ATR
National Scientific User Facility

2013 Annual Report

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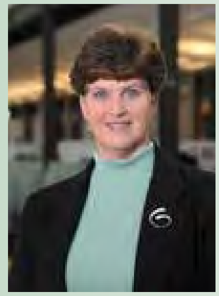

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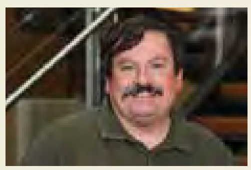

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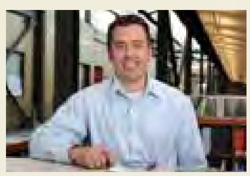

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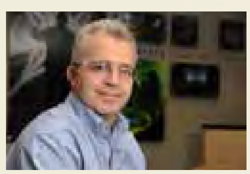

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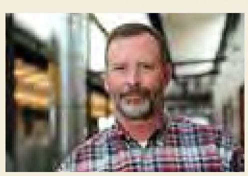

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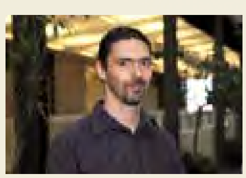

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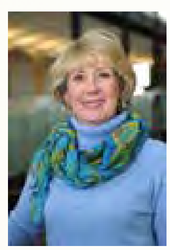

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Advanced Test Reactor National Scientific User Facility

2013 Annual Report

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Frances Marshall, Interim Director
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(This report covers the period beginning October 1, 2012, through September 30, 2013)

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

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Table of Contents

Table of Contents (cont.)

Acronym List

AECL .... Atomic Energy of Canada Limited
AES .... auger electron spectroscopy
AFCI .... Advanced Fuel Cycle Initiative
AFM .... atomic force microscopy
AMS .... accelerator mass spectrometry
ANIAC .... ATR NSUF Industry Advisory Committee
ANL .... Argonne National Laboratory
ANS .... American Nuclear Society
APS. .... Advanced Photon Source
APT .... atom probe tomography
ATLAS .... Argonne Tandem Linac Accelerator System
ATR .... Advanced Test Reactor
ATRC .... Advanced Test Reactor Critical
BWR .... boiling water reactor
CAES .... Center for Advanced Energy Studies
CEA .... Commissariat à l'énergie atomique
CRPs .... copper-rich precipitates
CSL .... coincidence site lattice
CVD .... chemical vapor deposition
DNB .... departure from nucleate boiling
DOE. .... Department of Energy
dpa. .... displacements per atom
DSC .... differential scanning calorimeter
EBR-II .... Experimental Breeder Reactor II
EBSD. .... electron backscatter diffraction
ECAP .... equal channel angular pressing
ED .... electro-deposition
EDS .... energy dispersive X-ray spectroscopy
EELS .... electron energy loss spectroscopy
EFRC .... Energy Frontier Research Center
EFTEM .... energy-filtered transmission electron microscopy
EFPD. .... effective full power days
EML .... Electron Microscopy Laboratory
ENDF .... Evaluated Nuclear Data File
EPRI .... Electric Power Research Institute
EUVR .... extreme ultraviolet reflectometry
EXAFS .... extended X-ray absorption fine structure
FCC .... face-centered cubic
FCRD .... Fuel Cycle Research and Development

FEG ....field-emission gun
FFTF ....Fast Flux Test Facility
FIB.. ....focused ion beam
FIMA.. fission per initial metal atom
FM.. ferritic-martensitic
FSI..fluid-structure interactions
FSRT ....Faculty/Student Research Team
FSW .... friction-stir welding
GTRI .... Global Threat Reduction Initiative
HEDM...high energy diffraction microscopy
HFEF...... ....Hot Fuel Examination Facility
HFIR ....High Flux Isotope Reactor
HIP ....hot isostatic pressing
HRTEM............................ .... high resolution transmission electron microscopy
HT-UPS .... high-temperature ultafine-precipitation-strengthened
HTGR..high temperature gas reactor
IASCC................. .... irradiation-assisted stress corrosion cracking
ICPMS.. inductively coupled plasma mass spectrometry
IFEL....................... ....Irradiated Fuels Examination Laboratory
IIT.......................... ....Illinois Institute of Technology
IMC............................. ....Irradiated Materials Complex
IMET.. ....Irradiated Materials Examination and Testing Facility
IMPACT.......................... ....Interaction of Materials with Particles and Components Testing
INL.. ....Idaho National Laboratory
INLO .... in-situ lift-out
ISU... .... Idaho State University
ITER...International Thermonuclear Experimental Reactor
IVEM .... intermediate voltage electron microscope
LAMDA............................. .... Low Activation Materials Development and Analysis
LANL.. ....Los Alamos National Laboratory
LBE... ....lead-bismuth eutectic
LBP...late blooming phases
LEAP ....local electrode atom probe
LBP ....late blooming phases
LM ....liquid metal
LOCA...loss-of-coolant accident
LWR... ....light water reactor
MaCS ....Microscopy and Characterization Suite
MANTRA ....Measurement of Actinide Neutronic Transmutation Rates with Accelerator Mass Spectrometry

Acronym List (cont.)

