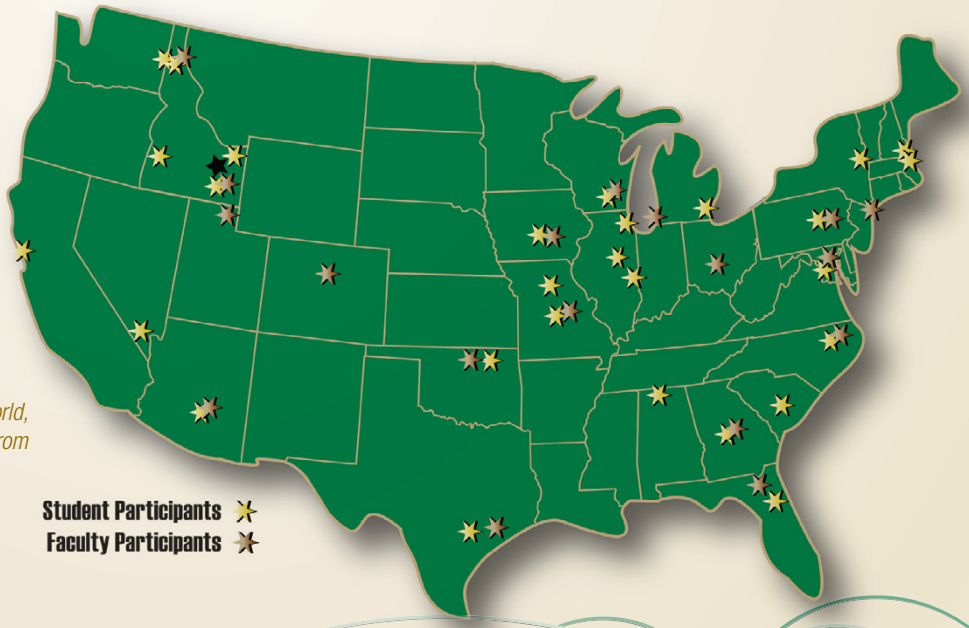
Each year, there are a variety of workshops and courses, which may vary from year to year. The courses focus on a variety of topic-specific areas, such as in-reactor instrumentation, fuels and materials, or how to conduct radiation experiments.

Participants are always offered an opportunity to tour the Advanced Test Reactor as well as INL's Materials and Fuels Complex where many post-irradiation examination facilities are housed.

An annual research forum was initiated in 2009. The forum offers an opportunity for university, industry and national laboratory researchers to present their latest findings in a collaborative environment. The forum's primary focus will be on ATR NSUF-awarded research experiments as they progress.

**For more information**

http://atrnsuf.inl.gov/Users/UsersWeek/tabid/164/Default.aspx

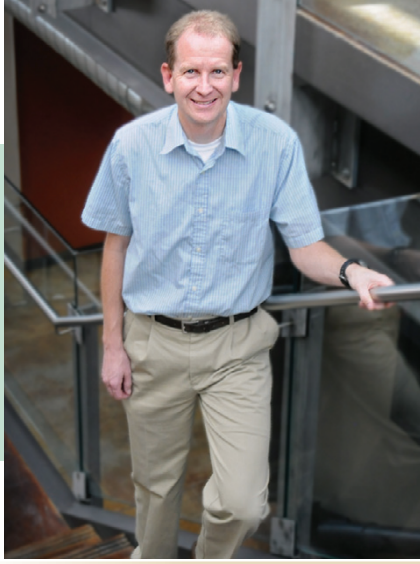



*Since its inception in 2007, ATR NSUF Users Weeks has welcomed more than 326 participants from around the world, including faculty and students from 32 universities across the U.S.*

*User's Week provides researchers and students with opportunities for learning, networking and collaborating with others who share an interest in nuclear researcher.*

*"The big outcome [for two of the students I worked with on ATR NSUF experiments] was the contacts they made working at the ATR NSUF. After working at the User Facility, they came back and were hired."*

— Dr. Jeff King, the lead for a faculty-student research team from Colorado School of Mines and the Missouri University of Science and Technology



**For more information**

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# Additional Educational Programs and Opportunities

## Faculty/Student Research Teams

This unique research opportunity provides faculty and students with a chance to spend part of a summer performing research in collaboration with an INL scientist or engineer. Projects are selected (depending on funding availability) through a special call for proposals, which is openly advertised and posted each fall on the ATR NSUF website.

Proposals are accepted for scientifically meritorious projects that result in increased research capability for the ATR NSUF. Specific areas of interest include:

- Ramp testing of fuel
- Instrumentation test capsule design
- In-canal measurements
- Integrated computational modeling for analysis of irradiation experiments
- In-reactor ultrasonic measurement
- Analysis of materials using advanced techniques.

## Proposals

Proposals should be designed to meet the following criteria:

- Project lead must be a faculty member from an accredited U.S. university
- Proposal must include at least two research participants, preferably graduate students
- Participants must commit to spend 10 to 12 weeks at the INL Site, preferably during the summer
- Mutual agreement about the project must be reached by the faculty member and assigned INL researcher prior to arrival.

Proposals are awarded in January and funded through a contract to the university faculty project lead.

## Graduate and Undergraduate Internships

Each year, a number of internships are offered through the INL intern program. These internships are designed to provide students real-life experience in science or engineering in a national laboratory setting and to introduce students to the issues and opportunities in nuclear operations, nuclear science and technology, materials and fuels research. Graduate students may also use an internship to conduct thesis or dissertation research.

## Learn More

Visit the ATR NSUF webpage to learn more about educational programs and opportunities at **http://atrnsuf.inl.gov/FacultyandStudents/Internships/tabid/75/Default.aspx**

*The ATR reactor vessel is a cylinder that is 12 feet in diameter, 36 feet in height and constructed of stainless steel. The reactor's core, which includes 40 fuel elements, is 4 feet in diameter and height.*

# *Key to Reading the Project Summaries*

Each ATR NSUF research project summarized in this report can be reviewed at a glance.

A green sidebar at the left side of each project summary highlights the DOE-NE research and development needs that the project addresses.

The U.S. map at the upper left of the summary highlights one of ATR NSUF's distinguishing characteristics — its broadly collaborative nature. For each project, the highlighted states represent the locations of those involved, including:

- ATR NSUF as well as any partner facilities
- Research team members — principal investigators, project engineers, students and other collaborators from universities and national laboratories from across the U.S. The locations of collaborators from industries or other nations are not highlighted on the map.

Below the map is an array that represents the ATR NSUF distributed partnership. It highlights the specific partners involved in the research project.

The table, located at the bottom left, includes important details about the project, including:

- ATR NSUF and partner capabilities utilized
- Materials or instrumentation addressed in the research as well as specific related information
- A list of team members and collaborators.

DOE-NE Need: Advanced Structural Materials / Pilot Project

Irradiation Test Plan for the ATR National Scientific User Facility (Pilot Project)

Kumar Sridharan, principal investigator (University of Wisconsin–Madison)
e-mail: kumar@engr.wisc.edu

Introduction

The safety and effectiveness of many proposed advanced nuclear systems will rely on the performance of the materials used for cladding, ducting and other structural components. Because these systems would generate higher temperatures and total radiation dosages than typically seen in light water reactors, it is essential to gain abetter understanding of the stability of the large number of materials or material systems that have been developed for high-temperature or high radiation environments.

This project was the pilot project for the ATR NSUF program. Not only was it instrumental in streamlining technical and programmatic management of irradiation and post-irradiation examination experiments, it paved the way for successful and efficient interaction between the ATR NSUF program and university users in the future.

Example

2 • ATR NSUF FY–2010 Annual Report

## Acronyms Used in This Report

| | |
|---|---|
| ATR | Advanced Test Reactor |
| ATR-C | Advanced Test Reactor-Critical facility |
| ATR NSUF | Advanced Test Reactor National Scientific User Facility |
| DOE-NE | U.S. Department of Energy Office of Nuclear Energy |
| dpa | displacements per atom |
| IIT | Illinois Institute of Technology |
| INL | Idaho National Laboratory |
| MIT | Massachusetts Institute of Technology |
| nm | nanometer |
| NC State | North Carolina State University |
| PIE | post-irradiation examination |
| PIE | post-irradiation examination |
| SEM | scanning electron microscopy |
| TEM | transmission electron microscopy |
| µm | micrometer |
| U-M | University of Michigan |
| UW-M | University of Wisconsin–Madison |
| UNLV | University of Nevada, Las Vegas |

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# Irradiation Test Plan for the ATR National Scientific User Facility (Pilot Project)

Kumar Sridharan, principal investigator (University of Wisconsin–Madison)
e-mail: kumar@engr.wisc.edu

## Introduction

The safety and effectiveness of many proposed advanced nuclear systems will rely on the performance of the materials used for cladding, ducting and other structural components. Because these systems would experience higher temperatures and total radiation dosages than typically seen in light water reactors, it is essential to gain a better understanding of the stability of the large number of materials or material systems that have been developed for high-temperature or high radiation environments.

This research program was the pilot project for the ATR NSUF program. Not only was it instrumental in streamlining technical and programmatic management of irradiation and post-irradiation examination (PIE) experiments, it paved the way for successful and efficient interaction between the ATR NSUF program and university users in the future.

**Table 1. Project Details — Irradiation Test Plan for the ATR NSUF (pilot project)**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| UW–M | PIE facilities |
| UNLV | PIE facilities |
| **Materials** | **Description** |
| Ferritic-martensitic steels<br>• HT-9 (from INL, ORNL and LANL)[a]<br>• T91<br>• NF616<br>• 9Cr nanostructured oxide-dispersion strengthened (ODS) steel (from JAEA)[a]<br>• grain boundary engineered HCM12A<br>• Fe-Cr binary alloys | All samples irradiated to 3 and 6 dpa at 300, 400, 500, 700°C |
| Austenitic alloys (from UW-M)<br>• 800H<br>• NF709<br>• D9<br>• Super 304H<br>• HT-UPS-AX-6 (developed at ORNL)[a] | |
| Fe-Cr-Mo-B metallic glasses (from UW-M) | |
| Ceramic materials<br>• SiC<br>• $ZrO_2$-MgO | |
| Metals and a refractory alloy<br>• W (from Westinghouse)[a]<br>• Ag (from Westinghouse)[a]<br>• ODS-Mo | |
| **Team Members/Collaborators[a]** | |
| • UW-M — Kumar Sridharan (principal investigator); Yong Yang, Jon McCarthy (co-investigators); Peng Xu (post-doctoral investigator); Alicia Certain, Kevin Field, Tyler Gerczak (graduate students); Shuhong Nie (visiting scientist)<br>• INL — Heather MacLean (principal investigator)<br>• University of Idaho, INL — Ram Prabhakaran (graduate student)<br>• U-M — Gary Was and George Jiao (collaborators); Janelle Warley (graduate student)<br>• Pennsylvania State University — Arthur Motta (collaborator); Cem Topbasi (graduate student)<br>• University of South Carolina — Djamel Kaoumi (collaborator) | |

a. Additional participants/collaborators included Oak Ridge National Laboratory (ORNL), including Shared Research Equipment (SHaRE) program; Los Alamos National Laboratory (LANL), including Los Alamos Neutron Science Center (LANSCE); Japan Atomic Energy Agency (JAEA); Westinghouse; Alabama A&M; University of California, Berkeley; and National Institute of Standards and Technology (NIST).

## Project Description

The objectives of this project are to:

- Perform neutron irradiations of a broad spectrum of structural materials relevant to present and future nuclear reactor systems, followed by structural characterization and mechanical property testing
- Contribute a large number of irradiated samples to the ATR NSUF sample library for use by research teams across the U.S.

More than 500 individual samples of advanced and model materials (Table 1) are currently undergoing irradiation and examination. These materials include:

- Ferritic/martensitic steels
- Austenitic alloys
- Five compositions of iron-chromium-molybdenum-boron (Fe-Cr-Mo-B) metallic glasses prepared by melt spinning
- Ceramic materials
- Pure metals and a refractory alloy, oxide dispersion strengthened (ODS)-Mo.

The geometries of the samples (Figure 1) will include:

- 3-mm disks (intended for transmission electron microscopy(TEM))
- Miniature 16-mm long tensile samples
- Rectangular SiC rods (used for indirect temperature measurements).

Irradiations are being performed at 300, 400, 500 and 700˚C to dose accumulations of 3 and 6 dpa. Post-irradiation analyses will be performed at UW-M, INL and UNLV as well as

at Oak Ridge National Laboratory (ORNL), which is not an affiliated ATR NSUF partner. Analyses will include high-resolution TEM, atom probe analysis, micro-diffraction, tensile testing and shear punch and micro-hardness tests. Select samples will be subjected to Small-Angle Neutron Scattering (SANS) analysis at National Institute of Standards and Technology (NIST) and Los Alamos National Laboratory.

## Accomplishments

The project began in January 2008 with bimonthly project planning meetings. In June, team members at UW-M began preparing, documenting and loading samples into irradiation capsules. In September, sample irradiations began. By May 2010, irradiation to a dose accumulation of 3 dpa was complete and planning for post-irradiation examination was well under way.

Activities pertaining to PIE work have begun at several locations. Electron microscopy equipment for characterizing irradiated samples has been put in place at the UW-M Characterization Lab for Irradiated Materials. Besides a TEM (JEOL 200CX) that was already available, a new SEM (JEOL 6610), electropolisher and dimpling machine and ion mill were procured and installed.

The mechanical properties of the irradiated samples will be evaluated at INL using an Instron tensile testing machine as well as a shear punch and microhardness testers, which have been calibrated and prepared. Structural characterization will be undertaken at ORNL, where a proposal funded by the SHaRE program has enabled access to a Phillips CM200 FEG-TEM/STEM and an Imago Scientific LEAP microscope.

By May 2011, a dose accumulation of 6 dpa is expected to be complete.

## Future Activities

The primary focus this year (FY 2011) will be PIE activities and analyses of the 3-dpa samples. Researchers will examine the irradiated alloy samples at INL by conducting tensile testing as well as shear punch and microhardness tests on a portion of the 3-mm disks. The remaining 3-mm disks will be divided into two batches and sent for structural analysis — high-resolution TEM and atom probe studies — at UW-M, UNLV and ORNL, but a new atom probe and FEG-STEM at INL will be utilized when they become available.

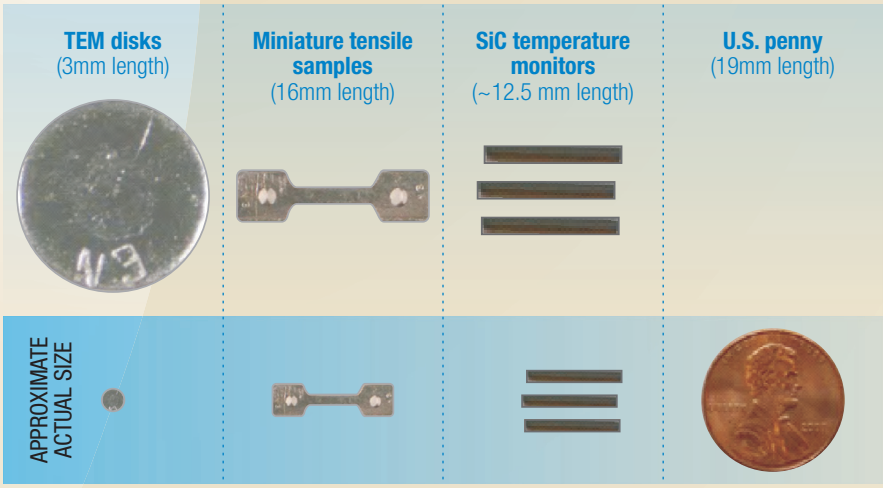
Many of the irradiated samples will be transferred to the ATR NSUF sample library for use by research teams across the U.S.

