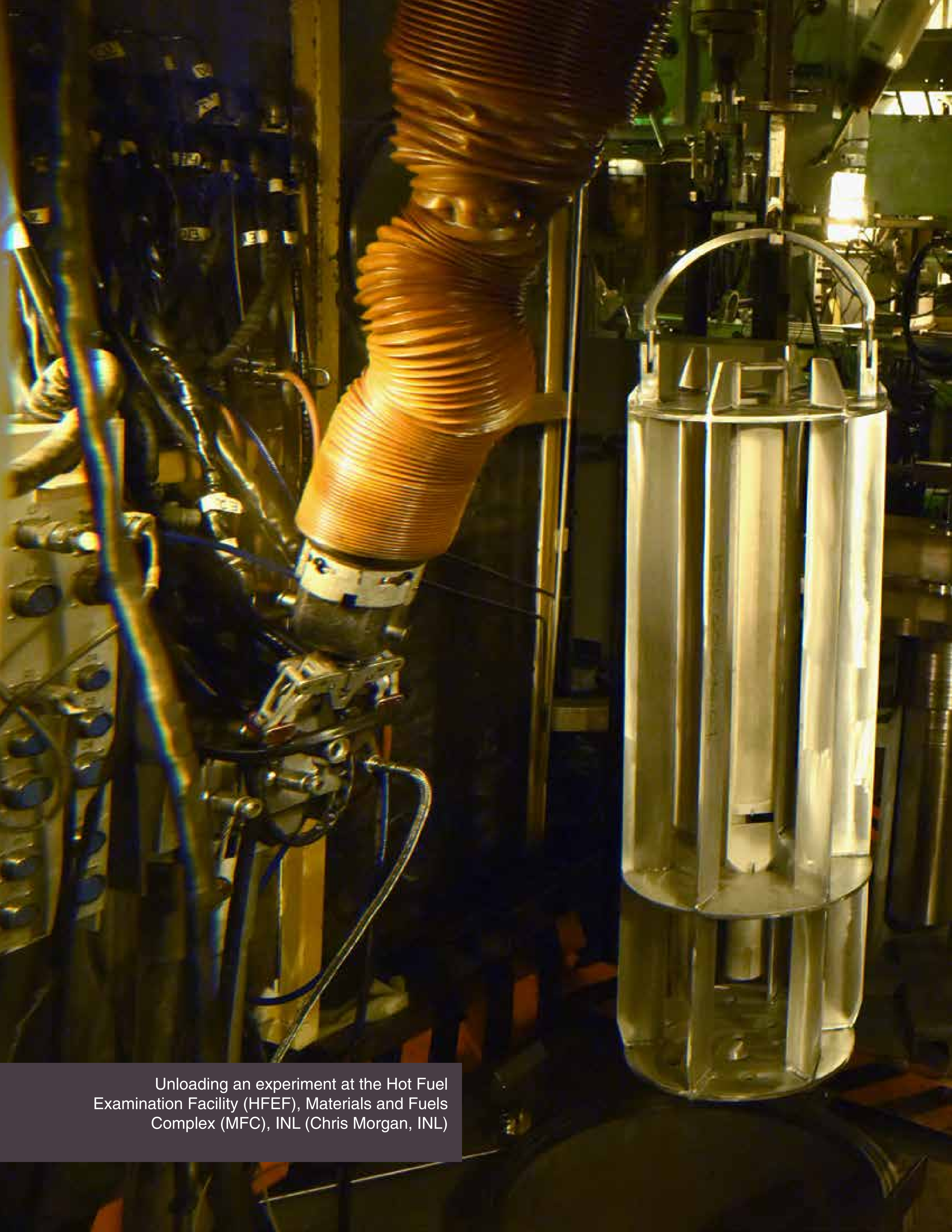
2017
Annual Report
Nuclear Science User Facilities



Unloading an experiment at the Hot Fuel Examination Facility (HFEF), Materials and Fuels Complex (MFC), INL (Chris Morgan, INL)

Nuclear Science User Facilities
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nsuf.inl.gov

INL/EXT-_ _-_ _ _ _

Prepared for the U.S. Department of Energy,
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(This report covers the period beginning October 1, 2016, through September 30, 2017)

Cover Image: A model of the Advanced Test Reactor (ATR) produced by advanced simulation capabilities at Idaho National Laboratory (INL).

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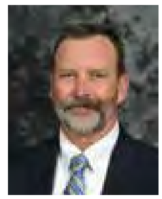

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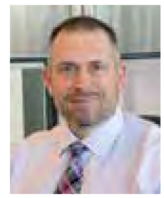

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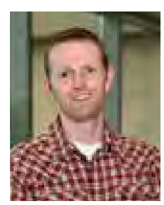

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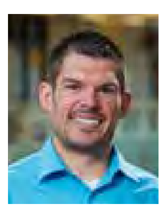

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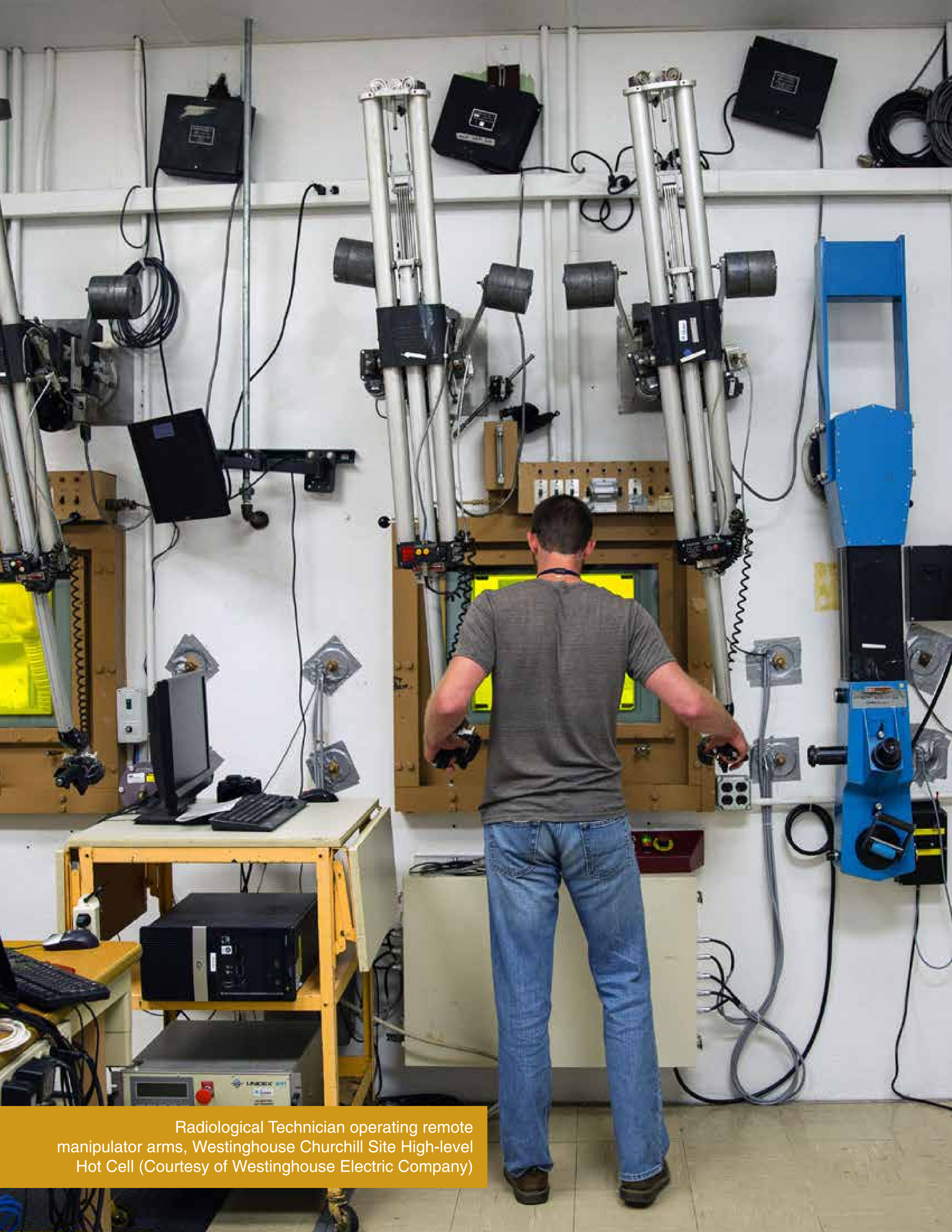

Radiological Technician operating remote manipulator arms, Westinghouse Churchill Site High-level Hot Cell (Courtesy of Westinghouse Electric Company)

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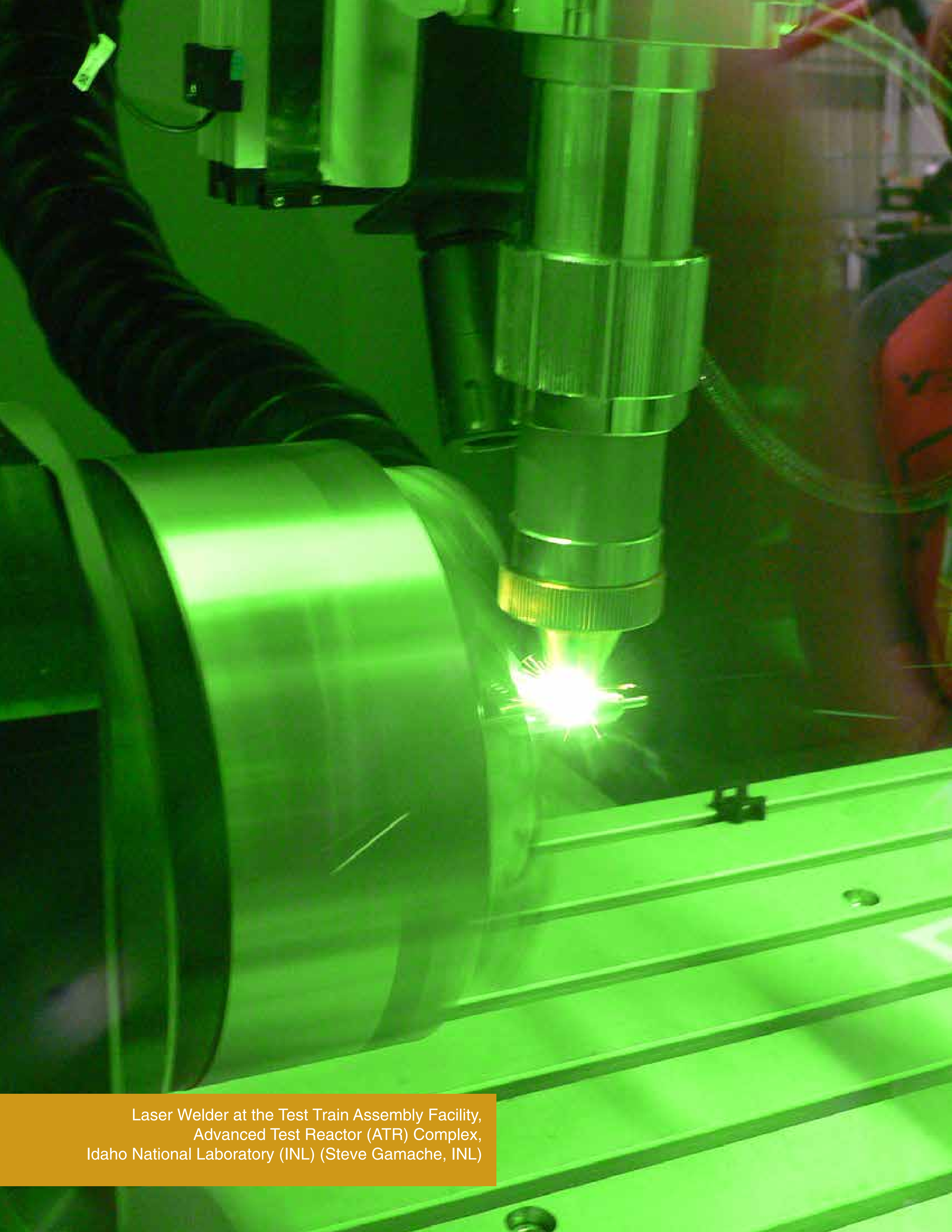

Laser Welder at the Test Train Assembly Facility,
Advanced Test Reactor (ATR) Complex,
Idaho National Laboratory (INL) (Steve Gamache, INL)

FROM THE NSUF DIRECTOR

J. Rory Kennedy
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The Nuclear Science User Facilities (NSUF) celebrated our 10th anniversary in 2017, a year that also marked the 50th anniversary of the Advanced Test Reactor (ATR), the founding facility of the NSUF.

Fiscal year (FY) 2017 was another exceptional year for the NSUF in a number of areas. The demand for NSUF award opportunities continued to expand. The Consolidated Innovative Nuclear Research (CINR) Funding Opportunity Announcement (FOA) solicitation results saw, once again, the NSUF achieving record numbers: 124 Letters of Intent (LOIs) leading to 108 preproposal submissions from which 50 full proposals were invited. $11 million in direct project funding allowed us to award 15 projects ranging in cost from $60,000 to $3.6 million. Five of the 15 awards went to industry leads, five went to university leads, and five went to national laboratory leads. The NSUF also saw a record number of applications and awards for the FY 2017 Rapid Turnaround Experiments (RTEs). A total of 180 RTEs proposals were received from 48 institutions in FY 2017 (a 140 percent increase from FY 2016), from which 92 projects were awarded (a 136 percent increase from FY 2016).

Seven new partner facilities were accepted into the NSUF. The parent institutions include Brookhaven National Laboratory, Lawrence Livermore National Laboratory, Los Alamos National Laboratory, Sandia National Laboratories, The Ohio State University, University of Florida, and Texas A&M University. The NSUF has now evolved to include 20 partner facilities with each facility bringing exceptional capabilities to the relationship, including: reactors, beamlines, instruments, hot cells, and importantly, technical and scientific expertise with the capabilities that are so critical to obtain quality results.

The NSUF also added our first international affiliate, Belgium's Studiecentrum voor Kernenergie/Centre d'Etude de l'Energie Nucléaire (SCK•CEN) Belgian Nuclear Research Centre that houses the Belgium Reactor-2 (BR-2) and associated Laboratory for High and Medium Activity (LHMA). Department of Energy (DOE) and SCK•CEN signed a Memorandum of Understanding (MOU) concerning cooperation in nuclear energy research and development in January 2017. This MOU established the basis for in-kind collaboration on projects of mutual interest, employing the BR-2 and LHMA together with the ATR, the Transient Reactor Test (TREAT) Facility, and associated facilities located at Idaho National Laboratory (INL), as well as other facilities that are part of the NSUF.

The steady growth in the number of NSUF partners led to the NSUF hosting the first NSUF Partner Facilities Working Group meeting, May 24-25, 2017, at the Center for Advanced Energy Studies (CAES) in Idaho Falls, Idaho. The working group meeting was intended to provide the partner facilities with an avenue to self-organize in order to increase their involvement in programmatic activities and to establish a baseline for evaluating shared interests and concerns among the participating institutions. The meeting was well-attended and quite educational with respect to the partner facilities gaining a better understanding of the breadth of capabilities and activities of the NSUF.

We welcomed three new NSUF team members in FY 2017. After two years of searching, our chief post-irradiation scientist position was finally filled by Simon Pimblott, Ph.D. Simon comes to us from the United Kingdom, where he was Chaired Professor of Radiation Chemistry at Manchester University and founding director of the Dalton Cumbrian Facility. Simon was already quite familiar with the NSUF, having served as the international member of the NSUF Science Review Board since 2012. One of his tasks in FY 2018 will be to measure the overall research impact of the NSUF over the last 10 years.

With the steady addition of new partner facilities and demand for NSUF funding opportunities, we foresaw the need for an additional planning and financial control specialist. Travis Howell now fulfills this essential role for the NSUF. His work experience includes three years at an engineering, fabrication, and manufacturing company managing projects constructing components for the commercial nuclear sector. He assists NSUF managers in making strategic operational decisions in an efficient and productive manner.

Laura Scheele joined the NSUF as communications liaison, replacing Sarah Robertson, who moved to another position. We will miss Sarah but know our communications are in good hands with Laura. She previously handled media relations for INL and has worked for the American Nuclear Society in a communications and public policy role. Please feel free to contact her with any questions about the NSUF and plan to see her at several of our conference exhibits.

The NSUF has also increased dedicated technical resources to support the ever-growing demand for projects. Keith Jewell, Ph.D., has transitioned into a full-time technical lead, and the NSUF has secured the expertise of Nick Meacham and Katie Anderson as full time experiment managers. Tom Maddock will remain a part-time technical lead (international). The NSUF continues to seek technical leads to partner with principal investigators (PIs) to plan and execute experiments to ensure that research projects proceed smoothly and according to schedule. It is important to note that the NSUF technical leads are scientific professionals, and I thank our supported PIs for recognizing this by having the technical leads become scientifically and technically involved in the projects NSUF supports.

The NSUF furthered our efforts in our charge from DOE-NE to provide information to best manage infrastructure when we teamed with the Gateway for Accelerated Innovation in Nuclear (GAIN) and co-hosted a thermal hydraulics workshop in July 2017. The workshop objective was to develop a ranked list of thermal-hydraulic research and development needs in the reactor technology areas of light water reactors, fast reactors, high-temperature gas reactors, and molten salt reactors. The workshop was well-attended with approximately 70 participants from national laboratories, universities, industry and foreign nuclear organizations, as well as Department of Energy Office of Nuclear Energy (DOE-NE) and Department of Energy Idaho Operations Office (DOE-ID). The NSUF's industry programs will continue to evolve as we ensure that proposals focus on priority areas to support today's reactor fleet and tomorrow's energy systems. The NSUF will remain a key contributor to and supporter of the GAIN initiative in its charge to aid industry in accelerating innovation. Through our solicitation processes, the NSUF offers the opportunity to address and solve targeted issues of importance to the nuclear industry.

In a first-of-its-kind endeavor, the NSUF brought together experts and stakeholders in the area of ion beam irradiations to prepare a document addressing the application of ion beam technologies to advancing nuclear energy. The work is titled, "Roadmap for the Application of Ion Beam Technologies to Challenges for the Advancement and Implementation of Nuclear Energy Technologies," and should be available to the public in early FY 2018. Again, this is the first such road map addressing the issue that we are aware of and we expect it to have a significant impact on the field.

10TH ANNIVERSARY

Celebrating our past
Inspiring our future

2007 – 2017

NSUF Nuclear Science User Facilities

Gamma Irradiation Test Loop, Fuels and Applied Science Building (FASB), MFC, INL (Chris Morgan, INL)

Building upon the continued success and growth of the Nuclear Fuels and Materials Library (NFML) and the Nuclear Energy Infrastructure Database (NEID) – and the goal to provide a completely integrated web-based suite of research tools – the NSUF established the Combined Materials Experiment Toolkit (CoMET). It will eventually not only link the NFML and NEID, but also databases of scientific and technological expertise and project knowledge, including references and links, where appropriate, to publications and reports that contain the information and knowledge gained from those projects.

The NSUF maintained an active presence at scientific conferences and meetings. By expanding awareness of the opportunities and capabilities provided by DOE-NE through the NSUF, we increased the impact and recognition of NSUF-awarded research. Now reaping the benefits of 10 years of NSUF-supported research, the American Nuclear Society (ANS) Materials Science and Technology Division organized, together with the NSUF, three NSUF sessions at the 2017 ANS Annual Meeting, during which 18 NSUF-supported research papers were presented. The NSUF exhibited and was invited to present at several conferences and meetings in the U.S. (See Table 1)

Table 1.

NSUF Conference and Meetings Engagement: FY 2017
NSUF Exhibits
Nuclear Materials Conference (NuMat)
American Nuclear Society (ANS) Winter Meeting
Materials Research Society Winter Meeting
ANS Student Conference
The Minerals, Metals and Materials Society (TMS) Annual Meeting
Environmental Degradation Conference
NSUF Invited Presentations
Advanced Sensors and Instrumentation Program review
GE Hitachi Advanced Manufacturing Works
EPRI Primary Systems Corrosion Technical Advisory Committee
Accelerator Applications in Research and Industry Conference
Toshiba/CRIEPI "U-Free" TRU Burner Concepts meeting
China National Nuclear Corporation (CNNC) Nuclear Power Institute of China (NPIC) meeting
U.S. Nuclear Regulatory Commission (NRC) Materials Harvesting Workshop
Montana State University
4th Technical Meeting of the Advanced Reactor Research and Development, Fuel Cycle Research and Development and Waste Management, and Light-Water Reactor Research and Development Sub-Working Groups of the Civil Nuclear Energy Research and Development Working Group (CNWG)
INL/ORNL ICERR Assessment
MeV Summer School
DOE-NE Cross-cut Coordination meeting
National Organization of Test, Research, and Training Reactors (TRTR) Conference
Utilities Service Alliance

"As we move into FY 2018, the NSUF will continue to build upon its foundational success."

International interest in the structure and work of the NSUF is growing. Our unique distributed partnership model, as well as our NFML and NEID, are increasingly being considered by other countries. The NSUF was invited to present at the Nuclear Materials Conference in France, the Hot Lab Conference in Japan, the Global Conference in South Korea, and the Nuclear Academics Discussion Meeting (NADM) in England. In addition to representing the NSUF, these events provide the opportunity to assess international capabilities and resources that may be of interest to the NSUF community.

As we move into FY 2018, the NSUF will continue to build upon its foundational success. We must continue to invest in the capabilities, resources, and scientific expertise needed to support the anticipated growth in CINR and RTE projects. We will continue to enhance and streamline the NEID and NFML and to expand them into CoMET to provide seamless information management for researchers.

High Performance Computing (HPC) is a valuable offering available through the CINR and RTE calls. The NSUF is making available approximately 30 percent of computing time on INL's Falcon, an SGI ICE-X distributed memory system with 34,992 cores, 121 terabytes of memory and a LINPACK rating of 1,087 teraflops. HPC supports a wide range of research activities, including performance of materials in harsh environments (including the effects of irradiation and high temperatures) and multiscale multiphysics analysis of nuclear fuel performance. I strongly encourage our users and the nuclear community to take advantage of this capability.

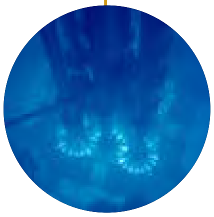
Please take a few moments to read through this report and familiarize yourself with our organization, our research, and the opportunities available through the NSUF. We put a good amount of effort into our annual reports to keep our users informed, and take great pride in them. As a note of recognition, the NSUF 2015 Annual Report received an Award of Distinction in the 2017 Communicator Competition, as judged by the Academy of Interactive and Visual Arts. I thank the NSUF staff, partners, users, and both DOE-NE and DOE-ID for their hard work to make the NSUF a successful strategic research asset.

J. Rory Kennedy

Bryan Forsmann, Center for Advanced Energy Studies (CAES), preparing samples in the Materials Laboratory, CAES (Chris Morgan, INL)

NSUF TIMELINE

2007



The NSUF (INL) established as DOE-NE's first user facility – Neutron Irradiation, Hot Cells, Gamma Irradiation, Low Activity Laboratories

2008

MIT joins the NSUF – Neutron Irradiation

2009

CAES joins the NSUF – Low Activity Laboratories

Illinois Institute of Technology joins the NSUF – Beamline (x-ray)

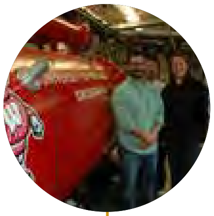
North Carolina State University joins the NSUF – Beamline (positron), Neutron Irradiation

University of Michigan joins the NSUF – Beamline (ion), Hot Cells

University of Nevada, Las Vegas joins the NSUF – Low Activity Laboratories

Purdue University joins the NSUF – Beamline (ion)

University of Wisconsin joins the NSUF – Beamline (ion), Low Activity Laboratories



Oak Ridge National Laboratory joins the NSUF – Neutron Irradiation, Gamma Irradiation, Hot Cells, Low Activity Laboratories

University of California, Berkeley joins the NSUF – Low Activity Laboratories

Pacific Northwest National Laboratory joins the NSUF – Hot Cells, Low Activity Laboratories

2010

2011

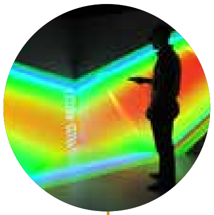
2012

Sandia National Laboratories joins the NSUF – Gamma Irradiation, Neutron Irradiation, Beamline (ion)

Westinghouse joins the NSUF – Hot Cells, Low Activity Laboratories

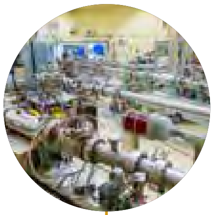
2013

INL offers High Performance Computing capabilities



2015

Argonne National Laboratory joins the NSUF – Beamline (ion)



2016

Brookhaven National Laboratory joins the NSUF – Beamline (x-ray)

2017

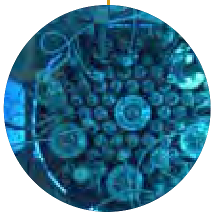
Lawrence Livermore National Laboratory joins the NSUF – Beamline (ion)

Los Alamos National Laboratory joins the NSUF – Beamline (neutron), Hot Cells, Low Activity Laboratories

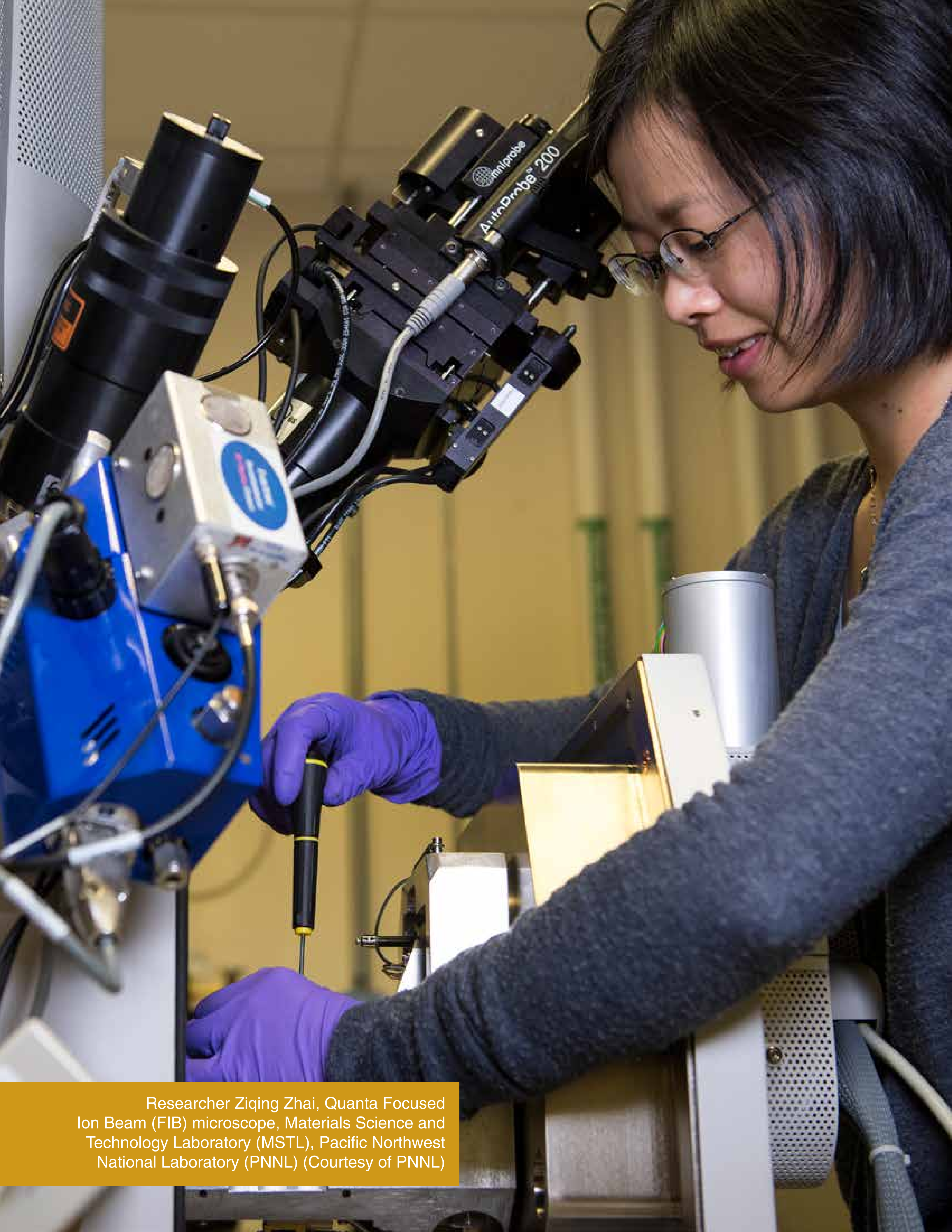
University of Florida joins the NSUF – Low Activity Laboratories

The Ohio State University joins the NSUF – Neutron Irradiation

Texas A&M joins the NSUF – Beamline (ion)



SCK•CEN joins the NSUF as the first international affiliate – Neutron Irradiation, Hot Cells, Low Activity Laboratories



Researcher Ziqing Zhai, Quanta Focused Ion Beam (FIB) microscope, Materials Science and Technology Laboratory (MSTL), Pacific Northwest National Laboratory (PNNL) (Courtesy of PNNL)

DOE-ID PROGRAM MANAGER RETIRES

Brooks Weingartner reflects on his time overseeing the NSUF

"Verify and validate" were the key words for Brooks Weingartner in the time he was the U.S. Department of Energy's Idaho Operations Office point man for contractual oversight of programs including the Nuclear Science User Facilities (NSUF).

Weingartner retired from his position at the end of 2017, looking forward to returning to environmental engineering, his original field of expertise. He received a bachelor's in geological engineering from Montana Tech of the University of Montana in 1988 and a master's in environmental engineering there in 1992.

He took his DOE job as NSUF program manager in 2013, about the same time as NSUF Director Rory Kennedy came in to oversee the NSUF. Weingartner's responsibilities included helping to put together work packages and keeping track of milestones. The time he spent as DOE-Idaho's program manager gave him some special insight.

"Rory has assembled a first-rate team and they are doing first-rate work," he said. The most gratifying aspect of the job was "seeing the breadth of work across the nation and how it supported DOE-NE's mission."

Established at Idaho National Laboratory in 2007, the NSUF expanded to partner facilities outside INL the following year and has continued to do so into 2018. If there is one particular challenge for the NSUF, it is balancing the resources with the increasing interest in and demand for new funding awards to facilitate research.

"As the NSUF has acquired new partner facilities to expand the capabilities available to researchers, the pie has stayed the same size," said Weingartner. "Even though the funding has stayed flat, the number of proposals has been growing. That's good news in terms of the high demand for NSUF capabilities, but it also requires a strategic approach to addressing research gaps."

"Rory has assembled a first-rate team and they are doing first-rate work."

FOCUS ON NEW STAFF

Simon Pimblott

NSUF Chief Post-Irradiation Scientist

Interested in the future of research priorities? Spend a few minutes with Simon Pimblott, the NSUF Chief Post-Irradiation Scientist and INL Directorate Fellow, who joined the NSUF in September 2017. His involvement with the NSUF began about five years earlier, in 2012, when then-NSUF Director Todd Allen recruited him as the international member of the NSUF Science Review Board.

The NSUF Science Review Board provides independent overview and input to the NSUF on the broad range of NSUF programs and initiatives to provide access to nuclear energy research capabilities, to generate impactful results and to maintain and enhance the infrastructure, capabilities and expertise available in the United States. An international perspective on the Science Review Board is essential to maintaining the NSUF's status as DOE-NE's world-class user facilities organization.

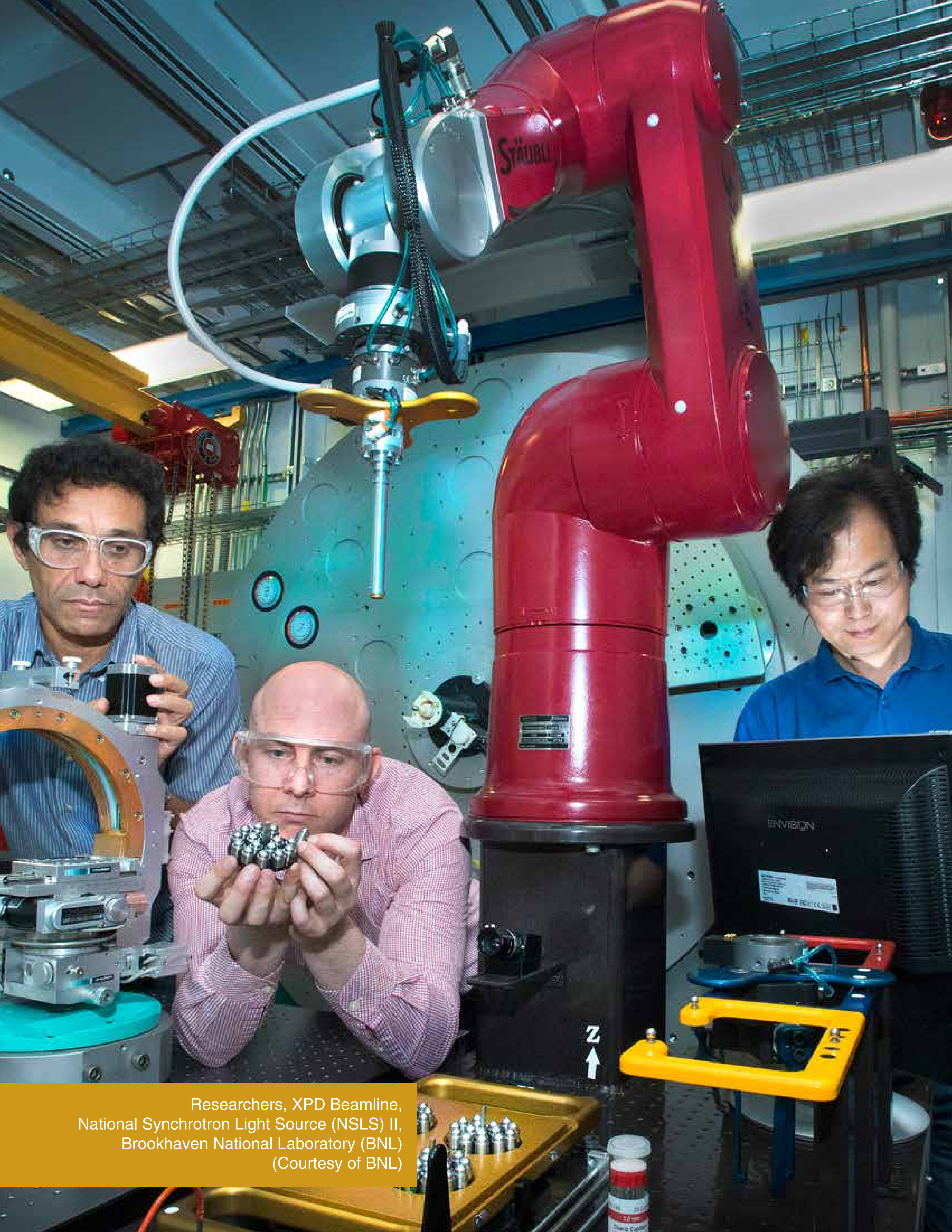
"The United States has to understand how the international community prioritizes, maintains and uses nuclear energy research capabilities," said Pimblott. "Getting international feedback helps DOE-NE and the NSUF drive strategic decisions on research infrastructure priorities."

Pimblott was well-suited to provide an international perspective. He hails from Derby, Great Britain. His interest in nuclear energy was sparked when he received a scholarship to St. Peter's College at the University of Oxford from the U.K. Central Electricity Generation Board.

"The scholarship required me to work on CEGB projects prior to going to university and during the summer vacations," explains Pimblott. "My first project was to study SOX and NOX production by fossil fuels and their contributions to acid rain. My introduction to nuclear was on a project at Berkeley Nuclear Laboratories, on the use of low oxidation state metal ions for the decontamination of steam generator pipework. This work provided me with technical insight into the benefits of generation that doesn't rely upon combustion, with fission generation being the most robust."

Pimblott pursued his doctorate (D.Phil.) in the Physical Chemistry Laboratory at the University of Oxford and at Harwell with a scholarship from the U.K. Atomic Energy Authority. Following his D.Phil.,

Ph.D. students Bill Chuirazzi (left) and
Ibrahim Oksuz (right), Nuclear Reactor Laboratory,
The Ohio State University Research Reactor (OSURR),
The Ohio State University
(Courtesy of The Ohio State University)



Researchers, XPD Beamline,
National Synchrotron Light Source (NSLS) II,
Brookhaven National Laboratory (BNL)
(Courtesy of BNL)

Pimblott joined the Radiation Laboratory and later the Department of Physics at the University of Notre Dame. Among his motivations for making the move? "I wanted to see the United States," he said.

He not only saw the United States, but he met Nancy "Hedge" Harridge, his future wife, while attending a radiation research conference in Nashville, Tennessee. Pimblott says he and Hedge have been married too many years to count. Their family has grown to include two daughters Robyn and Erin, plus two sons-in-law and five grandchildren who live in Ohio and Tennessee.

Always ready for challenge, Pimblott left Notre Dame in 2006 to establish the Dalton Cumbrian Facility for The University of Manchester and the U.K. Nuclear Decommissioning Authority. The laboratory initially focused on radiation effects on materials and robotics for use in nuclear facilities and grew under Pimblott's leadership to include work in fuel cycle, water chemistry, waste packaging, nuclear fuel performance and reprocessing chemistry.

Malcom Gladwell, in his book "Outliers," popularized the notion that achievement is talent plus preparation. The concept is based on psychology research that indicated that role preparation plays a bigger part than innate talent in the careers of the gifted. In cognitively demanding fields, 10,000 hours (or 10 years), seems to be a rule of thumb for the amount of time necessary to excel at complex tasks.

Pimblott takes this notion to heart. "I spent eleven years building Dalton Cumbria into a multidisciplinary and financially solvent laboratory," said Pimblott. "The timing was right for me to move into a new position with new opportunities and challenges that match my expertise." The NSUF offered a perfect fit.

He has seen the NSUF experience dramatic growth over the past 10 years. "The NSUF offers a broader range of capabilities. Now the NSUF is way beyond reactor experiments and is active in simulation, analysis and the interrogation of irradiated materials," said Pimblott. "The NSUF today provides world-leading science and engineering research capabilities. The key is to strategically balance NSUF capabilities to fulfill emerging research needs."

"The NSUF today provides world-leading science and engineering research capabilities. The key is to strategically balance NSUF capabilities to fulfill emerging research needs."

Travis Howell

Minding the Numbers

Behind every experiment is someone whose job it is to keep track of the money. Even before the addition of six new partner facilities in 2017, directors of the Nuclear Science User Facilities (NSUF) knew they were going to need an extra planning and financial control specialist (PFC).

Travis Howell, a native of Blackfoot, Idaho, was brought into the NSUF fold at the beginning of 2017 to help handle the planning and finances for a rising number of experiments. Howell earned his MBA with an emphasis in finance from Idaho State University in 2013. His work experience includes three years at an engineering, fabrication and manufacturing company, where he managed projects involving the construction of components for the commercial nuclear sector. Howell then spent one year doing cost analysis and accounting for a health and wellness company. Both of these positions cultivated proficiencies required in his current role at INL within the NSUF.

Howell's focus is on the numbers, not the engineering. In the planning and setting up of any experiment, milestones are scheduled. As milestones are met, Howell's job is to communicate with experiment managers to provide planning and controls support to assigned projects. Howell provides a wealth of services, including supporting the development, implementation, analysis, and monitoring of scope, schedule, budget and cost; and ensuring cost plus commitments do not exceed approved funding ceilings. PFCs also assist with proposal preparation and development of cost estimates; preparing monthly performance reports and confirming that the actual cost is appropriate and correct for designated work scope; and implementing project closeout procedures.

Keyou Mao, Purdue University, using the Transmission Electron Microscope, Microscopy and Characterization Suite, CAES

Loading a non-fueled test,
Laser Welder, TTAF, ATR Complex, INL
(Steve Gamache, INL)

Communication with work scope managers and other business management staff is essential to success. There must be a foundation of trust and respect which enables work scope managers and PFCs to work in tandem using individual aptitude and training to aid project achievement. His work also requires knowledge and use of a wide array of business systems. These applications assemble a large amount of information and help PFCs organize it into a manner that can assist managers in making strategic, prudent and operational decisions in an efficient and productive manner.

Working at the NSUF has been a revelation for Howell. "I enjoy the people," he said. "The NSUF has a great team of individuals that are very passionate about what they do. They are customer focused and results driven. They really want to make a difference in our world's energy future. Working with people like that is contagious. The NSUF is a great program and I feel extremely fortunate to be a part of it."

Growing up in eastern Idaho, Howell says he'd always been aware of Idaho National Laboratory and the fact that nuclear research was conducted there. But seeing the projects and meeting the people has given him a deeper understanding. "I didn't grasp the magnitude and importance of INL's research and mission until I had the opportunity to witness the focus and drive of world class scientists, researchers, managers and support staff with a common goal," he explained.

"The NSUF has a great team of individuals that are very passionate about what they do. They are customer focused and results driven. They really want to make a difference in our world's energy future."

Laura Scheele

Getting the Word Out

While she has had an appreciation for nuclear energy since she was getting her master's, it's only been the past year that Laura Scheele has taken a deep dive into to the workings of how the wheels turn at the most basic level.

Scheele became communications lead for Nuclear Science User Facilities (NSUF) in January 2017. The previous two years she had been media relations lead for Idaho National Laboratory. In that job, the focus was wider, on getting word out to the world about the work going on at the lab. At the NSUF, she has learned a lot more about how nuclear energy research happens and how the U.S. Department of Energy supports it.

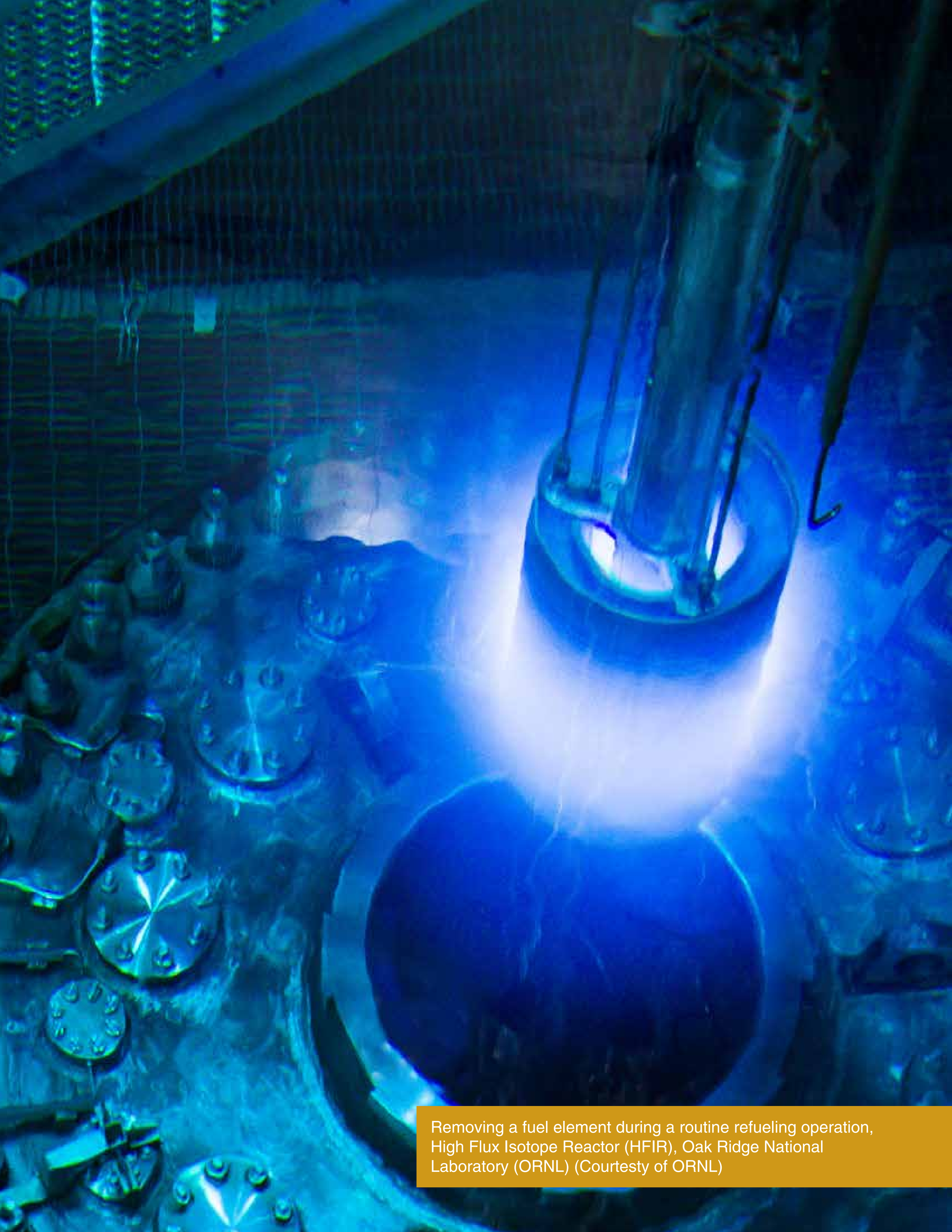
"I went from a high level appreciation of it to a more nuts-and-bolts understanding of what goes on in the industry," she said.