MCOE .... Materials Center of Excellence Laboratories
MFC... .... Materials and Fuels Complex
MIBL........ Michigan Ion Beam Laboratory
MIT........ Massachusetts Institute of Technology
MITR........ Massachusetts Institute of Technology Reactor
MOOSE........ Multiphysics Object-Oriented Simulation Environment
MOX.. .... mixed oxide
MPa .... megapascal
MPC........ multi-purpose coupons
MRCAT........ Materials Research Collaborative Access Team
MSTL........ Materials Science and Technology Laboratory
MX .... M=metal, X=metalloid
NCSU........ North Carolina State University
NE........ Nuclear Energy
NERI........ Nuclear Energy Research Initiative
NEUP........ Nuclear Energy University Programs
NFS........ nanostructured ferritic steels
NIST........ National Institute of Standards and Technology
NMMU .... Nelson Mandela Metropolitan University
NRC .... Nuclear Regulatory Commission
NSUF .... National Scientific User Facility
NUFO .... National User Facility Organization
ODS........ oxide dispersion strengthened
ORNL........ Oak Ridge National Laboratory
PECS........ pulsed electric current sintering
PI........ Principal Investigator
PIE........ post-irradiation examination
PNNL........ Pacific Northwest National Laboratory
PWR .... pressurized water reactor
RAFM........ reduced activation ferritic-martensitic
REDC .... Radiochemical Engineering Development Center
RERTR .... Reduced Enrichment Research and Test Reactors
RIA .... reactivity insertion accident
RID .... radiation-induced depletion
RIS........ radiation-induced segregation
RPL........ Radiochemistry Processing Laboratory
RPV. .... reactor pressure vessel
RTE .... rapid turnaround experiment

SANS ... small angle neutron scattering
SCC ... stress corrosion cracking
SDTM ... scanning differential thermal microscope
SEM ... scanning electron microscopy
SHaRE ... Shared Research Equipment Collaborative Research Center
SIMS ... secondary ion mass spectroscopy
SPS ... spark plasma sintering
SPT ... shear punch test
SRIM ... Stopping and Range of Ions in Matter
SRNL ... Savannah River National Laboratory
SS ... stainless steels
STEM ... scanning transmission electron microscopy
TAMU ... Texas A&M University
TEM ... transmission electron microscopy
TIMS ... thermal ionization mass spectrometer
TMS ... The Minerals, Metals, & Materials Society
TMT ... thermo-mechanical treatment
TRIGA ... Training Research Isotope General Atomics
TRIM ... transport of ions in matter
TRISO ... tristructural isotropic
TTS ... transition temperature shift
UCB ... University of California, Berkeley
UCSB ... University of California, Santa Barbara
UF ... University of Florida
UFG ... ultra-fine grained
UI ... University of Idaho or Illinois
UM ... University of Michigan
UNLV ... University of Nevada, Las Vegas
USU ... Utah State University
UTS ... ultimate tensile stress
UW ... University of Wisconsin
V&V ... validation and verification
VHTR ... very-high temperature reactor
XANES ... X-ray absorption near-edge spectroscopy
XAS ... X-ray absorption spectroscopy
XPS ... X-ray photoelectron spectroscopy
XRD ... X-ray diffraction
µm ... micrometre

Welcome & Introduction

Frances Marshall
Interim ATR NSUF Director
and ATR NSUF Program Manager

It is a pleasure to introduce the Advanced Test Reactor National Scientific User Facility (ATR NSUF) Annual Report, as the interim director for 2013. As most of you know, Dr. Todd Allen resigned from his position as scientific director of ATR NSUF in December 2012 to become the deputy laboratory director of Science and Technology for Idaho National Laboratory (INL). In this new position, Todd has continued to provide oversight to ATR NSUF activities and helped guide the program forward from his initial vision into a more substantial program for collaborative research, using our network of users and facilities.

In addition to facilitating experiment access to a wide variety of research capabilities for our user community, 2013 was a year of several "firsts" for ATR NSUF.

We accepted a proposal by Westinghouse Electric Company to become one of our partner facilities, making it the first industrial organization to join the ranks of ATR NSUF partners. The addition of Westinghouse, a leading supplier of nuclear plant products and technologies, provides ATR NSUF researchers access to an even wider variety of capabilities for nuclear materials and fuels research. As an ATR NSUF partner, Westinghouse is offering its Materials Center of Excellence Laboratories (MCOE) Hot Cell Facility and accompanying laboratories to provide experimental support to ATR-related nuclear energy materials research programs. The Westinghouse facilities are housed in four cells that provide a broad range of testing, evaluation and characterization capabilities for both unirradiated and irradiated materials. This partnership will enable our university researchers to more easily collaborate with commercial nuclear power organizations. The addition of Westinghouse makes a total of 11 ATR NSUF partner facilities: eight university partners, two national laboratory partners and now one industry partner.

And speaking of partner facilities and their impact, in 2013 we awarded 19 new research projects, many of them rapid turnaround experiments (RTE) that were performed at a variety of INL and partner facilities. As the ATR NSUF program continues to align with other DOE-NE programs, our partner facilities will increase in significance. RTEs lend themselves well to partner facilities, and in fact, already in 2013 we have seen the number of RTE proposals double what we received last year. This is due in large part to the diversity of capabilities partners bring to ATR NSUF, and I would like to take this opportunity to thank our partners for opening their doors to an experiment in collaborative research, the likes of which has not been seen before.