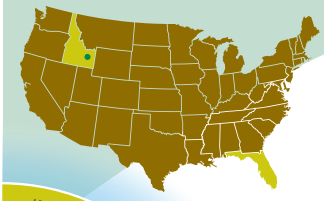
> *"The ATR NSUF program initiated by DOE is emblematic of the nuclear energy renaissance in the United States… we feel fortunate to be a part of the pilot project for this growing program. This project puts this team of researchers at the forefront of the DOE's goal of pairing unique national assets with key nuclear energy researchers."*
>
> — Kumar Sridharan, Ph.D., Fellow of American Society for Materials, Distinguished Research Professor, College of Engineering, University of Wisconsin–Madison

## Publications, Presentations and Patents

- "Getting Ready for Materials Testing in Advanced Test Reactor – A User's Perspective"; K. Sridharan, et al., ATR NSUF Workshop, Idaho National Laboratory, Idaho Falls, ID, 2008.
- "Pilot Project for Irradiation Testing of Materials at the ATR-National Scientific User Facility"; H.J. MacLean, et al., Trans. American Nucl. Society Annual Conference, Atlanta, GA, June 2009

*Figure 1. The geometries of 3- and 6-dpa irradiated samples — 3-mm TEM disks; miniature tensile samples and rectangular silicon-carbide temperature monitors — are shown enlarged (top) and at their approximate actual size (bottom). A U.S. penny is shown as a basis for comparison.*





project at-a-glance

# Irradiation of Potential Inert Matrix Materials

Juan Claudio Nino, principal investigator (University of Florida)
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## Introduction

Safe, ecologically friendly and economically sensible disposal of radiotoxic nuclear waste, such as weapons- and reactor-grade plutonium (Pu), neptunium (Np), americium (Am) and curium (Cm), is a priority for our national and international security. There is an increasing radiotoxic inventory of this type of nuclear waste from spent nuclear fuel and weapons programs.

One approach for decreasing the volume and hazards associated with this radiotoxic waste is transmutation in nuclear reactors, a process that converts the radioactive constituents of the waste into more stable elements. Transmutation of mixed oxide (MOX)-based fuels, however, leads to the generation of new transuranium actinides. A more promising alternative is using an inert matrix to burn Pu and other transuranic elements as an inert matrix fuel (IMF). This approach would generate less radioactive waste.

A recent Nuclear Energy Research Initiative (NERI) project at the University of Florida extended INL's work in developing magnesium oxide (MgO)-based ceramic-ceramic (cercer) inert matrix materials.[1] The university's researchers have developed and investigated the synthesis and thermophysical properties of magnesium oxide-neodymium zirconate cercer composites ($MgO\text{-}Nd_2Zr_2O_7$) and single-phase magnesium-based spinel compounds as potential inert matrix materials.[2] The next step for the researchers is to investigate the behavior of these materials under irradiation.

**Table 1. Project Details — Irradiation of Potential Inert Matrix Materials**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| **Materials** | **Description** |
| • $MgO{\cdot}1.5Al_2O_3$<br>• $MgAl_2O_4$<br>• $MgO$<br>• $Nd_2Zr_2O_7$<br>• $0.7MgO\text{-}0.3Nd_2Zr_2O_7$<br>• $Mg_2SnO_4$ | All samples irradiated to 1 and 2 dpa at 350 and 700°C |
| **Team Members/Collaborators** | |
| • University of Florida — Juan Claudio Nino (principal investigator); Donald Moore and Peng Xu (graduate students)<br>• INL — Pavel Medvedev (principal investigator); Mitchell Meyer (manager); Gregg Wachs (project engineer) | |

## Project Description

The objectives of this project are to:

- Investigate the behavior of $MgO\text{-}Nd_2Zr_2O_7$ cercer composites as an inert matrix in irradiation environments (Table 1)
- Investigate the behavior of single-phase Mg-based spinel compounds as an inert matrix in irradiation environments
- Characterize the effects of irradiation on the microstructure of the materials.

Ceramic disc samples will be irradiated at approximately 350 and 700°C to dose accumulations of 1 and 2 dpa. Post-irradiation analyses will be performed at INL's Materials and Fuels Complex.

## Accomplishments

The thermal and safety analysis for the experiment was performed and new hardware designed and developed. Ceramic disc samples for thermal diffusivity measurements and TEM analyses were fabricated (Figure 1), loaded into three capsules and irradiated. The post-irradiation examination plan for the first capsule was completed and PIE activities began.

## Future Activities

Post-irradiation examination, which will begin in 2010, will encompass a number of characterization techniques.

Neutron radiography will be used to evaluate the sample integrity and movement as well as condition of the newly developed hardware.

Sample volumetric change due to irradiation will be measured by comparing pre- and post-irradiation thickness and diameter of the thermal diffusivity samples and quantifying the percentage of swelling.

Defect formation in neutron-irradiated materials will be characterized. Samples will be mechanically polished then ion milled so they are electron transparent for TEM analyses.

SEM will be used to characterize grain size, micro-cracks, voids and other deteriorative microstructural features, though changes in the samples' microstructure are not anticipated.

Thermal diffusivity of irradiated samples will be measured and correlated with radiation doses, radiation temperatures and defect formation.

The mechanism of thermal diffusivity degradation will be studied.

### Publications, Presentations and Patents

- 2009 ATR NSUF Users Week and poster session
- 2010 ATR NSUF Colloquium, "Investigation of MgO-Pyrochlore Composites and Spinel Compounds as Potential Inert Matrix Materials," Idaho National Laboratory, May 20, 2010

### *References*

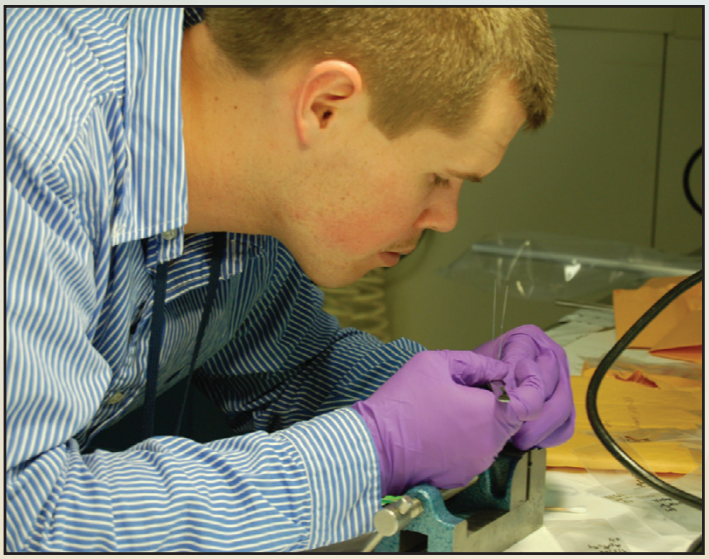

[1] Medvedev, P. G. S.M. Frank, T.P. O'Holleran and M.K. Meyer. Dual phase MgO-$ZrO_2$ ceramics for use in LWR inert matrix fuel. J Nucl Mater 342, 48-62. (2005)

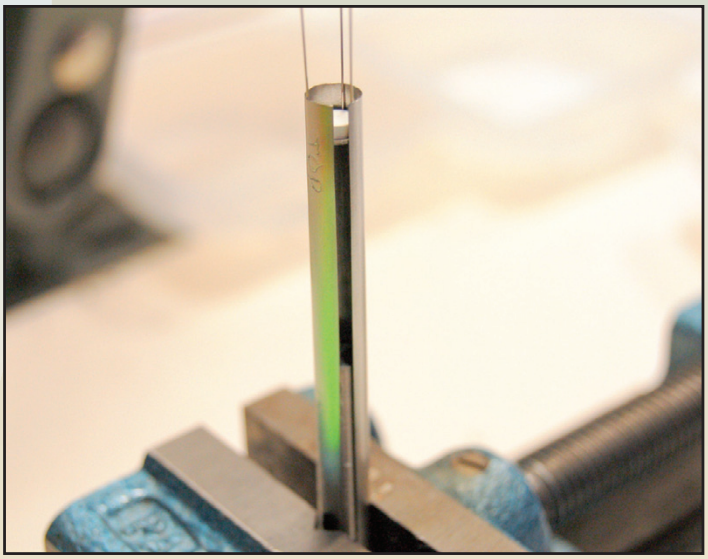
[2] Nino, J. C. NERI final technical report DE-FC07-05ID14647: Optimization of oxide compounds for advanced inert matrix materials. Report No. DOE/ID/14647-Final (2009)

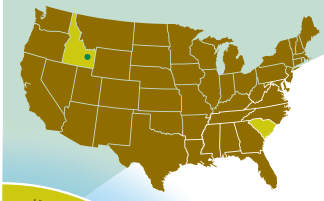

*"When you think about it, it is a great story. We developed new candidate materials for IM [inert matrix] applications here in our labs at the University of Florida. We used computational simulation to guide the material selection, we optimized the processing and fabricated samples and test them out-of-pile to select the best candidates. Now as part of the ATR NSUF program, we have the unique opportunity to test these materials under reactor conditions and learn a whole lot about their behavior. Taking a new material from the university lab all the way to the test reactor in a couple years is simply fantastic!"*

— Juan Claudio Nino, Ph.D., Associate Professor, Materials Science and Engineering, University of Florida, Gainesville, Fla.

*Figure 1. (clockwise from top left): Pavel Medvedev, INL principal investigator and Donald Moore, a University of Florida graduate student, prepare to load samples at INL; the new hardware design for loading TEM samples; closeup of the assembly hardware at the beginning of sample loading; Moore loads samples onto the sleeve and basket fixture assembly.*







project at-a-glance

# Influence of Fast Neutron Irradiation on the Mechanical Properties and Microstructure of Nanostructured Metals/Alloys

K. L. Murty, principal investigator (North Carolina State University)
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## Introduction

Materials that are resistant to radiation damage must be developed to support the development of a new generation of nuclear reactors and future fusion reactors. Radiation damage in nanostructured metals and alloys is expected to be much lower due to the relatively large volume fraction of interfaces (grain boundaries) that could act as sinks for radiation-produced defects.

## Project Description

The major objective of this project is to examine the effects of neutron radiation on the mechanical properties of nanostructured metals and alloys.

The materials studied (Table 1) will include nano-grain (nanocrystalline) structured copper (nc-Cu) and nickel (nc-Ni), ultrafine grain-sized carbon steel and the conventional Cu, Ni and steel counterparts as well as the oxide dispersion-strengthened alloys, MA-956 and MA-754.

In addition to low-dose radiation studies at the NC State PULSTAR reactor facility, high fluence effects will be studied at the Advanced Test Reactor facilities.

**Table 1. Project Details — Influence of Fast Neutron Irradiation on the Mechanical Properties and Microstructure of Nanostructured Metals/Alloys**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| NC State | PULSTAR reactor, PIE facilities |
| **Materials** | **Description** |
| • Cu<br>- nc-Cu<br>- conventional-Cu<br>• Nickel<br>- nc-Ni<br>- conventional Ni<br>• Steel<br>- ultrafine-grained<br>- conventional<br>• Oxide-dispersed strengthened alloys<br>- MA-956<br>- MA-754 | • Cu samples irradiated in PULSTAR reactor up to 0.34 dpa and in Advanced Test Reactor to 1 and 2 dpa<br>• Remaining samples irradiated to 1 and 2 dpa |
| **Team Members/Collaborators** | |
| • NC State — K. L. Murty (principal investigator); Walid Mohamed (Ph.D. candidate)<br>• University of Idaho — Indrajit Charit (co-principal investigator); Ramprashad Prabhakaran (Ph.D. candidate)<br>• INL — Douglas Porter (principal investigator) | |

## Accomplishments

Nanostructured Nc-Cu samples (28-nm grain size) and conventional grain structured Cu (21-µm grain size) were irradiated up to 0.34 dpa in the PULSTAR reactor and PIE activities were completed. The irradiated conventional Cu revealed radiation hardening and embrittlement, as commonly observed, while nc-Cu became softer following irradiation (Figure 1).

Microhardness measurements exhibited similarly increased and decreased hardness following radiation (Figure 2). As expected, the changes were relatively small due to relatively low fluence of 0.34 dpa, but it is expected that irradiations in the Advanced Test Reactor will correspond to higher fluence effects.

Grain-size measurements (both optical and TEM) were made before and after irradiation. A distinct increase in grain size, from 28nm to 87 nm, was observed for the nc-Cu, which suggests an indirect relationship between grain size and the decreased hardening exhibited by the irradiated nc-Cu. The temperature of irradiation within the PULSTAR reactor was not measured and no arrangements were made to maintain the sample temperature, but it was assumed to be the same as the coolant temperature.

The nc-Cu, nc-Ni, ultrafine grained steel and their conventional counterparts as well as the oxide dispersion-strengthened alloys were irradiated in the Advanced Test Reactor to dose accumulations of 1 dpa and 2 dpa. PIE activities, including micro-hardness and tensile tests have been completed for the samples irradiated to 1 dpa (Figure 3).

## Future Activities

The 2-dpa samples of nc-Cu, nc-Ni, ultrafine grained steel and their conventional counterparts as well as the oxide dispersion-strengthened alloys are currently undergoing PIE activities, including micro-hardness, TEM analyses and tensile tests.

> *"If we can characterize (these materials), we will understand what is happening after these special materials are exposed to radiation. Then we may be able to come up with better and more advanced materials for nuclear applications."*
>
> — K. L. Murty, Ph.D., Director of Graduate Programs and Professor, Nuclear Engineering, and Materials Science and Engineering, North Carolina State University

*Figure 1. Stress-strain curves for conventional copper (below left) and nanocrystalline copper (below right) show radiation-induced hardening for conventional copper.*

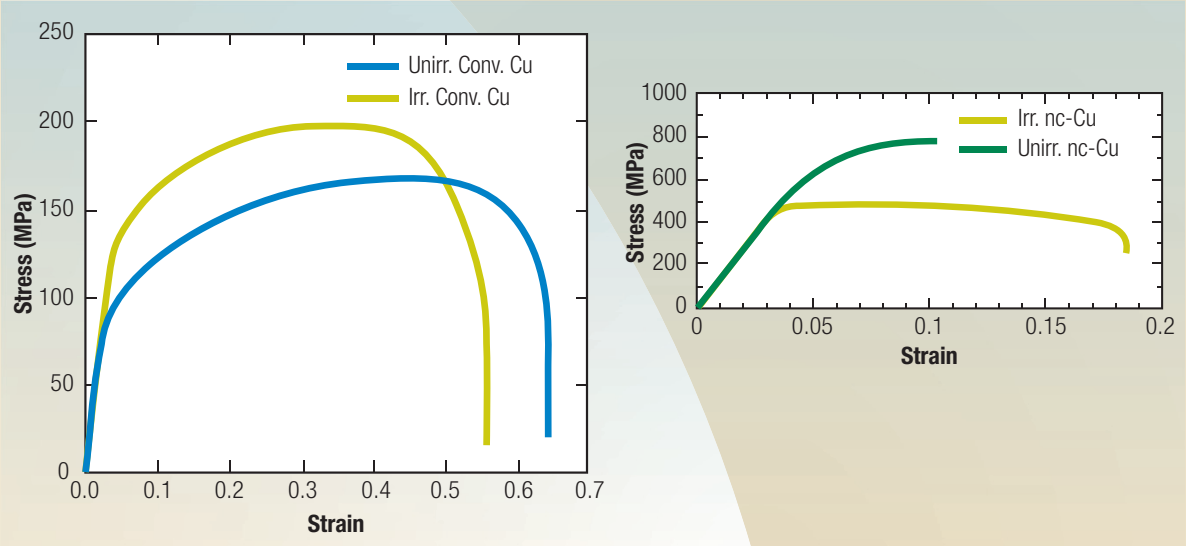



*Figure 2. Microhardness measurements of the nanocrystalline copper show radiation-induced hardness variations relative to variations in dosage levels (dpa).*