She has become more adept at web layout. "You have to get the news up fast," she said. "It can get very busy when you've got three calls for rapid turnaround experiments each year, as well as major research and infrastructure projects."

A native of Indiana, part of the greater Chicago area, Scheele earned her bachelor's in political science from Vassar College. She then studied public and environmental policy at the University of Colorado. She was attracted to nuclear energy because of "the elegance of the fission process" and the benefits of a baseload energy source that didn't put massive amounts of carbon dioxide into the atmosphere.

From 2008 to 2012 she was communications and policy manager for the American Nuclear Society, managing media relations and developing strategic messaging, especially in the rapidly emerging world of social media. She moved to Richland, Washington, in 2013 to become senior public affairs analyst and external relationship manager for Energy Northwest, a 27-member public power consortium.

In the course of her career, Scheele said she has seen a shift in the prospects for nuclear energy. "I think it has changed a lot as millennials come into their own. Younger people, they see climate change and the threat it poses, and they're more open to technological solutions like nuclear energy. You see a lot of interest and excitement about new innovations, and the NSUF supports a lot of the research that will get us there."



Removing a fuel element during a routine refueling operation, High Flux Isotope Reactor (HFIR), Oak Ridge National Laboratory (ORNL) (Courtesty of ORNL)

Megan Isabelle, KaeCee Holden, and Kelly Verner, NSUF Interns, CAES (Chris Morgan, INL)

NSUF SUMMER INTERNS

NSUF interns power summer research work

When they began their 2017 Nuclear Science User Facilities (NSUF) summer internships, KaeCee Holden, Megan Isabelle and Kelley Verner were somewhat surprised to find themselves without male colleagues in their select group.

At school – the University of Idaho (UI) for Holden and Verner, North Carolina (NC) State for Isabelle – they'd been in the minority. While not dwelling on this – they had work to do, after all – they inadvertently hit on an issue the nuclear industry has noticed and is seeking to address.

"One of the most important things we can do is to encourage outstanding women scientists and engineers to enter and to remain in our field," said Richard Lester, head of Massachusetts Institute of Technology (MIT's) Nuclear Science & Engineering Department, at a 2015 symposium.

The enthusiasm Holden, Isabelle and Verner have shown for their chosen field ought to be cause for hope if not celebration, and the support they've gotten from their NSUF mentor, Brenden Heidrich, has greatly reinforced their determination to pursue careers in nuclear energy.

"I believe nuclear power will be an essential component in the future energy production of the United States and the world," said Holden, who grew up in central Kansas. She became fascinated with all things nuclear during her sophomore year chemistry class in high school. She earned her bachelor's in applied physics, with an emphasis in nuclear science and a minor in astronomy, from Brigham Young University in 2016, and is now working on a master's in nuclear engineering at University of Idaho – Idaho Falls.

Isabelle got interested in nuclear research in high school when she was in NASA's Virginia Aerospace Science and Technology Scholars program. They planned, designed and evaluated a manned mission to Mars. "With any manned deep space mission, radiation is of the highest concern, which is how I found myself studying nuclear engineering," she said. Her fascination grew at NC State University, where she became interested in someday participating in experiments involving the PULSTAR reactor's core.

"It just gets cooler and cooler," she said.

For Verner, the internship this summer has been a natural extension of the work she has been doing at the Center for Advanced Energy Studies, where the NSUF has its offices. A native of Idaho Falls with a master's in

I believe nuclear power will be an essential component in the future energy production of the United States and the world."

— Kaecee Holden, University of Idaho and NSUF Summer Intern

biological engineering, she is now pursuing a doctorate in nuclear engineering from UI. She has also become a key member of Students for Nuclear (http://www.students4nuclear.org/), a nationwide advocacy group.

All three interns' work has involved computer simulations. Verner focused on a neutron damage calculator aimed at helping researchers determine which NSUF research reactor is best suited to the kind of research they want to do. This involves modeling the reactor, calculating the amount of damage incurred by a number of material options in different experimental reactor positions, and building a tool that will be on the NSUF website for users to access.

Holden's project has been a radioactivity calculator to estimate the radioactivity levels of materials post-irradiation. "Materials with short half-lives are able to decay prior to the sample being removed from the reactor. Materials with longer half-lives must be handled with caution and often stored until their radioactivity levels are low enough for examination," she said. "The purpose of my project was to design a radioactivity calculator, which will estimate the radioactivity levels of materials post-irradiation. The calculator is intended to be an easily accessible tool for NSUF researchers during the conceptual design phase of their experiments."

Making it easier to estimate the radioactivity of a sample before it is ever irradiated will have three benefits, she said: increased worker safety awareness, improved efficiency by planning the examination work at the appropriate facility, and information that will allow researchers to plan project timelines efficiently due to a better understanding of required decay time.

Both Holden and Verner say the NSUF's material library and research database has been essential to their work. "They have access to many of the reactor simulation models," said Verner.

Isabelle said what the NSUF has made available to her relates directly to the peripheral experiments she did at NC State. "My objective was to analyze whether experimental flexibility could be improved by increasing or changing the locations of sample irradiation tubes," she said. The availability of a central irradiation experiment location, within the NC State reactor has shown a threefold increase in neutron flux exposure for experiment samples. This should be a benefit to PULSTAR, especially where it relates to the effects of increased fuel enrichment.

"Interns always have new ideas and new ways of looking at things," Heidrich said. "I like to think I'm flexible, but I've been at this awhile. I have to rethink a lot of things when I have to re-explain them. Sometimes, we get results we don't expect."

He credited all three as "total self-starters," which is a benefit for intern research projects. Isabelle's research ought to be invaluable to NC State and the PULSTAR. "All three projects involved developing tools to help researchers design better experiments, which is what the NSUF is all about," said Heidrich. Verner and Holden, who are staying in Idaho Falls to pursue their degrees, will have time to finish up their NSUF projects.

As for having three women for protégés, Heidrich said it was "just kind of how it fell out. There were a lot of applicants. These three were the best fit for the NSUF."

Kelly Verner, NSUF Intern, and J. Rory Kennedy, NSUF Director, at the Summer 2017 INL Intern Poster Session. (Chris Morgan, INL)

FOCUS ON RESEARCH

Dr. Peter Hosemann

Dr. Peter Hosemann values exchange of capabilities, ideas through the NSUF

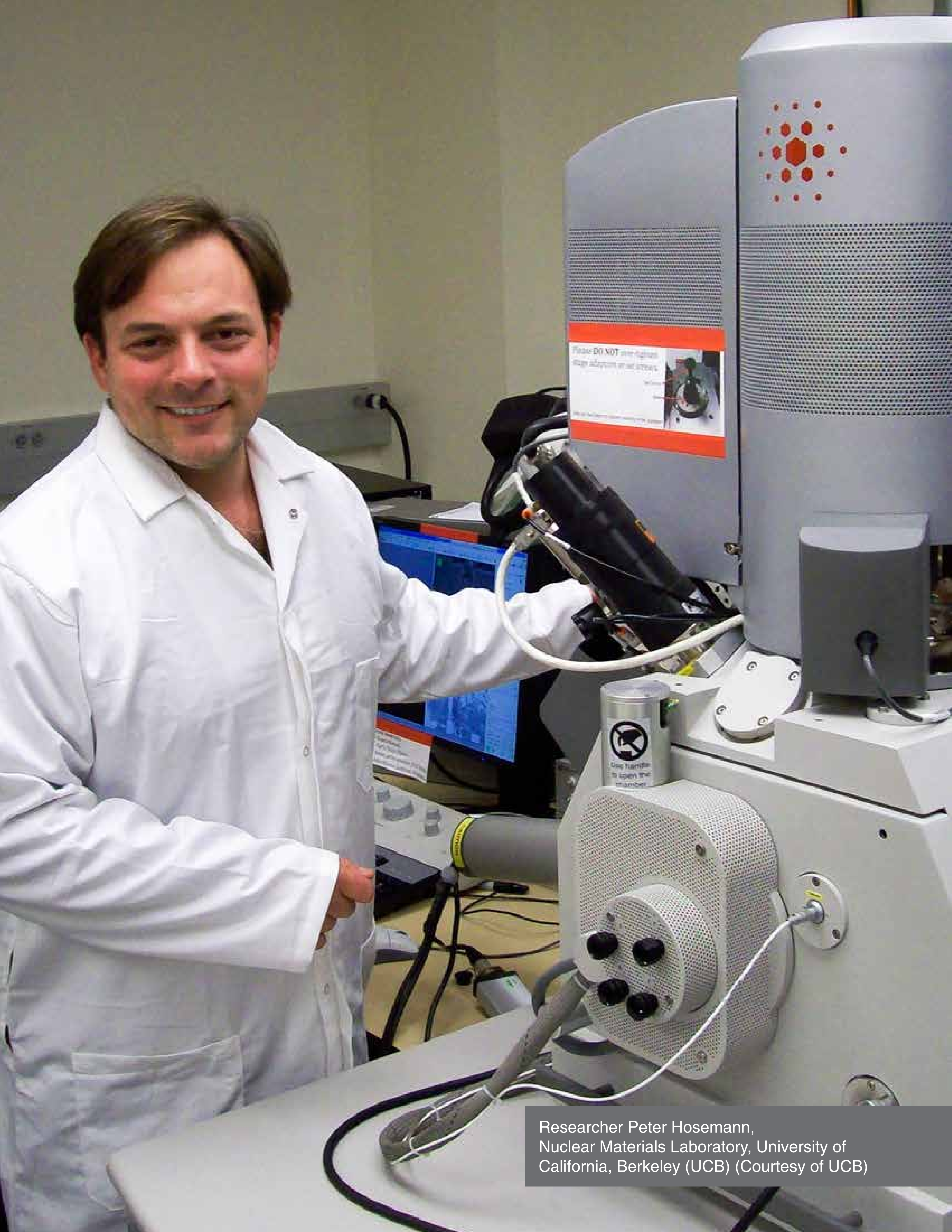
Dr. Peter Hosemann of the University of California, Berkeley Nuclear Engineering (UCBNE) is the latest chairman of the Nuclear Science User Facilities (NSUF) Users Organization. Elected in November 2017, he acts as liaison between NSUF users and facilities and NSUF management. Part of the NSUF partnership since 2011, UCBNE assists nuclear material scientists by making the institution's materials equipment available to interested researchers.

Hosemann's enthusiasm for the benefits of the NSUF drove his interest in the Users Organization. "The NSUF is a genius idea," he said. "By connecting new researchers with new ideas with the necessary partner facilities, the NSUF also raises intellectual nuclear science capital across the research community."

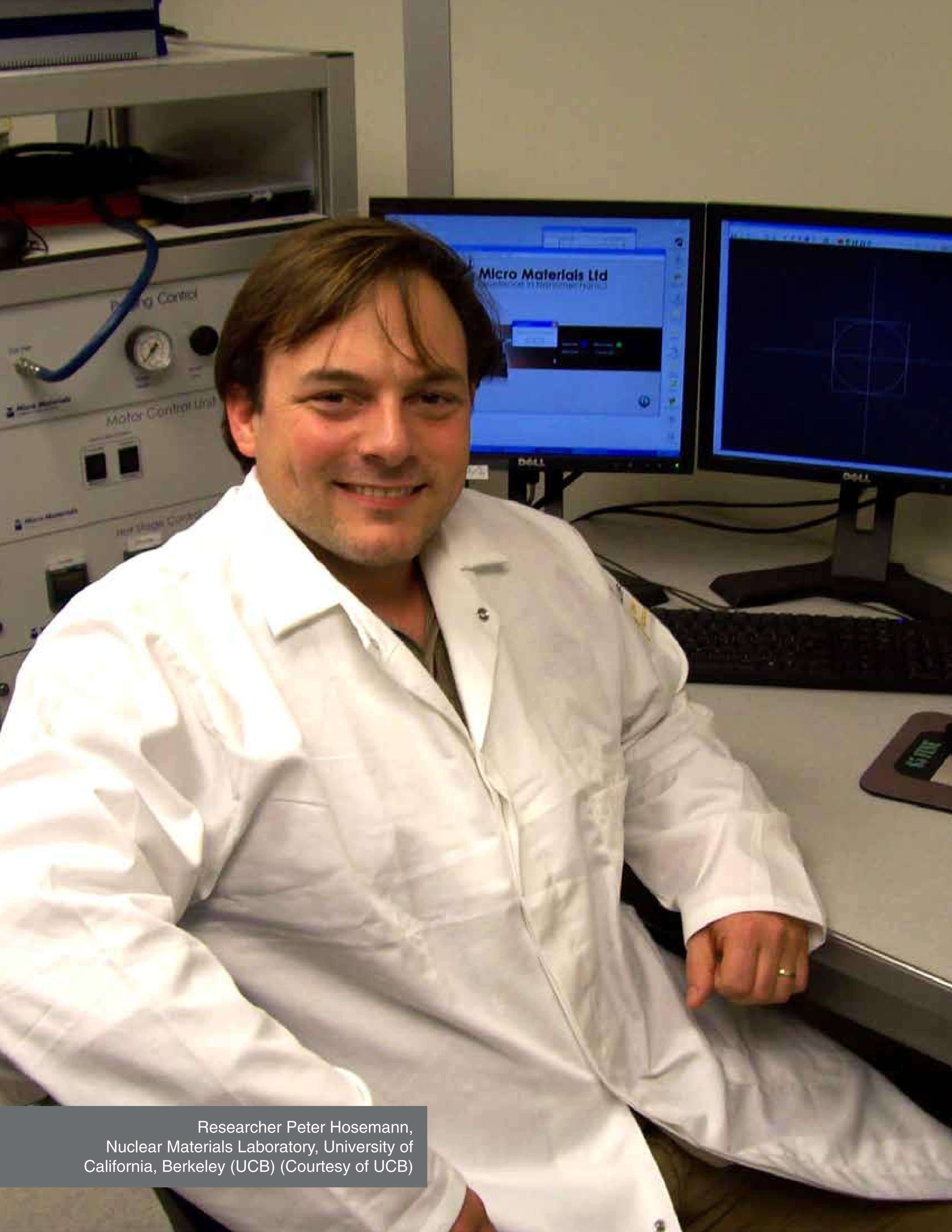
Hosemann joined the Berkeley faculty in 2010 as assistant professor and was promoted to associate professor in 2014. He is department co-chair, head graduate adviser, and UC Berkeley's radiation safety committee chair. He received his doctorate in material science from the Montanuniversität Leoben, Austria, in 2008, while conducting research on ion beam irradiations and microscale mechanical testing as well as liquid metal corrosion at Los Alamos National Laboratory (LANL).

Hosemann's interest in materials and radiation began as an undergraduate in Switzerland and got into full swing in 2003, when he spent a summer studying at LANL. In the Materials Science in Radiation and Dynamics Extremes group, he developed an interest in how materials respond in extreme environments. "Radiation adds essentially another dimension, another axis on your diagram," he said. "You tackle problems that don't exist anywhere else. It was different and more interesting than anything I'd ever encountered."

He returned to LANL as a graduate student in 2005 – a lot of people at LANL call New Mexico the "Land of Entrapment," he joked – and became a postdoc in 2008. "The variety of expertise available was considerable," he said. "You could approach almost anyone for help and advice and they'd be willing to give it. I also appreciated the fact that my ideas were treated as equal to anyone else's. Being young or a student didn't matter as long as the idea and science was sound."



Researcher Peter Hosemann,
Nuclear Materials Laboratory, University of California, Berkeley (UCB) (Courtesy of UCB)



Researcher Peter Hosemann,
Nuclear Materials Laboratory, University of
California, Berkeley (UCB) (Courtesy of UCB)

After getting his doctorate that year, he continued his research at LANL, focusing on structural materials used for nuclear components and developing a basic understanding of degradation processes in a nuclear environment. He worked extensively in the Ion Beam Materials Laboratory at Los Alamos, crediting the people and the lab's overall culture with the outlook he brought to Berkeley.

Under Hosemann's leadership, UCBNE continues to develop new tools and techniques as part of the NSUF system. Hosemann welcomes researchers who find uses for UCBNE capabilities that he might not have considered.

"Truly, everybody who wants to use it can. The NSUF gets you engaged in other areas and broadens everyone's horizons in the nuclear community. This was paid for by taxpayers, and to get the best use of taxpayer money, the tools and techniques developed should be available. What we are doing needs to be relevant to the broader DOE nuclear community."

His current research focus can be broken down into three parts:

- Small-scale materials testing on irradiated and unirradiated structural materials. This is aimed at reducing the necessary sample volume to a minimum in order to assess the materials state while investigating the basic effects of radiation damage.
- New advanced structural materials concepts (e.g., oxide dispersion-strengthened steels) for nuclear applications using accelerated materials testing via ion beam irradiations.
- Liquid metal corrosion of structural materials for nuclear applications. This involves a fundamental understanding of mechanisms that lead to improved alloying concepts and system operating techniques, with the overall goal of reducing corrosion issues.

Since 2008, Hosemann has authored more than 130 peer-reviewed articles. In 2014, he was named best reviewer by the Journal of Nuclear Materials and also received the American Nuclear Society's literature award. In 2015, he won the The Minerals, Metals and Materials Society (TMS) Early Career Faculty Fellow Award and The American Institute of Mining, Metallurgical, and Petroleum Engineers (AIME) Robert Lansing Hardy Award.

Hosemann also leads the UCB Bladesmithing team, which won the title of "best example of a traditional blade" for UCB, and is the lead faculty member for the CalSol solar car racing team, which won the American Solar challenge for Berkeley in 2017.

As for the NSUF Users Organization, Hosemann intends to expand communications among users through improved use of the Users Organization website, hosted by the NSUF at nsuf.inl.gov, and through the NSUF Users Organization email distribution list.

"The NSUF is a genius idea," he said. "By connecting new researchers with new ideas with the necessary partner facilities, the NSUF also raises intellectual nuclear science capital across the research community."

Riley Parrish

The NSUF launches a research career

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

University of Florida graduate student Riley Parrish freely concedes that without the resources of the Nuclear Science User Facilities he would not have been able to accomplish what he has since the beginning of 2017.

With NSUF funding and resources, he has completed multiple Rapid Turnaround Experiments focused on three-dimensional microstructural and chemical characterization of mixed oxide (MOX) fuels at varying stages of burnup. The funding would not likely have been available from any other source, and the resources definitely would not have been. "The NSUF was able to provide access to unique capabilities compared to universities and has been a great benefit to my research area," Parrish said.

After completing his undergraduate degree at Boise State University in 2015, Parrish spent two summers at INL working on energy storage systems in the Summer Undergraduate Laboratory Internship (SULI) program. He joined Dr. Assel Aitkaliyeva at the University of Florida in spring 2017 and has traveled back to Idaho on multiple occasions to conduct his work at INL's Materials & Fuels Complex.

Parrish said the more he learns about nuclear materials and fuels, the more fascinating he finds them. While most materials involve looking at only one or two major stimuli – high temperatures, corrosion, mechanical strength, stability – all of these considerations must be accounted for in a reactor environment.

"It's a complex puzzle that has been studied for decades now, but we're still actively learning about the intricacies of what happens to the fuel in such a hostile environment," he said. "It binds so many materials disciplines together to work toward a singular goal of safe and efficient fuel performance."

MOX fuels will be an important cog in the process of closing the nuclear fuel cycle, he said. They can incorporate plutonium from spent nuclear fuel and decommissioned nuclear weapons to eliminate dangerous long half-life elements from storage and promote nonproliferation.

"Nuclear fuel reprocessing will be critical to the success of limiting the potential environment impacts associated with spent nuclear fuel, and MOX fuels are just one form of fuel we can use to make sure that the energy is produced as efficiently as possible," he said. "My work is looking to study the fuel as it evolves in the reactor, to extend the lifetime of the fuel in a safe and effective manner."

Parrish's Rapid Turnaround Experiments have been sequential, examining MOX fuel specimens that were first irradiated in the mid-1980s as part of a core demonstration

Riley Parrish, University of Florida, using the Focused Ion Beam (FIB), the Electron Microscopy Laboratory (EML), MFC, INL (Chris Morgan, INL)

Riley Parrish, University of Florida,
using the FIB, EML, MFC, INL
(Chris Morgan, INL)

experiment at the Fast Flux Test Facility, located at DOE's Hanford Site. Taking advantage of the sophisticated characterization tools needed to understand fuel chemical and structural characteristics, he has been conducting detailed microstructural examination of the prototypic fast reactor fuel pins. The specific focus is on the ACO-3 fuel subassembly, which achieved a peak burnup of ~21 percent. The fuel pins had been previously sectioned and stored in the Materials and Fuels Complex's Hot Fuel Examination Facility (HFEF).

Fuel pins are being thoroughly examined using scanning electron microscopy (SEM) to both characterize the fuel microstructure and identify regions of interest for lamella and block extraction. A radial approach is being used to create tomography blocks and transmission electron microscopy lamella aimed at helping understand the effects of irradiation conditions on the local microstructures of the fuel pellets. Microstructural and microchemical examination of the prepared samples provides insight into the effects of radial position in the fuel pellet on structure and composition of the samples.

INL's Focused Ion Beam (FIB) tool is being used to prepare tomography blocks, which are then stored on site at INL for future use. Parrish estimated his time will be split roughly 60-40 between the University of Florida, home to the Nuclear Fuels and Materials Characterization Facility, an NSUF partner facility, and INL.

Parrish was on the cover of the February 2018 edition of Nuclear News, which featured a special section on NSUF capabilities. A native of south-central Idaho, he enjoys biking, backpacking, and climbing. He admits that he is still adjusting to what Florida has to offer.

Beyond graduation, Parrish believes environmental conservation will be the greatest challenge to the world in the next 50 years, and he is passionate about clean energy advocacy and advancement. "I believe that new nuclear reactor technologies will need to play a significant role in the future energy landscape and I would love to contribute to the promotion of the science, whether that is through active research at one of the national labs or through policy and public communications, to promote opportunities for advancement in the U.S.," he said. "Nuclear energy has never had a technology problem. It has always been a perception problem and I believe public outreach and activism is the best way to brighten the future of nuclear energy production."

"The NSUF was able to provide access to unique capabilities compared to universities and has been a great benefit to my research area," Parrish said.

Luca Capriotti

Introducing NSUF Instrument Scientists

Luca Capriotti, NSUF Instrument Scientist, always knew he wanted to work and conduct research in the United States. "The nuclear industry is more developed and the national laboratory system offers great research and professional development opportunities," he explained. Capriotti is fully qualified on the Physical Properties Measurement System (PPMS), which will be installed and up and running in INL's Irradiated Materials Characterization Laboratory (IMCL) by Summer 2018 for radioactive materials. A second instrument is also available in the INL Research Center for cold samples and depleted uranium.

Growing up in San Benedetto del Tronto, a town on Italy's east coast, Capriotti knew that he wanted to work in the applied sciences. After high school, he moved to Milan and earned his Bachelor's and Master's degrees in energy engineering.

Nuclear sparked his interest early on. "Nuclear physics had more depth and interest to me than mechanical engineering," he said. "The nuclear engineering department was a great learning environment. It was a small department and we were like a family."

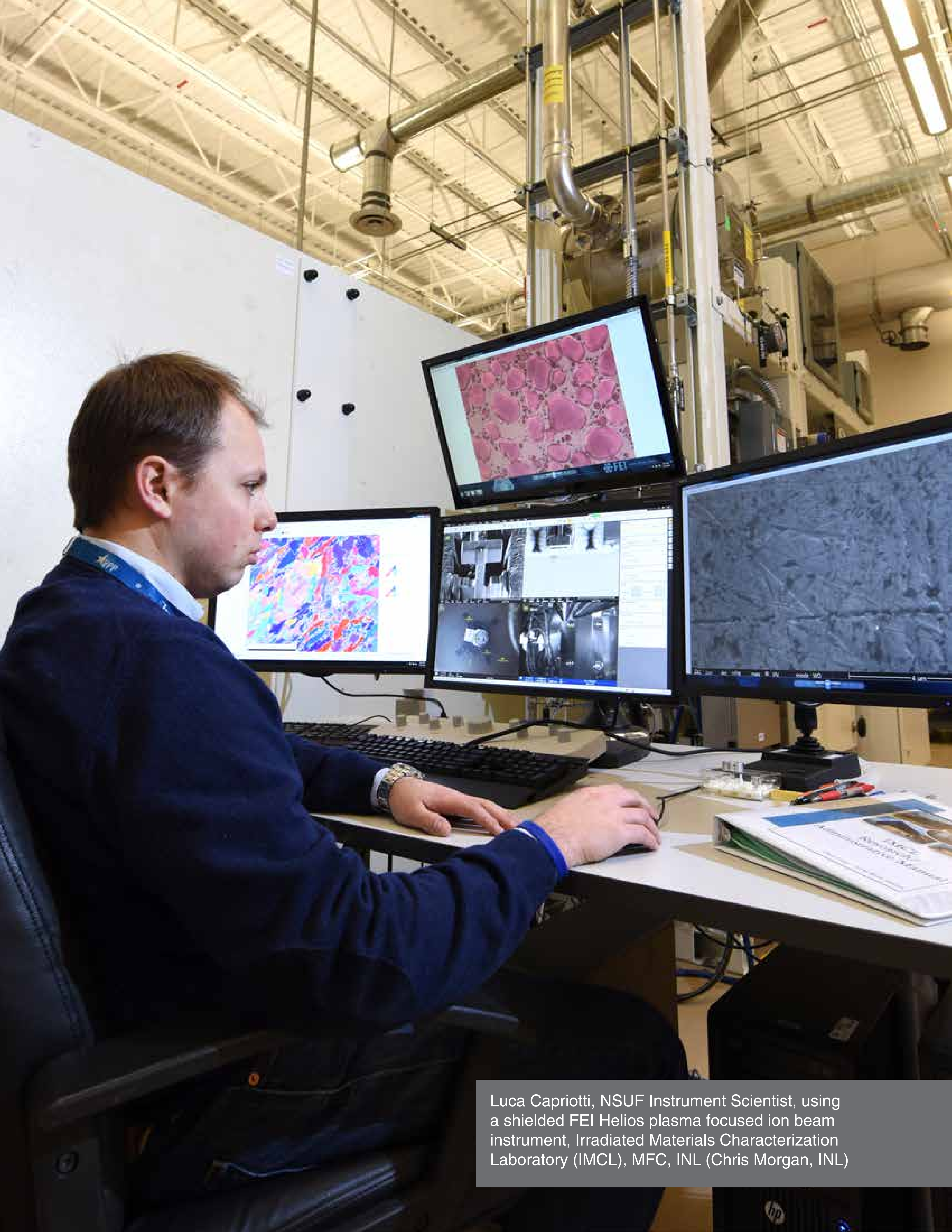
Nuclear engineering also provided an opportunity for experimental research, which captivated him. Capriotti knew this was his calling. In 2011, he moved to Karlsruhe, Germany, to work with the European Commission on his graduate project: laser techniques for high temperature melting point behavior of oxide materials. His research involved quite of bit of glovebox work (more on that later!).

Capriotti's research activities introduced him to several NSUF and INL scientists and leaders, including NSUF Director Rory Kennedy and HFEF Director Heather Chichester. His Ph.D. research project started in 2013 and was on post irradiation examination of metallic fuel with minor actinides. Six months before the end of his Ph.D. work at the European commission, he began to scan the INL career website on a regular basis and successfully applied for his current position in July 2015. Since then, time has flown by.

"When friends and family ask how I am, I always say that I'm living the dream," said Capriotti. In addition to his NSUF research, he works on the Advanced Fuel Campaign, concentrating on post irradiation examination (PIE) activities for Dr. Jason Harp. He is one of two scientists fully qualified on the PPMS, along with INL scientist Krystoff Goffryk, who is the scientific supervisor.

His request to NSUF researchers interested in how they can use the PPMS in their work is straightforward: "Please call or email me. I like talking about new projects and areas of research, and I can work with you on project feasibility. The earlier, the better, since we can discuss approaches and timing considerations, which can then be incorporated into your NSUF proposal."

"I like talking about new projects and areas of research, and I can work with you on project feasibility."



Luca Capriotti, NSUF Instrument Scientist, using a shielded FEI Helios plasma focused ion beam instrument, Irradiated Materials Characterization Laboratory (IMCL), MFC, INL (Chris Morgan, INL)

Fidelma Di Lemma, NSUF Instrument Scientist, using a Scanning Electron Microscope (SEM), FASB, MFC, INL (Chris Morgan, INL)

Fidelma Di Lemma

Fidelma Di Lemma pursues the urgency of doing

Fidelma Di Lemma was born an inventor with a passion for adventure. "My father is an engineer and many of my family members are in engineering, so I wanted to try something new and unexpected," she explained. Her journey to joining the NSUF as an instrument scientist spans the globe and encompasses her novel approach to the sciences and engineering.

Growing up in a small Italian village outside of Rome, Italy, she had ready access to world-renowned art history for inspiration, as well as a treasure trove of toys (and the occasional appliance) that she relished disassembling. "As a kid I would break things to see how they would work," she said. With an Irish mother and an Italian father, travel was a way of life — a familiarity that would serve her well as an adult. She grew up bilingual in English and Italian and learned additional languages through travel.

A fateful decision in high school broadened Di Lemma's career options to include engineering. For her graduation project, she decided to focus on the petrol crisis in the 1970s. Di Lemma became absorbed by how the world would meet growing energy demands moving forward. "I always took mathematics and science classes. I'm adept in mathematics and enjoy both disciplines."

Her fascination with novel and innovative approaches has also shaped her approach to engineering. "Nuclear engineering added an additional dimension and complexity to electricity generation beyond the need to turn a turbine, Di Lemma said. "I became more interested in nuclear science and engineering than in electricity production, per se." She pursued a B.S. in energy engineering, followed by an M.S. in nuclear engineering.

"Please contact me if you are writing a proposal. I like to talk with researchers as they're developing a proposal. I can help with scheduling and feasibility and make life easier for everyone involved with the project."

Leonardo da Vinci said, "I have been impressed with the urgency of doing. Knowing is not enough; we must apply. Being willing is not enough; we must do." Always interested in the practice as much as the theory, Di Lemma enthusiastically began her research at the European Commission Joint Research Laboratory in Karlsruhe, Germany. Her Ph.D. research focused on using laser heating techniques for aerosol production to simulate radiation dispersal devices, which required frequent use of a glovebox for research. The story of the glovebox begins on page 50.

Di Lemma joined INL in 2016, following a post-doc stint in Tokai-mura, Muramatsu, Japan, with the Japan Atomic Energy Agency. "I liked my work and coworkers in Japan, but my life was calling to me to Idaho," she said. Her staff position put her in place to become fully qualified for two instruments: the scanning electron microscope (SEM) for fresh fuel, which will soon be moved from INL's Fuels and Applied Sciences Building (FASB) to INL's Irradiated Materials Characterization Laboratory (IMCL), and the electron probe microscope (EPMA) for irradiated materials, located in the IMCL. She is fully qualified on the SEM and partially qualified on the EPMA.

Di Lemma is also working to develop techniques for sample preparation for electron backscatter diffraction (EBSD) research. The techniques permit the determination of grain size and orientation, and to investigate stress and deformation in materials and require keeping current in new research approaches as well as a sound basis in current practices.

What advice does she have for researchers interested in using INL capabilities?

"Please contact me if you are writing a proposal. I like to talk with researchers as they're developing a proposal. I can help with scheduling and feasibility and make life easier for everyone involved with the project."

Fidelma Di Lemma, NSUF Instrument Scientist, using an SEM, Fuels and Applied Sciences Building, MFC, INL (Chris Morgan, INL)

THE GLOVEBOX

A Tale of Actinides and Love

Luca Capriotti and Fidelma Di Lemma met while working side-by-side with adjoining gloveboxes at the Institute for Transuranium Elements in Karlsruhe, Germany. Their respective work both focused on decoding the magic of nuclear science – more specifically, the behavior of actinide materials subjected to high temperatures.

Actinides are any of the series of fifteen metallic elements from actinium (atomic number 89) to lawrencium (atomic number 103) in the periodic table. They are all radioactive, the heavier members being unstable and human-made. Although quite interesting to nuclear scientists and engineers who are always seeking improvements in nuclear fuels, actinides do not typically fill the dreams of little boys and girls imagining the great romance that will signal the arrival of their life partner.

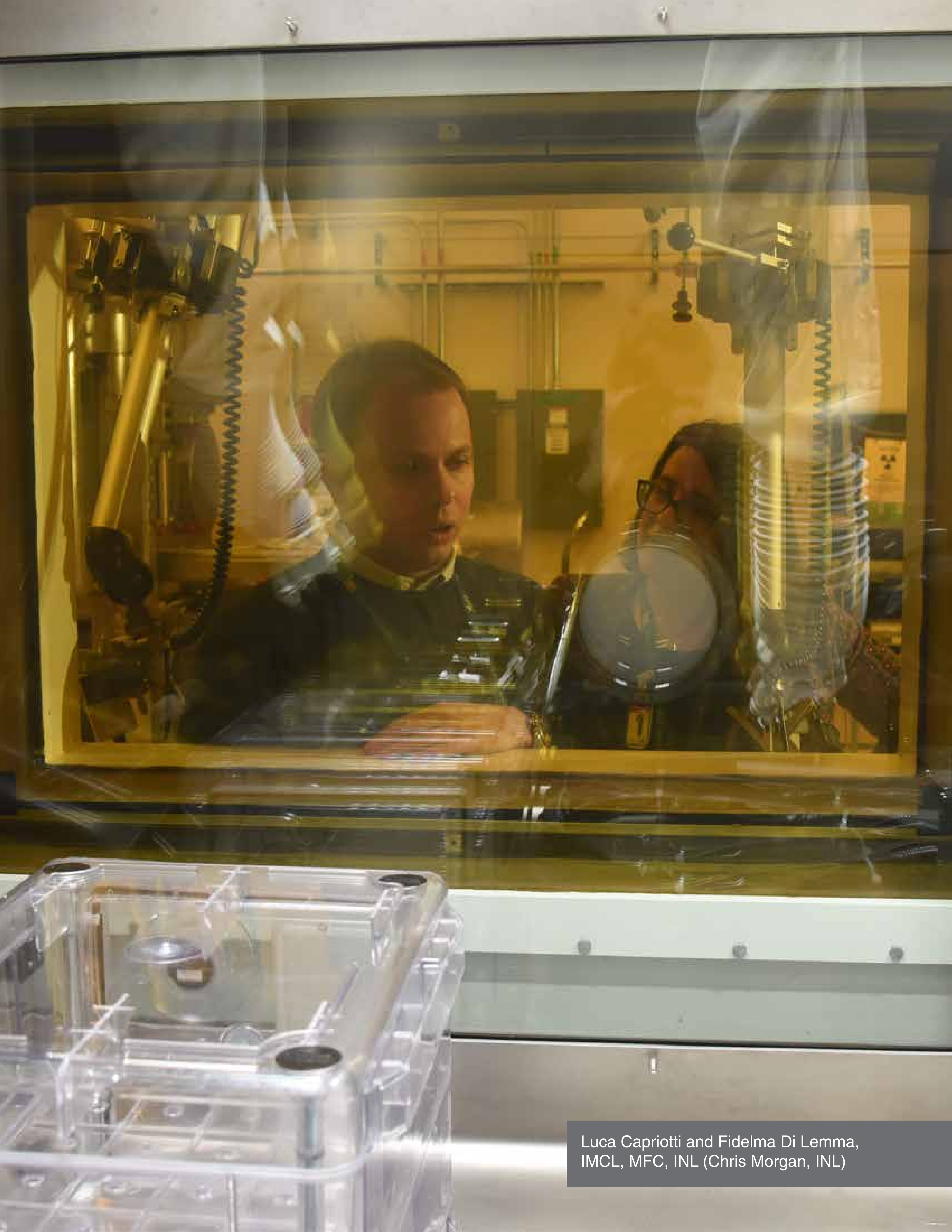
What stray atomic attraction brought Capriotti and Di Lemma together?

"We were working side by side and we're both Italian," explains Di Lemma, laughing. "It would have been more strange if we hadn't met."

Talking over their respective work soon led to discussions about their respective lives and backgrounds and future plans and dreams. Italian is a romance language, which no doubt helped. A shared interest in understanding nuclear energy needs and barriers across the globe created their first joint venture: participation in international organizations of nuclear professionals, including the International Youth Nuclear Congress.

"I wanted to be better connected to the nuclear industry and understand nuclear research needs in Germany and around the world," said Capriotti.

His research landed him opportunities with the European Space Agency in Madrid, Spain; Ph.D. studies at the Technische Universität München in Munich, Germany; and the European Commission Joint Research Centre in Eggenstein-Leopoldshafen, Germany. Meanwhile, the Institute for Transuranium Elements asked Di Lemma to stay to commence a contract as a Ph.D student. She then started a new post doctoral research fellow position with the Japan Atomic Energy in Muramatsu, Japan, while Capriotti remained in Germany.



Luca Capriotti and Fidelma Di Lemma, IMCL, MFC, INL (Chris Morgan, INL)

Luca Capriotti and Fidelma Di Lemma at the HFEF, MFC, INL. Capriotti is using a new inverted optical microscope (Leica model DMi8, modified for hot cell settings). (Chris Morgan, INL)

"We were both in International Youth Nuclear Congress (IYNC) leadership positions," said Di Lemma. "Sometimes the IYNC meetings were the only times we were in the same country."

They became engaged in March 2015 just before Di Lemma went to Japan. Capriotti was first to join INL in early 2016. Di Lemma accepted a position with INL in late 2016. They married June 2017 in Rome, Italy.

Involvement in the IYNC does not guarantee marriage for everyone, although Di Lemma says that she knows of another happily married couple who met through the organization. Capriotti and Di Lemma maintain their strong participation for professional reasons. In fact, Capriotti was elected in March 2018 to a two-year term as IYNC President at a biennial conference. At the same conference, Di Lemma managed the first-ever session of the IYNC International Innovation Congress I4N (Innovation for Nuclear), which saw teams from all over the world proposing innovative solution to the challenges facing the nuclear industry.

"IYNC fosters leadership skills, especially in coordinating the efforts of a team," said Capriotti. "Involvement helped me to gain a broad perspective on culture and learn protocols for business."

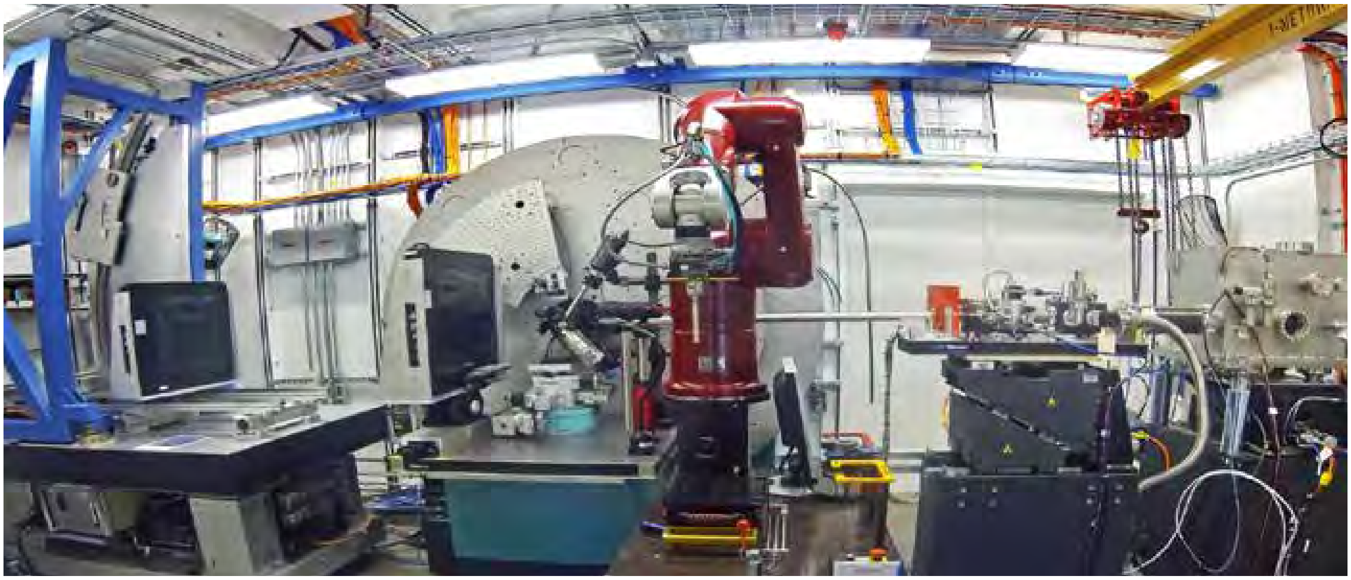
"The global function of IYNC provides members with a better understanding of the nuclear communities around the world," said Di Lemma. "In terms of managing teams, I've learned to be clear with what the "ask" is and the timeline necessary to meet commitment – and to give participants a way to opt out if necessary, as we are volunteers dedicating whatever time we can find."

What's next in store for this research duo?

"I'm enjoying becoming certified on the electron probe microscope (EPMA) for irradiated materials," said Di Lemma. "The opportunities the NSUF has provided, like presenting my research at the annual program review, are giving me a valuable perspective on how a successful research program operates."

As for Capriotti? "I'm already living the dream," he said with a smile. "I have the opportunity for research and professional development. I'm happy."

NEW NSUF PARTNERSHIPS



XPD Beamline, NSLS-II, Brookhaven National Laboratory

The Nuclear Science Users Facility (NSUF), a network of nuclear energy research institutions across the United States, added seven new partner facilities in FY 2017 and one international affiliate.

While ATR – the only materials test reactor in the United States that can replicate multiple reactor environments concurrently – remains the crown jewel, the NSUF has expanded its scope over ten years to incorporate a wide variety of reactor and research facilities from coast to coast.

Partner Facilities Added in FY 2017

Brookhaven National Laboratory

National Synchrotron Light Source II

Core Functions: X-ray beams.

Brookhaven National Laboratory's NSLS-II enables the study of material properties and functions with nanoscale resolution and exquisite sensitivity by providing world-leading capabilities for X-ray imaging and high-resolution energy analysis. The NSLS-II is a medium energy (3.0 GeV) electron storage ring designed to deliver photons with high average spectral brightness exceeding 10^{21} ph/s in the 2–10 keV energy range and a flux density exceeding 10^{15} ph/s in all spectral ranges.

Lawrence Livermore National Laboratory

Center for Accelerator Mass Spectrometry

Core Functions: Accelerator ion irradiation, ion beam analysis and mass spectroscopy.

CAMS hosts a 10-MV FN tandem Van de Graaff accelerator, a NEC 1-MV tandem accelerator, and a soon to be commissioned 250KV single stage accelerator mass spectrometry (AMS) deck will perform up to 25,000 AMS measurement per year. The Center also has an NEC 1.7-MV tandem accelrator for ion beam analysis and microscopy. The research and development made possible by AMS and ion beam analytical techniques is diverse and includes material analysis and modification studies, as well as nuclear physics cross-section measurements and nuclear chemistry studies.

The research team at the Center for Accelerator Mass Spectrometry (CAMS), Lawrence Livermore National Laboratory (LLNL) (Courtesy of LLNL).

Los Alamos National Laboratory

Lujan Center at Los Alamos Neutron Science Center (LANS)

Core Functions: Cold neutron scattering and diffraction techniques.

The LANS features instruments that operate in time-of-flight mode, receiving neutrons from a tungsten spallation target. Four moderators provide epi-thermal, thermal and cold neutrons to specialized beamlines. The available instrument suite includes the Spectrometer for Materials Research at Temperature and Stress (SMARTS); High-Pressure-Preferred Orientation instrument (HIPPO); Flight Path 5 for energy-resolved neutron imagining; and other beamlines, as available.

Researchers preparing for an experiment on the High Pressure Preferred Orientation (HIPPO) Instrument in the Lujan Center at the Los Alamos Neutron Science Center (LANS), Los Alamos National Laboratory (LANL) (Courtesy of LANL).