In 2013, a newly available ATR capability – a restarted pressurized water irradiation loop – was used to support a nuclear power industry experiment for the first time. The Electric Power Research Institute (EPRI) is collaborating with ATR NSUF on a pilot project focused on post-irradiation examination of reactor structural materials to determine how metal alloys behave under irradiation in reactor environments. Specifically, the project's objective is to explore the growth behavior of irradiation-assisted stress corrosion cracks (IASCC) in two particular alloys, X-750 (a nickel-based alloy) and XM-19 (a nitrogen-strengthened austenitic stainless steel) within a typical light water reactor environment. In addition to the loop in ATR, two new test cells were assembled at the Materials and Fuels Complex (MFC) for testing crack growth on irradiation specimens. Irradiation of the test specimens was initiated in 2013 and testing of the irradiated specimens in the IASCC cells will begin in 2014. This work is significant because it is a very high priority for the commercial nuclear power industry, and until now, this capability has not been not available to industry or university researchers in the United States.

In June, we hosted the ATR NSUF's sixth annual Users Week, and with the addition of more than 60 researchers, we exceeded 500 users who have attended the ATR NSUF Users Week since we hosted the first one in 2008. The agenda for the week included many presentations about the partner facility capabilities and time for discussions

The newly constructed irradiation-assisted stress corrosion crack (IASCC) test rig at INL's Materials and Fuels Complex.

with partner facility representatives. Similarly, there were several presentations by ATR NSUF researchers, and many opportunities for new users to discuss research projects with more seasoned experimenters.

After a hiatus of a year, the ATR NSUF awarded two faculty-student research team projects in 2013, both to Wade Marcum of Oregon State University, but focused on different technical areas and objectives. For one project, Marcum and his students used a new modeling and simulation platform to model the Transient Reactor Test (TREAT) facility, which is being restarted to simulate nuclear reactor accidents and the impact on fuels. The other project, also involving modeling and simulation, was to develop a fluid-structure interaction (FSI) validation and verification (V&V) methodology for multiphysics models that are being developed. Much progress was made to understand the current V&V protocols and evaluate the current codes being developed; however, there is more work to be performed.

A key foundation of the ATR NSUF is the ability to offer our user community the most current experimental capabilities and tools. Toward that end, the ATR NSUF program sponsors some research to develop new testing techniques, such as instrumentation, to help researchers better understand the effects during the irradiation portion of the experiment, rather than waiting until the test specimens are unloaded into PIE hot cells. Dr. Joy Rempe is leading a team of INL researchers to develop these new instruments. In 2013, an experiment was awarded to Pennsylvania State University to collaborate with INL on development of ultrasonic transducers that can be inserted into the reactor with test specimens. Additional work is ongoing to develop new temperature and flux detection methods, and new detectors will be tested in reactors in 2014. It is expected that these new instrumentation techniques will provide valuable capabilities to the ATR NSUF user community for future experiments.

As 2013 has transitioned into 2014, there are some changes to the staffing of the ATR NSUF. Dr. Rory Kennedy, previously a manager in INL's Nuclear Fuels and Materials Division, accepted the position of ATR NSUF scientific director in January 2014, and will fill the role as a full-time scientific director. Rory has a long history and understanding of material properties, including fuel fabrication and characterization, reactor-based testing and post-irradiation examination, all of which lend itself well to the scientific director position.

Mary Catherine Thelen, our first ATR NSUF program administrator who had been with the program since its inception, retired from INL in February 2014. Her duties were split between Jeff Benson and Julie Ulrich, with Jeff serving as the lead point of contact for the proposal solicitations. In April 2014, Julie Ulrich, our communications specialist, accepted a new position at INL with the Center for Advanced Energy Studies; her duties will transition to Sarah Robertson, who formerly supported internal communications at INL.

Lastly, in May 2014, I will retire from INL. I had the opportunity to work on the ATR NSUF for the last seven and a half years, starting with development of the original proposal for establishment of the program, participating in the transition to start up the program, and following the program to the mature, but ever-changing, state that exists today. My duties will be split between Rory Kennedy and Dan Ogden, who has been with the program for five years.

In closing, it has been my pleasure serving as interim director for ATR NSUF. I have had the privilege to lead a wonderful team of INL employees who work tirelessly to support the ATR NSUF user community, seek innovative ways of facilitating collaborative research projects, and continue to implement the vision set by our first scientific director, Dr. Todd Allen. Our user community has matured and continues to partner with us to seek the best capabilities to support their research missions. The next ATR NSUF scientific director will find a creative user community, demanding researchers, and a staff willing to test new ideas as we continue to expand the ATR NSUF into a national complex serving ever-increasing nuclear energy research needs.

Sincerely,

Research Perspectives:

A Peek Inside the Imagination and Innovation of ATR NSUF Scientists

The 38 individual scientific projects you'll read about in this annual report all have one thing in common. They all aim to make nuclear energy a better, safer, more effective, more cost-efficient source of electricity for the United States and the world. This article discusses three of those projects from the perspectives of some of the scientists most closely involved with them. Together, they provide a glimpse into the processes, innovations, and value of the various sciences used to advance nuclear energy.

Tom Maddock

Experiment Manager

Advanced Test Reactor National Scientific User Facility

In a conference room at the Center for Advanced Energy Studies (CAES) in Idaho Falls, Idaho, Tom Maddock considers the question, "What is the value and benefit to the American taxpayer of what we're doing here?"

As an experiment manager for ATR NSUF, he coordinates experiments and the scientists who work on them from the initial idea to final post-irradiation examination (PIE), so he understands more clearly than most people what researchers are trying to accomplish at ATR NSUF and the challenges they face. Today, he's working on a project that focuses on extending the life of the reactors we have online now. "We're looking at the long-term degradation of the materials in our current fleet of nuclear reactors," he says, "many of which are approaching 40 years of service. The question the principal investigator is trying to answer is, 'Can these reactors make it to 60 years of service? Can they make it to 80 years?'"