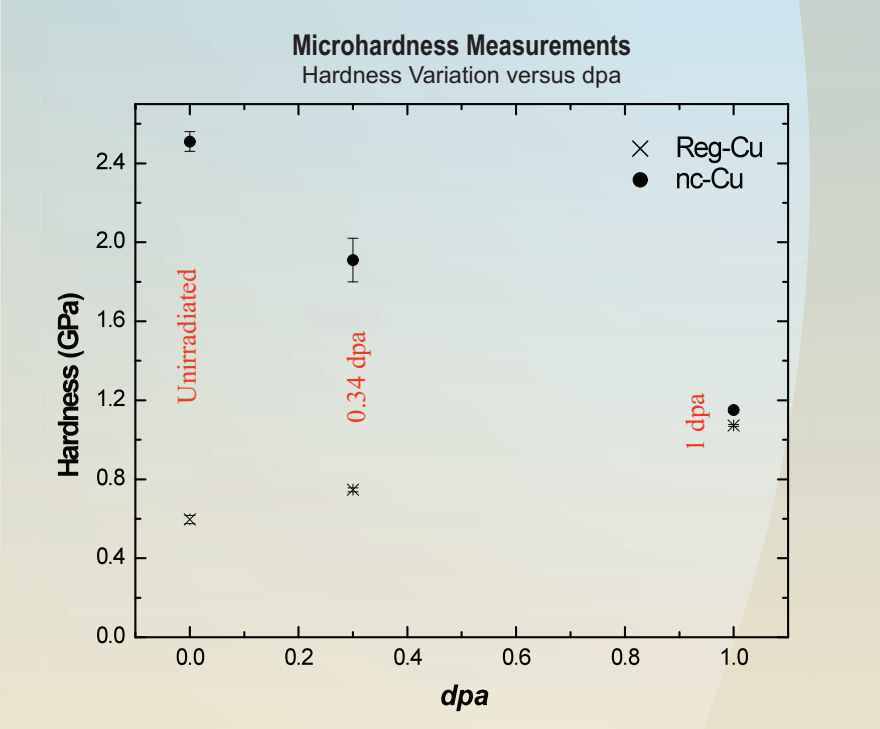
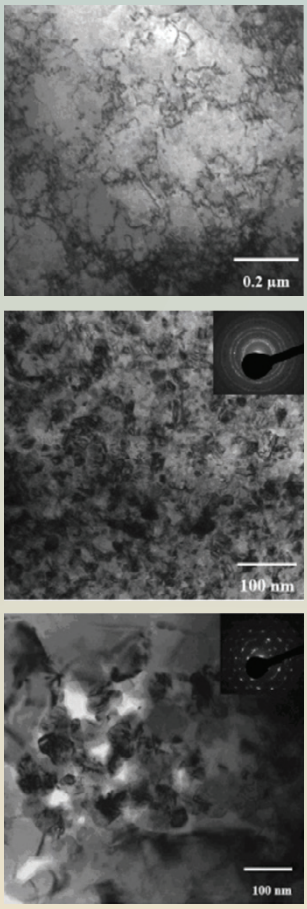


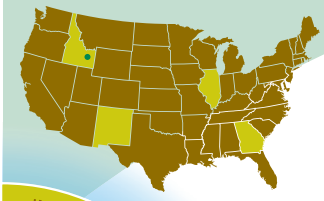

*Figure 3. Microstructure evolution is shown in TEM samples: conventional copper (below, top) and nanocrystalline copper (middle and bottom).*




Conventional copper, after irradiation

Nanocrystalline copper, before irradiation

Nanocrystalline copper, following irradiation to 0.34 dpa



# Irradiation Performance of Fe-Cr Base Alloys

James F. Stubbins, principal investigator (University of Illinois)
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project at-a-glance

## Introduction

Ferritic alloys are candidate materials for advanced nuclear power system components. Compared to austenitic stainless steels, ferritic alloys have excellent resistance to void swelling, better thermal conductivity, lower thermal expansion and acceptable high-temperature mechanical strength.

The iron-chromium (Fe-Cr)-based alloy system is considered the lead alloy system for a variety of advanced reactor components and applications.

## Project Description

The objective of this project is to gain new insights into the performance of ferritic alloys in advanced reactor applications by assessing the impacts of neutron irradiation on model, commercial and developmental Fe-Cr-based alloys (Table 1) using TEM analyses and miniature tensile testing. This effort will acquire the data needed to develop and validate computational models used to predict alloy performance.

The experimental test matrix for this project includes 12 materials, three irradiation temperatures, six irradiation levels (or doses) and two sample geometries — TEM and tensile test samples (Figure 1).

Samples will be irradiated at the Advanced Test Reactor. Low target dose irradiations (0.01–0.1 dpa) will be completed with capsules inserted at position B-7 (the hydraulic shuttle system or "rabbit"). Higher dose irradiations (0.5–10 dpa) will be completed with capsules inserted at position A-11.

## Accomplishments

TEM and tensile testing samples and capsules for high-dose irradiations (0.5–10 dpa) were machined and delivered to INL, where capsules were loaded, inspected, tested and inserted into the Advanced Test Reactor. Two target irradiation conditions, 0.5 and 1 dpa at 300°C, were completed. Higher dose irradiations have completed the third cycle and are ready to start the fourth cycle.

The project team overcame machining difficulties and restrictions on sample geometries. The single crystalline Fe-19Cr samples required the design and fabrication of a new fixture that would hold smaller pieces. The highest target dose capsules (5 and 10 dpa at 300°C) required a slight adjustment to maintain low temperature at high flux axial position. For these capsules, the sample diameters were increased to reduce the gas gap within the capsule.

## Future Activities

After the high dose irradiated samples have completed a suitable cooling-down period completed, the capsules will be transferred to INL's Hot Fuel Examination Facility for PIE activities.

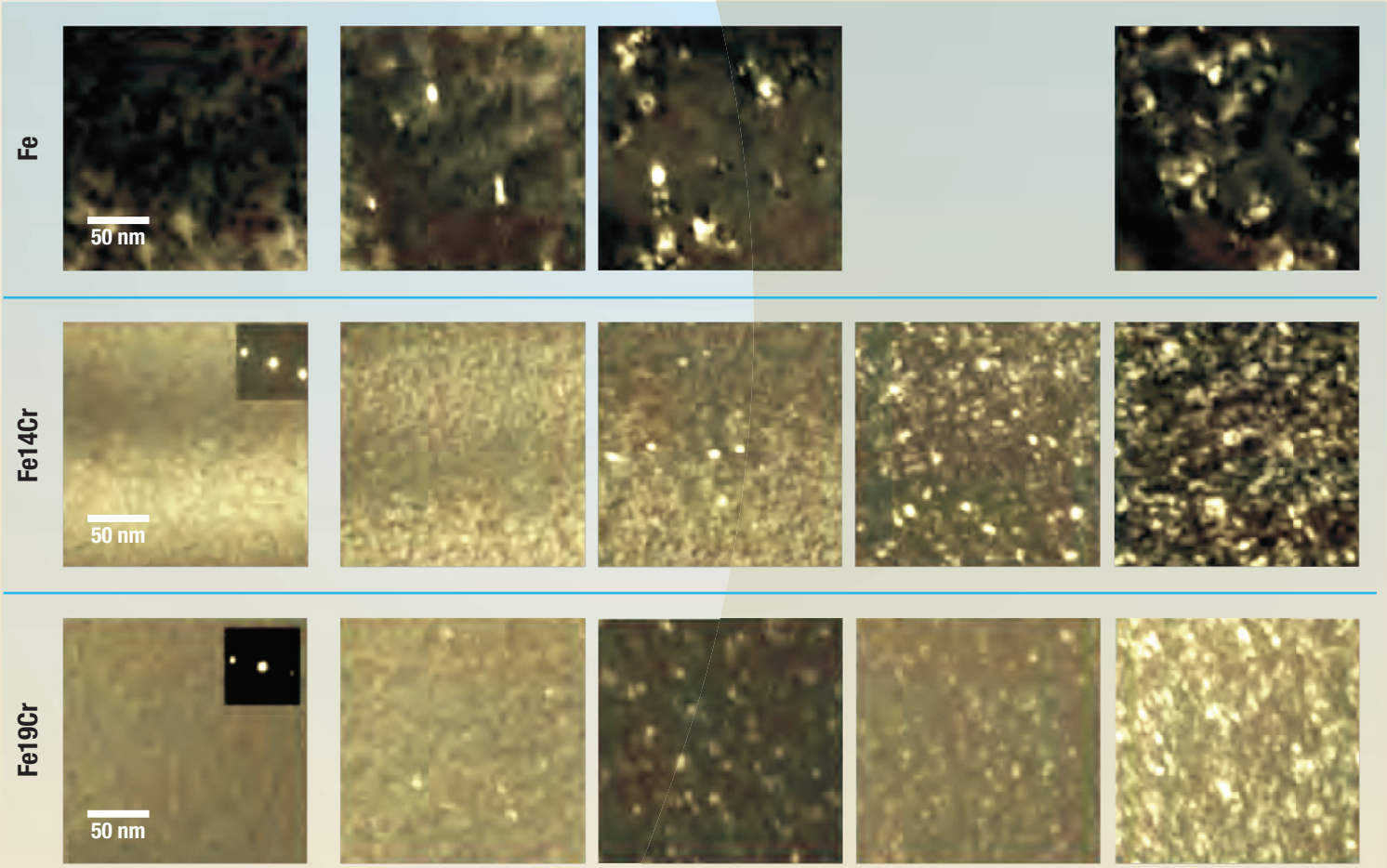
**Table 1. Project Details — Irradiation Performance of Fe-Cr Base Alloys**

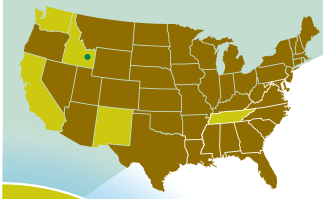
| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| **Materials** | **Description** |
| Model alloys<br>• Fe<br>• Fe-9Cr<br>• Fe-9Cr-0.1C<br>• Fe-9Cr-0.5C<br>• Fe-12Cr<br>• Fe-12Cr-0.2C<br>• Fe-12Cr-0.5C<br>• Fe-14Cr*<br>• Fe-19Cr*<br>Commercial alloys<br>• T91<br>• HT-9<br>Developmental alloy<br>• MA-957<br>* Single crystal materials, no miniature tensile samples | All samples irradiated to 0.01, 0.1, 0.5, 1.0, 5.0 and 10.0 dpa at 300, 450, 550°C |
| **Team Members/Collaborators** | |
| • University of Illinois — James Stubbins (principal investigator)<br>• INL — Jian Gan (principal investigator); Maria Okuniewski (co-investigator); Gregg Wachs (project engineer)<br>• University of Idaho — Carolyn Tomchik (graduate student)<br>• Georgia Institute of Technology — Chaitanya Deo (collaborator)<br>• General Electric Company — Eric Loewen (collaborator)<br>• Los Alamos National Laboratory — Stuart Maloy (collaborator) | |

The team will finalize the sample layout and geometries in the titanium capsule for low-dose irradiations (target doses of 0.01 and 0.1 dpa) and machine the samples. The capsules for the rabbit will be loaded, inspected and tested. After the low dose irradiations are completed, the capsules will also be transferred to INL's Hot Fuel Examination Facility. PIE sample preparation, testing and TEM analyses will begin and is expected to continue into 2011.

> *"We are looking forward to becoming the first users of the "rabbit" facility later this summer. This facility will allow us to perform some short-term, low dose irradiation exposures at temperature to study the early evolution of radiation damage in our Fe-Cr alloy series."*
>
> — J. F. Stubbins, Ph.D., Department Head and Professor, Nuclear, Plasma and Radiological Engineering, University of Illinois

*Figure 1. Dark field microscopic images show 50-nm samples of pure Fe, Fe-14Cr and Fe-19Cr single crystal material that were irradiated at the Intermediate Voltage Electron Microscope/Tandem Facility at Argonne National Laboratory. The samples were irradiated at 450°C with irradiation doses of (each row, from left): 0, 1 x $10^{14}$, 4 x $10^{14}$, 7 x $10^{14}$ and 1 x $10^{15}$ ion/$cm^2$. Alloy samples of the same materials will be subjected to neutron irradiation at similar dosage levels in the Advanced Test Reactor and re-examined.*







# Characterization of Advanced Structural Alloys for Radiation Service

G. R. Odette, principal investigator (University of California, Santa Barbara)
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## Introduction

A challenge to the development of advanced sources of nuclear energy is the deleterious effects of radiation damage on structural materials. A better understanding is needed of the radiation-induced degradation of these materials' mechanical properties and microstructures.

Conducting side-by-side alloy irradiation and developing a comprehensive database will lead to better understanding, enabling the development of models development of models of irradiation effects.

## Project Description

The objective of this project is to create a large library of irradiated alloys and sample types. To accomplish this, 44 structural and model alloys — tempered martensitic steels, nanostructured ferritic alloys, a stainless steel, a set of Mn-Mo-Ni bainitic pressure vessel steels and a variety of simple model alloys — will be irradiated (Table 1).

The alloys will be irradiated to a range of 1.5–6 dpa at seven temperatures between 300 and 750°C to build a database of neutron induced hardening and softening phenomena. Changes in their properties will be assessed with micro-hardness measurements, supplemented by tensile tests on a subset of materials. Irradiating many alloys side-by-side will enable the database to provide a unique opportunity for understanding and models for the effects of metallurgical variables on irradiation effects on constitutive properties.

Fracture studies using compact tension samples will be carried in the framework of the Master Curve method on a subset of alloys to measure shifts in the 100 MPa√m reference temperatures, $\Delta T_o$. The compact tension tests will be supplemented by mini bend bar tests on other alloys.

Model alloys will be used to study various key mechanisms. For example, 0-18% chromium (Cr) iron (Fe-Cr) binary alloys irradiated over a wide range of irradiation temperatures will be used to study alloying effects on microstructures and hardness.

Diffusion multiples, a "lab-on-a-chip" combination of materials, will enable characterization of thermokinetic parameters and phase fields in multi-constituent alloys under irradiation.

State-of-the-art tools will be used to relate mechanical property tests of the irradiated materials to microstructural characterization studies. In situ helium implantation studies will also be conducted.

The majority of the specimens — approximately 1,380 samples — are disc multi-purpose coupons, but also included are sub-sized tensile, disc compact tension fracture, deformation and fracture mini-beam, chevron notch wedge fracture and cylindrical compression specimens.

**Table 1. Project Details — Characterization of Advanced Structural Alloys for Radiation Service**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| **Materials** | **Description** |
| • Tempered martensitic steels<br>• Nanostructured ferritic alloys<br>• Mn-Mo-Ni bainitic pressure vessel steels<br>• Stainless steel<br>• Simple model alloys | • Samples irradiated to approximately 1.5-6 dpa at temperatures between 300 and 750°C |
| **Team Members/Collaborators** | |
| • University of California, Santa Barbara — G. R. Odette (principal investigator) David Gragg, Doug Klingensmith, Ben Sams, Takuya Yamamoto (co-principal investigators); Nicholas Cunningham (Ph.D. candidate)<br>• INL — Jim Cole (principal investigator); Paul Murray, Gregg Wachs, Tony Walters (project engineers)<br>• Los Alamos National Laboratory — S. Maloy (collaborator)<br>• Oak Ridge National Laboratory — J. Busby (collaborator)<br>• Pacific Northwest National Laboratory — R. Kurtz, M. Toloczko (collaborator)<br>• University of California, Berkeley — B. D. Wirth (collaborator) | |

## Accomplishments

Team members from University of California, Santa Barbara (UCSB) began the project in January 2009. By June, the required documents were prepared, materials were collected, samples and capsule parts were fabricated and sample packets were assembled. The specimens were loaded in 11 sub-capsules containing a total of 32 isothermal temperature packets. Figure 1 shows the configuration of a typical diffusion multiple sample and nickel foil wedge

sample stack-up for in-situ helium implantation studies.

