


2015 | ANNUAL REPORT
Nuclear Science User Facilities

NSUF
Nuclear Science
User Facilities

Nuclear Science User Facilities
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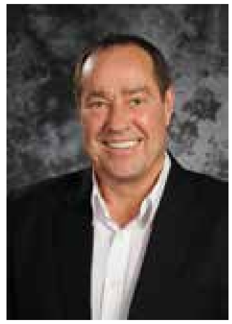
OUR NSUF TEAM



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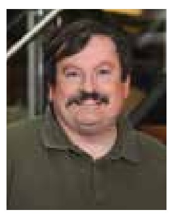

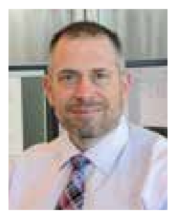

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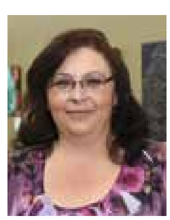

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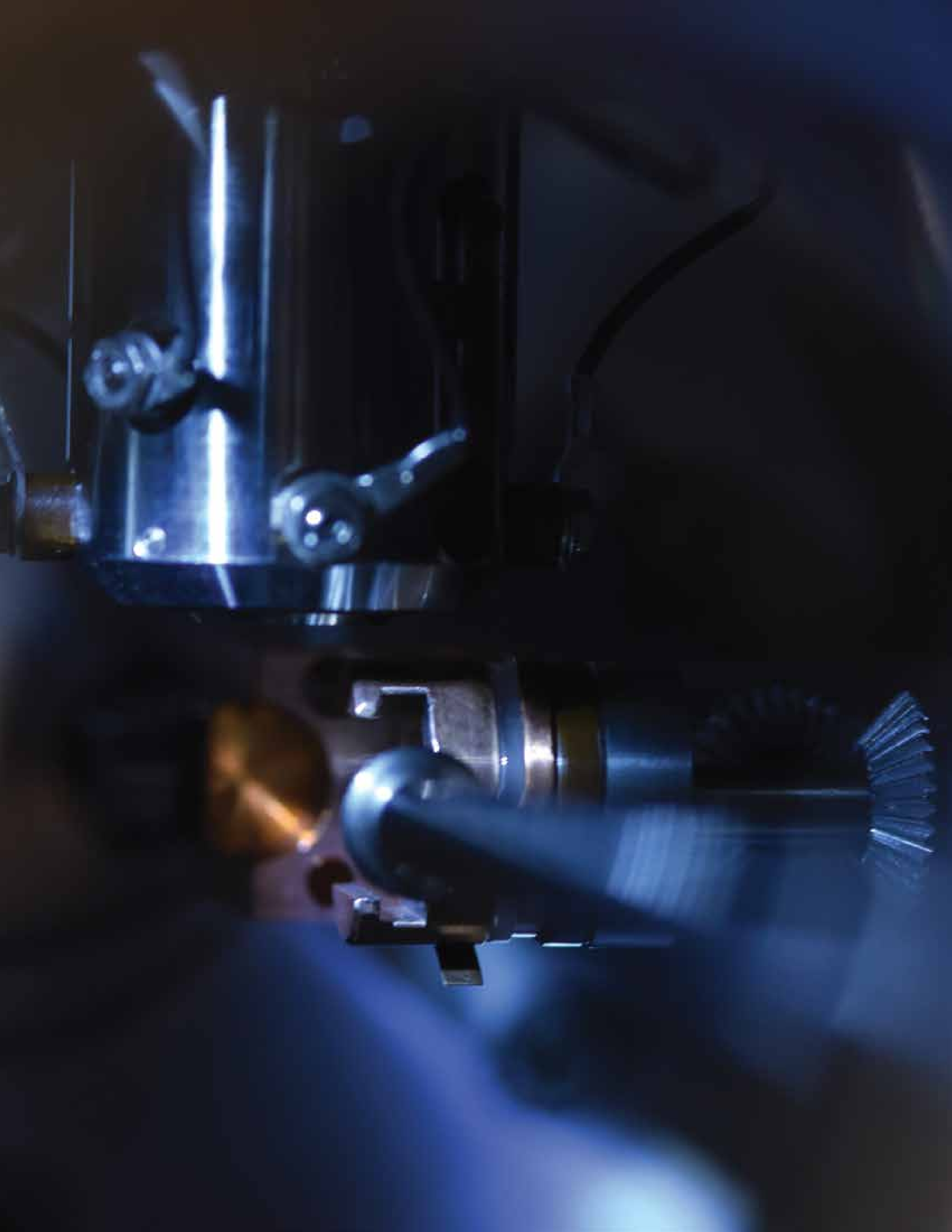

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FROM THE NSUF DIRECTOR

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In the eight years since the Nuclear Science User Facilities program was conceived, the staff has worked tirelessly to create a framework that enables nuclear energy research. This year, we have built on this framework with the creation of the Nuclear Energy Infrastructure Database. It's a web-based, searchable resource of nuclear research-related capabilities including reactors, instrumentation and other information from U.S. institutions. Interest appears to be robust with the expanding set of NEID users from 34 federal government and national laboratories, 22 universities and nongovernmental organizations and five industry organizations. The database was deployed in September 2015 and received a great deal of attention when it was promoted in November as part of the Gateway for Accelerated Innovation in Nuclear, or GAIN, initiative.

Speaking of GAIN, I'm excited about the NSUF's role in this important initiative. It will lead to easier access by business entities to technical and regulatory support to move new or advanced nuclear reactor concepts and designs toward commercialization while ensuring the continued safe, reliable and economic operation of the existing reactor fleet. That's a big deal for NSUF and falls right in line with our mission as the nation's only designated nuclear energy user facility. We will continue to facilitate fundamental research that will serve as a foundation for GAIN as it aims to elevate innovative concepts to a commercial readiness level.

As our program has matured, more users are learning about the opportunities we offer for nuclear research and, as such, more research is being proposed. In FY 2015, the NSUF conducted three solicitations for Rapid Turnaround Experiments and, for the first time, integrated its annual call into the 2015 Consolidated Innovative Nuclear Research Funding Opportunity Announcement. The RTE solicitations drew 47 proposals and resulted in 30 awards to researchers from 17 different institutions. The FOA drew 41 letters of intent with final submissions of 17 full applications resulting in five awards totaling $4.2 million in support. We expect to grow and be able to support more in FY 2016.

Our researchers are gaining scientific prominence by publicizing and documenting research results through peer-reviewed journals and presentations at conferences. We recorded the highest number of journal publications in NSUF history in FY 2015 with 33. That beat the previous high of 28 in 2013. We've also made it easier for people to access these publications online by modifying the NSUF website to include a field for a URL in the publication database. Now with just one click, it's possible to actually read or download an article instead of only being able to view a citation.

This year, we established dedicated technical leads to assist and partner with our users as they propose and conduct research. These leads – Thomas Maddock, Assel Aitkaliyeva and Donna Post-Guillen are some of INL's best and brightest and their expertise has been exceptionally valuable to our organization. You can read more about the tech leads and their individual scientific accomplishments in an article in this publication.

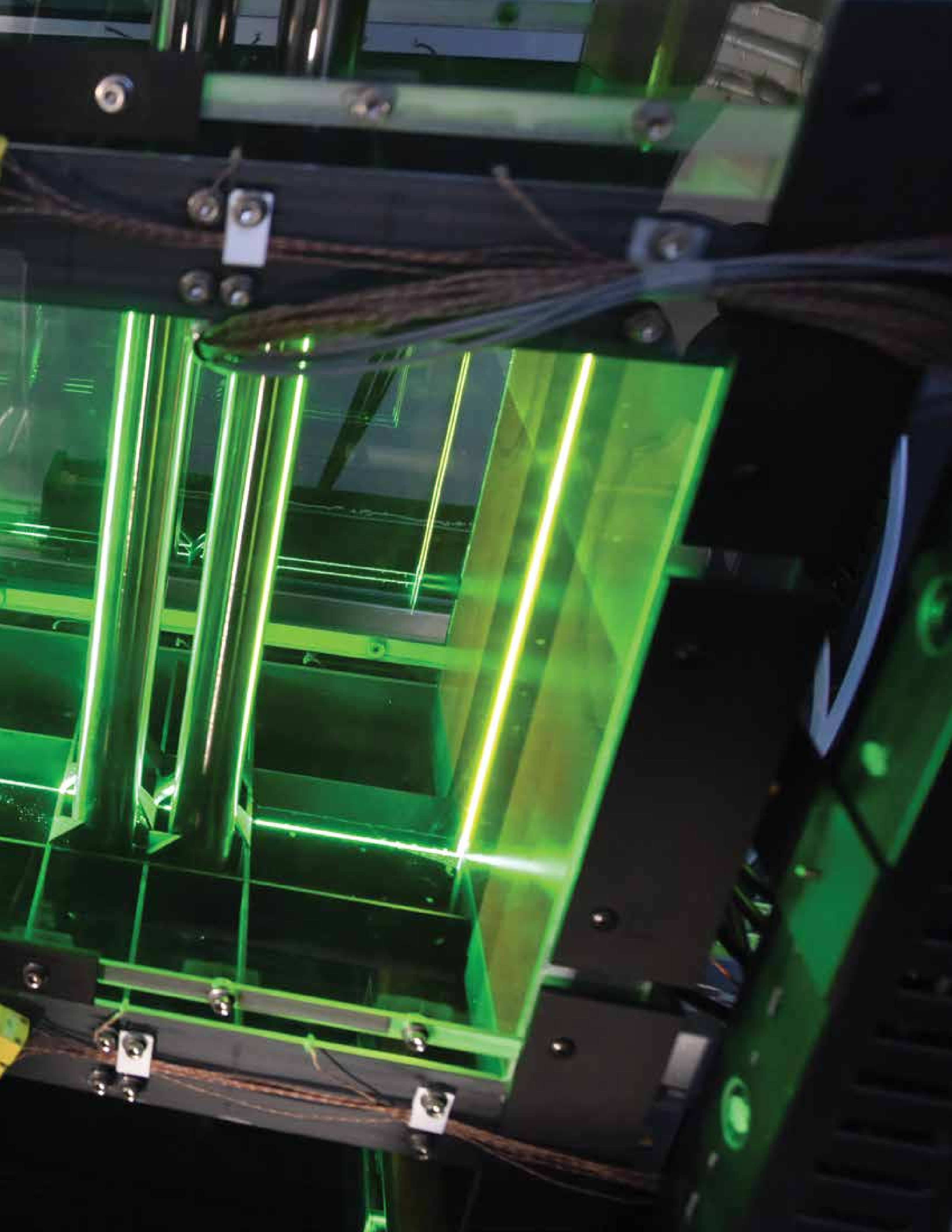
Finally, our Industry Program continues to receive very strong interest. In 2015, fracture toughness tests on irradiated light water reactor internal materials – alloys X-750 and XM-19 – were completed. The work, which supports the Electric Power Research Institute, represented the first use of new world-class Irradiation-Assisted Stress Corrosion Cracking facilities at INL's Materials & Fuels Complex to perform fracture toughness tests. The resulting data, when combined with previously obtained IASCC data, will represent the most complete picture on the irradiated mechanical properties for these alloys.

Looking ahead, I'd like to give you a brief preview of things to come in FY 2016. There is a major initiative to further enhance the Nuclear Fuels and Materials Library making it accessible online, searchable and coupled with the NEID. We will continue our efforts to bring more materials with good provenance into the library. An entirely new website that will incorporate a single sign-on for the proposal system and the NEID, as well as links to many other user-friendly features, will be rolled out soon. We are working to provide our users access to more capabilities, such as the Intermediate Voltage Electron Microscopy Tandem Facility at Argonne National Laboratory; as well as increasing our relationship with Office of Science user facilities, such as the National Synchrotron Light Source II (NSLS-II) at Brookhaven National Laboratory. International interest in nuclear energy user facilities is growing; therefore, we are expanding on our international relationships while seeking out new opportunities for collaboration.

Please take a few moments to learn more about all the important nuclear research facilitated by NSUF at Idaho National Laboratory and at our diverse mix of affiliated partner institutions across the U.S.

Sincerely,

Dr. J. Rory Kennedy
Director



Q&A WITH ALISON

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Q&A with the NSUF DOE-NE Program Manager Alison Hahn

Nuclear Science User Facilities fills a unique role for the Department of Energy as it is the only user facility overseen by the Office of Nuclear Energy: most of the other scientific user facilities fall under the Office of Science.

Alison Hahn has the important job of overseeing the program from DOE Headquarters; acting as a liaison between the NSUF leadership in Idaho Falls and DOE-NE leadership in Washington, D.C. She ensures that the program understands the objectives and priorities of the Office of Nuclear Energy and that the research performed by NSUF users addresses these goals.

Earlier this year, Hahn sat down with Sarah Robertson, the NSUF communications liaison, to discuss the NSUF.

Q: *What do you see as the NSUF's greatest accomplishments in 2015?*

A: The development of the interactive Nuclear Energy Infrastructure Database was probably the most visible and significant program accomplishment in 2015. The database concept was briefed to the Nuclear Energy Advisory Board (NEAC) and received highly positive feedback as it is designed to address fundamental research facilities and issues of interest to NEAC for over a decade. Also, the database was cited prominently in the Nov. 6, 2015, White House press release announcing U.S. government support and activities associated with the recently launched Gateway for Accelerated Innovation in Nuclear (GAIN) initiative.

We are also proud to have fully integrated, for the first time, the processes for receiving, evaluating and awarding NSUF access awards with requests for Office of Nuclear Energy programmatic research funding. If successful, this "one-stop shopping" approach will increase efficiency, reduce researcher uncertainty, and improve the transparency of the full range of NE research investment decisions.

Q: *What is the NEID and how do you foresee it benefiting DOE-NE?*

A: When fully populated, functioning and socialized, the NEID will address the decades long need of both the department and the nuclear energy research community to have ready access to reliable information on the whole range of supporting capabilities in the United States and, as appropriate, other parts of the world. The database will be especially impactful for its ability to provide not only capabilities information, but also schedule availability and points of contact.

Q: *Why is this information important to NE?*

A: This capability will allow NE to avoid making duplicative purchases through the CINR infrastructure solicitation and better leverage limited infrastructure funding to purchase the highest priority capabilities. In addition, gaps will be identified to assist in new infrastructure investments or CINR infrastructure work scope developments.

Q: *How will this information be a benefit to others in the nuclear community?*

A: By releasing this database into the public domain, the nuclear energy community will also be able to identify necessary capabilities without waiting for new infrastructure funding, helping to accelerate current research and development (R&D). For example, a university professor will be able to search the database for something, such as a FIB or TEM, and see that another university has one that's only a short day trip away, or even in a different department at the same university.

Q: *Can you tell me more about the CINR accomplishment?*

A: Of course, in 2015 NSUF also resumed making large irradiation and post-irradiation examination awards through the CINR. We were able to make three NSUF access-only awards and one NSUF access award coupled with R&D funding. This marks the first time the program has been able to make these large awards since 2012. In previous years, we found it difficult for PIs to receive both R&D funding from the Nuclear Energy University Program (NEUP) and follow-on access awards because of the two separate review processes. With this improvement, the proposal is reviewed all at once by both the R&D technical reviewers and the NSUF staff.

Q: *Did anything else memorable occur this year with the program?*

A: Yes, NSUF is committed to ensuring the nuclear energy community's R&D needs are met and also supporting our partner facilities. Members of the NSUF staff traveled to each of our partner facilities in 2015 to discuss how the program can continue to offer our users the best capabilities available.

Q: *Why were these partner visits important to the program?*

A: With 11 partner facilities, it's not always feasible to have a face-to-face meeting, but it is the best way to foster communication. There have been changes at many of the partner facilities and at the NSUF in recent years. These meetings made it possible for everyone to get to know one another and for the new leaders at NSUF to tour the facilities and learn more about what the partners can offer to researchers.

Q: *Can you give us any insight into what the future may hold for the NSUF?*

A: The NSUF program is growing. We expect to support more research in the future. That includes research proposed in both the RTE and the CINR solicitations.

Q: *What do you think this might mean for our program on the larger international stage?*

A: The NSUF is actively discussing collaborations with several international entities, which share the same mission goals as the DOE-NE. In addition, we have seen more proposals originating from principal investigators outside of the United States. We expect to see an increase in our international relationships.

Q: *Can you tell me more about what you do at DOE Headquarters (HQ)? What are some of your other responsibilities?*

A: Sure, I am currently the program manager for the Advanced Methods for Manufacturing program under Nuclear Energy Enabling Technologies – Crosscutting Technology Development and the federal program manager of the Nuclear Science User Facilities. I'm also the DOE-HQ point of contact for Idaho National Laboratory's Laboratory Directed Research and Development program.

Q: *How long have you been at DOE-NE?*

A: It's been about five years. I began my career at DOE-NE right after I graduated in 2011.

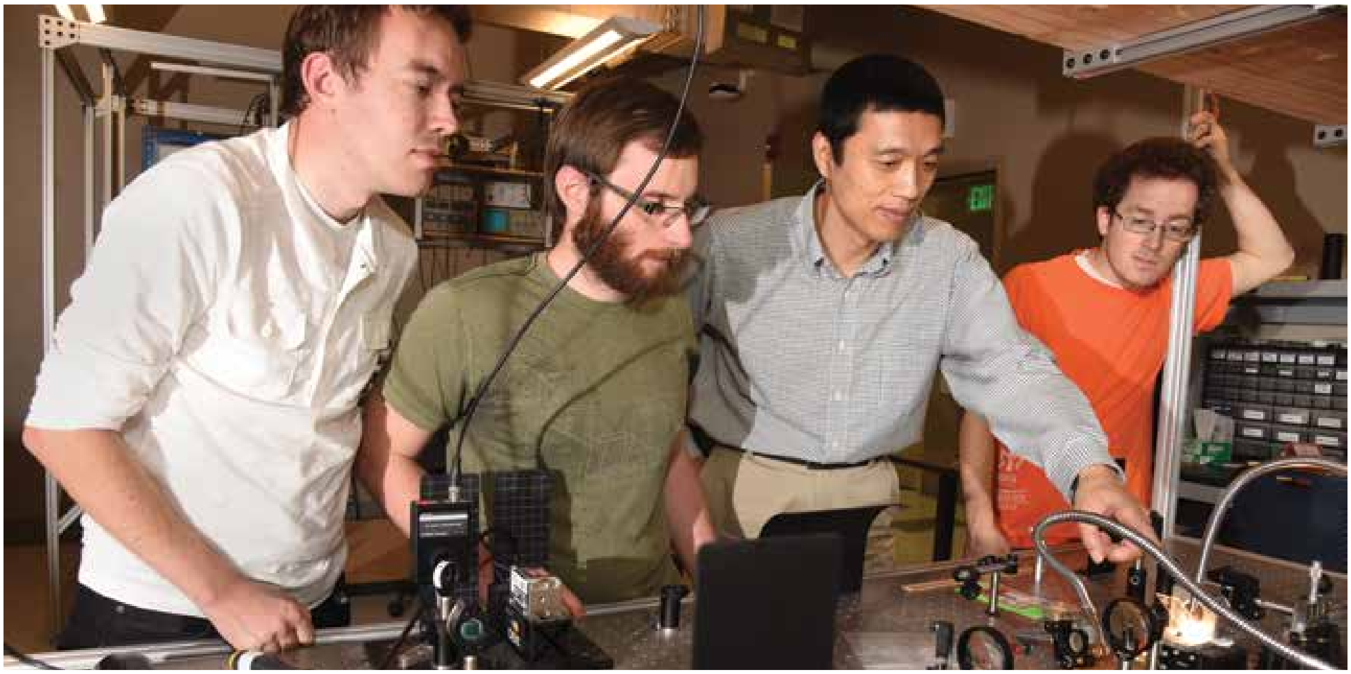
Q: *What is your educational background?*

A. I received my bachelor's degree in nuclear engineering from Pennsylvania State University in 2011 and a Master of Science in environmental engineering from John's Hopkins University in 2013.

Q: *What do you like to do outside of the office?*

A: I enjoy running, traveling and corralling my two young children. Luckily my kids enjoy the stroller, so I'm able to take them with me on long runs.

FOCUS ON TECHNICAL LEADS



Nuclear Science User Facilities users gained important allies in FY 2015 with the introduction of technical leads. A technical lead is assigned early on in the proposal process and works closely with the principal investigator to establish the scope and feasibility of the project. As the experiment progresses, the tech lead ensures the project is running smoothly.

"Basically, we are the experts on the capabilities and various instruments, which makes us able to advise the PIs on the intricacies of the experiment," said Assel Aitkaliyeva, an NSUF technical lead.

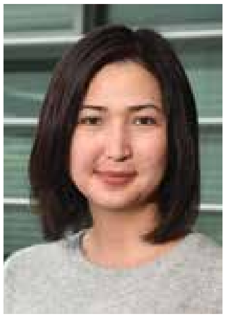
It's the technical lead's job to keep the project on track, on schedule, and to resolve any issues that could cause delays. Part of this process includes a thorough feasibility review to determine if the objectives in the proposal can be accomplished or if the scope of the project will need refining.

"We need to make sure that what the researcher wants to do is compatible with our limits," said Donna Post Guillen, an NSUF technical lead. "We have to know where those limitations are so we can push the frontiers of science while being safe."

As with any close collaboration, it's important to keep the lines of communication flowing between the principal investigator (PI), the tech lead and the rest of the collaborators on the experiment. It's not uncommon for a tech lead to juggle several NSUF experiments in addition to the work he or she is doing in other areas of the lab. If the tech lead is left out of communications relating to the experiment, it's much more difficult to help.

"The PI needs to be very engaged in order for us to have successful outcomes, because that's how we will deliver the most value for the NSUF and for the nuclear science community," Guillen said. "It's our job to make sure these experiments succeed and provide value for the customer."

NSUF users will benefit from working with a technical lead who can help researchers navigate the experiment process while avoiding issues. Tribal knowledge is common in every lab, so it makes sense to team up with others who have experience and know the ins and outs of a specific facility. For instance, Idaho National Laboratory has a strict quality assurance program that includes planning and documentation requirements for items, processes and services. These requirements can be a stumbling block to users from universities or other labs who might not be accustomed to these procedures.



Assel Aitkaliyeva, Ph.D.

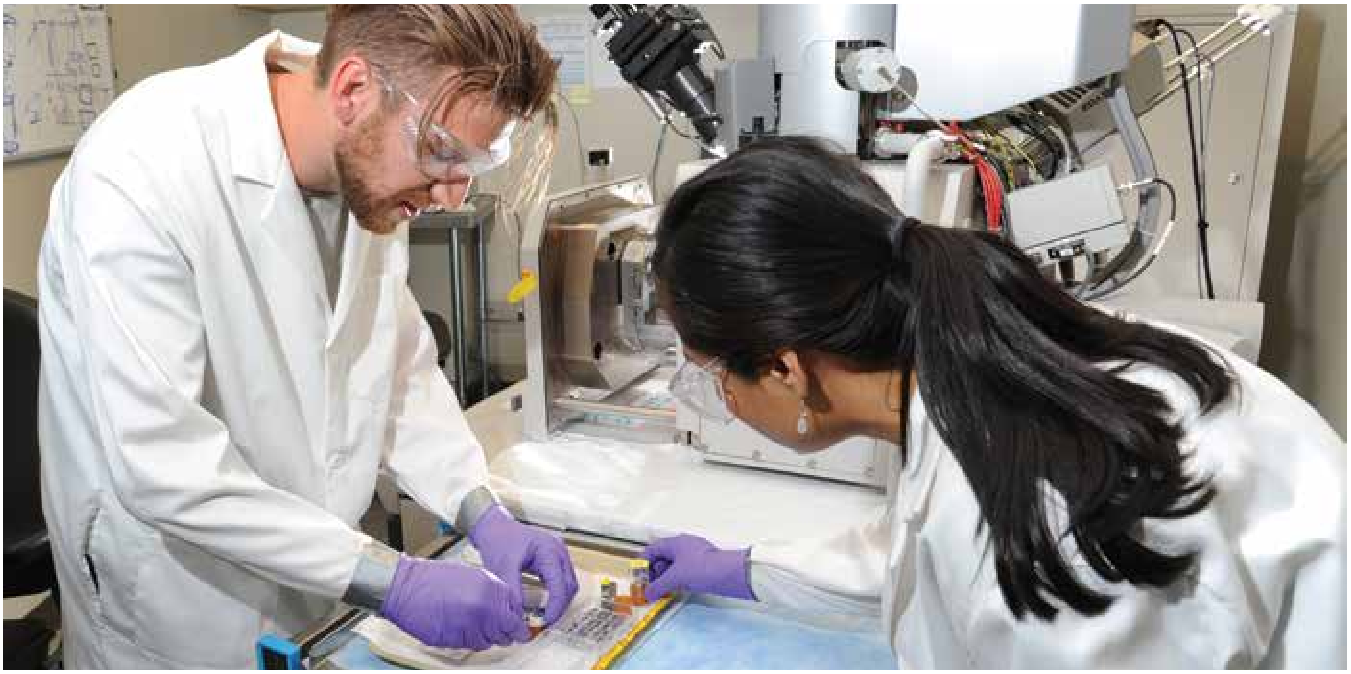
B.S. in physics from Kazakh National University, M.S. in nuclear engineering and a Ph.D. in materials science from Texas A&M University

Assel Aitkaliyeva started a yearlong internship at INL in 2011, and joined INL full time as an NSUF technical lead following her graduation in December 2012.

Her scientific work at INL is focused on understanding the equilibria and kinetics of plutonium-based fuels in contact with cladding materials. Her studies are designed to close the gap in the understanding of the progression of the fuel-cladding chemical interaction (FCCI).

Her work in pioneering focused ion-beam sample preparation methods has made her widely sought after as an adviser by domestic and international laboratories.

She has authored more than 20 peer-reviewed journal publications and 30 presentations at professional meetings. She has published six peer-reviewed journal papers based on her INL research. As a principal investigator, Aitkaliyeva has secured more than $3.2 million in research funding for her work, including a $593,000 grant for research equipment that benefits many others within the lab.



"Getting materials accepted at the laboratory is not as easy as it would be between universities. We are required to have certifications for materials and we commonly need extra specimens to perform further analysis to confirm sample chemistry," said Tom Maddock, an NSUF technical lead and INL experiment manager.

Maddock stressed the importance of communicating early and regularly to make sure there aren't surprises down the road. "If you have questions about flux, geometry or instrumentation, you should reach out early to your technical lead or to an experiment manager," Maddock said. "Also, if you are preparing a sample that will be going in the reactor or any instrumentation, we need to make sure that it can go through our Quality Assurance (QA) and get here in a reasonable amount of time."

The NSUF technical leads agreed that the best part of the job is being involved in many different projects as well as having an opportunity to work with a diverse set of researchers from all over the world.

"Every project we have is interesting and we get to work with people from different places that we probably wouldn't meet otherwise," said Aitkaliyeva.

With each new project comes a new opportunity for learning. "There is always a new aspect of what you're doing, whether it's a capsule design, a new material, developing new capabilities for PIE, or coming up with new methods for doing things," said Guillen. "These diverse collaborations are a great way to cross-fertilize new ideas."

Donna Post Guillen, Ph.D.

B.S. in mechanical engineering from Rutgers University, M.S. in aeronautics from Caltech, Ph.D. in engineering and applied science from Idaho State University

Donna Post Guillen has been a researcher at INL since 1993. She is currently a distinguished research engineer and a registered professional engineer licensed to practice mechanical engineering in the state of Idaho.

She has served as principal investigator for several multidisciplinary research projects on the topics of waste heat recovery, synthetic fuels production, nuclear reactor fuels and materials experiments, and waste glass processing. The focus of her research is on multiphase computational fluid dynamics and thermal hydraulics for sustainable energy technologies. She applies numerical modeling techniques to provide understanding of a wide variety of complex systems, from greenhouse gas generation/sequestration for dairies to waste vitrification for the Hanford Waste Treatment Plant.

She is the lead inventor on two patents related to the development of a new metal matrix composite material to produce a fast reactor environment within a pressurized water reactor, such as Advanced Test Reactor (ATR). She actively mentors students, routinely chairs and organizes technical meetings and serves in a leadership capacity for professional societies. She has published over 100 conference papers, reports and journal articles, and written/edited three books.

From start to finish, it takes a small army of support staff to see an experiment through to its conclusion. As an experiment manager, Maddock's position provides him a very broad and complete overview about what's going on everywhere in the lab. "It starts with the researchers who propose and define the experiment and then the experiment managers who help with the budget and schedule," Maddock said. "Then it's onto physics, structural and thermal analysts and then operations specialists who work on irradiation at the ATR and then PIE at MFC."

Each experiment is unique. Some projects require more one-on-one time from the technical leads, but this varies greatly depending on the complexity of the project and the experience level of the PI.

"I find the more complex projects are more challenging and interesting, but sometimes it's nice to work on a simpler project that we can complete quickly," Aitkaliyeva said. "It actually is refreshing that each PI looks at the project with a different perspective."

Thomas Maddock, M.S.

B.S. in physics and M.S. in nuclear engineering from University of Utah

Thomas Maddock has more than 13 years of experience in experiment design and project engineering. He has experience working at both a Mark I and a Mark II Training Research Isotope General Atomics (TRIGA) reactor-designing, fabricating and installing new irradiation facilities. He also worked as the Neutron Radiography (NRAD) system engineer to convert the reactor from high-to low-enriched fuel. He currently works as an experiment manager for the NSUF program and he is Project Management Professional (PMP) certified.

Maddock has given a number of presentations at INL and at various professional conferences. He presented a series of conferences at the ATR to different operations organizations on NSUF experiments and their importance to the nuclear community. He also gave a presentation at the National Organization of Test, Research and Training Reactors (TRTR) conference on the experiment design process and on ATR capabilities. Before beginning work for NSUF, he wrote a paper for the Physics of Reactors (PHYSOR) conference and gave a presentation on a control rod malfunction at the NRAD reactor.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INTRODUCING THE NEID

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Nuclear energy researchers gained a new tool this year with the launch of the Nuclear Energy Infrastructure Database (NEID), which was announced by President Obama as part of the Gateway for Accelerated Innovation in Nuclear (GAIN) initiative. The user-searchable database includes 804 nuclear energy-related instruments in 445 facilities at 124 institutions across the United States and abroad.

The DOE-NE identified a need to take an inventory of the nuclear energy-related infrastructure. Once assembled, the database would be used to establish needs, identify redundancies, look for efficiencies and better understand how the current infrastructure is distributed. This information would help inform the content of future infrastructure calls and also be a resource for the NSUF and the greater nuclear research community.

Brenden Heidrich, NSUF R&D capabilities scientist, took on the task of managing the project including compiling all the data. He explained that unlike other infrastructure databases, the NEID has a wider reach. "There are databases on hot cells or research reactors, but the NEID is more focused on all of the nuclear capabilities in the U.S., although we do have information on some international facilities," Heidrich said. "The other big difference is that anyone can use it, after we verify their identity."

According to Heidrich, acquiring the data has been the biggest hurdle in developing the database. "I spent the first year really by myself, getting the data off websites, reports and publications," he said. "Getting good quality data, that is not outdated, has been difficult. Oddly enough, the national labs were the hardest ones, because there's little public information readily available on most facilities."

The data was compiled from a variety of sources, such as the International Atomic Energy Agency, the DOE, the National Research Council and other federal and civilian resources. As new facilities come online and new instruments are purchased, they will be added either from a direct pull from available asset databases or they can be manually entered by the facility owners. NSUF plans on releasing a formal request for information (RFI) in 2016 through the DOE-NE, which will allow anyone to propose their facility for inclusion in the NEID.

Users	Count	Percentage
National Laboratory	48	41%
U.S. University	25	21%
Industry	21	18%
U.S. Government	12	10%
International Government	4	3%
NGOs	4	3%
International University	2	2%
International Industry	2	2%

Institutions in the database	Count	Percentage
U.S. University	50	41%
National Laboratory	27	22%
U.S. Industry	22	18%
International Government	14	11%
International University	5	4%
International Industry	4	3%

In addition to initially gathering the data, it's important to make sure that it stays current. "We can have the database email [the facility owners] directly and then they can click on a link and update their own facilities and verify that the information we have on file is still current," Heidrich said. "Our challenge is to make facility owners understand why this is important and what's in it for them, such as the ability to advertise their capabilities for free."

The Gap Analysis

Once the major nuclear energy capabilities were identified and the near-, mid- and long-range R&D goals were investigated, the next step was to perform a gap analysis. The analysis was based on two foundations: a capabilities analysis of the existing infrastructure, and the projected needs based on the anticipated direction of nuclear energy research and development. The goal was to provide a set of recommendations for potential funding based on the types of instrumentation and facilities that will be needed to perform the work.

As expected, the initial analysis pointed out the "elephant in the room" as an ongoing need for irradiated material characterization equipment, such as focused ion beam microscopes that can look at radioactive fuel and ion beam irradiation capabilities. In response, the NSUF and the DOE-NE organized an ion


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beam workshop at INL that took place in March 2016. During the workshop, representatives from 16 different ion beam facilities, as well as DOE-NE research programs and the stakeholders from the nuclear power industry, came together to discuss the needs and talk about development options. The discussion is ongoing with a road map exercise anticipated in 2017.

What's next?