With 20 percent of America's electricity generated by nuclear energy and few new plants being built, the country has a huge stake in answering that question. If this experiment demonstrates that the current U.S. fleet of reactors can provide the electricity a growing population (now approaching 325,000,000) needs, then we've bought ourselves some time developing a new generation of nuclear plants. If they cannot, then the possibility exists that we must either find alternative sources for our power fairly quickly or start taking faster, colder showers.

Maddock's first ATR NSUF project originated with Dr. R. G. Odette of the University of California, Santa Barbara, who used the ATR NSUF to irradiate and perform PIE on approximately 1,400 samples of 49 individual materials under several different, very specific, carefully monitored conditions. For Odette and his team, a project of this scope presented a unique opportunity to identify, understand and model materials used in nuclear energy systems in order to build a comprehensive database for understanding radiation damage in structural alloys and ultimately aid in predicting and improving the behavior of these materials in radiation environments.

It also demonstrated the innovative, literally "out-of-the-reactor" thinking ATR NSUF staff must sometimes do. "Due to some unplanned, unscheduled reactor outages," Maddock explains, "we realized we'd have to take the experiment out of the reactor at some point and put it back in later to complete the experiment." Of course, this was not in the original plan.

Removing the experiment was not a simple matter. It had to be done without damaging the seventeen-foot, bow-shaped tube that houses the samples, so it could be reinserted back into the reactor.

"It takes two days to get the tube out of the reactor," Maddock continues. "Twelve people on top of the reactor with ropes and lifting tools are pulling, twisting, rotating until we get the tail end of the tube out, and that's where the innovative thinking comes to play."

In order to get the experiment all the way out of the reactor, it was necessary to grasp the bottom end of the tube to keep it from rolling over and coming out of the reactor the wrong way. However, applying too much pressure can crimp or crush the end of the tube, complicating if not eliminating any chance of returning the experiment to the reactor.

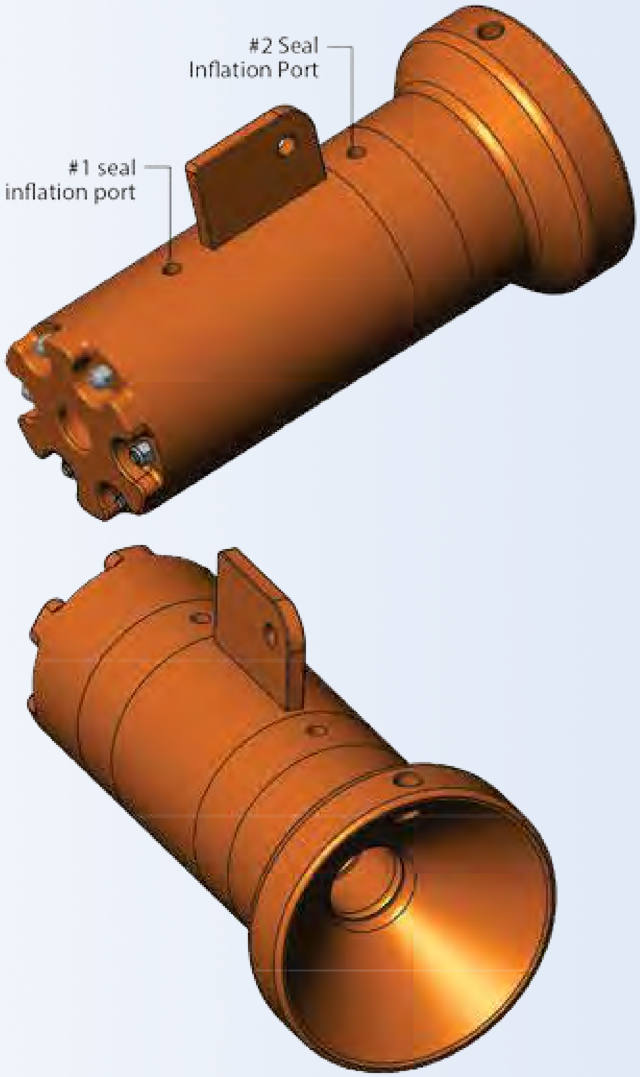
"So the experiment designer came up with an idea," Maddock says, "to slip inflatable rings over the bottom of the tube. When we inflated the rings, they'd wrap around the tube, and we could adjust the air pressure in the rings so they would grab the tube, but not crush it."

After an intense search, the team found a company in Arizona with the expertise to build the tool to its specifications.

Positioning the rings on the container was complicated by the fact that they had to be applied while the tube was 25 feet underwater. "It's tricky," Maddock adds. "You've got a huge pole and a small-diameter air hose going down through two-and-a-half stories of liquid and equipment, but once we got the rings on the capsule and inflated, we could use the tool to maneuver the experiment up safely past all the obstacles in the canal."

Did this innovative problem-solving meet the challenge? In his project summary in this annual report (page 99), Odette states, "Even after testing a handful of specimens, the ATR-1 samples produced some seminal and enormously high-impact results."

Of course, this sort of resourcefulness is not an unusual occurrence at ATR NSUF. "With every project," Maddock notes, "it seems like you're starting over and doing new things that haven't been done before. So we're getting used to coming up with new ways of doing things."



A mockup of the innovative tool researchers used to extract the capsule from the reactor. Drawing courtesy of Mechanical Research & Design, Inc.

Research Perspectives:

A Peek Inside the Imagination and Innovation of ATR NSUF Scientists (cont.)