An innovative capsule design was used to optimize the precision and control of the irradiation temperatures. The design involved establishing axial insulating sections that force radial heat transfer primarily though only one press fit interface and a gas gap. Isothermal temperatures were maintained in the specimen packets by varying the thickness of a profiled gas gap between the packets and the cooled capsule wall in a way that effectively accounts for axial variations in the nuclear heat generation rate (Figure 2).

In July 2009, the sample packets were shipped to INL with detailed documentation for insertion in the ASME pressure boundary capsule and A-10 basket irradiation vehicle of the Advanced Test Reactor.

Irradiation began in August 2009 at seven temperatures, ranging from approximately 300–550°C in 50-degree increments and at 650 and 750°C, up to a peak dose of 6 dpa. A low-dpa, high-flux capsule has already been removed and a replacement capsule reinserted.

## Future Activities

Irradiation will be completed in June 2010. A plan for sample post-irradiation examinations will be developed. The majority of PIE analyses will take place at INL and other national laboratory hot cells. Focused ion beam micro-machined samples will be prepared and added to the ATR NSUF sample library where they can be distributed for post-irradiation examination at universities and research institutions around the world.

> *"I have been involved with irradiation experiments for more than 40 years, including a number of very large-scale studies. I can say that working with the INL staff to design, build and successfully initiate the irradiation for the very scientifically aggressive, multi-faceted UCSB-ATR experiment has been one of the most satisfying experiences of my career."*
>
> — G. R. Odette, Ph.D., Professor, Mechanical Engineering and Materials, University of California Santa Barbara

*Figure 1. An example of a 300°C isothermal temperature packet.*

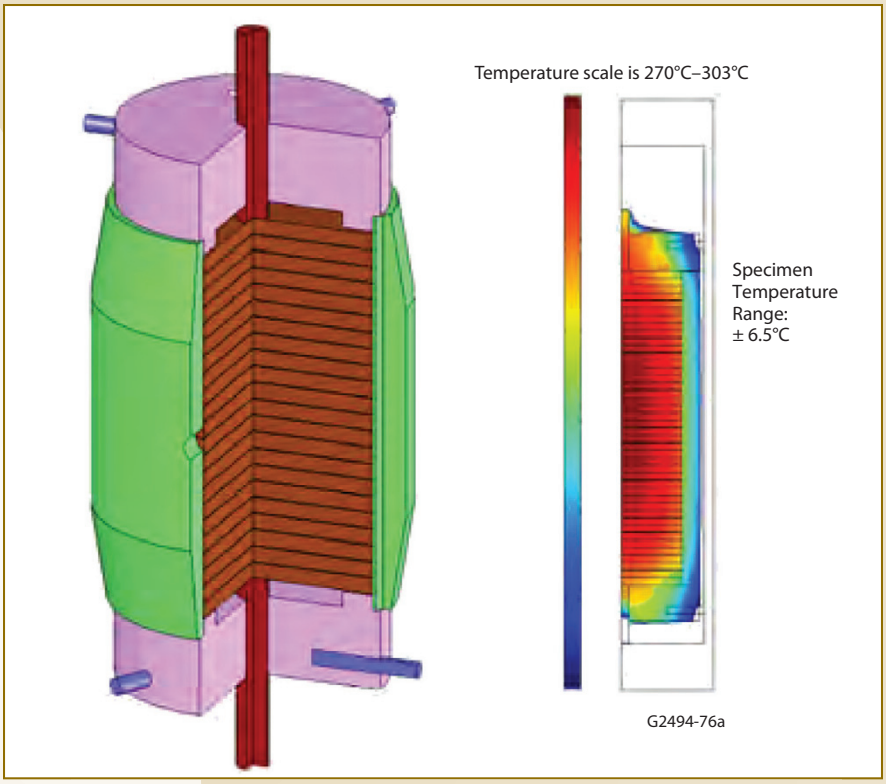
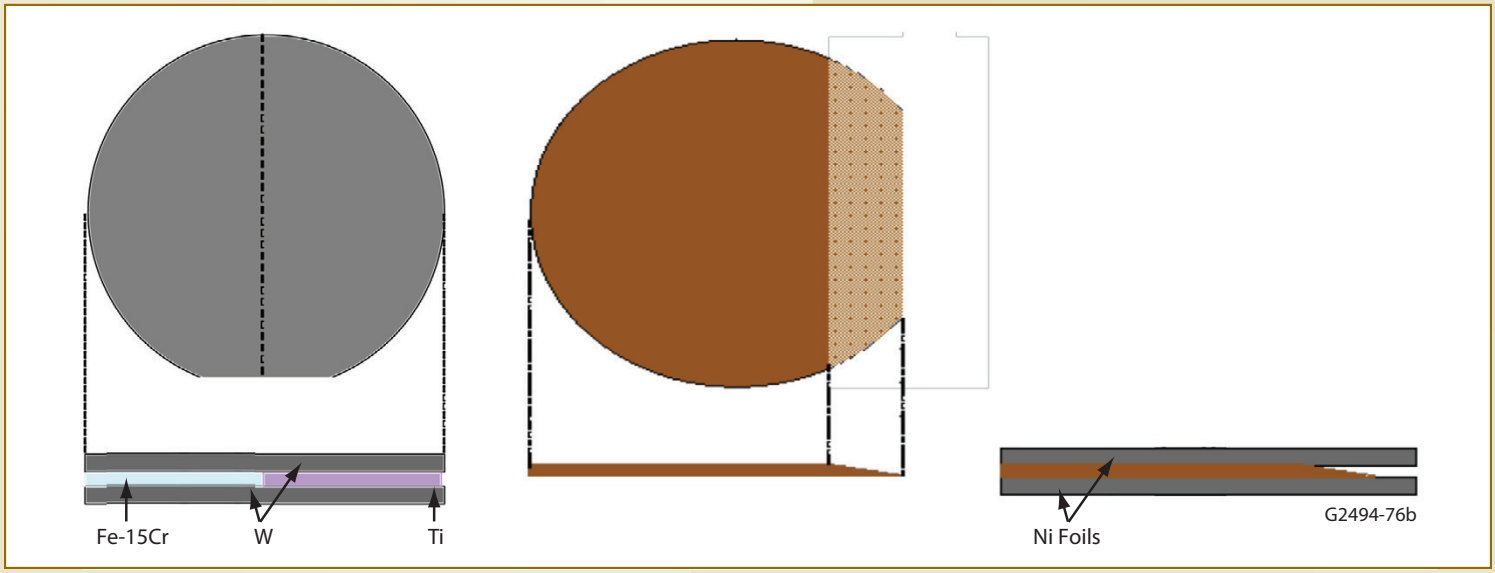
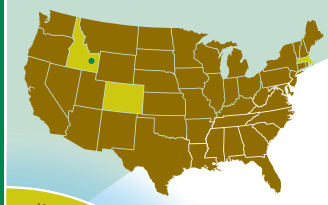



*Figure 2. Two sample configurations include: (left) a typical diffusion multiple specimen for Fe-W-Cr-Ti and (right) a nickel foil wedge specimen stack-up for in situ helium implantation studies.*





project at-a-glance

# Advanced Non-Destructive Assessment Technology to Determine the Aging of Silicon-Containing Materials for Generation IV Nuclear Reactors

David L. Olson, principal investigator (Colorado School of Mines)
e-mail: dolson@mines.edu

## Introduction

The higher operating temperatures of next generation nuclear reactors will require high-temperature structural materials in the reactor core. Non-destructive assessment tools are needed for assessing irradiation damage (aging) in these structural materials as well as for in-situ monitoring.

Many potential sensory materials are silicon-based, such as silicon carbide (SiC), silicon nitride ($Si_3N_4$) and possibly sialons, aluminium-silicon-oxyniride alloys of silicon nitride. These materials have a semiconductor or non-stoichiometric ionic structure (excess oxygen or metal ions) which, when damaged, will cause measurable changes in their electronic concentrations. A calibrated sensor for a given nuclear reactor core material could correlate the irradiation-induced changes in the electronic properties of these semiconductor sensors with a knowledge base of structurally damaged materials.

Note: A related ATR NSUF project, "Radiation Stability of Ceramics for Advanced Fuel Applications," is discussed on page 24 of this report.

## Project Description

The objective of this project is to investigate the application of neutron transmutation doping in silicon-containing ceramic materials used as radiation damage sensors via the neutron transformation of silicon to phosphorus. The silicon-neutron reaction — $^{30}Si\ (n, \gamma)\ ^{31}Si \rightarrow\ ^{31}P + \beta$ — produces a phosphorus-doped semiconductor that exhibits n-type behavior.

Neutron transmutation doping offers a method to easily, non-destructively and continuously evaluate aging nuclear materials by using thermoelectric power contact sensors, low-frequency impedance contact measurements, Hall coefficient measurements, gamma spectroscopy (for activated residuals) and ultrasound resonance spectroscopy. These methods allow for the measurement of changes that indicate neutron fluence and its effects on electronic carriers, structural damage and modification of the polytype of the SiC.

The project will develop and assess electronic-property measurement methodology for rapid in situ non-destructive assessment of structural nuclear materials during reactor operation as well as passive detection. This will include:

- Characterizing the radiation damage on SiC core materials, including p-doped additions that may assist in the healing; damage will be monitored by using the n-type neutron doping that results from transmutation of silicon into phosphorous
- Utilizing chemically p-doped SiC, which will allow increased sensitivity in measuring the irradiation damage by annihilation of the n-type neutron transmutation dopant and easier measurements of electronic properties
- Developing practices to assess the alteration of the electronic properties of the SiC and other core materials by monitoring the resulting n-type neutron doping.

The materials used initially will be semiconductor-grade SiC materials, sourced from MIT. INL researchers have expressed interest in using SiC as a core material and have promoted initial preliminary activities in the behavior of SiC materials under a fluence of neutrons.

Using SiC for advanced electronic and elastic wave sensors will require understanding how it is altered by neutron flux as well as understanding the correlation, calibration and standardization of SiC as a radiation damage sensor. To develop this understanding, electronic

**Table 1. Project Details — Advanced Non-Destructive Assessment Technology to Determine the Ageing of Silicon-Containing Materials for Generation IV Nuclear Reactors**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | PIE facilities |
| MIT | MIT reactor, PIE facilities |
| **Materials** | **Description** |
| • SiC | Samples irradiated to fluence of $10^{19}$ $cm^{-2}$ |

**Team Members/Collaborators**

- Colorado School of Mines — David L. Olson (principal investigator); Michael Kaufman, Jeff King, Brajendra Mishra (investigator/project engineers); Travis Koenig (Ph.D. candidate)
- INL — Mitchell Meyer (principal investigator); Jim Cole, Rory Kennedy (investigators)
- MIT — Lin-wen Hu, Gordon Kohse (collaborators)

properties will be nondestructively assessed with LCR (eddy current), elastic wave (ultrasonic), Hall coefficient, thermoelectric power, and gamma spectroscopy measurement systems. These measurement systems will be used to assess acoustic and electronic impedances as well as their related measurements. Hall coefficient measurements will be made to assess the electronic carrier content, which is essential for the development of an electronic sensor for n-type neutron doping of the SiC. Irradiation causes changes in both carrier content and specific polytype, so multiple measurement systems are needed to completely assess the effects of the irradiation.

Structural damage will also be assessed using synchrotron radiation analysis and transmission electron microscopy, which will identify the changes in the polytype of SiC being generated.

## Accomplishments

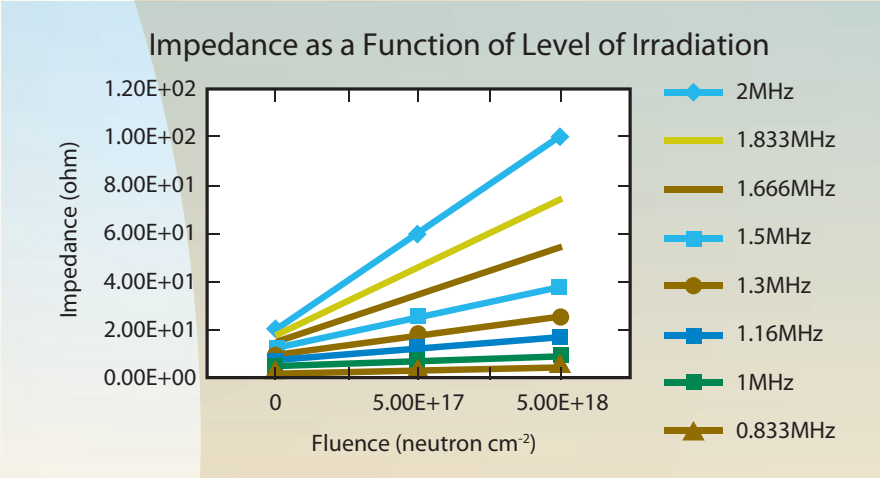
The project team, in collaboration with INL and the MIT reactor group, performed initial tests on SiC material up to $10^{19}$ $cm^{-2}$ of fluence. During spring 2010, senior Colorado School of Mines students assisted on the preliminary effort.

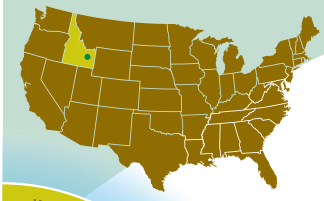
Preliminary results show an excellent correlation between electronic impedance measurements and radiation damage in SiC (Figure 1). Ultrasound resonance frequency analysis was used to assess the radiation damage in the irradiated SiC samples.

Structural damage in the SiC was seen to dominate over the issues of neutron electrical doping. Adding cadmium coatings of various thicknesses to the SiC sensor material would allow for a comparative interpretation between the difference in properties from lattice damage increase in carrier content. These results indicate a high probability of success in the development of an inexpensive, non-contact, in-situ, nondestructive sensor for nuclear reactor cores.

> *"This experiment has shown encouraging preliminary results and can provide useful nondestructive assessment tools for monitoring radiation damage in future nuclear reactors."*
>
> — David L. Olson, Ph.D., PE, John Henry Moore Distinguished Professor of Metallurgical Engineering; Professor of Metallurgical and Materials Engineering Lead Scientist, Materials Science Program, Colorado School of Mines

*Figure 1. The preliminary results indicate a correlation between irradiation level and impedance in irradiated silicon carbide, a future reactive fuel clad material.*





project at-a-glance

# Real-Time ATR-C Flux Sensors

George Imel and Jason Harris, principal investigators (Idaho State University)
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## Introduction

Real-time methods for detecting thermal neutron flux and fission reaction rates for irradiation capsules are lacking at both the Advanced Test Reactor (ATR) and Advanced Test Reactor-Critical (ATR-C) facility. The ATR has the ability to measure fast neutron flux in real-time as it relates to individual reactor lobe power, but it is only utilized for reactor operations and not for individual experiments.

Newly developed sensor technologies have the potential to directly measure the actual power deposited into a test without resorting to complicated correction factors. These sensors may also provide the ability to directly measure minor actinide fission reaction rates and to provide time-dependent monitoring of the fission reaction rate or fast/thermal flux during transient testing.

## Project Description

The objective of this project is to investigate the feasibility of using neutron flux detectors to provide online measurements of the fission reaction rate in the ATR-C. The detectors assessed will include:

- Sub-miniature fission chambers, real-time state-of-the-art in-pile flux detection instrumentation developed by the Commissariat de Energie Atomique (CEA) in France (Figure 1)
- Self-powered neutron detectors (Figure 2)
- Back-to-back fission chambers designed for highly accurate absolute measurements of fission reaction rates (Figure 3).