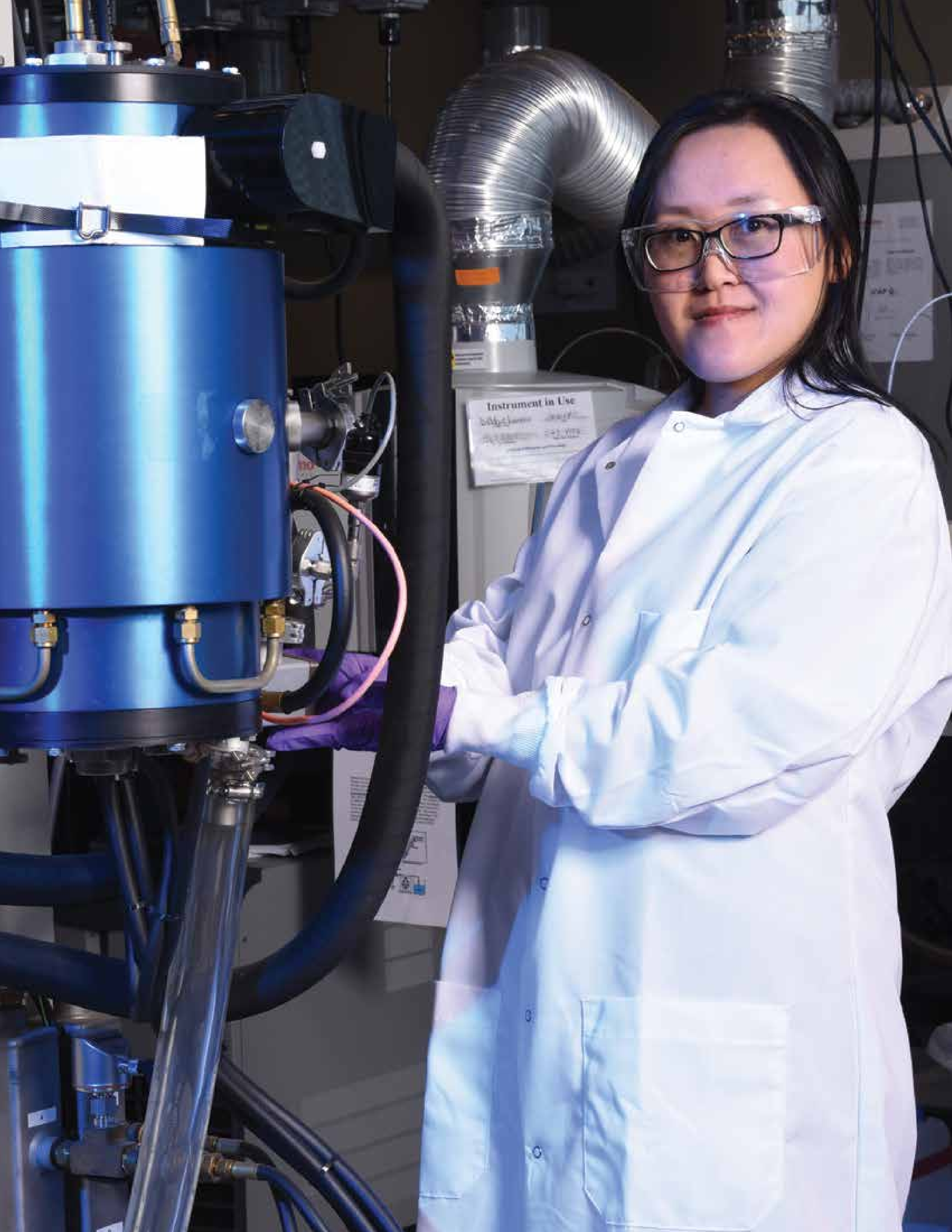
While keeping the database up-to-date will remain a top priority, the NEID in its current configuration is populated with information from the facilities that are most relevant to the nuclear science community. The work does not stop there. This year's goal is to tie in the NSUF Fuels and Materials Library, and the NEUP and Office of Science and Technical Information (OSTI) databases will be incorporated after that.

"The hope is to tie facilities, people and research all together," said Heidrich. The big idea is to create a pathway to getting research done. "It's figuring out the steps from the proposal process, to experiment mock-up and then on to irradiation, PIE and then billing," he said. "And then we will see if there's a way to make the pathway 'smart' by showing all the different combinations and ways it could get done — shortest geography, lowest cost, best availability."

Get involved

The database is open to everyone, but users must register to get access at https://nsuf-infrastructure.inl.gov/. Once the new NSUF website is launched in the summer of 2016, one set of credentials will get you into the NSUF site, including the RTE proposal system as well as the NEID and Fuels and Materials Library. There is no cost to use the database.

NSUF users are encouraged to register and see what's out there and to share their ideas and opinions on the NEID. Every page includes an email link to provide feedback to the administrators.


Instrument in Use

PROGRAM OVERVIEW

NSUF: A Model for Collaboration

NSUF and its partner facilities represent a prototype laboratory for the future. This unique model utilizes a distributed partnership with each facility bringing exceptional capabilities to the relationship including reactors, beamlines, state-of-the-art instruments, hot cells, and most importantly, expert mentors. Together, these capabilities and people create a nationwide infrastructure that allows the best ideas to be proven using the most advanced capabilities. Through NSUF, university researchers and their collaborators are building on current knowledge to better understand the complex behavior of materials and fuels in a nuclear reactor.

In 2015, NSUF's partnership program had eight universities, two national laboratories, and one industry partner. The partner facility capabilities greatly expand the types of research offered to users. The avenues opened through these partnerships facilitate cooperative research across the country, matching people with capabilities and students with mentors. In 2015, NSUF included INL and the following institutions:

- Illinois Institute of Technology
- Massachusetts Institute of Technology
- North Carolina State University
- Oak Ridge National Laboratory
- Pacific Northwest National Laboratory
- Purdue University
- University of California, Berkeley
- University of Michigan
- University of Nevada, Las Vegas
- University of Wisconsin
- Westinghouse

The pages that follow contain specific details on the capabilities of NSUF, its partners, and how to access these capabilities through the calls for proposals. There is also information on the Users Meeting, a yearly event hosted by NSUF designed to instruct and inform. This event is free of charge to interested persons, and a number of scholarships for travel and hotel are offered to students and faculty. Take time to familiarize yourself with the many opportunities offered by NSUF and consider submitting a proposal or two.

NSUF Research Supports DOE-NE Missions

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

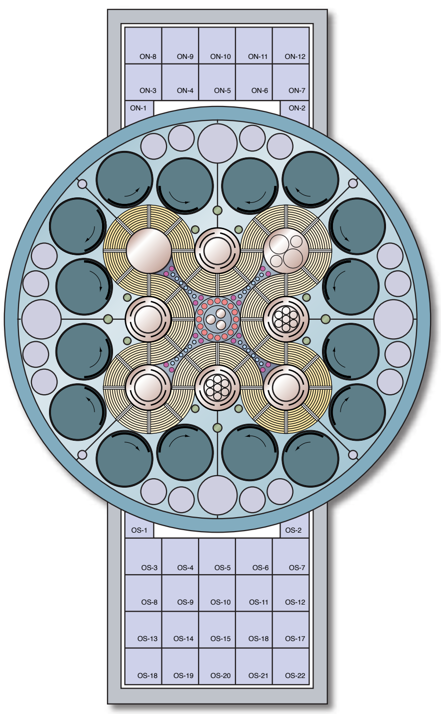
- Develop technologies and other solutions that can improve the reliability, sustain the safety, and extend the life of current reactors.
- Develop improvements in the affordability of new reactors to enable nuclear energy to help meet the administration's energy security and climate change goals.
-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

NSUF research addresses a number of these mission needs. Most of the research contained in this report looks at either understanding the mechanisms of radiation on materials and fuels to address the challenges of the aging current fleet, or looks at materials and fuels for the next generation of reactors. To be eligible as an NSUF research project, the research must support at least one of the DOE-NE missions. For specific information on DOE missions, go to http://www.energy.gov/ne/mission.

To learn more about proposing a research project, visit the NSUF website: http://nsuf.inl.gov.

U.S. DEPARTMENT OF ENERGY
Nuclear Energy

REACTOR Capabilities



ATR's serpentine design allows a variety of experiment configurations.

NSUF offers access to a number of reactors. ATR is located at the ATR Complex (ATR) on the INL Site and has been operating continuously since 1967. In recent years, the reactor has been used for a wide variety of government and privately sponsored research. The Advanced Test Reactor Critical Facility (ATRC) reactor is a low-power version of ATR.

The MIT reactor is a 5-MW reactor with positions for in-core fuels and materials experiments. Oak Ridge National Laboratory's (ORNL) HFIR is an 85-MW reactor offering steady-state neutron flux and a variety of experiment positions. The PULSTAR reactor at North Carolina State University is a pool-type reactor that offers response characteristics similar to commercial light water power reactors.

Idaho National Laboratory Advanced Test Reactor

ATR is a water-cooled, high-flux test reactor, with a unique serpentine design that allows large power variations among its flux traps. 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. Experiment positions vary in size from 0.5 to 5 inches in diameter (1.27 to 12.7 centimeters) and all are 48 inches (121.92 centimeters) long. The peak thermal flux is 1 X 1015 n/cm^2-sec and fast flux is 5 X 1014 n/cm -sec when operating at full power of 250 MW. 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.

Idaho National Laboratory Advanced Test Reactor Critical Facility

ATRC is a low-power version (same size and geometry) of the higher-powered ATR core. It is operated at power levels less than 5 KW with typical operating power levels of 600 W or less. ATRC is primarily used to provide data for the design and safe operation of experiments for ATR. ATRC is also used to supply core performance data for the restart of ATR after periodic core internals replacement. Occasionally, ATRC is used to perform low-power irradiation of experiments.

Oak Ridge National Laboratory High Flux Isotope Reactor

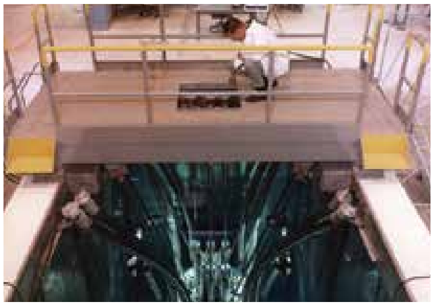
High Flux Isotope Reactor (HFIR) is a versatile 85-MW research reactor offering the highest steady-state neutron flux in the western world. With a peak thermal flux of 2.5 x 10^{15} n/cm^2-s and a peak fast flux of 1.1 x 10^{15} n/cm^2-s, HFIR is able to quickly generate isotopes that require multiple neutron captures and perform materials irradiations that simulate lifetimes of power reactor use in a fraction of the time. HFIR typically operates seven cycles per year, each cycle lasting between 23 and 26 days. Associated irradiation processing facilities include the Hydraulic Tube Facility, Pneumatic Tube Facilities for Neutron Activation Analysis (NAA) and Gamma Irradiation Facility.

Massachusetts Institute of Technology Reactor

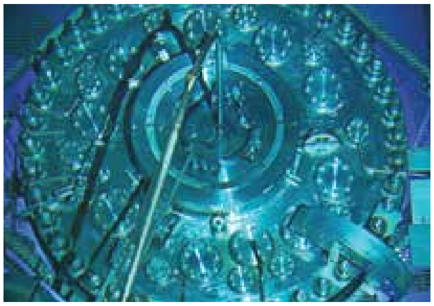
Massachusetts Institute of Technology Reactor (MITR) is a 5-MW tank-type research reactor. It has three positions available for in-core fuel and materials experiments over a wide range of conditions. Water loops at pressurized water reactor/boiling water reactor (PWR/BWR) conditions, high-temperature gas reactor environments at temperatures up to 1400°C and fuel tests at light water 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 1014 and 5 x 1014 n/cm^2-s. MITR has received approval from the Nuclear Regulatory Commission for a power increase to 6 MW, which will enhance the neutron fluxes by 20 percent.

North Carolina State University PULSTAR Reactor

The PULSTAR reactor is a 1-MW pool-type nuclear research reactor located in North Carolina State University's (NCSU) Burlington Engineering Laboratories. The reactor, one of two PULSTAR reactors built and the only one still in operation, uses 4 percent 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.



Aerial view of the ATRC reactor core and bridge.



Top of the HFIR reactor.

Annular fuel rig in the MITR core.

Downward view of the PULSTAR reactor pool.

POST-IRRADIATION EXAMINATION Capabilities

Hot Fuel Examination Facility, located at the Materials and Fuels Complex at DOE's INL Site in Idaho.

NSUF offers researchers access to a broad range of PIE facilities.

These include capabilities at INL's MFC; the Microscopy and Characterization Suite (MaCS) at the Center for Advanced Energy Studies; the Nuclear Services Laboratories at North Carolina State University; hot cells, radiological laboratories and the Low Activation Materials Development and Analysis (LAMDA) facility at Oak Ridge National Laboratory; the Radiochemistry and Materials Science and Technology Laboratories at Pacific Northwest National Laboratory; the Interaction of Materials with Particles and Components Testing (IMPACT) facility at Purdue University; several instruments from the Nuclear Materials Laboratory at University of California, Berkeley; the Irradiated Materials Complex at the University of Michigan; the Harry Reid Center Radiochemistry Laboratories at the University of Nevada, Las Vegas; and the Characterization Laboratory for Irradiated Materials at the University of Wisconsin.

Transmission electron microscope, one of many PIE capabilities in the Microscopy & Characterization Suite (MaCS) at the Center for Advanced Energy Studies in Idaho Falls, Idaho.

Idaho National Laboratory: Hot Fuel Examination Facility Analytical Laboratory, Electron Microscopy Laboratory

Hot Fuel Examination Facility (HFEF) is a large alpha-gamma hot cell facility dedicated to remote examination of highly irradiated fuel and structural materials. Its capabilities include nondestructive and destructive examinations. The facility also offers a 250-kWth Training, Research, Isotopes 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 a wide range of spectrometric techniques.

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

The Scanning Electron Microscope in Oak Ridge National Laboratory's LAMDA facility.

A hot cell in the Radiochemistry Processing Laboratory at Pacific Northwest National Laboratory.

The Electron Microscopy Laboratory (EML) is dedicated to materials characterization, primarily using transmission electron, scanning electron and optical microscopy. The EML also houses a focused ion beam (FIB) that allows examination and small-sample preparation of radioactive materials.

Center for Advanced Energy Studies Microscopy and Characterization Suite

The MaCS is equipped to handle low-level radiological samples as well as nonradiological samples. MaCS offers several high-end pieces of equipment, including a local electrode atom probe (LEAP), automated hardness tester, scanning electron microscope (SEM), nanoindenter and atomic force microscope, transmission electron microscope (TEM) and focused ion beam.

North Carolina State University Nuclear Services Laboratories

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

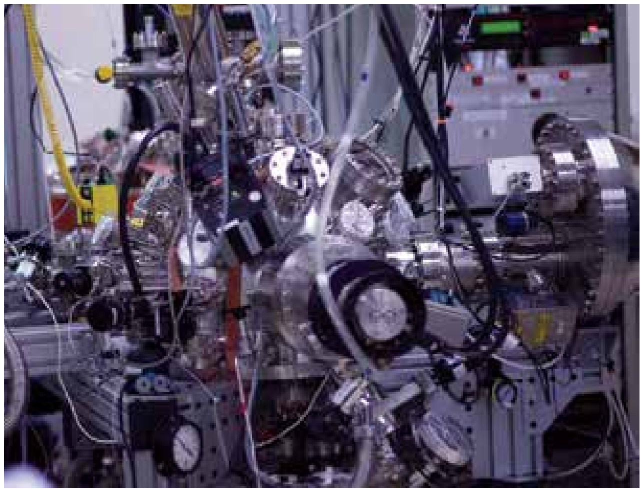
Oak Ridge National Laboratory Hot Cells, Radiological Laboratories, LAMDA Facility

ORNL hot cells and radiological laboratories offer a wide variety of research and development and production capabilities from radiochemistry and isotope packaging to materials testing to irradiated fuels examination. Facilities include the Irradiated Materials Examination and Testing (IMET) facility, Irradiated Fuels Examination Laboratory (IFEL) and Radiochemical Engineering Development Center (REDC).

The LAMDA Laboratory, added in 2012, offers post-irradiation examination capabilities including refractory element test stands for tensile testing, optical and scanning electron microscopes, and thermal diffusivity and density measurement equipment.

Pacific Northwest National Laboratory Radiochemistry Processing Laboratory, Materials Science and Technology Laboratory

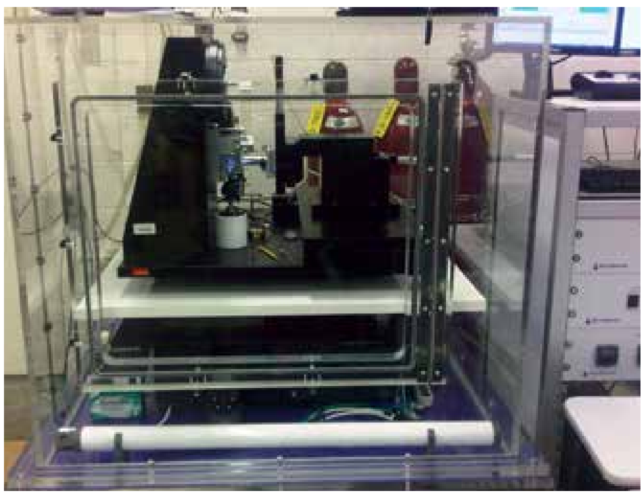
The RPL and the Materials Science and Technology Laboratory (MSTL) offer a wide range of specialized equipment for handling and testing fuels and materials. Capabilities include experiment hardware design, fabrication and assembly, testing facilities for both nonradioactive and radioactive structural materials, and the advanced characterization of unirradiated and irradiated fuels and materials using instruments including TEM, SEM and optical microscopy.



The IMPACT facility at Purdue University.

Purdue University IMPACT Facility

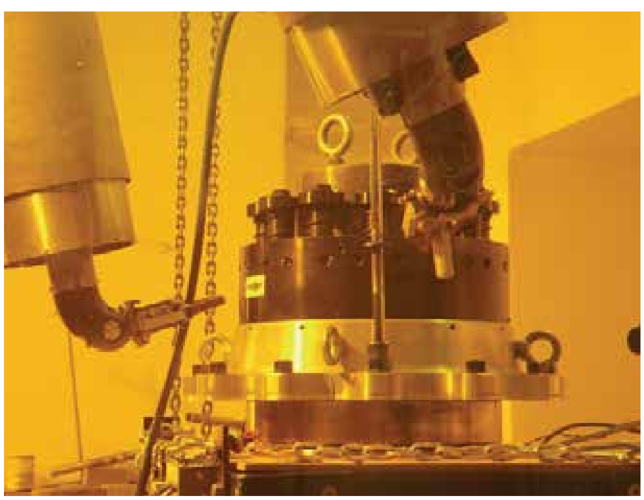
The IMPACT facility offers a wide range of spectroscopy techniques to study the surface of materials. The IMPACT facility houses a variety of examination instruments including low-energy scattering spectroscopy, X-ray photoelectron spectroscopy, auger electron spectroscopy, extreme ultraviolet reflectometry, extreme ultraviolet photoelectron spectroscopy and mass spectrometry.



UC Berkeley nano-indentation system.

University of California, Berkeley Nuclear Materials Laboratory

The Nuclear Materials Laboratory provides several capabilities for examining irradiated material samples including a nanoindentation system for nano and microscale hardness testing at ambient and elevated temperature and inert environments, positron annihilation spectroscopy, and warm sample preparation (polishing, cutting, grinding and mounting).



Capability at the Irradiated Materials Complex on the UM campus at Ann Arbor, Michigan.

University of Michigan Irradiated Materials Complex

The Irradiated Materials Complex provides laboratories and hot cells with capabilities 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.

University of Nevada, Las Vegas Harry Reid Center Radiochemistry Laboratories

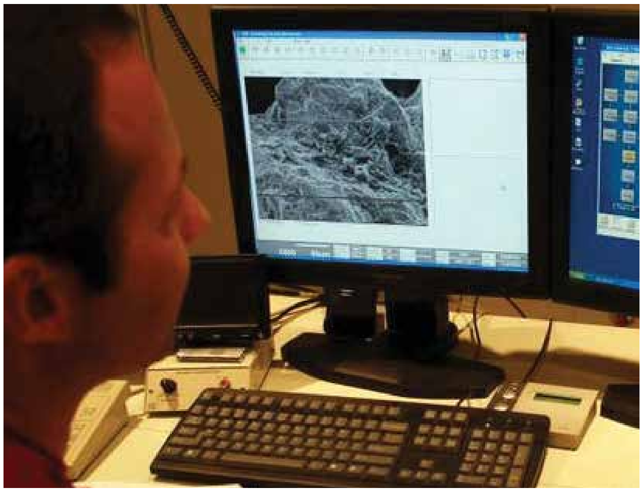
Post-irradiation examination capabilities at the Radiochemistry Laboratories include metallographic microscopy, X-ray powder diffraction, Rietveld analysis, SEM and TEM, electron probe microanalysis, and X-ray fluorescence spectrometry.

University of Wisconsin Characterization Laboratory for Irradiated Materials

The Characterization Laboratory for Irradiated Materials offers PIE capabilities including SEM and TEM on neutron-irradiated materials.

Westinghouse Materials Center of Excellence Laboratories

Westinghouse offers its Materials Center of Excellence Laboratories (MCOE) Hot Cell Facility and accompanying laboratories to provide experimental support to ATR-related nuclear energy materials research programs. The Westinghouse facilities in Churchill, Pennsylvania, are housed in four cells that provide a broad range of testing, evaluation and characterization capabilities for both unirradiated and irradiated materials. In-place capabilities include the ability to test under a variety of environments, an extensive mechanical testing laboratory, a specialized corrosion and stress corrosion cracking lab, and materials microstructure and chemical characterization instruments. Specialized facilities are also available to measure the radioactivity properties of materials under investigation as well as neutron and gamma sources facilities, which can be employed to assess materials' response to in-situ radiation.



PIE capabilities at the Harry Reid Center Radiochemistry Laboratories, located on the UNLV campus in Las Vegas, Nevada.

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

Operators use manipulators to perform work at the Westinghouse Hot Cell Lab.

BEAMLINE Capabilities

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

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 2015, the NSUF program offered researchers access to four university partner beamline 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 Tandem Accelerator Ion Beam.

Illinois Institute of Technology (IIT) MRCAT at Argonne National Laboratory's Advanced Photon Source

The 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.

North Carolina State University 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.

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

University of Michigan 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.

Michigan Ion Beam Laboratory for Surface Modification and Analysis, located on the UM campus in Ann Arbor, Michigan.

University of Wisconsin Tandem Accelerator Ion Beam

A 1.7-MV terminal voltage tandem ion accelerator (Model 5SDH-4, National Electrostatics Corporation Pelletron accelerator) installed at University of Wisconsin (UW) 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.

Tandem Ion Beam Accelerator, located on the UW campus in Madison, Wisconsin.

CALLS FOR PROPOSALS

Jeff Benson
Program Administrator

The 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.

The NSUF offers research proposal options through an online submittal system that helps prospective researchers develop, edit, review and submit their proposals. 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. All NSUF research must be nonproprietary and results are expected to be published. 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.

Calls for Irradiation, Post-irradiation Examination and Synchrotron Radiation Experiments

Applications are submitted annually through the Consolidated Innovative Nuclear Research Funding Opportunity Announcement. More information is available on the Nuclear Energy University Programs (NEUP) website, www.neup.gov.

While priority will be given to proposals that further the direction of DOE's nuclear energy research programs, the NSUF will consider all technically feasible proposals for scientific merit and selection.

- Irradiation/post-irradiation examination of materials or fuels.
- Post-irradiation examination of previously irradiated materials or fuels from the NSUF Fuels & Materials Library.
- 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 the DOE-NE missions and cost. The results are compiled and provided to a panel committee that performs a final review and ranks the proposals. The ranking is given to the 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 NSUF and partner capabilities as determined by the program.

Calls for Rapid Turnaround Experiments

Rapid turnaround experiments are experiments that can be performed quickly — typically in two months or less — and include, but are not limited to, PIE requiring use of an instrument (FIB, TEM, SEM, etc.), irradiations in the PULSTAR reactor, ion beam irradiation and neutron scattering experiments. Proposals for rapid turnaround experiments are reviewed three times per year 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 the proposal for the next call.

For more information, visit the NSUF website, www.nsuf.inl.gov

NSUF Fuels & Materials Library

The NSUF has established a Fuels & Materials Library as an additional pathway for research. The library contains irradiated and unirradiated samples in a wide range of material types, from steel samples irradiated in fast reactors to ceramic materials irradiated in the ATR. Many samples are from previous DOE-funded material and fuel development programs. University researchers can propose to analyze these samples in a PIE-only experiment. Samples from the library may be used for proposals for open calls and rapid turnaround experiments.

As the NSUF program continues to grow, so will the library. To review an online list of available specimens, visit the NSUF electronic system at the address above.

USERS MEETING

The annual NSUF Users Meeting offers researchers 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.

Users Meeting is not just a way to learn more about the 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 NSUF's calls for proposals. Users Meeting supports 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 seven years since its inception, the NSUF Users Meeting has hosted 680 participants from 30 countries and 38 U.S. universities.

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

What to expect at Users Meeting

Users Meeting kicks off with an introductory workshop to the NSUF, which includes a description of current and upcoming research capabilities offered by INL and its university partners, a briefing on the solicitation process and a welcome from DOE.

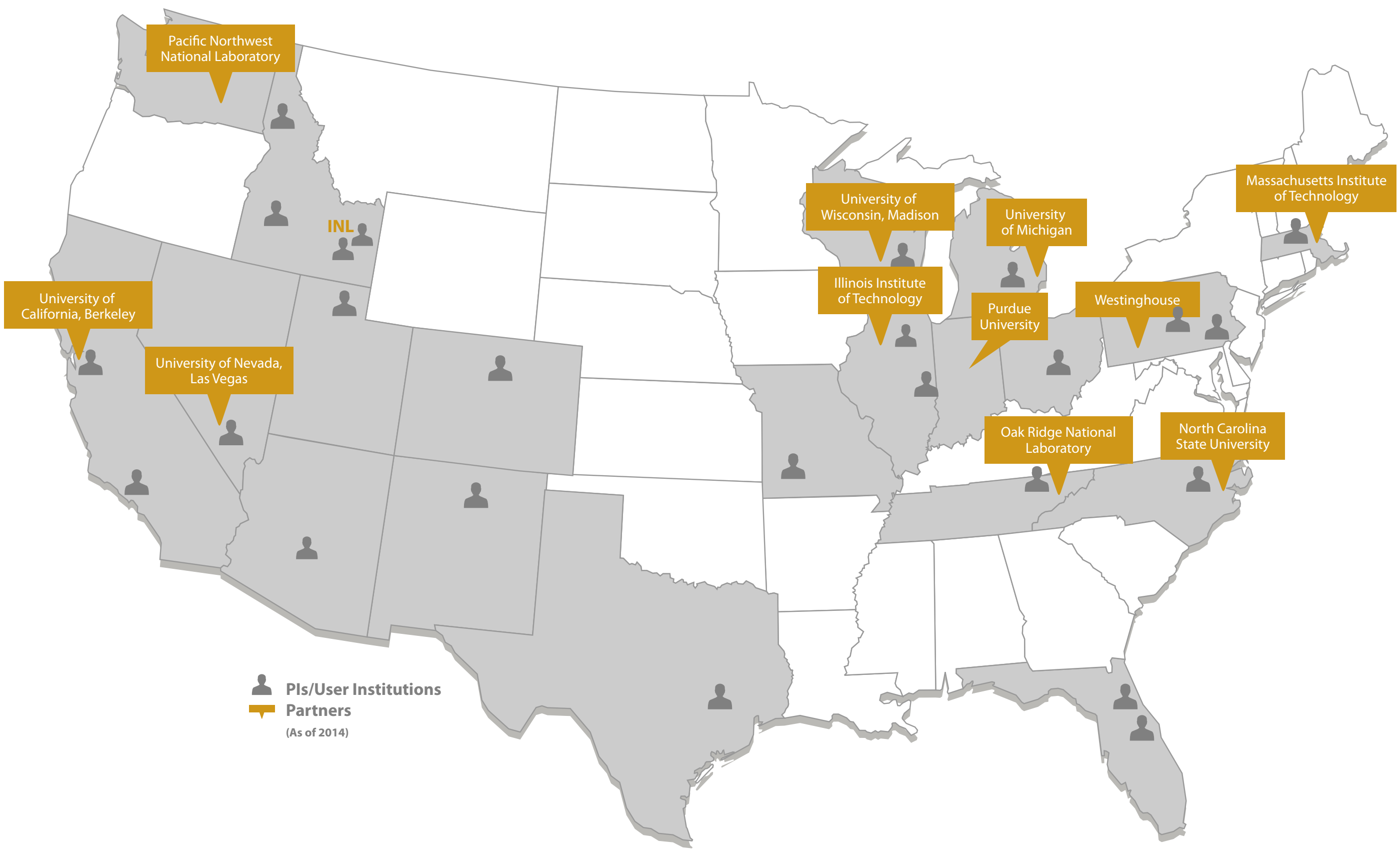
Each year, Users Meeting offers a number of workshops and courses for students to participate in. These may vary from year to year, but courses generally focus on a variety of topic-specific areas, such as in-reactor instrumentation, fuels and materials, or how to conduct radiation experiments.

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.

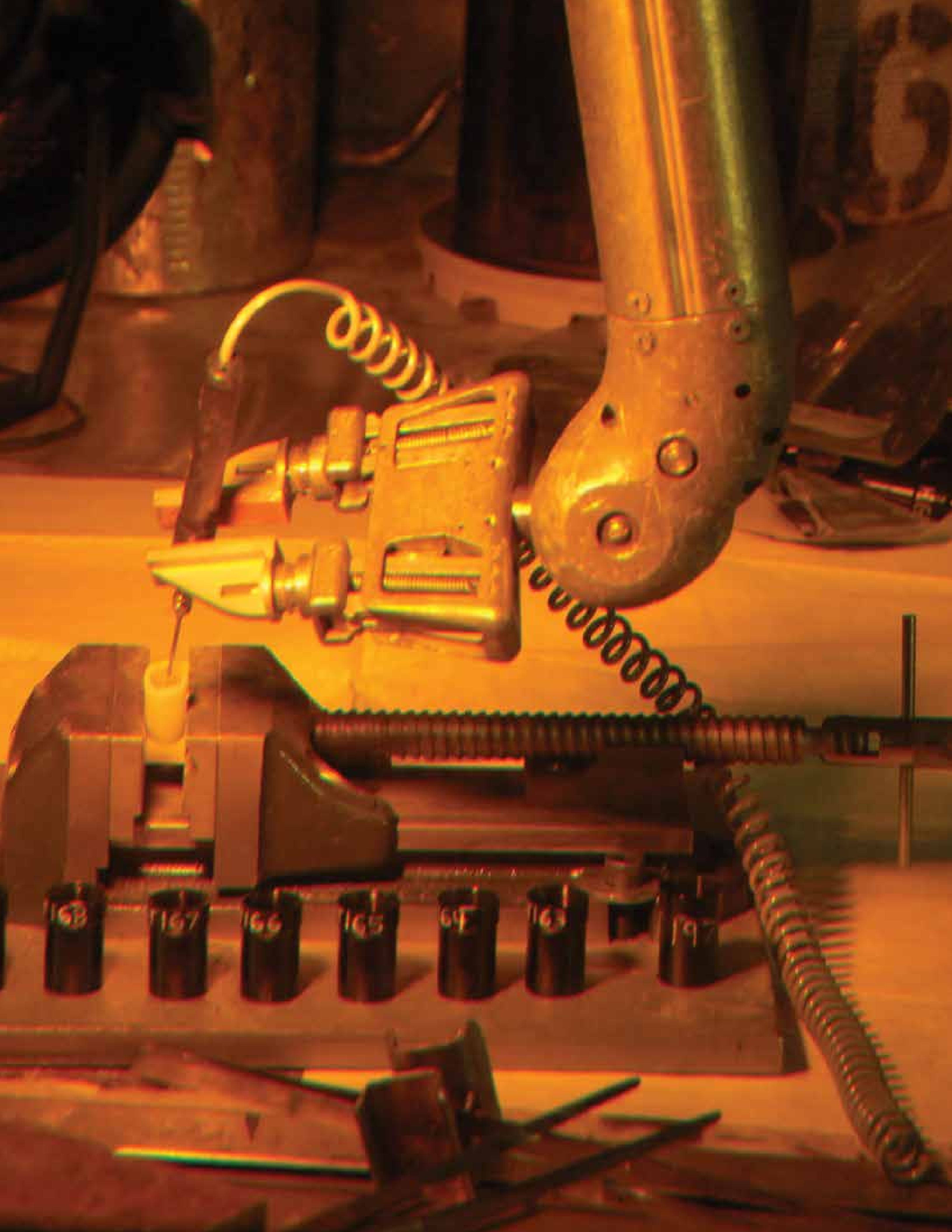
Users who are not able to attend in person have the ability to participate in the meeting online. For more information, visit our website, nsuf.inl.gov.


ADVANCED TEST REACTOR

DISTRIBUTED PARTNERSHIP



Berkeley UNIVERSITY OF CALIFORNIA
ILLINOIS INSTITUTE OF TECHNOLOGY
MIT Massachusetts Institute of Technology
Westinghouse
Pacific Northwest NATIONAL LABORATORY
WISCONSIN UNIVERSITY OF WISCONSIN–MADISON
OAK RIDGE National Laboratory
UNLV
NC STATE UNIVERSITY
PURDUE UNIVERSITY
M UNIVERSITY OF MICHIGAN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NSUF AWARDED PROJECTS

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

Heng Ban – Utah State University (USU) – heng.ban@usu.edu

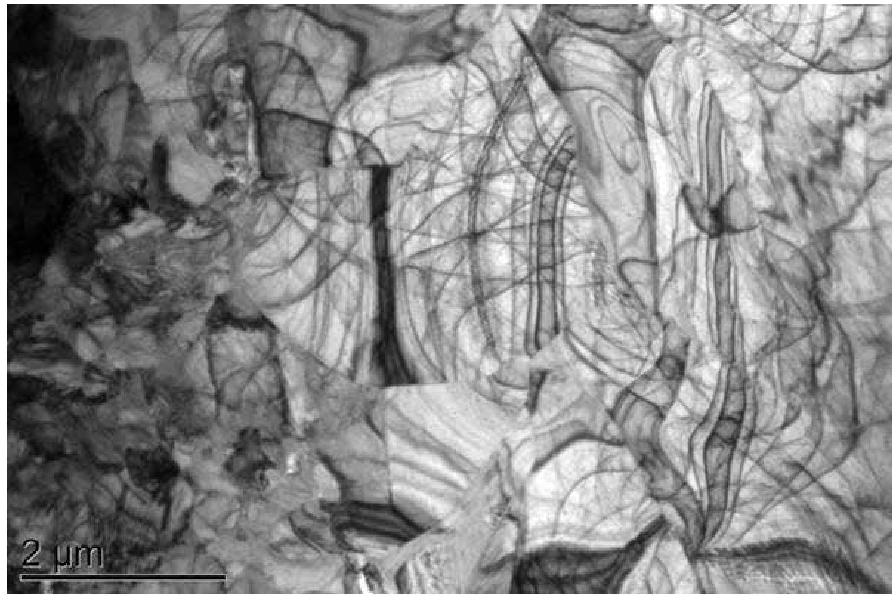


Figure 1. TEM bright-field image of irradiated material showing smaller grains and bend contour, which indicates strain-release during annealing process.

A metal matrix composite material comprised of hafnium aluminide (Al_3Hf) intermetallic particles in an aluminum matrix has been identified as a promising material for fast-flux irradiation testing applications. This material can filter thermal neutrons while simultaneously providing high rates of conductive cooling for experiment capsules, which experience significant heating in the reactor. An experiment was performed wherein material specimens were irradiated in the Advanced Test Reactor (ATR). Thermal and mechanical properties of the material were measured on irradiated specimens and compared to those for the unirradiated material. The effects of irradiation on this new material are being assessed.