Wing 9 Hot Cells

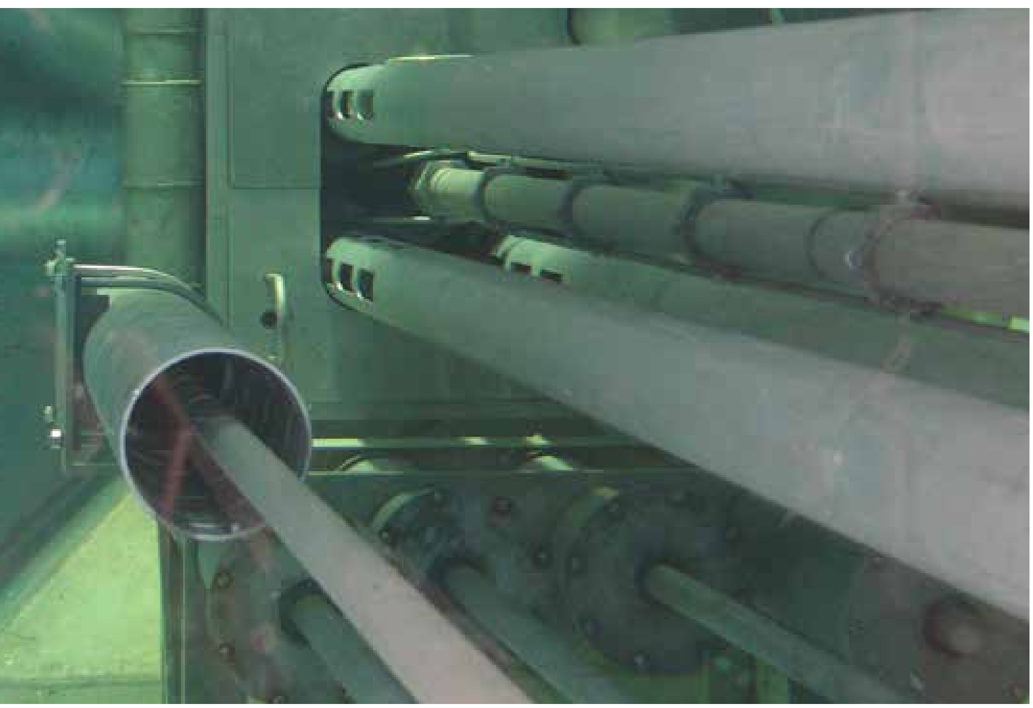
Core functions: remote testing.

The Los Alamos National Laboratory Wing 9 Hot Cells consist of four shielded cells for mechanical testing (including tension, compression, bend bar, ring pull and harness testing); machining/cutting/polishing; sample handling and storage; cleaning; and shipping and receiving. Support equipment includes a 10-ton crane for interior sample movement and a 25-ton crane for equipment. The Wing 9 Hot Cells accept beta-gamma materials only.

Plutonium Surface Science Laboratory

Core Functions: Oxide Fuel Fabrication (PuO_2) and characterization.

The Plutonium Surface Science Laboratory provides capabilities in scanning tunneling microscopy and spectroscopy, atomic force microscopy, infrared reflection-absorption spectroscopy, secondary ion mass spectroscopy, x-ray and ultraviolet photoemission spectroscapies, profilometer, and gas chromatograph mass spectroscopy.



View from the top of the pool at the Ohio State University Research Reactor, The Ohio State University (Courtesy of The Ohio State University)

The Ohio State University

The Ohio State University Nuclear Reactor Laboratory (OSU-NRL)

Core Functions: Thermal neutron irradiation of nuclear fuels and materials and instrumentation.

The OSU-NRL offers the unique capability of reactor irradiations in external large-volume experiment dry tubes at temperatures from 4 K to 1873 K. Uses include experiments involving instrumented, high-temperature irradiations of prototype instrumentation for next generation reactors, sensors, and sensor materials, as well as optical fibers designed for up to 1600°C.

Researcher Khalid Hattar using the In Situ Ion Irradiation Transmission Electron Microscope (I3TEM) in the Ion Beam Laboratory, Sandia National Laboratories (Courtesy of SNL).

Sandia National Laboratories

Ion Beam Laboratory (SIBL)

Core Functions: Ion irradiation, ion beam modification, ion beam analysis and high magnification imaging.

The *In Situ* Ion Irradiation Transmission Electron Microscope (I3TEM) Facility at the SIBL offers ion irradiation, including *in situ* irradiation in a TEM with specialty specimen stages available, such as heating, cooling, strain, compression, and changes in specimen environment. The I3TEM Facility offers the capabilities of a 200 kV JEOL 2100 high-contrast TEM combined with the implantation/irradiation capabilities of the 10 kV Colutron and the 6 MV Tandem accelerators housed in the SIBL. SIBL's eight accelerators cover a wide range of energies and ions.

Gamma Irradiation Facility (GIF)

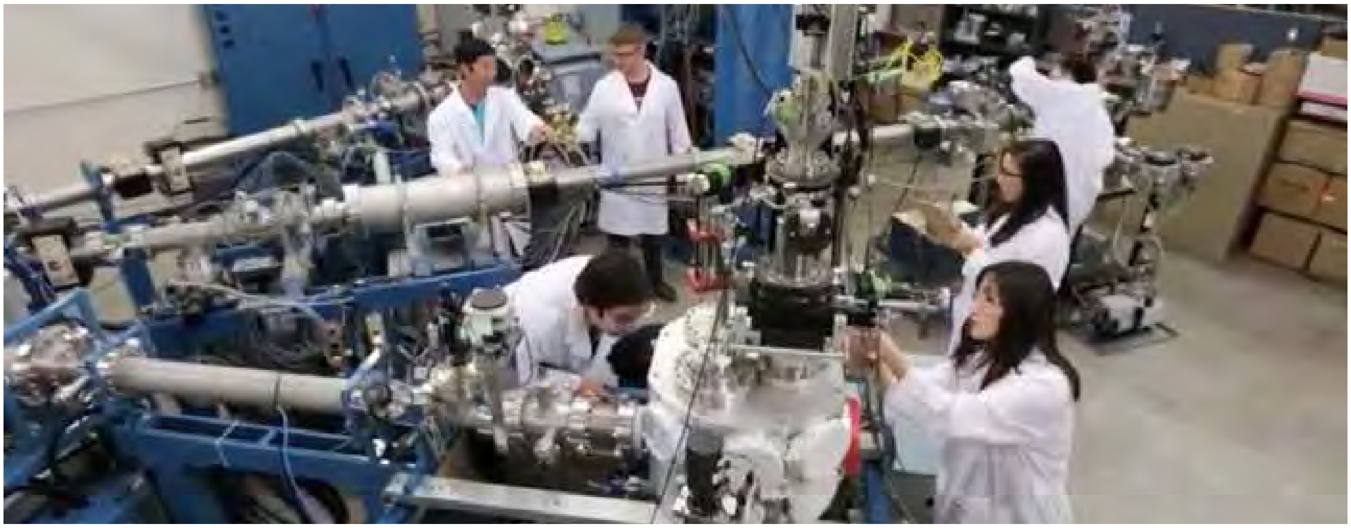
Core Functions: Gamma irradiation of materials and sensors using Co-60 sources.

The GIF produces a wide range of gamma radiation environments using Co-60 sources. The GIF is capable of irradiating objects as small as bacteria and as large as an Abrams M1 tank (although SNL typically irradiate electronic components, equipment and samples of various materials). The GIF provides in-cell dry irradiations in test cells and in-pool submerged irradiations in the pool. The GIF has three concrete dry test cells: two cells are 3 m × 3 m, one cell is 5.5 m × 9.1 m, and an 18-foot deep pool. The facility offers gamma dose rates from 10^{-3} rad/s to over 1000 rad/s.

Annular Core Research Reactor (ACRR)

Core Functions: Safety testing of nuclear fuel samples and instrumentation.

The ACRR is an epi-thermal pool-type reactor which uses cylindrical UO_2-BeO fuel elements. Researchers perform sample irradiations in typical research reactor steady-state mode or in a high-power pulse mode, reaching powers as high as 30GW for a few milliseconds. There are four main experimental cavities at the ACRR facility: central cavity, FREC-II cavity, thermal neutron beam tube (the neutron radiography facility), and the Tri-Element facility. The ACRR is complementary to TREAT (INL), focusing more on electronics testing for the National Nuclear Security Administration.



Researchers in the Texas A&M Accelerator Laboratory, Texas A&M University (Courtesy of Texas A&M University).

Texas A&M University

Texas A&M Accelerator Laboratory

Core Functions: Ion irradiation and ion beam analysis.

The Texas A&M Accelerator Laboratory is one of the largest university ion irradiation facilities in the United States. Key facilities in the lab include: a 10 kV ion accelerator (with a gas ion source); a 150 kV Ion Accelerator (with a universal ion source); a 200 kV ion accelerator (with a universal ion source); a 1 MV Ionex Tandetron Accelerator (with a RF plasma source and a Source of Negative Ions by Cesium Sputtering (SNICS) source); a 1.7 MV Ionex Tandetron Accelerator (with an RF plasma source and a SNICS source); a high temperature vacuum furnace; a high temperature gas furnace; a four-point-probe resistivity measurement; and various heating and cooling systems for ion irradiations at different temperatures.

University of Florida

Nuclear Fuels and Materials Characterization Facility (NFMC)

Core Functions: Materials characterization of irradiated materials.

The NFMC provides capabilities in microstructural characterization and mechanical properties evaluation of materials-related research with an emphasis on nuclear. The laboratory is dedicated to supporting radiological work and the University of Florida Teaching Reactor.

Dual Beam Focused Ion Beam (FIB) and Scanning Electron Microscope (SEM) at the Nuclear Fuels and Materials Characterization Facility, University of Florida (Courtesy of University of Florida).

International Affiliate Added in 2017

SCK•CEN

Belgian Reactor 2 (BR2)

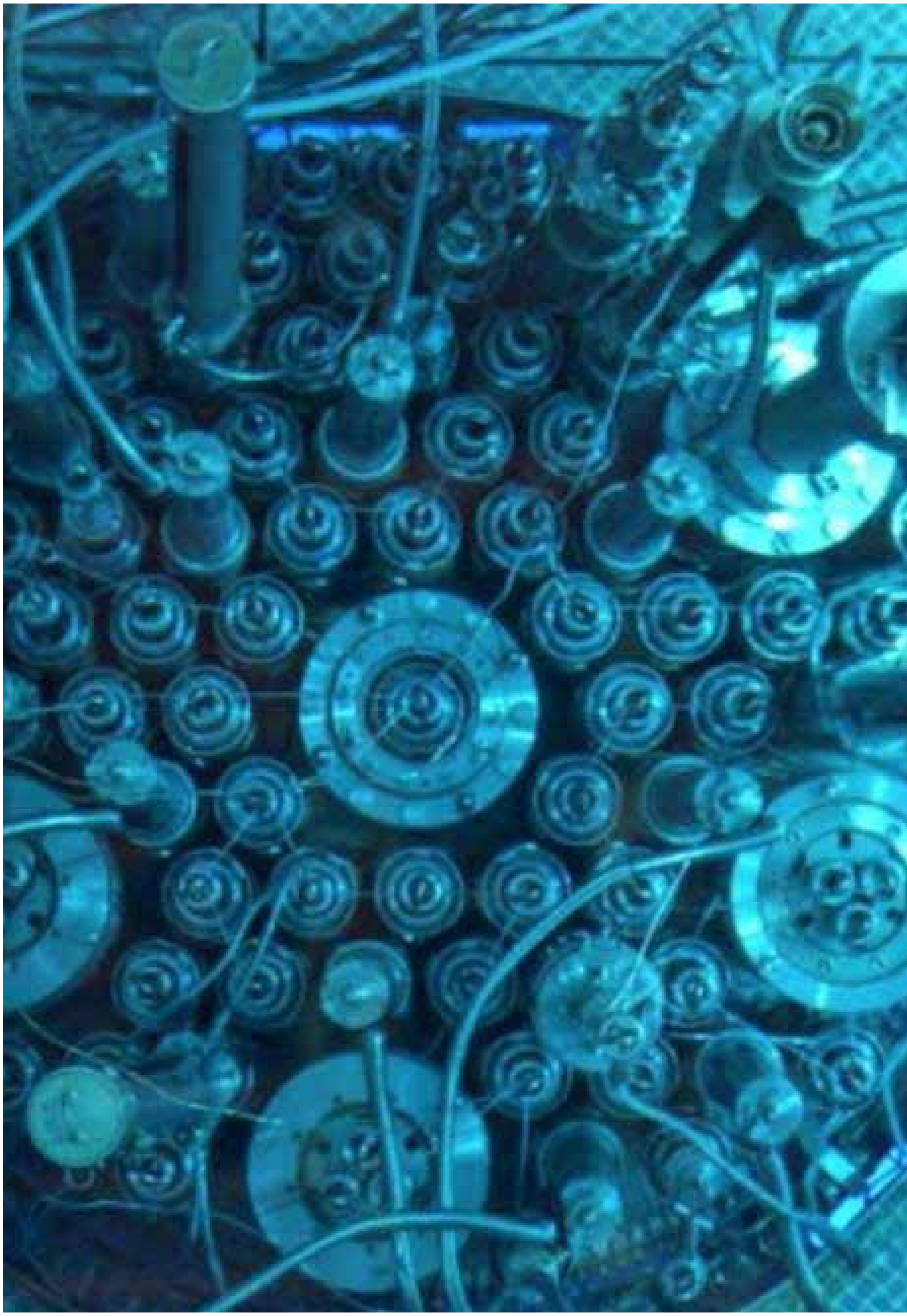
Core Functions: Advanced fuel and materials irradiation. Versatile core configurations.

BR2 is among the most powerful and flexible research reactors in the world. BR2 irradiates all kinds of nuclear fuels and materials for different types of reactors and the European nuclear fusion program. The intense radiation allows researchers to study aging of irradiated materials. The core can be reconfigured to accommodate custom experimental items.

Laboratory for High and Medium Activity (LHMA)

Core Functions: Materials characterization of highly irradiated materials.

The LHMA focuses on the effects of radiation on materials, such as in the pressure vessels of nuclear reactors or the effects of uranium fission in the reactor fuel. The laboratory has the necessary infrastructure to handle highly radioactive substances safely. The study of fuel pins from reactors around the world is supported.



View of the reactor core of the Belgian Reactor 2 (BR2), SCK•CEN.

NSUF-GAIN SYNERGY

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Synergy of the NSUF with the DOE GAIN Initiative

The Gateway for Accelerated Innovation in Nuclear (GAIN) initiative was established to address issues that constrain innovation in the domestic nuclear industry. As such, GAIN influences the direction and priorities of relevant U.S. Department of Energy, Office of Nuclear Energy (DOE-NE) research, development and deployment (RD&D) programs and functions as a framework for private-public partnerships. The GAIN initiative offers a single access point to a broad range of expertise and capabilities over many subject areas and promotes working relationships between nuclear technology developers and DOE national laboratory expertise.

The Nuclear Science User Facilities (NSUF) program is focused on understanding irradiation effects in nuclear fuels and materials and, for this particular subject area, is a key contributor to the GAIN initiative. The NSUF offers the nuclear community access to capabilities and expertise at the unique facilities at Idaho National Laboratory (INL) and 20 additional partner facilities. The nuclear community that the NSUF supports includes researchers from the nuclear industry as well as those from universities, government laboratories, and small businesses. The GAIN initiative and the NSUF program thus represent a powerful combination of resources for those contributing to re-establishing the U.S. leadership position in nuclear power.

In principle, the advanced and innovative fuels and materials vetted through NSUF experimental research would be picked up by GAIN and its network of industry members to move forward with commercialization and deployment of these materials. The DOE-NE Memorandum of Understanding with the U.S. Nuclear Regulatory Commission (NRC) on implementation of GAIN is one tool to apply here. The NSUF in 2017 awarded industry-led Consolidated Innovative Nuclear Research (CINR) projects addressing the joining of advanced SiC-SiC cladding to General Atomics, advanced neutron absorbing materials to AREVA, an additive manufacturing process from Westinghouse, and two projects to Electric Power Research Institute (EPRI), one on high burnup fuel and another on the hydrogen pickup mechanism in

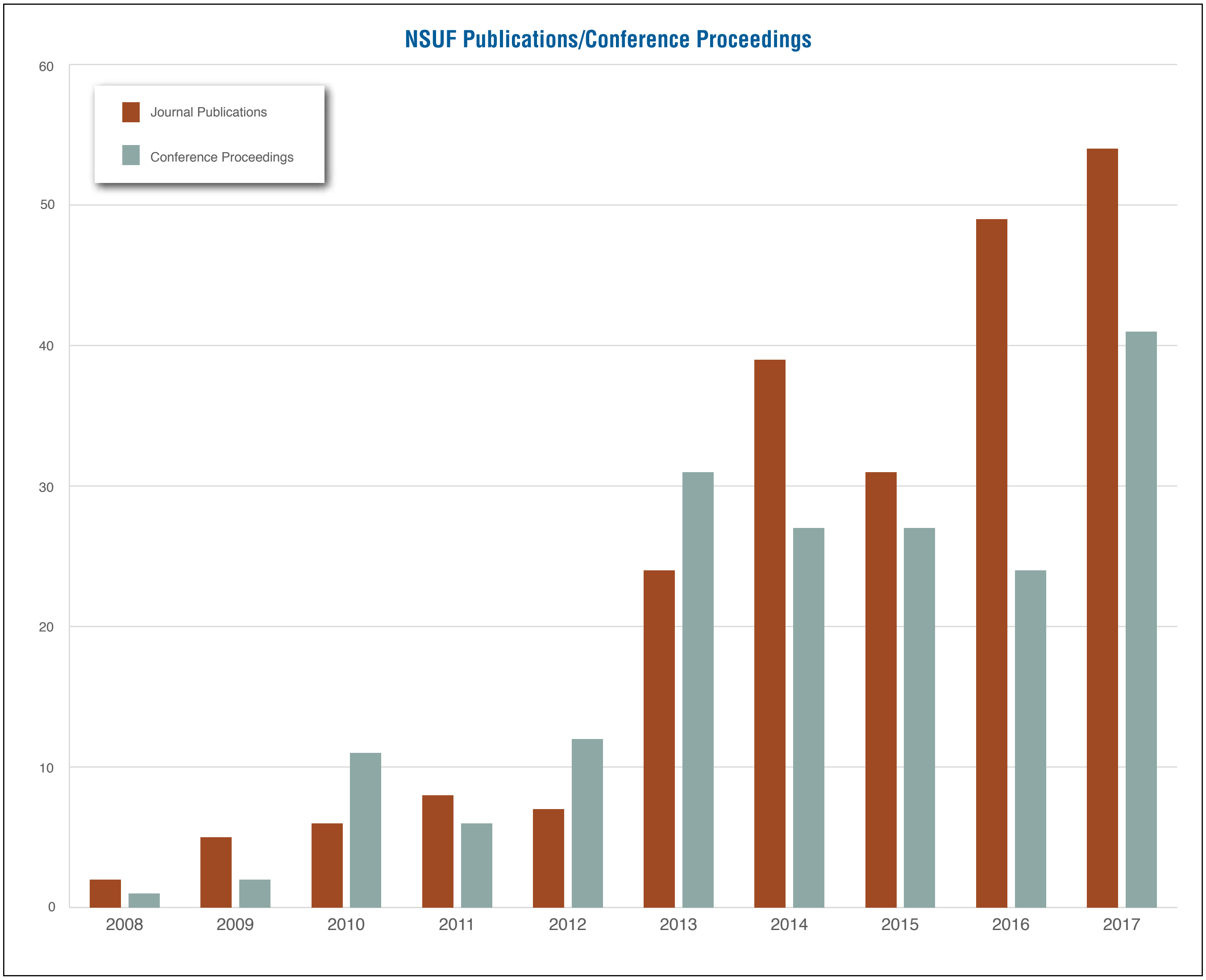
Zircaloy-2. These examples indicate how the NSUF operates at all Technology Readiness Levels in addressing specific scientific questions of interest to industry (See table).

Beginning in 2017, the NSUF and GAIN began collaborating on industry outreach workshops in order to maintain and improve (where necessary) responsiveness to industry needs that the DOE-NE can address. The NSUF thermal hydraulics workshop was held in cooperation with GAIN in order to assess and prioritize thermal hydraulics facilities that might contribute most to the mission of DOE-NE in advancing technologies, particularly for small modular reactor designs. The NSUF recently included a GAIN representative in its workshop to develop a Fuels and Materials Understanding Scale (FaMUS) that will provide a means to indicate the state of understanding of the behavior of a particular material in reactor. Implementation of this scale will aid in assessing the impact of NSUF research on the nuclear industry in addressing questions of importance. Going forward, the NSUF and GAIN will collaborate on additional workshops to support the current fleet as well as advanced reactor concepts. Examples that cover both areas would be potential workshops on Advanced Manufacturing and Advanced Modeling and Simulation capabilities. The NSUF also regularly invites GAIN to participate in its Industry Advisory Committee meetings.

FY 2017 Consolidated Innovative Nuclear Research (CINR) Awards to Industry

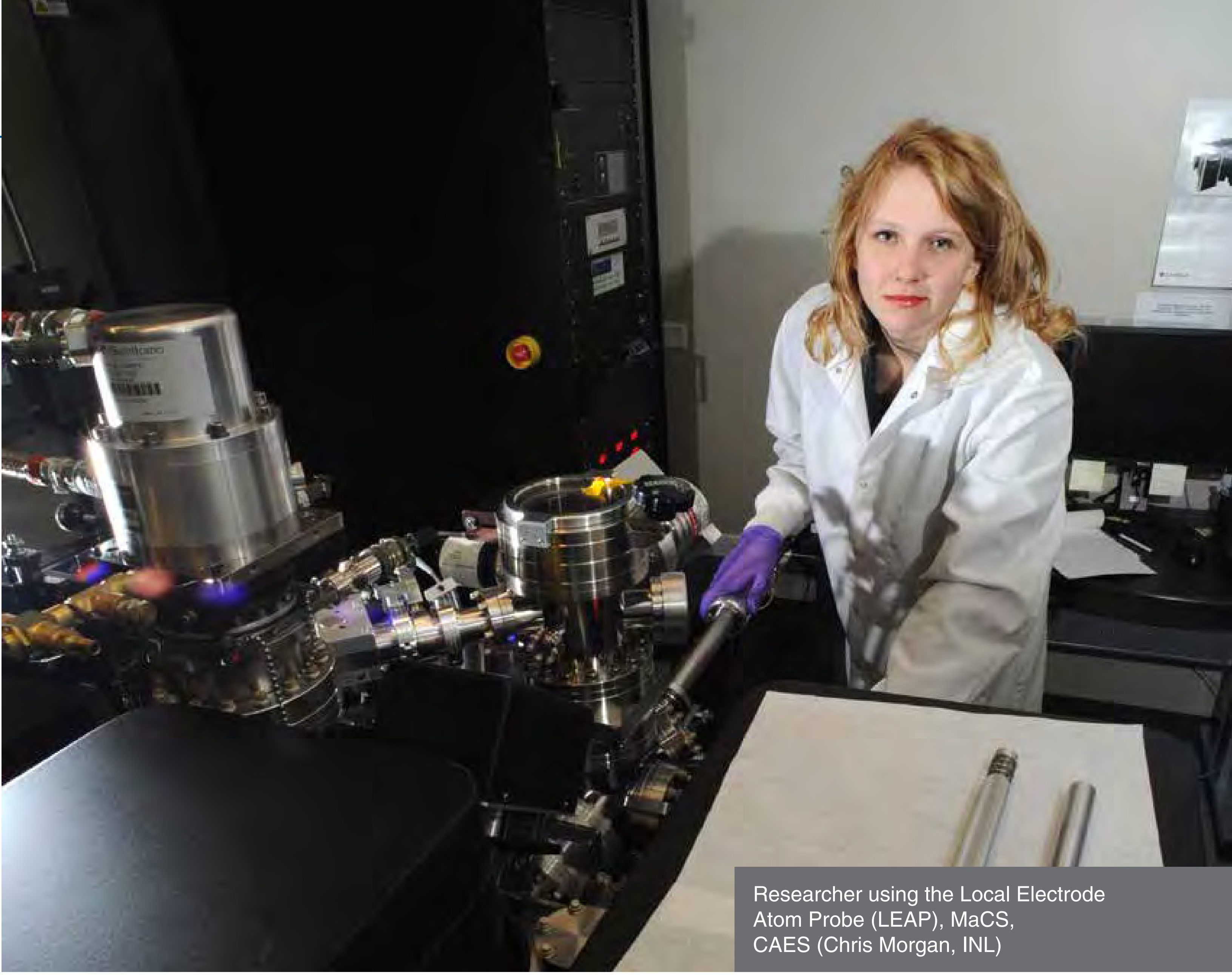
General Atomics	Performance of SiC-SiC Cladding and Endplug Joints Under Neutron Irradiation with a Thermal Gradient
AREVA	Irradiation of Advanced Neutron Absorbing Material to Support Accident Tolerant Fuel
Electric Power Research Institute, Inc.	Irradiation, Transient Testing and Post Irradiation Examination of Ultra High Burnup Fuel
Westinghouse Electric Company	Radiation Effects on Zirconium Alloys Produced by Powder Bed Fusion Additive Manufacturing Processes
Electric Power Research Institute	Improved Understanding of Zircaloy-2 Hydrogen Pickup Mechanism in BWRs

MEASURING IMPACT



	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017
Journal Publications	2	5	6	8	7	24	39	31	49	54
Conference Proceedings	1	2	11	6	12	31	27	27	34	41

Figure 1. NSUF Publications and Conference Proceedings by calendar year



Researcher using the Local Electrode Atom Probe (LEAP), MaCS, CAES (Chris Morgan, INL)

A Peek Inside the NSUF

NSUF Publications and Conference Proceedings

In order for the Nuclear Science User Facilities (NSUF) to fulfill the DOE-NE's mission to advance nuclear energy, NSUF researchers must publish and document research results through peer-reviewed research journals and presentations at conferences.

The NSUF has tracked publications and conference proceedings since 2008, and took additional steps in FY 2017 to increase the number of self-reported publications and to improve the consistency in the data capture process for collecting the lists of publications and conference proceedings.

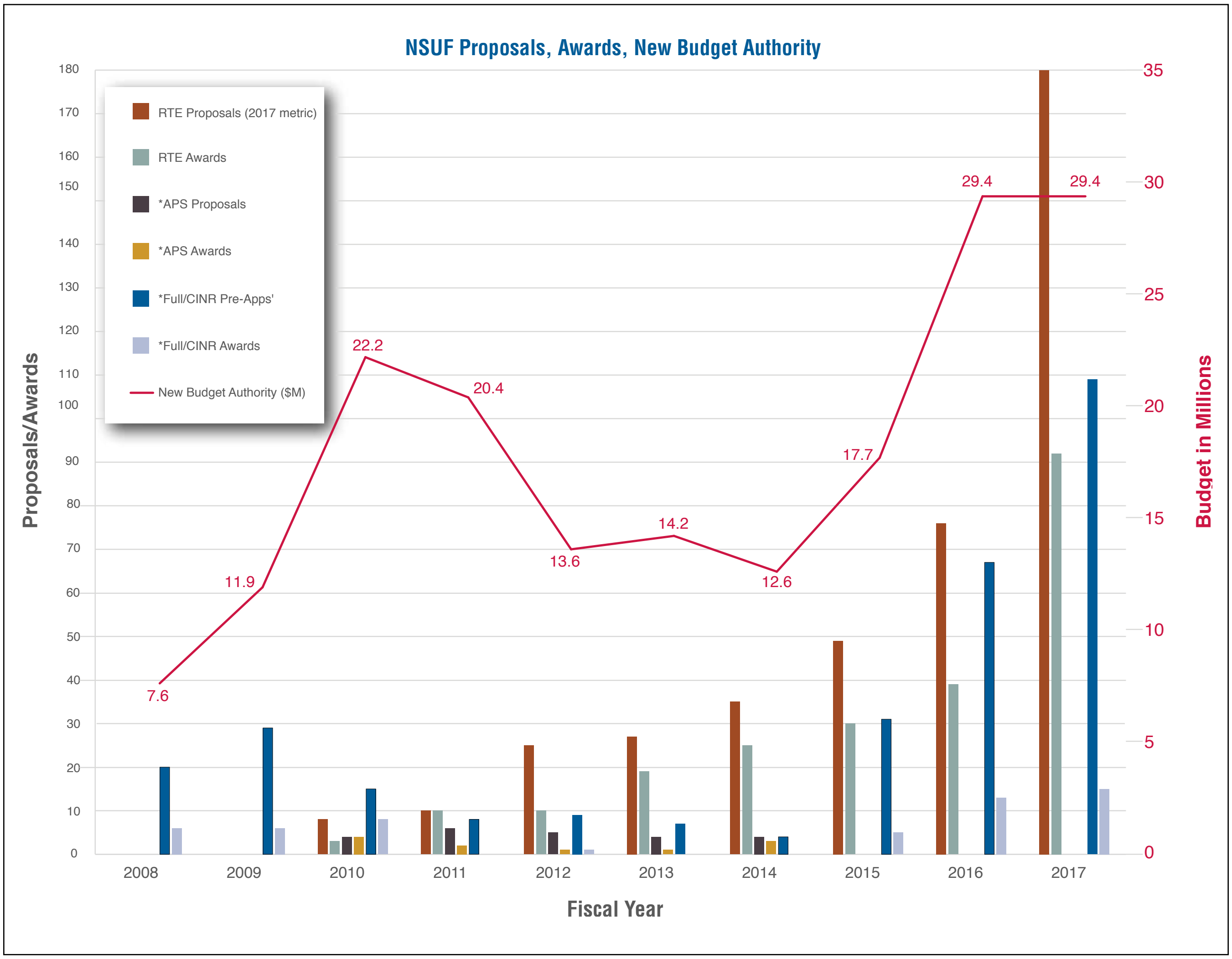
NSUF Experiment, Test Train Assembly Facility, ATR Complex, INL (Chris Morgan, INL)

Out of the 225 total publications for the NSUF in 60 different journals, 89 were published in the Journal of Nuclear Materials. This indicates that NSUF projects are producing quality scientific output and that the Journal of Nuclear Materials is the most important journal to the NSUF nuclear community from a relevancy viewpoint. The importance of this journal is also emphasized by the fact that 26 of the publications coming out of the NSUF were published in this journal in 2017.

Expansion and Diversification of the NSUF User Community

In order to evaluate how the NSUF is growing, several metrics are tracked over time:

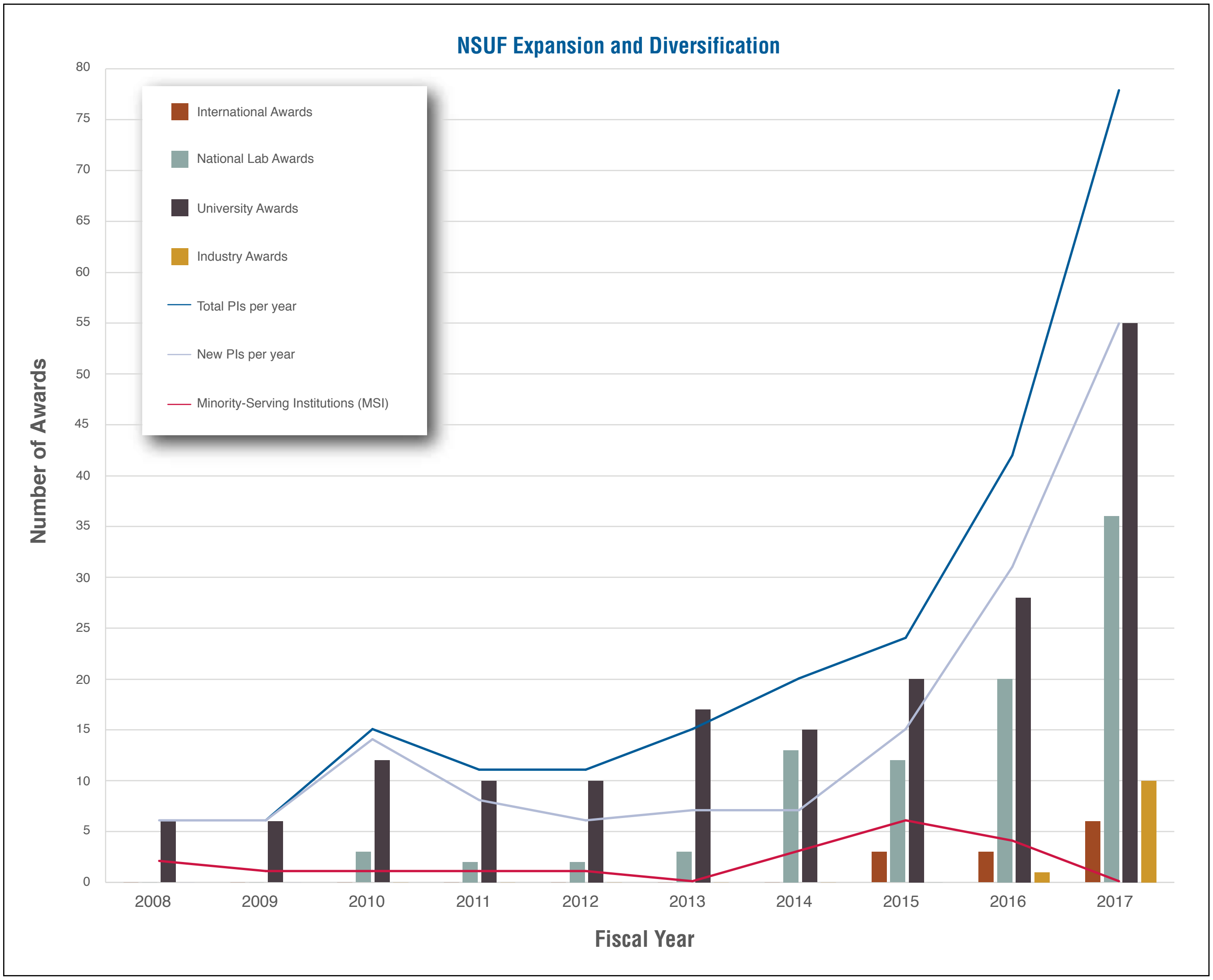
- the number of Rapid Turnaround Experiment (RTE) proposals
- the number of RTE awards
- the number of Consolidated Innovative Nuclear Research (CINR) pre-applications
- the number of CINR full applications, and
- the number of CINR awards



	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017
*APS Proposals			4	6	5	4	4			
*APS Awards			4	2	1	1	3			
RTE Proposals			8	10	25	27	35	49	76	180
RTE Awards			3	10	10	19	25	30	39	92
*Full/CINR Pre-Apps	20	29	15	8	9	7	4	31	67	108
*Full/CINR Awards	6	6	8		1			5	13	15
New Budget Authority ($M)	7.6	11.9	22.2	20.4	13.6	14.2	12.6	17.7	29.4	29.4

* Full/APS Awards transitioned to CINR Award in 2015

Figure 2. NSUF Proposals, Awards, New Budget Authority ($M)



	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017
International Awards	0	0	0	0	0	0	0	3	3	6
National Lab Awards	0	0	3	2	2	3	13	12	20	36
University Awards	6	6	12	10	10	17	15	20	28	55
Industry Awards	0	0	0	0	0	0	0	0	1	10
Total PIs per year	6	6	15	11	11	15	20	24	42	78
New PIs per year	0	6	14	8	6	7	7	15	31	55
Minority-Serving Institutions (MSI)	2	1	1	1	1	0	3	6	4	0

FY 17 Data was analyzed against the UNITED STATES DEPARTMENT OF EDUCATION ACCREDITED POSTSECONDARY MINORITY INSTITUTIONS List: https://www2.ed.gov/about/offices/list/ocr/edlite-minorityinst-list-tab.html

Figure 3. NSUF Expansion and Diversification

Quality Assurance inspection of upper locking mechanisms of reactor vessel surveillance capsules, Westinghouse Churchill Site (Courtesy of Westinghouse Electric Company).

To help evaluate expansion and diversification, the NSUF tracks the number of combined CINR and RTE awards by year made to international, national laboratory, industrial and university institutions. A plot of the total number of Principal Investigators (PIs) and new PIs per year is also tracked.

The NSUF continued its expansion and diversification to international and industry requesters in FY 2017. NSUF research opportunities have received a significant increase in user interest from industry applicants. The increase in industry proposals demonstrates that industry regards NSUF as a viable asset.

The increase in the industry usage of the NSUF is due in part to the NSUF's proactive engagement with the Gateway for Accelerated Innovation in Nuclear (GAIN) initiative. GAIN is a DOE initiative that is focused on addressing universally recognized issues that currently constrain the domestic nuclear industry. The NSUF supports industry as follows:

- Provides federally funded experimental data for all phases of the innovation cycle
- Maintains a database for critical experimental facilities and equipment that are of interest to GAIN participants
- Develops and maintains the scientific infrastructure through identified equipment needs, measurement technique development and associated expertise.

Proposal submissions to the NSUF have grown at a rate that is outpacing available funding. As a result, the awards have become more competitive.

AWARDED PROJECTS

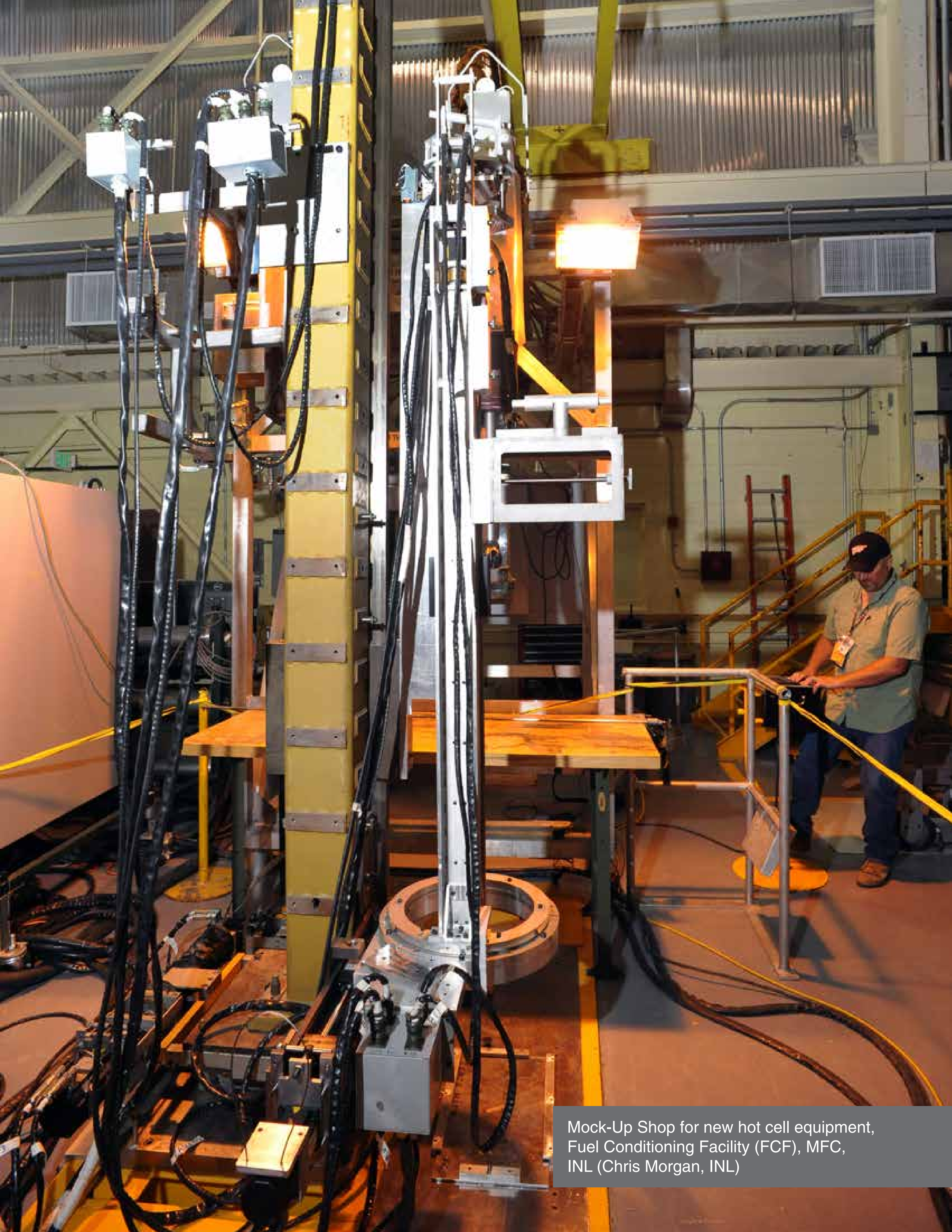
The NSUF offers rapid turnaround experiments (RTEs) three times per year. DOE in FY 2017 awarded a total of 94 RTE projects that will make use of NSUF's capabilities at INL, the Microscopy and Characterization Suite (MaCS) at the Center for Advanced Energy Studies (CAES), and over its distributed network of partner facilities. The tables of FY 2017 awards for the three calls for RTE proposals are below.