This project will compare the accuracy, response time and long-duration performance of these detectors and their ability to provide online regional power measurement in the ATR-C.

This project offers the potential to increase the current ATR-C power limit and the ability to perform low-level irradiation experiments. It is complemented by current activities to improve advanced in-reactor software tools, computational protocols and in-core instrumentation for the ATR.

## Accomplishments

Self-powered neutron detectors and back-to-back fission chambers that were used in experiments at Argonne National Laboratory were transferred to Idaho State University's Nuclear Engineering Laboratory where they underwent testing. All necessary electronics were gathered. A draft test plan was generated and sent to CEA for approval.

## Future Activities

In October 2010, the fixtures for inserting sensors into the ATR-C were installed, then testing of the CEA miniature fission chambers and self-powered neutron detectors was initiated. The evaluations proposed in the test plan will be completed during FY 2011.

Techniques for assessing flux detector performance will be used to develop detailed procedures for initial and follow-on sensor calibrations. In addition to comparing data obtained from each type of detector, calculations will be performed to assess sensor performance, reduce uncertainties in flux detection sensors and compare sensor data with existing integral methods employed at the ATR-C.

**Table 1. Project Details — Real Time ATR-C Flux Sensors**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| **In-reactor Instrumentation** | **Description** |
| • Sub-miniature fission chambers<br>• Self-powered neutron detectors (SPNDs)<br>• Back-to-back fission chambers (BTBs) | Detectors installed in Advanced Test Reactor-Critical facility and compared |
| **Team Members/Collaborators** | |
| • Idaho State University — George Imel, Jason Harris (principal investigators); Eric Bonebreak (M.S. candidate, mechanical engineering)<br>• INL — Joy Rempe (principal investigator) | |

> *"The ATR-C capability developed in this project provides researchers from ISU, INL, CEA [Commissariat de Energie Atomique in France] and other organizations a unique opportunity for evaluating real-time flux detectors."*
>
> — George Imel, Ph.D., Professor, Founding Dean,
> College of Science and Engineering, Idaho State University

*Figure 1. A sub-miniature fission chamber (bottom), a real-time state-of-the-art in-pile flux detection instrument developed by the Commissariat de Energie Atomique, is one of the flux detector technologies that will be assessed at the Advanced Test Reactor. A matchstick is shown as a basis for comparison.*

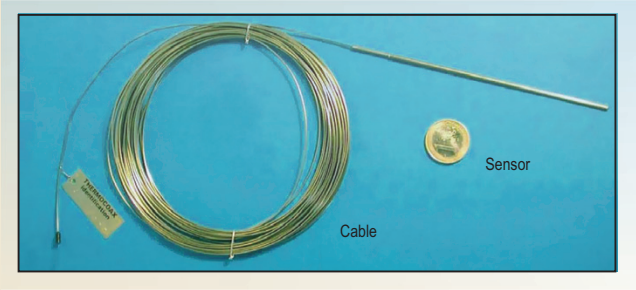
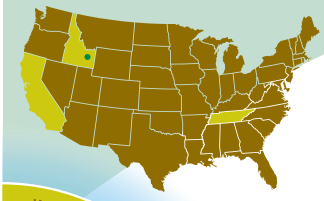



*Figure 2. Another flux detector that will be assessed is a commercially available self-powered neutron detector (SPND).*

*Figure 3. The third type of flux detector that will be assessed is a back-to-back (BTB) fission chamber, shown closed above right and open below.*





# A High Fluence Embrittlement Database and ATR Irradiation Facility for Light Water Reactor Vessel-Life Extension

G. R. Odette, principal investigator (University of California, Santa Barbara)
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## Introduction

Our nation's energy stability depends on the safe operation of our current fleet of pressurized light water reactors at longer lifetimes. Extending lifetimes up to 80 years, for example, will require greater understanding of the properties of reactor pressure vessel steels during long-term exposure to neutron bombardment.

It is known that neutron flux has strong and very complex effects on irradiation hardening and ductile to brittle transition temperature shifts, both of which are manifestations of irradiation embrittlement. Depending on all the other irradiation and material conditions, higher flux can increase, decrease or leave hardening and transition temperature shifts unaffected.

Currently, there is little low flux reactor pressure vessel surveillance data on transition temperature shifts (TTS) at high fluence levels (greater than approximately $5x10^{19}$ n/cm$^2$). Also, current low flux embrittlement models systematically and significantly under-predict the transition temperature shifts in the existing high flux, accelerated test reactor TTS database.

To help resolve these issues, new experiments are needed that will provide a basis for accurate predictions of reactor pressure vessel transition temperature shifts at high fluences.

## Project Description

The objectives of this project are to:

- Develop a new Advanced Test Reactor test rig for precisely controlled intermediate flux neutron irradiations of pressure vessel steels
- Characterize a large number of irradiated reactor pressure vessel steels and model alloys — more than 1,500 samples — to fill major gaps regarding the combined effects of composition, temperature, flux and fluence on embrittlement.

Most notably, this experiment will provide a new high fluence database at an intermediate flux that will be linked with other test reactor and surveillance databases over a much wider range of flux to refine and validate predictive physically based transition temperature shift models, and to resolve a umber of other outstanding embrittlement issues.

The project will focus on:

- Assessing flux effects, including the use of post-irradiation annealing, to evaluate the contributions of various irradiation hardening features as a function of flux and other embrittlement variables
- Identifying alloy-irradiation conditions leading to the formation of "late-blooming" phases that could lead to severe, but currently unaccounted for, embrittlement
- Conducting extensive post-irradiation microstructural characterization and mechanism studies
- Conducting post-irradiation annealing recovery experiments as a potential embrittlement mitigation strategy
- Irradiating new reactor pressure vessel alloys, including candidates for use in advanced reactors
- Evaluating the master curve method for measuring fracture toughness at high fluence in sensitive alloys
- Addressing the surrogacy issue associated with alloy conditions in the actual vessel, which typically differ from nominally limiting steels for particular reactor vessels.

The flux-fluence bridging provided by the Advanced Test Reactor's reactor pressure vessel irradiation (Figure 1) will include a variety of

**Table 1. Project Details — A High Fluence Embrittlement Database and ATR Irradiation Facility for Light Water Reactor Vessel-Life Extension**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| **Materials** | **Description** |
| • Reactor pressure vessel steels (variety to be determined)<br>• Model alloys (variety to be determined)[a] | Samples irradiated to approximately $3.8x10^{12}$ n/cm$^2$-s at 255, 270, 290, 310°C |
| **Team Members/Collaborators[a]** | |
| • University of California, Santa Barbara — G. R. Odette (principal investigator); Takuya Yamamoto (co-principal investigator); Doug Klingensmith (investigator/project engineer); N. Reilly-Shapiro, S. Cun, J. Krafve, S. Kabachek (undergraduate research team)<br>• INL — Mitchell Meyer (principal investigator); Paul Murray, M. Sprenger (project engineers)<br>• Oak Ridge National Laboratory — Randy Nanstad (collaborator)<br>• University of California, Berkeley — B. D. Wirth (collaborator) | |

a. Additional participants/collaborators in the effort to identify and acquire alloys include: Bill Server at ATI Consulting; Rolls Royce Marine Power; Electric Power Research Institute; Bettis Atomic Power Laboratory; Central Research Institute of Electric Power Industry (Japan)

sample types. The steels used in this project will be primarily selected from alloys that were previously irradiated over a wide range of flux, including in the lower flux Irradiation Variable Program conducted by the University of California, Santa Barbara (UCSB), as well as other test reactors. Surveillance alloys as well as 50 new specially prepared steels with a wider range of composition than is represented in the current database will also be included in this irradiation project.

The project will include disc multipurpose coupons, disc compact tension fracture specimens and subsized tensile specimens.

## Accomplishments

A sophisticated design for the irradiation test rig was developed by a team of INL engineers in consultation with researchers at UCSB. As just one of the many examples of meeting design objectives, thin gadolinium thermal neutron shield sleeves will be used to minimize the sample activation. Sample temperatures will be controlled by profiled gas gaps containing a variable mixture of helium and argon that can adjust for time dependent and axial variations in nuclear heating. The specimen temperatures will be monitored by 28 thermocouples that will be used to control the gas mixtures in three separate compartments. The capsule cross section and axial temperature map are shown in Figure 2.

A mechanical engineering senior design project team at UCSB developed a scaled mock-up of the irradiation capsule.

> *"The ATR experiment will make a major contribution to resolving questions about RPV [reactor pressure vessel] embrittlement at high fluence in a timeframe that will contribute to the viability of life extension to beyond 60 years of our largest source of carbon-free energy from our existing fleet of LWR [light water reactor] nuclear power plants."*
>
> — G. R. Odette, Ph.D., Professor, Mechanical Engineering and Materials, University of California Santa Barbara

Oak Ridge National Laboratory, Rolls Royce Marine Power, Electric Power Research Institute, Bettis Atomic Power Laboratory and, in Japan, the Central Research Institute of Electric Power Industry have expressed interest in this experiment and have identified and acquired alloys that may be included.

## Future Activities

By the end of January 2011, all the alloys will be acquired and samples will be fabricated and loaded into the inner irradiation assembly at UCSB, coinciding with the completion of all necessary documentation and certifications.

The loaded capsule will be shipped to INL for assembly in the irradiation vehicle for insertion into the Advanced Test Reactor at position I-10 for irradiation beginning April 2011. Irradiations will occur at nominal temperatures of 255, 270, 290 and 310°C. In approximately 1.2 years, peak flux levels of approximately $3.8 \times 10^{12}$ n/cm$^2$-s will produce a fluence of approximately $10^{20}$ n/cm$^2$.

*Figure 1. Flux-fluence map for reactor pressure vessel steel irradiations.*

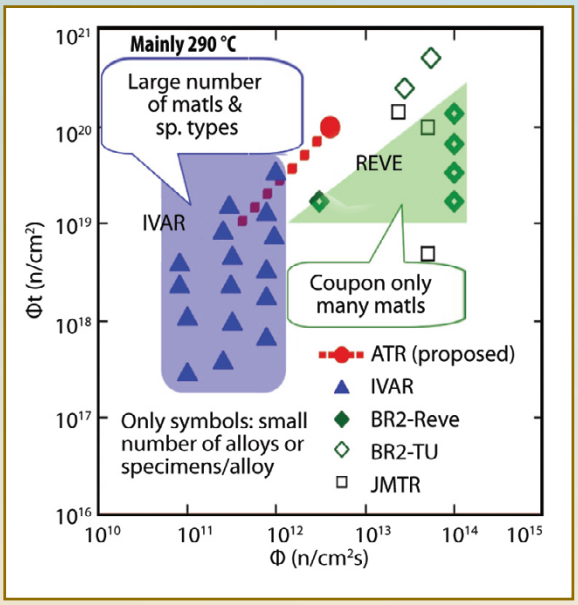
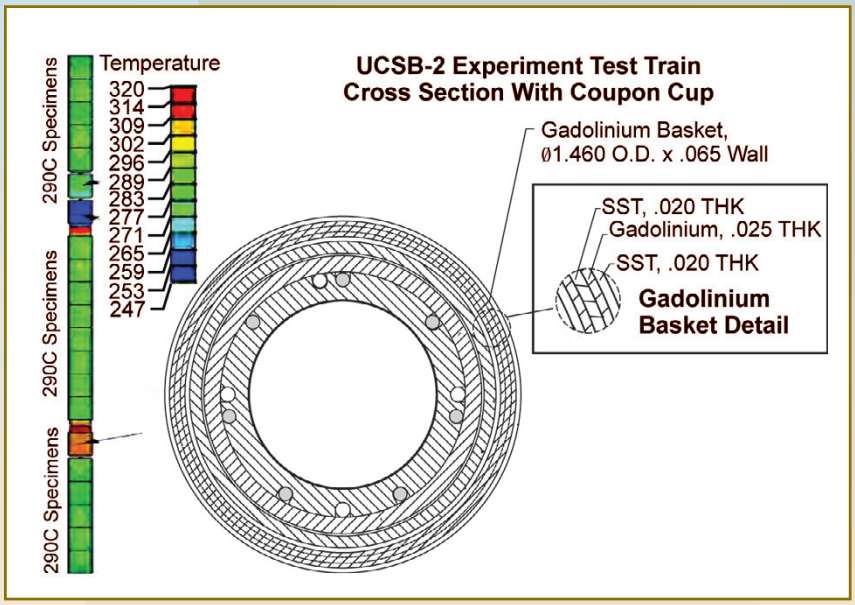
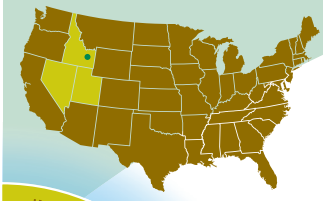



*Figure 2. The preliminary design of the irradiation capsule shows the cross section and gas gap controlled temperature profile.*





project at-a-glance

# Irradiation Effect on Thermophysical Properties of Hafnium-Aluminide Composite: A Concept for Fast Neutron Testing at ATR

Heng Ban, principal investigator (Utah State University)
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## Introduction

The development of advanced nuclear reactors has been hindered by the lack of capability for conducting fast neutron irradiation tests in a domestic facility. For example, advanced materials and fast reactor fuels have been sent to France or Japan for testing.

An absorber material comprised of hafnium aluminide ($Al_3Hf$) particles (approximately 23% by volume) in an aluminum matrix ($Al_3Hf$-Al) can absorb thermal neutrons and transfer heat to pressurized water-cooling channels. Thermal analyses conducted on a candidate configuration confirm that the design of a water-cooled $Al_3Hf$-Al absorber block is capable of maintaining all system components below their maximum allowable temperature limits. However, the thermophysical properties of $Al_3Hf$ have never been measured and the effect of irradiation on these properties has never been determined.

These materials could change the local characteristics of a reactor experiment, allowing an improved ability to tailor experiments to a customer's needs.

## Project Description

The objectives of this project are to determine the necessary properties and behavior of hafnium-aluminide alloys to determine their long-term stability in a reactor environment. The information gained will provide the necessary data for the development of fast neutron test capability at the Advanced Test Reactor as well as advance the understanding of the basic properties of $Al_3Hf$ and $Al_3Hf$-Al and how they are affected by irradiation.

The project will include:

- Investigating the use of one of the Advanced Test Reactor corner lobes with the addition of a thermal neutron filter to absorb the thermal neutrons and booster fuel to augment the neutron flux
- Assessing the irradiation effects on the thermophysical and mechanical properties of the $Al_3Hf$ intermetallic and $Al_3Hf$-Al composite as well as collecting other information, including corrosion behavior and radioactive decay products, that is necessary to proceed with the design and optimization. (The material specimens that will be irradiated are part of a unique, patent-pending design.)

Emphasis will be placed on studying:

- Thermophysical and mechanical properties of $Al_3Hf$ intermetallic and $Al_3Hf$-Al composite at different temperatures (Figure 1)
- Effects of irradiation on the thermophysical and material properties of the $Al_3Hf$ intermetallic and $Al_3Hf$-Al composite as well as the physical/morphological, metallurgical and microstructural changes of the $Al_3Hf$-Al composite after different cycles of irradiation
- Decay products of hafnium — such as Hf-179m1 versus Hf-179m2 — and corrosion behavior of the $Al_3Hf$-Al composite.

## Accomplishments

The team completed initial sample fabrication and measurement of properties. An irradiation test plan was prepared and reviewed by March 2010. The efforts to prepare a post-irradiation test plan also began.