Project Description

The capability for conducting fast neutron irradiation tests is essential to meet fuels and materials development requirements for future nuclear reactors. The lack of domestic fast neutron testing capability hinders the development of advanced reactors. The concept behind this project is to use one of the ATR corner lobes with the addition of a thermal neutron filter to absorb the thermal neutrons and booster fuel to augment the neutron flux. An absorber material comprised of hafnium aluminide (Al_3Hf) particles (~28.4% by volume) in an aluminum matrix (Al_3Hf-Al) can absorb thermal neutrons and transfer heat from the experiment to pressurized water cooling channels. Thermal analyses

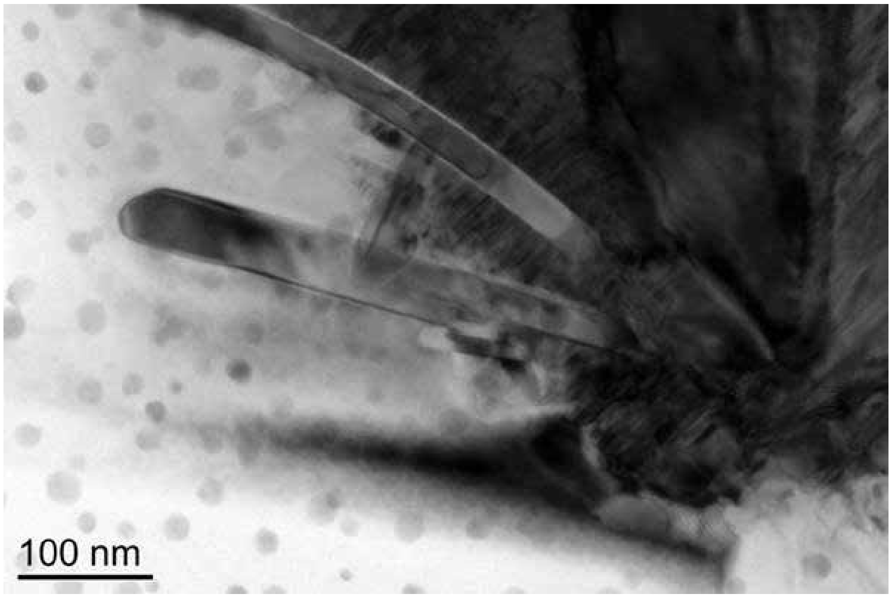


Figure 2. TEM bright-field image of irradiated material showing columnar-shaped grains at phase boundary; the smaller sphere-like particles are oxidation contamination.

conducted on a candidate configuration confirmed that the design of the 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. It is essential to obtain data on the effect of irradiation on the thermophysical and mechanical properties of the Al_3Hf intermetallic and Al_3Hf-Al composite. Other information, such as corrosion behavior and radioactive decay products, are also necessary to proceed with the design and optimization. The purpose of the project is to determine the necessary properties and behavior of this new material. Specific objectives are to determine the:

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

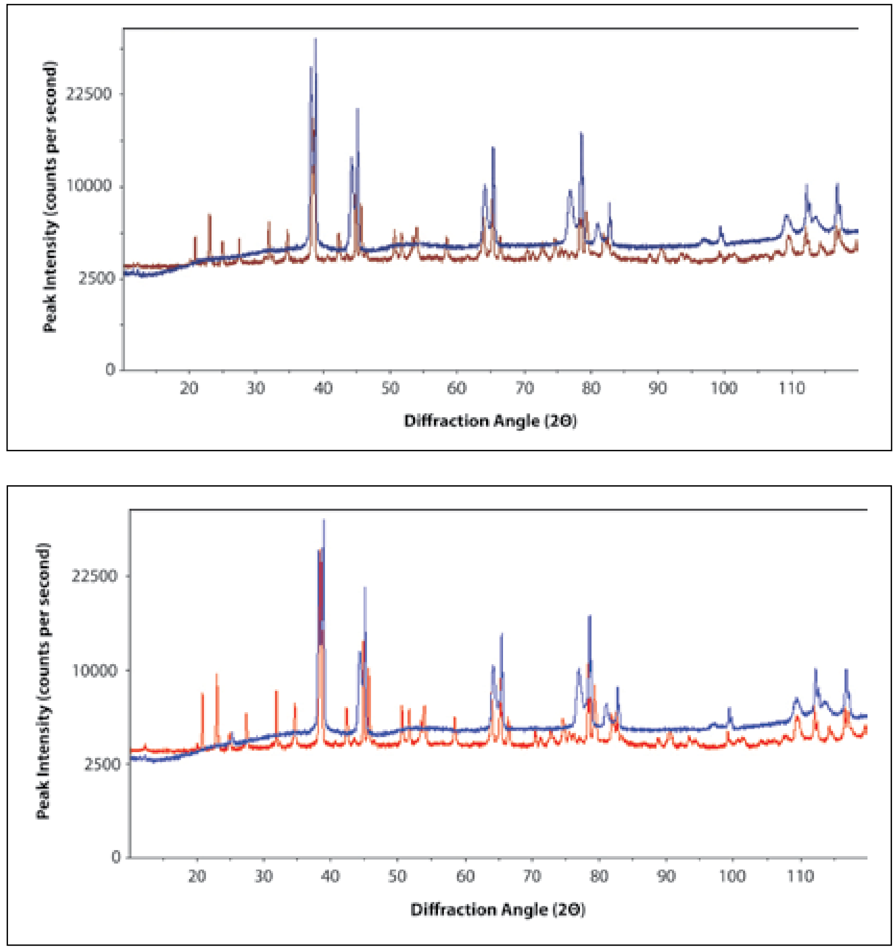
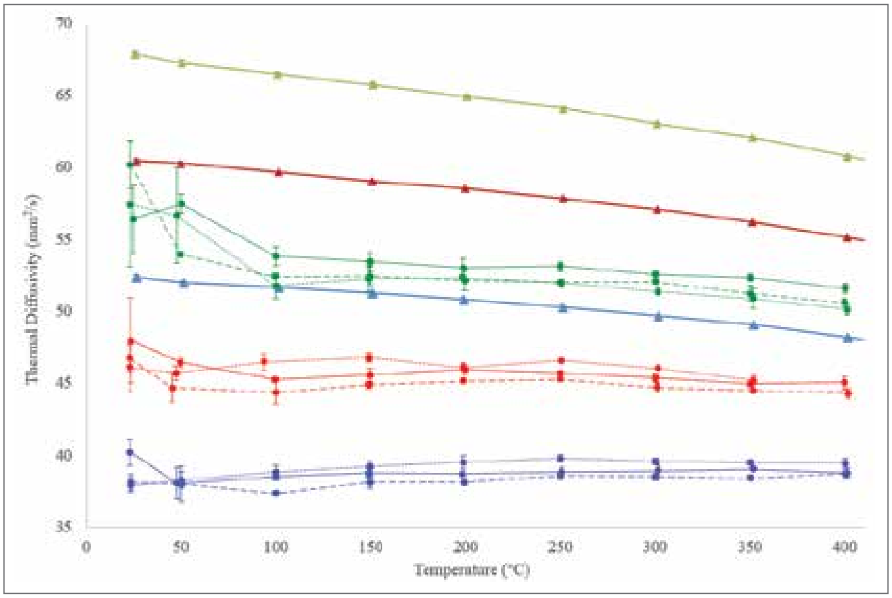


Figure 3. Diffraction patterns of heat treated and non-heat treated samples that have undergone (a) three cycles of irradiation, and (b) four cycles of irradiation.

Successful completion of the project will: (1) provide necessary data for the development of fast neutron test capability at ATR, (2) fill a knowledge gap on the basic properties of the Al_3Hf intermetallic and Al_3Hf-Al composite, and (3) advance the scientific understanding of the irradiation effects on these materials. The end result, in terms of data and fundamental understanding obtained, will directly support DOE's mission and benefit the science community in general.

Accomplishments

During FY 2015, a 3-D microstructural reconstruction was performed for irradiated specimens of the $HfAl_3$-Al metal matrix composite material developed for the Utah State University NSUF project. Focused Ion Beam (FIB) milling and Electron Backscatter Diffraction was done using the FEI Quanta 3-D field emission gun (FEG) located at the Center for Advanced Energy Studies (CAES). The gallium ions from the FIB were found to be



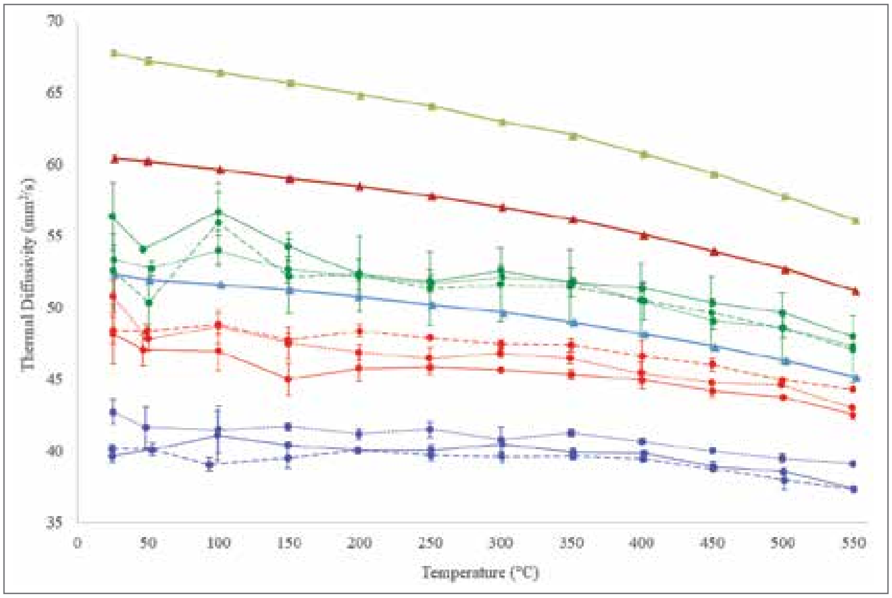


Figure 4. Comparison of measured thermal diffusivity of unirradiated with unannealed irradiated material (top) and annealed irradiated material (bottom).

Irradiated Data: 36.5 vol%, 28.4 vol%, 20 vol% Al_3Hf

Unirradiated Data: 36.5 vol%, 28.4 vol%, 20 vol% Al_3Hf

"The insight provided into the changes in properties of this new material resulting from irradiation have enormous value to the nuclear community, as well as to the students who have been inspired to pursue advanced degrees."

— Donna Post Guillen, Distinguished Research Engineer

very damaging to the $HfAl_3$-Al; thus, a new procedure was developed to enable the acquisition of acceptable Kikuchi patterns. The serial scans were reconstructed using Dream.3D software and visualized using Para-View. This work is pioneering in that 3-D microstructural reconstruction has never before been attempted for this material.

Post-irradiation examination completed during FY 2015 includes: (1) measurements of thermal conductivity using the laser flash method and specific heat using differential scanning calorimetry on nine specimens, (2) thermal expansion measurements on three specimens, (3) 3-D EBSD of one specimen, and (4) transmission electron microscope (TEM)/local electrode atom probe (LEAP) of one specimen.

Future Activities

Research yet to be completed on the irradiated material includes tensile and hardness tests on 12 specimens. This will be conducted at PNNL's Material Science and Technology Laboratory. Also to be completed are thermal diffusivity and differential scanning calorimetry measurements on the irradiated intermetallic material.

Publications and Presentations*

1. Guillen, D. P., and H. Ban, "Characterization and Modeling of a New Material for Nuclear Reactor Applications," *CAES Materials, Modeling, Simulation, and Visualization Workshop, Shore Lodge, McCall, Idaho, May 13–14, 2015.*

2. Guillen, D. P., and H. Ban, "Radiation Effects on the Thermophysical Properties of a New Neutron Absorbing Material," *2015 TMS Meeting, Orlando, Florida, March 15–19, 2015.*

3. Guillen, D. P., and H. Ban, "Development of a Metal Matrix Composite Material for Nuclear Reactor Applications," *2015 TMS Meeting, Orlando, Florida, March 15–19, 2015.*

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

This NSUF project has enabled us to obtain the first-ever data on a new two-phase metal matrix composite material developed to enable the testing of fast reactor fuels and materials in an existing LWR.

Distributed Partnership at a Glance

NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor, Hot Fuel Examination Facility Analytical Laboratory, Electron Microscopy Laboratory
Pacific Northwest National Laboratory	Materials Science & Technology Laboratory
Collaborators	
Idaho National Laboratory	Donna Post Guillen (principal investigator)
University of Nevada, Las Vegas	Thomas Hartmann (co-principal investigator)
Utah State University	Heng Ban (principal investigator), Zilong Hua (postdoctoral researcher)

Advanced Damage-Tolerant Ceramics:
Candidates for Nuclear Structural Applications

Michel W. Barsoum – Drexel University – barsoumw@drexel.edu

The MAX phases, a class of machinable, layered, ternary carbides, and nitrides, have great promise for use in the next-generation of nuclear reactors. This is the first time the MAX phases have been neutron irradiated at temperatures as high as those carried out here.

This project is a collaborative effort between the Idaho National Laboratory (INL), Savannah River National Laboratory, and Drexel University aiming to explore the neutron irradiation response of MAX phases (i.e., Ti_3SiC_2 and Ti_3AlC_2) for advanced nuclear applications. Samples of each composition were irradiated in the Advanced Test Reactor (ATR), with nominal irradiation conditions of 0.1, 1, and 9 dpa at 100, 500, and 1000°C. Post-irradiation examination was performed at the Center for Advanced Energy Studies, including X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), and resistivity testing.

Project Description

Robust materials are critical to meet evolving advanced reactor and fuel designs. These materials need to operate in extreme environments of elevated temperatures, corrosive media, and high-radiation fluences, with lifetime expectation of greater than 60 years. Full understanding of a material's response to irradiation is paramount to long-term, reliable service. The layered ternary carbides and nitrides, known as MAX phases, have the potential to be used in the next-generation nuclear reactors. All MAX phases are fully machinable even though some of them, such as Ti_3SiC_2 and Ti_3AlC_2, are similar to titanium metal in density, but are three times as stiff. The thermal and electrical conductivities are high and metal-like. They have relatively high-fracture toughness values and some are chemically stable in corrosive environments. They also have shown irradiation damage tolerance in heavy ion studies.

The aim of this project is to investigate the damage in Ti_3SiC_2, Ti_3AlC_2, and chemical vapor deposition SiC (for comparison) after exposure to a spectrum of neutron irradiations consistent with conditions found in light water nuclear reactors. The carbides are exposed to a series of neutron fluence levels (0.1, 1, and 9 dpa) at moderate to high irradiation temperatures (100, 500, and 1000°C) in the ATR at INL. The damage to the microstructures and the effects of the radiation on the mechanical and

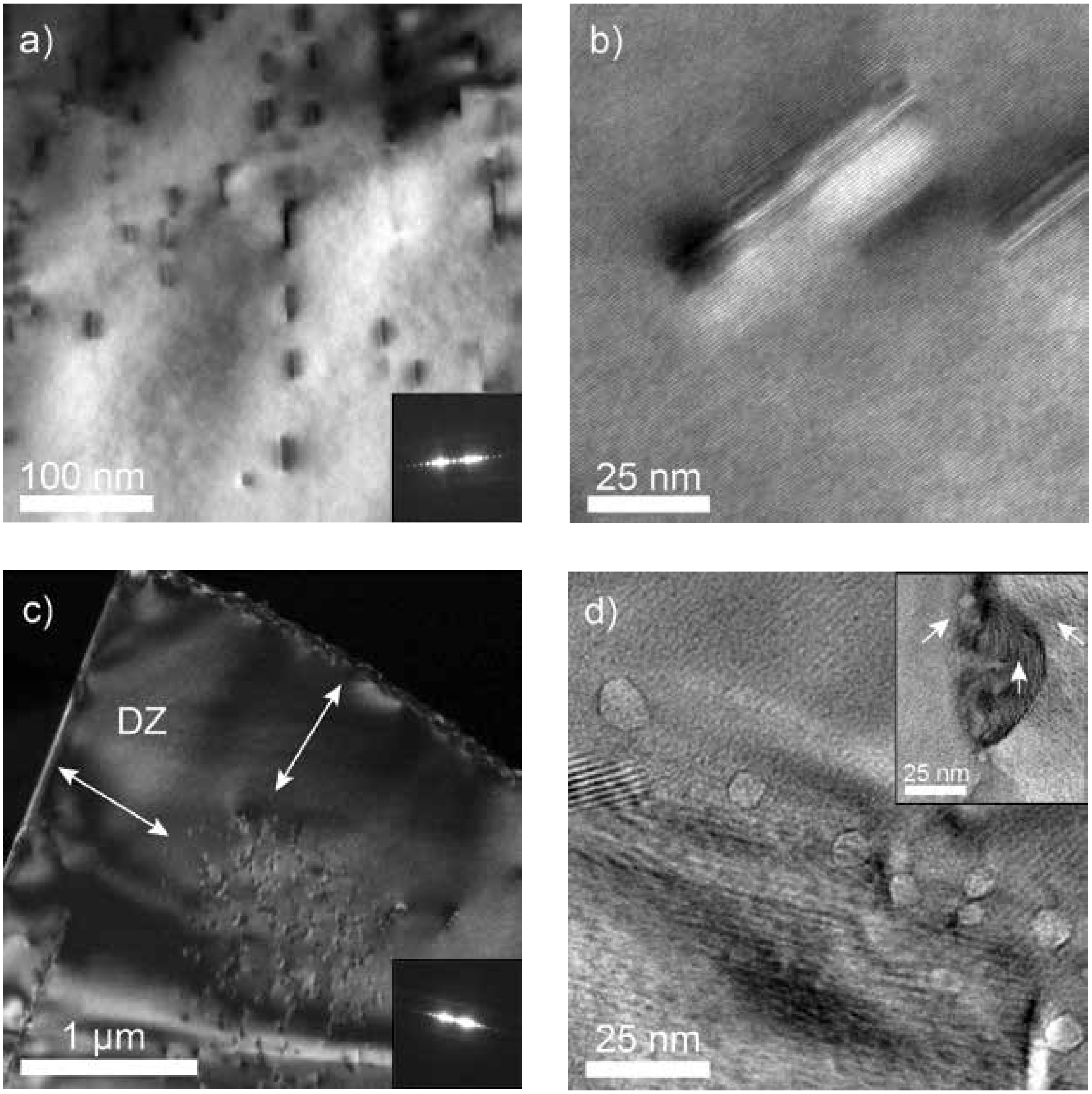


Figure 1. Brightfield TEM micrographs of Ti_3SiC_2 irradiated to 9 dpa at (a) 500°C showing dislocation loops imaged near the [11 $\bar{2}$0] zone axis, with an average loop diameter of 30(8) nm and a loop density of 2 x 1020 loops/m^3. (b) High resolution image of several loops in (a) exhibit a complex interaction of several loop stain fields arrayed along the c-axis. (c) Darkfield TEM micrographs of the same sample showing denuded zones, DZ (arrows), of 860(90) nm wide along both the a and c-lattice directions. (d) Brightfield micrograph showing spherical voids, which formed occasionally at the grain boundaries at this condition, with an average diameter of 7(2) nm. Inset of (d) shows small nanograins of Ti_3SiC_2 that were observed at the boundaries, which grew in different orientations to either parent grains (white arrows denoting a-axis direction).

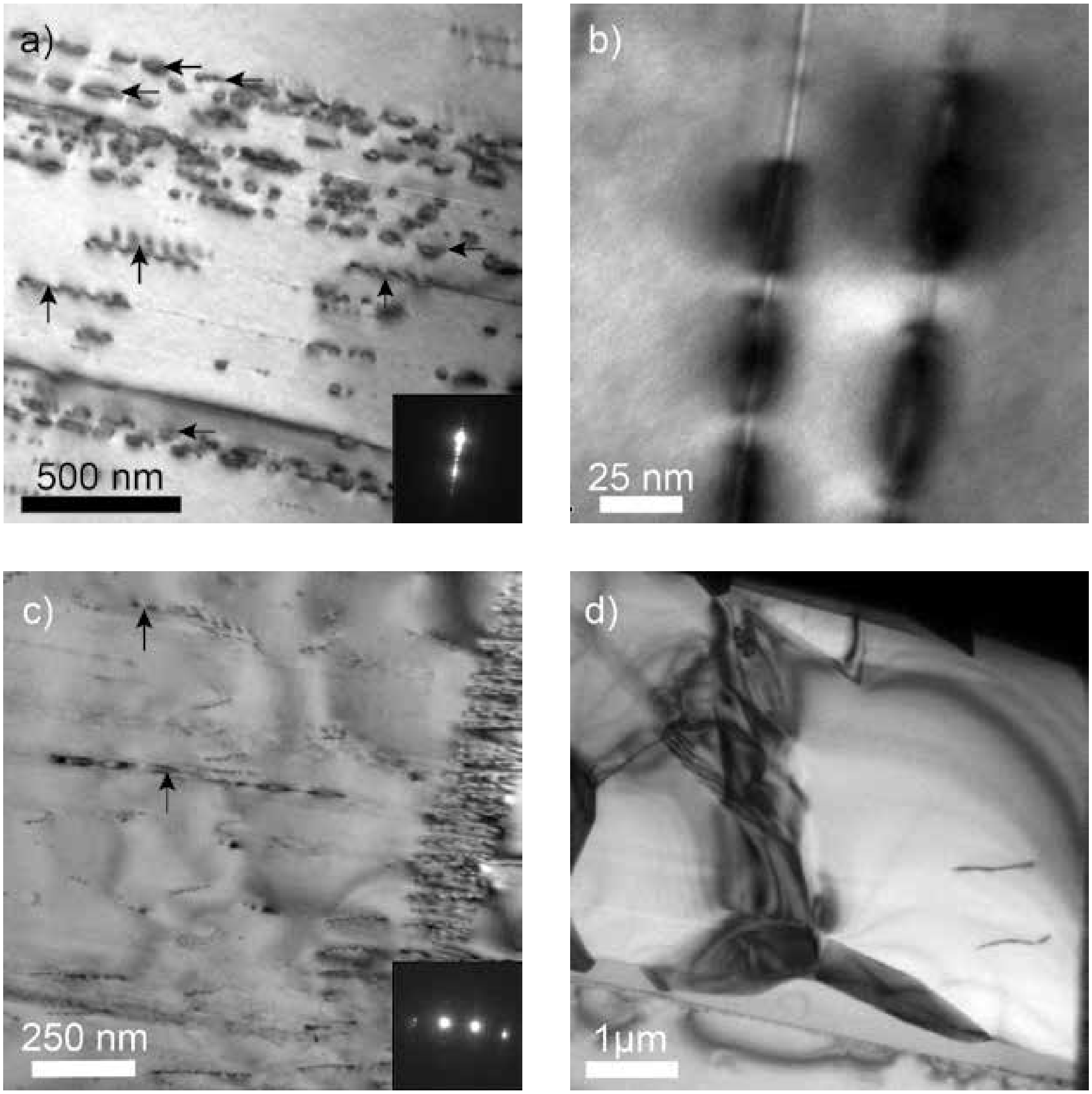


Figure 2. Bright Field TEM micrographs of Ti_3SiC_2 irradiated to 9 dpa at 1000°C reveal (a) basal dislocations (vertical arrows) and dislocation loops (horizontal arrows) pinned at stacking faults dispersed throughout a large grain, on average 63(25) nm in dia. with a density of 4 x 1019 loops/m^3, only seen near stacking faults. (b) High-resolution micrographs of the loops in (a) reveal the c-axis strain contrast of the loop. (c) Tilting of this sample to g $1\bar{1}00$ results in the loss of contrast for the b = ½ [0001] loops, while basal dislocations and arrays remain visible. (d) Smaller grains, and others without stacking faults, show no signs of irradiation induced dislocation loops.

electrical properties of the materials will be characterized during post-irradiation examinations. The results will provide an initial database that can be used to assess the micro-structural responses and mechanical performances of these ternaries.

Accomplishments

As of FY 2015, this project has been deemed completed. The irradiation of samples of Ti_3SiC_2 and Ti_3AlC_2 was conducted throughout the first two years of this project, which were then left to cool awaiting characterization. Due to unavoidable delays within the INL facilities, work on this project was delayed throughout FY 2013. In FY 2014, the receipt, cask unloading, experiment disassembly, and cataloguing of specimens were successfully accomplished, led by Collin Knight and Karen Wright. Upon examination, several samples were found to be fused together, notably in the 100°C capsules at higher irradiation conditions, and were unavailable for characterization. Work continued throughout FY 2014 and into FY 2015 as samples of Ti_3SiC_2 and Ti_3AlC_2, most desirably those irradiated to 9 dpa, became available for characterization.

Samples were mounted in metallographic epoxy to protect the workers from radiation exposure, and were first analyzed using XRD for phase and structure analysis. According to the Rietveld refinement of the XRD patterns collected, both Ti_3SiC_2 and Ti_3AlC_2 remained crystalline after irradiation, and both resulted in limited TiC formation. After irradiation at 100°C, a-LPs decreased and the c-LPs increased. Conversely, the lattice parameters of samples irradiated at all doses at 500 and 1000°C, were close to the pristine values, indicating that dynamic recovery was occurring at temperatures as low as 500°C.

With the collaboration of Lingfeng He at INL, extensive TEM work was conducted to explore the irradiation induced defects in Ti_3SiC_2 and Ti_3AlC. Dislocation loops with b = 1/2[0001] were observed in samples of Ti_3SiC_2 irradiated at 500°C, 21(6) nm dia. at 1 dpa, and 30(8) nm at 9 dpa (Figure 1a,b). In the Ti_3SiC_2 samples irradiated to 9 dpa, both at 500 and 1000°C, voids were observed within the grain boundaries (Figure 1d).

The MAX phases show great potential for irradiation damage tolerance due to their nanolayered structure.

— Darin J. Tallman, Ph.D. Research Assistant

Further, at 63(25) nm and 4 X 1019 loops/m^3, the largest loops in Ti_3SiC_2 were observed after irradiation to 9 dpa at 1000°C (Figure 2). However, these were only observed near stacking faults, and the majority of grains imaged showed no signs of irradiation damage in the bulk (Figure 2d). At 70(25) nm, the dislocations loops in Ti_3AlC_2 after irradiation to 9 dpa at 500°C were larger, and more numerous with a density of 8 X 1020 loops/m3 (Figure 3). Basal perturbations were also observed in Ti_3AlC_2 at these conditions (Figure 3d). TiC impurity particles were significantly more susceptible to irradiation damage, forming extensive defect clusters and dislocation loop networks (Figure 4).

Even more notable is the appearance of a large defect-free denuded zone, nearing 1 µm in size, in Ti_3SiC_2 irradiated to 9 dpa at 500°C (Figure 1c). Furthermore, at 1000°C, most grains on the order of 3–5 µm appear to be free of damage altogether (Figure 2d). This finding unequivocally demonstrates the ease of mobility of defects along the basal planes, and the lack thereof in the impurity particles.

These results confirm our initial conjecture that we postulated when we started this work. Namely, the A-layer in the MAX phases, sandwiched between hard M3 X 2 blocks, would provide stable defect accommodation sites, allowing for point defect accumulation, migration, and their ultimate annihilation. With increased irradiation temperature, the ease of migration along the basal planes allows for enhanced recovery of irradiation defects, resulting in the formation of coherent dislocation loops or annihilation of defects at the grain boundaries. The results from this work show that the MAX phases, notably Ti_3SiC_2, are able to withstand neutron irradiation damage, and recover from microstructural distortion with high-temperature irradiation. The project has thus provided the foundation for future experimental and theoretical studies for this promising family of materials for nuclear applications.

Future Activities

This project has concluded. No further work is expect to be performed. Samples will be transferred to the NSUF Sample Library and are available for future proposals.

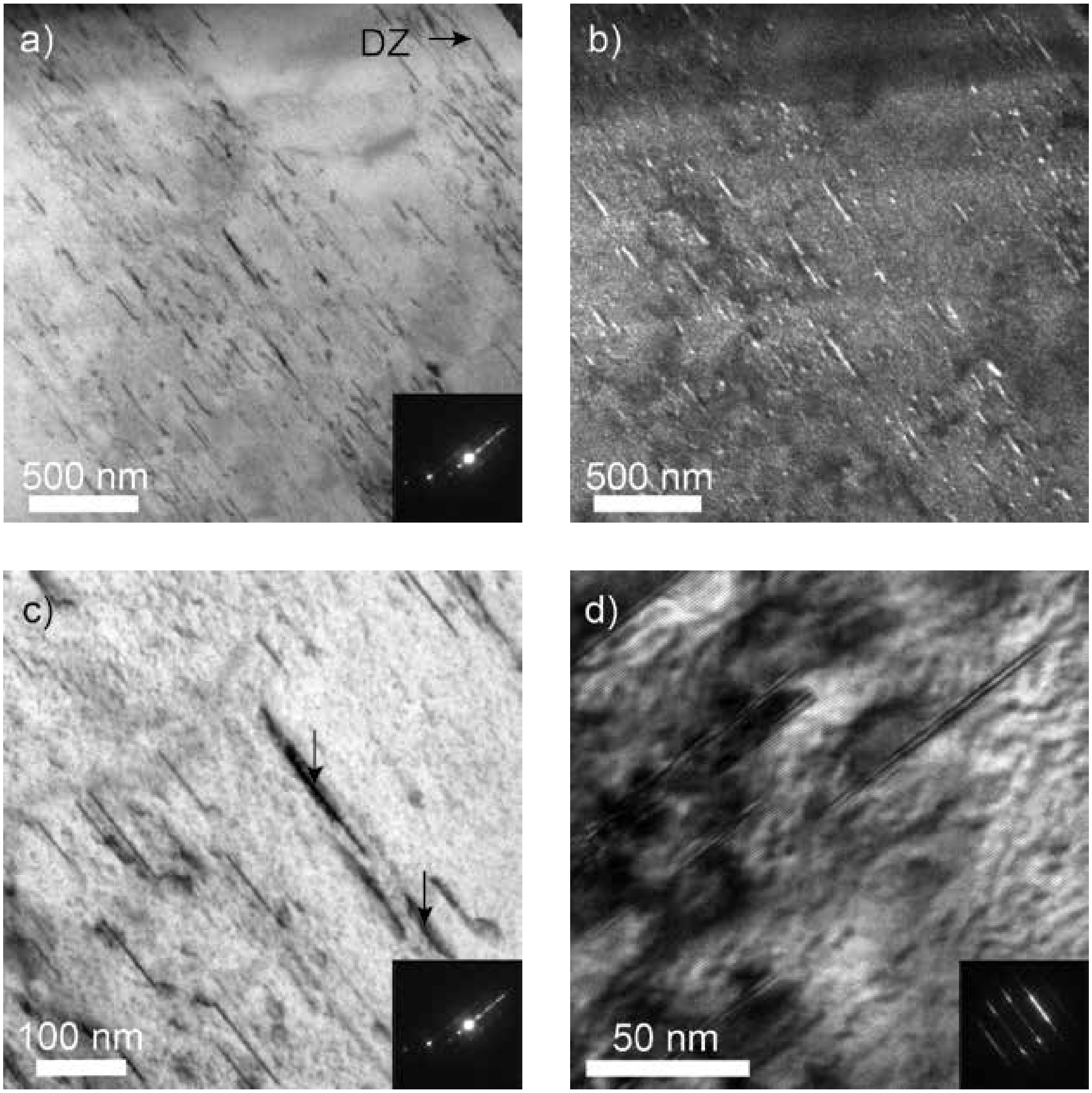


Figure 3. (a) Brightfield and (b) darkfield TEM micrographs of Ti_3SiC_2 irradiated to 9 dpa at 500°C showing dislocation loops and stacking faults imaged near the [11 $\bar{2}$0] zone axis with an average loop diameter of 70(25) nm and a loop density of 8 x 1020 loops/m^3. A denuded zone, DZ, of 140 nm can be seen at the grain boundary. (c) Higher magnification of a region in (a) reveals short stacking faults (black arrows), with a lack of Al content determined by EDS, and dislocation loops. (d) High-resolution micrographs of the loops in this region show extensive basal plane perturbation and loops within the basal planes.

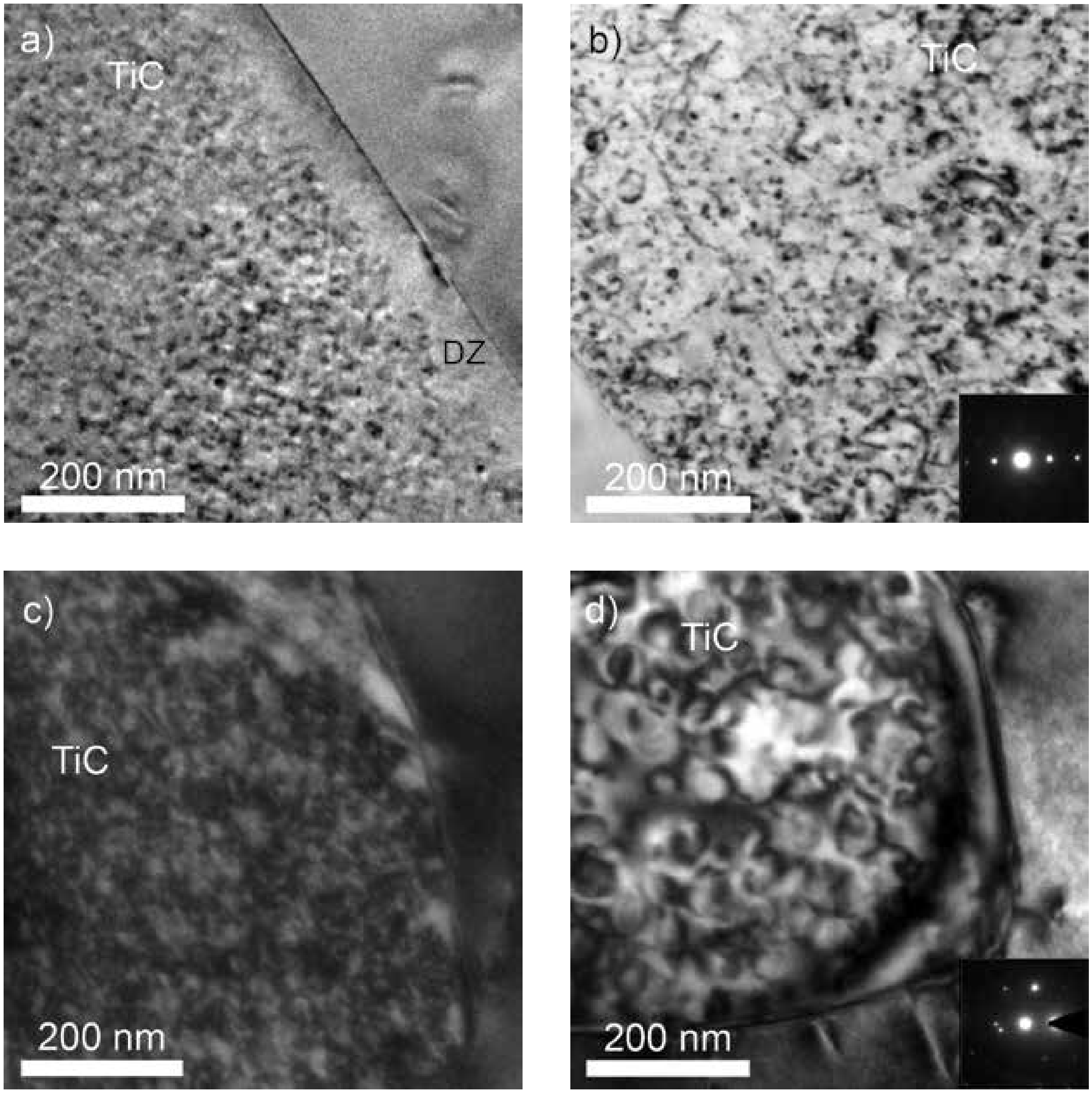


Figure 4. Brightfield TEM micrographs of TiC impurity particles in (a) Ti_3SiC_2 irradiated to 0.1 dpa at 1000°C, (b) 1dpa at 500°C, and (c) 9 dpa at 500°C, and (d) TiC in Ti_3AlC_2 irradiated to 0.1 dpa at 500°C. All TiC particles observed exhibited the formation of defect clusters and dislocation loops after irradiation, extensively more so than the surrounding MAX phase matrix.