Dr. Michele Manuel

University of Florida

> **"It could be a problem most people think can't be solved because it's so complicated. And maybe it can't be, but having all this brainpower working together gives us a lot better chance of success."**
>
> Dr. Michele Manuel, University of Florida

While many of the projects submitted to ATR NSUF investigate materials and fuels that are considered candidates for future nuclear reactor applications, Dr. Michele Manuel of the University of Florida is concerned, like Odette, with the present. "We're trying to understand nuclear fuels," she explains, "specifically uranium dioxide (UO_2), which is the most common fuel used in today's commercial reactors, and what happens to it as it's subjected to irradiation in a reactor during actual service."

As one might expect, the UO_2 that comes out of a reactor is much different than the material that goes in. "There are a lot of processes going on inside the reactor," Manuel points out, "and we're trying to figure out what's causing what and how those interactions are related to each other."

Making our current fuel better

The ultimate goal of her team's work is to gain a better understanding of how UO_2 behaves. "We're trying to find the linkages between how the material performs, what the microstructure looks like, and inevitably what its properties are," she said. Once they have a firm grasp on its quirks and eccentricities, they can engineer its microstructure to get the best performance possible out of the material.

Much of that work is being conducted in CAES, using atom probe tomography (APT). "The atom probe is really a unique instrument," Manuel said. "It allows us to see where the atoms are located inside the sample we're studying, so we can compare their locations to a sample that's been irradiated or heated. So what we're getting is a snapshot of what the material looks like at the atomic level."

Invented by Erwin Müller, J.A. Panitz, and S. Brooks McLane in 1967, the atom probe removes ions from a sample's surface with magnifications high enough that it can "see" individual atoms. As each successive surface layer of a material is removed, the instrument collects the data and builds a three-dimensional image of the sample, thus enabling scientists to study the atomic structure of the material in great detail.

A real team effort

One of the challenges Manuel has faced in her work is the lack of information on the study of oxides using the atom probe.

"There's a lot of established literature on the study of metals using the probe," she says, "but not on the study of oxides." Historically, acceptable results using atom probe tomography were only possible with materials that easily conducted electricity. However, recent improvements in the technology have expanded its capabilities, and now it can be used to examine less conductive materials like silicon and oxides. "We're using a relatively new instrument to look at a material that hasn't been looked at in this way before," she says, "and one of our challenges is determining how much the instrument influences our data and how much is real science."

One of the people helping Manuel and her students figure that out is Yaqiao Wu, a senior scientist at CAES, who personifies one of the major benefits of ATR NSUF's mission: providing students in the nuclear and materials sciences access to real-world research and investigative experiences.

"When we send students up to CAES to work on the experiment, they've already been trained to run the atom probe," Manuel says, "but Dr. Wu is there as the instrument scientist. He's in charge of getting the materials in and out of the atom probe and aligning the samples inside. And depending on a student's expertise with the instrument, he can act as an on-site mentor or guide."

Wu and the other ATR NSUF staff contribute much to the success of the project and the students' progress toward their degrees. However, they represent only one example of how the ATR NSUF partner program fosters an atmosphere of cooperation. "The thing about ATR NSUF," Manuel says, "is you have all these great partner institutions from across the country, so you have national lab scientists and staff working on projects together as a team. Each of them has their own role and their own area of expertise, and they bring all these extraordinary skills and knowledge together to solve this really hard problem. It could be a problem most people think can't be solved because it's so complicated. And maybe it can't be, but having all this brainpower focused on this one project gives us a lot better chance of success."

"For example, a lot of times, we'll be working on something and another scientist notices what we're doing and says, 'Hey, I've got an idea. Can you try this, so I can take a look at it with my instrument?' And they'll have a suggestion for a variation on the experiment. And we'll do it, and they'll look at it, and maybe we'll learn something we didn't suspect before. It might drive the project in a new direction or push it the same direction, only faster. It's a real team effort."

As we'll see in the final "chapter" of this article, the future—and the promise of new materials and technologies it holds—can be very exciting. But as Michele Manuel and Tom Maddock both demonstrate, pushing to improve the present holds the same potential for important advancements; ones that could bring the future much closer.

The local electrode atom probe (LEAP) in the Microscopy and Characterization Suite in CAES.

Dave Senor
Staff Engineer
Pacific Northwest
National Laboratory

Andy Casella
Experiment Manager
Pacific Northwest
National Laboratory

In the early part of the 21st Century, scientists at the University of California, Berkeley (UCB) were conducting laboratory research on hydride fuel as a possible power source for future light water reactors. The researchers recognized that this new material could be safer and more efficient than mixed oxide fuels that were—and still are—the predominant fuel in nuclear reactors.

"The original concept for this fuel came from Don Olander," says Dave Senor, the principal investigator of the PIE phase of the project at Pacific Northwest National Laboratory (PNNL) in Richland, Washington. "For a time, experiments on the new fuel were limited to the laboratory, but Olander and his team recognized that if they were going to push the project to the next stage, the work had to be shifted to, as they called it, 'more relevant environments'."

In 2009, Olander, Mehdi Balooch, and others at UCB submitted the project proposal to ATR NSUF, which awarded their proposal and funded the project. Shortly thereafter, samples of uranium-zirconium hydride arrived at the Massachusetts Institute of Technology Reactor (MITR), a 6-megawatt light water research reactor in Cambridge, Massachusetts. There, the samples were irradiated and then shipped back across the country to PNNL.