**Table 1. Project Details — Irradiation Effect on Thermophysical Properties of Hafnium-Aluminide Composite: A Concept for Fast Neutron Testing at ATR**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| **Materials** | **Description** |
| • $Al_3Hf$<br>• $Al_3Hf$-Al composite | Sample fabrication and measurement completed; irradiation test plan prepared |
| **Team Members/Collaborators** | |
| • Utah State University — Heng Ban (principal investigator)<br>• UNLV — Thomas Hartmann (collaborator); Adam Gerth, Kurt Harris, Heather Wamper (students)<br>• INL — Donna Post Guillen (principal investigator) | |

## Future Activities

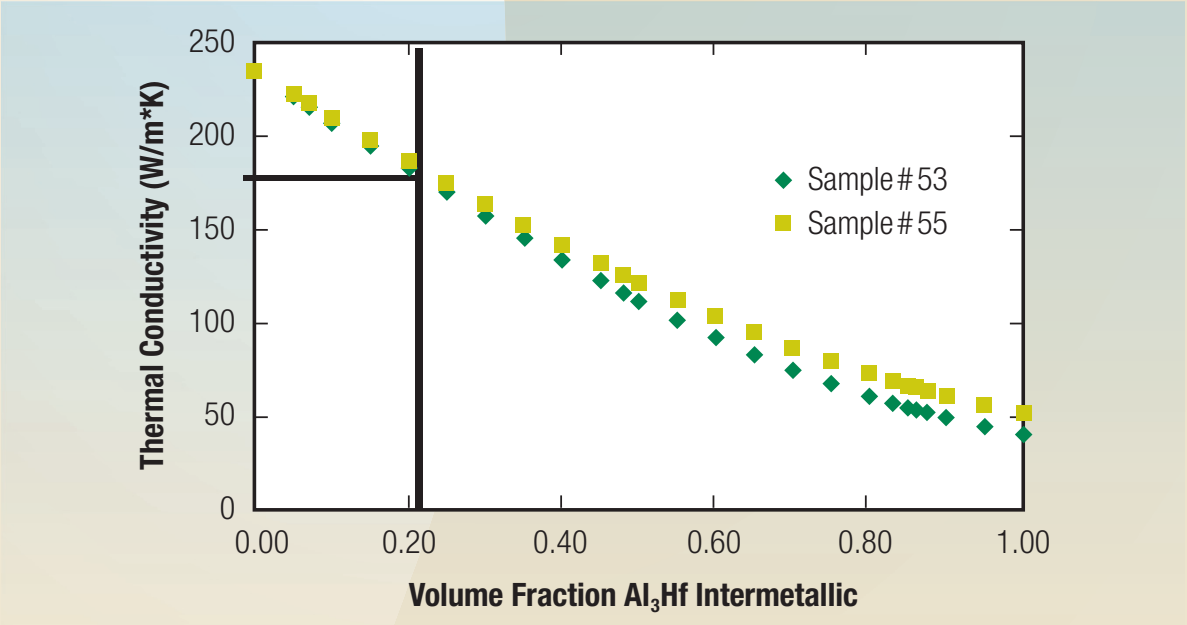
Samples will be prepared and characterized according to the irradiation test plan and inserted into the Advanced Test Reactor for testing before the end of 2010.

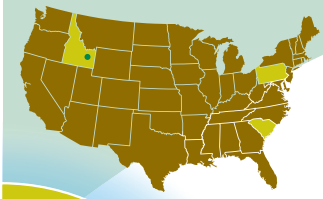
## Publications, Presentations and Patents

- D. P. Guillen, D.L. Porter, J.R. Parry, H. Ban, In-Pile Experiment of a New Hafnium Aluminide Composite Material to Enable Fast Neutron Testing in the Advanced Test Reactor, ANS ICAPP 10, San Diego, CA, June 13-17, 2010, Paper 10115.
- H. Wampler, A. Gerth, H. Ban, D.P. Guillen, D.L. Porter, C. Papesch and T. Hartmann, Fabrication and Characterization of a Conduction Cooled Thermal Neutron Filter, ANS ICAPP 10, San Diego, CA, June 13-17, 2010, Paper 10118.
- Irradiation Test Plan for USU ATR NSUF Project: Irradiation Effect on Thermophysical Properties of a Hafnium-Aluminide Composite: A Concept for Fast Neutron Testing at ATR, DOE Document ID: PLN-3269, March 15, 2010.
- Post Irradiation Examination Plan for USU ATR NSUF Project: Irradiation Effect on Thermophysical Properties of a Hafnium-Aluminide Composite: A Concept for Fast Neutron Testing at ATR, DOE Document ID: PLN-3446, March 23, 2010.

> *"This project is so fascinating that I decided to stay on as a Utah State University masters student."*
>
> — Adam Gerth, graduate student,
> Mechanical and Aerospace Engineering, Utah State University

*Figure 1. The graph shows the thermal conductivity for two samples of $Al_3Hf$ in an Al matrix at different volume fractions: sample #55 ($Al_3Zr$ type) with predominantly high-temperature phase and sample #53 ($Al_3Ti$ type) with low-temperature phase.*





project at-a-glance

# Advanced Damage-Tolerant Ceramics: Candidates for Nuclear Structural Applications

M. W. Barsoum, principal investigator (Drexel University)
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## Introduction

The MAX phases, a new class of machinable ternary carbides and nitrides (Figures 1-4), have potential applications for use in next generation nuclear reactors. All MAX phases are fully machinable despite the fact that some of them such as $Ti_3SiC_2$ and $Ti_2AlC$, are roughly as dense as titanium metal, but three times as stiff.

## Project Description

The objective of this project is to investigate the damage response of titanium silicon carbide ($Ti_3SiC_2$) and titanium aluminum carbides ($Ti_3AlC_2$ and $Ti_2AlC$) after they are exposed to a spectrum of irradiation consistent with light water reactor conditions.

The ternary carbide samples will be exposed to a series of neutron fluence levels at moderate to high irradiation temperatures in the Advanced Test Reactor. The evolution of the damage microstructure and the mechanical and electrical properties will be characterized.

The results will provide an initial database that can be used to assess the microstructural response and mechanical performance of these layered machinable ternary carbides.

## Accomplishments

Samples were synthesized at Drexel University and certified by an external company. The samples were machined and the experimental task plan was drafted. All samples are now ready to be inserted into the Advanced Test Reactor.

## Future Activities

After the experimental test plan is completed, the machined samples will be placed in the reactor during 2010 and irradiated. Testing before irradiation and PIE activities will include:

- Measurement of mechanical and electrical properties
- Microstructural characterization, including TEM.

## Publications, Presentations and Patents

- E. N. Hoffman, M. W. Barsoum, R. L. Sindelar, D. Tallman. 2010. MAX Phases and Their Potential for Nuclear Reactor Applications American Nuclear Society: 2010 Annual Meeting. San Diego, CA June 13-17, 2010

**Table 1. Project Details — Advanced Damage-Tolerant Ceramics: Candidates for Nuclear Structural Applications**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| **Materials** | **Description** |
| • $Ti_3SiC_2$<br>• $Ti_3AlC_2$<br>• $Ti_2AlC$ | samples machined; experimental test plan drafted |
| **Team Members/Collaborators** | |
| • Drexel University — Michel W. Barsoum (principal investigator); Darin Tallman (graduate student)<br>• Savannah River National Laboratory — E. N. Hoffman, R.L. Sindelar (collaborators)<br>• INL — Jian Gan (principal investigator) | |

> *" This is a new and exciting area for us to get involved in. We are very confident that some of the materials we are testing herein will turn out to be exceptionally tolerant to irradiation."*
>
> — Michel Barsoum, Ph.D., A. W. Grosvenor Professor, Department of Materials Science and Engineering, Drexel University

*Figure 1. An illustration below shows the unit cells of: (a) a $M_2AX$ phase, b) a $M_3AX_2$ phase and c) a $M_4AX_3$ phase.*

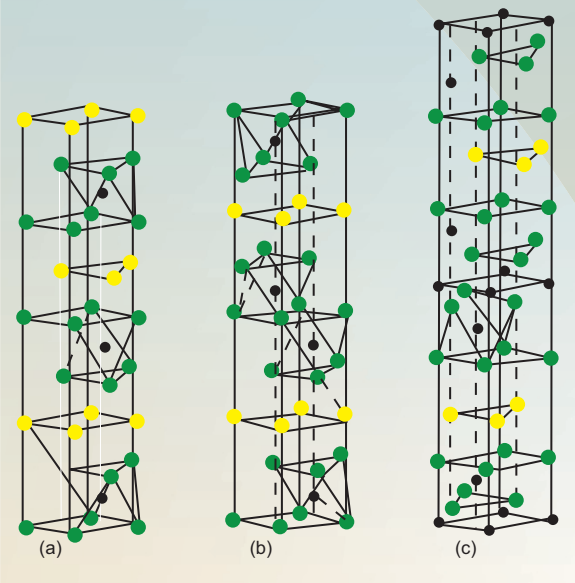


*Figure 2. A photo on the cover of the September 1997 issue of Journal of the American Ceramic Society shows the extraordinary extent that individual ultra-large grained $Ti_3SiC_2$ samples can deform at room temperature.*

*Figure 3. A closeup shows a bridged crack in a coarse-grained $Ti_3SiC_2$ sample tested at room temperature.*

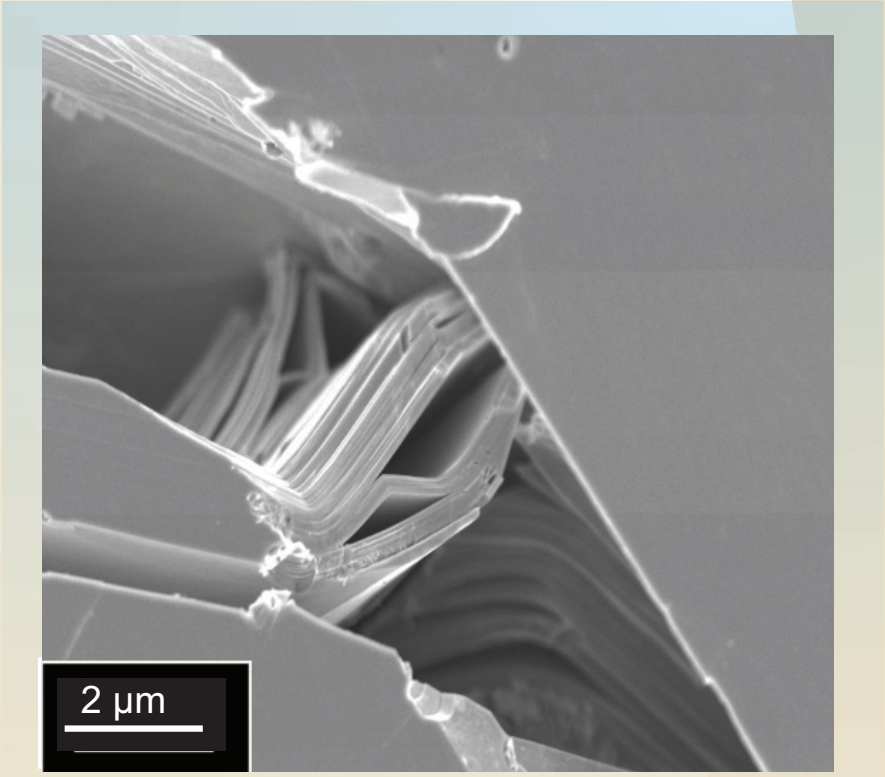
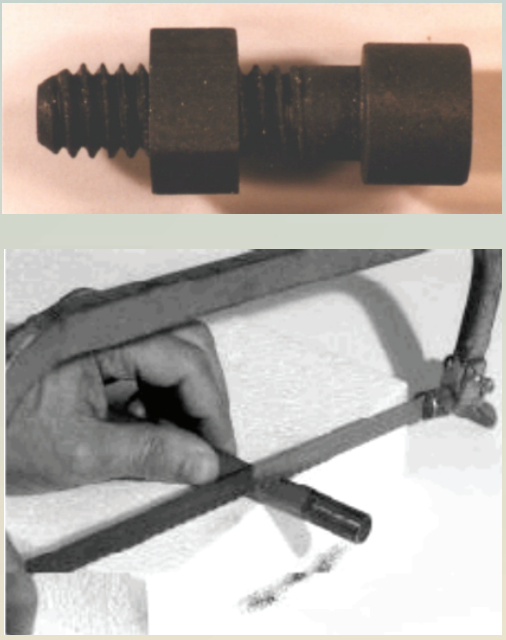
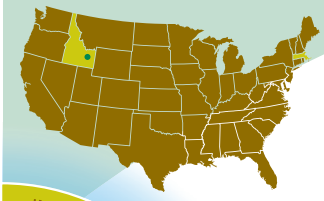



*Figure 4. A nut and screw machined out of $Ti_3SiC_2$ can be cut with a manual hack saw, showing that all the MAX phases are most readily machinable in their final fully dense state.*





project at-a-glance

# Irradiation and Examination Program for Triplex SiC Composite Tubing for Light Water Reactor Fuel Cladding Applications

Mujid S. Kazimi, principal investigator (Massachusetts Institute of Technology)
e-mail: kazimi@mit.edu

## Introduction

Longer-life fuels will be needed to extend the lifetime of light water reactors and improve their economic performance. A key element is improved pressurized water reactor fuel cladding as a replacement for zirconium alloy fuel cladding. Using silicon carbide (SiC) composite materials — Triplex Ceramic Cladding, an SiC/SiC composite — as cladding offers a variety of potential benefits. These benefits include better fuel behavior in loss of coolant accidents (LOCAs), departure from nucleate boiling transients and increased fuel burn-up. These benefits are the result of SiC's inherent high temperature strength and radiation resistance as well as the engineered features of the cladding.

If performance advantages can be demonstrated, then improved fuel can be designed to enhance the safety, reliability and economy of commercial reactors.

## Project Description

The primary objective of this project is to:

- Expose a variety of candidate tubing materials and bonding methodology samples to pressurized water reactor conditions (300°C, 10 MPa) in an in-core loop in the MIT research reactor
- Characterize the corrosion behavior and mechanical property evolution of the candidate samples.

| Table 1. Project Details — Irradiation and Examination Program for Triplex SiC Composite Tubing for Light Water Reactor Fuel Cladding Applications | |
|---|---|
| **ATR NSUF and Partners – Facilities and Capabilities** | |
| MIT | MIT Research Reactor (PIE activities also at MIT) |
| **Materials** | **Description** |
| • SiC/SiC composite "Triplex Ceramic Cladding" tubing | Samples irradiated to pressurized water reactor conditions (300°C, 10 MPa); required 12 months operation for approximately 1 dpa |
| **Team Members/Collaborators** | |
| • MIT — Mujid S. Kazimi (principal investigator); Gordon Kohse, Yakov Ostrovsky, Sung Joong Kim (co-investigators); David M. Carpenter (Ph.D. candidate); Jacob P. Dobisesky, John D. Stempien (M.S. candidates); Uuganbayar Otgonbaatar (undergraduate student)<br>• Ceramic Tubular Products, LLC — H. Feinroth (collaborator)<br>• Electric Power Research Institute — K. Yueh (collaborator)<br>• Westinghouse Electric Company, LLC — E. Lahoda (collaborator)<br>• INL — Mitchell Meyer (principal investigator) | |

The tube samples will be provided by Ceramic Tubular Products and Westinghouse Electric Company. The companies have already produced several generations of triplex clad tubing, some already irradiated at an irradiation facility constructed with the help of DOE funding.

The triplex clad tubing is comprised of:

- An inner monolithic SiC layer, which effectively seals against release of fission product gases
- An SiC fiber-based composite middle layer, which provides mechanical strength and a "graceful failure mode" that retains solid fission products and maintains a coolable geometry even under LOCA conditions
- An outer layer of chemical vapor-deposited SiC, which provides corrosion resistance and protection against fretting and debris damage.