FY 2017 First Rapid Turnaround Experiment (RTE) Awards (29)

PI Name	Institution	Title	Facility
Nathan Almirall	University of California Santa Barbara	Microstructural characterization of archival surveillance steels from the Advanced Test Reactor (ATR-2) neutron irradiation experiment	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Weiying Chen	Argonne National Laboratory	*In situ* Observation of Defect Clustering in High Entropy Alloys	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Yiren Chen	Argonne National Laboratory	Microstructural evolution of dual-phase cast stainless steels under irradiation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Mahmut Cinbiz	Oak Ridge National Laboratory	Hydride microstructure at the metal-oxide interface of Zircaloy-4 from H.B. Robinson Nuclear Reactor	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Philip Edmonson	Oak Ridge National Laboratory	Microstructural and -chemical Investigations of the short-term annealing of irradiation-induced late blooming phase precipitates in a high-Ni reactor pressure vessel steel weld	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
David Frazer	University of California, Berkeley	Localized mechanical property assessment of neutron irradiated SiC/SiC composites at elevated temperature	University of California, Berkeley

PI Name	Institution	Title	Facility
Marine Gaumé	CEA Saclay	Study of deformation mechanisms of zirconium alloys under irradiation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Jing Hu	Argonne National Laboratory	*In situ* ion irradiation and high resolution microstructure and microchemistry analysis of accident tolerant fuels	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Kookhyun Jeong	University of Florida	Irradiation of the vanadium carbide coating on HT-9 steel using protons	University of Michigan – Michigan Ion Beam Laboratory
Djamel Kaoumi	North Carolina State University	Ion beam radiation damage assessment in advanced Ferritic/Martensitic (F/M) alloys	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Jie Lian	Rensselaer Polytechnic Institute	Radiation response and microstructure of accident tolerant U_3Si_2 fuels by ion beam irradiation	Center for Advanced Energy Studies – Microscopy and Characterization Suite & Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Jie Lian	Rensselaer Polytechnic Institute	Micromechanical testing of sintered UO_2 fuel pellets with controlled microstructure	University of California, Berkeley
Yinbin Miao	Argonne National Laboratory	Fission gas bubble characterizations of high-energy Xe implanted U_3Si_2	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Yinbin Miao	Argonne National Laboratory	Xe bubble evolution in U_3Si_2: an *in situ* TEM Investigation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Arthur Motta	Pennsylvania State University	Cavity shrinkage in $Fe_{21}Cr_{32}Ni$ under *in situ* ion irradiation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Chad Parish	Oak Ridge National Laboratory	Microstructural recovery in irradiated nanostructured ferritic alloys	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)

PI Name	Institution	Title	Facility
Riley Parrish	University of Florida	Microstructural characterization of 23% burn-up MOX fuel	Idaho National Laboratory – Materials and Fuels Complex
Kumar Sridharan	University of Wisconsin - Madison	Investigation of deformation mechanisms of an intermetallic-strengthened alloy with and without heavy ion irradiation	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Cheng Sun	Idaho National Laboratory	Characterization of ferritic steels Fe-9Cr and 9Cr2WYT Oxide-Dispersion-Strengthened (ODS) alloys irradiated in ATR	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Cheng Sun	Idaho National Laboratory	The window of gas-bubble superlattice formation in bcc metals	University of Michigan – Michigan Ion Beam Laboratory
Benjamin Sutton	EPRI - Electric Power Research Institute	TEM investigation of irradiated austenitic stainless steel alloys	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Elena Tajuelo Rodriguez	Oak Ridge National Laboratory	Changes on viscoelastic behavior, morphology and chemical structure of gamma irradiated calcium silicate hydrates with respect to nonirradiated samples	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Lizhen Tan	Oak Ridge National Laboratory	Radiation-hardening and microstructural Stability of NF709 Austenitic Stainless Steel	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Peter Wells	University of California-Santa Barbara	Investigation of the thermal stability of Mn-Ni-Si precipitates in ion irradiated RPV steels	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Haiming Wen	Idaho State University	Ion irradiation of advanced materials – nanostructured steels and high entropy alloys	University of Wisconsin – Tandem Accelerator Ion Beam and the Characterization Laboratory for Irradiated Materials
Yong Yang	University of Florida	Low temperature Fe-ion irradiation of 15-15Ti steel in different thermo-mechanical states	University of Michigan – Michigan Ion Beam Laboratory
Xinghang Zhang	Purdue University	*In situ* studies of radiation damage in nanostructured austenitic stainless steels	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility



Mock-Up Shop for new hot cell equipment, Fuel Conditioning Facility (FCF), MFC, INL (Chris Morgan, INL)

FY 2017 Second Rapid Turnaround Experiment (RTE) Awards (30)

PI Name	Institution	Title	Facility
Aida Amroussia	Michigan State University	Post-irradiation characterization of ion irradiation damage in Ti-6Al-4V and CP-Ti : Influence of the microstructure and temperature	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Alicia Raftery	Purdue University	Pre-characterization of DISECT U-Mo fuel samples	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Caleb Massey	University of Tennessee Knoxville	Nano-precipitate response to neutron irradiation in model ODS FeCrAl Alloy 125YF	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Cheng Sun	Idaho National Laboratory	Nanoindentation testing of neutron irradiated 304 stainless steels hex-blocks	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Chris Grovenor	Oxford University	*In situ* ion irradiation of second phase particles in zirconium fuel cladding	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Elena Tajuelo Rodriguez	Oak Ridge National Laboratory	Changes on viscoelastic behavior, morphology and chemical structure of gamma irradiated calcium silicate hydrates to 0.96MGy with respect to non-irradiated samples	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Elizabeth Getto	United States Naval Academy	Radiation tolerance of friction stir welded ferritic oxide dispersed steel under ion irradiation	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Emmanuelle Marquis	University of Michigan	ά precipitation in neutron irradiated Fe-9/12Cr alloys	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Gabriel Meric de Bellefon	University of Wisconsin - Madison	Sample preparation for *ex situ* TEM study of deformation-induced twinning and martensite in two 316L austenitic stainless steels: role of stacking fault energy and grain orientation	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Haiming Wen	Idaho State University	APT study of neutron irradiated U-Mo fuel	Center for Advanced Energy Studies – Microscopy and Characterization Suite

PI Name	Institution	Title	Facility
Ian Robertson	University of Wisconsin-Madison	Enhancing radiation tolerance through increasing alloy complexity	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Jacqueline Stevens	AREVA NP Inc.	Hydrogen analysis and oxide characterization of reactor irradiated Zr-Nb alloy	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Janelle Wharry	Purdue University	Radiation induced segregation and phase separation in neutron irradiated FeCrAl alloys	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Jie Lian	Rensselaer Polytechnic Institute	Fission gas behavior and fuel swelling of accident tolerant U_3Si_2 fuels by ion beam irradiation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Jiming Bao	University of Houston	Post irradiation examination of fiber optic temperature sensors for in-pile temperature monitor and control for ATR	Idaho National Laboratory – Materials and Fuels Complex
Jonathan Tatman	Electric Power Research Institute	SEM, EBSD, and TEM investigation of irradiated austenitic stainless steel weldment	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Ju Li	Massachusetts Institute of Technology	The *in situ* observation of radiation resistance mechanism in metal-1D/2D nanocomposites for structural material and fuel cladding of next generation reactors	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Lingfeng He	Idaho National Laboratory	Investigation of gas bubble behavior under ion irradiation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Mahmut Cinbiz	Oak Ridge National Laboratory	*In situ* TEM study of the ion irradiation damage on hydrides in a zirconium alloy for nuclear fuel cladding	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Ming Tang	Los Alamos National Laboratory	Radiation stability study on nuclear waste/spent fuel materials	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility

Experiment Sample,
Materials Laboratory, CAES
(Chris Morgan, INL)

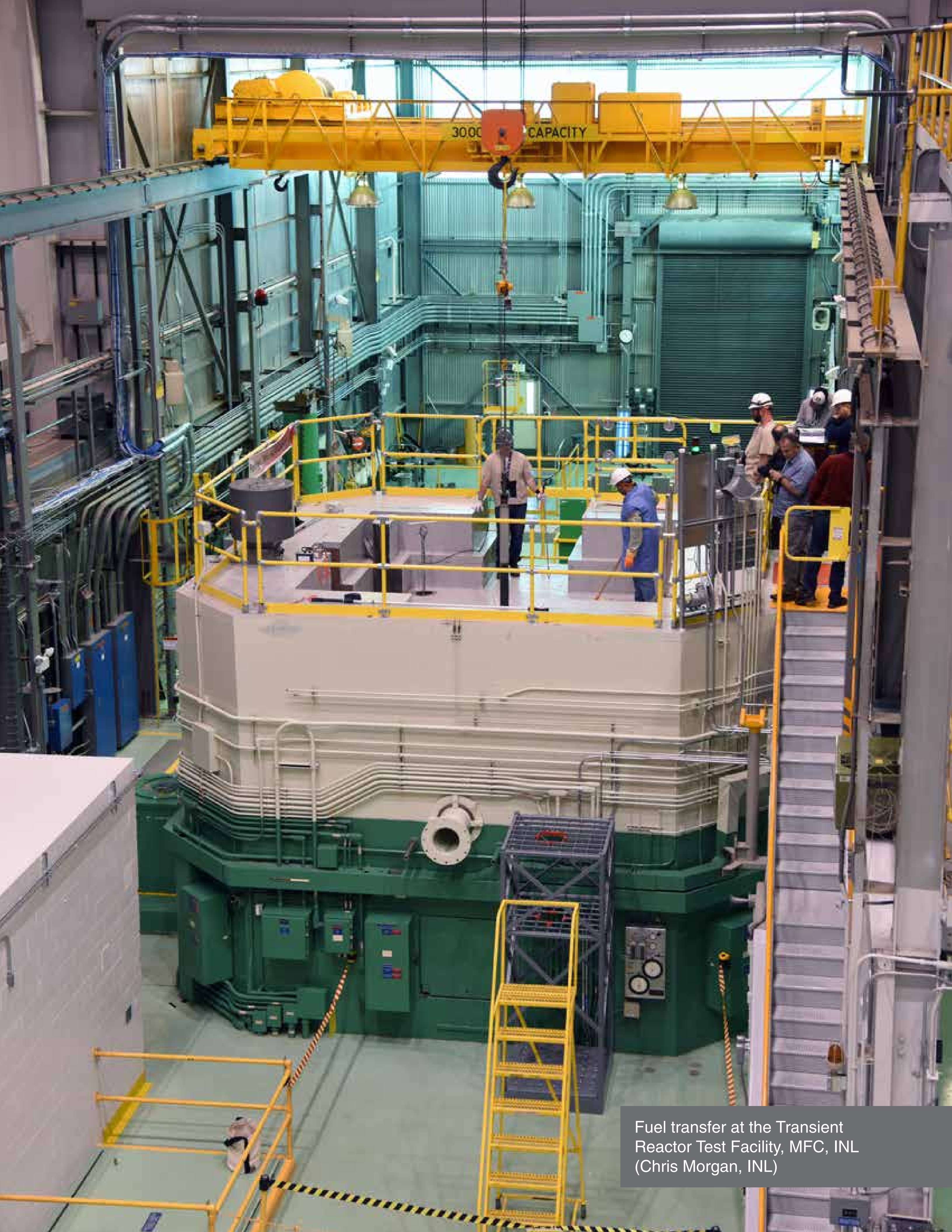
PI Name	Institution	Title	Facility
Mitra Taheri	Drexel University	Quantitative assessment of the role of interfaces and grain boundaries in the development of radiation tolerant nuclear materials	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Niels Cautaerts	SCK•CEN	Characterization of ion irradiated 15-15Ti steel by APT	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Osman El Atwani	Los Alamos National Laboratory	Performance of nanocrystalline and ultrafine Tungsten under irradiation and mechanical extremes	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Ramprashad Prabhakaran	Pacific Northwest National Laboratory	Mechanical characterization of neutron irradiated FSW ODS alloys	Pacific Northwest National Laboratory – Radiochemistry Processing Laboratory
Riley Parrish	University of Florida	Microstructural characterization of 3% burn-up MOX fuel	Idaho National Laboratory – Materials and Fuels Complex
Samuel A. Briggs	Sandia National Laboratories	Study of nanocluster stability in neutron- and ion-irradiated ODS FeCrAl alloys	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Vijay Vasudevan	University of Cincinnati	Effect of grain boundary character and surface treatment on irradiation tolerance of nuclear alloys	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Yutai Katoh	Oak Ridge National Laboratory	Micromechanical properties of interfacial elements in advanced SiC composite and its environmentally protective coatings	University of California, Berkeley
Zhangbo Li	University of Florida	TEM investigation of radiation damage of ferrite in CF-3	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Zheng Zhang	University of Florida	Pore size distribution in U-Mo fuel irradiated to high burnup	Idaho National Laboratory – Materials and Fuels Complex

FY 2017 Third Rapid Turnaround Experiment (RTE) Awards (35)

PI Name	Institution	Title	Facility
Adrien Couet	University of Wisconsin	Characterization of oxide layer on the surface of high temperature water corroded Zircaloy-4 in the presence of Neutron+Gamma and Gamma only	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Adrien Couet	University of Wisconsin	APT and TEM study of redistribution of alloying elements in ZrNb alloys following proton irradiation: effects on in-reactor corrosion kinetics.	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Bei Ye	Argonne National Laboratory	Investigation of irradiation-induced recrystallization in U-Mo fuel	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Ben Maier	University of Wisconsin	APT studies of irradiated cold spray coatings for accident tolerant cladding	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Benjamin Jenkins	University of Oxford	Site-specific APT characterisation of grain boundaries in archival surveillance steels from Advanced Test Reactor (ATR-2) neutron irradiation experiment	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Cem Topbasi	Electric Power Research Institute	*In situ* TEM study of radiation damage effects on the δ-Hydride microstructure in irradiated Zircaloy-4	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Chi Xu	University of Florida	An *in situ* TEM characterization of tensile testing of ion irradiated HT-UPS steel at RT and 400°C	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
David Carpenter	Massachusetts Institute of Technology	Post-irradiation analysis of hybrid metallic coatings on SiC after neutron irradiation 290-330°C	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Djamel Kaoumi	North Carolina State University	Ion irradiation response of nanostructured alloys: *in situ* TEM observations vs. *ex situ* characterization	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility

PI Name	Institution	Title	Facility
Elena Tajuelo Rodriguez	Oak Ridge National Laboratory	Changes on viscoelastic behavior, morphology and chemical structure of gamma irradiated calcium silicate hydrates to 1.94 MGy with respect to non-irradiated samples	Oak Ridge National Laboratory – Low Activation Materials Development and Analysis (LAMDA)
Erik Mader	Electric Power Research Institute	AsTeROID (follow-on AsTeR (Advanced Test Reactor) project to Optimize hydrogen-assisted Irradiation growth and Dimensional stability)	Idaho National Laboratory – Materials and Fuels Complex
Gabriel Meric de Bellefon	University of Wisconsin	The effectiveness of coherent and incoherent twin boundaries in alleviating radiation damage in heavy ion irradiated 316L austenitic stainless steels	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Elizabeth Getto	United States Naval Academy	Radiation tolerance of advanced joining techniques for oxide dispersion strengthened steels under ion irradiation	University of Michigan – Michigan Ion Beam Laboratory
Haiming Wen	Idaho State University	Enhanced irradiation tolerance of high-entropy alloys	University of Wisconsin – Tandem Accelerator Ion Beam and the Characterization Laboratory for Irradiated Materials
Konstantina Lambrinou	SCK•CEN	Study of the factors affecting the radiation tolerance of MAX phases for innovative fuel cladding concepts	University of Michigan – Michigan Ion Beam Laboratory
Kumar Sridharan	University of Wisconsin	Heavy ion irradiation and *ex situ* transmission electron microscopy study of the effectiveness of twin boundaries in alleviating radiation damage in 316 austenitic stainless steels	University of Wisconsin – Tandem Accelerator Ion Beam and the Characterization Laboratory for Irradiated Materials
Lin Shao	Texas A&M University	Post-irradiation observation following high rate self-ion irradiation of previously neutron irradiated 304 stainless steel	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Mukesh Bachhav	Idaho National Laboratory	Microstructure and microchemical characterization of neutron and proton irradiated alloy D9 using APT and TEM	Center for Advanced Energy Studies – Microscopy and Characterization Suite

PI Name	Institution	Title	Facility
Mukesh Bachhav	Idaho National Laboratory	Effect of Phosphorous (P) on precipitation and segregation behavior in neutron irradiated Reactor Pressure Vessel (RPV) steels in the Advanced Test Reactor (ATR-2): An atom probe study.	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Nathan Almirall	University of California, Santa Barbara	APT investigations of nm-scale precipitates in advanced RPV super clean steels in the UCSB Advanced Test Reactor (ATR-2) neutron irradiation experiment	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Niels Cautaerts	SCK•CEN	Hardness profiling of ion irradiated 15-15Ti cladding steel using CSM nano-indentation	University of California, Berkeley
Philip Edmondson	Oak Ridge National Laboratory	*In situ* amorphization studies of forsterite, diopside and quartz under ion irradiation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Riley Parrish	University of Florida	Microstructural characterization of 13% burn-up MOX fuel	Idaho National Laboratory – Materials and Fuels Complex
Riley Parrish	University of Florida	Microstructural characterization of 21% burn-up MOX fuel	Idaho National Laboratory – Materials and Fuels Complex
Rodney Ewing	Stanford University	Radiation tolerance of $M_{n+1}AX_n$ phase nuclear fuel cladding materials	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Sebastien Teysseyre	Idaho National Laboratory	Characterization of grain boundary damage in highly irradiated specimens exposed to irradiation assisted stress corrosion cracking	University of California, Berkeley
Sebastien Teysseyre	Idaho National Laboratory	Characterization of the stability of the microstructure of novel ODS alloys	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Steven Zinkle	University of Tennessee	Irradiation responses of ultrastrong nano precipitation martensite steel	Center for Advanced Energy Studies – Microscopy and Characterization Suite



Fuel transfer at the Transient Reactor Test Facility, MFC, INL (Chris Morgan, INL)

PI Name	Institution	Title	Facility
Tianyi Chen	Oak Ridge National Laboratory	Radiation hardening and microstructural stability of NF709 austenitic stainless steel	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Todd Allen	University of Wisconsin	Examining microstructural differences in irradiated HT9, correlated with differences in processing prior to irradiation	Idaho National Laboratory – Materials and Fuels Complex
Todd Allen	University of Wisconsin	IVEM investigation of defect evolution in FCC and BCC HEAs during heavy ion irradiation	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Weiying Chen	Argonne National Laboratory	Fundamental study of alloying complexity effects on the irradiation process in high entropy alloys	Argonne National Laboratory – Intermediate Voltage Electron Microscope Tandem Facility
Yong Yang	University of Florida	Characterize the irradiated microstructure and understand the fission product behavior in an irradiated and safety tested AGR-1 TRISO fuel particle new proposal	Center for Advanced Energy Studies – Microscopy and Characterization Suite
Yuanyuan Zhu	Pacific Northwest National Laboratory	Microstructural examination of neutron irradiated Al-$HfAl_3$ metal matrix composite materials for application to neutron spectrum modification in nuclear reactors	Pacific Northwest National Laboratory – Radiochemistry Processing Laboratory
Zheng Zhang	University of Florida	Pore size distribution in U-Mo fuel irradiated to low burnup	Idaho National Laboratory – Materials and Fuels Complex

FY 2017 Consolidated Innovative Nuclear Research (CINR) Awards

In FY 2017, DOE selected five university, five national laboratory, and five industry-led projects that will take advantage of NSUF capabilities to investigate important nuclear fuel and material applications. DOE will support six of these projects with a total of $2.3 million in research funds, and all 15 of these projects will be supported by more than $10 million in facility access costs and expertise for experimental neutron and ion irradiation testing, post-irradiation examination facilities, synchrotron beamline capabilities, and technical assistance for design and analysis of experiments through the NSUF.

Joint R&D with NSUF Access

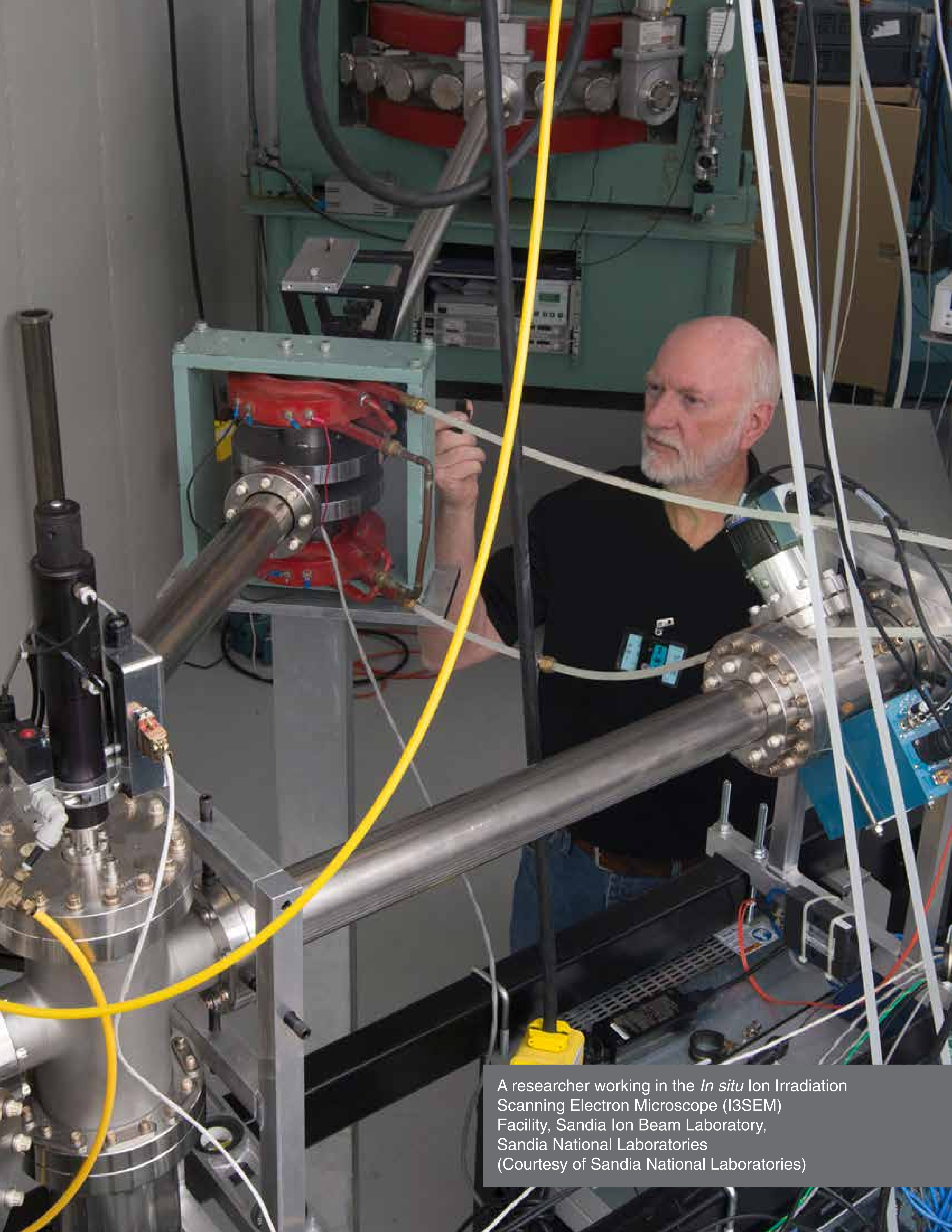
Title	Institution	Project Description
NSUF Project 17-12527: Additive Manufacturing of Thermal Sensors for in-pile Thermal Conductivity Measurement	Boise State University	Researchers will develop and demonstrate an additive manufacturing approach to fabricate nonintrusive and spatially resolved sensors for in-pile thermal conductivity measurement. The team will print thermal conductivity sensors onto fuel components using an aerosol jet printing approach, and study in-pile performance of the printed sensors through irradiation and post-irradiation testing. This research has the potential to establish a new sensor manufacturing paradigm for the nuclear industry.
NSUF Project 17-12573: Performance of SiC-SiC Cladding and Endplug Joints under Neutron Irradiation with a Thermal Gradient	General Atomics	Researchers will investigate the effects of temperature and thermal gradients on the irradiation performance and stability of joints in silicon carbide (SiC) cladding and endplug geometries. The project will fill a gap in understanding the performance SiC joint performance which will advance the development of accident tolerant fuels.
NSUF Project 17-13004: Capacitive Discharge Resistance Welding of 14YWT for Cladding Applications	Los Alamos National Laboratory	Researchers will join cladding tubes of 14YWT alloy and a ferritic ODS alloy using capacitor-discharge resistance welding (CDRW), a rapid, solid-state welding process with very low heat input. The CDRW process is especially well suited for cladding applications. The project will provide a significant advance in the state of the knowledge for joining of 14YWT and ferritic ODS materials and will permit their broader use with increased confidence.

Title	Institution	Project Description
NSUF Project 17-13050: Correlation between Microstructure and Mechanical Properties of Neutron-Irradiated Ferritic-Martensitic and Austenitic Steels	Oak Ridge National Laboratory	Researchers will identify correlations between microstructures and mechanical properties of neutron irradiated advanced ferritic-martensitic and austenitic steels through comprehensive experimental post-irradiation examinations, coupled with thermodynamics, kinetics and microstructural hardening modeling of selected samples that are relevant to Light Water Reactors. Results from other ongoing studies and literature data of similar alloys will be collected and compared to complement the correlations.
NSUF Project 17-12797: *In Situ* Ion Irradiation to Add Irradiation Assisted Grain Growth to the MARMOT Tool	Pennsylvania State University	Researchers will add the capability to model irradiation assisted grain growth to the MARMOT tool by using *in situ* ion irradiation of UO_2 to quantify the effect of irradiation on grain growth. The team will investigate the hypothesis that irradiation assisted grain growth is caused by thermal spikes resulting from atom collisions. The model added to MARMOT will couple the existing grain growth model to a heat conduction simulation using a stochastic heat source describing the thermal spike.
NSUF Project 17-13073: Radiation Effects on Optical Fiber Sensor Fused Smart Alloy Parts with Graded Alloy Composition Manufactured by Additive Manufacturing Processes	University of Pittsburgh	Researchers will establish the foundation for converging disciplines of multi-functional fiber optic sensors and additive manufacturing for smart part fabrications for nuclear energy applications, especially for in-pile applications. Using advanced laser fabrication techniques, the team will develop both high-temperature stable point sensors and distributed fiber sensors for high spatial resolution measurements in radiation-hardened silica and sapphire fibers.

NSUF Access Only

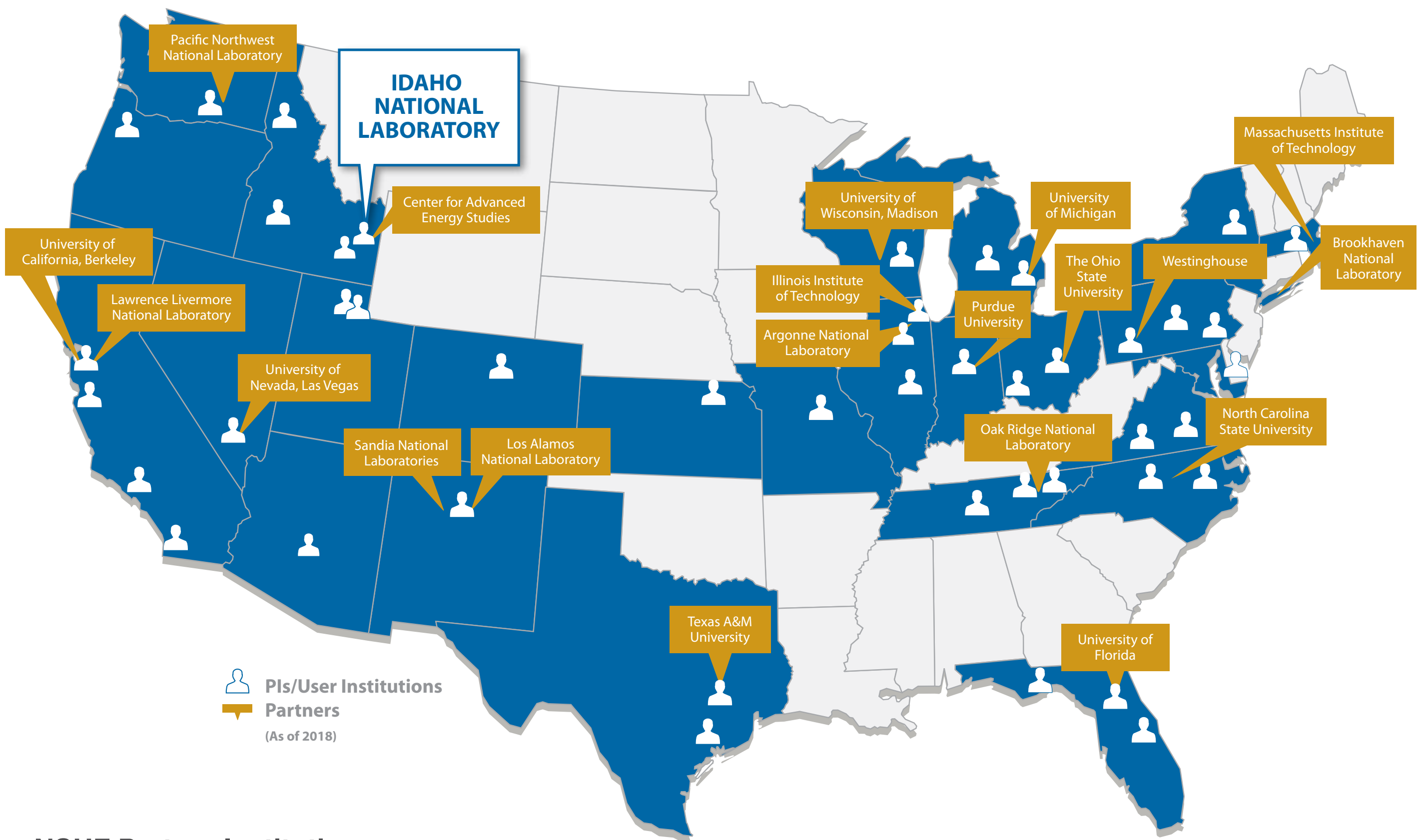
Title	Institution	Project Description
NSUF Project 17-13007: Irradiation of Advanced Neutron Absorbing Material to Support Accident Tolerant Fuel	AREVA	To provide irradiation and post-irradiation examination program for four neutron absorber materials. The team will evaluate four pellets of each absorber type irradiated to target doses of 1.3 and 2.7 x 10^{22} n/cm^2. Following neutron irradiation, examinations will focus on pellet integrity using optical microscopy and dimensional measurements to characterize irradiation induced swelling. This scope of work will utilize HFIR and hot cells at ORNL.
NSUF Project 17-12985: Irradiation, Transient Testing and Post Irradiation Examination of Ultra High Burnup Fuel	Electric Power Research Institute, Inc.	Researchers will provide experimental data on fuel fragmentation's role in fuel burnup to make the case for increasing the regulatory burnup limit past 62 Gwd/MTU. The scope of work involves re-irradiation of high burnup fuel at the appropriate power levels in ATR followed by transient testing, both out of reactor and in TREAT.
NSUF Project 17-12957: X-ray Characterization of Atomistic Defects Causing Irradiation Creep of SiC	Oak Ridge National Laboratory	The proposed work at the NSLS-II Facility will conduct synchrotron-based XRD experiments on high-purity SiC neutron irradiated with and without applied stress. The outcome from this work will provide critical experimental data to understand underlying mechanism of irradiation creep of SiC and will consequently advance the thermo-mechanical model of the SiC cladding of LWRs.
NSUF Project 17-13088: Improved Understanding of Zircaloy-2 Hydrogen Pickup Mechanism in BWRs	Electric Power Research Institute, Inc.	Researchers will study why Zircaloy-2 material shows high hydrogen pickup and variability in BWR environments by investigating the correlation between the irradiated Zircaloy-2 oxide layer resistivity and hydrogen pickup. The scope of work will include *in situ* electrochemical impedance spectroscopy (EIS) measurements on pre-irradiated channel and water rod samples as well as post-irradiation characterization of the same materials using Transmission Electron Microscopy and Scanning Electron Microscopy at PNNL.
NSUF Project 17-12976: Study of the Irradiation Behavior of Fast Reactor Mixed Oxide Annular Fuel with Modern Microstructural Characterization to Support Science Based Model Validation	Idaho National Laboratory	Researchers will grow the available database of post irradiation data available for annular mixed oxide (MOX) fuel irradiated in fast spectrum reactors by examining irradiated fuel from the FO-2 irradiation. The data collected in this project would be used to validate models currently being developed at the Japanese Atomic Energy Agency (JAEA) for fuel performance models that seek to simulate MOX fuel behavior and will be implemented in BISON.

Title	Institution	Project Description
NSUF Project 17-12849: Simulation of Radiation and Thermal Effects in Advanced Cladding Materials	Pacific Northwest National Laboratory	Researchers will develop atomic scale data on the phase stability and thermo-mechanical properties of FeCrAl accident tolerant cladding under the combined effects of radiation and elevated temperature. The goal is to ultimately provide materials parameters for the MARMOT code and develop predictive physics-based models for the BISON code.
NSUF Project 17-13211: Positron Annihilation Studies of Neutron Irradiated Ferritic Alloys	University of Illinois, Urbana-Champaign	Researchers will quantitatively measure sub-5nm defect structures, particularly tiny vacancy clusters, which are inaccessible using any other microstructural analysis techniques. The project will use North Carolina State University's PALS and DBS systems to study nanoscale defect structures in ATR neutron irradiated ferritic and ferritic/martenistic alloys.
NSUF Project 17-12853: HPC Access to Advance Understanding of Fission Gas Behavior in Nuclear Fuel	University of Tennessee, Knoxville	Researchers will develop high-performance simulation tools to predict fission gas bubble evolution in nuclear fuel. The scope of work in this project includes access to 10 million CPU hours of high performance computing (HPC) resources each year for two years.
NSUF Project 17-13106: Radiation Effects on Zirconium Alloys Produced by Powder Bed Fusion Additive Manufacturing Processes	Westing-house Electric Company	Researchers will collect post irradiation examination data for additive manufactured Zircalloy-2 materials for LWR fuel applications. The scope of work includes PIE of a previously irradiated zirconium material that was fabricated using laser powder bed fusion. The sample was irradiated at MIT's reactor and PIE will be conducted at Westinghouse's Churchill hot cell facility.



A researcher working in the *In situ* Ion Irradiation Scanning Electron Microscope (I3SEM) Facility, Sandia Ion Beam Laboratory, Sandia National Laboratories (Courtesy of Sandia National Laboratories)

NSUF ACROSS THE NATION



NSUF Partner Institutions

Argonne National Laboratory
Brookhaven National Laboratory
CAES Center for Advanced Energy Studies
Berkeley University of California
UF University of Florida
Illinois Institute of Technology
UNLV
Lawrence Livermore National Laboratory
University of Michigan
Massachusetts Institute of Technology
NC State University
Oak Ridge National Laboratory
The Ohio State University
Pacific Northwest National Laboratory
Purdue University
Sandia National Laboratories
SCK•CEN Studiecentrum voor Kernenergie Centre d'Etude de l'Energie Nucléaire
A&M
Westinghouse
Wisconsin University of Wisconsin–Madison

INL Idaho National Laboratory

Participants at the first NSUF Partner Facilities Workshop, Center for Advanced Energy Studies (CAES) (May 2017) (Chris Morgan, INL)

NSUF User Institutions

Arizona
Arizona State University

California
Electric Power Research Institute (EPRI)
General Atomics
Stanford University
University of California, Berkeley
University of California, Santa Barbara

Colorado
Colorado School of Mines

Florida
Florida State University
University of Central Florida
University of Florida

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

Illinois
Argonne National Laboratory
Illinois Institute of Technology
University of Illinois

Indiana
Purdue University

Kansas
Kansas State University

Maryland
The Johns Hopkins University
United States Naval Academy

Massachusetts
Massachusetts Institute of Technology

Michigan
Michigan State University
University of Michigan

Missouri
University of Missouri

New York
Rensselaer Polytechnic Institute (RPI)

Nevada
University of Nevada – Las Vegas

New Mexico
Los Alamos National Laboratory
Sandia National Laboratories

North Carolina
GE Hitachi Nuclear Energy
North Carolina State University

Ohio
The Ohio State University
University of Cincinnati

Oregon
Oregon State University

Pennsylvania
Drexel University
Pennsylvania State University
University of Pittsburgh
Westinghouse

Tennessee
Oak Ridge National Laboratory
University of Tennessee
Vanderbilt University

Texas
Texas A&M University
University of Houston

Utah
University of Utah
Utah State University

Virginia
AREVA NP
Virginia Commonwealth University

Washington
Pacific Northwest National Laboratory

Wisconsin
University of Wisconsin

Australia
Australian Nuclear Science and Technology Organization

Belgium
SCK•CEN

France
CEA Saclay

United Kingdom
Oxford University
University of Liverpool
University of Manchester

The Control Room for the Advanced Test Reactor (ATR), INL (Chris Morgan, INL)

NSUF AWARDED PROJECTS

This section contains reports on projects awarded through the NSUF and completed in FY 2017.

INL Advanced Test Reactor Complex Named ANS Nuclear Historic Landmark

May 2017

The American Nuclear Society (ANS) dedicated the Idaho National Laboratory (INL) Advanced Test Reactor Complex as an ANS Nuclear Historic Landmark at a May 2017 award ceremony. The complex housed the original Materials Testing Reactor (MTR) built in 1952, the Engineering Test Reactor (ETR) in 1957, and the Advanced Test Reactor (ATR) that began operation in 1967.

At the INL Advanced Reactor Complex dedication, ANS President Andrew Klein said, "The INL Advanced Reactor Complex has valuable history in nuclear energy research, safety, and security. The work performed at the complex today will help us meet current nuclear challenges and develop future advanced reactor technologies."

Today, the ATR remains the largest test reactor in the world with a unique serpentine fuel arrangement. The available experimental space of the ATR is shared by the U.S. Department of Energy, commercial users, other nations, Nuclear Science User Facilities users, and the U.S. Navy. It is still revered as the nation's premier resource for fuels and materials irradiation testing, nuclear safety research, and nuclear isotope production.

The INL Advanced Test Reactor was recognized in 2016 with the ANS Meritorious Performance in Operations Award. The national award recognizes outstanding performance in the operation of nuclear facilities. The Advanced Test Reactor is marking its 50-year anniversary in 2017.

Nanohardness measurements on neutron irradiated steel samples for next generation reactors

Tarik Saleh – Los Alamos National Laboratory – tsaleh@lanl.gov

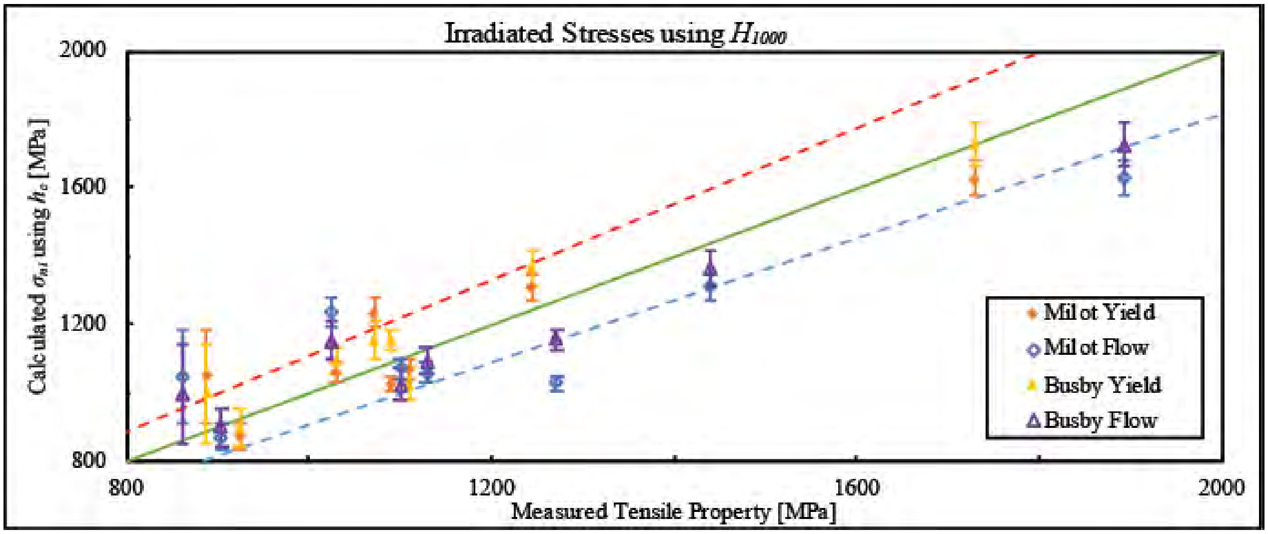


Figure 1. Plot comparing predicted σ_{ni} using H_{1000} values from h_c to σ_y and σ_{flow} for the ATR-irradiated samples. Note that $\sigma_{ni,y} = \sigma_{ni,flow}$ for the Milot correlation. Green line indicates 1:1 agreement, while red/blue lines indicate +/– 10%.

Los Alamos National Laboratory has samples from a large number of neutron irradiation experiments in their hot cells. Most of these samples have, or will have, macroscopic mechanical test data (tensile or shear punch tests) performed on them. This proposal covered sending undeformed sections of these irradiated samples to UC Berkeley for nanoindentation and imaging of samples. The combination of the macroscopic mechanical property measurements and the nanoindentation data fed into constitutive modeling of irradiated materials, resulting in a mechanistic understanding of irradiation effects on the mechanical behavior of next generation and accident tolerant cladding materials.

Project Description

The mechanical testing data at different length scales (macroscopic to nano) at varying doses and irradiation temperatures in assorted materials will increase the understanding of fundamental irradiation effects in candidate cladding materials for next generation reactors and current reactor accident tolerant claddings (Figure 1). The data collected from this work can feed directly into finite element method (FEM)-based models of true stress-strain constitutive behavior of irradiated materials, including strain rate and multiscale behavior. This ability to extrapolate macroscopic mechanical property data from small samples via small scale testing and modeling will allow for more efficient use of neutron irradiation experiments, leading to advances in understanding high dose materials behavior.

Figure 2. Photograph of polishing setup in radioactive material fume hood in 1140 Etcheverry Hall, UC Berkeley. An overview of the space including a MiniMet semiautomatic polisher, lead walls, and surface sheets for shielding.

Accomplishments

Initial work, beginning in 2016, included shipping samples from Los Alamos National Laboratory Wing 9 hot cells to UC Berkeley and continued development of a robust shielded sample holder for polishing the irradiated samples in hoods, and handling and transfer between instruments (Figures 2 and 3). Work on characterizing the Advanced Test Reactor (ATR) irradiated samples continued in 2017. Both micro and nano scale Berkovich indentation was performed on all eight samples in both irradiated and as-received conditions. This data was cross-compared and also compared to previous Vickers microhardness data to determine effects of test size and indenter tip geometry. This analysis led to determining nonstandard data processing methods to accurately compare data between traditional and instrumented indentation methods that depends on expected sample strain-hardening characteristics. As many of the empirical correlations linking hardness to bulk tensile properties use optically measured Vickers microhardness, understanding the link between that measure of hardness and the results from nanoindentation is vital.

After determining best practices to process the nanoindentation data, hardness results were directly compared to LANL-measured tensile properties utilizing three different empirical relationships. The efficacy of each correlation was analyzed on an alloy-by-alloy and overall basis

Access to LANL expertise and irradiated samples through the NSUF has been pivotal in the development of radioactive material handling and testing capabilities at UC Berkeley. In addition, the data generated from this collaboration has played a key role in advancing not only my thesis work but other collaborative projects within DOE NEUP.

— David Krumwiede, Student

Figure 3. (Left) Photograph and (right) cross-sectional schematic of updated three-piece sample holder design for polishing small irradiated samples in hoods, shielded storage, and secure transferring of samples between hoods and instruments.

NSUF Fuels & Materials Library
08-139 UCSB samples

for a multitude of indentation curve processing techniques. Details of these analyses have been published in journal articles (publications [1,2]) and a Ph.D. dissertation. In summary, with a proper prediction of a sample's strain-hardening behavior, both the yield stress and 8% flow stress can be estimated to within 5% of the measured property. Figure 1 shows correlation of measured tensile and flow stress on macroscopic irradiated samples compared to predicted data derived from nanohardness data that was measured on the same samples. For a full discussion, see publications [2].

The ability to use nanoindentation to predict tensile properties is important for future works for two reasons. First, future experiments will investigate samples from BOR60 that were irradiated as TEM discs, which were tested in a shear punch system. The shear properties from these tests can be correlated with tensile properties. As such, nanohardness can be correlated with standard penetration test data via a two-step process. This work validates one of those steps. Second, analysis of ion irradiated samples requires the use of small scale mechanical testing, such as nanoindentation. The ability to extrapolate to bulk properties is an essential screening tool for an alloy's irradiation performance.

Future Activities

This research will expand beyond the initial irradiation conditions in the Spallation Target Irradiation Program (STIP) and ATR irradiations to encompass samples from different irradiation conditions in the ATR and much higher dose irradiations including BOR60 irradiations as well as archived samples from the Fast

Formalizing relations between nanoscale and macroscopic mechanical data via experiment modeling will allow for more efficient testing and better understanding of high dose cladding material for next generation reactors.

Flux Test Facility. This will generate a bigger database of macro and nano mechanical properties for model and technique validation.

Publications

[1.] Krumwiede, David L; Abad, M.D.; Saleh, Tarik A.; Maloy, Stuart Andrew; Odette, G. Robert ; et al. "Initial Studies on the Correlation of Nanohardness to Engineering-Scale Properties of Neutron-Irradiated Steels." International Congress on Advances in Nuclear Power Plants, ICAPP 2016 Volume 1, 2016, Pages 224-229

[2.] D.L. Krumwiede, T. Yamamoto, T. Saleh, S.A. Maloy, G.R. Odette, and P. Hosemann, "Direct comparison of nanoindentation and tensile test results on reactor-irradiated materials," Journal of Nuclear Materials. Volume 504, June 2018, Pages 135-143

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Los Alamos National Laboratory	Chemical and Metallurgical Research Facility (Wing 9)
University of California, Berkeley	Nuclear Materials Laboratory
Collaborators	
Los Alamos National Laboratory	Dr. Tarik A. Saleh (principal investigator), Dr. Stuart A. Maloy (co principal investigator)
University of California, Berkeley	Dr. Peter Hosemann (collaborator), David Krumwiede (collaborator)

Critical evaluation of radiation induced segregation in nickel base alloys subjected to proton irradiation

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The data related to radiation induced segregation can impact the material selection for the life extension of current operating light water reactors with better safety and efficiency.

Radiation induced segregation (RIS) behavior is used as one of the indicators of the radiation resistance of materials for its potential impacts on irradiation assisted stress corrosion cracking (IASCC). The RIS behavior of several commercial austenitic alloys (alloy 625, 625Plus, 625DA, 725, 800 and 310) was investigated. All the alloys were proton irradiated to 5 dpa at 360°C in the Michigan Ion Beam Laboratory (MIBL). Energy-dispersive X-ray spectroscopy (EDX) scans were performed on a Talos scanning transmission electron microscope (STEM) in the Low Activation Materials Design and Analysis Laboratory at ORNL. RIS results show that all the random high-angle grain boundaries of these austenitic alloys follow the same pattern of significant Cr and Fe depletion and Ni enrichment. RIS of minor elements such as Si in austenitic steels were tracked as well. These results can provide the knowledge to identify the most promising radiation tolerant alloys with good resistance to IASCC.