A challenging assignment

"Our role on the project," Senor says, "is to disassemble the MIT capsule the materials were irradiated in and conduct various characterizations and analyses on the fuel to see how it performs in a more robust reactor like MITR."

Because the samples are highly radioactive, all the work has to be done in hot cell or shielded facilities. Mechanical manipulators take the place of human hands, so the PNNL scientists must be very careful about cutting open the capsule and extracting the components without damaging the samples or introducing outside artifacts.

The project faced other challenges, as well. "Because this fuel is a hydride fuel, Mehdi was concerned about the propensity of the material to react with air during sectioning," explains Andy Casella, the PNNL experiment manager, "which would change the hydride concentration and adversely affect the data."

"In order to address the concern of hydrogen migration during irradiation, we had to break the fuel into smaller pieces to determine the percentage of hydrogen in the fuel

The hot cell at the Pacific Northwest National Laboratory.

as a function of its location.” That’s easier said than done, since many of the techniques normally used to determine the concentration of elements within a material don’t work well with hydrogen. “So we had to very carefully slice off a piece of the sample,” Casella continues, “but we have to do it in an inert gas environment to preserve the surface condition of the fuel.”

“After that, we have to determine the sample’s mass, heat it up to drive off the hydrogen, and then put the gas through a mass spectrometer to determine how much of it is hydrogen and how much is fission gas.”

As you might expect, no one instrument can perform this entire process. So a number of instruments were recruited to take a new kind of measurement on this new kind of fuel.

“That might be one thing PNNL offers that other facilities do not,” Senor adds. “We’re set up with varying degrees of shielding, different sized hot cells, and different glove boxes. So we have the facilities and expertise to get the measurements Mehdi and his team need.”

When the PIE is finished, and in a bow to the teamwork to which Michele Manuel alluded, PNNL will send the capsule, the samples and a report containing the collected data to both ATR NSUF and the scientists at UCB. “There are some characterization capabilities the Berkeley folks are interested in that we don’t have,” Casella notes. “So shipping the material to the ATR NSUF sample library means they’ll be available for further examination, and the data gives future researchers a starting point for their work.”

The Pacific Northwest National Laboratory near Richland, Washington.

Anything we can do to improve the efficiency and safety of the energy sources we have available, the better things are going to be for everyone.

Higher efficiency. Greater safety. Is it enough?

Does uranium-zirconium hydride hold enough promise for future researchers to continue Olander and Balooch’s efforts? Senor thinks so. “Since the accident at Fukushima in 2011,” he says, “reactor fuels that can withstand loss-of-coolant accidents are getting more attention. And this fuel operates at a much lower temperature than oxide fuels. It doesn’t store as much heat after a reactor shut down either, so the hope is it’s much more forgiving in an accident situation.”

The fuel is also much more efficient. “The reactor can extract more energy from it and achieve higher burnup,” Senor adds, “so you get more energy and less waste from the same amount of fuel.” Obviously, Senor sees great potential in uranium-zirconium hydride. But is it enough to replace the uranium-dioxide fuel Michele Manuel is investigating?

On the surface, it might appear that the two scientists in opposite corners of the country are working at cross-purposes. But, as Senor points out, they, along with Tom Maddock and the other scientists, engineers, and staff involved in America’s nuclear energy research, are all striving to achieve the same goal.

“Energy is fundamental to everything we do in the world,” he points out. “Anything we can do to improve the efficiency and safety of the energy sources we have available, from nuclear to hydroelectric, even solar and wind power, the better things are going to be for everyone. And that’s exactly what we’re trying to do. Not only with this experiment, but with every project in every nuclear research facility in the country.”

User Friendly:

Fostering Collaboration and Innovation in Nuclear Research and Development

In 2007, the U.S. Department of Energy (DOE) added the Advanced Test Reactor (ATR) National Scientific User Facility (NSUF) at Idaho National Laboratory (INL) to its list of some 40 national user facilities across the country, making it the nation's first and only designated DOE nuclear user facility. In doing so, DOE brought the world's most advanced test reactor into play in the race to catch up a lost generation of nuclear scientists in the United States.

For Dr. Kemal Pasamehmetoglu, that race began more than 30 years ago. When he came to the U.S. from Turkey in 1980 to attend graduate school at the University of Central Florida, the DOE budget for nuclear R&D, which had been declining in this country for a number of years, had dwindled to zero. As a result, very few people were looking at careers in nuclear science, and many universities were closing down their nuclear energy departments. A few years later, armed with a Ph.D. in mechanical engineering, the young scientist went to work at Los Alamos National Laboratory in New Mexico. In 1989, the federal government made a push to

"What I'm trying to do in the classroom really can't be taught effectively unless my students come to Users Week and interact with other people. I always say, 'See, you're not competing with your classmates, you're competing with the world.'"

Dr. Yongho Sohn, professor,
University of Central Florida

Researcher Curtis Clark shows Users Week attendees one of the many testing capabilities at INL's site facilities.

re-introduce the fuel cycle into this country's nuclear energy research portfolio, and Pasamehmetoglu was tasked with assembling a team to start developing potential reactor fuels.

"I reached out to the most brilliant materials scientists in the country," Pasamehmetoglu said, "and I was only able to find about six people who had ever seen nuclear fuel in their lives."