## Accomplishments

The first irradiation phase (under pressurized water reactor conditions) began in June 2009 and continued through early December 2009. The samples that were exposed included:

- A set of nine samples from round 6-generation tubing, which were already irradiated for approximately one year in the in-core loop at the MIT reactor
- A set of twelve samples of non-irradiated round 7-generation tubing (which represents the current state-of-the-art in triplex tubing and incorporates refinements that resulted from previous irradiations and other testing).

Interim post-irradiation examinations, weight loss measurements, visual examinations and photography to document the physical state of the tubing were performed in December 2009 and January 2010. Figure 1 shows typical round 6- and round 7-generation tube samples after the initial irradiation period.

After members of the project team evaluated the interim results, a second irradiation period began in March 2010.

Eight of the round 7-generation samples were returned for further exposure. Three pairs of bond test samples were also added.

### Future Activities

Ongoing irradiation will continue until the end of April 2010, when there will be an extended shutdown of the MIT reactor for major maintenance and heat exchanger upgrades. During the outage, interim examinations will be conducted on the bond test samples.

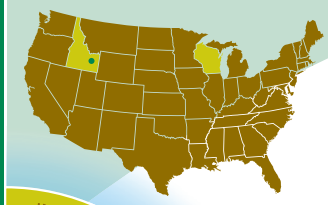
When further irradiation can continue, it will include samples with bonds that remained intact and samples from the round 6- and 7-generation set as agreed upon with our collaborators. This irradiation will continue from December 2010 to June 2011.

Further post-irradiation examination, including mechanical property testing and SEM, will be performed on the Round 6- and 7-generation samples that are not returned to irradiation.

*"This project has enabled us to significantly expand examination of the innovative idea of using a ceramic material as the cladding of nuclear fuel, instead of the traditional zirconium alloys. It is an example of what can be produced in a collaboration among universities, national laboratories and industry. If successful, this will enable extraction of more energy from the fuel before it has to be discharged from the reactor. It might even be possible to think of fuel that will operate at higher power densities, thus reducing the cost of energy production."*

— David M. Carpenter, Ph.D. candidate,
Department of Nuclear Science & Engineering,
Massachusetts Institute of Technology

*Figure 1. Triplex SiC composite clad tube samples — round 7-generation (near right) and round 6-generation (far right) — after Phase 1 irradiation under pressurized water reactor conditions in an in-core loop at the MIT reactor.*



project at-a-glance

# Radiation Stability of Ceramics for Advanced Fuel Applications

Yong Yang, principal investigator (University of Wisconsin–Madison; currently located at University of Florida)
e-mail: yongyang@ufl.edu

## Introduction

High-temperature gas-cooled fast reactors will require advanced materials, like some ceramics, that show good stability under higher temperatures. Examining the microstructural evolution of proposed ceramic materials under neutron irradiation at high temperatures could be the basis for a better understanding of their potential stability in gas-cooled fast reactors.

## Project Description

The objective of the project is to examine the impact of neutron irradiation on the microstructure of proposed candidate ceramics — zirconium carbide (ZrC), titanium carbide (TiC), zirconium nitride (ZrN) and titanium nitride (TiN) — that were irradiated in the Advanced Test Reactor to 1 dpa at 800ºC.[1]

The samples — 3 mm discs and 20 mm long rods — will be examined using TEM to understand the effect of radiation on lattice stability, phase change, void growth and development of other microstructural features, such as dislocation loops and stacking fault tetrahedra.

PIE activities will also include micro-hardness tests and immersion density measurements.

## Accomplishments

To prepare samples for TEM analysis, disks were dimpled to about 10-µm thick at the center and then finished to electron transparency using an ion mill (Figure 1). Besides controlling loose contamination, this sample preparation method is highly repeatable.

To perform the micro-hardness measurement, a well-polished flat surface was developed by using a 25-mm diameter stainless steel flattening wheel on the dimpling grinder. The sample was left with a dished surface with less than 10-µm depth and a surface finish to 0.05 µm.

Along with the development of sample preparation techniques, a new water chiller and a new objective aperture assembly were installed on the JEOL 200CX TEM.

## Future Activities

Samples will be prepared for the irradiated microstructure study, including lattice stability, phase stability and voids and dislocation structures, and TEM will be performed.

The micro-hardness test samples will also be prepared using the developed procedure and the micro-hardness will be evaluated using a Vickers hardness test.

The density measurement will be performed at INL while a similar capability is also developed for the UW-M radioactive examination facility. Finally, the microstructural comparison between the neutron and proton irradiated samples will be made and analyzed.

**Table 1. Project Details — Radiation Stability of Ceramics for Advanced Fuel Applications**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | PIE facilities |
| UW–M | PIE facilities |
| **Materials** | **Description** |
| • ZrC<br>• TiC<br>• ZrN<br>• TiN | Samples previously irradiated at Advanced Test Reactor to 1 dpa at 800°C |
| **Team Members/Collaborators**[a] | |
| • UW-M — Yong Yang (principal investigator); Clayton Dickerson (graduate student); Robert Agasie (director, UW-M reactor laboratory)<br>• INL — Jian Gan (principal investigator), David Duncan (project manager) | |

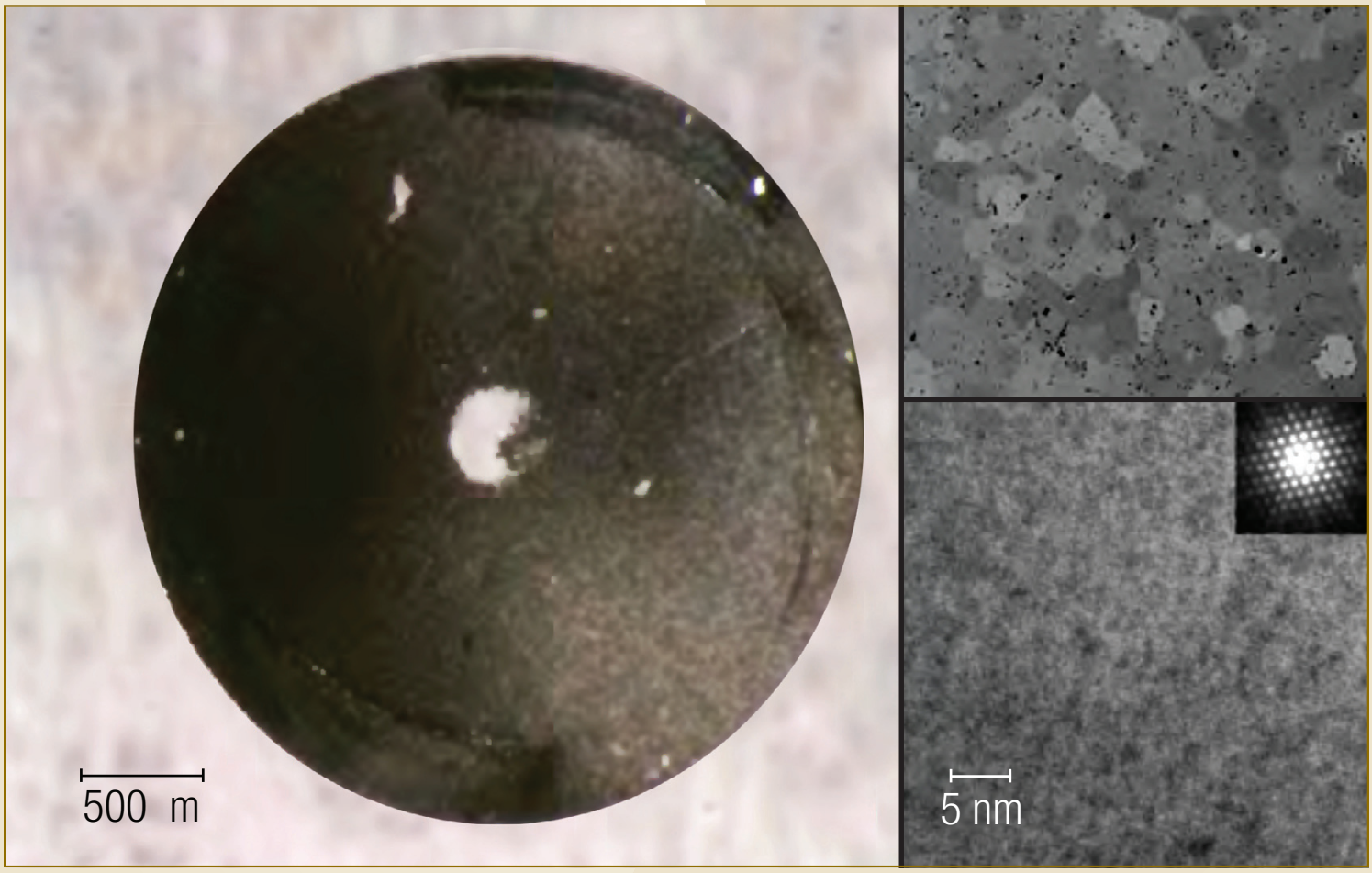
Note: A related ATR NSUF project, "Advanced Non-Destructive Assessment Technology to Determine the Aging of Silicon-Containing Materials for Generation IV Nuclear Reactors," which is using ion irradiation to study the radiation response of these ceramic materials, is discussed on page 12 of this report.

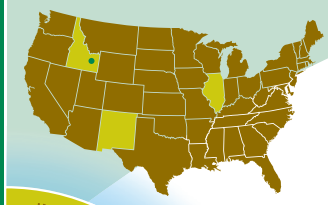
> *"The new designation of ATR as a National Scientific User Facility can really charge our research with neutrons."*
>
> — Yong Yang, Assistant Professor, Nuclear and Radiological Engineering, University of Florida

## References


1. Terry, J., et al., "Synchrotron X-Ray Diffraction and X-Ray Absorption Fine Structure Study of Irradiated Binary Carbides". This report, pp. 28.


*Figure 1. A ZrC TEM sample was prepared using the dimpling and ion milling method.*







# Study of an Irradiated Ferritic Steel by Synchrotron X-Ray Diffraction and X-Ray Absorption Spectroscopy

Meimei Li, principal investigator (Argonne National Laboratory)
e-mail: mli@anl.gov

## Introduction

The advanced power generation industry requires radiation-sustained materials with appropriate thermal and mechanical properties. Ferritic and martensitic stainless steel alloys are candidate materials due to their superior properties: higher resistance to radiation-induced swelling, higher thermal conductivity, lower thermal expansion, good oxidation and corrosion resistance at elevated temperatures.

Using new synchrotron radiation techniques to examine advanced ferritic/martensitic alloys will provide valuable insight into the future applications of synchrotron radiation for nuclear materials research. The ultra-brilliant third-generation synchrotron x-rays are an invaluable tool for non-destructive examination of nuclear fuels and materials.

## Project Description

The objective of this study is to explore using synchrotron x-ray absorption spectroscopy (XAS) and x-ray diffraction (XRD) for characterizing radiation-induced microstructural changes in advanced ferritic-martensitic steels.

Samples of the material used for this project, including a mod.9Cr-1Mo steel, were obtained from the pre-irradiated ATR NSUF sample library.

## Accomplishments

Some of the samples were irradiated to 1, 4 and 10 dpa at 32–57°C, to a mixed proton and spallation neutron flux, at the Los Alamos National Laboratory (LANL) Neutron Science Center. After the irradiated samples were retrieved from the LANL hot cell facility, they were measured, cleaned, packaged and shipped to the Argonne National Laboratory Irradiated Materials Laboratory where they were unpacked, measured, cleaned and cut to small pieces to reduce radioactivity. Both non-irradiated and irradiated samples were individually mounted in triple-contained sample holders then transferred to the Advanced Photo Source. The entire process was completed within three weeks.

Synchrotron XAS measurements were carried out on the non-irradiated and irradiated samples at the Advanced Photon Source Materials Research Collaborative Access Team (MRCAT) beamline 10-ID.

The extended x-ray absorption fine structure (EXAFS) spectroscopy data at the K-edges of the iron (Fe), chromium (Cr), molybdenum (Mo) and niobium (Nb) were obtained and the local structure close to the x-ray absorbing atom determined. The graphs in Figure 1 show the Fourier transformations of the K-edges EXAFS spectra as function of the dose.

The information on the nearest neighbors of the solute atoms enables a better understanding of the radiation defect structure and distribution in this complex alloy.

## Future Activities

The project is considered complete. However, in future proposals, synchrotron XRD measurements will be undertaken to complement electron microscopy and synchrotron XAS characterization. Together, these methods will characterize microstructural changes that occur under irradiation. For a complex alloy like mod.9Cr-1Mo ferritic-martensitic steel, a variety of advanced characterization tools are required to fully understand irradiation defect production and accumulation and irradiation-induced/enhanced precipitation or dissolution.

**Table 1. Project Details — Study of an Irradiated Ferritic Steel by Synchrotron X-Ray Diffraction and X-Ray Absorption Spectroscopy**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | PIE facilities |
| IIT | Advanced Photon Source |
| **Materials** | **Description** |
| • Ferritic/martensitic steel (mod.9Cr-1Mo) | Samples irradiated to 1, 4, 10 dpa at 32-57°C (to a mixed proton and spallation neutron flux) at LANL |

**Team Members/Collaborators**[a]

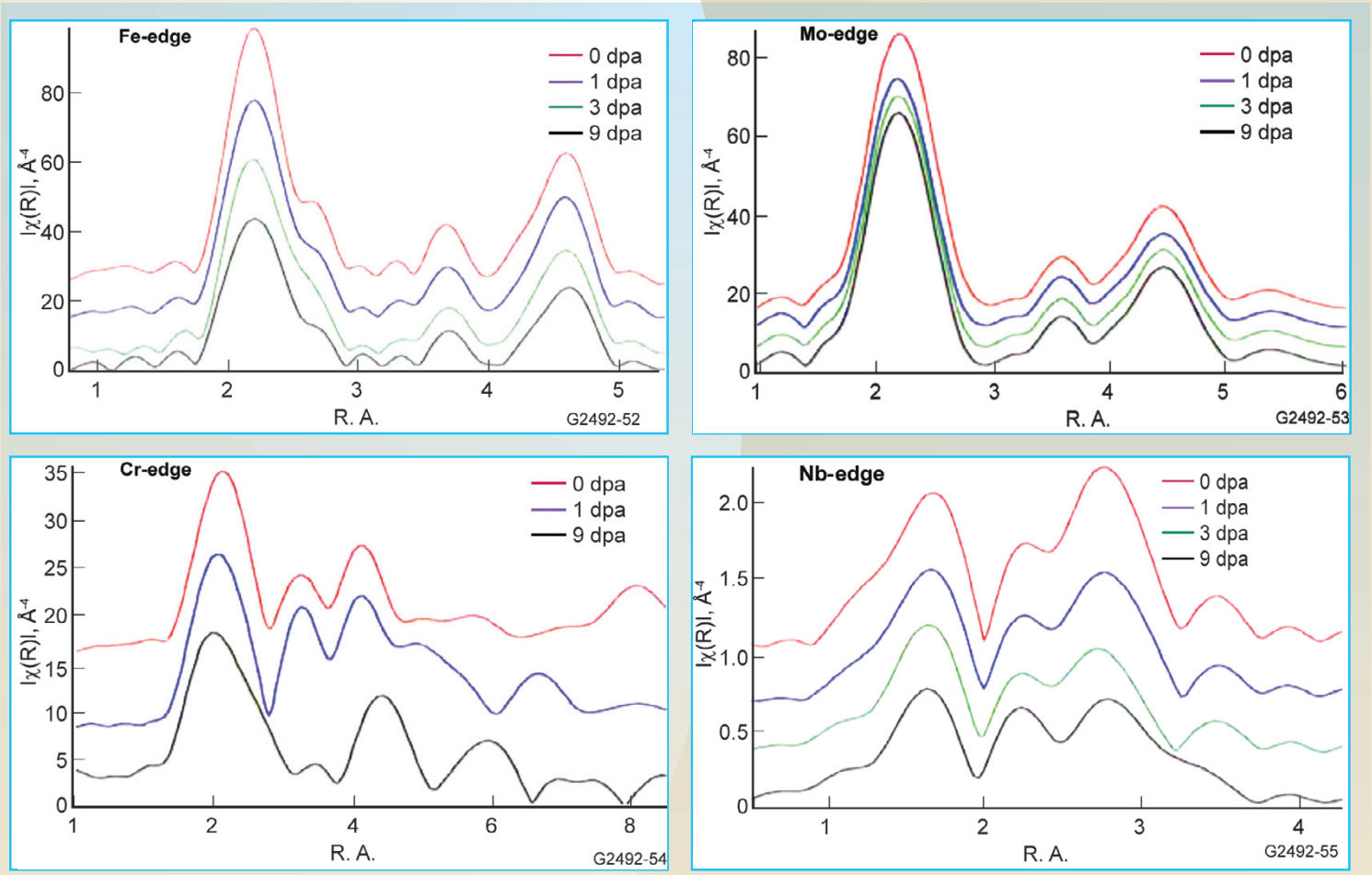
- Argonne National Laboratory — Meimei Li (principal investigator)
- IIT — Jeff Terry (principal investigator); Yulia Trenikhina, Dan Olive, Hasitha Ganegoda (graduate students)
- INL — Mitchell Meyer (collaborator)
- Los Alamos National Laboratory — Stuart Maloy (collaborator)

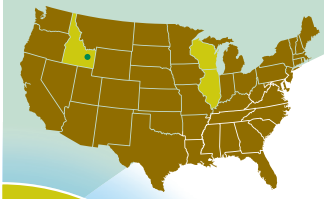
## Publications, Presentations and Patents

- Meimei Li, Yulia Trenikhina, Dan Olive, Hasitha Ganegoda, Jeff Terry and Stuart A. Maloy, "Study of Irradiated Mod.9Cr-1Mo Steel by Synchrotron XAS," ANS Transactions, June 2010.