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1. Tallman, D. J., L. He, E. N. Hoffman, E. N. Caspi, B. L. Garcia-Diaz, G. Kohse, R. L. Sindelar, M. W. Barsoum, "Neutron Irradiation of MAX Phases," *Presented at Il Trovatore Meeting 2, Philadelphia, Pennsylvania, July 24, 2015.*

2. Tallman, D. J., L. He, E. N. Hoffman, E. N. Caspi, B. L. Garcia-Diaz, G. Kohse, R. L. Sindelar, M. W. Barsoum, "Nuclear Scientific User Facility: Neutron Irradiation of MAX Phases," *Presented at NSUF User's Week, Idaho Falls, Idaho, June 23, 2015.*

3. Tallman, D. J., L. He, G. Bentzel, E. N. Hoffman, B. L. Garcia-Diaz, G. Kohse, R. L. Sindelar, M. W. Barsoum, "Microstructural Defects in Neutron Irradiated Ti_3SiC_2 and Ti2AlC," *Presented at ICACC'15, Daytona Beach, Florida, January 28, 2015.*

4. Tallman, D. J., L. He, B. L. Garcia-Diaz, E. N. Hoffman, G. Kohse, R. L. Sindelar, M. W. Barsoum, Effect of neutron irradiation on defect evolution in Ti_3SiC_2 and Ti_2AlC, *Journal of Nuclear Materials, 468, 2016, pp. 1–13.*

5. Tallman, D. J., "On the potential of MAX phases for nuclear applications," Ph.D. Thesis, Drexel University, Philadelphia, Pennsylvania, 2015.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and characterization Suite
Idaho National Laboratory	Advanced Test Reactor, Hot Fuel Examination Facility Analytical Laboratory
Massachusetts Institute of Technology	Nuclear Reactor Laboratory
Collaborators	
Drexel University	Michel Barsoum (principal investigator), Darin Tallman (collaborator)
Idaho National Laboratory	Jian Gan (principal investigator), Lingfeng He (collaborator)
Savannah River National Laboratory	Brenda L. Garcia-Diaz (collaborator), Elizabeth N. Hoffman (collaborator)

Hydride LWR Fuel Rod Irradiation

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The principal difference between oxide and hydride fuel is the high-thermal conductivity of the latter. This feature greatly decreases the temperature drop over the fuel during operation, thereby reducing the release of fission gases to the fraction due only to recoil. The maximum fuel temperature can be further reduced by filling fuel-cladding gap with low-melting LM instead of He.

The objectives of this experimental research are to study the materials issues associated with the use of a hydride fuel for power production in light water reactors (LWRs) and to explore the use of Pb-Bi Eutectic liquid metal (LM) as a replacement for helium in hydride fuel elements. Hydrides provide a number of improvements, including the addition of hydrogen neutron moderation within the fuel, thermally induced hydrogen up-scattering that accompanies Doppler Feedback that improves safety, and higher efficiency in elimination of plutonium than achievable with mixed oxide (MOX). The liquid metal bounded fuel-cladding gap assist in lowering the temperature of the fuel.

Project Description

Feasibility and benefits of incorporation of hydride nuclear fuels into the current fleet of LWRs have been investigated in detail using neutronic and thermal-hydraulic calculations and laboratory-scale materials experiments by the materials group in Nuclear Engineering Department at the University of California at Berkeley. Recognizing the necessary shift from laboratory-scale experiments to more relevant environments, an irradiation experiment was then conducted to evaluate the feasibility of an LWR hydride fuel. Sealed fuel rodlets were constructed with U0.17ZrH1.6 fuel pellets and conventional Zircaloy-2 cladding with Bi-Pb LM filled-gap, inner and outer surfaces oxidized to nominal 1 micron to prevent hydrogen attack. They were housed in Ti capsules surrounded with the same LM used for filling gaps between the fuel and cladding. Three irradiated Ti capsules were then irradiated at the Massachusetts Institute of Technology (MIT) research reactor to burnups of 0.19, 0.17, and 0.29 FIMA (%) with up to a maximum 6-MW thermal power. Along with a fourth unirradiated rodlet for reference use, the capsules were then transferred to PNNL for extensive post-irradiation evaluations. In this fiscal year, novel tools were designed and fabricated to handle and extract the highest-burnup rodlet (0.29% FIMA) from the outer Ti capsule. Thin-sliced samples were prepared at two axial locations of the rodlet: close to the axial mid-level of the rod where TC tips were located, and near to the end of rodlet. The full-round cross-sections were mounted and polished to observe the fuel, gap LM, outer LM, cladding, and their interfaces. Because of radioactivity and pyrophoricity concerns, metallographic samples preparation were performed in hot cells under flowing inert gas.

The irradiated samples were evaluated in detail and compared to unirradiated sliced sample from the similar axial location through visual inspection for possible crack development, optical microscopy for detecting dimensional changes in the fuel, cladding, and the gaps and scanning electron microscopy

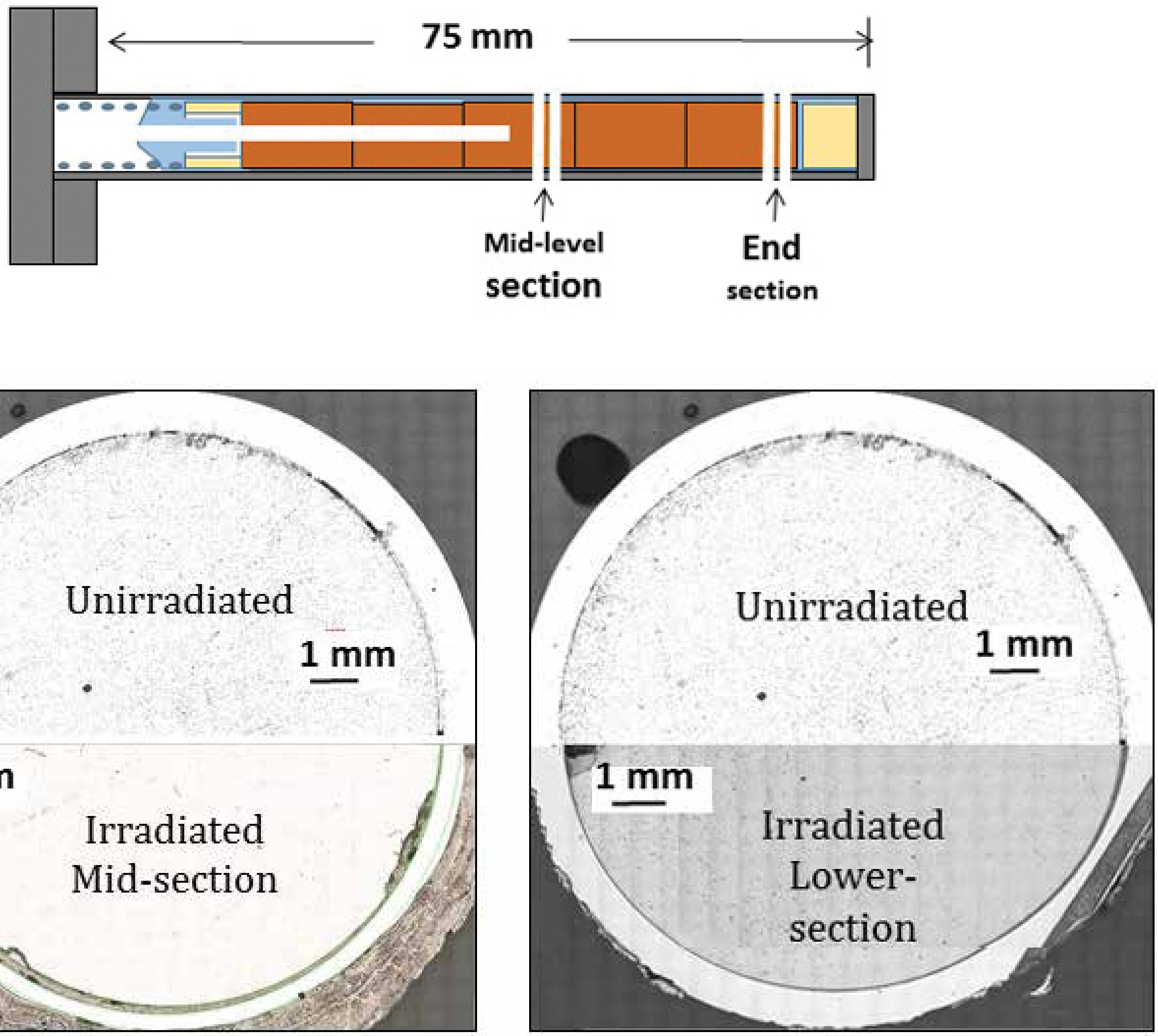

Figure 1. Mid- and lower-level cross-sectional optical images of irradiated fuel rodlet as overlapped to unirradiated fuel.

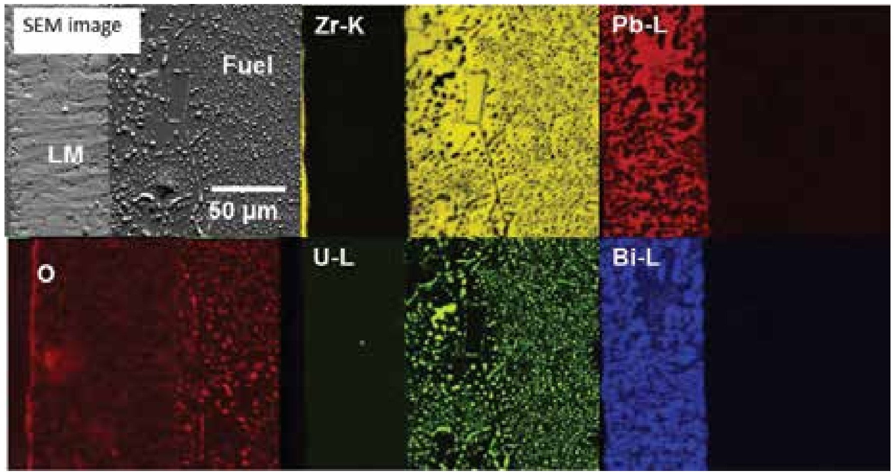

Figure 2. Elemental map including inner liquid metal, fuel, and cladding of the unirradiated fuel rodlet.

Collaboration with first-class material scientists and access to PNNL advanced facilities resulted in the successful completion of the project.

— Mehdi Balooch, Co-principal Investigator

with X-ray microanalysis (SEM/ energy dispersive X-ray spectroscopy [EDS]) for detection of swelling, void formation, corrosion of cladding, dissolution of zircaloy and fuel in LM gap, and finally LM chemical changes due to temperature cycles during irradiation.

Accomplishments

In this progress report, we present the post-irradiation analysis of one of the rodlets, the 0.29% FIMA, using high-resolution optical and SEM/EDS at two height-level axial positions (mid- and lower-level).

Comparing the unirradiated with irradiated fuel rod images by high-resolution optical microscopy (Figure 1) we have observed extensive thinning, mainly from the outside surface of the cladding at the mid-level cross section, where the cladding prematurely failed during irradiation at the MIT reactor. This persuaded, but to much lesser extent, at lower-level cross section where it was limited to ~8%. Limited availability of the LM for dissolution of Zr in the fuel-cladding gap impeded thinning of the inside surface of the cladding to negligible amount. In contrast, the larger outer LM reservoir was a more-efficient sink for dissolution of the cladding, which resulted in massive and surprising thinning of the cladding at mid-level. This zircaloy loss is an artifact of the experiment. In actual use, the outer cladding surface is contacted by water, not by LM.

The loss of cladding material, suggests the dissolution of Zr in LM passes beyond the solubility limit. But it continued by generating new phases such as $ZrBi_2$, ZrBi, Zr_3Bi, Zr_5Pb_3, and possibly $Zr_{3.33}$ $Pb_{0.67}$. U phase of UBi_2 may also be formed. High-resolution elemental imaging of unirradiated and irradiated fuel (Figures 2 and 3) reveal the dissolved Zr was distributed uniformly in the LM gap. The fuel surface interaction with the LM gap was limited—some evidence of dispersed micron-size uranium phase of the fuel in the LM gap adjacent to the fuel surface and its aggregation close to the inner cladding surface was found. The bulk of the hydride fuel appeared intact—no detectable swelling of the fuel, or its microstructural changes within the capabilities of the instruments was observed.

The dissolution of 8% of zircaloy-2 cladding in lead-bismuth eutectic Bi-Pb LM during 1000 hours of exposure was found excessive for use as a gap-filler in a nuclear reactor. However, the addition of

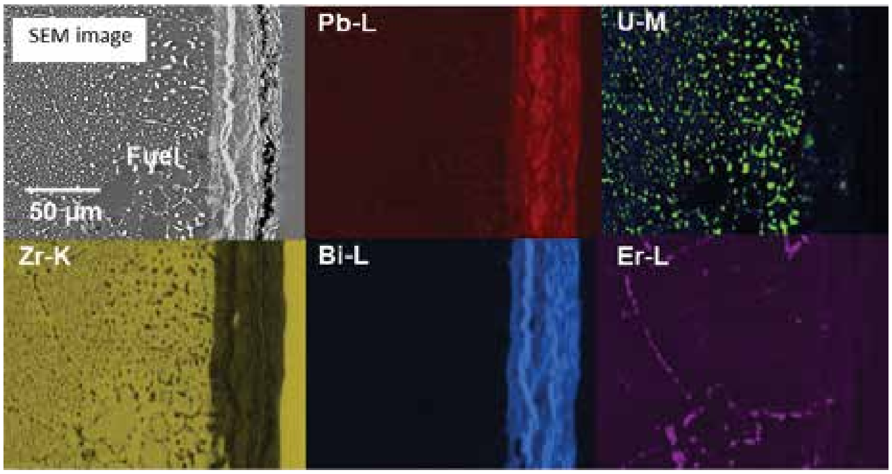

Figure 3. SEM and elemental maps of Pb, Bi, U, and Zr and Er revealing fuel and cladding interaction at their interface of the lower-level section of irradiated fuel rodlet.

third component such as Sn, may limit the dissolution by forming intermetallic compounds that provide a barrier to some and extend kinetically, bringing it closer to an acceptable level.

Future Activities

This project is now complete.

Publications and Presentations*

1. Casella, A. M., D. J. Senor, E. C. Buck, D. R. Olander, K. A. Terrani, P. Hosemann, M. Balooch, "Post Irradiation Experiments on Hydride LWR Fuel Rodlet" In preparation.
2. Kima, S. J., D. Carpentera, G. Kohsea, L-w. Hua, "Hydride fuel irradiation in MITR-II: Thermal design and validation results," *J. Nuc. Mat.*, Vol. 277, 2014, pp. 1–14.
3. Terrani, K. A., M. Balooch, and D. R. Olander, "LWR Hydride Fuel Irradiation: Experiment Design and Fabrication" In preparation.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Massachusetts Institute of Technology	Nuclear Reactor Laboratory
Collaborators	
Idaho National Laboratory	James Cole (principal investigator)
Pacific Northwest National Laboratory	Andrew Casella (collaborator), David Senor (collaborator)
University of California, Berkeley	Donald Olander(principal investigator), Mehdi Balooch (co-principal investigator)

Transducers for In-pile Ultrasonic Measurements of Fuels and Materials Evolution

Bernhard Tittmann – Pennsylvania State University – brt4@psu.edu

Figure 1. 3-D renderings of the ULTRA-capsule as constructed for the irradiation. The piezoelectric sensors are shown in yellow, the magnetostrictive sensors are shown in green, the piezoelectric drop in specimens are shown as gray disks, while the magnetostrictive drop-in samples are shown in a peach cylinder.

Current generation light water reactors (LWRs), sodium cooled fast reactors (SFRs), small modular reactors (SMRs), and Next Generation Nuclear Plants (NGNPs) produce harsh environments in and near the reactor core that can severely tax material performance and limit component operational life. To address this issue, several Department of Energy Office of Nuclear Energy (DOE-NE) research programs are evaluating the long-duration irradiation performance of fuel and structural materials used in existing and new reactors. To maximize the amount of information obtained from Material Testing Reactor (MTR) irradiations, DOE is also funding development of enhanced instrumentation that will be able to obtain in-situ, real-time data on key material characteristics and properties with unprecedented accuracy and resolution. Such data are required to validate new multi-scale, multi-physics modeling tools under development as part of a science-based, engineering driven approach to reactor development. It is not feasible to obtain high-resolution/microscale data with the current state of instrumentation technology.

Project Description

Ultrasound-based sensors offer the ability to obtain in-situ data if it is demonstrated that these sensors and their associated transducers are resistant to high-neutron flux, high-gamma radiation, and high temperature. To address this need, the Nuclear Science User Facilities (NSUF) funded an irradiation, led by Pennsylvania State University (PSU), at the Massachusetts Institute of Technology (MIT) Research Reactor to test the survivability of ultrasound transducers.

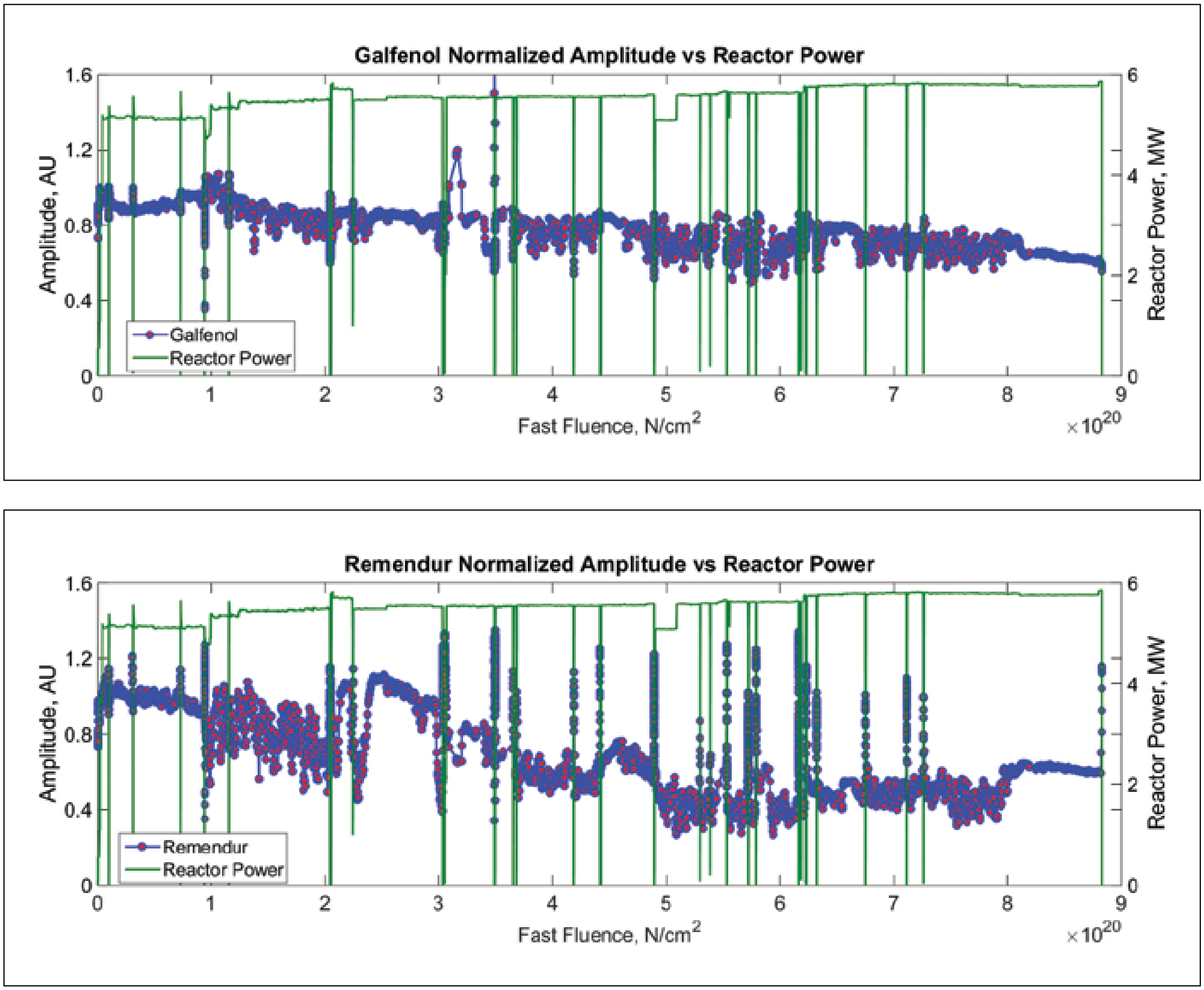

Figure 2. Pulse-echo amplitude for magnetostrictive transducers as a function of reactor power.

The feasibility of ultrasonic transducers in a nuclear reactor has been established. This opens the door to leave-in-place sensors for in-reactor conditions and materials.

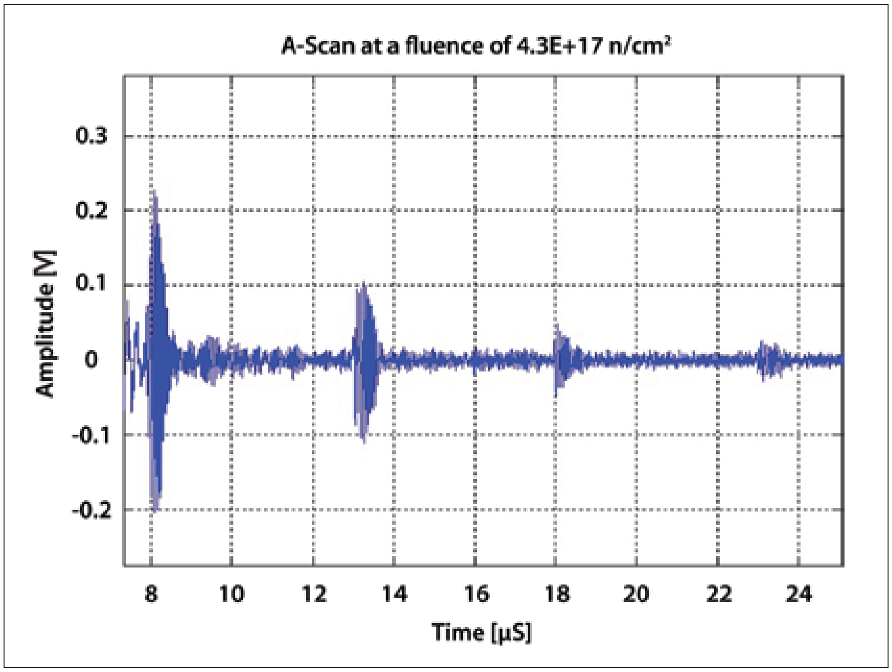

Figure 3. Example waveform from an elapsed time of 4 days.

AlN survived 18 months of irradiation.

— Bernhard Tittmann, Professor

As part of this effort, PSU and collaborators have designed, fabricated, and tested piezoelectric and magnetostrictive transducers that are optimized to perform in harsh, high flux environments. Four piezoelectric transducers were fabricated with aluminum nitride (AlN), zinc oxide (ZnO), and bismuth titanate (BiT) as the active elements and two magnetostrictive transducers were fabricated with remendur and galfenol as the active elements.

Accomplishments

The irradiation was performed in the MIT Reactor for a period of 18 months. First and most importantly, the successful operation of the transducers are shown at integrated neutron fluence of approximately 8.68 E+20 n/cm^2 for n >1 MeV, temperatures in excess of 420°C, and a gamma fluence of 7.23 Gy/cm^2. Although the sensors could perform in such environments, it was not without some troubles. Some of the sensors had issues with electrical connection and mechanical coupling to the waveguide. This is demonstrated and explained in the context of the pulse-echo signals. Overall, this is the longest exposure experiment conducted to the researchers' knowledge on the chosen sensor materials and the first instrumented lead test for many of these materials, aside for aluminum nitride, which had previously been tested at PSU's Brezeale Research reactor. The magnetostrictive transducers were monitored by Dr. Joshua Daw from INL and were found unaffected by the irradiation.

Future Activities

Additional funding has been sought from Bettis and obtained for post-irradiation examination of the ultrasonic transducers to obtain detailed measurements after the capsules have been dissassembled. Physical appearance, electrical impedance values, ultrasonic pulse-echo amplitudes, piezoelectric performance, and vibrational Q values will be obtained.

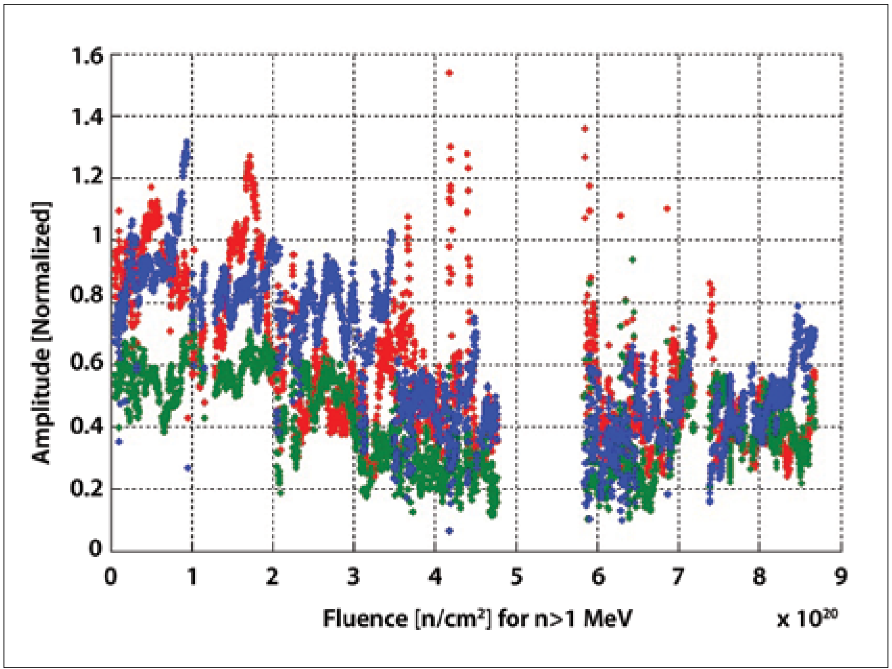

Figure 4. Change in ultrasonic pulse echo amplitude as a function of fluence for AlN transducer; only data points for which the power was greater than 5 MW are shown. The gap in data occurred during extended shutdown of the reactor.

Publications and Presentations*

1. Reinhardt, B., B. Tittmann, and A. Suprock, 2015, "Nuclear Radiation Tolerance of Single Crystal Aluminum Nitride Ultrasonic Transducer," *Physics Procedia*, 2015, in press.
2. Daw, J., J. Palmer, P. Ramuhalli, P. Keller, R. Montgomery, H-T. Chien, B. Tittmann, B. Reinhardt, G. Kohse, J. Rempe, J-F Villard, 2015, "Updated Results of Ultrasonic Transducer Irradiation Test," *4th Advancements in Nuclear Instrumentation Measurement Methods and their Applications (ANIMMA 2015) Lisbon, Portugal, April 20–24, 2015.*
3. Daw, J., J. Rempe, J. Palmer, P. Ramuhalli, R. Montgomery, H-T. Chien, B. Tittmann, B. Reinhardt, and G. Kohse "Ultrasonic Transducer Irradiation Test Results," *9th International Conference on Nuclear Plant Instrumentation, Control & Human–Machine Interface Technologies (NPIC & HMIT 2015), Charlotte, North Carolina, February 21–26, 2015.*

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

Distributed Partnership at a Glance

NSUF and Partners	Facilities and Capabilities
Massachusetts Institute of Technology	Nuclear Reactor Laboratory
Collaborators	
Argonne National Laboratory	H.T. Chien (collaborator)
Bettis	Ben Wernsman (collaborator)
Commissariat à l'Energie Atomique	Brian Reinhardt (collaborator)
Idaho National Laboratory	Joy Rempe (principal investigator), Joshua Daw (co-principal investigator)
Massachusetts Institute of Technology	Gordon Kohse (collaborator)
Pacific Northwest National Laboratory	Pradeep Ramuhalli (collaborator)
Pennsylvania State University	Bernhard Tittmann (principal investigator)

Synchrotron X-Ray Characterizations of Advanced Accident-tolerant Cladding

Kun Mo – Argonne National Laboratory – mokun2004@gmail.com

Advanced intermetallic coatings on zircaloy cladding have great potential for improving resistance to oxidation and radiation damage.

This research aims to characterize the microstructural development of intermetallic coated zircaloy, an advanced cladding material, after heavy ion irradiation. The intermetallic coatings on the zircaloy were developed using atomic layer deposition (ALD) technique. With the protection of the coating, the oxidation resistance of the zircaloy has been significantly improved. However, the performance of the coating in a highly radioactive environment is not yet understood. In this study, the synchrotron X-ray available at the Materials Research Collaboratory Access Team (MRCAT) beamline at the Advanced Photon Source (APS) was utilized to study the materials' performance after high-energy heavy ion irradiation. An X-ray absorption fine structure (XAFS) experiment was conducted to study the ion radiation damage on the zircaloy sample with advanced coatings.

Project Description

Advanced intermetallic coatings have great potential for improving resistance to oxidation and radiation damage in nuclear materials. The Fukushima Daiichi nuclear accident has clearly demonstrated to the world that improvement of the safety performance of operating light water nuclear reactors (LWRs) is an imminent need. The reactors at the Fukushima Daiichi Nuclear Power Plant experienced a loss of coolant accident (LOCA) during which the zirconium alloy (zircaloy) cladding (sealed tubes that protect the UO^2 fuel material from contact with the environment) experienced severe oxidation. Large amounts of hydrogen were generated as steam reacted with zirconium at high temperature, eventually leading to hydrogen explosions. In response to the accident, the U.S. Department of Energy (DOE) Office of Nuclear Energy (NE) has placed strong strategic importance on developing LWR fuels with enhanced accident

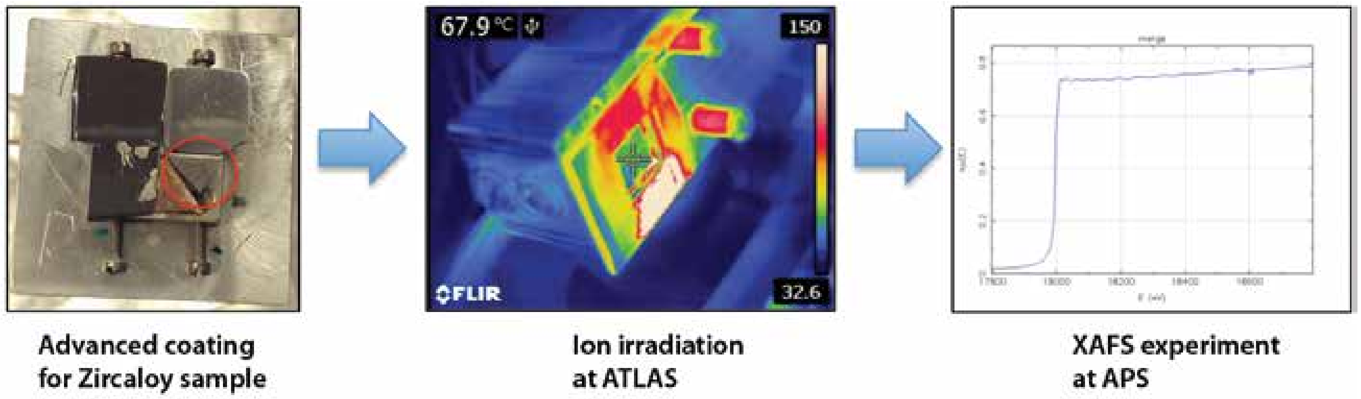

Figure 1. Experimental procedure.

tolerance characteristics. In comparison with standard UO_2+Zircaloy systems, an enhanced accident tolerant fuel/cladding system will release significantly less energy, resultant hydrogen, and fission products to the environment during beyond-design-basis LOCA events. In this context, improving the performance of zircaloy cladding under accident conditions has become a central issue of great interest to both DOE and the nuclear industry. Corrosion resistance (resistance to oxidation) of zircaloy has been studied since long before the Fukushima accident. Meanwhile, the ALD technique available at Argonne National Laboratory (ANL) has been demonstrated as a very effective coating technique, which significantly improves adhesion between the coating and the zircaloy compared to other coating techniques used in the past, such as vapor deposition techniques. Although the oxidation resistance has been significantly improved, the radiation resistance of the coating is unknown. This experiment aims to study the microstructural development of advanced cladding materials following heavy ion radiation. A comprehensive understanding of the performance of advanced cladding materials will be needed to validate their improvement in performance during both normal and transient scenarios.

Synchrotron X-rays provide us with unique insight about the radiation damage in advanced fuel cladding materials.