Project Description

The life extension of current existing reactors and design of next generation nuclear reactors require advanced materials that can maintain structural integrity in harsh radiation environments. Over the past years, the Electric Power Research Institute (EPRI) tried to identify all possible degradation mechanisms and their potential effects on the reactor components. Void swelling, stress corrosion cracking (SCC), IASCC, and fracture toughness are the major concerns as materials experience high irradiation levels. However, most in-core structures were built with austenitic stainless steels, which are susceptible to degradation at a relative early time during service. Thus, replacement components may become a necessity. EPRI initiated the Advanced Radiation Resistant Materials (ARRM) program to address these issues. The ARRM project is aimed at identifying promising candidate alloys that can replace austenitic stainless steels, which suffer from serious IASCC in light water reactor (LWR) environments. Reactors using these alloys can operate with better efficiency and lower cost of maintenance and repair. The successful completion of this proposed project will facilitate the completion of the ARRM project, which aligns well with the first program goal: development of new nuclear generation technologies and proliferation-resistant LWR and fuel cycle technologies, and development and deployment of next

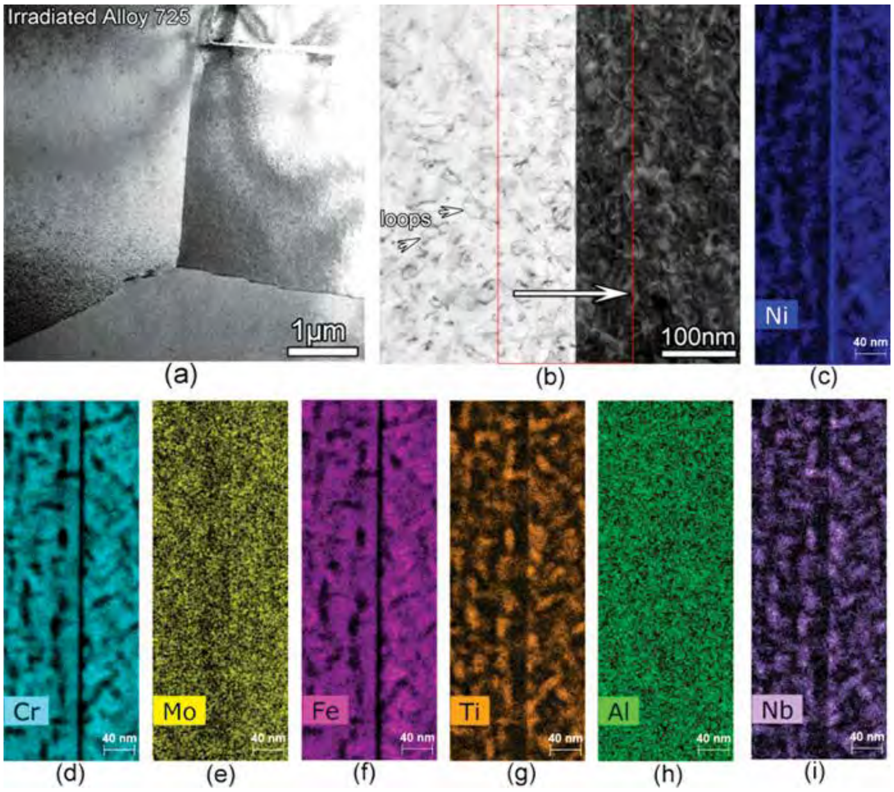


Figure 1. EDX mapping of irradiated alloy 725: (a) bright field image of a triple junction, (b) the target grain boundary and mapping area. EDX mapping results of individual elements (c) Ni (d) Cr (e) Mo (f) Fe (g) Ti (h) Al and (i) Nb.

generation advanced reactors and fuel cycles in the longer term. Also, the second phase of the ARRM project is targeting the development of superior degradation resistant alloys, which would allow a larger margin of design and even tolerance for beyond-design-basis events. This addresses the second program goal: enhancing the safety of the nation's nuclear infrastructure to meet national energy and environmental needs.

Accomplishments

IASCC is identified as one of the primary degradation mechanisms for materials of core components in LWR systems. Among all the factors, RIS, especially Cr depletion at grain boundaries (GBs), is suspected to be a contributor to the susceptibility of IASCC. Ni base alloys and austenitic stainless steels typically exhibit

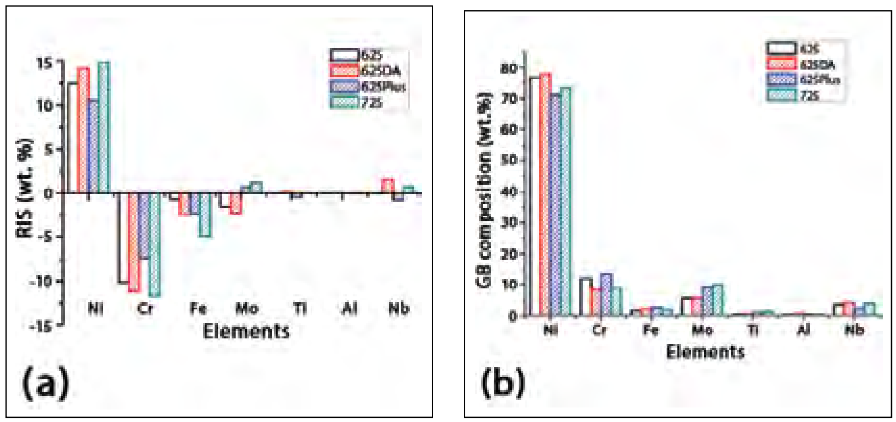


Figure 2. Comparison of (a) irradiation induced segregation and (b) GB chemistry in several Ni based alloys.

excellent mechanical properties and good resistance to corrosion. However, the data related to RIS are limited to alloys 316 and 304. The goal of this study is to evaluate the RIS behavior of nickel- and iron-based austenitic alloys 625, 625Plus, 625DA, 725, 800 and 310, which are rarely reported in literature.

Random high-angle GBs are typically susceptible to IASCC. These GBs possess a length fraction of ~30% in alloy 625, ~22% in alloy 625Plus, ~46% in alloy 718, ~32% in alloy 725, ~49.3% in alloy 310, and ~32.4% in alloy 800, based on the electron backscatter diffraction (EBSD) study.

The first issue is to determine random high-angle grain boundaries. The triple junctions are applied to distinguish the random high-angle GB from a twin boundary. The Kikuchi patterns for both grains are also recorded for precise misorientation calculation. After the targeted GB type is determined, the grain boundary is tilted to an edge-on condition for EDX analysis. The EDX mappings are performed across the GBs. The 2D mapping data are transferred into 1D lines, corresponding in area to the width of the box (>100nm) across the GBs in Figure 1. Good signal to noise ratio was achieved.

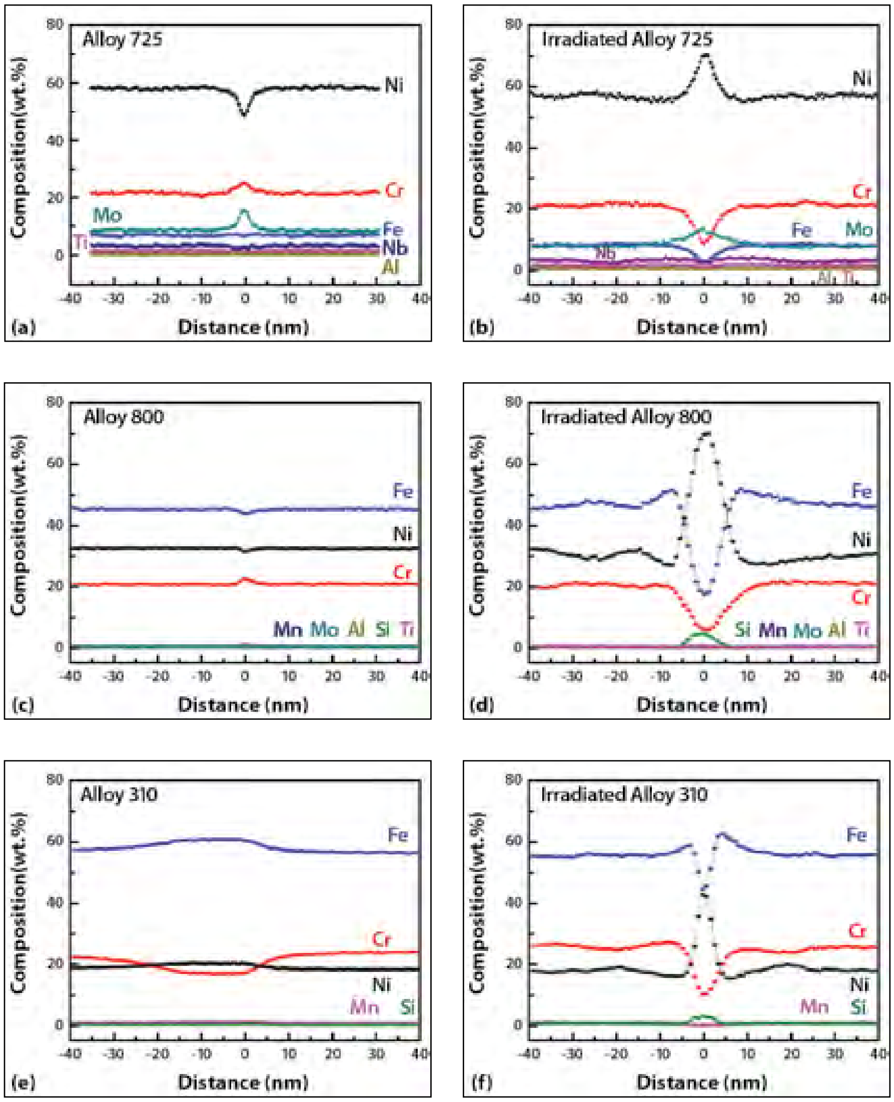


Figure 3. Grain boundary segregation before and after irradiation in several commercial grade alloys: (a) as-received (AR) alloy 725 and (b) irradiated alloy 725, (c) AR alloy 800 and (d) irradiated alloy 800, (e) AR alloy 310, and (f) irradiated alloy 310.

Based on this method, grain boundary segregation was observed in all alloys investigated. In Ni based alloys, depletion of Ni and enrichment of Cr and Mo were observed in the as-received alloys. The segregation seems smaller in alloy 625DA, likely due to a lower aging temperature. After irradiation, significant Ni enhancement was observed in the Ni based alloy while Cr and Fe were always depleted. Mo was a slow diffuser that is enriched in the as-received materials and depleted under irradiation. The segregation of other elements is either insignificant or beyond the resolution of equipment. A comparison of RIS in the Ni based alloys is summarized in Figure 2. 625DA shows the lowest GB Cr level of around 8 wt%. The Ni concentration increases up to 70 wt%, which is around 10 wt% above the bulk composition. The difference between different Ni based alloys is insignificant. The RIS in stainless steels are presented separately due to their different chemical composition.

Segregation in as-received stainless steels is insignificant, as shown in Figure 3 where only a small amount of segregation was observed. However, the RIS is significant. The general pattern is the same as in the Ni based alloys in which significant Ni diffuses to the GB and Cr and Fe diffuse away from the GB. The RIS of Si is pronounced, typically about three times its nominal value. In alloy 800, the amount of Si at the GB is comparable to that of Cr while the nominal composition of Cr is more than 40 times that of Si in the alloy.

Alloy 800, with 31Ni-22Cr-45Fe, shows the highest magnitude of radiation induced segregation. Both Ni based and Fe based alloys show less segregation compared to alloy 800. The segregation behaviors of the elements are consistent among different alloys: Ni is always enriched after irradiation, and Cr and Fe are depleted. These data constitute important knowledge of the RIS in commercial grade alloys.

Dr. Chad M. Parish at Oak Ridge National Laboratory is acknowledged for the successful completion of the project.

Publications

[1.] M. Song, M. Wang, G. Was, L. Nelson, R. Pathania, Irradiation Assisted Stress Corrosion Cracking (IASCC) of Nickel-base Alloys in Light Water Reactors Environments Part I: Microstructure Characterization, 18th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, August 13-17, 2017, Portland, Oregon, USA

[2.] W. Kuang, M. Song, C. Parish, G. Was, Microstructural Study on the Stress Corrosion Cracking of Alloy 690 in Simulated Pressurized Water Reactor Primary Environment, 18th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, August 13-17, 2017, Portland, Oregon, USA

Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Oak Ridge National Laboratory	Low Activation Materials Design and Analysis Laboratory
Collaborators	
Electric Power Research Institute	Raj Pathania (co principal investigator)
Oak Ridge National Laboratory	Keith Leonard (co principal investigator)
University of Michigan	Gary Was (co principal investigator), Miao Song (principal investigator), Mi Wang (co principal investigator)

A comparison of mechanical properties of ion and neutron irradiated Fe-9Cr

Professor Steve Roberts – University of Oxford – steve.roberts@materials.ox.ac.uk

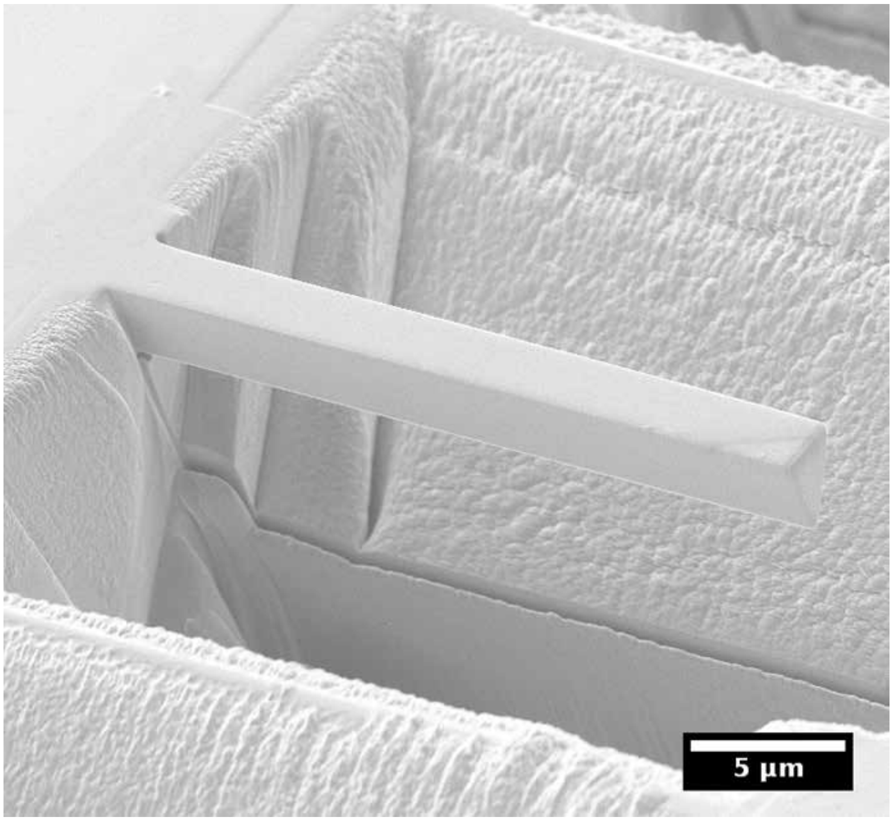


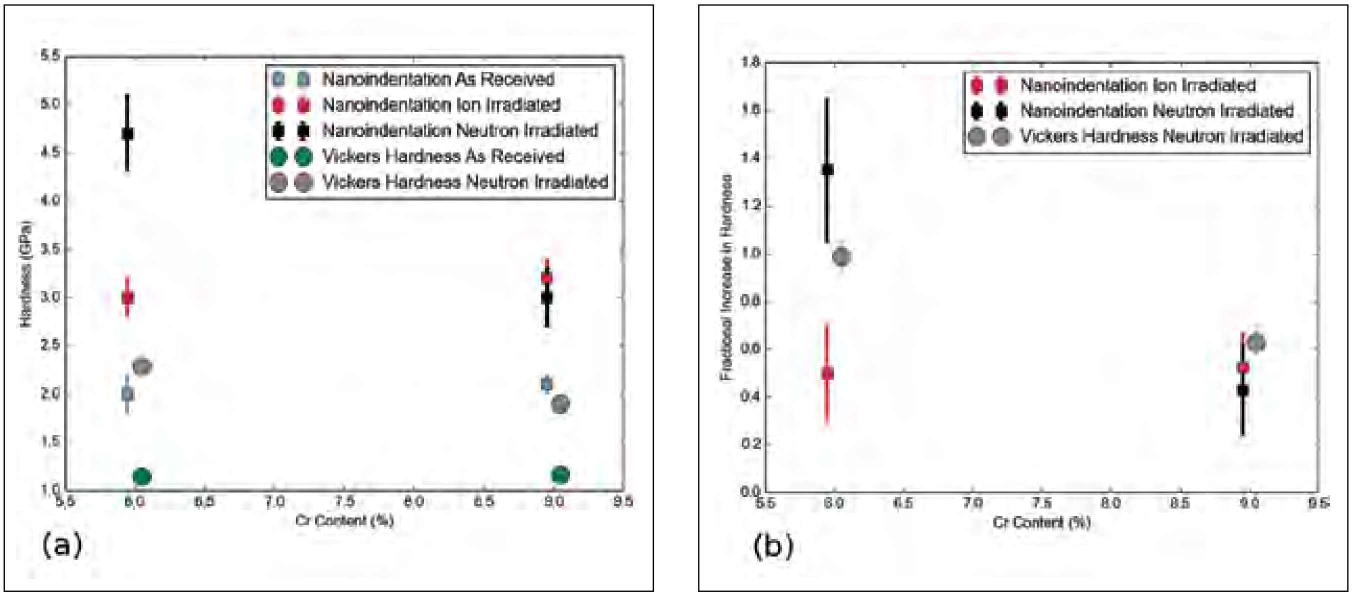
Figure 1. A FIB-milled microcantilever in neutron irradiated Fe-9Cr alloy. These cantilevers are deformed, using a nanoindenter as a loading device, to give yield and flow data from very small volumes of material.

Ion irradiation can be a cheaper and faster surrogate for neutron irradiation in the study of radiation damage effects, but whether it is a valid surrogate or not can depend strongly on small changes in alloy composition. These experiments have helped us understand this better.

Neutron irradiation campaigns are expensive, take a significant amount of time to complete, and offer only limited control over experimental conditions. Radioactivity is also induced in samples, requiring specialized handling facilities, such as provided by the NSUF, that are not routinely available to most researchers.

Heavy ion irradiation provides a faster, cheaper alternative to neutron irradiation where good control over experimental parameters allows a methodical approach towards building an understanding of radiation damage, without the need for irradiated material handling facilities.

This work uses small scale mechanical testing and microstructural analysis techniques to study how well heavy ion irradiation replicates neutron irradiation in a nuclear relevant model alloy.



Project Description

The aim of this project is to compare the mechanical properties and microstructure of samples of an Fe-9Cr model alloy irradiated under the same conditions using heavy ions at the Joint Accelerators for Nanosciences and NUclear Simulation (JANNuS) facility in Saclay-Orsay, France, and with neutrons in the Advanced Test Reactor (ATR) at INL.

Low activation ferritic/martensitic steels with 9–12% Cr are favored candidate materials for use in future fission and fusion reactors due to both their superior swelling resistance at high doses compared to austenitic steels and an observed minimum in ductile to brittle transition temperature increase at this composition. Understanding the microstructural development of these alloys under irradiation is critical for the design, safe operation, and commercial viability of future reactors.

Heavy ion irradiation has often been used to study radiation damage in these materials. The damage produced by this technique has several neutron atypical characteristics, so it is important to validate these experiments through comparisons with neutron irradiation made possible through access to facilities provided by the NSUF.

The shallow damage layer produced by heavy ion irradiation necessitates the use of small scale mechanical

Figure 2. (a) Hardness and (b) fractional increase in hardness after ion and neutron irradiation for FeCr model alloys. Points of the same composition have been spread horizontally slightly for ease of reading. Nanoindentation hardness data were taken at 300 nm indenter penetration using the cross-section method. Fe6Cr nanoindentation data from C.Hardie (University of Oxford/ CCFE), Fe-9Cr nanoindentation data from L.Hewitt (University of Oxford). Vickers hardness data (500 g) provided by T. Milot (INL).

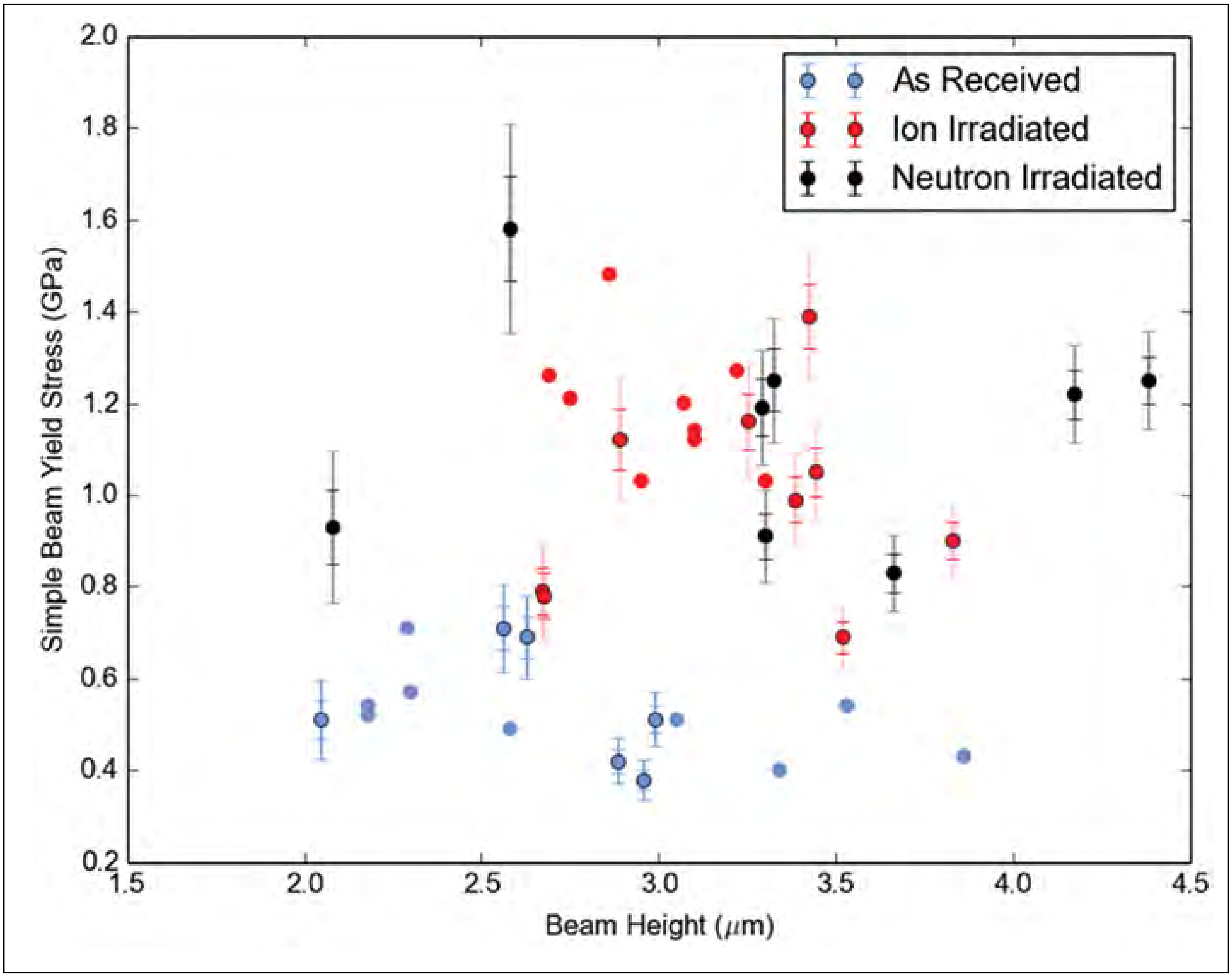


Figure 3. Yield stress measured using simple beam theory on Fe-9Cr microcantilevers as a function of beam height for ion irradiated, neutron irradiated, and as-received samples. Each point represents a different microcantilever beam. Data produced by L. Hewitt (University of Oxford).

testing methods such as nanoindentation and micromechanical testing. The development of these techniques is also of use for active materials—here, reducing the size of samples required reduces their activity, facilitating easier handling and testing.

Accomplishments

The neutron irradiated sample had already been irradiated to 1.7dpa at 288°C as part of the University of California at Santa Barbara (UCSB) NSUF Irradiation Experiment lead by G.R. Odette. The sample was provided and prepared by P. Wells and D. Gragg at UCSB and shipped to the Center for Advanced Energy Studies (CAES). Microcantilever specimens (Figure 1) were then manufactured using the NSUF-funded access to the focused ion beam (FIB) at the Microscopy and Characterization Suite (MaCS) before being shipped to the Materials Research Facility at the Culham Centre for Fusion Energy (CCFE), Culham, U.K., for testing. Access to the MaCS facility was essential to the feasibility of the research, as such active-material facilities were not otherwise available to L. Hewitt.

The ion irradiated sample was irradiated at the JANNuS facility at the same temperature, but due to technical issues received a dose around 0.4–0.8 times that targeted. Because the ion irradiation was performed over a much shorter time, the dose rate differs significantly between the two irradiation conditions.

The FIB work on this and a reference as-received sample was performed at the University of Oxford, with testing and nanoindentation for all samples performed at CCFE.

Changes in nanoindentation hardness are shown in Figure 2, alongside similar work on an Fe-6Cr alloy from this specimen series, performed as part of another NSUF project by C. Hardie of University of Oxford/CCFE. Data provided by T. Milot of INL showing changes in Vickers hardness after neutron irradiation for both alloys are also shown.

Both the nanoindentation and Vickers hardness data show, for neutron irradiation, a larger increase in hardness (absolute or relative) for the Fe-6Cr alloy compared to the Fe-9Cr alloy. The nanoindentation data show a greater disparity in the increase in hardness for ion and neutron irradiation in the Fe-6Cr alloy than in the Fe-9Cr alloy. This is thought to owe to dose-rate sensitivity of radiation induced changes in hardness, which has been shown to vary strongly with Cr content (Journal of Nuclear Materials 439 [2013] 33–40).

Irradiation effects are complicated – and the more experiments we do, the more complicated we find they are. But we now have the tools to understand what's happening.

— Steve Roberts, Professor of Materials, Oxford University, UK

The yield stress measured by microcantilever testing shown in Figure 3 for the Fe-9Cr alloy also displays similar changes after ion and neutron irradiation. Data are more scattered than for the indentation studies (this is thought to be due to small variations in beam dimensions, to which the results are very sensitive: further analysis is in progress). Both ion irradiated beams and neutron irradiated beams show higher yield stress values than unirradiated material.

Analysis of the ion irradiated Fe-9Cr sample by atom probe tomography at the University of Oxford revealed an absence of the alpha prime precipitates in neutron irradiated Fe-9Cr materials found by M. Bachhav of the University of Michigan (Scripta Materialia 74 (2014) 48–51); this likely owes to the large differences in irradiation rate between the ion (fast ~3–6 × 10^{-5} dpa/s) and neutron (slow ~3 × 10^{-7} dpa/s) irradiation. Because changes in hardness and properties under neutron irradiation were well reproduced by ion irradiation for this Cr content, it appears that the irradiation induced hardening is not sensitive to possible radiation induced clustering of Cr, but depends more strongly on the presence of other radiation induced features such as dislocation loops, which are less sensitive to irradiation rate.

Note that the earlier work by Hardie (also carried out with NSUF support) indicates that the sensitivity of irradiation hardening to dose rate (and hence irradiation type, ion or neutron) varies strongly with Cr content; for his Fe-6Cr, the slower irradiation with neutrons gave a greater degree of hardening than that for the rapid irradiation with ions, which was not the case for the Fe-9Cr studied here. This may be due to a segregation of Cr to dislocation loops in Fe-6Cr. Further investigation is needed as these dose rate/%Cr/temperature (probably) effects have a strong and controlling effect on the extent to which hardening effects of neutron irradiation in this material type can be evaluated by ion irradiation.

This work highlights the need to validate heavy ion irradiation experiments with work on neutron irradiated samples, only possible through access to facilities such as those in MaCS at CAES. It also shows the need for more work to improve understanding of Cr behavior under irradiation for the design of ferritic/martensitic steels for future nuclear applications.

Future Activities

This work will be written up for journal publication in 2018.

Further Vickers hardness tests will be performed with lower loads to investigate size effects and provide continuity with the nanoindentation tests.

Further microstructural analysis of the samples will be carried out, particularly of dislocation loop chemistry, in order to understand and link the differences in mechanical properties observed for the different compositions to damage microstructure.

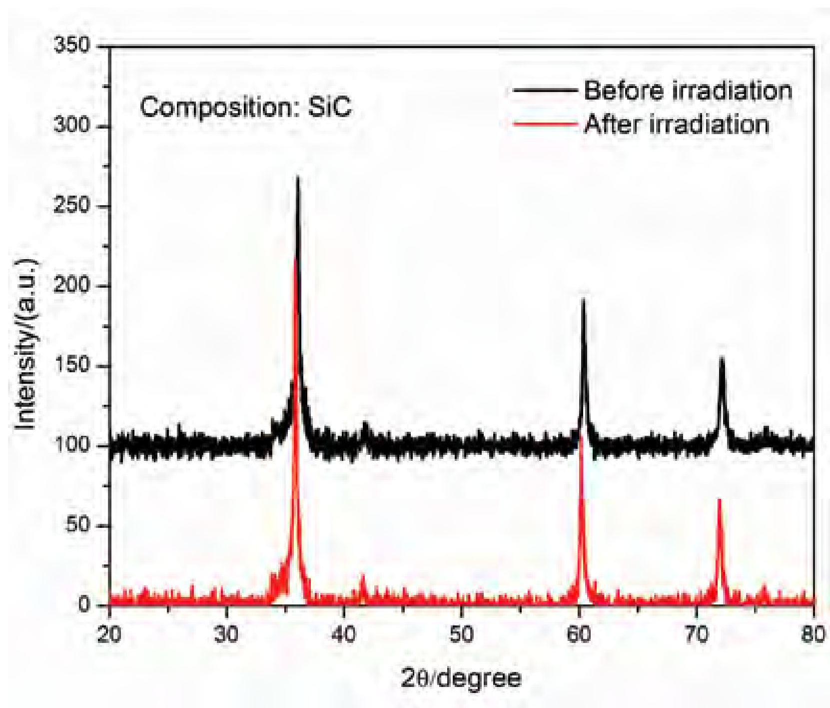
NSUF Fuels & Materials Library
08-139 UCSB samples

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
Collaborators	
University of Idaho	Joanna Taylor (collaborator)
University of California Santa Barbara	David Gragg (collaborator), G.R. Odette (collaborator), Peter Wells (collaborator)
University of Oxford	Luke Hewitt (collaborator), Professor Steve Roberts (principal investigator)

Influence of neutron irradiation on the microstructures and electrical properties of polymer derived ceramic sensing material

Dr. Cheryl Xu – Florida State University – cxu@eng.famu.fsu.edu

Figure 1. XRD pattern of 1600°C sintered SiC samples before and after irradiation.



If our hypothesis—that the microstructures and electrical properties are maintained under irradiation—is correct, these materials will later be used to create temperature sensors in a nuclear reactor for in-core temperature measurement during the next phase of research.

Sensors fabricated of polymer-derived ceramics (PDCs) are widely investigated. This kind of material has shown a great potential for applications in high temperature and harsh environments that are not survivable for most commercial sensors available. Unlike conventional polycrystalline ceramics, PDCs possess a unique structure that can be generally described as an amorphous matrix containing self-assembled carbon phase (named free carbon in later discussion). Such a structure leads to many unusual properties, such as high temperature stability, creep resistance, semiconducting behavior, and piezoresistive behavior up to temperatures higher than 1600°C. In the development of a sensor for use in high temperature and harsh environments, PDCs are one of only a few choices available. In nuclear reactors, irradiation energy and temperature cause most materials to degrade with time. For PDCs to be useful as a temperature sensor inside a reactor, their irradiation resistance is very important. In this project, investigation into irradiation influence on the microstructure and electrical properties of PDCs will proceed to determine the irradiation resistance of PDCs.

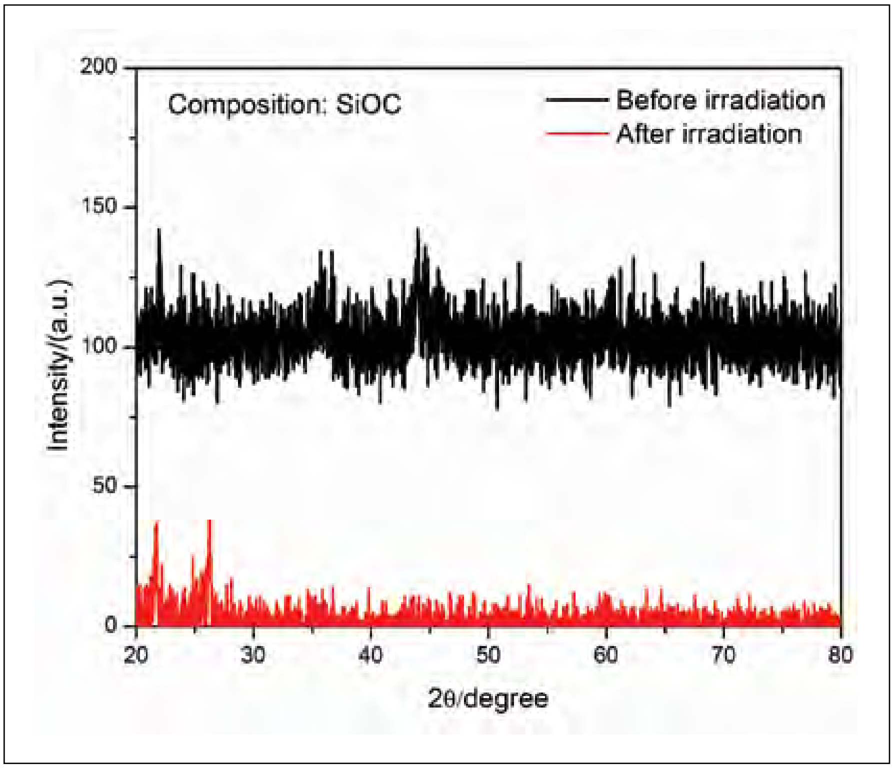


Figure 2. XRD pattern of 1600°C sintered SiOC samples before and after irradiation.

Project Description

The objective of this project is to perform neutron irradiation damage tests on silicon-based PDCs. If our hypothesis—that microstructure and electrical properties are maintained under irradiation—is correct, such material will later be designed as a temperature sensor in a nuclear reactor for in-core temperature measurement. This will be the goal for the next phase of research. Irradiation stability is one of the most important factors in ensuring the performance of PDC sensor material for nuclear applications. It is well known that the root cause of radiation damage is the continuous generation of vacancies and interstitials in atomic displacement cascades and defect accumulation. Crystallinity of the material is thus an important factor in radiation damage. PDCs are mostly used at non crystalline state, which makes them damage resistant. PDCs also exhibit a more stable structure than crystalline SiC and Si_3N_4, which is inferred from their higher creep resistance and higher thermal stability. It was further demonstrated that PDCs are thermodynamically stable as compared to the crystalline mixtures ($SiC + Si_3N_4 +$ graphite) of the same

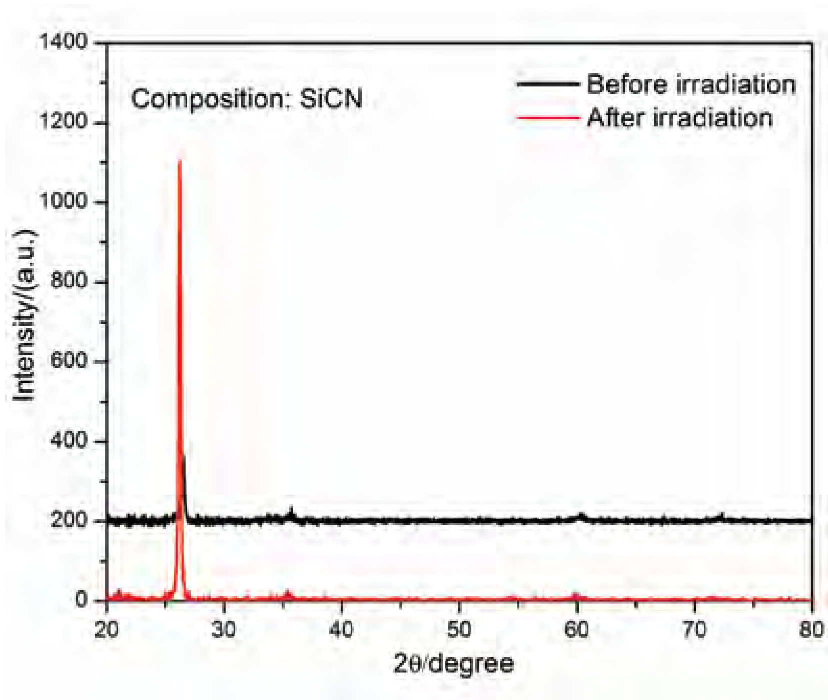


Figure 3. XRD pattern of 1600°C sintered SiCN samples before and after irradiation.

compositions. Unlike conventional materials, the PDCs consist of nanodomains created by intertwined graphene (aromatic carbon) sheets about 1–5 nm in size. The unique structure of PDCs can effectively promote defect recombination to mitigate radiation damage. The variations of these structures under irradiation damage would be critical for the development of the sensor in harsh environments.

Accomplishments

In the experiment, we first cured the liquid ceramic precursor and then crushed it into powders using ball milling. To get different compositions, especially those not commercially available such SiBCN and SiAlCN, we did chemical modifications on the ceramic precursor, including polysilazane. After crushing, we prepared the amorphous samples in our own laboratory by pressing each powder into bullets and then doing pyrolysis

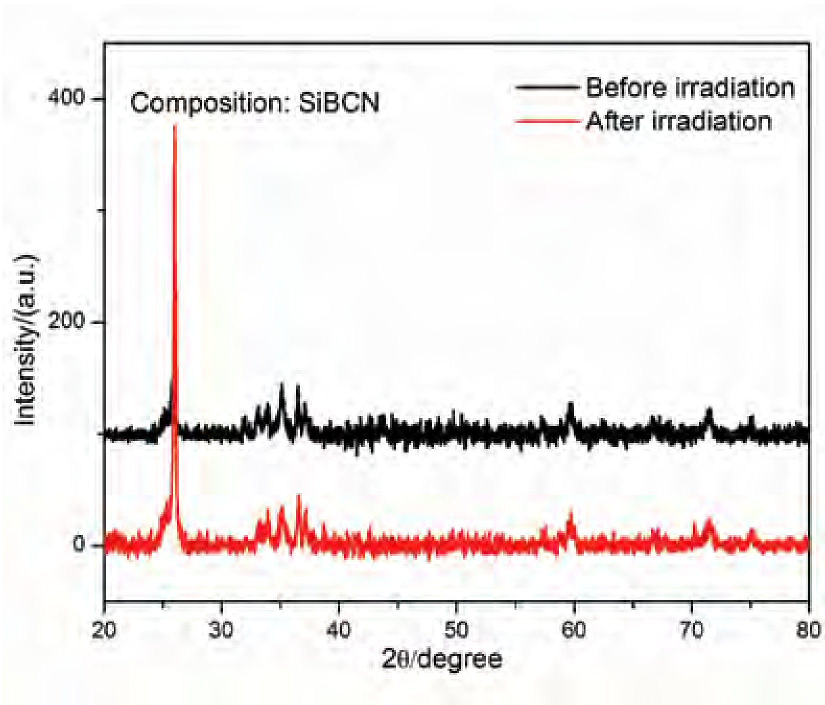


Figure 4. XRD pattern of 1800°C sintered SiBCN samples before and after irradiation.

under one atmosphere pressure in nitrogen. In order to get dense and crystallized PDC samples, our collaborator at the National Aeronautics and Space Administration (NASA), Glenn, helped us to prepare the samples using higher temperatures (over 1400°C) in a hot press. Our collaborator at North Carolina State University coordinated and finished the radiation tests. We did our characterization in High Performance Materials Institute and Chemistry department at Florida State University.

For amorphous samples, no pressure is applied, and the dwelling temperature is 1000°C during pyrolysis. For crystallized samples, the pyrolysis temperature is 1600°C (SiC, SiCN, SiOC) or 1800°C (SiAlCN, SiBCN) and the sintering pressure is 8 ksi. Note that only ceramic or pyrolyzed powders can be used as a feedstock for hot press treatment. For every sample irradiated, an extra, unirradiated sample from the same batch is archived for comparison.

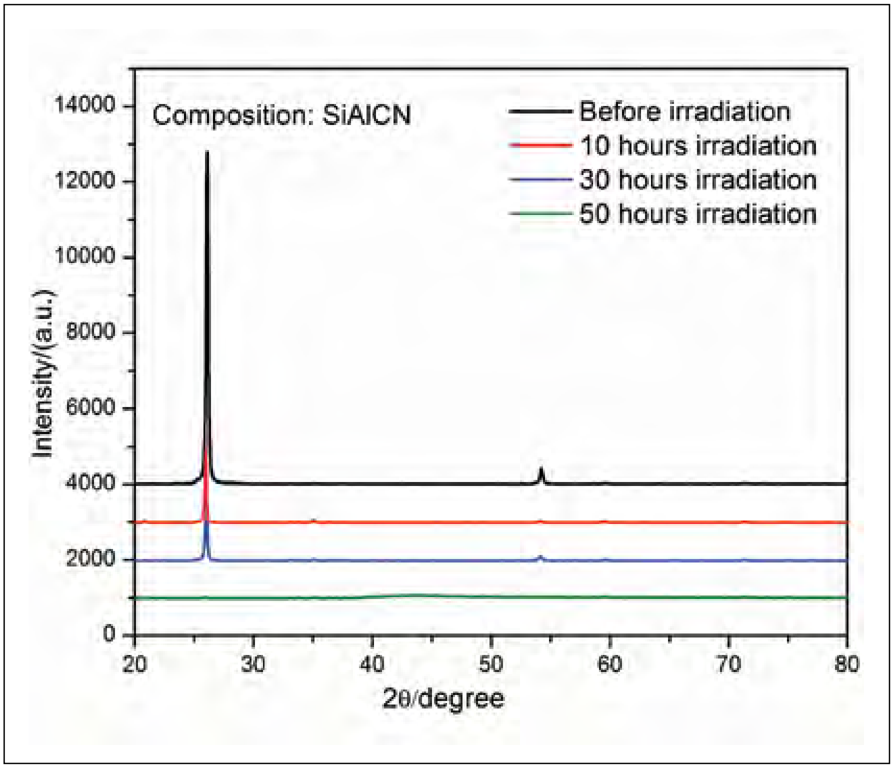


Figure 5. XRD pattern of 1800°C sintered SiAlCN samples before and after irradiation.

Visually, the irradiated and unirradiated samples are almost the same. For crystallized samples (samples treated at higher temperature in a hot-press), according to the x-ray diffraction (XRD) results:

1. SiC and SiOC samples show little change on the crystallinity structure after 10 hours of irradiation (Fig. 1). SiC maintained its crystallinity over 10 hours of irradiation. SiOC is not crystallized even after 1600°C sintering (Fig. 2). Its amorphous structures are similar before and after irradiation.
2. For SiCN (Fig. 3) and SiBCN (Fig. 4) samples, irradiation increased the crystallinity. The (002) peak of graphite is the only major peak in XRD, which means that crystallization of Si-B-C-N composites is not complete. After irradiation for 10 hours, the graphite peak for both SiCN and SiBCN samples has a higher intensity, which means that the input irradiation energy increased crystallinity.

3. For SiAlCN samples, irradiation destroys the crystal structure gradually (Fig. 5). The main crystallized peak is also the (002) peak of graphite. With increasing the peak irradiation time, intensity decreases continuously, and the crystallized peak disappeared after 50 hours irradiation, which means that neutrons have destroyed the crystal structure of the sample.

In summary, SiC (crystalized) and SiOC (amorphorous) show better irradiation resistance than other compositions. Irradiation increased the crystallinity of SiCN and SiBCN while, for SiAlCN samples, the irradiation energy destroys the crystal structure after 50 hours of irradiation.

Scanning electronic microscopy (SEM) was used to inspect the microstructure of the samples. No obvious extra defects were detected for any sample under SEM measurement. In principle, neutron irradiation will generate atomic-scale defects such as interstitials, displacement, stacking faults, and Frenkel defects. These defects are difficult to detect by SEM because the scale is small, and defects do not lie on the surface.