> *"It was no miracle [we could complete the research so quickly]; it only happened because we got great support from a lot of people at the ATR NSUF, Argonne National Laboratory and Los Alamos National Laboratory. This helped us to [accomplish a lot] in a very short amount of time."*
>
> — Meimei Li, Nuclear Engineering Division, Argonne National Laboratory

*Figure 1. The graphs below show Fourier transformations of the Fe–, Mo–, Cr– and Nb–edges EXAFS spectra as functions of dose in irradiated mod.9Cr-1Mo steel.*





project at-a-glance

# Synchrotron X-Ray Diffraction and X-Ray Absorption Fine Structure Study of Irradiated Binary Carbides

Jeff Terry, principal investigator (Illinois Institute of Technology)
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## Introduction

In complex alloys, radiation-induced segregation and the creation of other stable phases can lead to irradiation hardening and embrittlement. Radiation damage can include the addition of phases in the material, irradiation defect clusters, radiation-produced precipitates, chemical changes and voids in the irradiated samples.

The mechanisms of damage are much less clear in simple binary carbide and nitride compounds. The information that is obtained from this study will help predict which elements may have better radiation tolerance as constituents of ternary carbides.

## Project Description

The project's objective is to characterize the mechanisms of radiation damage in simple binary carbide and nitride compounds. Damage will be tracked as a function of the atomic number (Z) of the anions carbon (C) and nitrogen (N) in relation to the constituent cations — zirconium (Zr), aluminum (Al), titanium (Ti) and silicon (Si).

The sample materials included aluminum nitride (AlN), silicon carbide (SiC), titanium carbide (ZrC), titanium nitride (ZrN), zirconium carbide (ZrC) and zirconium nitride (ZrN). The materials were subjected to neutron irradiation in the Advanced Test Reactor at 800°C to 1 dpa.[1]

**Table 1. Project Details — Synchrotron X-Ray Diffraction and X-Ray Absorption Study of Irradiated Binary Cables**

| ATR NSUF and Partners – Facilities and Capabilities | |
|---|---|
| INL | Advanced Test Reactor, PIE facilities |
| UW–M | PIE facilities |
| IIT | Advanced Photon Source |
| **Materials** | **Description** |
| • AlN<br>• SiC<br>• TiC<br>• TiN<br>• ZrC<br>• ZrN | Samples irradiated to 1 dpa at 800°C |
| **Team Members/Collaborators** | |
| • IIT — Jeff Terry (principal investigator); Hasitha Ganegoda, Dan Olive, Yulia Trenikhina (graduate students in physics)<br>• INL — Mitchell Meyer (principal investigator)<br>• UW-M — collaborated in obtaining and mounting sample materials | |

## Accomplishments

The samples were mounted in approved triple containment sample cells and shipped to the Advanced Photon Source Materials Research Collaborative Access Team (MRCAT) beamline for x-ray measurements (Figure 1). Measurements were completed for the ZrC and ZrN samples only; it was not possible to complete measurements for all the samples within the two-day beam time allocated for the project.

While the measurements show similar results for both ZrC and ZrN (Figure 2), the main difference is that shorter inter-atomic distances were observed for ZrN. The initial observations indicated that at irradiation up to 1 dpa, no new phases formed in the binary Zr materials. The ZrC retained the cubic sodium chloride (NaCl) structure.[2]

Within experimental error, there was no expansion of the lattice under these irradiation conditions, which is consistent with results on ion-irradiated ZrC.1[1] However, while the overall structure is retained, there is a loss of coordination at larger interatomic distances, which is consistent with radiation effects in materials with low–Z members.

It is difficult to end up with many unterminated Zr atoms in a material of this type. Adventitious carbon will usually find unterminated Zr created by irradiation and terminate the Zr–C bond. This results in the number of nearest neighbors remaining saturated, as was observed in this study. However, at larger inter-atomic distances, the coordination is significantly reduced. This indicates that defect sites have resulted in smaller regions of perfect crystallinity.

## Future Activities

The project has been completed.

Future proposals will be written to complete the analysis of the ZrC and ZrN spectroscopic data and compare it with TEM analyses. The team expects to make x-ray diffraction (XRD) measurements on the samples of ZrC and ZrN as well as on samples of TiC, TiN, AlN and SiC. Modeling will determine the nature and

density of defect sites. Combining the theoretical modeling of the x-ray absorption fine structure (XAFS) spectroscopy data with TEM observations will allow loss of coordination to be correlated with observed grain size, dislocation loop formation or other observed defects.

> *"This experiment required great coordination and cooperation between multiple institutions. The University of Wisconsin–Madison could not have been more helpful by providing a facility where the samples could be mounted."*
>
> — Jeff Terry, Ph.D., Assistant Professor of Physics, Illinois Institute of Technology

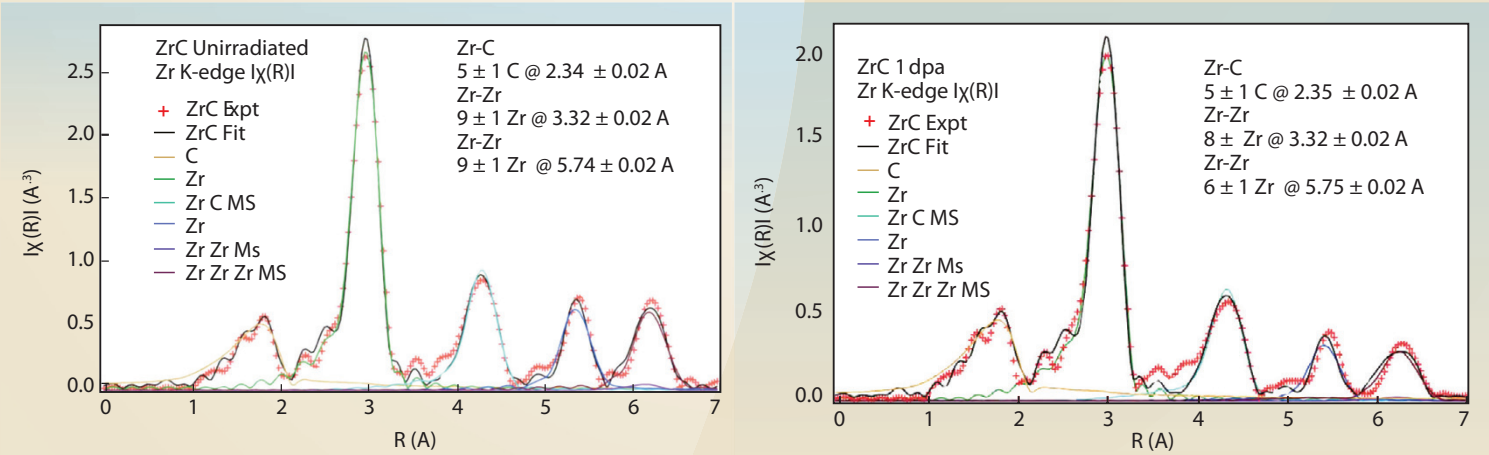
## References


1. Yang, Y. et al., "Radiation Stability of Ceramics for Advanced Fuel Applications". This report, pp. 24.

2. Gosset, D., et al., "Structural evolution of zirconium carbide under ion irradiation." Journal of Nuclear Materials 373, 123 (2008).

*Figure 1. An aerial view shows the Advanced Photon Source at Argonne National Laboratory. The large building in the left foreground is the Center For Nanoscale Materials, one of five DOE nanoscale science research centers.*

*Figure 2. Measurements of ZrC and ZrN show similar results, though shorter interatomic distances were observed for ZrN.*



# Summary of All Awarded Projects (FY 2008 – FY 2010)

## FY 2008 Projects

**Irradiation Test Plan for the ATR National Scientific User Facility (Pilot Project)**

Proposed by: Kumar Sridharan, University of Wisconsin–Madison

DOE-NE need addressed: Advanced Structural Materials

**Nonstoichiometric Spinel as Inert Matrix** *(also titled "Irradiation Behavior of Oxide Ceramics for Inert Matrices")*

Proposed by Juan Nino, University of Florida

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**Irradiation Performance of Fe-Cr Base Alloys**

Proposed by James Stubbins, University of Illinois

DOE-NE need addressed: Advanced Structural Materials

**Influence of Fast Neutron Irradiation on the Mechanical Properties and Microstructure of Nanostructured Metals/Alloys**

Proposed by K. L. Murty, North Carolina State University

DOE-NE need addressed: Advanced Structural Materials

**Advanced Non-Destructive Assessment Technology to Determine the Aging of Structural Materials for Generation IV Nuclear Reactor**

Proposed by David Olson, Colorado School of Mines

DOE-NE need addressed: Advanced Structural Materials

**Characterization of the Microstructures and Mechanical Properties of Advanced Structural Alloys for Radiation Service: A Comprehensive Library of Advanced Test Reactor Irradiated Alloys and Specimen**

Proposed by Robert Odette, University of California, Santa Barbara

DOE-NE need addressed: Advanced Structural Materials

## FY 2009 Projects

**Radiation Stability of Ceramics for Advanced Fuel Applications**

Proposed by Yong Yang, University of Wisconsin-Madison

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**High Fluence Embrittlement Database and Advanced Test Reactor Irradiation Facility for Light Water Reactor Vessel Life Extension**

Proposed by Robert Odette, University of California, Santa Barbara

DOE-NE need addressed: Advanced Structural Materials

**Irradiation Behavior of Triplex SiC/SiC Tubing Under Pressure Water Vessel (PWR) Conditions**

Proposed by Mujid Kazimi, Massachusetts Institute of Technology

DOE-NE needs addressed: Advanced Structural Materials, Improved/Advanced Nuclear Fuels

**Irradiation Effect on Thermophysical Properties of Hafnium-Aluminide ($Hf_3Al$-Al) Composite: A Concept for Fast Neutron Testing at Advanced Test Reactor**

Proposed by Heng Ban, Utah State University

DOE-NE needs addressed: Advanced Structural Materials

**Real-time Advanced Test Reactor-Criticality (ATR-C) Flux Sensors**

Proposed by George Imel, Idaho State University

DOE-NE needs addressed: Advanced Instrumentation and Controls, Advanced Structural Materials, Improved/Advanced Nuclear Fuels

**Advanced Damage Tolerant Ceramics: Candidates for Nuclear Structural Applications**

Proposed by Michel W. Barsoum, Drexel University

DOE-NE need addressed: Advanced Structural Materials

# Summary of All Awarded Projects (FY 2008 – FY 2010)

## FY 2010 Projects

**Measurement of Actinide Neutronic Transmutation Rates with Accelerator Mass Spectroscopy**

Proposed by George Imel, Idaho State University

DOE-NE needs addressed: Advanced Structural Materials, Improved/Advanced Nuclear Fuels

**Hydride Light Water Reactor (LWR) Fuel Rod Irradiation**

Proposed by Donald Olander, University of California, Berkeley

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**Development and Validation of an Advanced Test Reactor-Criticality (ATR-C) Radiation Transport Model**

Proposed by Denis Beller, University of Nevada, Las Vegas

DOE-NE need addressed: Advanced instrumentation and controls

**Low Fluence Behavior of Metallic Fuels**

Proposed by Yongho Sohn, University of Central Florida

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**Radiation-Induced Segregation/Depletion at Grain Boundaries in Neutron-Irradiated 304 Stainless Steel (304SS) at Low Dose Rates**

Proposed by Emmanuelle Marquis, University of Michigan

DOE-NE need addressed: Advanced Structural Materials

**Multi-scale Investigation of the Influence of Grain Boundary Character on Radiation-Induced Segregation (RIS) and Mechanical Behavior in Light Water Reactor (LWR) Steels**

Proposed by Mitra Taheri, Drexel University

DOE-NE need addressed: Advanced Structural Materials

**High Temperature In-Pile Irradiation Test of Single Phase $U_3Si_2$**

Proposed by Darryl Butt, Boise State University

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**Irradiation Behavior and Performance of a Uranium-Zirconium Metal Alloy Fuel**

Proposed by Sean McDeavitt, Texas A&M University

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**Investigation of the Thermodynamics of Plutonium/Iron Solubility in Brines Using X-Ray Absorption Spectroscopy (XAS)**

Proposed by Donald Reed, Los Alamos National Laboratory

DOE-NE need addressed: Stable Waste Forms

**Synchrotron X-Ray Diffraction Measurements of Spatially Resolved Strain Fields in Nuclear Fuel Plates**

Proposed by Maria Okuniewski, Idaho National Laboratory

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**Study of an Irradiated Ferritic Steel by Synchrotron X-Ray Diffraction (XRD) and X-Ray Absorption Spectroscopy (XAS)**

Proposed by Meimei Li, Argonne National Laboratory

DOE-NE need addressed: Advanced Structural Materials

**Synchrotron X-Ray Diffraction (XRD) and X-Ray Absorption Fine-Structure (XAFS) Study of Irradiated Binary Carbides**

Proposed by Jeff Terry, Illinois Institute of Technology

DOE-NE need addressed: Advanced Structural Materials

## FY 2010 Rapid-Turnaround Experiments

**Microstructural and Irradiation Effects on Silver (Ag) and Cesium (Cs) Diffusion in Chemical Vapor Deposition Silicon Carbide (CVD-SiC)**

Proposed by Izabela Szlufarska, University of Wisconsin–Madison

DOE-NE need addressed: Improved/Advanced Nuclear Fuels

**Radiation Effects on Ceramic Coating of Advanced Cladding for Fast Reactors**

Proposed by Jian Gan, Idaho National Laboratory

DOE-NE needs addressed: Improved/Advanced Nuclear Fuels, Stable Waste Forms

**Developing a Mechanistic Understanding of Radiation Tolerant Materials**

Proposed by David Hoelzer, Oak Ridge National Laboratory

DOE-NE needs addressed: Advanced Structural Materials, Improved/Advanced Nuclear Fuels

Idaho National Laboratory