— Kun Mo, Materials Scientist, Nuclear Engineering Division, Argonne National Laboratory

Accomplishments

Zircaloy samples were coated with ALD techniques and thermally treated to form intermetallic coatings at ANL. The coated samples were then irradiated by 55-MeV Fe ions at the Argonne Tandem Linac Accelerator System (ATLAS) at ANL. The samples were exposed to a 1pnA beam for 1 hour, then a 50pnA beam for 15 hours at ~100°C. After irradiation, the samples were characterized using XAFS technique at the MRCAT beamline at APS. Since the ion-irradiation damage zone is only a few µm from the surface, the samples were tilted to three different angles (90°C, 45°C, and 25°C) to the X-ray beam to study radiation damage at different depths. The experimental procedure is summarized in Figure 1. A typical K-edge absorption spectra for an irradiated zircaloy specimen (25°C to the X-ray) is shown in Figure 2. Further data analysis and interpretation of the X-ray Absorption Fine Structure data are needed. This research was conducted primarily by Kun Mo and Jeff Terry, and facilitated in large part by staff in the Nuclear Engineering Division at ANL and Illinois Institute of Technology, including Yinbkin Miao (ANL), Sumit Bhattacharya (ANL), Laura M. Jamison (ANL), Bei Ye (ANL), Walid Mohamed (ANL), Di Yun (ANL), Abdellatif M. Yacout (ANL), Daniel Velázquez (IIT), and Rachel Seibert (IIT).

Future Activities

The experiment is completed. Data processing and results analysis will be finished in FY 2016.

Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Illinois Institute of Technology	Materials Research Collaborative Access Team (MRCAT) facility at Argonne National Laboratory's Advanced Photon Source
Collaborators	
Argonne National Laboratory	Kun Mo (principal investigator), Di Yun (collaborator), Walid Mohamed (collaborator)
Illinois Institute of Technology	Jeff Terry (collaborator)

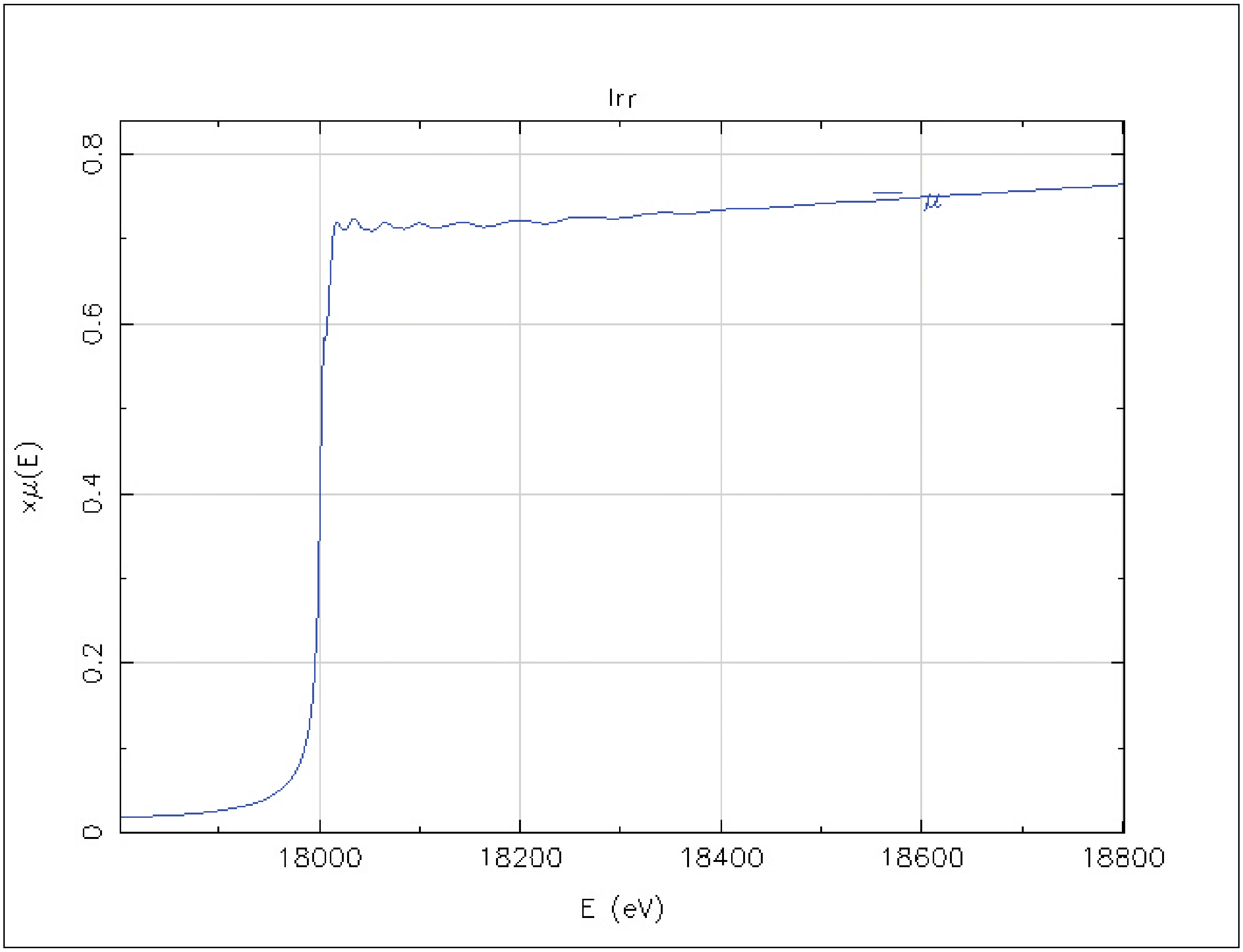

Figure 2. K-edge absorption spectra for irradiated coated zircaloy specimen.

Investigation of the Chemical State of Ag and Pd in SiC Shell of Irradiated TRISO Particles via XAFS

Kurt Terrani – Oak Ridge National Laboratory – terranika@ornl.gov

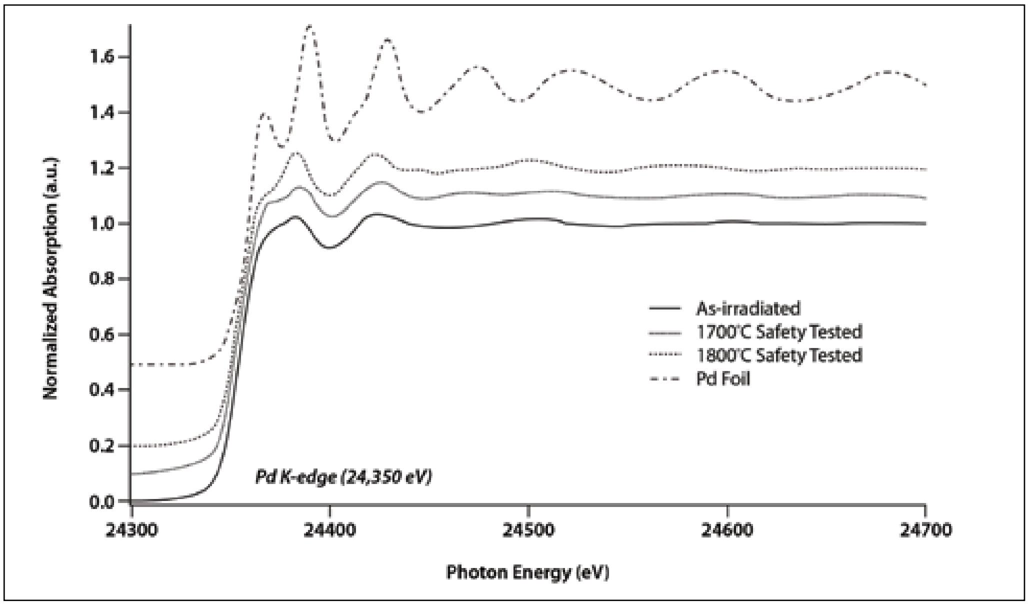

Figure. 1. Palladium XAFS in SiC shells from the various particles compared with metallic Pd foil that clearly shows a different character.

This experiment for the first time provides insights into specific mechanisms of radionuclide transport in irradiated TRISO particles, providing the basis for fundamental understating that in turn allows informed enhancements to the fuel design.

Tristructural isotropic (TRISO) fuel particles consist of a fuel kernel contained within a multiple ceramic spherical coating shells. The purpose of these ceramic coating layers, namely graphite and silicon carbide (SiC), is to limit the release of radionuclide fission products from the fuel kernel. However, a number of these fission products, particularly precious metals such as Ag, do transport within these layers under reactor normal operating and off-normal conditions. This study examines the mechanism of transport of these species in the SiC coating layer of TRISO particles.

Project Description

TRISO fuel form has enjoyed decades of development and testing and is currently considered a mature technology. This fuel form, originally developed for high temperature gas-cooled reactor (HTGR) applications, is now being considered for a number of other reactor platforms. To further enhance the safety of these reactors, the mechanism of transport and release of a few select problematic radionuclides, namely Ag and Pd, from these particles needs to be understood. A multitude of computational studies exist that have examined the various possibilities for the transport of these species

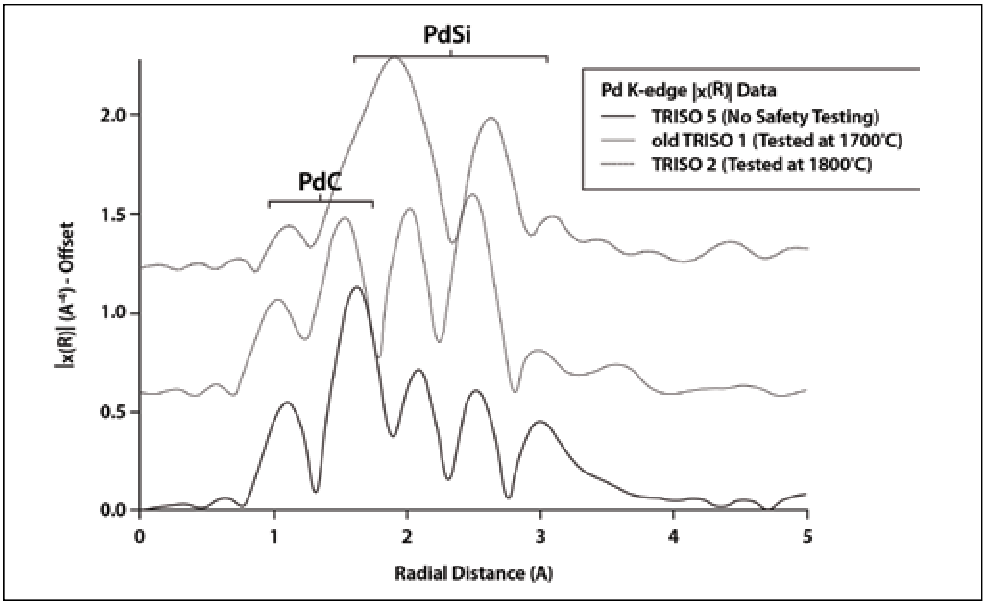

Figure 2. Radial Distribution Function (RDF) around Pd in the various SiC shells showing different chemical forms of Pd.

inside the SiC coating layer of TRISO particles. These computational studies are complemented by only a handful of experimental studies, most of which have poorly simulated the prototypical fuel operating environment. Therefore, the computational studies merely serve to contemplate various scenarios without any constraints and experimental data to narrow down any particular mechanism. In this study, care was taken to extract the SiC coating layer from actual TRISO fuel particles irradiated inside the reactor to high burnups. These SiC fragments were then examined via X-ray absorption fine structure (XAFS) technique, using a synchrotron, to specifically and accurately probe particular chemical species of interest. The results of these studies can pinpoint the chemical state of the species of interest after various fuel operating and testing scenarios. In this manner, given the experimental data, specific mechanisms of transport and release may be reinforced or dismissed. These studies will provide the mechanistic understating of radionuclide transport and release in the coated particle fuel forms. Accordingly, this will allow fuel scientists to enhance safety in the reactors employing this fuel form.

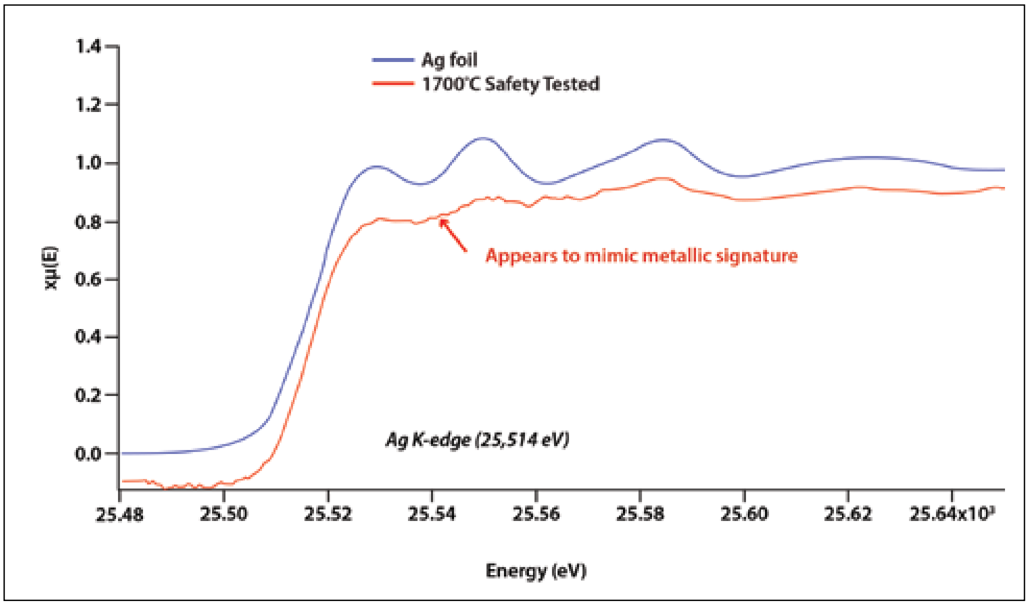

Figure. 3. Silver XAFS in 1700°C safety-tested SiC shell compared to a metallic Ag foil. The data show a metallic character for the Ag in the SiC shell.

Accomplishments

SiC fragments from three specific TRISO fuel particles were selected for synchrotron XAFS examination. One of the particles was in the as-irradiated state, while the other two had been safety tested (after irradiation, held at elevated temperature isothermal conditions to simulated off-normal conditions) at 1700 and 1800°C. The particles had undergone gamma assay to determine the inventory of Ag still retained in the particles, the amount of which varied widely between these three particles. The particles were crushed, burned to remove all the graphite layers, and leached with acid to remove all the fuel. This process was repeated twice to then end up with pristine SiC coating shells. These shells were then extracted from Oak Ridge National Laboratory's (ORNL's) fuel hot-cell and shipped to Materials Research Collaboratory Access Team (MRCAT) for synchrotron characterization. The basic fluorescence spectra showed the presence of Pd, U, and Pu in these shells for all the particles. However, Ag, in reasonable detectable amounts, was only present in the SiC shell from the 1700°C safety-tested particle. XAFS studies were carried out on all of these shells and the dominant chemical state of Pd and U for all the shells from the various particles was determined. Long collection times were dedicated to collection of XAFS data for Ag in the 1700°C safety-tested SiC shell. The data closely resemble that of metallic Ag, though a metallic alloy cannot be ruled out at this time. Longer collection times are needed to probe the XAFS regime to determine the bond length of the Ag near neighbors to answer this question. However, this is a major finding that implies Ag does not transport inside the SiC as a single atomic solute. Instead, it is in metallic clusters. The graduate student collaborator in this project, Rachel Seibert (IIT), meticulously and patiently collected and analyzed the XAFS data.

Future Activities

Future activities involve electron microscope characterization of these SiC shells. The shells have already been returned to ORNL and the microscopy characterization is currently underway in FY 2016.

The ability, facilitated via NSUF, for preparation of samples at hot cell facilities and subsequent examination at synchrotron resources provides unprecedented experimental data.

— Kurt Terrani, Staff Scientist - ORNL

Distributed Partnership at a Glance

NSUF and Partners	Facilities and Capabilities
Illinois Institute of Technology	Materials Research Collaborative Access Team (MRCAT) facility at Argonne National Laboratory's Advanced Photon Source
Collaborators	
Illinois Institute of Technology	Jeff Terry (collaborator), Rachel Seibert (collaborator)
Oak Ridge National Laboratory	Kurt Terrani (principal investigator)

Irradiation Effects in Oxide Nanoparticle Stability and Matrix Microstructure in ODS Steel Neutron Irradiated to 3 dpa at 500°C

Kumar Sridharan – University of Wisconsin, Madison – kumar@engr.wisc.edu

ODS steels are deemed to be among the most promising candidate materials for cladding and core internals of next generation nuclear reactors.

One of the major steps toward the implementation of Generation IV nuclear reactor systems is the development of appropriate materials for cladding and other core internal components that will be able to withstand high doses and temperatures. Oxide dispersion strengthened (ODS) steels are deemed as being among the promising candidate materials for cladding and core internals for advanced nuclear reactors, by virtue of their superior mechanical properties at high temperatures. These properties arise from a fine dispersion of Y-Ti oxide nanoparticles in the steel matrix, which serve as pinning points for dislocation motion and point defect annihilation sites. Additionally, these oxide nanoclusters are expected to trap transmutation-produced helium in small, high-pressure bubbles. To apply these materials in the extreme environments of next generation nuclear reactors, a complete understanding of their radiation response is necessary.

Project Description

In the proposed research, a 9%Cr ODS steel in unirradiated and irradiated (with neutron to 3 dpa at 500°C) conditions, were analyzed with high-resolution microscopy techniques for microstructural and microchemical changes induced by neutron irradiation. The analysis was performed utilizing Transmission Electron Microscopy (TEM), Scanning-TEM coupled with Energy Dispersive Spectroscopy (EDS), and Local Electrode Atom Probe (LEAP, also known as atomic probe tomography, [APT]). Many studies in literature have investigated the stability of the Y-Ti oxide nanoclusters in ODS steels using proton and heavy ion irradiations, but there have been relatively few studies the effects of neutron radiation on these oxide nanoclusters. Specifically, this research focused on the changes caused by the neutron irradiation on the size, density, and composition of the nanoclusters as well as on the formation of extended defects (dislocation

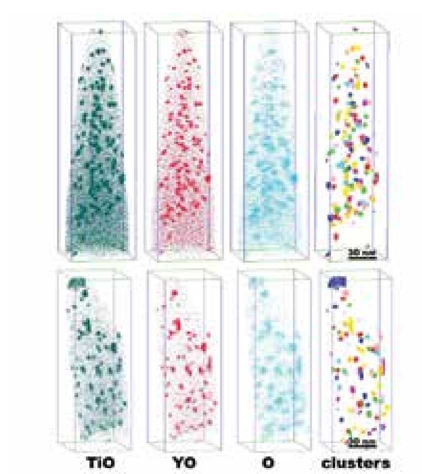

Figure 1. LEAP images showing elemental distribution maps at near-atomic level for the as-received (top row) and neutron irradiated (bottom row) ODS steels.

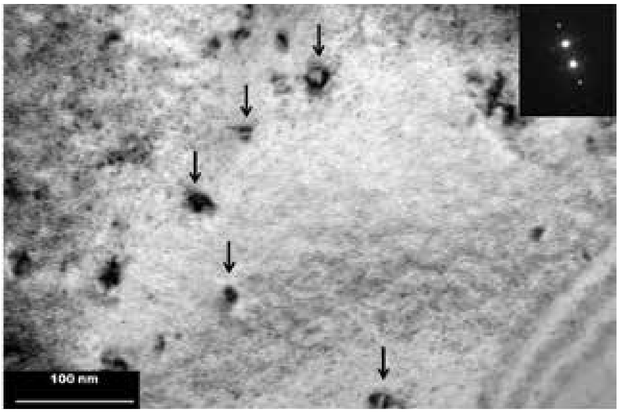

Figure 2. Two beam bright field image of a grain of the neutron irradiated ODS steels showing dislocation loops (identified are indicated by black arrows).

loops) and on the radiation-induced segregation of elements in the steel matrix. The synergistic deployment of multiple atomic resolution analytical techniques was intended to provide a comprehensive understanding of the response of this material to neutron irradiation. Understanding the influence of neutron irradiation-induced changes to the microstructure and microchemistry of ODS steels is key to the successful application of these steels as advanced fission reactor components, and is therefore highly relevant to the DOE-NE program.

Accomplishments

The objective of the proposed research was to develop a fundamental understanding of the structural response of a 9C%Cr ODS steel to neutron irradiation. Specific emphasis was placed on the size and size distribution of the nanoclusters before and after radiation, their compositional changes, as well as the formation of extended defects in the matrix, and radiation-induced elemental segregation.

Analysis of the size, size distribution and chemistry of the smallest precipitates in the matrix (size <5 nm), which were difficult to be imaged by conventional TEM and Scanning Transmission Electron Microscopy (STEM) techniques, was performed by LEAP. After irradiation, it was observed that the average size of the nanoclusters increased by a small fraction, from 2.58 ± 1.19 nm to 3.01 ± 1.71 nm. Correspondingly, the number density of the nanoparticles decreased slightly, from 1.8 X 10^{23} m^{-3} to 1.2 X 10^{23} m^{-3}. Figure 1 shows the LEAP 3-D reconstruction of the samples in the

Understanding the stability of the microstructure of oxide dispersion strengthened (ODS) steels under neutron irradiation is critically important to the application of these steels to next generation advanced nuclear reactors.

— Kumar Sridharan
Distinguished Research Professor, Engineering Physics Department, University of Wisconsin-Madison

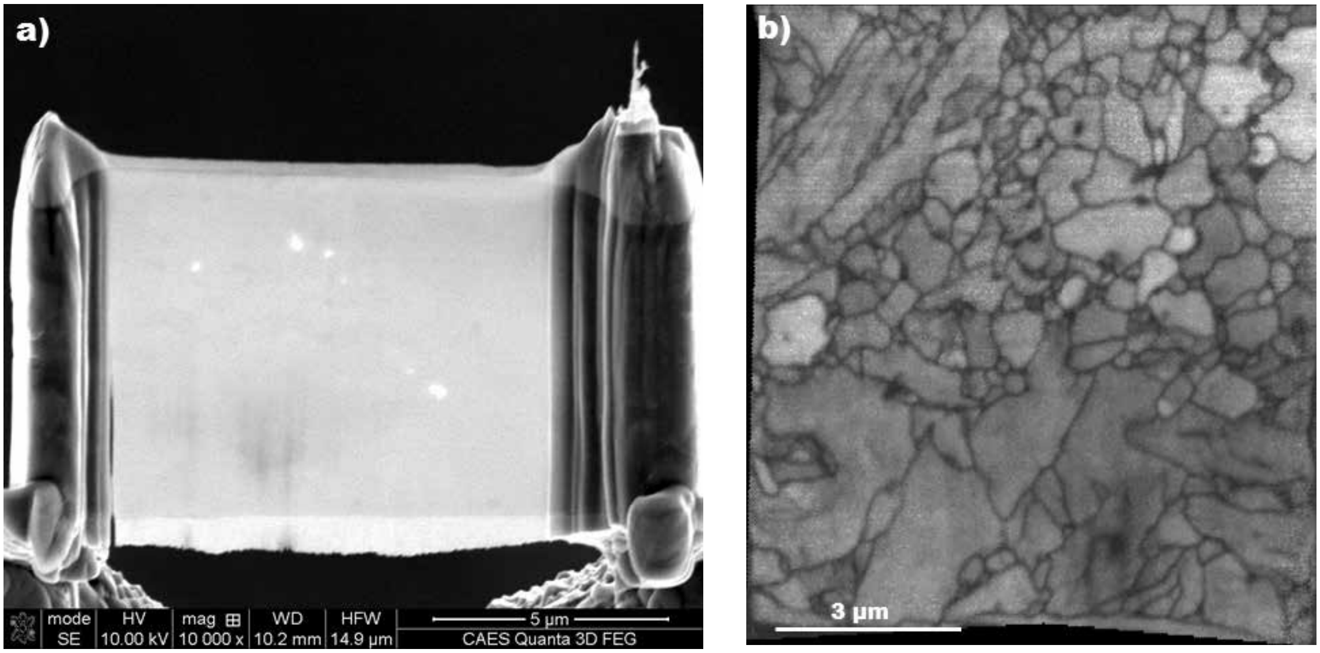

Figure 3. (a) SEM image of a TEM sample of the neutron irradiated specimen, and (b) image quality map of the same sample as obtained by t-EBSD analysis. This analysis was used to determine grain boundary character distribution of the samples.

as-received and irradiated conditions. The chemistry of very small nanoclusters was also investigated. It was found that both the Ti/Y and (Ti+Y)/O ratios decrease after radiation, going from 1.22 to 1.09 and from 1.37 to 1.29, respectively.

Nanoclusters of sizes greater than 5 nm, were identified by means of STEM Z-contrast imaging, by virtue of their differences in chemical composition with respect to the matrix (the presence of Y and Ti in these precipitates was confirmed by EDS). It was noted that, in accordance with the results obtained from LEAP analysis for the smallest precipitates, the average size decreased slightly and the number density correspondingly increased going from 7.62±2.0 nm and 2.17 x 10^{22} m^{-3} to 8.77±2.1 nm and 2.01 x 10^{23} m^{-3}, respectively, in the as-received and irradiated conditions. The very small changes in the sizes of the nanoclusters after radiation generally indicates that the nanoclusters are very stable under irradiation.

Defect analysis was also performed on the sample after irradiation. Two beam condition images close to [001], [011], and [111] zone axis were acquired and the dislocation loops identified were quantified. A typical micrograph showing dislocation loops in the neutron irradiated sample is shown in Figure 2. The analysis performed yielded an estimate defect size of 9.1 nm ± 2 nm and a density of 3.3 x 10^{21} m^{-3}.

Finally, elemental segregation across grain boundaries was performed on a few grain boundaries of the neutron irradiated sample. To reveal the nature of the grain boundary under investigation, t-EBSD maps were collected from the samples under investigation.

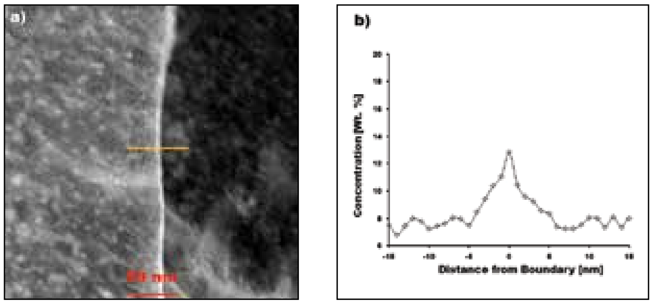

Figure 4. Segregation profile of Cr across a high angle grain boundary (θ=49°C) of the neutron irradiated specimen. (a) STEM image,(b) segregation profile obtained obtained form EDS analysis.

Figure 3 shows an SEM image of the TEM sample from the irradiated specimen along with the image quality maps obtained from t-EBSD analysis. Figure 4 shows a STEM-EDS line scan performed along a high-angle grain boundary of the irradiated specimen. This analysis shows that after radiation, high-angle grain boundaries are prone to radiation-induced segregation phenomena, with enrichment of Cr and, consequently, depletion of Fe, observed at the grain boundaries.

Future Activities

The research is completed.

Publications and Presentations

1. Mairov, A., J. He, K. Sridharan, T. Allen, 2015, "Irradiation Effects in Oxide Nanoparticle Stability in Oxide Dispersion Strengthened (ODS) Steel," *Oral presentation at the Metallurgical Society (TMS) Annual Meeting, March 15–19, 2015, Orlando, Florida*.

2. Mairov, A., J. He, K. Sridharan, 2015, "Structural Effect in Oxide Dispersion Strengthen (ODS) Steel Neutron Irradiated to 3 dpa at 500°C," *Microscopy and Microanalysis*, Vol. 21, Supplement S3, 2015 (Proceedings of Microscopy & Microanalysis 2015).

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
University of Wisconsin, Madison	Kumar Sridharan (principal investigator), Alexander Mairov (collaborator)

Investigation of Thermal Stability of nm-scale Ni-Mn-Si Precipitates in Highly Irradiated Reactor Pressure Vessel Steels

G. Robert Odette – University of California, Santa Barbara – odette@engineering.ucsb.edu

The results from this experiment have greatly improved our understanding of Mn-Ni-Si precipitates and enabled more accurate predictive models for RPV embrittlement at 80 years of operation or more, which is absolutely critical to light water reactor life extension.

One potential barrier to extending nuclear light water reactors lifetimes to 80 years is embrittlement of their massive reactor pressure vessels (RPV). Embrittlement is primarily due to the formation of nm-scale precipitates, which cause hardening and a corresponding increase in the ductile to brittle transition temperature. Recent work has shown that a new embrittlement mechanism, formation of a large volume fraction of Mn-Ni-Si precipitates, may emerge at high fluences. Notably, these precipitates are not accounted for in current predictive models. These features must be better understood to ensure the safe operation of the RPV at extended life fluences.

Project Description

While the existence of Mn-Ni-Si precipitates (MNSPs) at high fluence is no longer debated, there is still a large disagreement in literature regarding their formation mechanism. For example, some groups have argued that the MNSPs are solute clusters formed by a radiation-induced segregation (RIS) mechanism, while other groups have used thermodynamic models to suggest that MNSPs are equilibrium phases whose formation is accelerated by the high-vacancy concentration created under irradiation. Resolving this question is critical to developing accurate predictive models of low-flux, high-fluence embrittlement, especially considering that most data used to calibrate such models will come from high-flux test reactor irradiations where RIS may be enhanced.

Thus, the purpose of this work is to elucidate the MNSP formation mechanism through the use of long-term post irradiation annealing treatments at 425°C on a Cu-free, high Ni (1.6%) steel. This steel was previously irradiated in the Advanced Test Reactor to very high neutron fluence and a large volume fractions (f) of precipitates was found in the as-irradiated condition. Thus, if the precipitates remain stable and begin coarsening under annealing, they are equilibrium phases and not segregated solute clusters, which would be expected to dissolve under annealing. Atom Probe Tomography (APT) was used to measure the precipitate (f) at various annealing times.

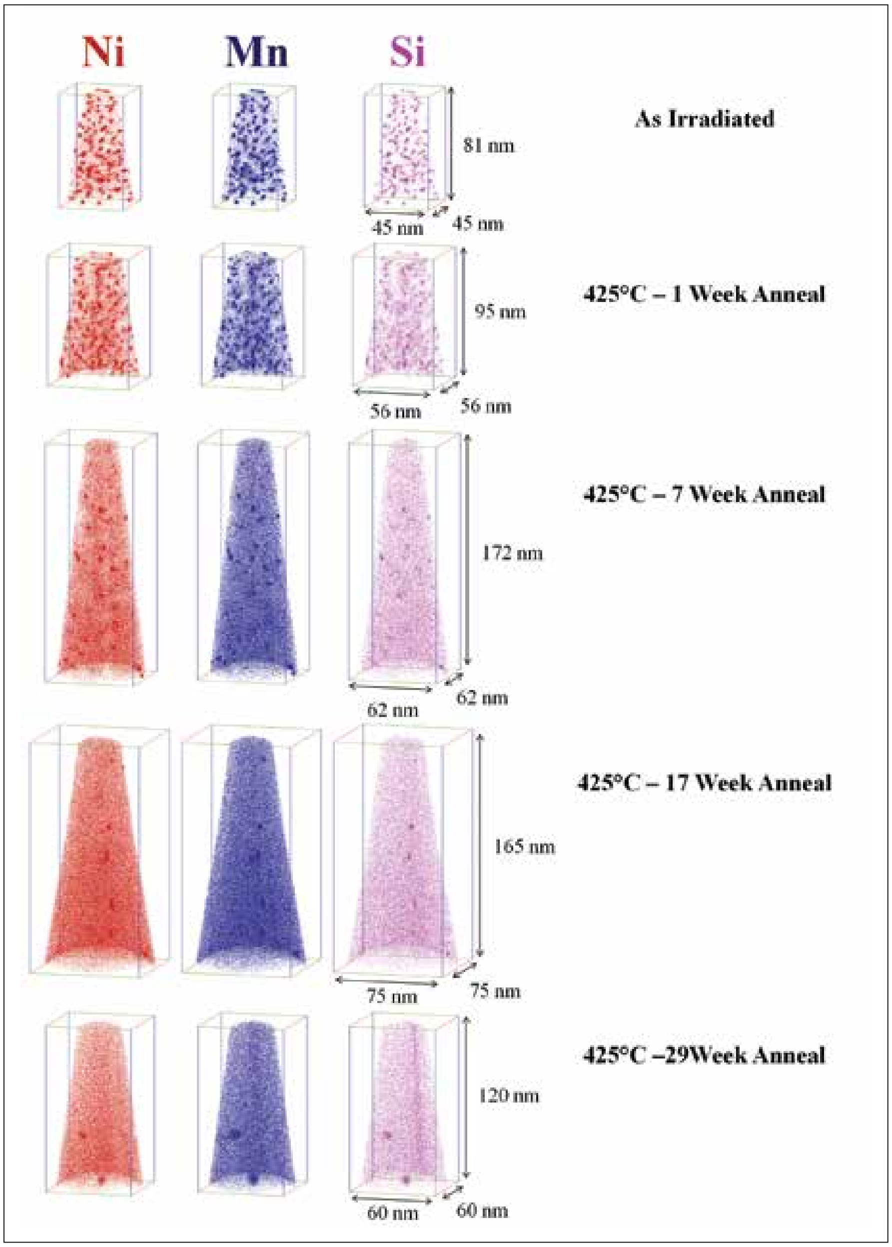

Figure 1. Atom maps from the Cu-free, 1.6% Ni steel showing a reduction in number density with increasing annealing time, but apparent coarsening at the longest annealing time.

The post-irradiation examination facilities at the Center for Advanced Energy Studies offer us an outstanding opportunity to examine changes in material microstructures and properties after irradiation that are vital for evaluating light water reactor life extension.

— Nathan Almirall, Graduate Student Researcher

Accomplishments

APT atom maps from the high (1.6%) Ni steel after various annealing times are shown in Figure 1. While the high Ni steel showing a reduction in MNSP number density with each additional time increment, some precipitates remained in the 29 week annealed condition, the longest time investigated. In addition, it appears that the remaining precipitates have begun coarsening, as the measured precipitates in the 29-week annealed condition have over twice the number of Mn, Ni, and Si atoms as the largest precipitates in the as-irradiated condition. Cluster dynamics modelling is ongoing to better understand the dissolution process, but these results strongly suggest that the MNSPs are in fact equilibrium phases.

Future Activities

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

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

Effects of Minor Element Additions in AA6061 on the Microstructural Evolution of the Interaction Region between U-Mo Alloys and AA6061 Claddings

Emmanuel Perez – Idaho National Laboratory – Emmanuel.Perez@inl.gov

The U.S. Material Management and Minimization Reactor Conversion program is developing low-enriched molybdenum-stabilized uranium alloy fuels systems for use in research and test reactors. Monolithic and dispersion fuel plates have local regions where the U-Mo fuel can come into contact with the Al Alloy 6061 (AA6061) cladding. U-Mo alloys in contact with Al undergo diffusional interactions that result in the development of interdiffusion zones with complex fine-grained microstructures with multiple phases. In this study, the microstructural development of a diffusion couple consisting of U-10wt.%Mo vs. AA6061, annealed at 600°C for 24 hours was analyzed in detail by transmission electron microscopy with X-ray energy dispersive spectroscopy. The diffusion couple developed complex interaction regions where phase development was significantly influenced by the alloying additions of the AA6061. This study seeks to determine the effect of the minor element additions in the AA6061 and to identify any phases that may have developed as a result of these additions.