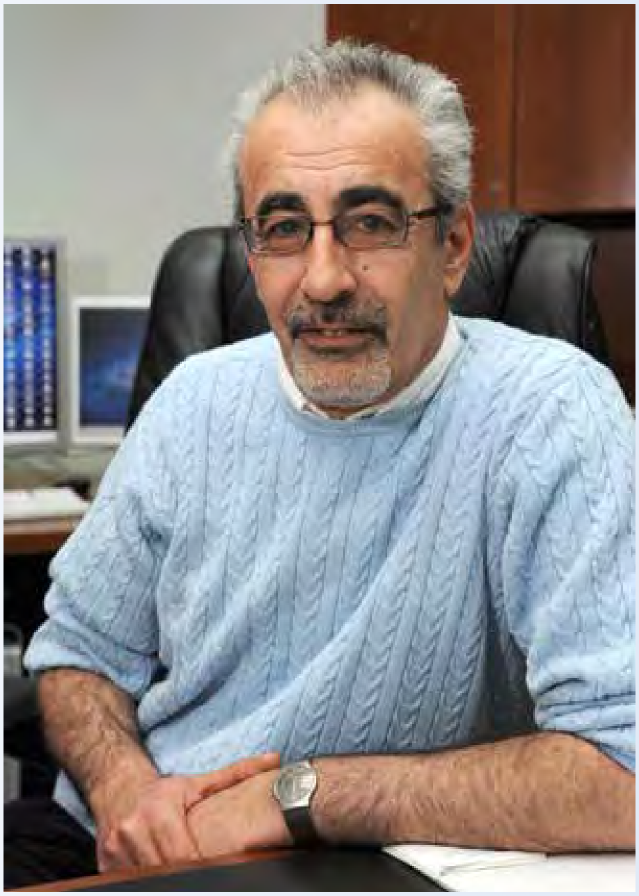
The nuclear research hiatus in the U.S. had taken its toll. But efforts by Pasamehmetoglu and others in the field over the past 25 years have paid off, and today INL's Nuclear Science and Technology Directorate, which he heads, is thriving, with more than 120 scientists working solely on nuclear fuel research.

"In the United States, nuclear energy is a commercial entity," said Pasamehmetoglu, "and it is industry's job to solve today's problems. Our job is to look into the future and establish the research and development base to enhance nuclear energy going forward. And one area that always filters to the top in that is fuels and materials. They are the heart of any nuclear energy system we deploy, but they also take the longest to develop."

Indeed, the race is far from over. The lost generation of nuclear scientists may be well on its way to being restored, but new challenges loom on the horizon, and a new generation of nuclear scientists must be ready to tackle them. With some 20 percent of the electricity in the U.S. generated by nuclear reactors, the nuclear industry in this country is approaching a critical juncture. The current fleet of reactors is showing its age. Many of them are approaching the end of their licensing periods. The development of new nuclear fuels and materials may allow them to continue producing nuclear energy safely and efficiently for many years to come, but time is of the essence.

"There are innovative concepts that can improve on existing technologies," Pasamehmetoglu said, "but many of them take five or six years to develop. The end result is, if you start working on a concept for a brand new fuel today, it might be 25 years before it can be put into commercial use. That is why ATR NSUF puts such an emphasis on programs like Users Week. It's an important part of finding and developing the talent and the facilities to help move that research along faster."

ATR NSUF takes its role as a user facility very seriously. So much so that in 2009, the facility's then scientific



Dr. Kemal Pasamehmetoglu, director of INL's Nuclear Science and Technology Directorate.

director, Dr. Todd Allen, spearheaded an effort to grow the user facility beyond its own physical boundaries by inviting other research facilities to form a distributed partnership program. Thus far eight universities, two other national laboratories and, in 2013, the first private industry partner, Westinghouse, have come on board in an unprecedented effort to share capabilities through an innovative proposal program coordinated by ATR NSUF. The result is a broad base of nuclear research capabilities that make it unique in the DOE's family of user facilities.

This collaborative network is overseen by the ATR National Scientific User Facility Users Organization, which provides direction on capability enhancements, research areas of interest, and calls for proposals. Early on, it proposed an annual meeting at which users and researchers could discuss ongoing research as well as present new ideas. That task fell to ATR NSUF's then education coordinator, Jeff Benson.

User Friendly:

Fostering Collaboration and Innovation in Nuclear Research and Development (cont.)

Jeff Benson leads Users Week attendees on a tour of INL's Materials and Fuels Complex.

"Users Week is not a new idea," said Benson, whose job has recently been expanded to program administrator. "Most of the user facilities within the DOE complex have similar meetings, but we've got 11 partners involved, so we needed an entire week to introduce all our capabilities and cover all the research topics that were interesting to our users. It's a great opportunity to bring all the partners together and introduce them to the users, as well as extend an open invitation to any other scientists and students who want to attend. The intent is to provide an environment where they can talk about the issues that are out there in the materials science world. What specific questions are nuclear scientists discussing? What kinds of experiments are being proposed? Where is the research leading?"

The first one, in 2008, was simply an introduction to ATR NSUF and what it does. That inaugural program focused primarily on materials issues, so in 2009, it delved into fuels issues. The following year, materials took center stage again. Then, in 2011, ATR NSUF combined its annual meeting with that of the Energy Frontier Research Center (EFRC).

"Because both conferences were featuring many of the same speakers," Benson said, "the joint event became a synergistic meeting of the minds that was hugely successful."

Riding that momentum into the fifth year, Benson joined forces with another group. The Modeling Experimentation Validation (MeV) School was holding its annual summer program at Oak Ridge National Laboratory (ORNL), whose High Flux Isotope Reactor (HFIR) had become an ATR NSUF partner facility in 2011.