Future Activities

We will do furthur inspection on the samples in order to determine what happened during irradiation. First, we will measure electrical conductivity changes before and after irradiation. Then, high temperature dielectrical properties will be tested using our high temperature dielectric properties measurement system (funded by DOD DURIP, Award No. W911NF-16-1-0516). Third, ultrasonic scanning will be performed in order to confirm the large-scale defect distribution throughout the sample. X-ray fluorescence (XRF) characterization will be used to confirm the composition change (i.e., change in element ratios) for the samples before and after irradiation.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
North Carolina State University	PULSTAR Reactor Facility
Collaborators	
Florida State University	Dr. Cheryl Xu (principal investigator)

Beamline examination of a Hf-Al metal-matrix composite material

Donna Post Guillen – Idaho National Laboratory – donna.guillen@inl.gov

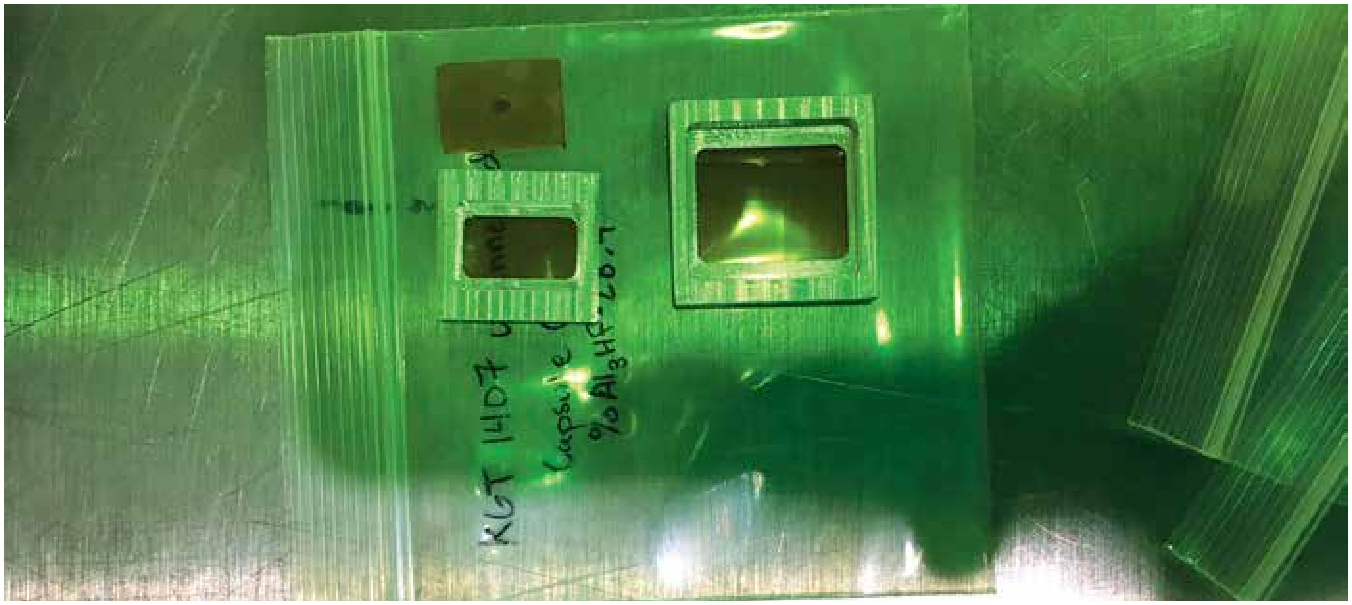


Figure 1. Irradiated specimen mounted in a triple contained sample holder sealed with Kapton tape for EXAFS measurements.

The capability for conducting fast neutron irradiation tests in a domestic facility is needed to meet fuels and materials testing requirements for advanced reactor programs. DOE has examined options to augment existing nuclear facilities to enable fast neutron testing, which could be brought on line in a few years, much sooner than–and at a fraction of the cost of–building a new fast flux test reactor. A fast flux test capability can be achieved by incorporating a special testing rig into one of the ATR corner lobes. A new material, comprised of hafnium aluminide ($HfAl_3$) particles in an aluminum matrix has been developed to achieve the required neutronic and thermal characteristics of such a system. This metal matrix composite material is very promising for use in the Advanced Test Reactor as a conduction cooled neutron absorber and has the potential to be useful as a shroud for a variety of fuel and material experiments.

The overall objective of this research is to determine the effects of radiation damage on the material microstructure, and in turn how the resultant microstructure affects the thermal and mechanical properties of the material, which ultimately affect performance and function of reactor components fabricated from this material. Additionally, there is interest in the $HfAl_3$ as an elevated temperature alloy for the semiconductor industry and other applications.

The end result, in terms of the data and fundamental understanding obtained, directly supports DOE's mission of providing clean, reliable energy technologies and benefits the science community in general.

Accomplishments

Synchrotron extended X-ray absorption fine structure (EXAFS) spectroscopy measurements were performed to study the structural changes in local atomic environments, defect formation, and the evolution of Hf-Al phases caused by irradiation and annealing in $HfAl_3$-Al metal matrix composites. The experiments were performed at the Advanced Photon Source (APS) Materials Research Collaborative Access Team (MRCAT) beamline 10-ID-B, which is equipped with a Si(111) monochromator consisting of a cryo-cooled first crystal and a 250 mm long second crystal, providing an energy range of 4.8–30 keV with the first harmonic. The absorption spectra were recorded at room temperature at the Hf L3-edge (E_o=9.561 keV) and the Zr K-edge (E_o=17.998 keV) in fluorescence geometry for the solid samples and transmission geometry for the powders. Reference materials consisted of Hf and Zr foils and powders of HfO_2, $HfAl_3$, and H_2Al_5. From the Al-Hf phase diagram, various additional intermetallic phases can form, including Al_3Hf, Al_2Hf, Al_3Hf_2, AlHf, Al_3Hf_4, Al_2Hf_3, and $AlHf_2$. The objective of this research is to determine which phases are initially present in the material and which phases form as a function of neutron irradiation and subsequent annealing. The key question to be answered by this study is, "Do new phases form as a result of irradiation or annealing?"

The materials examined were comprised of either 100 vol % $HfAl_3$ or 28.4 vol % $HfAl_3$-Al. The chemical composition of the $HfAl_3$ 100 vol % alloy was 69.7 wt% Hf, 29.4 wt% Al, with 8300 to 9400 ppm of Zr and trace amounts of other elements, and the chemical composition of the $HfAl_3$ 28.4 vol % alloy was 67.04 wt % Al, 32.96 wt % Hf, with 8300 to 9400 ppm of Zr and trace amounts of other elements. The $HfAl_3$ intermetallic was produced by casting Hf and Al in a centrifugal caster. The $HfAl_3$ castings were crushed into powder that was mixed with (for the 28.4 vol% material) or without (for the 100 vol% material) aluminum powder and uniaxially hot pressed to form pucks from which specimens were fabricated by electrical discharge machining.

The specimens examined here were 5 mm dia. x 2.5 mm thick, 5 mm dia. x 0.8 mm thick, or 3 mm dia. x 0.3 mm thick disks. Samples were irradiated for 1, 2, 3 or 4 cycles (800.6, 1965.5, 3184.0 or 3984.6 MWd) in the Advanced Test Reactor at INL. The materials were annealed after irradiation when differential scanning calorimetry was performed. The irradiated samples are from the NSUF Fuels and Materials Library (USU KGT-1407, 1389, 1533, 1401, 1456, 1496). Each sample was taped into a Kel-F insert inside of a beamline holder with Kapton tape, forming a triple layer of confinement (seen in Figure 1). The triple-contained specimens were then shipped from the INL Materials and Fuels Complex to the APS for the synchrotron radiation experiments.

The EXAFS measurements were conducted at room temperature on three different states of the material: (1) unirradiated, (2) irradiated-unannealed,

The discovery of new materials opens up new realms of possibilities and makes possible what was not before possible.

— Donna Post Guillen, Distinguished Research Engineer

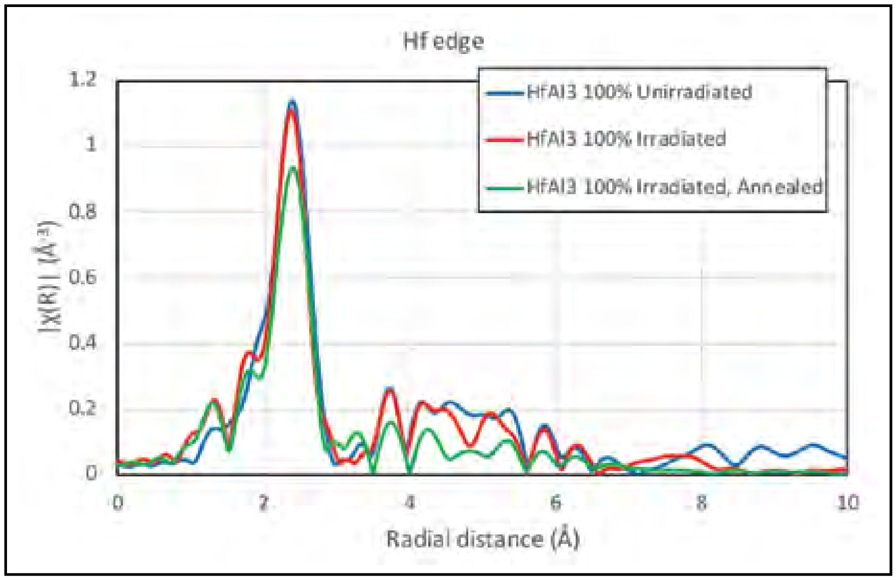


Figure 2. Fourier transform of the k^2-weighted spectrum as a function of bond distance for the 100 vol% $HfAl_3$ material at the Hf edge for three different material states.

Successful completion of this project will provide necessary data for the development of a fast neutron test capability in existing light water reactors, fill a knowledge gap on the basic properties of the $HfAl_3$ intermetallic and $HfAl_3$-Al neutron absorbers, and advance the scientific understanding of the irradiation effects on these materials.

and (3) irradiated-annealed specimens exposed to 0.40, 0.93, 1.05, 2.57, 3.51, and 3.65 displacements per atom (dpa) at 95-150 °C. The EXAFS data for the Hf L3-edge and Zr K-edge were recorded, and the local structure close to the X-ray absorbing atom was determined. X-ray absorption data for all samples were processed and analyzed using Athena and Artemis software. The X-ray absorption spectrum for each sample is an average of 10-15 scans that were initially processed using standard pre-edge background subtraction, edge-step normalization, and energy calibration procedures. The first large peak in the Fourier transform of the k2-weighted spectrum, $\mathcal{X}(R)$, as a function of bond distance, R, seen in Figures 2 and 3 is indicative of the signal from the first shell of atoms. A significant reduction in peak amplitude occurs in the Hf L3-edge and Zr K-edge spectra for the irradiated-unannealed specimens. The irradiated-annealed specimens display an increase, but not a full restoration, of the peak amplitude. Irradiation damage is manifested by the creation of local disorder within the crystal structure of the material, which is partially restored by annealing. The three different material states exhibit a decrease in the amplitude of $\mathcal{X}(R)$ from higher order shells ($R > 3A^{\circ}$). As the signal is a superposition of the photoelectron scattering functions for each shell, this suggests that in the annealed sample, the nearest neighbors around the Hf or Zr atoms are well defined, but the outer atomic shell structure is more disordered. These results demonstrate that EXAFS can provide an atomic level description of radiation damage in metal matrix composite systems.

The PIs are grateful to Professor Jeff Terry for his expert knowledge in conducting beamline experiments with irradiated materials at MRCAT and to Ph.D. student Rachel Siebert for her invaluable guidance on analyzing the EXAFS data.

Future Activities

We will continue to analyze the data obtained at the MRCAT beamline in March 2017. Pair distribution function data from synchrotron diffraction experiments was obtained at the Advanced Photon Source beamline 6-ID in July 2017. This complementary data will be helpful to identify the phases present and the associated bond

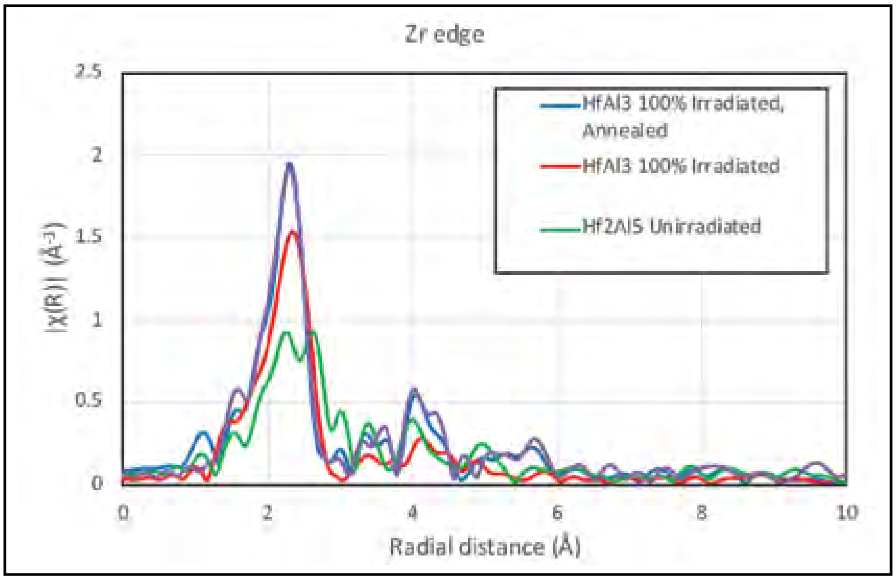


Figure 3. Fourier transform of the k^2-weighted spectrum as a function of bond distance for the 100 vol% $HfAL_3$ irradiated material and Hf_2Al_5 reference at the Zr edge.

distances. EXAFS data will be analyzed to quantitatively determine average bond distance (r (Å)), coordination number (n (atoms)), and disorder (Debye-Waller factor or σ_2 ($Å_2$)) of shells of atoms around the absorbing atom. A journal publication will be prepared with these results.

Additional future work planned to commence in spring 2018 includes another ATR irradiation experiment of the $HfAl_3$-Al materials at sustained temperatures of 300°C±50°C and 400°C±50°C (~0.45T_m and 0.61T_m of the Al matrix) to radiation damage levels of 1 and 3 dpa. This will provide needed information on the high temperature stability of these materials in an irradiation environment.

Publications

[1.] Cheng, S. and Guillen, D.P., Characterizing Local Structure of Nuclear Materials Using X-Ray Diffraction and Spectroscopy Techniques, presented at the INL Intern Expo, August 2017. *See additional publications from other years in the Media Library on the NSUF website at nsuf.inl.gov.

NSUF Fuels & Materials Library
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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 (APS)
Collaborators	
Idaho National Laboratory	Donna Post Guillen (principal investigator), Douglas Porter (principal investigator)
Rutgers University	Steven Cheng (intern)

Atom Probe Tomography characterization of irradiated UO_2 fuel from the BR3 Belgium Reactor

Brandon Miller – Idaho National Laboratory – brandon.miller@inl.gov

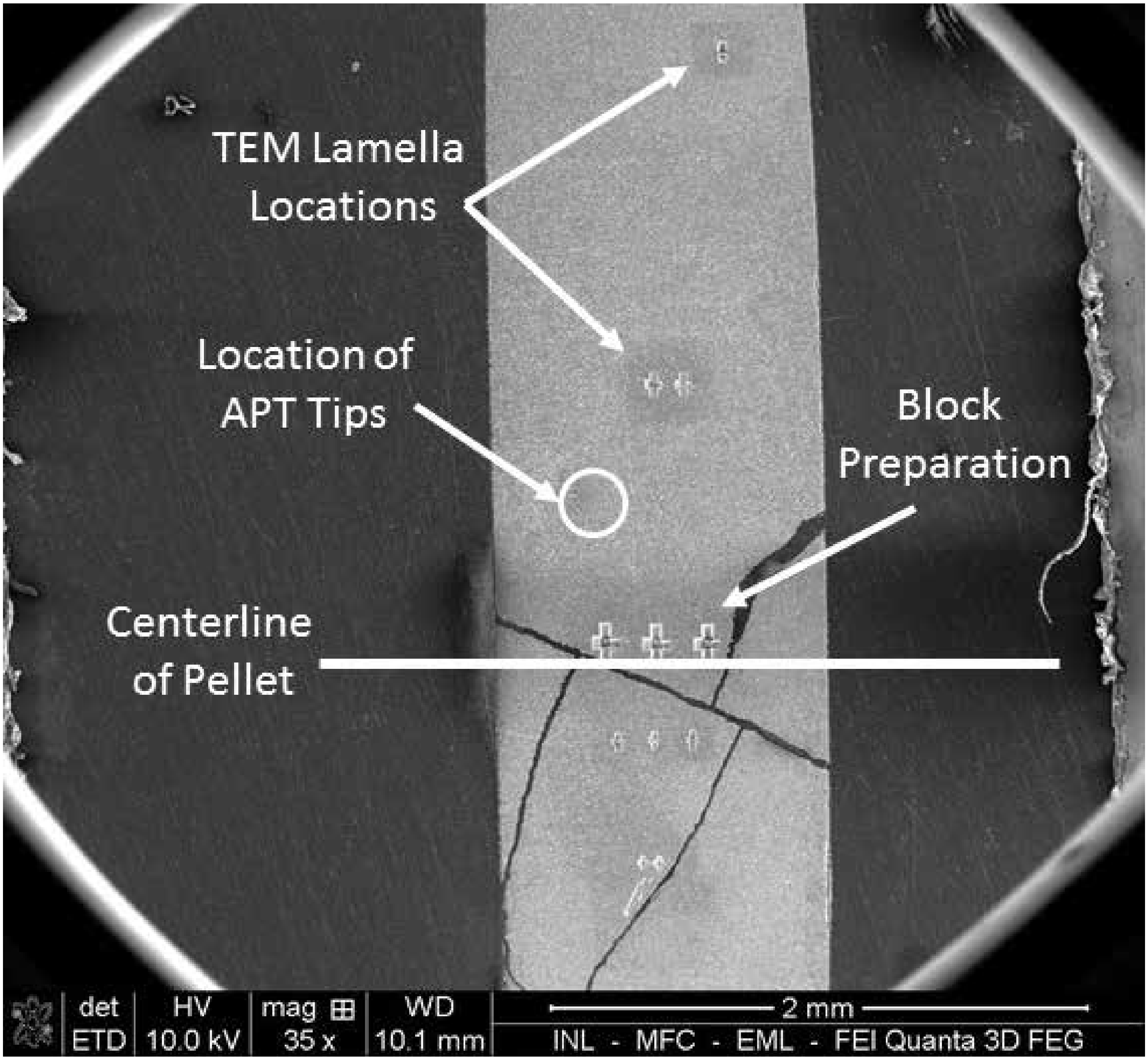


Figure 1. APT sample preparation locations in an irradiated UO_2 fuel cross section.

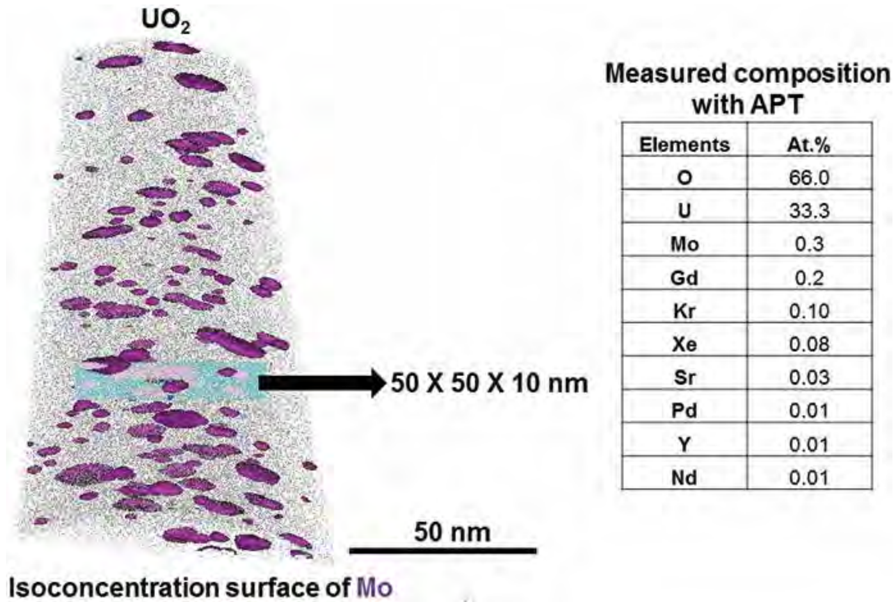
Atom Probe Tomography (APT) was performed to better understand defect and elemental composition of an irradiated UO_2 fuel sample. Focused ion beam, transmission electron microscopy, and scanning electron microscopy were assimilated together to characterize the microstructure of the UO_2 fuel cross section. These techniques provide useful information that can be incorporated into fuel code models. By creating higher accuracy models, the life time of fuel elements can potentially be extended. This research supports the Office of Nuclear Energy's (DOE-NE) Light Water Reactor Sustainability (LWRS) Program and focuses on materials integrity in the extension of the lifetime of existing LWR fuels.

Project Description

The purpose of this study was to perform APT of an irradiated UO_2 fuel sample to better understand the defect and elemental compositional makeup of UO_2 at various locations in a fuel pellet. Inside a commercial UO_2 fuel rod, the UO_2 fuel will be under differing irradiation conditions with various irradiation parameters affecting the local microstructure. Some irradiation parameters that can affect microstructure include irradiation temperature, fission rate, fission density, and precipitation of fission products. Using a cross section of a commercial fuel pellet from Belgium Reactor 3 (BR3), APT tips were created from a known radius location in the pellet. The locations of where the APT tips were obtained can be seen in Figure 1. At the time of this report, the local irradiation parameters of the APT location are unknown. Twenty APT tips were prepared using a Focused Ion Beam at INL's Materials and Fuels Complex. Transmission electron microscopy of the APT tips was performed prior to performing APT analysis to compliment the APT results. The APT tips were run using the CAMECA LEAP at CAES. Preliminary results show that various fission products are present in the APT tips. Figure 2 shows a preliminary isoconcentration surface of the UO_2 focusing on Mo content. Figure 3 shows a 50 x 50 x 10 nm section of one APT tip with isoconcentrations surfaces of other anticipated fission products. Mo precipitates can be seen throughout the APT tips. Xe, Pd, Sr, and Y show increased concentrations at the locations of the Mo precipitates. Gd is an artifact in the APT results.

Atom probe tomography of irradiated fuel provides information on fission product behavior in irradiated UO_2 fuels, which help support modeling efforts to extend fuel lifetimes in commercial power reactors.

— Brandon Miller, Research Scientist, Idaho National Laboratory



Elements	At.%
O	66.0
U	33.3
Mo	0.3
Gd	0.2
Kr	0.10
Xe	0.08
Sr	0.03
Pd	0.01
Y	0.01
Nd	0.01

Figure 2. APT isoconcentration surface of Mo in the irradiated UO_2 fuel.

Accomplishments

With little-to-no publications of APT of irradiated fuels, this work provided first-of-a-kind data on behavior of fission products in irradiated commercial UO_2 at the atomic level. Understanding fission product behavior in irradiated fuels supports modeling efforts and can potentially help extend the lifetime of commercial fuels, leading to increased efficiency of the current nuclear reactor fleet.

Future Activities

Data analysis has commenced for the project and a journal article will be created from the work once all data has been compiled and the irradiation history of the APT locations is finalized.

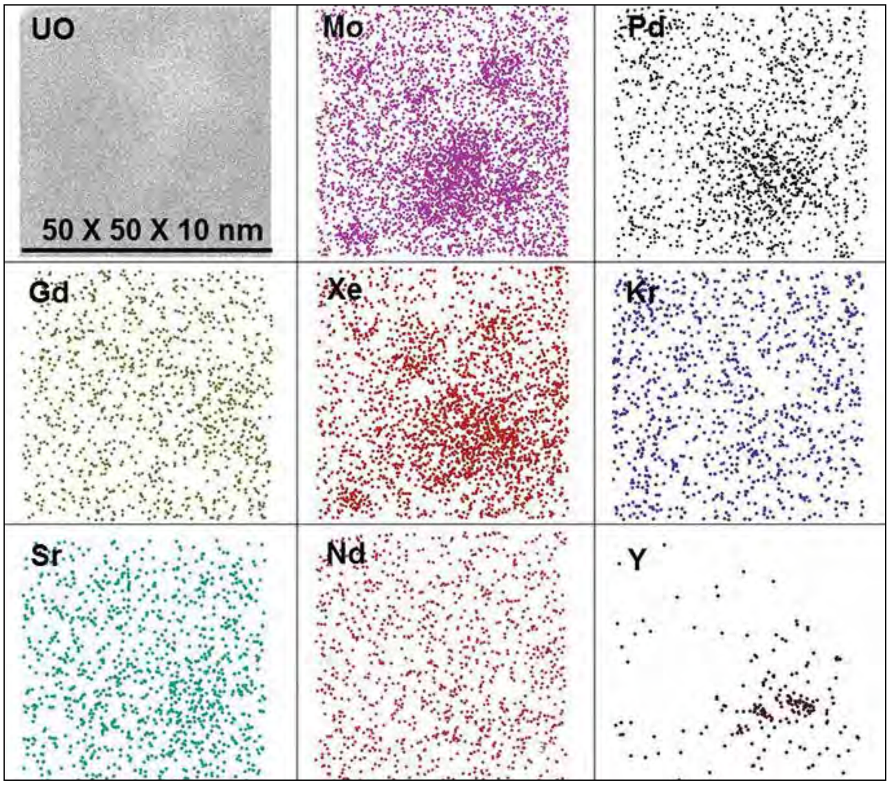


Figure 3. APT isoconcentration surfaces of multiple fission products.

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Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Electron Microscopy Laboratory
Collaborators	
Idaho National Laboratory	Brandon Miller (co principal investigator), Mukesh Bachhav (collaborator)
University of Florida	Assel Aitkaliyeva (co principal investigator)

In situ study of defect accumulation in Ti-6Al-4V under heavy ion irradiation: Influence of the microstructure and temperature

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This project investigates the irradiation damage in Ti alloys for the development of engineering components for the Facility for Rare Isotope Beams at Michigan State University.

Due to their high specific strength, good fatigue and creep properties, corrosion resistance, and commercial availability, titanium (Ti) alloys have been widely used in industrial, aerospace, and biomedical applications. High compatibility with coolants (e.g., lithium, helium, water) and low activation in radioactive environments also make Ti alloys attractive for nuclear applications. Specifically, Ti-6Al-4V (wt.%) was selected to be a structural material for the beam dump in the Facility for Rare Isotope Beams (FRIB) at Michigan State University (MSU). Limited studies have investigated the irradiation damage in these Ti alloys, specifically commercially available Ti-6Al-4V. Manufacturing the complex shape of the Ti alloy FRIB beam dump presents difficult challenges. Additive manufacturing constitutes an attractive alternative to casting and wrought processing due to its capability to produce near net shape components with less production time and material waste.

Project Description

One objective was to investigate the effect of microstrucuture, dose, and temperature on irradiation damage in titanium alloys. Both irradiation doses and temperature affect the mechanical properties of Ti-6Al-4V. Microstructure evolution under irradiation at 25 and 350°C induces different obstacles to the dislocation motion. Ti-6Al-4V is known for the dependence of its mechanical properties on thermomechanical processing. Thermomechanical processing influences grain size and phase compositions. Improving the resistance of materials to irradiation damage has been the subject of studies that focused on the effect of grain boundaries and grain size. The literature indicates that a higher density of grain boundaries, such as in nanocrystalline materials, tends to exhibit a higher irradiation resistance. In addition, the effect of the grain size on irradiation induced void formation was investigated in copper and steel. Our study investigates the irradiation damage in Ti-6Al-4V samples processed through two different thermomechanical processes: powder metallurgy (PM) rolled and additive manufacturing (AM). The latter was processed by direct metal laser sintering (DMLS) followed by hot isostatic pressing (HIP). As shown in Figures 1 and 2, the samples exhibited different microstructures. The PM rolled sample exhibited equiaxed α-phase grains, with the β-phase typically present at grain-boundary locations whereas the AM sample exhibited a lamellar $\alpha+\beta$ microstructure. This study proposed to provide quantitative data for the microstructural changes (defect formation, defect clustering, defect densities) and mechanical properties of these microstructures as a function of irradition dose.

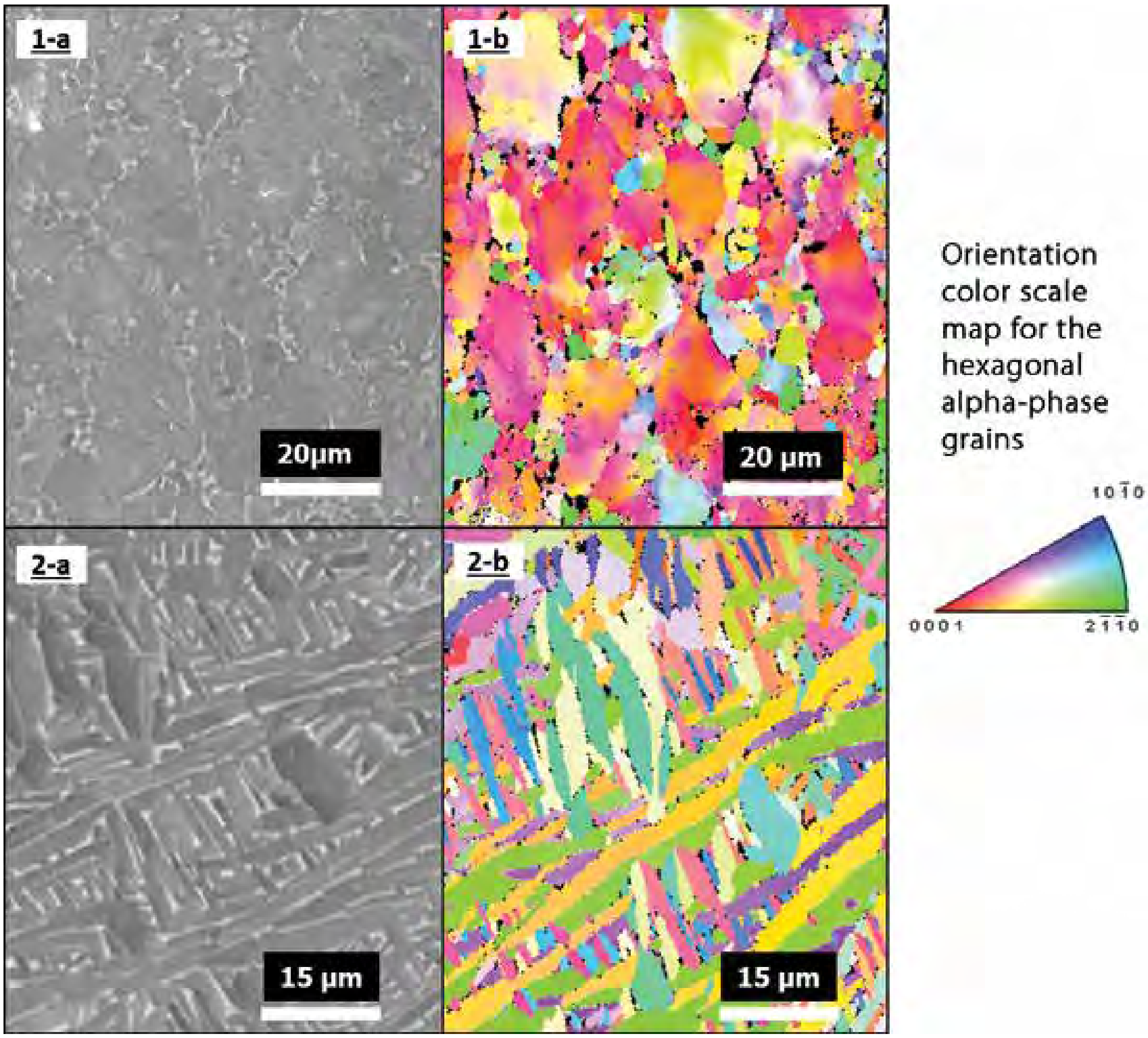

Figure 1. From upper left: (a) Backscattered electron scanning electron microscope photomicrographs and (b) electron backscattered diffraction inverse pole figures illustrating representative microstructures of the two studied as-processed Ti-6Al-4V samples; 1) PM rolled and 2) additively manufactured

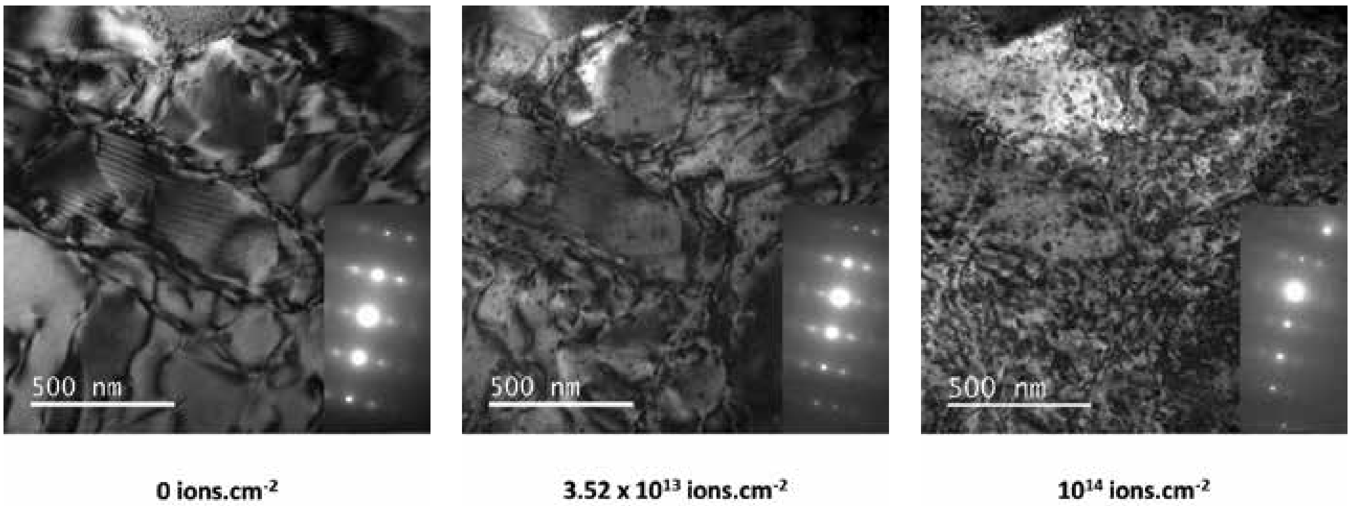

Figure 2. Bright field TEM micrographs obtained at the IVEM–Tandem facility during irradiation for an additively manufactured Ti-6Al-4V alloy. The insets show the selected area diffraction patterns for the central grain in the image. These images show the progression of damage that occurred at the different fluences (indicated under the images) which resulted from irradiation at 450°C with 1MeV Kr ions.

Accomplishments

For this NSUF rapid-turnaround experiment (RTE) project, we performed two different *in situ* transmission electron microscopy irradiation experiments at the Intermediate Voltage Electron Microscope (IVEM)–Tandem Facility at Argonne National Laboratory (ANL) with the assistance of the following ANL staff: Dr. Meimei Li, Dr. Mark Kirk, Mr. Pete Baldo, and Mr. Ed Ryans, and postdoctoral associate Dr. Jing Hu. During the first irradiation experiment at 350°C, we irradiated the two Ti-6Al-4V alloys that underwent different thermomechanical processing treatment. Their as-processed microstructures are shown in Figure 1. The PM rolled microstructure exhibited an equiaxed α-phase morphology with the β-phase decorating the grain boundaries.

The AM microstructure exhibited a lenticular morphology of the α+β phases. The objective of this study was to evaluate the progression of damage that occurs in Ti alloys and to evaluate the influence of microstructure on the damage accumulation. For the 350°C irradiation experiments, a relatively high dose of 24 dpa was achieved, and transmission electron microscopy bright-field images were acquired at different stages to show damage accumulation. The preliminary qualitative results suggest that the morphology of the α phase has an effect on defect accumulation. However, an in-depth evaluation of the crystal orientations of the α phase in the irradiated areas has yet to be performed and, thus, crystallographic effects cannot be ruled out. The second irradiation experiment was performed at 450°C on the same

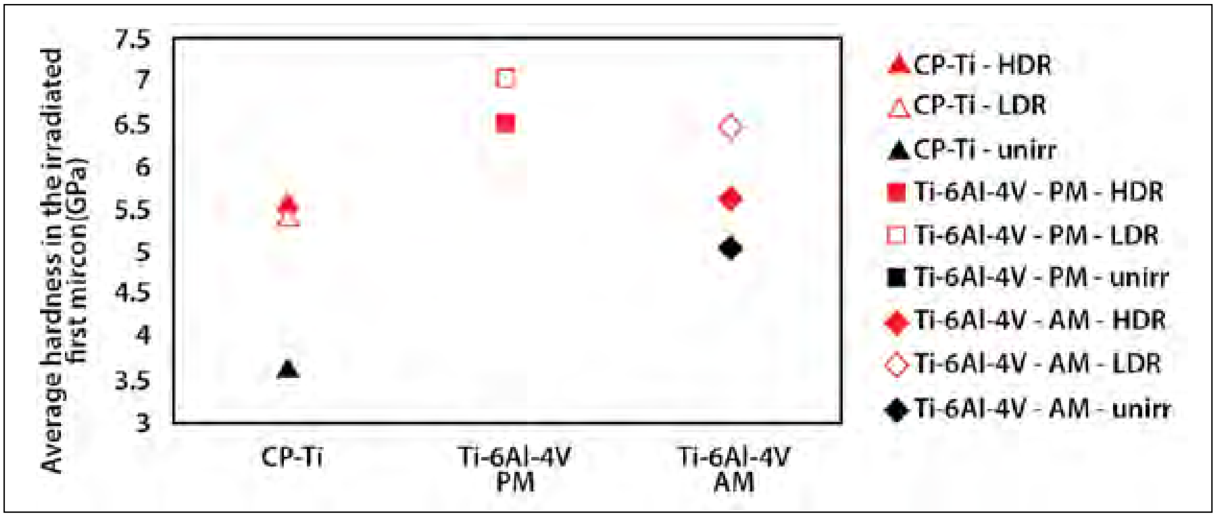

Figure 3. Average hardness measured for the three different Ti samples irradiated at the Notre Dame Tandem with 4 MeV Ar ions. Red symbols represent irradiated samples while the black symbols represent the unirradiated samples; solid symbols represent high dose rate (HDR, 13.4 dpa/hr) conditions while the empty symbols represent the low dose rate (LDR) (0.8 dpa/hr).

materials along with commercially pure titanium (CP-Ti). We stopped the experiments at lower doses in order enable <a> loop nucleation in the α phase without the extensive degradation associated with high dose rates that complicate such analysis. Bright field TEM images were acquired *in situ* throughout both irradiation experiments. Figure 2 shows the evolution of irradiation induced defects in an AM Ti-6Al-4V sample irradiated up to 0.24 dpa at 450°C. The nature of the defects present has yet to be determined.

Post irradiation TEM characterization of the irradiated CP Ti sample is currently being performed as part of an awarded NSUF proposal at the Low Activation Materials Design and Analysis (LAMDA) Laboratory at Oak Ridge National Laboratory (ORNL). The objective of this characterization work is to determine the defect structures and, in particular, establish whether <a> dislocation loops were formed in the α phase as a result of the irradiation exposure, and if so, to characterize their density.

In addition to the aforementioned *in situ* irradiation experiments, we are investigating the effect of irradiation exposure on the nanoindentation hardness for each of these materials. We irradiated bulk samples using a 4 MeV Ar ion beam at both 25°C (i.e., room temperature[RT]) and 350°C. This irradiation was performed using the University of Notre Dame's 5U accelerator in collaboration with The Institute for Structure and Nuclear Astrophysics (ISNAP) within the framework of the Radiation Damage In Accelerator Target Environments (RaDIATE) collaboration. The samples irradiated at RT were exposed to two different dose rates, 0.8 dpa/h

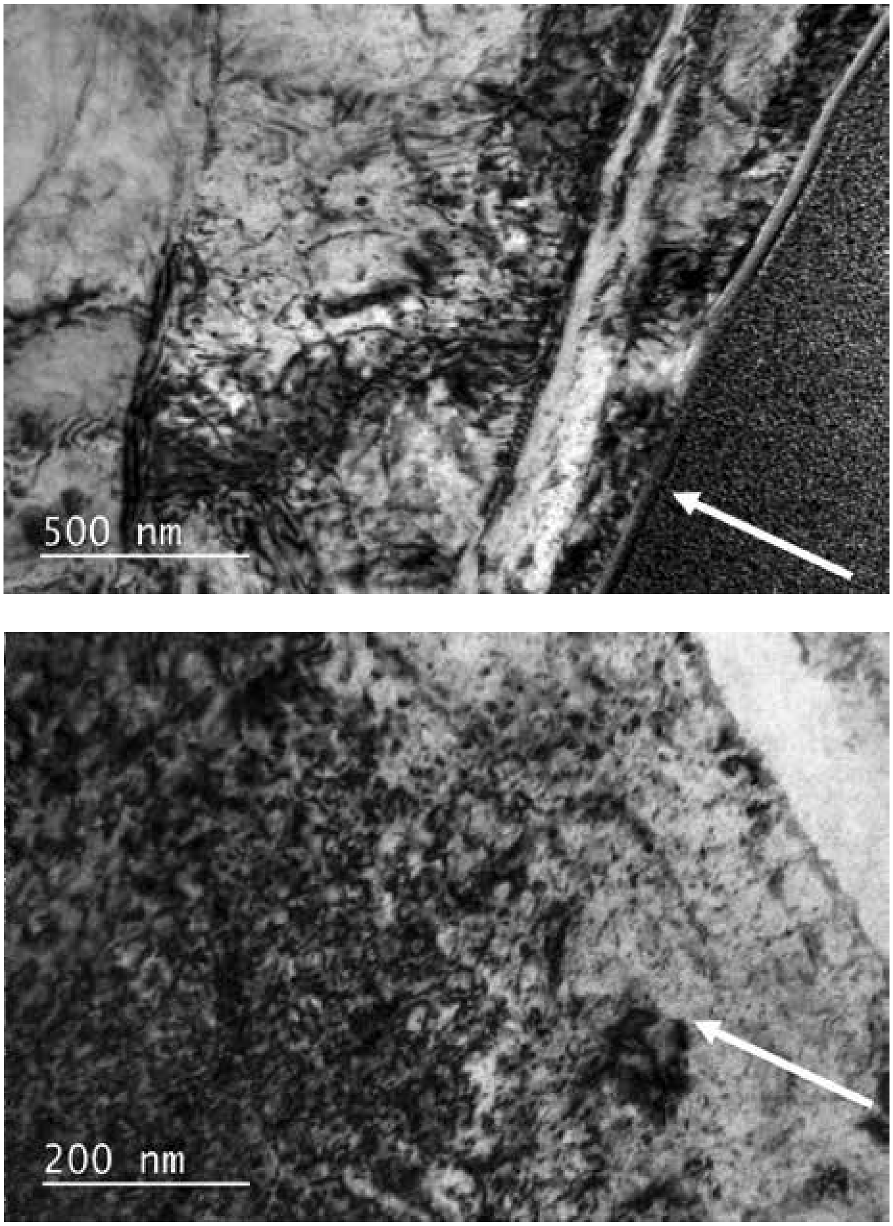

Figure 4. (a) Low magnification and (b) high magnification TEM bright field micrographs of the same area of a CP-Ti sample after irradiation at RT with 4 MeV Ar ions at LDR of 0.8 dpa/hr. Both arrows indicate the direction of the Ar ion beam and the arrow in (a) points to the border between the platinum layer deposited on the surface which was used to assist the FIB lift out.

and 13.4 dpa/h, and reached the same final dose of 7.3 dpa within 1 μm of the surface. Nanoindentation measurements were carried out using an Agilent Technologies G200 nano indenter in collaboration with the LAMDA facility at ORNL. Figure 3 summarizes the nanoindentation hardness results for the six irradiated samples. We have observed that CP-Ti exhibited the highest irradiation induced hardening whereas the nanohardness of the AM Ti-6Al-4V was the most sensitive to the dose-rate effect. Focused ion beam (FIB) liftouts were extracted from the CP-Ti samples with the assistance of the researchers at the LAMDA facility at ORNL. TEM characterization is ongoing with the help of ORNL postdoctoral fellow Dr. Boopathy Kombaiah. Preliminary results are shown in Figure 4, displaying the evolution of defects as a function of depth in the CP-Ti sample irradiated at the lower dose rate.

Future Activities

Further investigation and TEM characterization is ongoing at the LAMDA facility to understand the different damage structures in irradiated Ti-alloy samples at different doses. The results from the TEM and nanoindentation characterization of the irradiated Ti alloy will be published. As discussed previously, the microstcutures of the Ti-6Al-4V alloys examined were significantly different due to the different processing methods utlized. The PM rolled sample exhibited equiaxed α-phase grains with the β-phase typically present at grain boundary locations whereas the AM sample exhibited a lamellar

Figure 5. Picture of the ANL IVEM in use during in situ ion irradiation experiment.