Project Description

U-Mo fuel plates encased in aluminum undergo chemical interactions that result in the development of a complex interaction region between the U-Mo fuel and the aluminum cladding. This study aimed to characterize the interaction region that develops between U-Mo alloys in contact with AA6061. Emphasis was given to the effects of the minor element additions in the AA6061 onto the interaction region between the U-Mo and the AA6061. Although more recent designs of U-Mo fuel plate systems place a Zr-diffusion barrier between the U-Mo and the AA6061, due to failure of the barrier or shearing of the ends of the fuel foils, the fuel plates can have regions where the U-Mo comes into contact

The often ignored minor element additions in aluminum alloys play a significant factor in the evolution of the interaction regions that develop between uranium alloys in contact with aluminum alloys.

with the aluminum. Thus, a clear understanding of the U-Mo/AA6061 interaction is required.

Prior characterization of diffusion couples of U-Mo in contact with AA6061 showed that the minor element additions in the AA6061 penetrate into the interaction region and affect its microstructural evolution. Comparison to the interaction regions that develop between U-Mo in contact with high-purity aluminum, the interaction regions between U-Mo and AA6061 develop more complex microstructures with phases that appear to contain mainly the elements from the AA6061 additions. These phases, if present, can significantly impact the fuel system performance during service. Thus, the phases must be conclusively identified so that any detrimental effects can be minimized or eliminated.

This study supports the qualification efforts of the fuel system under consideration. The data that this study generates will result in improvements in reliability and service life of the fuel systems. The acquired knowledge will also be generally available for any future system where U-alloys interact with aluminum alloys.

This work will contribute significant insight into the interactions of U-Mo with Al-alloys

— Emmanuel Perez, Materials Engineer, Idaho National Laboratory

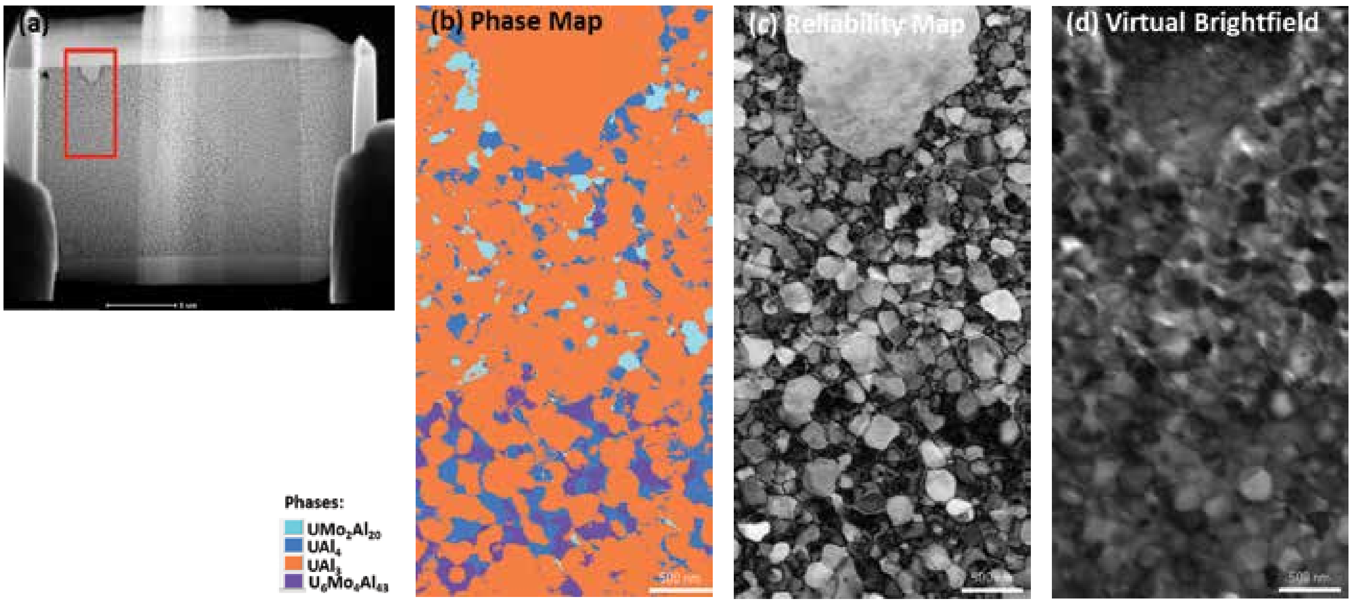

Figure 1. TEM characterization via the ASTAR system in a selected region of a TEM sample from the interaction region of a U-10Mo versus AA6061 diffusion couple annealed at 600°C for 24 hours. The micrographs show (a) the TEM sample, (b) a phase map identifying the UAl^3, UAl^4, $U^6Mo^4Al^{43}$, and UMo^2Al^{20} phases, (c) and (d) a reliability map and a virtual Brightfield micrographs detailing the microstructure of the selected region.

Accomplishments

Samples for characterization by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were prepared via focused ion beam (FIB) at CAES. A total of nine TEM samples were successfully prepared and analyzed in detail. The samples captured sections of the interaction regions in a U-10Mo vs. AA6061 diffusion couple annealed at 600°C for 24 hours that represented the different microstructural features observed in the interaction region between the U-10Mo and the AA6061. SEM analysis was carried out in the FIB using its SEM capabilities. TEM analysis was carried by high-angle annular dark-field (HAADF) imaging, energy dispersive spectroscopy (EDS), selected area electron diffraction (SAED) on individual grains, and by precession diffraction analysis using the ASTAR system installed in the TEM. At of the end of FY 2015, analysis of the data was still in progress.

This work was facilitated by the CAES staff: Jatuporn Burns, Yaqiao Wu, Joanna Taylor, and Kristi Moser-McIntire.

Future Activities

The project aims to complete analysis of the collected data in FY 2016. This work is scheduled to be presented at the 2016 Minerals, Metals and Materials (TMS2016) annual conference. A publication of this study is planned, likely in the *Journal of Nuclear Materials*.

Publications and Presentations

This work is scheduled to be presented at the 2016 *Minerals, Metals and Materials* (*TMS*2016) annual conference.

A publication of this study is planned, likely in the *Journal of Nuclear Materials*.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Idaho National Laboratory	Emmanuel Perez (principal investigator), Dennis D. Keiser (co-principal investigator)

Synergistic Effects of Thermal Aging and Neutron Irradiation in 308L Welds

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

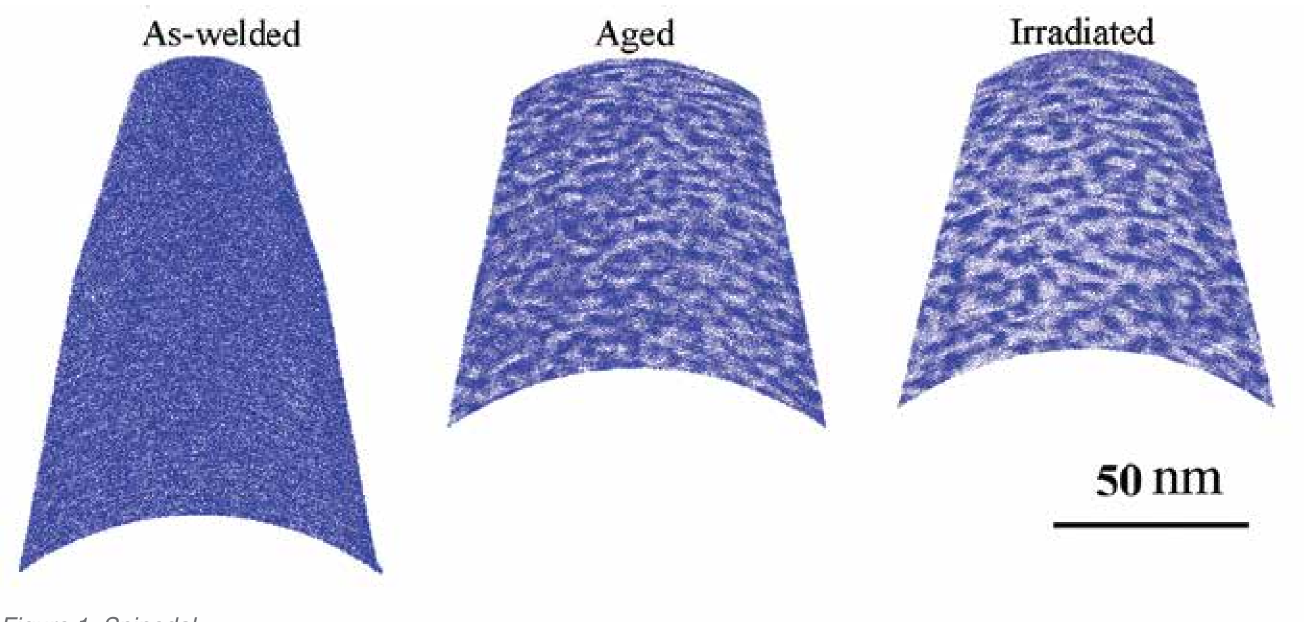

Figure 1. Spinodal decomposition structure (Cr map) of ferrite in the as-welded, aged and irradiated 308L weld.

Project Description

The research project is aimed to provide some of the first data and a direct comparison of the micro-structural evolution and elemental redistribution behaviors among the as-welds, thermally aged and neutron irradiated austenitic stainless steel welds. The research objective is well aligned with the Department of Energy Office of Nuclear Energy (DOE-NE) Sustainability of the Light Water Reactor (LWR) program, and to evaluate the materials' integrity is a key need for considering the life extension for the existing LWRs.

Accomplishments

The thermal aging was performed at 400°C for up to 2,220 hours on a 308L weld, and the irradiation was conducted in the Halden reactor at ~315°C to 0.08 dpa (5.6 x 1019 n/cm^2, E>1 MeV). The microstructural evolution of the ferrites was characterized using atom probe tomography (APT) and auxiliary transmission electron microscope studies. Spinodal decomposition and Ni-Mn-Si solute clusters were observed in both the

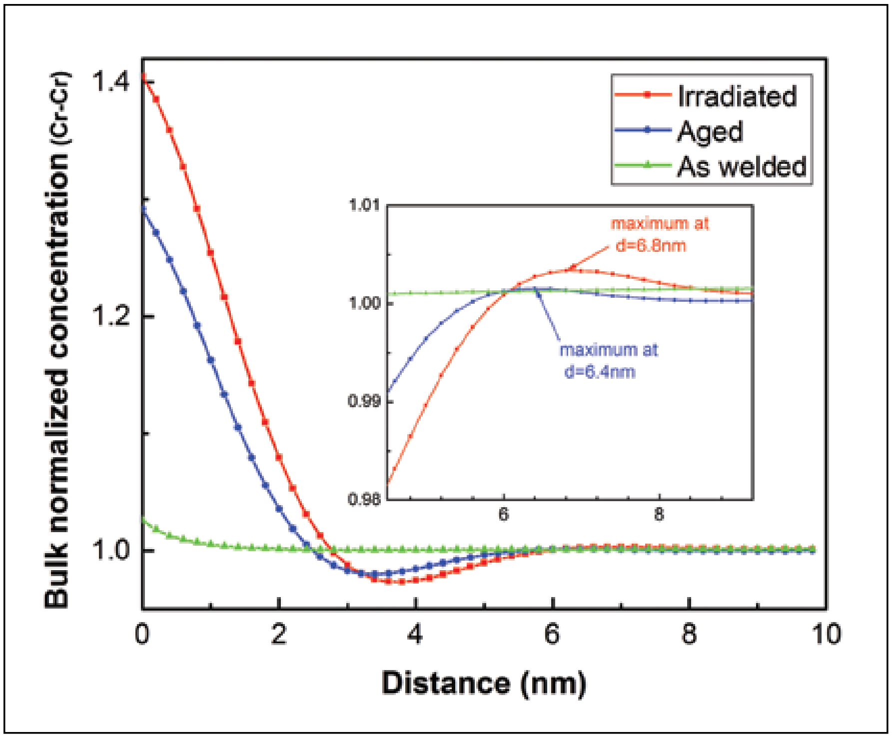

Figure 2. Radial distribution functions of Cr-Cr ions in aged and irradiated ferrites in 308L weld.

To fundamentally understand the thermal and radiation stability of duplex stainless steel welds serving in LWRs would significantly contribute to the LWRs' sustainability by ensuring the reactor component integrity in its extended lifetime.

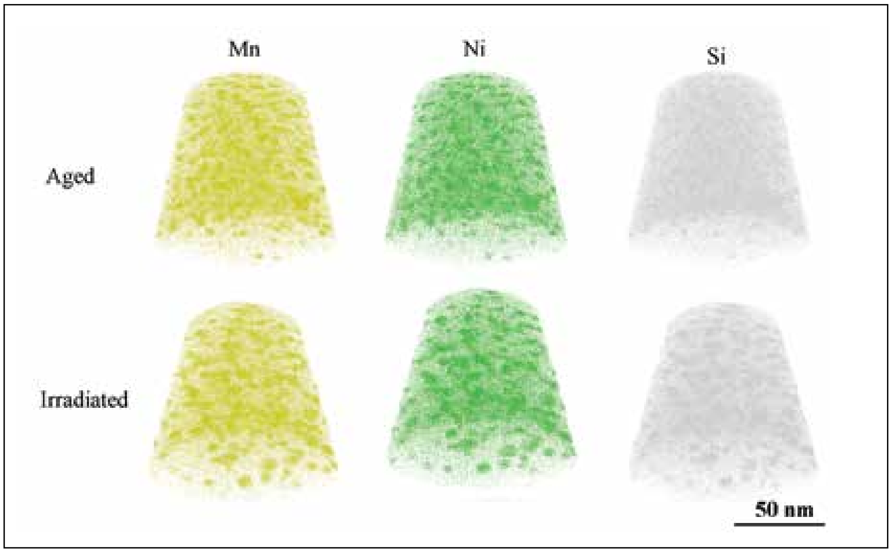

Figure 3. Mn-Ni-Si clusters in aged and irradiated ferrites in 308L weld.

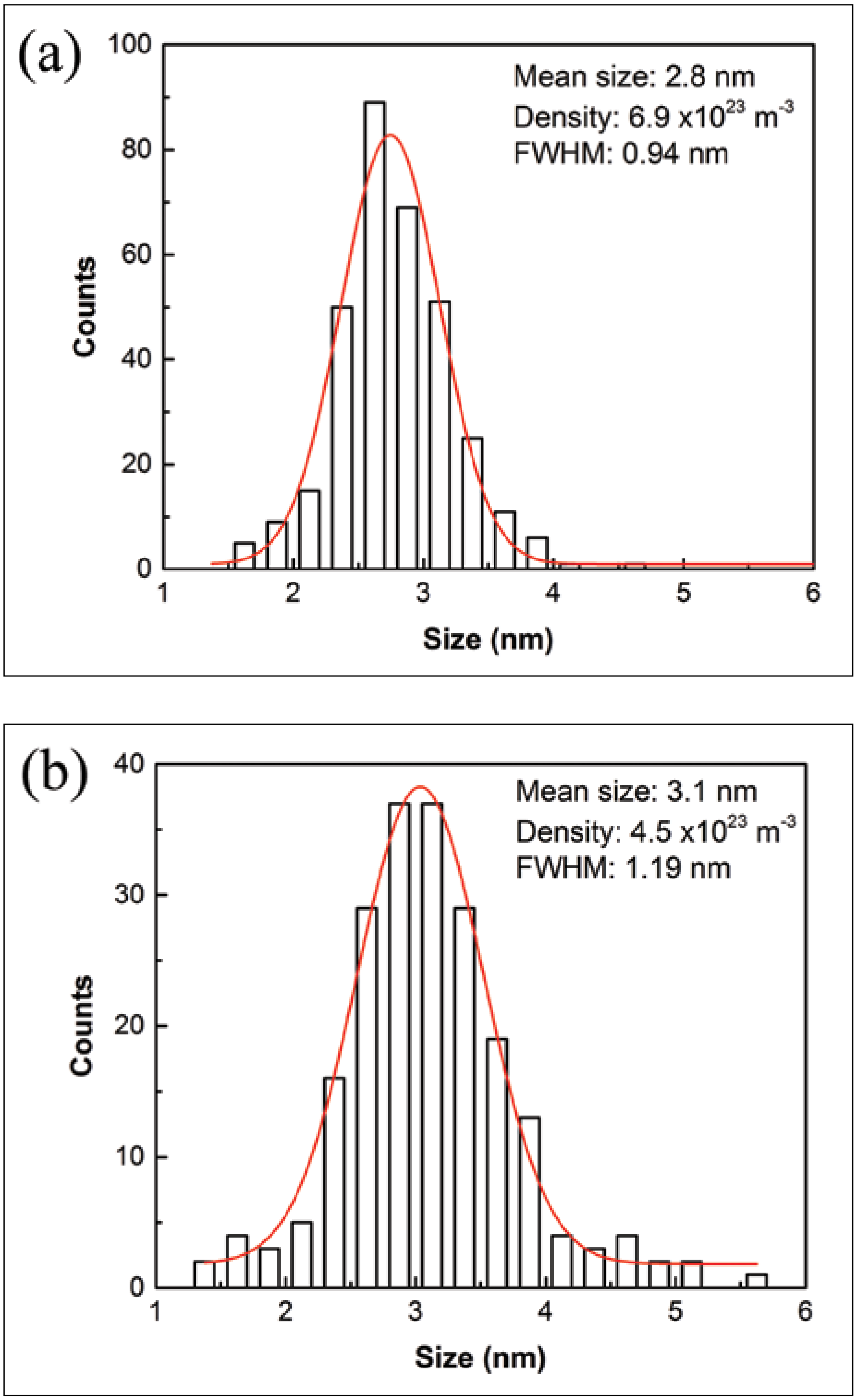

Figure 4. Size distribution, mean size and number density of G-phase precipitates: (a) aged and (b) irradiated.

thermally aged and neutron irradiated 308L welds. As compared with thermal aging, low-dose neutron irradiation induced slightly more significant spinodal decomposition with larger Cr concentration fluctuation wavelength and amplitude. The solute clusters in irradiated ferrite phase also show a larger mean size, a wider size distribution, but a lower number density as compared with those in thermally aged ferrite phase. In addition, the neutron irradiation significantly promotes segregation of other trace elements, particularly phosphorus, at the Ni-Mn-Si solute clusters.

Future Activities

The project was completed.

Publications and Presentations

1. Li, Z., Y. Chen, A. S. Rao, and Y. Yang, "Effects of thermal aging and low dose neutron irradiation on the ferrites in a 308L weld," submitted to *Journal of Nuclear Materials*, under revision.

Nuclear Materials research is highly empowered by the atom probe tomography and other advanced characterization tools.

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

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

Atom Probe Tomography of Fe-U-Pu-Zr Systems

Assel Aitkaliyeva – Idaho National Laboratory – assel.aitkaliyeva@inl.gov

The results are amongst three known attempts to prepare transuranic-bearing nuclear fuels in the FIB and examine these fuels in TEM. This is a first-documented attempt to characterize this type of fuel in a LEAP instrument.

Project Description

The objectives of this work were to perform advanced characterization of fuel-cladding chemical interaction products and determine chemical composition of complex intermetallic phases formed between U-Pu-Zr fuels and Fe cladding upon exposure to high temperatures. This study investigated the interdiffusion between metal fuel and cladding via diffusion couple technique, formation of various intermetallic phases upon exposure to high temperatures, and fuel-cladding microstructural development at the atomic scale using the atom probe tomography (APT) technique in a local electrode atom probe (LEAP) instrument. The results allowed comprehensive 3-D analysis of the formed phases, which was not previously attainable due to the inherent difficulties associated with sample preparation. Implementation of focused ion beam technology allowed preparation of site-specific lift-outs from the formed brittle fuel-cladding interaction layers with minimal hands-on sample processing, which allowed a thorough cross-sectional examination of all phases formed during annealing.

Accomplishments

Specimens were fabricated using focused ion beam at Electron Microscopy Laboratory at INL and then analyzed in transmission electron microscope (TEM) and LEAP instruments. The goal of this project, to visualize the complex phases formed between U-Pu-Zr fuels and Fe cladding, has been achieved and successfully incorporated into a publication in the *Metallurgical and Materials Transactions E*. A second manuscript is being prepared at this time.

Future Activities

The project is now complete.

Publications and Presentations

1. Aitkaliyeva, A., J. Madden, B. Miller, C. Papesch, J. Cole, 2015, "Fuel-cladding interaction between U-Pu_Ze fuel and Fe," *Metall. Mater. Trans. E.*, Vol. 2, No. 4, 2015, pp. 220–228.

Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Electron Microscopy Laboratory
Collaborators	
Idaho National Laboratory	Assel Aitkaliyeva (principal investigator), Cynthia Papesch (collaborator)

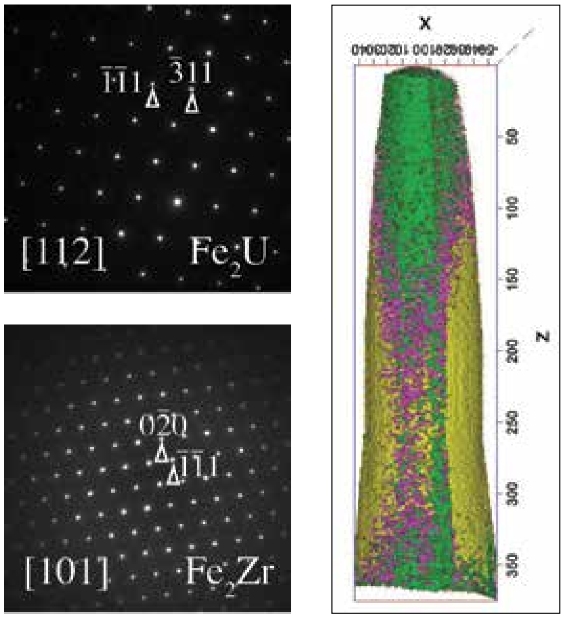

Figure 1. Atom probe tomography data of Fe^2U and Fe^2Zr phases formed within fuel-cladding chemical interaction layer and selected area (electron) diffraction patterns of the phases.

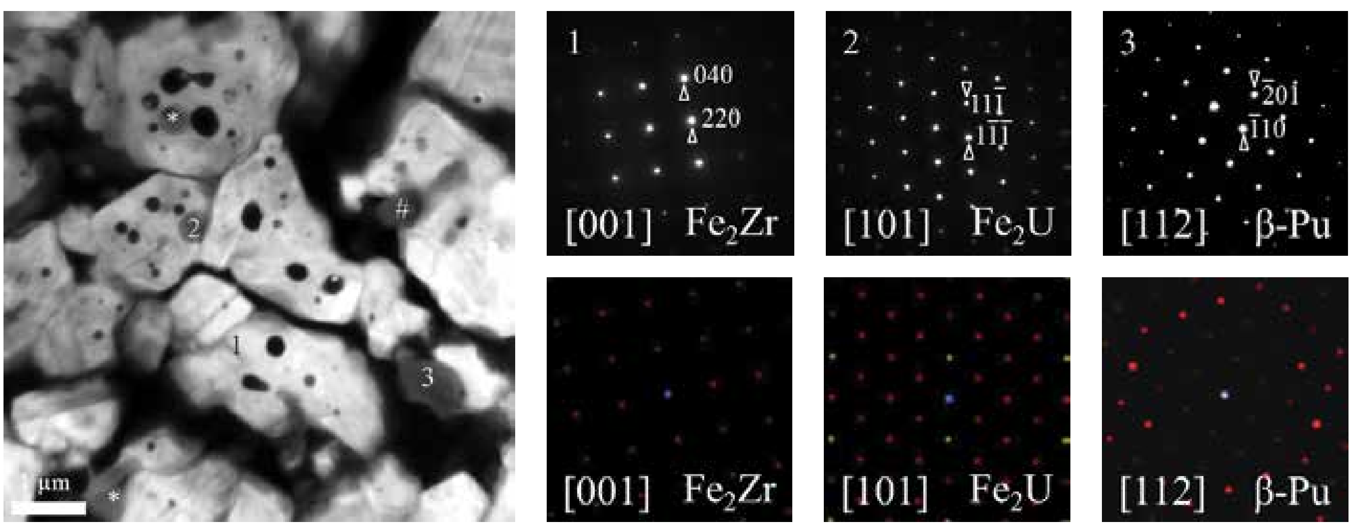

Figure 2. Transmission electron micrograp and corresponding experimental (top row) and simulated selected area (electron) diffraction patterns (bottom row). Scale bar denotes 1 um. The asterisk denotes the location of other identified Fe^2U phases and the # denotes the location of b-Pu in the micrograph.

Evaluation of Charged Particle and Neutron Irradiations on Fe-9%Cr ODS Steel

Janelle P. Wharry – Boise State University – janellewharry@boisestate.edu

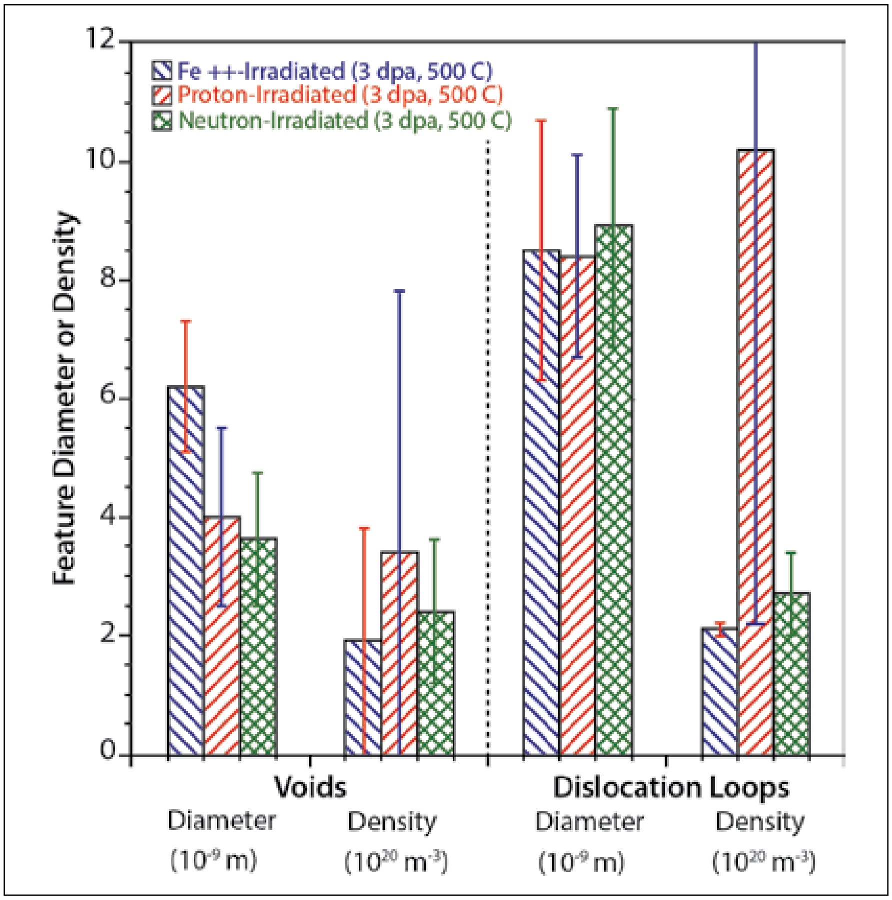

Figure 1. Comparison of void and dislocation loops measurements after self-ion-, proton- or neutron-irradiated to 3 dpa, 500°C in Fe-9%Cr ODS.

The U.S. Material Management and Minimization Reactor Conversion program is developing low-enriched molybdenum-stabilized uranium alloy fuels systems for use in research and test reactors. Monolithic and dispersion fuel plates have local regions where the U-Mo fuel can come into contact with the Al Alloy 6061 (AA6061) cladding. U-Mo alloys in contact with Al undergo diffusional interactions that result in the development of interdiffusion zones with complex fine-grained microstructures with multiple phases. In this study, the microstructural development of a diffusion couple consisting of U-10wt.%Mo vs. AA6061, annealed at 600°C for 24 hours was analyzed in detail by transmission electron microscopy with x-ray energy dispersive spectroscopy. The diffusion couple developed complex interaction regions where phase development was significantly influenced by

Oxide nanocluster evolution in Fe-9%Cr ODS differs between heavy ion, proton, and neutron irradiation.

the alloying additions of the AA6061. This study seeks to determine the effect of the minor element additions in the AA6061 and to identify any phases that may have developed as a result of these additions.

Project Description

Specimens of a model Fe-9%Cr oxide dispersion strengthened (ODS) alloy have been irradiated with neutrons in the Advanced Test Reactor (dose rate $\sim10^{-7}$ dpa/sec), with 2.0 MeV protons ($\sim10^{-5}$ dpa/sec) and with 5.0 MeV self-ions ($\sim10^{-4}$ dpa/sec). All irradiations were carried out to 3 dpa at a temperature of 500°C. The microstructure of each specimen was characterized using transmission electron microscopy and atom probe tomography (with cluster analysis) and compared to the microstructure of the original as-received sample. Nanoindentation was used to measure any relative change in hardness as a result of each irradiation.

Because this project aims to (1) understand the response of advanced reactor candidate structural materials to irradiation, and (2) assess the ability of proton and heavy ion irradiations to emulate in-reactor neutron irradiation damage, this project has direct relevance to the Department of Energy Office of Nuclear Energy's (DOE-NE's) Advanced Reactor Technologies program. The primary DOE-NE mission is to advance nuclear power as a resource capable of meeting the nation's energy, environmental, and national security needs. Generation IV advanced reactor designs, such as high-temperature reactors and fast neutron spectrum reactors, fulfill this mission by coupling high-efficiency power generation with the environmental and national security benefits of consuming long-lived radioactive isotopes found in used nuclear fuel. However, with the promise of Generation IV designs comes the challenge of finding suitable structural materials that will withstand the harsh in-reactor operating conditions. Ensuring the integrity of these materials under high temperatures, corrosive environments, cyclic loading, and high-irradiation damage, is paramount to the safety, performance, and long-term success of the Generation IV nuclear fleet.

Accomplishments

In this project, we compared the microstructure evolution of the identical heat of a model Fe-9%Cr ODS under self-ion, proton, and neutron irradiation, all carried out at 500°C to 3 dpa. NSUF enabled the team to access neutron-irradiated specimens from the Advanced Test Reactor sample library, conducted through the University of Wisconsin ATR Pilot Program. The team then conducted charged particle irradiations using 2.0-MeV protons or 5.0-MeV Fe++ ions at the Michigan Ion Beam Laboratory, through an NSUF rapid turnaround irradiation experiment. A follow-on rapid turnaround experiment enabled the team to complete a comprehensive microstructure characterization of all irradiated specimens using a combination of transmission electron microscopy (TEM) and local electrode atom probe (LEAP). All post-irradiation materials characterization work was conducted in the Microscopy and Characterization Suite (MaCS) at the Center for Advanced Energy Studies (CAES), utilizing the FEI Quanta focused ion beam (FIB), CAMECA 4000X HR LEAP, and FEI Tecnai S-Twin TEM.

TEM results show that grain size, dislocation line density, and carbide precipitates remain unchanged upon self-ion, proton, and neutron irradiation. Irradiation-induced dislocation loops and voids were also observed by TEM in all three irradiated samples. Notably, dislocation loop size and number densities were consistent across all irradiations (Figure 1). Only a few voids were observed, and they had diameters ~5 nm and were sparsely populated. These results suggest that at TEM resolutions, the neutron-irradiated microstructure in Fe-9%Cr ODS can generally be replicated using either self-ion or proton irradiation carried out at identical doses and temperature.

To complement our TEM analysis, LEAP enabled atomic-level resolution characterization of the oxide nanoclusters prior to and after each irradiation. Self-ion and proton irradiation led to decreases in the average nanocluster size by ~0.9-1.2 nm. However, neutron irradiation induced a more significant decrease in nanocluster size by more than 2.5 nm. The evolution of the particle size distributions (Figure 2) and number density of the nanoclusters after all irradiations suggest partial dissolution of the nanoclusters, but the extent of dissolution varies by irradiating particle. These results suggest that charged particle irradiations can produce comparable micro-scale features (e.g., dislocation loops and voids) as neutron irradiation, without a temperature or dose shift. However, at the nanoscale, the irradiating particle and/or irradiation dose rate has a greater effect, as charged particles are unable to produce consistent nanoscale features as neutron irradiation.