$\alpha+\beta$ microstructure. Future work is intended to provide quantitative data for the microstructural changes (defect formation, defect clustering, defect densities) and mechanical properties of these microstructures as a function of irradition dose. We have qualitatively shown the defect accumulation during irradiation and we now need to measure the defect densities as a function of irradiation dose and time and temperature.

The IVEM facility provides unique capabilities to investigate, in situ, the evolution of ion irradiation damage.

— Aida Amroussia, PhD Graduate student

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NSUF and Partners	**Facilities and Capabilities**
Argonne National Laboratory	The Intermediate Voltage Electron Microscopy (IVEM)–Tandem Facility
Collaborators	
Argonne National Laboratory IVEM – Tandem Facility	Meimei Li (collaborator)
Michigan State University	Aida Amroussia (co principal investigator), Carl Boehlert (principal investigator)
National Superconducting Cyclotron Laboratory (NSCL) - Facility for Rare Isotope Beams (FRIB)	Frederique Pellemoine (collaborator), Wolfgang Mittig (collaborator)

Irradiation effect on the heterogeneous hardening of cast austenitic stainless steels

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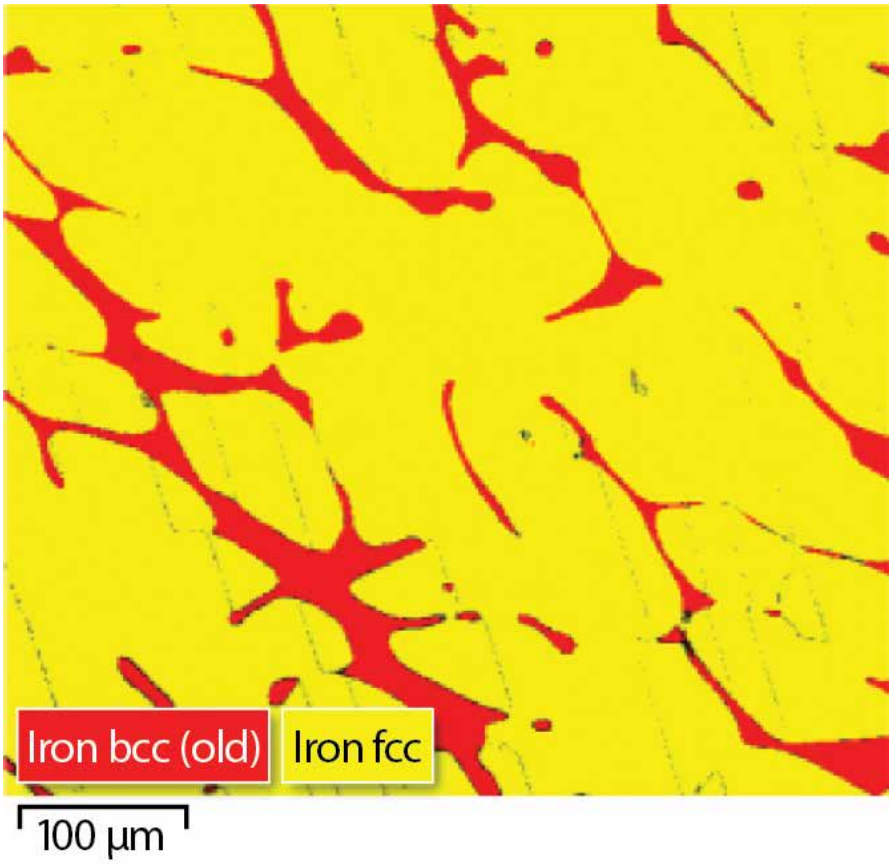

Figure 1. EBSD map shows the morphology of a CASS CF 8 where the yellow matrix is the austenite phase and the red islands are the ferrite phase.

Cast austenitic stainless steels (CASSs) are structural materials used for components at the primary pressure boundaries and core internals of light water reactors (LWRs). Consisting of a dual-phase microstructure of delta ferrite and austenite, as shown in Figure 1, CASS alloys are susceptible to thermal aging embrittlement and neutron irradiation damage; these phenomena are of concern after long-term exposure to reactor core environments. In this project, we investigated the embrittlement of CASS subjected to neutron irradiation and thermal aging treatment. The delta ferrite and austenite behave very differently under thermal and neutron

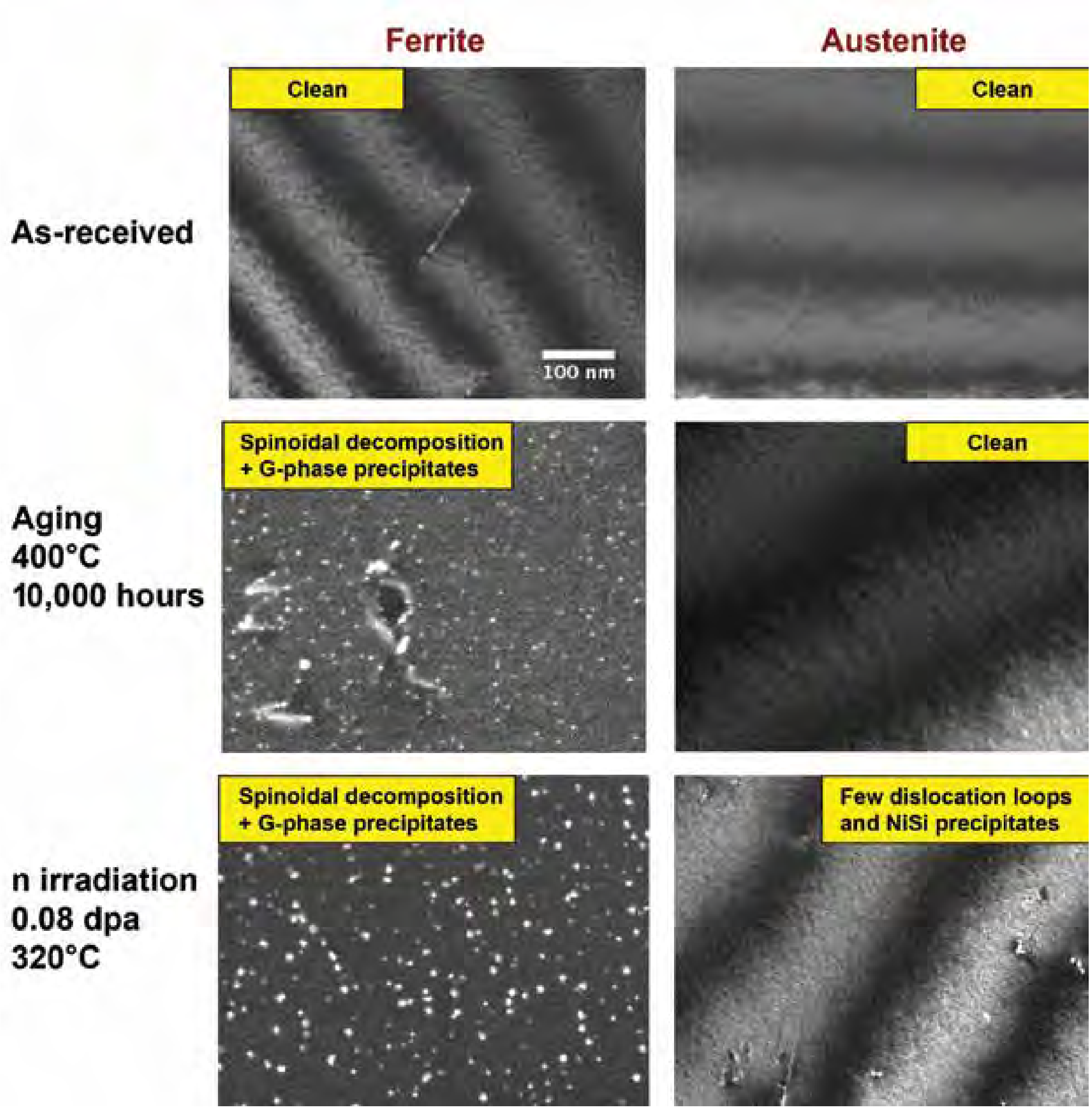

Figure 2. Dark field TEM images of the ferrite and austenite phases in CASS after thermal aging and neutron irradiation. The yellow text boxes summarize the microstructural characteristics observed by TEM and APT.

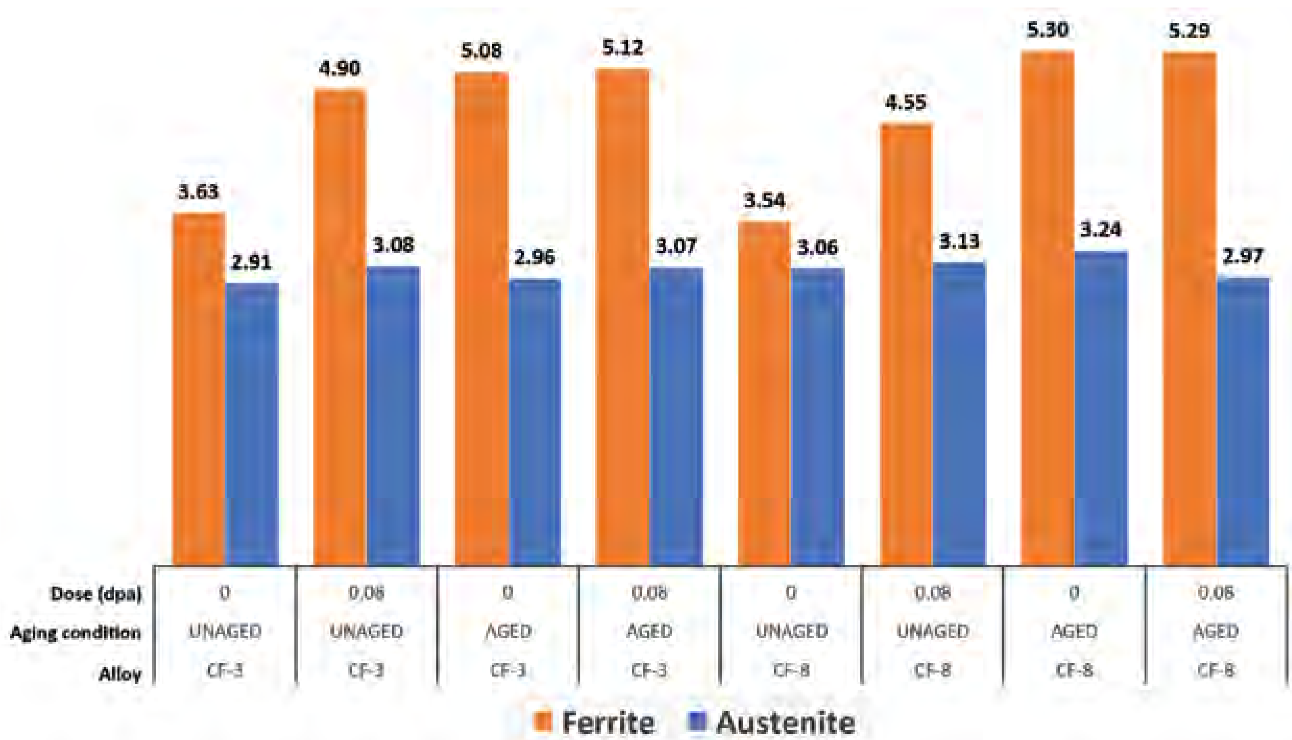

Figure 3. The hardness of the ferrite and austenite phase in CASS from nanoindentation in the unit of GPa.

exposure. Therefore, to understand the embrittlement mechanism in the dual phase microstructure of CASS, the hardening in delta ferrite and austenite need to be investigated separately. However, the thin shape of delta ferrite (a few microns in width) poses difficulty in traditional microhardness measurement.

With nano indentation measurements, we were able to measure the hardness evolution of delta ferrite and austenite individually with a fine scale. Combining the microstructural characterization and nanoindentation, we showed that the ferrite and austenite respond differently to thermal aging treatment and neutron irradiation, leading to an inhomogeneous development of hardness in two phases, which is critical for the deformation of dual phase microstructures and is contributing to the embrittlement of CASS alloys in service. The outcome of this study will help develop a scientific basis for assessing and managing the embrittlement of CASS components under LWR service conditions.

Project Description

In this project, we used nanoindentation to measure the hardness of the delta ferrite and austenite in a number of CASS CF3 and CF8 after thermal aging and neutron irradiation. The laboratory aging was performed at 400°C for 10,000 hours. The irradiation was performed in Halden reactor at 320°C to a dose of 0.08 dpa. The specimens were all in the shape of 3 mm discs with a thickness of about 200 μm. The specimens were electropolished to remove the surface deformation, and then mounted on SEM stubs using superglue.

Nanoindentation was performed in the Microscopy and Characterization Suite in the Center for Advanced Energy Studies (CAES). The delta ferrite and austenite phase were indented, each with multiple indents for good statistics. The hardness results were compared with the microstructural characterization using transmission electron microscopy and atom probe tomography (APT) to understand the relation between microstructural modification due to irradiation and thermal aging on the mechanical property of the delta ferrite and austenite in CASS. This work provides detailed understanding on the heterogeneous evolution of microstructure and mechanical property in CASS under the extreme conditions of nuclear reactors and serves as a stepping stone for developing a model to describe large scale mechanical properties such as fracture toughness, enhancing the ability to predict the long-term stability of CASS in nuclear reactors for an extended service life of 60 years.

The unique capability of nanohardness measurement on radioactive materials enables the study on materials of complex structure for use under the extreme conditions of nuclear applications

—Wei-Ying Chen, Postdoctoral Researcher

Table 1. List of specimens examined

Materials	Pre-irradiation Aging	Irradiation
CF3	unaged	0.08 dpa
CF3	400°C 1000 hours	0.08 dpa
CF8	unaged	0.08 dpa
CF8	400°C 1000 hours	0.08 dpa
CF3	unaged	No irradiation
CF3	400°C 1000 hours	No irradiation
CF8	unaged	No irradiation
CF8	400°C 1000 hours	No irradiation

The long-term degradation of cast austenitic stainless steel (CASS) is an important issue for light water reactor sustainability. This project is the first reported study to use nanoindentation to obtain the heterogeneous hardening in CASS as a result of thermal aging and neutron irradiation.

Accomplishments

We have successfully achieved the goal of the proposed scope to obtain the individual hardness of the delta ferrite and austenite in duplex CASS of various thermal aging and neutron irradiation conditions, and to correlate the hardness to the microstructures previously characterized with TEM and APT. TEM characterization was performed in the Intermediate Voltage Electron Microscope (IVEM)-Tandem Facility in Argonne National Laboratory. Zhangbo Li from the University of Florida carried out APT in CAES. As shown in Figure 2 and Figure 3, hardness and the microstructure can be consistently correlated. The ferrite experienced considerable hardening (roughly 40% more) after thermal aging as well as neutron irradiation due to the formation of G-phase precipitates and spinodal decomposition. The microstructures resulting from neutron irradiation and thermal aging were similar, leading to a comparable degree of hardening in spite of their distinct experimental conditions. Dislocation loops cannot be evidently observed in the ferrite phase after neutron irradiation.

By contrast, in the irradiated austenite phase, some dislocation loops can be observed along with the formation of the precipitates enriched in nickel and silicon. However, the total density of dislocation loops and precipitates was very low, so hardness did not increase. Thermal aging alone did not cause microstructural modification in the austenite phase and, therefore, did not change the hardness. The irradiation and thermal aging effects were qualitatively the same for CF3 and CF8.

Future Activities

We will continue this work by studying the same set of CF3 and CF8 CASS that had been neutron-irradiated to 3 dpa, a higher dose than the present study. Preliminary TEM examinations show that the austenite phase accumulated irradiation damage in the form of a high density of faulted dislocation loops while the ferrite phase seems to exhibit similar microstructure as its 0.08 dpa counterpart. We plan to use nanoindentation to investigate the heterogeneous hardening in these 3 dpa CASS specimens.

Publications

[1.] Y. Chen, W.-Y. Chen, B. Alexandreanu, K. Natesan and A.S. Rao, "Crack Growth Rate and Fracture Toughness of CF3 Cast Stainless Steels at ~3 DPA," Proceedings of the 18th International Conference on Environmental Degradation of Materials in Nuclear Power Systems—Water Reactors, August 13-17, Portland, OR.

[2.] Y. Chen, C. Xu, X. Zhang, W.-Y. Chen, J.-S. Park, J. Almer, M. Li, Z. Li, Y. Yang, A.S. Rao, B. Alexandreanu and K. Natesan, "Microstructure and Deformation Behavior of Thermally Aged Cast Austenitic Stainless Steels," Proceedings of the 18th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, August 13-17, Portland, OR.

[3.] W.-Y. Chen, Y. Chen, Z. Li, C. Xu, Y. Yang, N. Demas, "Effects of thermal aging and neutron irradiation on Cast Austenitic Stainless Steels," TMS Annual meeting, San Diego, CA 2017.

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Wigner Energy of SiC irradiated to high levels of swelling

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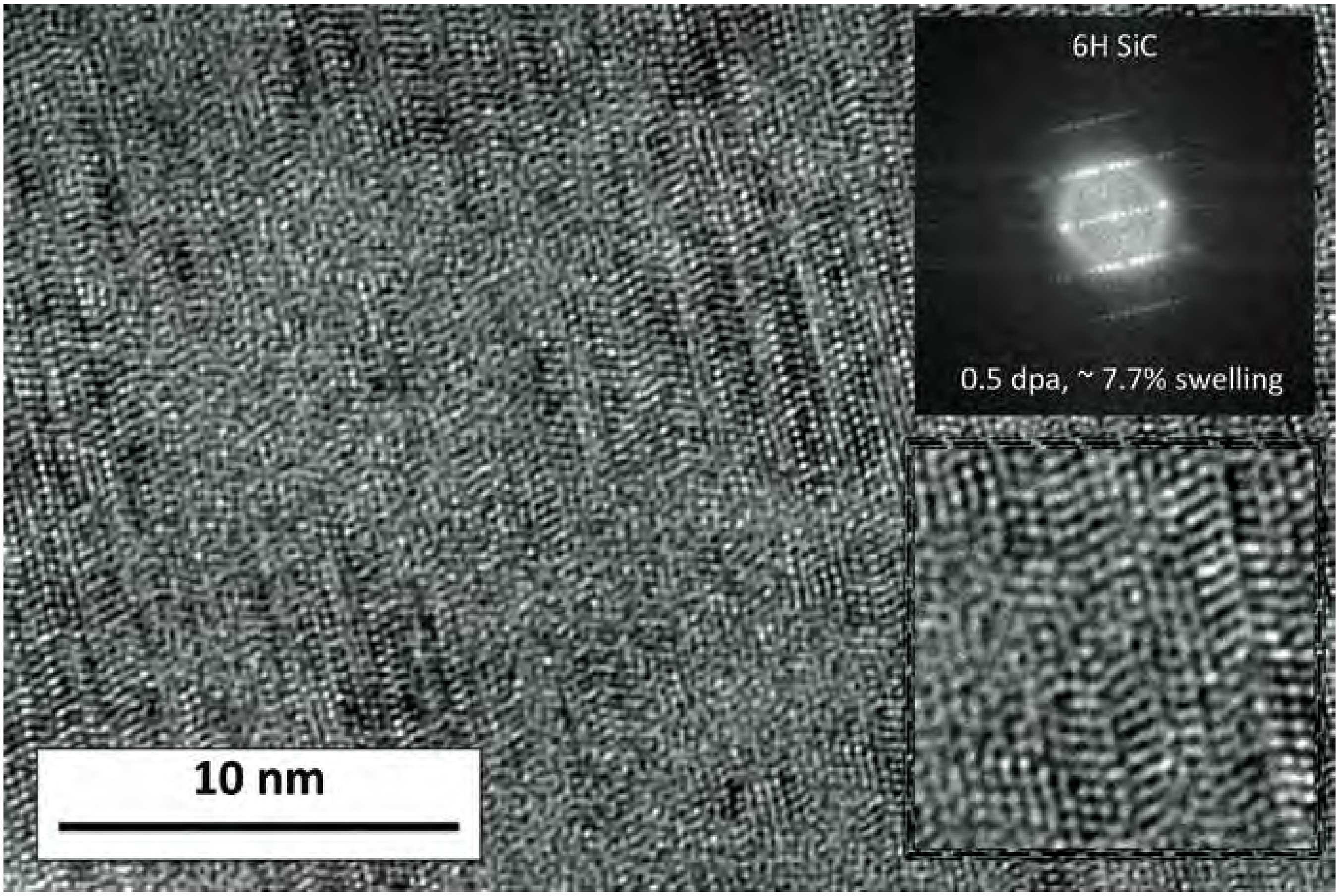

Figure 1: High resolution TEM Image of highly defected SiC sample

While stored energy is well understood conceptually and has been studied in detail for gas-cooled reactors, the Szilard Complication has been generally and practically thought to be unique to graphite. In other words, for practical nuclear systems the combined stored (Wigner) energy due to simple production is only an issue for graphite irradiated <200°C, where the subsequent energy release upon annealing greatly exceeds the materials' specific heat, driving an autocatalytic temperature rise. However, recent work on the gross swelling of silicon carbide has suggested significant Wigner energy in another important nuclear material (SiC) system may exist. (Snead et al. JNM 471 [2016], 92–96) In this project, the Wigner energy in highly defected, neutron irradiated SiC is determined and compared with physical swelling and lattice dilation. The extended goal of this research is to provide a full understanding of the SiC annealing kinetics irradiated at low to intermediate irradiation temperatures to judge whether Wigner energy could be an issue for the use of SiC at the relatively low temperature applications currently under consideration.

Project Description

The technical objectives for this project are two-fold. The first is the practical point of answering if Wigner energy exists in SiC to an appreciable amount, meaning can energy be stored and released such that an autocatalytic reaction takes place. Information on such a reaction would clearly be of concern for and necessary to reactor designers, which may be of increasing importance as SiC is now being considered for lower temperature applications (e.g., LWR cladding) as opposed to the historic HTGR application. In order to address this first point, a bounding set of materials was selected that would provide a maximum Wigner energy. The second goal of this work is to provide some fundamental and quantitative understanding of the process to help guide future research. This was accomplished by combining Wigner energy measurements with the study of underlying microstructural evolution.

The experimental approach was conceptually simple, taking advantage of high purity CVD SiC utilized in previous irradiation capsules irradiated in the High Flux Isotope Reactor. The specific designation of the historic samples were PC-1-M1 (0.02×10^{25} n/m^2), PC-2-M2(0.1×10^{25} n/m^2), PC-3-M3(0.05×10^{25} n/m^2), PC-4-M4(2×10^{25} n/m^2), and PC-5-M5(20×10^{25} n/m^2). Additionally, an opportunity to piggyback upon a set of irradiation vehicles of similar design (i.e, perforated HFIR rabbits) became available and was exploited. Specifically, Dr. Terrani arranged for space in a critical amorphization transition range. Data were also obtained on swelling at 0.762 and 1.34×10^{25} n/m^2, also at 90°C. In addition to the PI, the research team included Wally Porter, Takaaki Koyanagi, Yutai Katoh, and Kurt Terrani at ORNL. Energy release was carried out using a Netzch 404C DSC within the Low Activation Materials Development and Analysis (LAMDA) laboratory at ORNL. Supporting x-ray and transmission electron microscopy were also carried out to provide insight into the microstructure, also in LAMDA.

Accomplishments

In a previous paper by the author (Snead et al. 2016) an extraordinary level of swelling prior to amorphization in SiC was observed: ~8% <c> lattice expansion as measured by x-ray and density as measured by density gradient column. By straightforward calculation, assuming the annihilation of vacancies of ~5eV per vacancy-interstitial recombination, the restoration of the SiC structure through annealing would liberate ~1875 J/g. This suggests an energy release on the order of ~1.5 J/g-K as compared to an average specific heat for SiC

Similar to the well-known Wigner Energy release in irradiated graphite, for the first time this work demonstrates a significant energy release in a second important nuclear material, specifically silicon carbide.

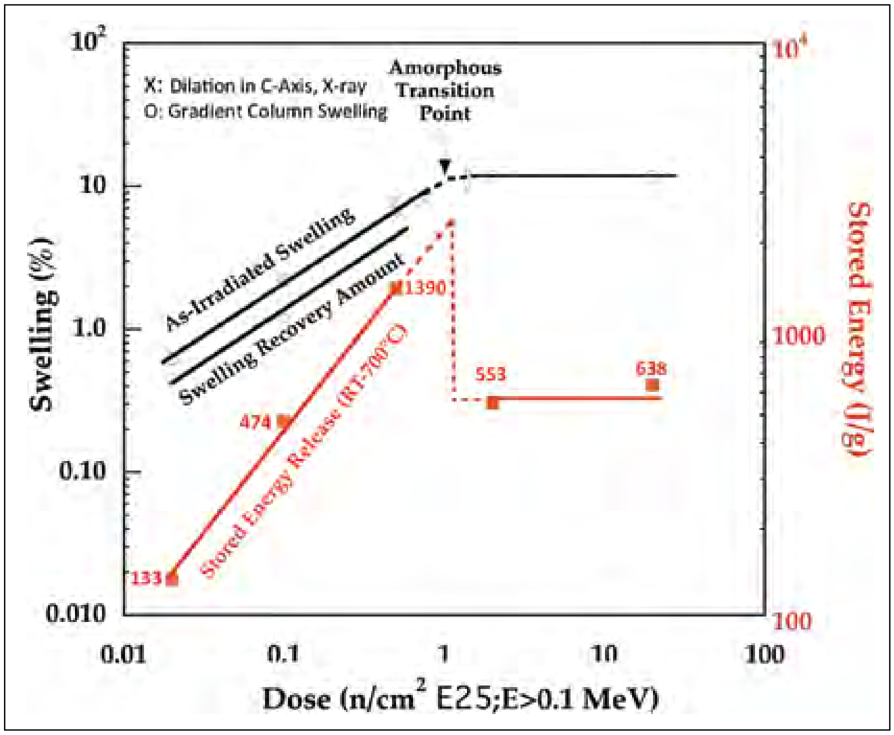

Figure 2: Swelling and energy release of irradiated SiC samples

(RT-700°C) of under ~1 J/g-K. Given this, it is a reasonable possibility that low-temperature irradiated SiC could have a Wigner energy release exceeding the specific heat and, therefore, leading to a positive temperature excursion. The primary goal of this work was to directly measure the energy release in such highly defected, highly strained material to scope the magnitude of the stored energy and rate of energy release in comparison with specific heat. An important secondary goal was to gain understanding of the underlying microstructure of the material in the irradiated and annealed material to guide future experiments.

An example of the highly defected structure is shown in Figure 1 for a highly pure CVD SiC material that underwent 7.7% swelling. Of interest is that the material has a large lattice strain due to the accumulation of irradiation induced defects. While the material clearly has remained crystalline, as evidenced by the diffraction pattern, discontinuous pockets of very highly damaged areas that approach amorphous materials are appearing. From the annealing literature, it is known that an approximately linear annealing curve behavior takes place in highly defected SiC with near complete annealing at or just above 1000°C. For this reason, it would be expected that a large number of the defects would annihilate upon high temperature annealing. Figure 2 provides results to date on irradiation induced swelling, swelling recovery, and Wigner energy release for the irradiated CVD SiC samples of this study (in this experiment, we were limited to 700°C annealing). From the figure, the swelling of the SiC is seen to follow a linear swelling with dose achieving a maximum of 8.13% volumetric expansion. The maximum energy release is 1390 J/g-K with a possible extrapolation to on the order of 2000 J/g-K. This amount of energy release, as discussed notionally above, is in excess of that necessary for a positive temperature excursion in SiC. As seen in the figure, above this dose and prior to the dose of 1.34×10^{25} n/m^2 (E >0.1 MeV) the crystal makes a transition from crystalline to amorphous, achieving a maximum volumetric expansion of ~11.7% (2.84 g/cm^3). Above the critical amorphization dose, the energy release substantially decreases, consistent with the notion of the material making a transition to a lower energy state.

The primary technical goal of identifying whether significant Wigner energy exists in SiC in any appreciable amount has been unambiguously demonstrated and for these irradiation conditions is clearly appreciable. It is noted that the irradiation conditions for executing this rapid turnaround experiment were chosen to provide maximum Wigner energy, and were for lower temperatures than LWR cladding or other currently conceived advanced reactor applications of SiC. In this light, a future, more comprehensive survey of Wigner energy for SiC as a function of irradiation and temperature would be prudent.

Future Activities

This rapid turnaround experiment has provided the first direct evidence of appreciable Wigner energy in SiC, albeit at temperatures lower than of general interest to nuclear applications. Follow-on work is suggested.

Publications

To be published

Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Oak Ridge National Laboratory	Low Activation Materials Design and Analysis Laboratory
Collaborators	
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In Situ observation of lunar-crater features in Xe irradiated UO_2 at high dose

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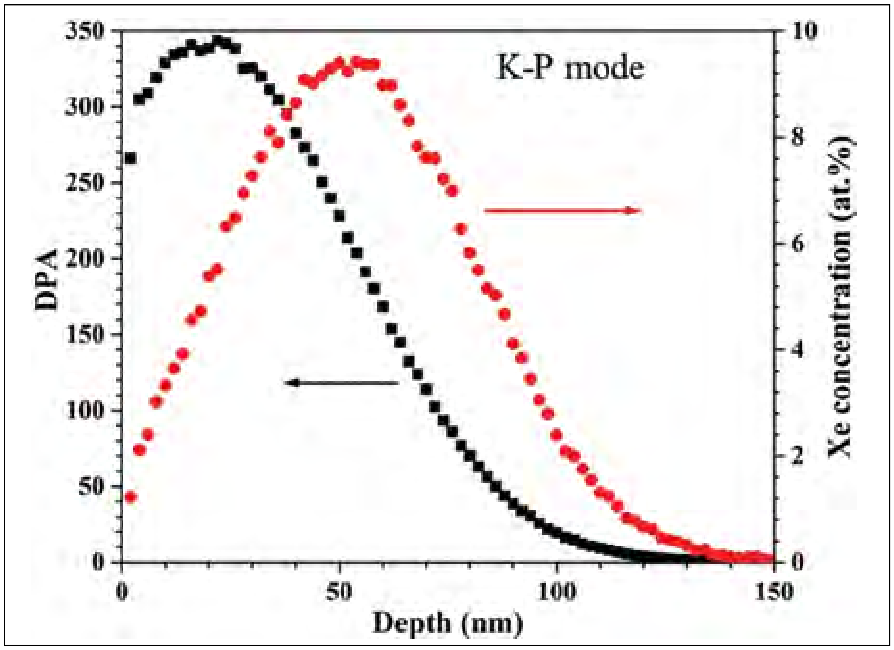

Figure 1. (Left) Argonne-IVEM for in situ TEM observation of UO_2 irradiated with 300 eV Xe. (Right) SRIM calculation showing the radiation damage level (dpa) and Xe concentration at 5×10^{16} ions/cm^2.

To simulate fission fragments damage and fission gas evolution in UO_2, the 300 keV Xe ion beam in the Intermediate Voltage Electron Microscope (IVEM)–Tandem facility at Argonne National Laboratory (ANL) was used to irradiate polycrystalline UO_2 lamella. The damage depth is around 150 nm, which is close to the thickness of UO_2 lamella and good for *in situ* observaton by transmission electron microscopy. This rapid turnaround experiment revealed the nucleation and evolution of lunar-crater features in UO_2 as a function of irradiation dose and temperature. The compositon and microstructure of lunar-crater features were analysed using dark field imaging, high resolution TEM imaging, energy dispersive x-ray spectroscopy (EDS), and energy-filtered TEM techniques. Small Xe bubbles were investigated by the underfocus and overfocus image techniques.

Project Description

Uranium dioxide (UO_2) is the most widely used nuclear fuel in commercial light water reactors. The cumulative radiation damage during the fission process causes severe degradation in the thermophysical properties of UO_2 fuels, which limits their lifetime and increases their operational cost. Therefore, investigating both defect production and evolution and fission product transport under irradiation to reveal their physical

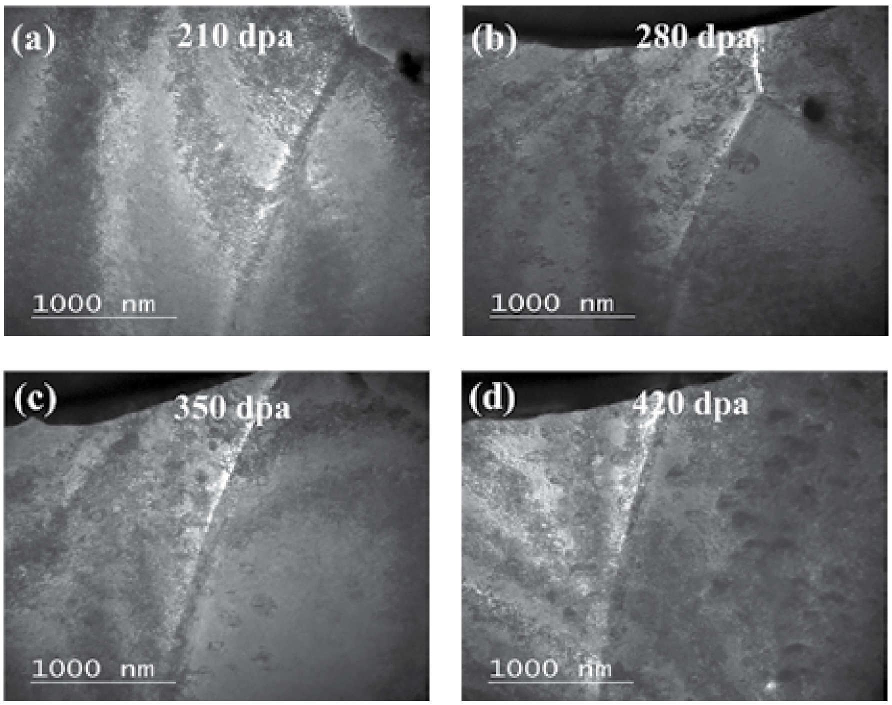

Figure 2. Lunar-crater feature evolution at 600°C as a function of dose (lunar-crater features occur at 280 dpa).

mechanisms is of great importance in understanding the degradation of thermophysical properties of UO_2 fuels. In this work, both *in situ* and *ex situ* TEM observation of defect nucleation and evolution under ion irradiation was conducted to understand the radiation damage mechanisms in UO_2 at high damage levels. The irradiation induced microstructure, including dislocation loops, inert gas bubbles, and lunar-crater features are characterized using state-of-the-art characterization tools. Revealing the nucleation and evolution of lunar-crater features at high dose is the main purpose of this study. The comparison between the microstructure features of UO_2 under various doses and temperatures will shed light on both the dose and temperature effects on lunar-crater features formation and evolution. Further, the experimental microstructure characterization will provide a fundamental foundation for atomic-level modeling, which is conducted at Idaho National Laboratory and Purdue University. This marks the first creation of lunar-crater features in UO_2. *In situ* studies of crater feature formation and evolution in UO_2 using the IVEM-Tandem facility provides a unique opportunity to study the effects of bubble pressure on microcracking, blistering, and exfoliation in UO_2. Our work has contributed to DOE's leading role in basic science research

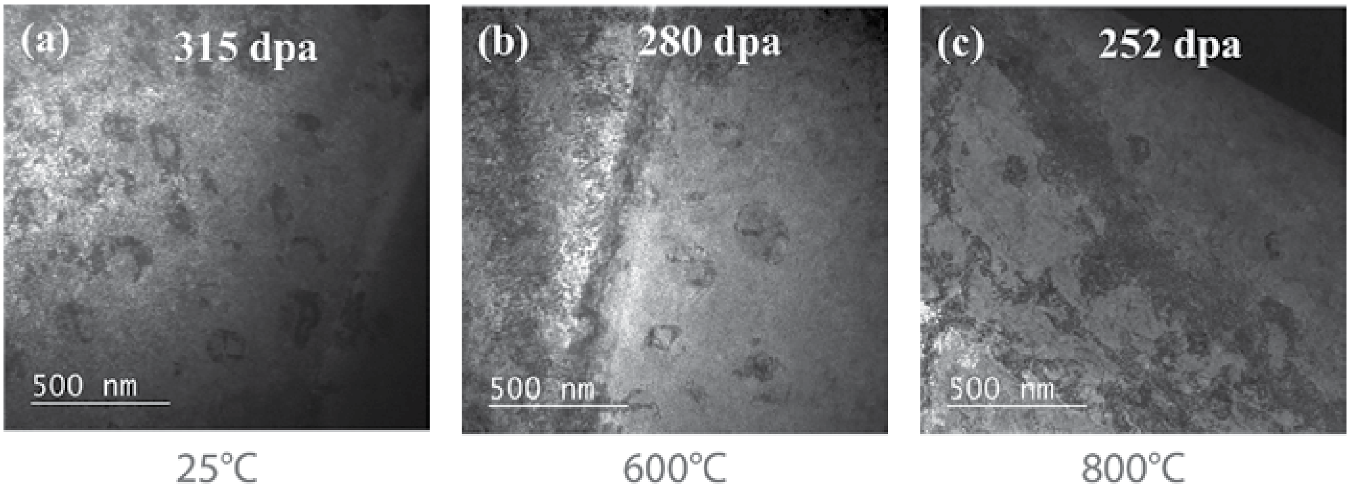

Figure 3. Lunar-crater feature evolution as a function of irradiation temperature (lunar-crater features occur at higher dose at lower temperature).

of materials behavior and performance in extreme environments. In addition, the microstructural characterization and modeling of radiation effects on UO_2 can guide the design of next generation nuclear fuels.

Accomplishments

The microstructure evolution under high dose irradiation in UO_2 is not yet well understood. The IVEM – Tandem Facility at Argonne National Laboratory was employed to study the defect evolution in Xe irradiated UO_2 at high dose (Figure 1). *In situ* TEM observation shows that the formation of crater featues depends on the ion dose and irradiation temperature (Figures 2 and 3). The higher the irradiation temperature, the lower the dose at which the lunar-crater featues occur. The crater features occur at a dose of 315 dpa at 25°C, at a dose of 280 dpa at 600°C, and at a dose of 252 dpa at 800°C. An extensive study on the chemical analysis using ChemiSTEM indicates the crater features are slighly rich in Xe compared to the matrix.

The formation mechanism of lunar crater features has been extensivly discussed. Xe-implantation induces various types of defects, such as interstitials and vacancies in UO_2. Vacancies act as efficient trapping centers for the implanted Xe and turn into Xe bubbles after trapping Xe (Figure 4). The implanted Xe segregates into bubbles, which thereby create internal pressure and reach an overpressurized state. When this internal pressure attains the fracture limit of UO_2, it ultimately lifts the implanted surface in the form of surface blistering and exfoliation and generates lunar-crater

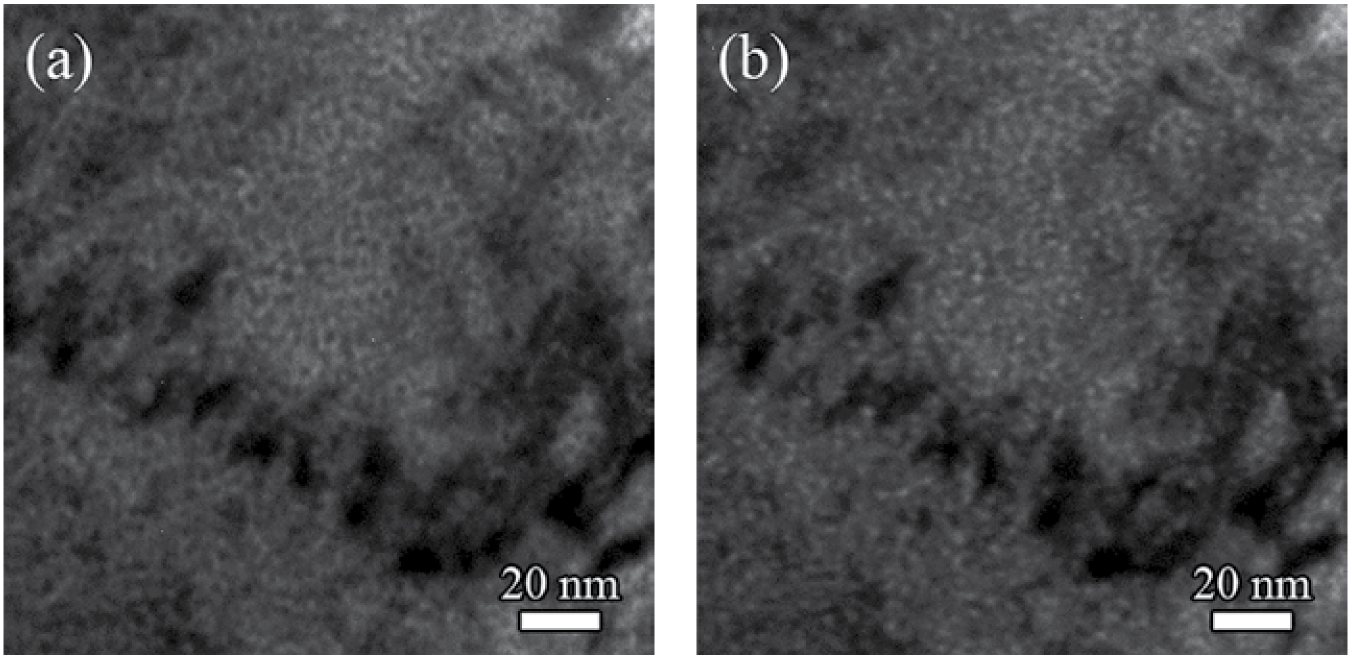

Figure 4. TEM images of bubbles in UO_2 irradiated with 300 keV Xe ions at 600°C up to a dose of 5×10^{16} ions/cm^2. (a) is an overfocus image, and (b) is an underfocus image. The bubble size and density are 2.3 ± 0.3 nm and $(8\pm4)\times10^{23}$ m^{-3}, respectively.

features in UO_2. The formation of lunar-crater features depends on both ion dose and temperature. The bubble pressure at high temperature is higher than that at low temperature, which makes the blistering and exfoliation occur at lower dose. However, the detailed mechanisms of the surface blistering and exfoliation are very complicated and more efforts are needed to understand them.

Matzke et al. investigated radiation effects in UO_2 under high dose Xe implantation by ion beam techniques (Rutherford backscattering and channeling) and found a critical dose typically around 3 to 5×10^{16} ions/cm^2 for the formation of a "polygonization peak" in single crystal UO_2 (Nuclear Instruments and Methods in Physics Research B 91 [1994] 294-300).

The implanted layer developed a structure of small crystallites, which were misaligned by a few degrees. The transformation could be produced at 77 K, room temperature, and 500°C in the same dose range. The dose level, Xe ion energy, and irradiation temperature in the present study are comparable to those in Matzke's work. The round features, therefore, could be the sign of the starting polygonization. HRTEM work by Matzke and Wang revealed the formation of subgrain boundaries in Xe irradiated UO_2 up to a dose of 5×10^{15} ions/cm^2, and the small subgrains of nanometer size were slightly rotated (about 1–2 degrees with respect to one another across the sub grain boundaries)

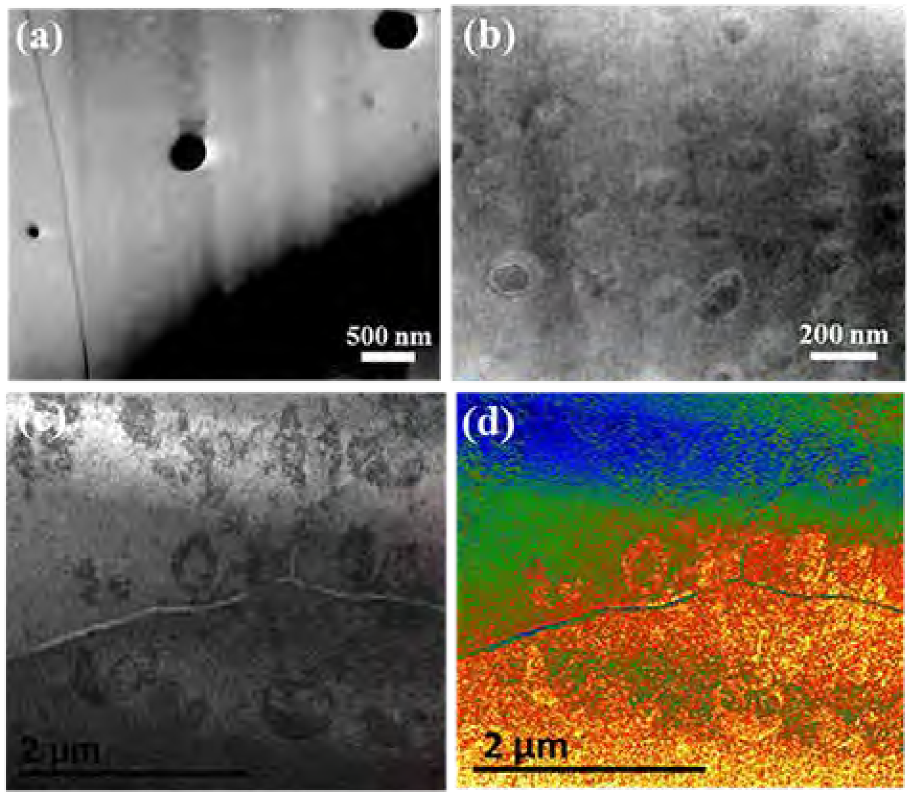

Figure 5. Lunar-crater features in Xe irradiated UO_2 at a dose of 350 dpa and 600°C. (a) low magnification and (b) high magnification STEM images; (c) TEM image; and (d) EFTEM image showing the thickness map of (c).