To correlate microstructure and nanostructure with macroscopic properties, the team conducted nanoindentation to evaluate the irradiation hardening of the neutron and charged particle irradiated samples. The nanohardness was

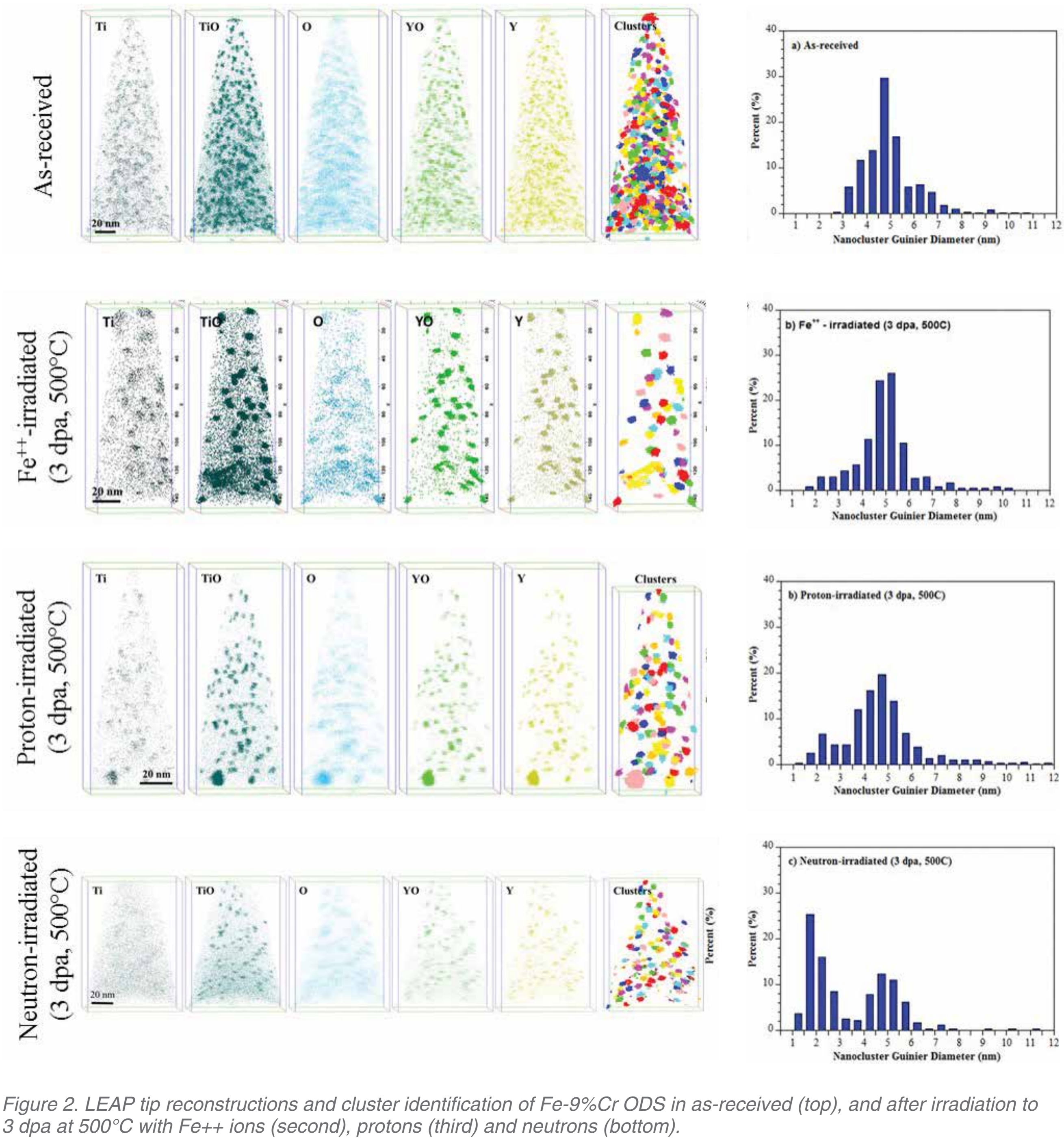

Figure 2. LEAP tip reconstructions and cluster identification of Fe-9%Cr ODS in as-received (top), and after irradiation to 3 dpa at 500°C with Fe++ ions (second), protons (third) and neutrons (bottom).

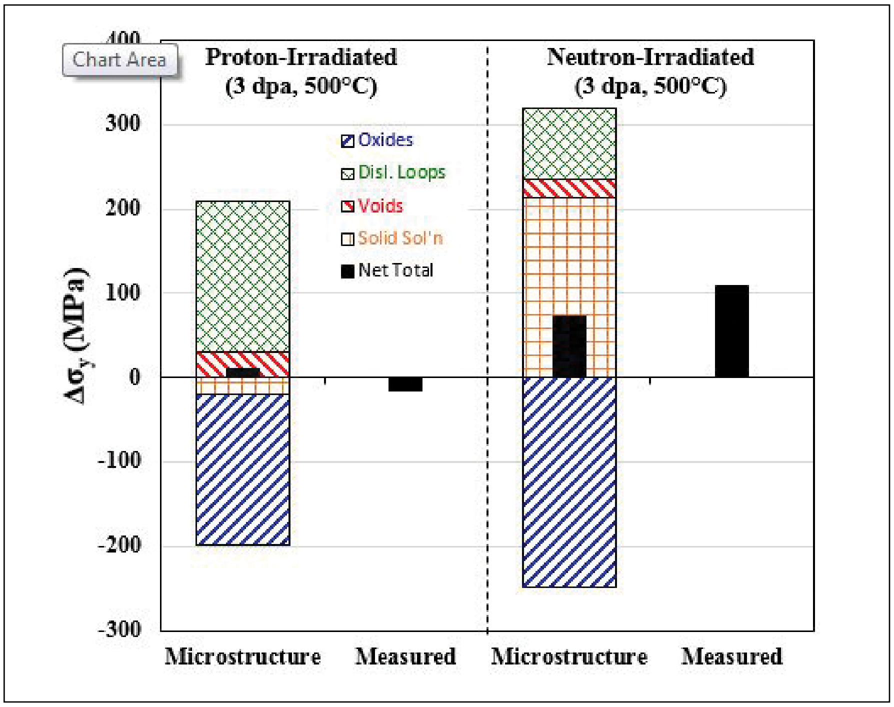

Figure 3. Contributions of dispersed barriers and solid solution to overall strengthening and comparison to measured strengthening via nanoindentation.

4.65 ± 0.26 GPa, 4.60 ± 0.13 GPa and 5.00 ± 0.06 GPa for the as-received, proton-irradiated, and neutron-irradiated, respectively, at 700–1000 nm indentation depths. The dispersed barrier hardening model was implemented to relate the microstructure evolution to the change in measured yield strength, but alone does not agree with measurements from nanoindentation. However, the dissolution of oxide nanoclusters and the subsequent re-entry of the oxide species into solid solution, must be considered. When a solid solution hardening model is appended to the dispersed barrier hardening model, calculations are able to predict measured changes in yield strength (Figure 3).

Work on this project was completed by Corey Dolph and Matthew Swenson, graduate students from Boise State University. The project team wishes to acknowledge Yaqiao Wu, Jatuporn Burns, and Joanna Taylor of CAES, who were instrumental in assisting with scheduling and instrument training and operation. The team also acknowledges Gary Was, Ovidiu Toader, Fabian Naab, and the team at the Michigan Ion Beam Laboratory, who assisted with charged particle irradiation experiment setup and monitoring.

Future Activities

Additional self-ion irradiations have been conducted at the Michigan Ion Beam Laboratory on Fe-9%Cr ODS to doses of 100 dpa at both 500°C and 400°C. The microstructural analysis of these irradiations will enable partial evaluation of dose and temperature dependence.

Publications and Presentations

1. Swenson, M. J., and J. P. Wharry, 2015, "The comparison of microstructure and nanocluster evolution in proton and neutron irradiated Fe-9%Cr ODS steel to 3 dpa at 500°C," *Journal of Nuclear Materials*, Vol. 467, 2015, pp. 97–112.

2. Swenson, M. J., C. K. Dolph, and J. P. Wharry, 2016, "The effects of oxide evolution on mechanical properties in irradiated Fe-9%Cr ODS," Manuscript in preparation, 2016.

3. Swenson, M. J., and J.P. Wharry, "Comparison of neutron, proton, and self-ion irradiation of Fe-9%Cr ODS at 3 dpa, 500°C," *The Minerals, Metals & Materials Society Annual Meeting*, *Orlando, Florida, February* 2016.

4. Swenson, M. J., C. Dolph, and J. P. Wharry, "The strengthening mechanism transition in nanofeatured ferritic-martensitic alloys," *The Minerals, Metals & Materials Society Annual Meeting, Orlando, Florida, March* 2015.

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

APT Study of Fission Product Transport in Unirradiated/Neutron Irradiated TRISO Fuel Particles

Yaqiao Wu – Boise State University – yaqiaowu@boisestate.edu

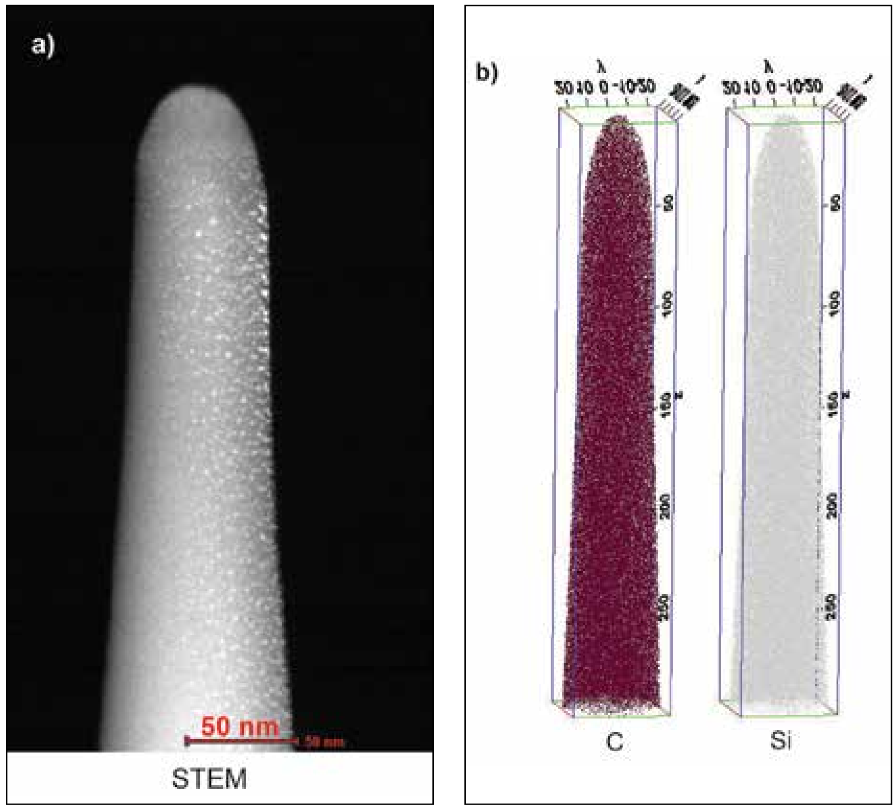

Figure 1. (a) STEM HAADF image of an unirradiated surrogate TRISO APT sample tip and (b) corresponding reconstructed three-dimensional APT maps of C and Si in a volume size of 48 nm x 49 nm x 293 nm.

Tristructural isotropic (TRISO) particle is a promising candidate as fuel for high-temperature gas reactors (HTGR). The TRISO coating is part of the HTGR functional containment and critical for the safety strategy for licensing purposes. Ag-rich phase was discovered by using Scanning Transmission Electron Microscopy (STEM) energy dispersive X-ray spectroscopy (EDS)/electron energy loss spectroscopy (EELS) techniques in previous studies along triple-junctions and grain boundaries, without in depth characterization of composition and distribution of the precipitates. The current research focuses on identifying fission products (FP), in particular Ag and Pd, released during post-irradiation examination (PIE) in TRISO particles by utilizing Atom Probe Tomography (APT) technique. It is critical to identify FPs and understand their transport mechanisms in SiC provide feedback to improve design of TRISO coating to reduce release of fission products from the TRISO coated particles.

Project Description

The TRISO particle is a promising candidate as fuel for HTGR. The TRISO coating is part of the HTGR functional containment and critical for the safety strategy for licensing purposes. This study aims at obtaining accurate compositional information and three-dimensional (3-D) spatial distribution of FPs in the SiC layer of neutron irradiated TRISO fuel by using the APT technique. This information will assist in gaining insight into the transport mechanisms of FP, particularly Ag, Pd, released from neutron irradiated TRISO fuel. Research that has been performed provided information on elements potentially present in the FP precipitates, but no in-depth characterization of composition and distribution of the precipitates has been concluded yet. Knowledge of the accurate composition and spatial distribution of the FPs is critical to understand their formation and transport. This proposed study continues the APT study part of completed RTE proposal with University of Wisconsin in 2014 (Title: STEM/LEAP Study of Fission Product Transportation in Neutron Irradiated TRISO Fuel Particles) and research conducted under the INL Advanced Gas Reactor (AGR) program. If successful, the obtained data will contribute to understanding the FP transport mechanisms in SiC and providing feedback to improve design of TRISO coating to reduce release of FP from the TRISO coated particles.

Accomplishments

The goals of this research are to identify and quantify fission products (in particular Ag and Pd). The research plan was designed by Yaqiao Wu and Isabella J van Rooyen. This research was conducted primarily by Yaqiao Wu (TEM, LEAP characterizations and data analyses) and the design, and availability of the APT research samples were primarily performed by Isabella J van Rooyen. Haiming Wen participated in an EML scheduling meeting by representing Idaho National Laboratory as the co-PI at this meeting. CAES MaCS staff, including Joanna Taylor and Kristi Moser-Mcintire, were of great help with handling TRISO fuels and scheduling equipment times. Two sample series of unirradiated surrogate TRISO fuel (supplied by John Hunn from Oak Ridge National Laboratory) and irradiated TRISO fuel from Compact 6-3-2 (from the AGR-1 experiment by INL) were characterized. APT tips of unirradiated surrogate samples were prepared at MaCS, Center for Advanced Energy Studies (CAES) by Jatuporn Burns using a FEI Quanta Focused Ion Beam (FIB). The irradiated samples were prepared at the MFC-EML by James Madden using a FEI Quanta FIB. TEM/EDS (FEI Tecnai G2 F30 FEG STEM) characterization on the tips before APT experiments (CAMECA LEAP 4000X HR) were conducted at MaCS, CAES. The APT data were analyzed by using CAMECA IVAS software package.

This is an unique result because for the first time we see Ag, Pd, and U together in a three-dimensional atomic structure. This is also the first time that APT was successfully applied to neutron irradiated SiC work to identify fission products.

— Dr. Isabella J. van Rooyen
TRISO Fuel Advanced Microscopy and Micro-Analysis Lead

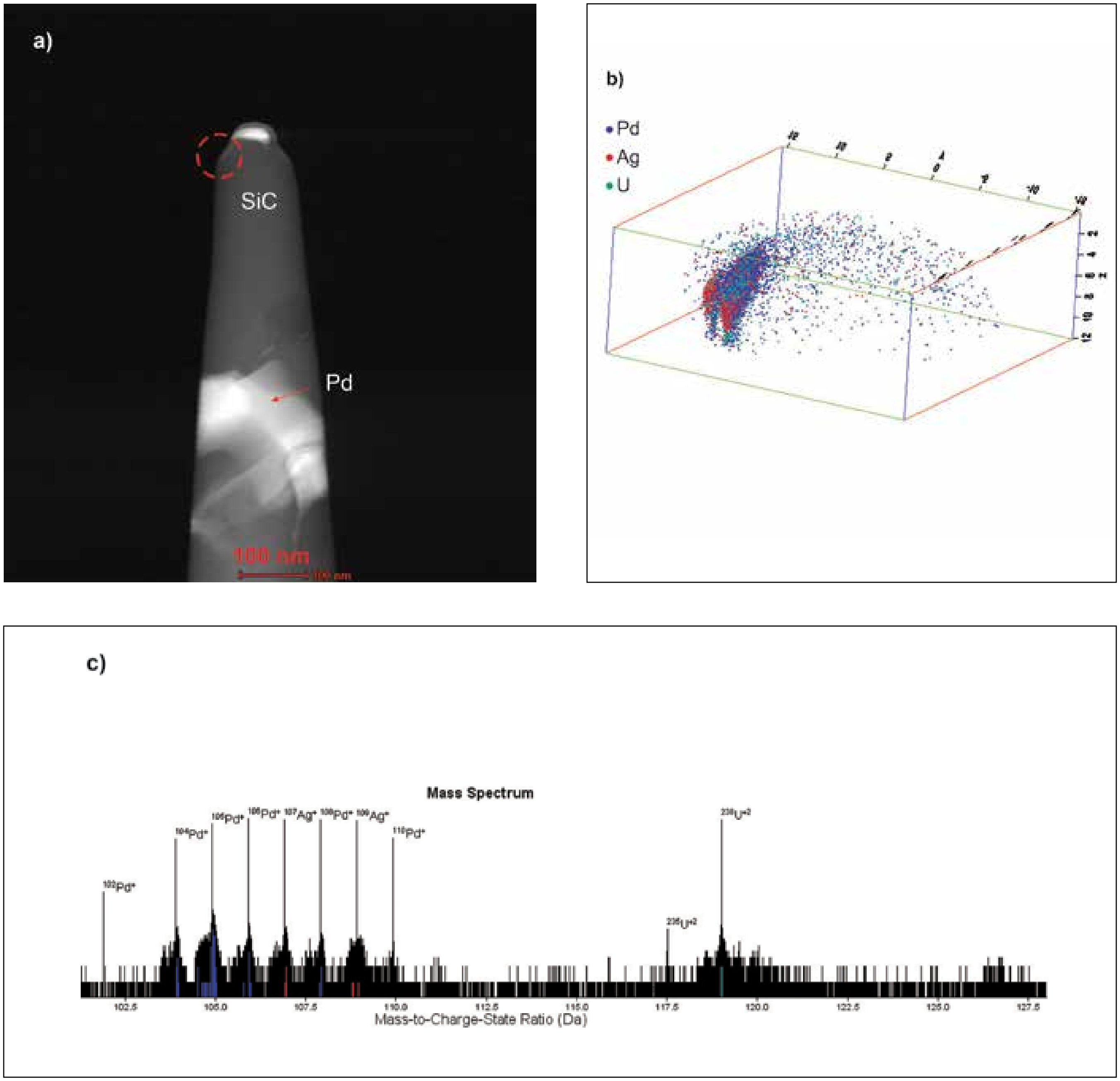

Figure 2. (a) STEM HAADF image of an irradiated TRISO APT sample tip, (b) corresponding reconstructed three-dimensional APT maps showing a Ag-Pd-U-rich phase in a volume size of 32 nm x 31 nm x 12 nm, (c) and mass spectrum showing detected isotopes of Ag, Pd, and U.

Knowing where fission products (Ag, Pd) are located and how they exist in TRISO fuels is very important to understand their transport mechanisms.

Challenges were encountered in APT experiments in (a) preparing LEAP sample tips containing Ag-/Pd-rich phases close to tip apex due to low number densities of Ag-rich phase and grain/phase boundaries; (b) obtaining large enough volume to encounter features of interest due to extremely brittle irradiated SiC sample that has low electric and thermoconductivities, as well as big differences in evaporation field needed in LEAP runs between C (142 V/nm C+, 103 V/nm C++) and Si (45 V/nm Si+ and 33 V/nm Si++). Therefore, nine tips were fabricated from both the unirradiated and irradiated samples, to increase the chance of obtaining quality data. By tailoring LEAP running parameters, the running mode, sample setting temperature, laser energy, and pulse frequency for unirradiated surrogate are laser, 90K, 100 pJ, 200 kHz, respectively; and those for irradiated TRISO samples are laser, 95K, 300 pJ, 125 kHz, respectively.

Representative scanning transmission electron microscope STEM image of an atom probe sample tip from unirradiated surrogate TRISO fuel and corresponding reconstructed 3-D elemental maps of C and Si are shown in Figure 1. The diameter of the sample tip apex is about 50 nm. C and Si atoms are uniformly distributed in the volume (48 nm x 49 nm x 293 nm). Quantitative analysis show the concentrations of C and Si within this volume are 44.813 at.% (error 0.014 at.%) and 53.965 at.% (error 0.014 at.%), respectively. About 1.2 at.% O was also detected within this volume, which is mostly attributed to the air. All APT data obtained from the unirradiated surrogate TRISO fuel samples show similar concentrations of C and Si of about 45 at.% and 54 at.%, respectively.

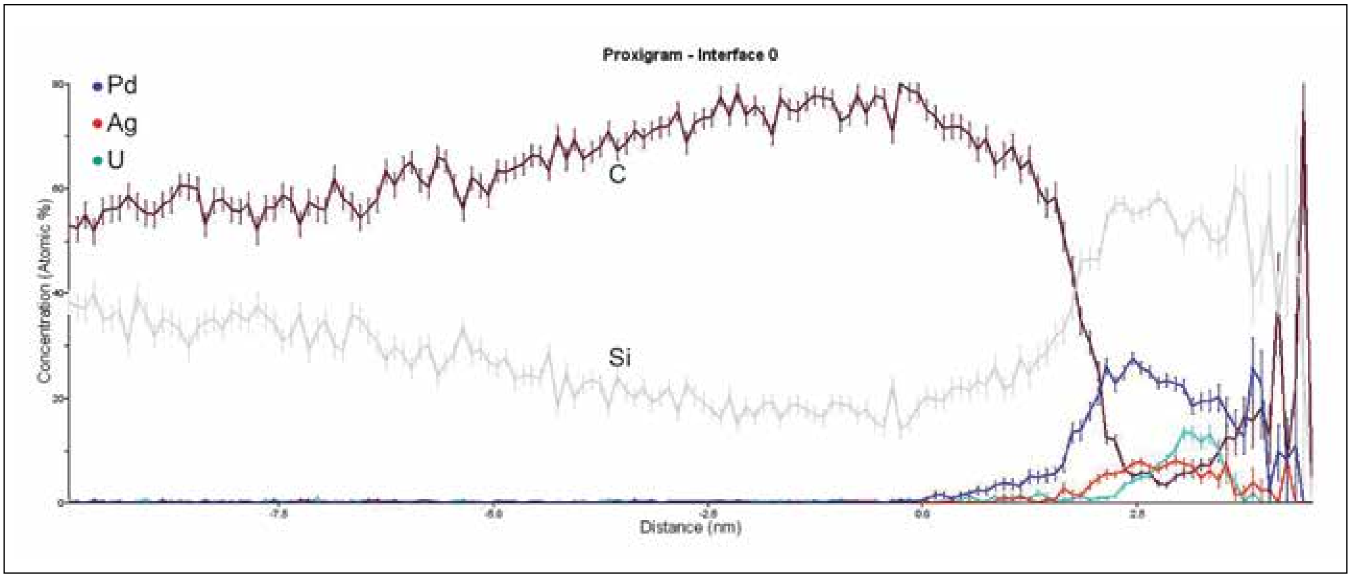

Figure 3. Proxigram with respect to a 3 at.%Pd isoconcentration surface, clearly indicating a Pd, Ag, and U-enriched phase. This phase also contains Si and some C.

Figure 2 shows a STEM image of an irradiated TRISO APT tip and corresponding LEAP data. The mass spectrum (Figure 2c) clearly indicate isotopes of 107Ag+ and 109Ag+ among Pd isotopes, as well as 235U+2 and 238U+2. An Ag Pd-U phase is identified at one side of the reconstructed volume (Figure 2b), which corresponds to the dashed red-line circled region in Figure 2a.

By selecting 3 at.%Pd as an interface, proxigram calculated with respect to the interface (Figure 3) clearly indicates a Pd, Ag, and U-enriched phase. This phase also contains Si and some C. From this proxigram, the local peak concentrations of Pd, Ag, U, Si, and C are about 27.6 at.%, 8.1 at.%, 13.4 at.%, 59.5 at.%, and 3.5 at.%, respectively. A 3-D elemental map of a volume of 6 nm x 6 nm x 6 nm is selected covering this Pd-Ag-U-rich phase is shown in Figure 4. Quantitative analysis within the Pd-Ag-U rich phase show that the concentrations of Pd, Ag, U, Si, and C are 12.813 at.% (error 0.451 at.%), 5.466 at.% (error 0.320 at.%), 6.020 at.% (error 0.312 at.%), 53.825 at.% (error 0.595 at.%) and 20.640 at.% (error 0.332 at.%), respectively.

Future Activities

Experimentation for this specific rapid turnaround experiment (RTE) project is now complete. A journal publication will be prepared and published in 2016.

Publications and Presentations

1. Wu, Y. Q., I. J. van Rooyen, H. M. Wen, J. Burns, and J. W. Madden, "Microstructure Characterization of TRISO fuels by Atom Probe Tomography," *TMS 2016, February 14–18, 2016*, accepted.
2. Wu, Y. Q., I. J. van Rooyen, J. W. Madden, J. Burns and H. M. Wen Journal paper in preparation.

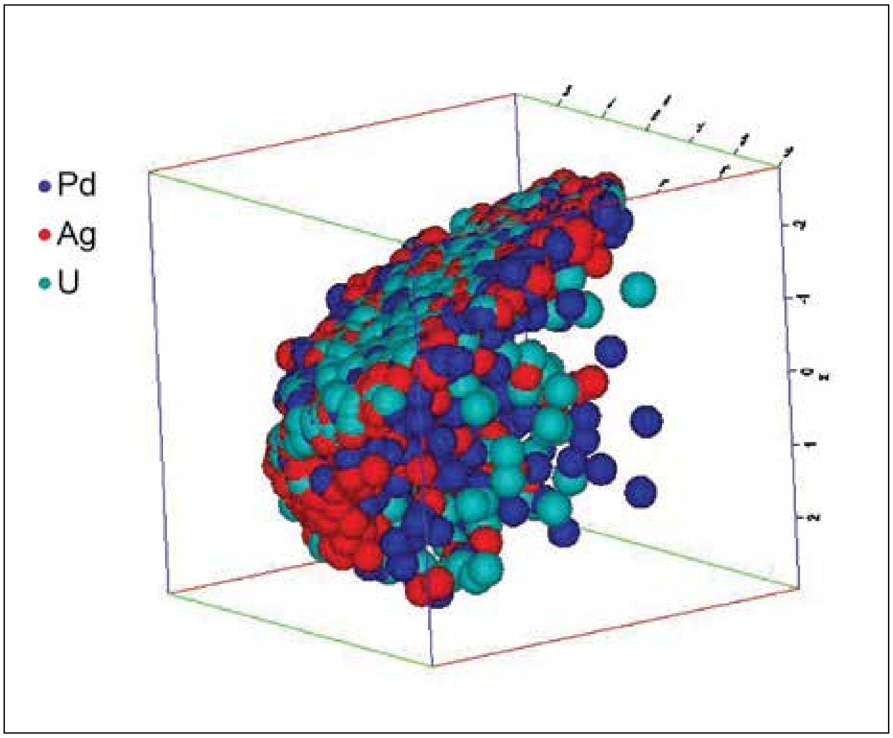

Figure 4. Three-dimensional elemental map of Pd, Ag, and U in a selected small volume of 6 nm x 6 nm x 6 nm.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Electron Microscopy Laboratory (EML)
Collaborators	
Boise State University	Yaqiao Wu (principal investigator), Jatuporn Burns (collaborator)
Idaho National Laboratory	Isabella J. van Rooyen (co-principal investigator), Haiming Wen (collaborator), James Madden (collaborator)

Investigation of Precipitate Formation Kinetics and Interactions in FeCrAl Alloys

Samuel A. Briggs – University of Wisconsin, Madison – sabriggs2@wisc.edu

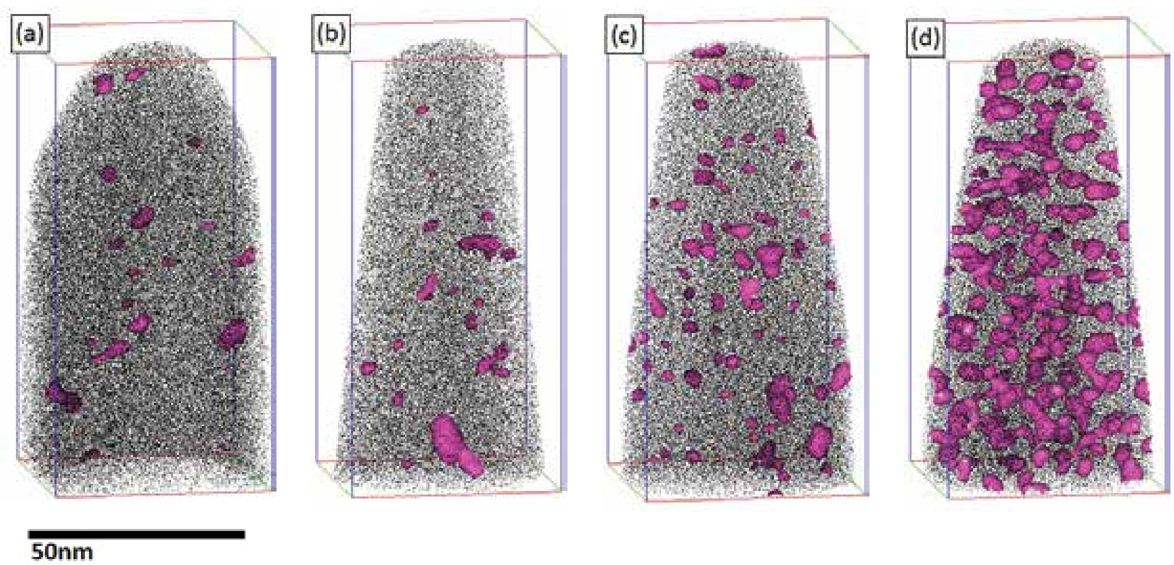

Figure 1. α' precipitates represented by 34 at.% Cr concentration isosurfaces for (a) Fe-10Cr-4.8Al, (b) Fe-12Cr-4.4Al, (c) Fe-15Cr-3.9Al, and (d) Fe-18Cr-2.9Al, neutron-irradiated in HFIR to 7dpa at 320°C. Black dots represent 2% of detected matrix Fe atoms.

FeCrAl alloys are currently being considered for accident-tolerant light water reactor (LWR) fuel cladding applications and show promise as a new alloy class for nuclear systems due to their superior oxidation and corrosion resistance in high temperature environments. However, high-Cr (>9 at.%) ferritic alloys are known to be susceptible to precipitation of a deleterious α' phase during long-term high-temperature exposure. This process has been shown to be accelerated by irradiation and tends to result in hardening and embrittlement of the material. As such, it is important to understand the factors that influence the formation of these precipitates to mitigate their effect on material properties when designing an alloy for nuclear applications.

Project Description

This work investigates the composition and dose dependencies for α' precipitation in neutron-irradiated FeCrAl alloys using atom probe tomography (APT) techniques as part of a rigorous irradiation and characterization campaign under the DOE Fuel Cycle Research and Development (FCRD) program. The data collected from the APT technique is reconstructed into a three-dimensional (3-D) rendering of the material in question and allows for detailed study of the spatial distribution of constituent elements, allowing for identification and study of the morphology and distribution of Cr-rich α' clusters in the α-Fe matrix with relative ease. Of particular interest are the shape, size, number density and composition of the precipi-

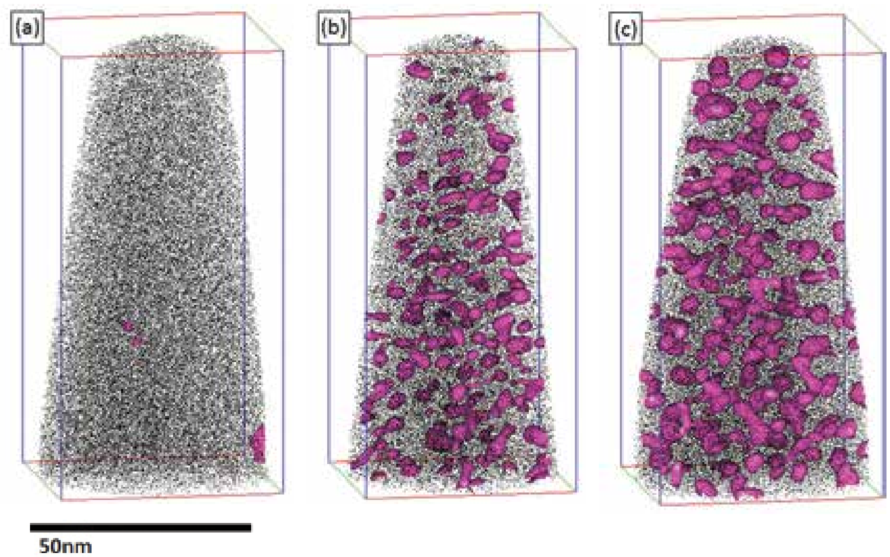

Figure 2. α' precipitates represented by 34 at.% Cr concentration isosurfaces for neutron-irradiated Fe-18Cr-2.9Al, irradiated to (a) 0.8 dpa at 355°C, (b) 1.8 dpa at 382°C, and (c) 7dpa at 320°C. Black dots represent 2% of detected matrix Fe atoms.

tates resulting from the irradiation treatment. Knowledge of the precipitate microstructure is to be coupled with mechanical testing and transmission electron microscopy (TEM) investigation of dislocation loop structure to develop hardening models for the FeCrAl alloy system.

Furthermore, this work directly correlates to previous studies in aged and irradiated FeCr binary systems as part of the ATR-1 and ATR-2 irradiation experiments. Al additions are expected to affect the α-α' phase boundary and lead to increased solubility of Cr, which may mitigate the irradiation-induced embrittlement issues that have been observed in similar ferritic systems. Ultimately, results of the current work will have broad reaching impacts on ferrous-based alloy development for nuclear power generation including cladding, structural materials, and corrosion barrier systems in both fusion and fission systems.

Accomplishments

Four FeCrAl model alloys with nominal compositions of Fe-10Cr-4.8Al, Fe-12Cr-4.4Al, Fe-15Cr-3.9Al, and Fe-18Cr-2.9Al have been fabricated at Oak Ridge National Laboratory (ORNL) and neutron-irradiated in the High Flux Isotope Reactor (HFIR) to nominal damage doses up to 7dpa at a target temperature of 320°C prior to FY 2015. In FY 2015, samples for atom probe tomography (APT) analysis

Understanding the mechanism and kinetics of α' clustering is FeCrAl alloys is essential in order to mitigate radiation-induced hardening and embrittlement effects for accident tolerant fuel cladding applications.

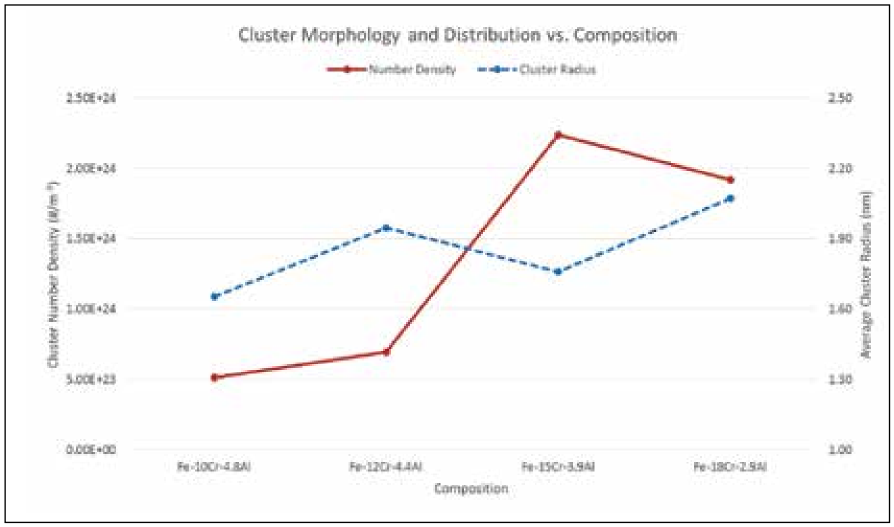

Figure 3. Trends in calculated cluster number density and average spherical equivalent cluster radius for α' precipitates in 7 dpa Fe-Cr-Al for the four model alloy compositions.

Atom probe tomography capabilities at the CAES facility has allowed for a detailed assessment of embrittlement mechanisms in candidate LWR accident-tolerant fuel cladding materials.