(Journal of Nuclear Materials 231 [1996] 155-158). These irradiation induced nano subgrains could be the nuclei for polygonization in UO_2, but the critical dose of the subgrain formation is one order of magnitude lower than that for the formation of polygonization peak and the round features found in this work. Alternatively, polygonization may also be induced by the overpressurized bubbles. The bubbles may cause cleavages and cracks on a very small scale, as well as amorphous tracks that may divide large grains. However, the direct evidence of the latter two mechanisms from TEM is still lacking. The size and density of the round features are also influenced by the foil thickness. Defects and Xe ions are more prone to accumulate at the thick foil regions as compared to the thin foil regions because the Xe out-diffusion is hindered and the surface cannot efficiently annihilate the irradiation defects. Thus, the high strain due to Xe build up and damage at the thick region may contribute to the formation of the observed round features.

This experiment will clarify the radiation damage mechanisms of UO_2 under ion irradiation at high dose.

Future Activities

Future goals for this research include simulating the nucleation and evolution of lunar-crater features in UO_2 under irradiation and bettering the understanding of formation mechanims. Lunar-crater features may also form in other materials, and *in situ* observation of formation and evolution of these features in other materials is worthy of further investigation. The grain boundary effects on the formation of lunar-crater features could be of importance, and further study is also needed. The polygonization mechanism in UO_2 is still under debate, and more efforts coupling experiments and computation are needed to clarify the mechanism.

Publications

[1.] L. He, X.M. Bai, J. Pakarinen, B. Jaques, J. Gan, A.T. Nelson, A. El-Azab, and T.R. Allen. "Bubble Evolution in Kr-irradiated UO_2 during Annealing," J. Nucl. Mater. 496 (2017), pp. 242–250.

[2.] L. He, J. Gan, M. Kirk, B. Tyburska-Pueschel, B. Jaques. "Radiation Damage on UO_2 and UN," TMS 2017 Annual Meeting & Exhibition; Feb 26-March 2, 2017, San Diego, California, USA (Invited).

The IVEM is a powerful instrument offering in situ observation of defect nucleation and evolution under irradiation

— Lingfeng He, Senior staff scientist

Distributed Partnership at a Glance

NSUF and Partners	Facilities and Capabilities
Argonne National Laboratory	The Intermediate Voltage Electron Microscopy – Tandem Facility
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Argonne National Laboratory	Marquis A. Kirk (collaborator)
Idaho National Laboratory	Jian Gan (collaborator), Lingfeng He (principal investigator)

Investigation of the interface strength for matrix-fiber interface of irradiated SiC composite materials

Yutai Katoh – Oak Ridge National Laboratory – katohy@ornl.gov

SiC/SiC composites push the boundary for operational performance in extreme environments. Detailed characterization and experimental understanding of interface properties will enable microstructurally informed models for improved engineering predictability.

SiC/SiC composites exhibit exceptional high-temperature strength and radiation-resistant properties, making them an advantageous choice for accident-tolerant nuclear fuel cladding. The composite architecture allows an intrinsically brittle material to exhibit sufficient structural toughness. This study evaluates the importance of the fiber/matrix interphase as it relates to composite toughening mechanisms. Small-scale mechanical testing along with atomic-force and transmission electron microscopy analyses have been employed to evaluate PyC interphase properties that play a key role in the overall mechanical behavior of the composite. The Mohr-Coulomb failure criterion allowed for the extraction of an internal friction coefficient and debonding shear strength as a function of the PyC layer thickness and irradiation.

Project Description

The technical objective of this research is to use experimental techniques to investigate the change in fiber-matrix interphase properties for irradiated SiC/SiC composite materials. In particular, the focus is on evaluating ultimate shear strength and friction properties in the irradiated specimens by preparing micropillars using scanning electron microscopy/focused ion beam (FIB) and *in situ* flat-punch compression testing via nano indentation. With a comprehensive and fundamental understanding, the property data and associated impact on mechanical behavior could be implemented into a predictive model, improving safety and reliability for accident tolerant fuel. Establishing this new state of knowledge has significant impact on academic theory of ceramic composite toughening mechanisms, as well as industrial application based component design. Macroscopic failure analysis has led to linear elastic fracture models that describe the role of the interphase in micro-crack deflection. However, experimental validation of these physics models has not been fully exhausted. Micropillar compression allows for a more intrinsic understanding of interface properties and is expected to aid in theory validation. For industrial application, traditional homogenized composite modelling techniques can implement these extracted property data directly. In this manner, the effects of irradiation on interphase-dependent mechanical behavior may be systematically explained. This will result in design improvement which can be implemented to enhance composite

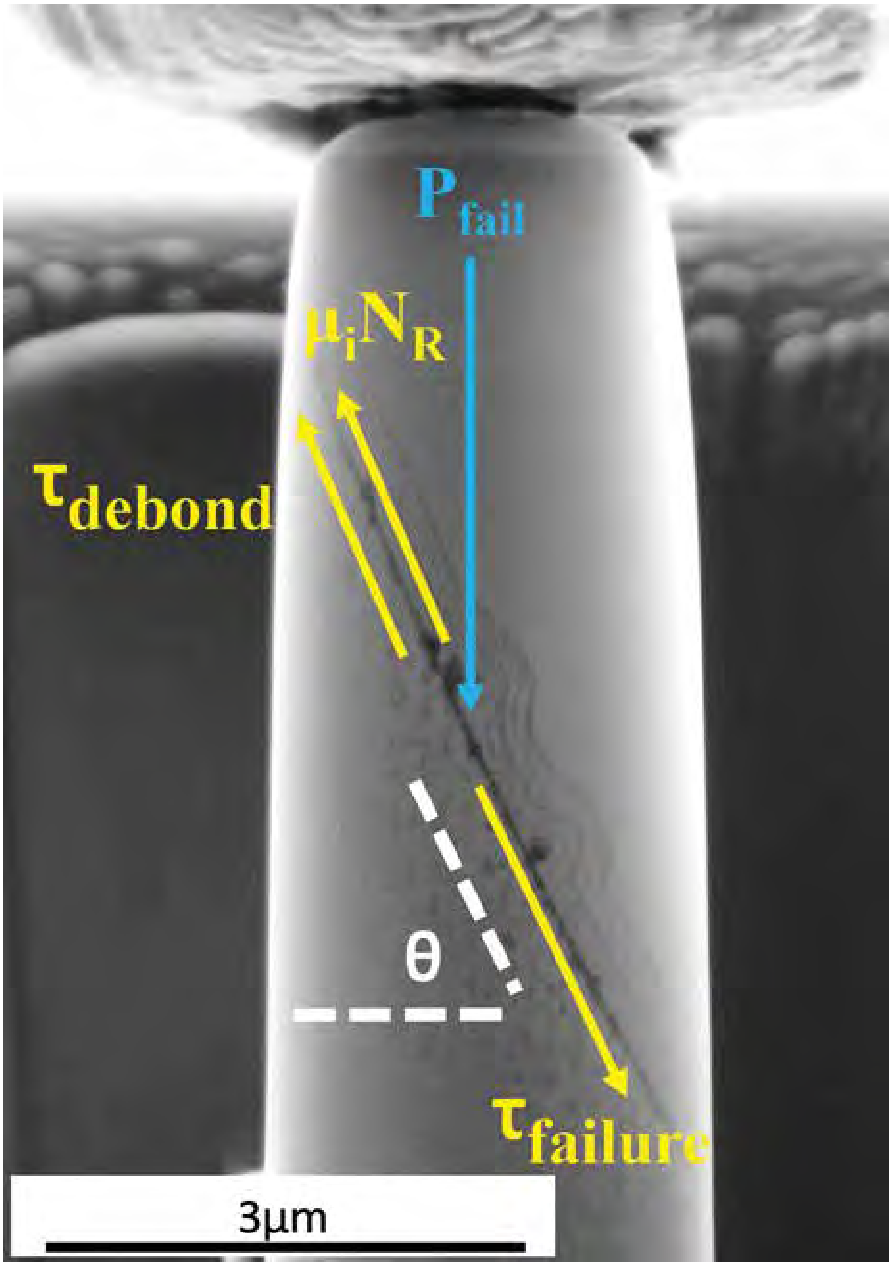

Figure 1. A typical 3 µm micro-pillar structure undergoing in situ compression test. The stress state and force balance that are used to extract debond shear strength and internal friction values are overlaid.

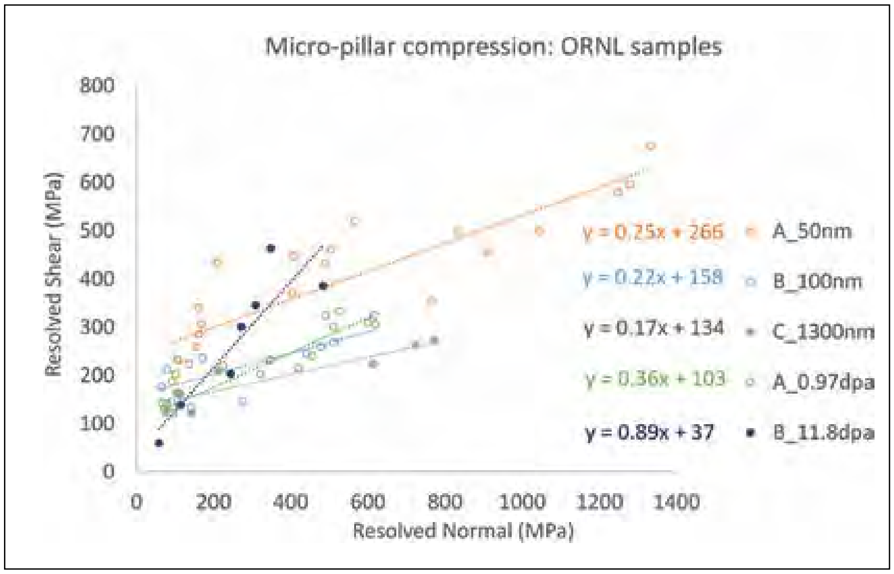

Figure 2. Extraction of the debond shear strength (intercept) and internal friction coefficient (slope) by application of the Mohr-Coulomb fracture criterion that plots the resolved shear stress versus the resolved normal stress at failure.

performance in neutron irradiation environments and will thereby support the mission of DOE-NE for reliable and efficient nuclear power systems.

Accomplishments

The technical goal of this research was to compare pristine and irradiated interface property data via micro-pillar compression. This goal was achieved through refinement of micro-pillar fabrication, increased sampling size, and application of the Mohr-Coulomb Mohr failure criterion. As the project moved forward, it quickly experienced success and additional samples with variable PyC interface thicknesses were also received, allowing for more thorough understanding of interface design space. Pragmatically, the goals of this research required strong cooperation and communication between all participants for handling and evaluation of the irradiated specimens. Takaaki Koyonagi of ORNL was a quick and reliable correspondent with attention to detail. He was responsible for obtaining, tracking, and communicating the expectations for the pre-irradiated SiC/SiC composite samples. With access to state-of-the-art facilities, he was also able to polish the irradiated samples prior to shipment to UC Berkeley, which simplified work for the university and allowed for expedited results. In total, two pristine control samples, two irradiated samples, and one thick PyC interface sample were obtained by Oak Ridge National Laboratory, followed by interface analysis at UC Berkeley. All samples contained HNLS nuclear grade fibers. Sample A and A_rad exhibited five alternating layers of PyC/SiC before full CVI SiC infiltration, while sample B and B_rad were fabricated to have a single monolayer of PyC at the fiber surface. Sample C exhibited a monolayer PyC interface ~1300 nm thick. Sample A_rad received 0.97 dpa at 300°C, and sample B_rad received 11.8dpa at 280°C.

The primary technique applied in this research was micro-pillar compression. Compared to fiber push out testing, micro-pillar structures can offer refined property extraction (τ_{debond} and μ), interface characterization, and consistent trials. Focused ion beam (FIB) milling techniques were used to fabricate the micro-mechanical structures. Pillars (~3 µm in diameter) were fabricated with a fiber/matrix interphase spanning its cross-section. All milling processes followed a similar cutting sequence with a rough cut (10–15 nA) followed by finishing cuts (0.1–0.5 nA). Once fabricated, the pillars were tested *in situ* with the SEM and Hysitron PI-85 Pico Indenter using a 5 µm flat punch diamond tip, with displacement controlled loading at 10 nm/s. The experimental work and data analysis were primarily carried out by UC Berkeley graduate student Joey Kabel. Figure 1 shows a representative micro-pillar structure and overlaid stress state during failure.

When samples are tested in compression, the Mohr-Coulomb criterion describes the stress state at failure with equation 1:

$$\frac{(P_{break} * \sin\theta)}{A} = \tau_{debond} + \frac{(P_{break} * \cos\theta)}{A} * \mu \qquad (1)$$

allowing for the extraction of τ_{debond} and μ. For each test, the failure load (P_{break}) was extracted from the load vs displacement curves and used to calculate the resolved normal stress and resolved shear stress during failure, at the failure plane. Each sample yielded data for its respective incline plane, which these data were then plotted. Applying the Mohr-Coulomb criterion allowed us to understand how the contributing debond resistance properties were evolving as a function of irradiation. In Figure 2, we plot the resolved shear stress versus the resolved normal to apply a linear fit, for which the slope is equivalent to the internal friction coefficient and the intercept equivalent to the chemical debonding stress. Table 1 summarizes the extracted properties.

The availability, preparation, and testing of the irradiated SiC/SiC samples has expanded and enriched the content of my PhD thesis. I'm grateful for this opportunity to collaborate.

— Joey Kabel,
Graduate Student Researcher

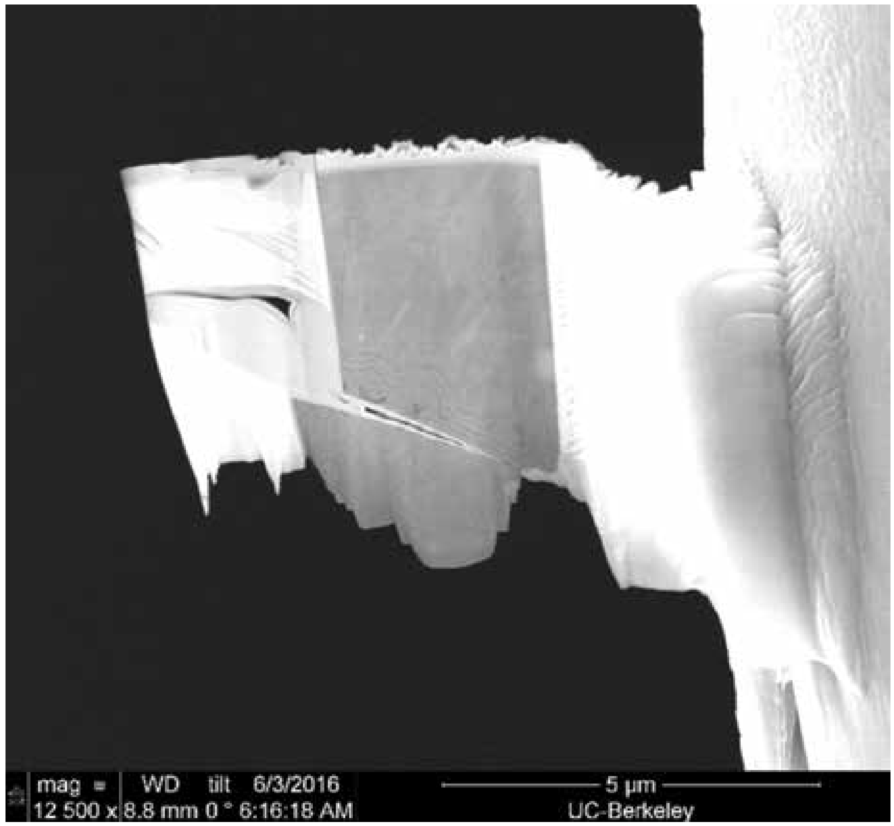

Figure 3. TEM foil lift out of the fracture pillar with PyC interface. The TEM analysis showed failure occurred within the PyC structure, suggesting properties are dependent on PyC structure.

TEM analysis of failed pillar interfaces, Figure 3, revealed that failure was occurring within graphite-like structure of the PyC interface, suggesting that extracted properties are inherent to the deposited PyC.

It can be observed that while the chemical debonding strength decreases with irradiation, the friction coefficient appears to increase. With respect to thickness, both the debond strength and internal friction coefficient are observed to decrease. This may be a result of graphite-like basal plane alignment/long range order for increased deposition thickness, and decreased long range order during irradiation. Another influence was the significant non-uniform porosity that evolved in the 11.8 dpa sample, shown in Figure 4.

It is believed that this porosity is responsible for the low statistical confidence in the extracted properties. However, the trend of increased internal friction and decreased strength is still observed, likely enhanced by the porosity.

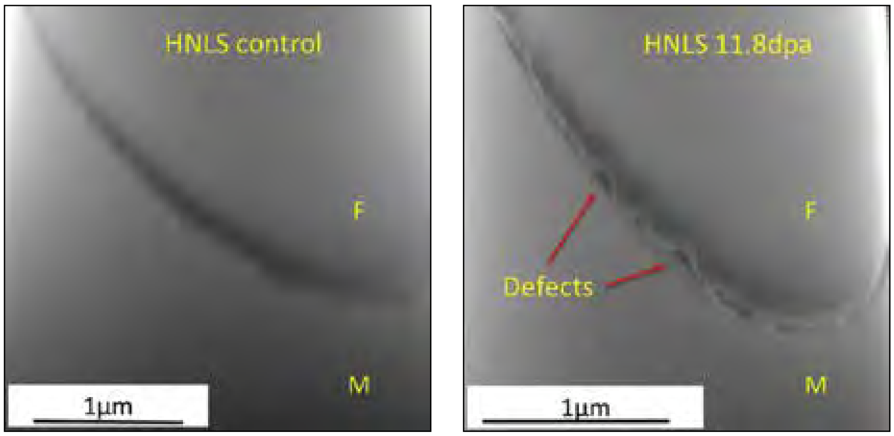

Figure 4. Observation of PyC interface degradation at 11.8 dpa sample irradiated at ~260–280°C. These high dose pillars showed increased internal friction and decreased debond shear strength.

Fiber	τdebond (MPa)	Internal friction coefficient (μ)	Successful tests
A (50nm PyC)	266 ± 23	0.25 ± 0.03	17
B (100nm PyC)	158 ± 19	0.22 ± 0.06	11
C (1300nm PyC)	133 ± 14	0.17 ± 0.03	8
A_{rad} (0.97 dpa)	103 ± 11	0.36 ± 0.03	16
B_{rad} (11.8 dpa)	37 ± 59	0.89 ± 0.20	7

In conclusion, micro-pillar compression was implemented to examine the interface properties of SiC-SiC composite interphases as a function of irradiation and thickness. SEM and TEM observations revealed insight into interface failure mechanisms and irradiation damage, providing some explanation of the observed PyC property evolution.

Future Activities

In the final month of 2017, continued data analysis and write up (for both journal submissions and conference procedings) are expected. Future goals for this project may include a more rigorous TEM analysis and understanding of PyC degradation and falure mechanisms as a funciton of dpa and thickness. It is also expected

Figure 5. Joey Kabel (graduate student) trains Ruijie Shao (undergraduate student) on micro-pillar fabrication techniques using FEI Quanta dual beam FIB at the University of California, Berkeley Nuclear Materials Laboratory, an NSUF Partner Facility.

that increasing the number of pillars tested, especially for the 11.8 dpa sample, will increase statistical fidelity and improve confidence in property implementation into finite element models. Another step forward would be to characterize the mode I fracture release rate energy as it applies directly to understanding limitations and interface design optimization for microcrack deflection. We believe the collaboration has been successful and are excited to continue our work through additional proposals and utilization of the Nuclear Science User Facilities.

Publications

[1.] J. Kabel, Y. Yang, M. Balooch, C. Howard, T. Koyanagi, K. A. Terrani, Y. Katoh, and P. Hosemann, "Micro-Mechanical Evaluation of SiC-SiC Composite Interphase Properties and Debond Mechanisms J. Kabel," Compos. Part B Eng., vol. 131, pp. 1–18 (Elsevier), 2017.

[2.] J. Kabel, P. Hosemann, Y. Zayachuk, D. E. J. Armstrong, T. Koyonagi, Y. Katoh, C. Deck. "Ceramic fiber-matrix composite interface property evaluation and testing on SiCf / PyC / SiCm composites." J. Mat. Res.

[3.] J. Kabel, M. Balooch, Y. Yang, T. Koyanagi, K. A. Terrani, P. Hosemann. "SiC-SiC Composite Interphase Evaluation via Small Scale Mechanical Testing." American Nuclear Society. Winter Meeting 2016; Las Vegas USA.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
University of California, Berkeley	Nuclear Materials Laboratory
Collaborators	
Oak Ridge National Laboratory	Takaaki Koyanagi (collaborator), Yutai Katoh (principal investigator)
University of California, Berkeley	Joey Kabel (collaborator), Peter Hosemann (collaborator)

A TEM study of proton, heavy-ion and neutron irradiated FeCr

Steve Roberts – University of Oxford – steve.roberts@materials.ox.ac.uk

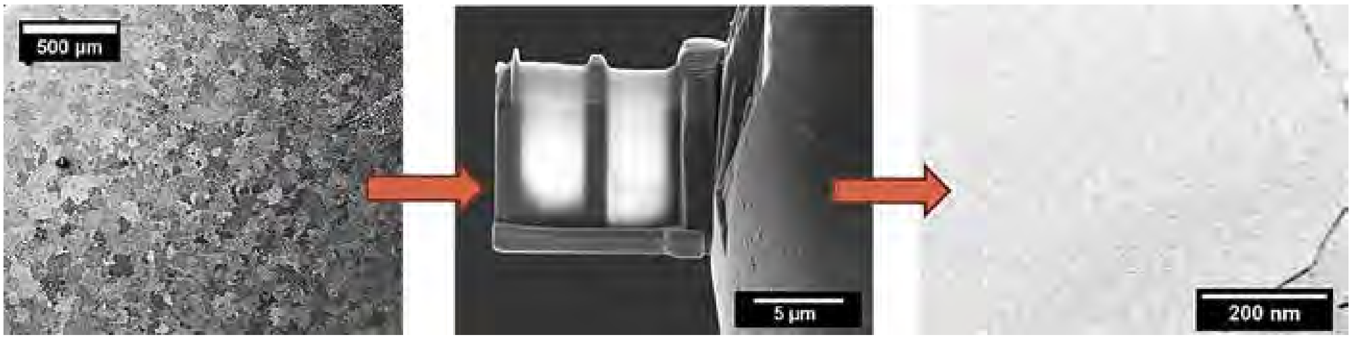

Figure 1: From left to right: SEM image of the grain structure; FIB lift out produced with two "windows" to reduce foil bending and TEM image of typical unirradiated microstructure.

The main focus of this project is to investigate the extent to which surrogate irradiations by protons and heavy ions can be used to reproduce the microstructural and mechanical changes brought on by neutrons in ferritic-martensitic (FM) materials.

Reduced activation ferritic-martensitic (RAFM) steels are promising materials for structural components and more accident tolerant fuel clad considered for future fission devices. Their advantages include reduced swelling compared to austenitic steels and no long term induced radioactivity. To predict their performance in a working reactor, the effects of neutron damage on RAFM materials must be tested and understood. Fission reactors are the closest analogue currently available to test materials in the radiation environment expected within such advanced nuclear devices. However, such experiments are complex, long, and expensive, with very little freedom to control the irradiation parameters. The alternative is to irradiate samples with charged particles such as heavy or light ions or electrons. Charged particles allow greater control over irradiation parameters, little or no radioactivity induced into samples, and far less time required to reach a particular dose. In order to validate the use of such surrogate particles, they must be directly compared with neutron irradiation under matching conditions.

Radiation damage is highly dependent on the chemical composition of a material. Complex RAFM steels, such as T91, have many elements which complicate the analysis of the radiation damage and comparisons with other steels. In order to understand radiation damage in such steels, it is essential to understand the radiation damage in simpler alloys. Therefore, this project studies binary alloys of FeCr.

Highlighting the differences among neutron, proton and self ion irradiation is crucial to understanding and interpreting irradiation experiments that attempt to mimic reactor conditions with charged particle beams.

Project Description

The objective of this work was to compare the microstructures of proton, heavy ion, and neutron irradiated FeCr binary alloys. Alloys of Fe6Cr and Fe9Cr were selected for their particular relevance to candidate RAFM steels. Of particular interest were the dislocation loops and cavities that can be produced during irradiation, and also changes (if any) to chromium distribution in the alloys.

The neutron irradiated alloys studied in this work are of considerable relevance to the development of RAFM steels as they will improve our understanding of the role chromium has in the evolution of microstructural defects during irradiation and subsequent radiation induced hardening. Understanding these mechanisms is invaluable to the development of new RAFM steels. Furthermore, comparisons to proton and heavy ion irradiation damage will lead to improvements in the design of irradiation experiments that use charged particles to replicate reactor conditions.

In order to achieve the objectives of this project, access to a facility capable of handling radioactive neutron irradiated samples was required. Neutron irradiated materials were produced in the University of California at Santa Barbara (UCSB)-Advanced Test Reactor (ATR) 1 irradiation campaign, carried out by Robert Odette in 2009. The materials originated from capsule 1A of this experiment. Since the irradiation experiment, these materials were stored at Idaho National Laboratory (INL) to allow radioactivity to reduce. The alloys were irradiated to ~1.8 dpa at 288°C over 200 days. The focused ion beam (FIB) microscope at the Center for Advanced Energy Studies (CAES) was used to prepare lift outs for transmission electron microscopy on each of these alloys. The CAES facility was ideal for this work because of the experience of CAES and INL staff with working on active material and close proximity to INL.

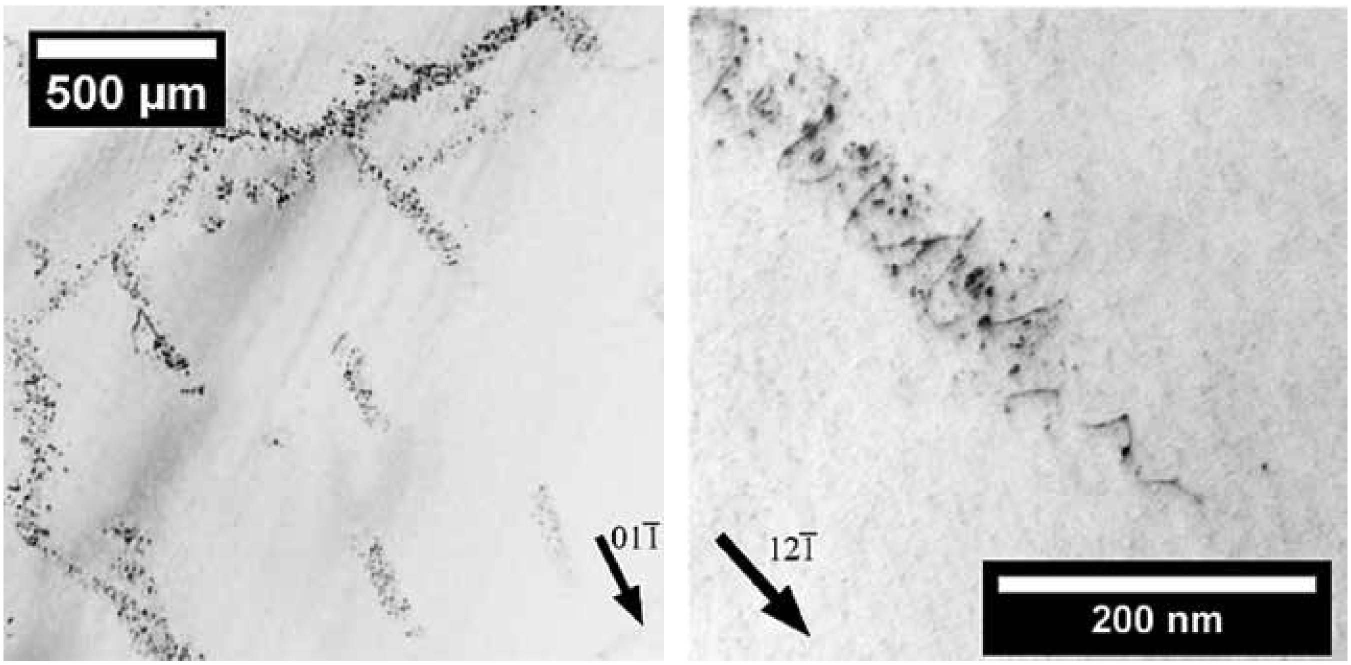

Figure 2. Neutron irradiated Fe9Cr microstructure. Dark field micrograph is shown with inverted contrast. Figure shows the pre-existing screw dislocations transformed into helices with a locally high population of dislocation loops.

Accomplishments

FIB lift outs were prepared initially at CAES before being shipped to the UK, where final thinning of the TEM foils was conducted (Figure 1). It was important to prepare the samples in this two-stage process in order to minimize the time between foil-thinning and analysis in the TEM. Successful sample preparation has been completed for both Fe6Cr and Fe9Cr alloys.

Ion irradiation and proton irradiation of the same materials has also been conducted to match the dpa dose and temperature of the neutron irradiated material as closely as possible.

Weak-beam dark field (WBDF) imaging in the TEM has shown the neutron irradiated Fe9Cr microstructure contains dislocation loops, which form from the point defects produced after collision cascades. Both 1/2<111> and <100> Burgers vector dislocation loops were found in the material, which can both cause hardening in the material by impeding dislocation motion. The defects have a highly heterogeneous spatial distribution in the grains; the majority of dislocation loops are found decorating dislocations that existed prior to irradiation (Figure 2).

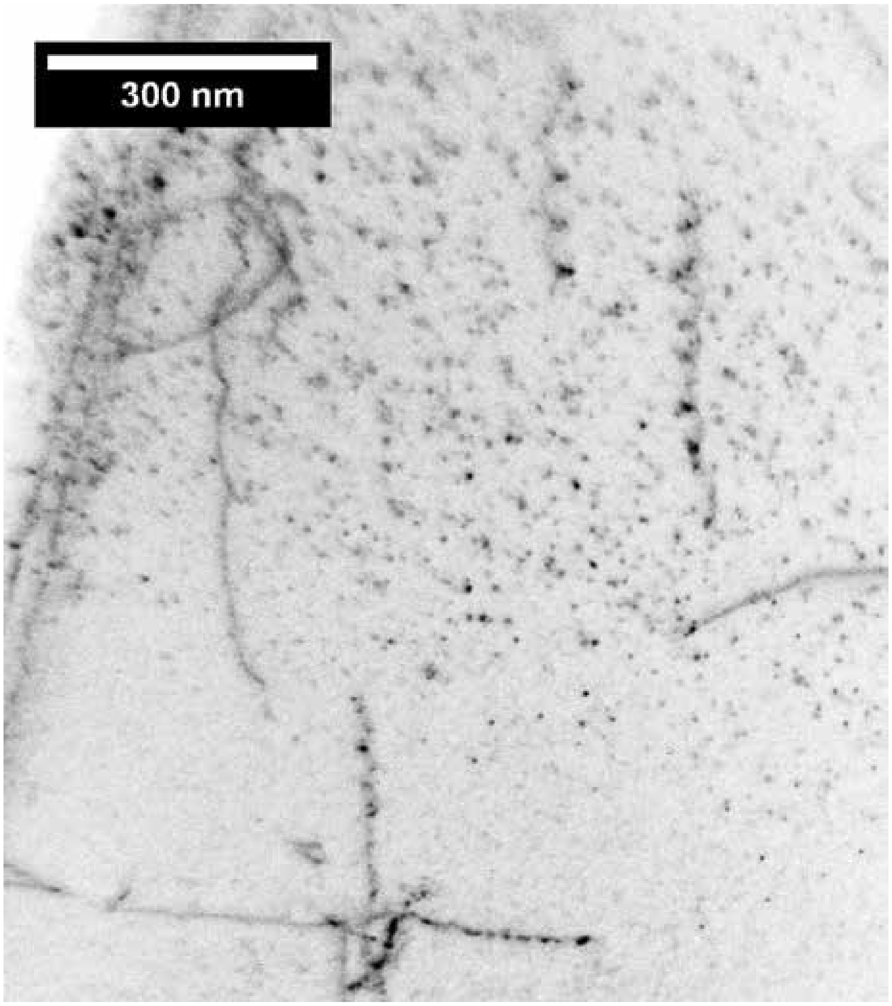

Figure 3. Ion irradiated Fe9Cr showing helical dislocations in the damaged region and straight dislocations in the unirradiated region.

The microscopes and personnel at the CAES facility are world class when it comes to working with active material, and the close ties with INL were invaluable.

— Jack Haley,
Co Principal Investigator

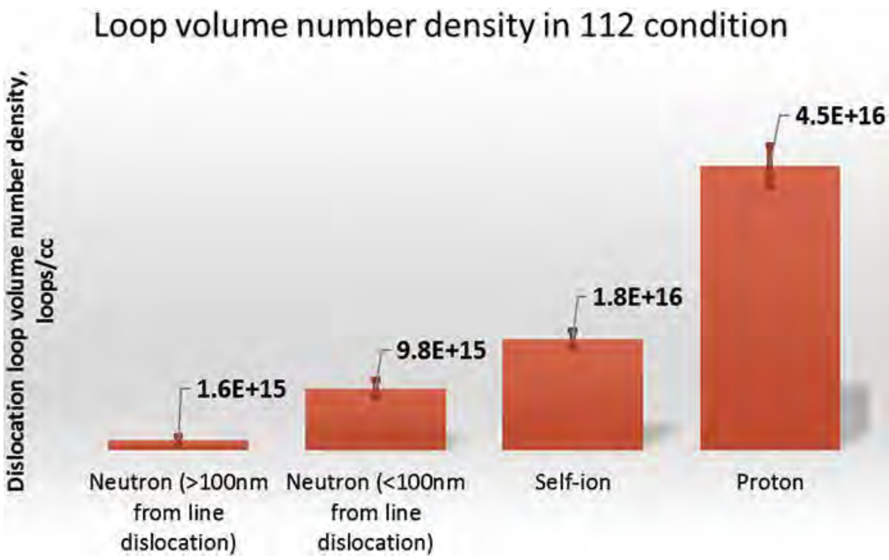

Figure 4. The number density of dislocation loops visible in a 112-like condition (with one variant of the <111> loops absent) is shown here.

An interesting observation is that the dislocations decorated with defects are helical. Ion irradiation has shown that the helical shape of the dislocation was initially straight and mainly screw in character (Figure 3). Screw dislocations can experience a climb in response to the absorption of point defects (interstitial or vacancy), which results in the dislocation taking a helical shape as it gains edge character, with a Burgers vector pointing along the axis of the helix.

Our analysis suggests that a bias of interstitial defects was the cause of the helical form.

Dislocation loops were counted and sized using micrographs from several g-vector conditions with varying visible proportions of loops with Burgers vectors of <100> and 1/2<111>. Loop counts were then derived based on statistical analysis. The work on the ion irradiated samples is ongoing, so the number density of dislocation loops visible in a 112-like condition (with one variant of the <111> loops absent) is shown in Figure 4.

This analysis shows that far more defects are present in the charged-particle irradiated alloys than the neutron irradiated alloy. The cause of this difference is likely the dose-rate effect because neutron irradiated alloys were damaged at a rate that was ~100 times slower than the charged particle irradiated alloys. For a high dose rate, cascade-damaged regions may be subject to multiple cascades in a short space of time, which can inhibit defects from migrating or annihilating. This favours dislocation-loop nucleation and may explain the larger number of visible defects in the proton and ion irradiated cases. This will also favor a homogeneous spatial distribution of dislocation loops.

The proton irradiated Fe9Cr alloy contains ~2.5 times as many loops as the equivalent ion irradiated alloy, despite having been irradiated at the same dose rate. This result could be due to differences in the primary knock-on atom (PKA) spectrum of the protons and ions (and neutrons). Proton irradiation favors lower PKA energies; hence, the collision cascades will be small compared to those produced by heavy ions or neutrons. Small cascades result in a greater proportion of surviving point defects than large cascades, which means that protons will create a larger number of surviving point defects for the same dpa dose as ions and neutrons.

Future Activities

Our future activities include completion of the analysis for the Fe6Cr and improvements to the analysis of the Fe9Cr. We intend to find the nature of the loops decorating the helices in both neutron irradiated alloys and for the ion and proton irradiated alloys, too. Chemical mapping via electron energy loss spectroscopy is also being planned to study chromium precipitation (if any).

Publications

None at present, but we expect the main results to be published in 2018.

Distributed Partnership at a Glance	
NSUF and Partners	**Facilities and Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
University of Oxford	Jack Haley (co principal investigator), Steve Roberts (principal investigator)
University of California, Santa Barbara	G. Robert Odette (collaborator)

Removing a fuel element during a routine refueling operation, High Flux Isotope Reactor (HFIR), ORNL (Courtesty of ORNL)

NSUF LIST OF ACRONYMS

ACRR .... Annular Core Research Reactor
AIME .... The American Institute of Mining, Metallurgical, and Petroleum Engineers
AM .... additive manufacturing
AMS .... accelerator mass spectrometry
ANL .... Argonne National Laboratory
ANS .... American Nuclear Society
APS .... Advanced Photon Source
APT .... Atom Probe Tomography
ARRM .... Advanced Radiation Resistant Materials
ATR .... Advanced Test Reactor
BNL .... Brookhaven National Laboratory
BR2 .... Belgian Reactor 2
BR3 .... Belgian Reactor 3
CAES .... Center for Advanced Energy Studies
CASS .... cast austenitic stainless steel
CCFE .... Culham Centre for Fusion Energy
CDRW .... capacitator-discharge resistance welding
CINR .... Consolidated Innovative Nuclear Research
CNNC .... China National Nuclear Corporation
CNWG .... Civil Nuclear Energy Research and Development Working Group
CoMET .... Combined Materials Experiment Toolkit
CP Ti .... commercially pure titanium
DMLS .... direct metal laser sintering

DOE .... U.S. Department of Energy

DOE-ID .... U.S. Department of Energy Idaho Operations Office

DOE-NE .... Department of Energy Office of Nuclear Energy

EBSD .... electron backscatter diffraction

EDX .... Energy-dispersive X-ray spectroscopy

EIS .... electrochemical impedance spectroscopy

EML .... Electron Microscopy Laboratory

EPMA .... electron probe microscope

EPRI .... Electric Power Research Institute

ETR .... Engineering Test Reactor

EXAFS .... X-ray absorption fine structure

FaMUS .... Fuels and Materials Understanding Scale

FASB .... Fuels and Applied Science Building

FCF .... Fuel Conditioning Facility

FEM .... finite element method

FIB .... Focused Ion Beam

FM .... ferritic-martensitic

FOA .... Funding Opportunity Announcement

FRIB .... Facility for Rare Isotope Beams

FY .... fiscal year

GAIN .... Gateway for Accelerated Innovation in Nuclear

GB .... grain boundary

GIF .... Gamma Irradiation Facility

HDR .... high dose rate

HFEF .... Hot Fuel Examination Facility

HFIR .... High Flux Isotope Reactor

HIP .... hot isostatic pressing

HIPPO .... High-Pressure-Preferred Orientation

HPC .... **High Performance Computing**

I3TEM .... *In Situ* **Ion Irradiation Transmission Electron Microscope**

IASCC .... **irradiation assisted stress corrosion cracking**

IMCL .... **Irradiated Materials Characterization Laboratory**

INL .... **Idaho National Laboratory**

ISNAP .... **Institute for Structure and Nuclear Astrophysics**

IVEM .... **Intermediate Voltage Electron Microscope**

IYMC .... **International Youth Nuclear Congress**

JAEA .... **Japanese Atomic Energy Agency**

JANNuS .... **Joint Accelerators for Nanosciences and Nuclear Simulation**

LAMDA .... **Low Activation Materials Development and Analysis**

LANL .... **Los Alamos National Laboratory**

LANS .... **Los Alamos Neutron Science Center**

LDR .... **low dose rate**

LEAP .... **Local Electrode Atom Probe**

LHMA .... **Laboratory for High and Medium Activity**

LOI .... **Letter of Intent**

LWR .... **light water reactor**

LWRS .... **Light Water Reactor Sustainability**

MaCS .... **Microscopy and Characterization Suite**

MFC .... **Materials and Fuels Complex**

MIBL .... **Michigan Ion Beam Laboratory**

MIT .... **Massachusetts Institute of Technology**

MOU .... **Memorandum of Understanding**

MOX .... **mixed oxide**

MRCAT .... **Materials Research Collaborative Access Team**

MSI .... **Minority-Serving Institutions**

MSTL .... **Materials Science and Technology Laboratory**

MSU .... Michigan State University

MTR .... Materials Testing Reactor

NADM .... Nuclear Academics Discussion Meeting

NASA .... National Aeronautics and Space Administration

NEID .... Nuclear Energy Infrastructure Database

NFMC .... Nuclear Fuels and Materials Characterization Facility

NFML .... Nuclear Fuels and Materials Library

NPIC .... Nuclear Power Institute of China

NRC .... U.S. Nuclear Regulatory Commission

NRL .... Nuclear Reactor Laboratory

NSCL .... National Superconducting Cyclotron Laboratory

NSLS .... National Synchrotron Light Source

NSUF .... Nuclear Science User Facilities

NuMat .... Nuclear Materials Conference

ODS .... Oxide-Dispersion-Strengthened

ORNL .... Oak Ridge National Laboratory

OSU .... The Ohio State University

OSURR .... The Ohio State University Research Reactor

PDC .... polymer-derived ceramics

PFC .... planning and financial control specialist

PIE .... post-irradiation examination

PKA .... primary knock-on atom

PM .... powder metallurgy

PNNL .... Pacific Northwest National Laboratory

PPMS .... Physical Properties Measurement System

RaDIATE .... Radiation Damage In Accelerator Target Environments

RAFM .... reduced activation ferritic-martensitic

RIS .... radiation induced segregation

RPV .... Reactor Pressure Vessel

RT .... room temperature

RTE .... Rapid Turnaround Experiment

SCC .... stress corrosion cracking

SCK•CEN ....
Studiecentrum voor Kernenergie • Centre d'Etude de l'Energie Nucleaire / Belgian Nuclear Research Centre

SEM .... scanning electron microscopy

SIBL .... Sandia National Laboratories Ion Beam Laboratory

SMARTS .... Spectrometer for Materials Research at Temperature and Stress

SNICS .... Source of Negative Ions by Cesium Sputtering

STIP .... Spallation Target Irradiation Program

SULI .... Summer Undergraduate Laboratory Internship

TEM .... transmission electron microscopy

TMS .... The Minerals, Metals and Materials Society

TREAT .... Transient Reactor Test Facility

TRTR .... Test, Research, and Training Reactors

TTAF .... Test Train Assembly Facility

UCB .... University of California, Berkeley

UCBNE .... University of California, Berkeley Nuclear Engineering

UCSB .... University of California, Santa Barbara

WBDF .... weak-beam dark field

XRD .... x-ray diffraction

XRF .... x-ray fluorescence

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NSUF & Partner Institutions: Facilities & Capabilities

Attendees at the NSUF-GAIN Thermal Hydraulics Workshop, INL Meeting Center, INL (Chris Morgan, INL)

Collaborators

Cherenkov radiation, The Ohio State University Research Reactor (OSURR), The Ohio State University (Courtesy of The Ohio State University)

Researchers working in the *In situ* Ion Irradiation Transmission Electron Microscope (I3TEM) Facility, Sandia Ion Beam Laboratory, Sandia National Laboratories (Courtesy of Sandia National Laboratories)

NSUF Nuclear Science User Facilities

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