— Samuel A. Briggs
Ph.D. Candidate, Department of Engineering Physics, University of Wisconsin-Madison

were prepared at the ORNL Low Activation Materials Development and Analysis (LAMDA) facility using standard focused ion beam (FIB) lift-out techniques. Atom probe data was collected using the CAMECA LEAP 4000X HR located at both the Center for Nanophase Materials Science (CNMS) at ORNL and the Microscopy and Characterization Suite (MaCS) at the Center for Advanced Energy Studies (CAES). The samples were analyzed in laser mode at 50 K, with a pulse frequency of 200 kHz, laser pule energy of 50 pJ, and a detection rate of 0.005–0.02 atoms per pulse. Data was reconstructed and analyzed using CAMECA's Integrated Visualization and Analysis Software (IVAS) Version 3.6.8.

Dependence of precipitation on Cr and Al content was determined by analyzing the 7-dpa condition of each model alloy composition. In addition, evolution of precipitate morphology with dose was investigated by analyzing specimens from the Fe-18Cr-2.9Al composition irradiated to 0.8, 1.8, and 7 dpa, in addition to the as-received state.

Representative reconstructions of the specimens of different compositions irradiated to 7 dpa are found in Figure 1. Reconstructions of the Fe-18Cr-2.9Al specimens irradiated to different nominal damage doses are found in Figure 2. It was found that the volume fraction and Cr content of precipitates both increased with increasing Cr content and decreasing Al content. However, the Cr content of the α' clusters in all FeCrAl alloys studied was consistently lower than the reported saturation composition of α' clusters observed in the FeCr binary system. Furthermore, cluster number density trended upward with increasing bulk Cr content, but saw a decrease at the highest bulk Cr content coupled with an increase in average cluster size. With regards to dose dependence, a high number density of smaller precipitates is seen in the lower dose specimens with volume fraction and cluster Cr content increasing asymptotically with increasing neutron fluence. This indicates that these clusters are beginning to nucleate and grow after short irradiation times and proceed to coarsen as irradiation continues. Plots illustrating the composition and dose dependencies of observed cluster size and number density are shown in Figures 3 and 4.

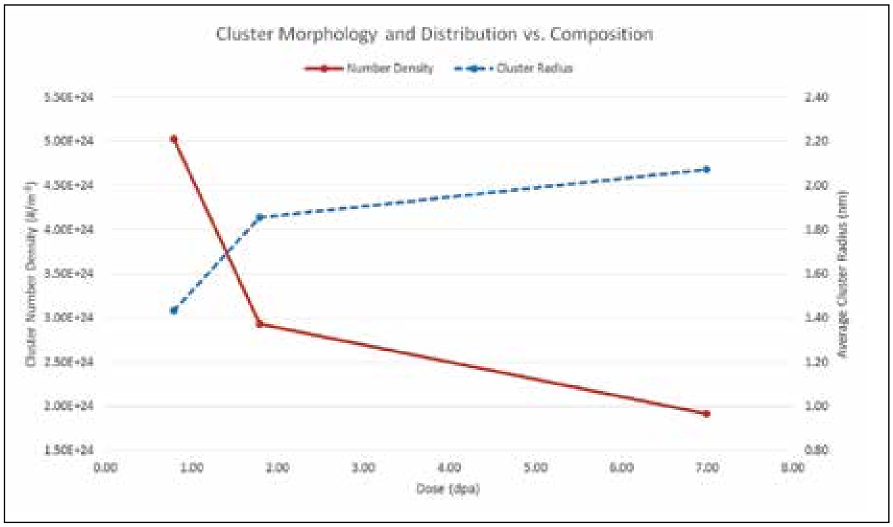

Figure 4. Trends in calculated cluster number density and average spherical equivalent cluster radius for α' precipitates in Fe-18Cr-2.9Al for different nominal radiation damage doses.

Future Activities

In FY 2016 the cluster morphology will be correlated with the results of tensile and impact testing to develop hardening models for the FeCrAl alloy system. Additionally, the APT data presented will be used to refine small-angle neutron scattering (SANS) data previously collected on the materials investigated. SANS analyses can be very sensitive to the assumed composition of the α-Fe and α' phases present. Coupling the SANS and APT data sets will assist in developing a complete and robust understanding of precipitation in the FeCrAl alloy system.

Publications and Presentations

1. Field, K. G., S. A. Briggs, P. D. Edmondson, X. Hu, K. C. Littrell, R. Howard, C. M. Parish, and Y. Yamamoto, 2015, *Evaluation of the effect of composition on radiation hardening and embrittlement in model FeCrAl alloys*, FY-15 FCRD Milestone Report: ORNL/TM-2015/518, September 2015.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Oak Ridge National Laboratory	High Flux Isotope Reactor, Irradiated Materials Examination and Testing Facility Hot Cells, Low Activation Materials Design and Analysis Laboratory
Collaborators	
Oak Ridge National Laboratory	Kinga A. Unocic (co-principal investigator), Kevin G. Field (collaborator)
University of Wisconsin-Madison	Samuel A. Briggs (co-principal investigator), Kumar Sridharan (co-principal investigator)

Investigating Alpha Prime Precipitation in a Neutron Irradiated NFA *(Nanostructured Ferritic Alloy)*

Kris Bhojwani – University of Oxford – kris.bhojwani@materials.ox.ac.uk

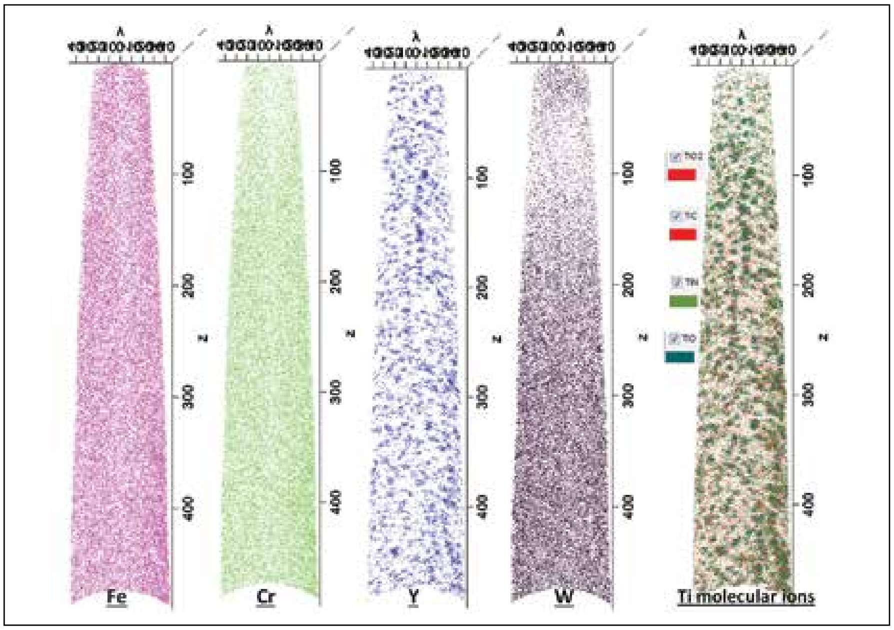

Figure 1. Atom probe tomographic ion maps from the neutron irradiated 14YWT NFA (dataset R33_4731). Shown here are Fe, Cr, Y, W, and Ti molecular ion maps. No Cr clustering can be seen. NFA dispersoids positions and morphology are represented by the Y and Ti molecular ion maps. Some high density W zones exist in the W ion map, but this requires further study.

To construct the first structural wall within the blanket of a nuclear fusion tokamak reactor, the candidate alloys are 14YWT non-ferrous alloys (NFAs). This is because they possess superior creep properties at high temperature and have a high tolerance to irradiation.

Coolants at temperatures between 300 and 500°C are likely to be used within the blanket. Fe-Cr phase diagrams indicate that the alloy will reside within the α- α' region in the miscibility gap at these temperatures. For Fe-Cr alloys with a low quantity of Cr, the α' phase manifests itself in the form of spherical precipitates. These α' precipitates induce embrittlement and increases in its hardness. As 14YWT is predominantly composed of Fe and Cr, they too could be susceptible to this phase formation.

This study aims to examine the microstructure of 14YWT that has undergone neutron irradiation to 1.7dpa at 288°C for 8 months, using atom probe tomography (APT).

Project Description

The prime objective of this research is to determine whether α' precipitates have formed within the neutron irradiated 14YWT NFA. If APT experiments are successful, an ion map of the local microstructure can be obtained. This will yield compositional and morphological information on any other features that are captured in the experiments.

Secondly, the atom probe data from this neutron-irradiated 14YWT will be

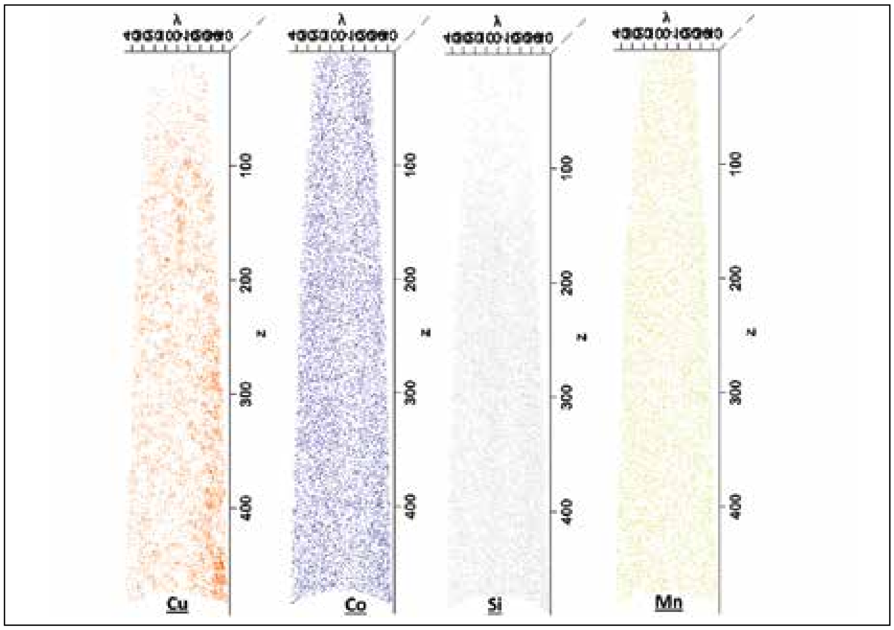

Figure 2. Atom probe tomographic ion maps from the neutron irradiated 14YWT NFA (dataset R33_4731). Shown here are Cu, Co, Si, and Mn molecular ion maps. High density Cu zones correspond to NFA dispersoid positions. It can be seen that Cu decorates the NFA clusters. Co and Mn are homogenous. Some Si high density zones occur, but this requires further study.

compared to 14YWT irradiated with Fe ions to the same peak dose and irradiation temperature as the neutron irradiated 14YWT (1.7 dpa and 288°C). This self-ion irradiation campaign has been completed at JANNUS in France, in 2014.

If 14YWT NFAs are susceptible to α' precipitation then APT will provide information required to calculate valuable metrics such as kinetic information on their formation. Additionally, data on the precipitates, such as their number density, radii, volumes, and compositions, can also be obtained. This data could also provide insight into whether preferential nucleation occurs at dispersoids or any other local features. This would be advantageous to theorists when trying to study the mechanisms underpinning α' precipitation within FeCr alloys generally. Secondly, alloy developers would have valuable local compositional information on solute movements, which may help refine their processing methods. Ultimately, the formation of α' precipitates induces mechanical changes that could shorten the lifetime of that component. Therefore, screening the microstructure of the 14YWT in this manner would provide tokamak engineers information on whether 14YWT could be placed close to the coolants.

The 14YWT NFAs have desirable properties for fission applications too. As one of the DOE's goals is to develop improvements in existing technologies the outcomes of this research may alter material choices for new components that are to be installed on fission plants.

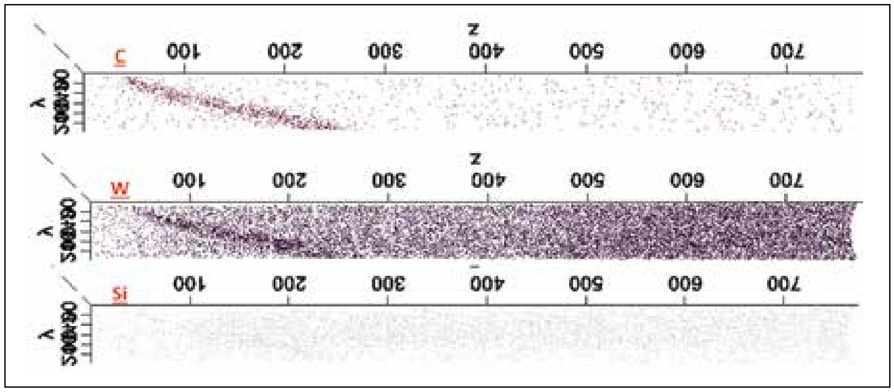

Figure 3. Atom probe tomographic ion map of neutron irradiated 14YWT NFA (dataset R33_4724). Shown here are C, W, and Si ion maps. As can be seen, there is segregation of these solutes to the grain boundary captured in this dataset.

The 14YWT NFA may be suitable for protecting more delicate components within a tokamak blanket at low temperatures and in a neutron irradiation environment.

Accomplishments

There are two main goals for this research. The first is to determine whether α' precipitates have formed within an irradiated 14YWT NFA using APT. The second is to compare the microstructure of the neutron irradiated 14YWT NFA with a 14YWT NFA irradiated with Fe ions. The ion irradiated 14YWT NFA has the same peak dose and the same irradiation temperature of 1.7 dpa and 288°C, respectively.

Following the proposal approval, APT experiments were successfully conducted at the Center of Advanced Energy Studies (CAES) in Idaho, on the neutron 14YWT NFA. APT samples were fabricated on the focused ion beam (FIB). Three datasets were acquired and all three have a low background noise at roughly ten counts across the spectrum. They are also very large by atom probe standards sampling as much as 5.21 x 10-21m^3 of the APT specimens, which equates to around 160 x 106 ions.

Due to the complexity of the alloy and the size of the datasets acquired, the APT reconstruction process has been challenging. In particular, magnification effects induced by the atom probe initially warped the dispersoids so that they look like ellipses. However, it can be reported that all three datasets have now been reconstructed and the dispersoids looks spherical.

From the APT data acquired, the neutron irradiated 14YWT NFA appear to not have formed any α' precipitates. Statistical analysis of the Cr ion maps shows a random distribution of Cr. Moreover, no other precipitate or intermetallic, apart from the NFA dispersoids, were found within the matrix.

There is significant W segregation as well as some P to the grain boundaries. Concentrations across the grain boundaries are to be determined. Si, which is an impurity in this alloy, also segregates to the grain boundary.

Cu is also present in the matrix and is another impurity. From the Cu ion map it can be seen that it decorates the dispersoids.

This research was conducted primarily by Kris Bhojwani and facilitated by Professor Bob Odette and Peter Wells at the University of California Santa Barbara, as well as the staff at CAES including Yaqaio Wu, Joanna Taylor, and Allyssa Bateman.

Future Activities

The dispersoid characteristics, such as radii, volume, and composition in the neutron irradiated 14YWT require further investigation and shall be conducted in 2016.

The second objective that was to compare this neutron irradiated 14YWT NFA has not been completed yet. Due to equipment challenges at the University of Oxford all the ion irradiated 14YWT APT datasets have not been acquired yet.

Access to the CAES facility has enabled researchers to conduct atom probe experiments on hot samples quickly and efficiently.

— Kris Bhojwani

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
University of Oxford	Kris Bhojwani (principal investigator), Professor S. G. Roberts (co-principal investigator)

INL-AECL Joint Project for Active FIB and TEM analysis of Irradiated X-750, CRADA 11-CR-16

John H. Jackson – Idaho National Laboratory – john.jackson@inl.gov

This work will provide much needed microstructural information toward understanding the evolution of material microstructure as a function of temperature and very high neutron fluence. The work will lead to a better understanding of the role of helium formation in nickel alloys as neutron damage accumulates.

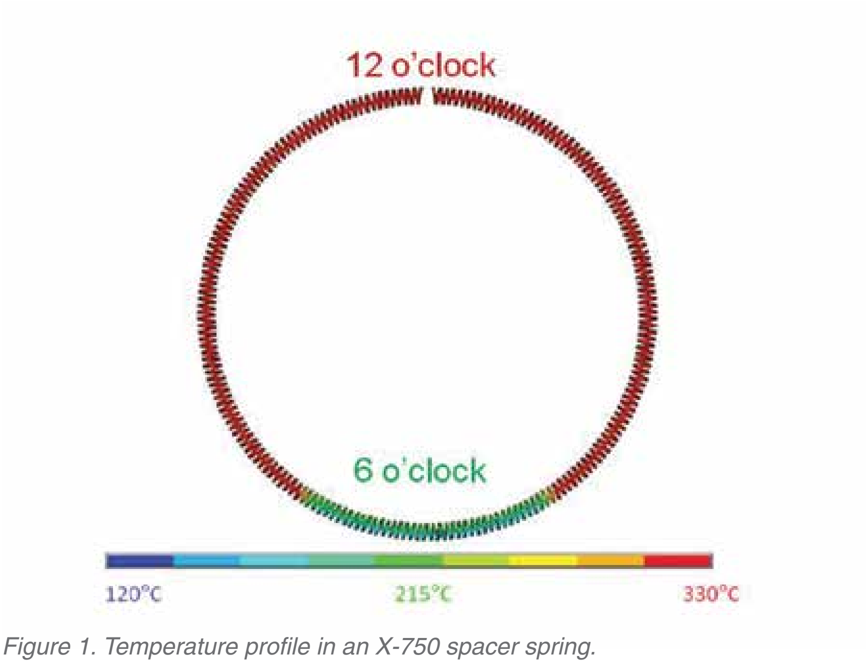

Figure 1. Temperature profile in an X-750 spacer spring.

Project Description

This joint project examines the microstructural changes of alloy X-750 as a function of neutron irradiation and temperature. Several garter spring sections that had been used in service as fuel channel spacers in Canada Deuterium Uranium (CANDU) reactors were provided to INL by the Atomic Energy of Canada's (AECL) Chalk River Laboratory (now Canadian Nuclear Laboratories Limited [CNL]). These spring sections had exhibited extreme loss of ductility during service inspections and have become the subject of fitness for service investigations by the CANDU reactor industry. Preliminary field inspections indicated a potential relationship between loss of ductility and location of spring sections; a thorough microstructural analysis was deemed necessary to understand the behavior of these spring materials.

Alloy X-750 is a nickle-based super alloy that is commonly used in light water reactor internal components as well as the CANDU reactor. Embrittlement caused by a neutron environment is of interest for lifetime prediction in the current fleet of reactors in the United States and the rest of the world. This work directly relates to relicensing efforts for light water reactors.

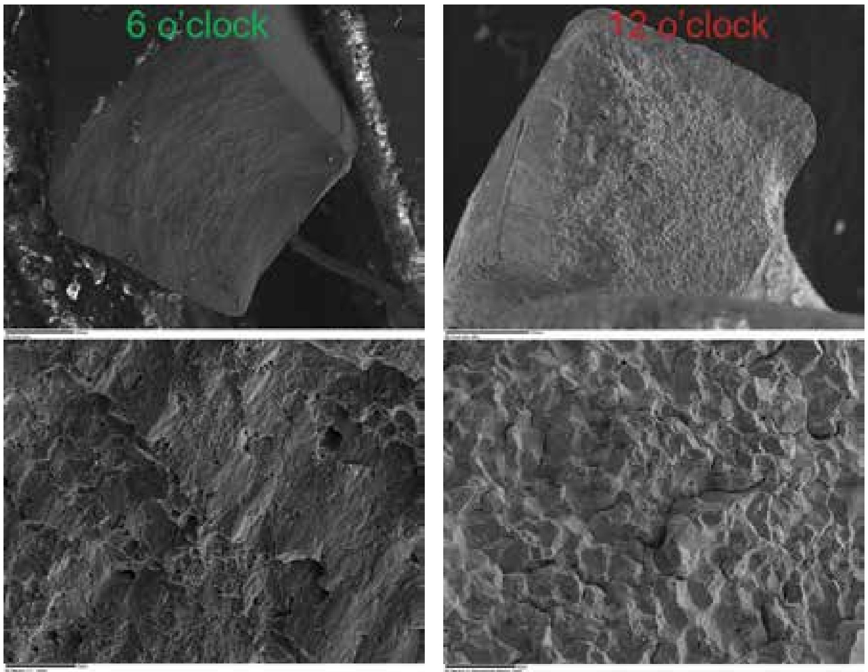

Figure 2. Fractography of 6 o'clock and 12 o'clock specimens at ~6 dpa and 1000 appm He.

The collaboration between CNL and INL has advanced our understanding of reactor core components through the use of state-of-the-art microscopy facilities.

— Colin D. Judge, R&D Scientist

Accomplishments

In late 2011, AECL shipped several small, 1 mm x 1 mm cubes of irradiated X-750 that had been removed from regions of interest in several garter springs. Figure 1 is a representation of a typical garter spring with the calculated temperature profile during service. Since temperature can contribute to embrittlement and it was noted that some regions of the springs exhibited ductility while others were severely embrittled, specimens were selected from spacer regions near the 6 o'clock position (lower temperature due to pinching against the cooler calandria tube) and the 12 o'clock position (higher temperature). In addition to temperature variation, specimens were extracted from springs that had accumulated DPA levels between 6 and 55 and that contained differing levels of He between 1500 and 18,000 appm.

During 2012 and into 2013, transmission electron microscopy (TEM) foils were produced using the active focused ion beam (FIB) at the INL's Electron Microscopy Laboratory (EML). TEM analyses were then performed by a visiting AECL scientist at the CAES Microscopy and Characterization Suite (MaCS). In addition to TEM analysis, hardness of the specimen sections was obtained using a shielded microhardness tester at the INL EML.

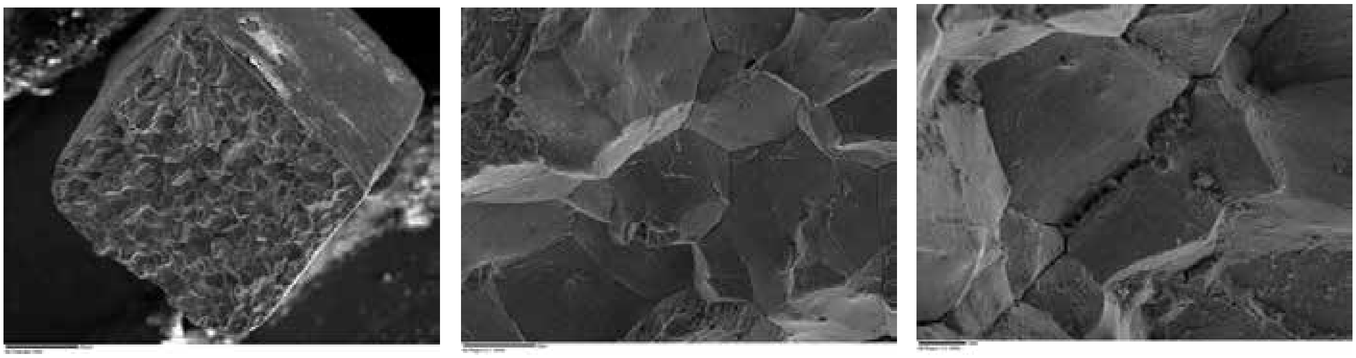

Figure 3. Fractography of 6 o'clock and 12 o'clock specimens at ~55 dpa and 18,000 appm He showing no appreciable difference.

It was noted by AECL following scanning electron microscope fractography studies that there was a definite difference in ductility (Figure 2) between the hot region of the spring and the cold region of the spring with the hotter, 12 o'clock region exhibiting brittle fracture features at lower DPA levels. Additionally, at higher DPA and He content there was no difference due to temperature (Figure 3). In correlation with this the TEM studies performed during 2012 and 2013 show a difference in cavity/bubble characteristics between high and low-temperature regions as well as cavity/bubble coalescence at higher DPA levels (Figure 4).

During Fiscal Years 2014 and 2015, additional material was shipped from Chalk River Laboratories to the INL EML to allow additional focus on helium bubble formation at grain boundaries in some of the more highly damaged regions of the garter springs. Sixteen additional TEM lamellae were fabricated using the active FIB at INL and have been partially examined at CAES and McMaster University. FY 2015 marks the completion of this project scope, but additional work scope may be pursued in a Work For Others agreement between Canadian Nuclear Laboratories and INL.

Future Activities

The NSUF portion of this project was completed in FY 2015. Additional studies will be carried out at McMaster University and at Canadian Nuclear Laboratories (CNL). Continued collaboration between CNL and INL is expected with a focus on the contribution of helium formation to embrittlement of these springs. There are papers being drafted for submission including the work performed this fiscal year, which will likely be published in 2016, including authorship of INL's Jim Cole, Jim Madden, and John Jackson.

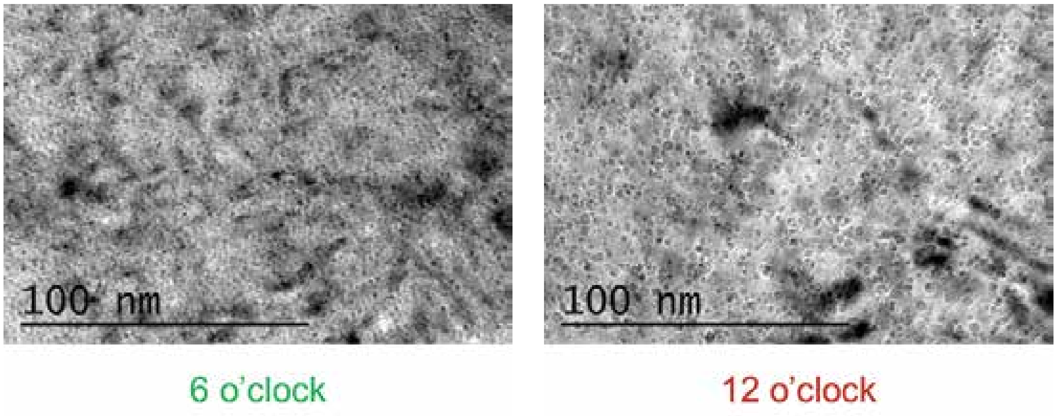

Figure 4. TEM images showing higher density of smaller cavities/ bubbles in cooler region.

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2. Griffiths, M., G. A. Bickel, S. A. Donohue, P. Feenstra, C. D. Judge, L. Walters, and M. D. Wright, 2013, "In Degradation of Ni alloy Components in CANDU Reactor Cores," *16th Int. Conference on Environmental Degradation of materials in Nuclear Power Systems - Water Reactors, Asheville, North Carolina, USA, August 11–15, 2013, Asheville, North Carolina.*

3. Judge, C. D., N. Gauquelin, M. Wright, J. I. Cole, J. Madden, G. A. Botton, and M. Griffiths, 2015, "Intergranular fracture in irradiated Inconel X-750 containing very high concentrations of helium and hydrogen," *Journal of Nuclear Materials*, Vol. 457, No. 0, 2015, pp. 165–172.

Staff exchanges

Colin Judge, the co-PI and an R&D scientist from Canadian Nuclear Laboratories was present at the CAES facility for some of the TEM examinations and for some of the FIB milling work.

Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Electron Microscopy Laboratory
Collaborators	
Canadian Nuclear Laboratories	Colin Judge (co-principal investigator), Malcolm Griffiths (collaborator)
Idaho National Laboratory	John H. Jackson (principal investigator), James Cole (collaborator)

NSUF LIST OF ACRONYMS

3-D	three-dimensional
AECL	Atomic Energy of Canada
AES	auger electron spectroscopy
AGR	Advanced Gas Reactor
ALD	atomic layer deposition
ANL	Argonne National Laboratory
APS	Advanced Photon Source
APT	Atom Probe Tomography
ATLAS	Argonne Tandem Linac Accelerator
ATR	Advanced Test Reactor
ATRC	Advanced Test Reactor Critical
BWR	boiling water reactor
CAES	Center for Advanced Energy Studies
CINR	Consolidated Innovative Nuclear Research
CNL	Canadian Nuclear Laboratories Limited
CNMS	Center for Nanophase Materials Science
DOE	Department of Energy
EDS	energy dispersive X-ray spectroscopy
EELS	electron energy loss spectroscopy
EML	Electron Microscopy Laboratory
EUV	extreme ultraviolet
EUVR	extreme ultraviolet reflectometry
FCCI	fuel-cladding chemical interaction
FDEG	field emission gun
FIB	focused ion beam
FP	fission product
FY	fiscal year

GAIN .... Gateway for Accelerated Innovation in Nuclear
HAADF .... high angle annular dark field
HFEF .... Hot Fuel Examination Facility
HFIR .... High Flux Isotope Reactor
HTGR .... high temperature gas-cooled reactor
IFEL .... Irradiated Fuels Examination Laboratory
IIT .... Illinois Institute of Technology
IMET .... Irradiated Materials Examination and Testing
IMPACT .... Interaction of Materials with Particles and Components Testing
INL .... Idaho National Laboratory
LAMDA .... Low Activation Materials Development and Analysis
LEAP .... local electrode atom probe
LEISS .... low-energy scattering spectroscopy
LM .... liquid metal
LOCA .... loss of coolant accident
LWR .... light water reactor
MaCS .... Microscopy and Characterization Suite
MFC .... Materials and Fuels Complex
MIT .... Massachusetts Institute of Technology
MITR .... Massachusetts Institute of Technology Reactor
MNSP .... Mn-Ni-Si precipitates
MOX .... mixed oxide
MRCAT .... Materials Research Collaborative Access Team
MSTL .... Materials Science and Technology Laboratory
MTR .... Material Testing Reactor
NAA .... Neutron Activation Analysis
NCSU .... North Carolina State University
NE .... Office of Nuclear Energy
NEID .... Nuclear Energy Infrastructure Database
NEUP .... Nuclear Energy University Program
NFA .... non-ferrous alloy
NGNP .... Next Generation Nuclear Plant
NRAD .... Neutron Radiography

NSUF .... Nuclear Science User Facilities
ODS .... oxide dispersion strengthened
ORNL .... Oak Ridge National Laboratory
OSTI .... Office of Science and Technical Information
PHYSOR .... Physics of Reactors
PI .... principal investigator
PIE .... post-irradiation examination
PMP .... Project Management Professional
PSU .... Pennsylvania State University
PWR .... pressurized water reactor
QA .... Quality Assurance
R&D .... research and development
RDF .... Radial Distribution Function
REDC .... Radiochemical Engineering Development Center
RF .... radio frequency
RFI .... request for information
RIS .... radiation induced segregation
RPV .... reactor pressure vessel
RTE .... rapid turnaround experiment
SAED .... selected area electron diffraction
SANS .... small-angle neutron scattering
SEM .... scanning electron microscope
SFR .... sodium cooled fast reactor
SMR .... small modular reactor
STEM .... Scanning Transmission Electron Microscopy
TEM .... transmission electron microscope
TRIGA .... Training Research Isotope General Atomics
TRISO .... tristructural isotropic
TRTR .... Test, Research and Training Reactors
XAFS .... X-ray absorption fine structure
XPS .... X-ray photoelectron spectroscopy
XRD .... X-ray diffraction

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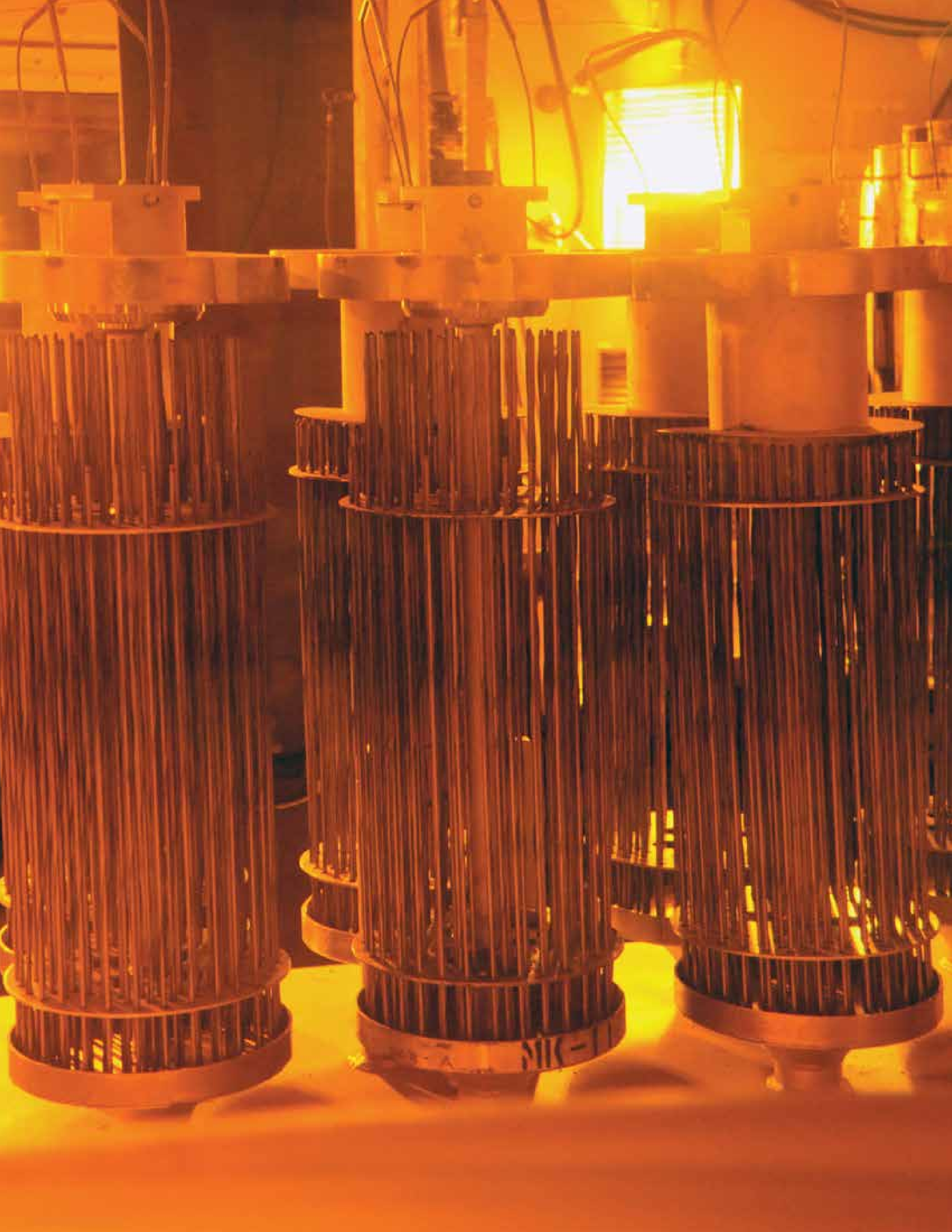

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