"Given our success the previous year," Benson said, "bringing these two events together seemed like another unique opportunity for Users Week participants and a natural next step in the evolution of the conference. It was a very intense, two-week school. We limited participation to 50 people who spent a week at Oak Ridge and then a week at INL. We brought in a world class lineup of nuclear scientists to talk about everything from fuel design and fabrication to fuels performance modeling and post-irradiation examination."

“I’ve attended Users Week for three years now. Our project started in 2008. The first part of it was done by a colleague who is now at Argonne National Laboratory, and I’m carrying on with the second part of it. We’ve gotten many of our ideas from this conference and learned a lot from the people here. I encourage everybody to attend.”

Ahmad Alsabbagh, Ph.D. candidate,
North Carolina State University

User Week attendees network at the poster session held in the CAES gallery.

The annual event continued to evolve in 2013, as research projects developed over the previous five years began to produce results. For the first time, scientists could hold in-depth discussions and present quantifiable data and analyses on research actually being conducted at ATR NSUF and its partner facilities. Users Week had come of age, and those in attendance heartily approved.

“There are a number of goals we try to accomplish with each of these conferences,” said Benson. “In addition to exploring current issues in fuels and materials, we also discuss the various tools and instruments at ATR NSUF and throughout our partnership network, and how these capabilities can help scientists accomplish their work in the most effective and efficient way. And the community environment allows people to figure out ways to solve problems together.”

The educational aspect of Users Week is arguably one of its most important attributes. Universities used to do a lot of nuclear-related testing, but because many reduced or eliminated nuclear programs during the research hiatus of the ’70s and ’80s, many scientists today, and especially students, have little or no experience in conducting a nuclear-related experiment.

“I like to come to Users Week to share some of my experiences and interact with the students; to help them become more familiar with some of the fuels and materials issues so they can write good proposals.”

Brandon Miller, INL research scientist

“I’m not calling it a dying art, necessarily,” Benson said, “but there are very few specialized places and not very many people who know how to do that. You don’t just decide to do a test and throw it in the reactor for a few days. There’s a process, from how to determine if your idea is viable to how to write a good proposal, and what to do once your proposal is accepted. One of the big things we do at Users Week is explain that process, and help researchers move ideas forward in the most productive way.”

“The preparation alone is a big part of doing the experiment,” said Pasamehmetoglu. “That’s one of the reasons it takes 25 years to get a project from concept to commercialization. Each individual experiment could take five years to complete. In the non-nuclear world, scientists can often go into the laboratory and do an experiment in one day. If it fails, you go back the next day and do another experiment. For us, one day is five years, and if it fails you have to start all over again.”

In addition to hearing presentations by world-renowned scientists and learning about the proposal process, all attendees are strongly encouraged to design a poster showcasing their work. Posters are judged on their technological approach, content, the novelty of the idea, and its potential impact on the scientific community and the world. Prizes are awarded to the top three winners.

“The poster session is where the collaboration part of the event really starts to coalesce,” said Benson. “In a research community, it’s always good for people to mingle with each other in a social situation and learn what kinds of work are being done. Everyone can look at the posters and see very quickly what someone else is doing and suggest

Peter Wells the student representative to the Users Organization, gives an update on the UCSB experiment that is compiling an unprecedented database of materials with the potential to extend the life of operating reactors.

an idea of their own. Or they might figure out that what they're doing relates to what someone else is doing, and they may be able to combine research and help each other out."

In 2014, Users Week is evolving once again, from a workshop and informational gathering to more of a research forum. The event will be shortened from one week to three days. Even the name is changing, to Users Meeting.

"It's a maturity thing," Benson said with pride. "The first few years we didn't have many experiments going on, but now we have lots of them, so we're pushing it more toward the technical discussion arena. We'll still do the poster session, but we won't offer that overview of the materials and fuels issues that are out there. Instead, our speakers will be discussing their actual, ongoing ATR NSUF experiments."

One successful carryover from the 2013 conference will be the inclusion of the annual meeting of the Users Organization. In a one-hour open session, attendees will be invited to discuss issues or problems they are working on and those who are not members will have a chance to join. Fostering collaboration and innovation will remain an integral part of the Users Meeting, and the discussion of

"Working with more experienced scientists in the community and being able to hear about some of the challenges that are faced by organizations within the nuclear industry has been very beneficial to me in looking at my career path."

Peter Wells, graduate student,
University of California, Santa Barbara

ongoing research will continue to plant the seeds of new ideas in the minds of attendees so they can start thinking about those problems from new perspectives.

“The biggest problem when it comes to nuclear research,” said Pasamehmetoglu, “is the 25 years thing. It’s a real turnoff. Private industry will not invest in a 25-year venture. Their horizon is 10 or 12 years. It’s the reason many people complain that the nuclear industry is not innovative enough. It’s not because we don’t have innovative ideas, there are plenty of those, but when you know that if you start a brand new idea just when you come out of graduate school, and by the end of your career you may, or may not, see it commercialized, it doesn’t excite a lot of people. My whole objective in that area is to get the time frame cut in half, so the whole process can be completed in 12 years. When I see ideas addressing that coming out of all these user facility proposals, many of which originate from Users Week, I get really excited.”

Technological progress is the keystone of Pasamehmetoglu’s belief that 10 years from today nuclear technology will be developed very differently. Modeling and simulation techniques are advancing at a rapid pace and will create a much stronger tie between user facility capabilities and the computational side of research.

“If we couple those two things properly,” Pasamehmetoglu said, “we can accelerate some of those trial and error processes. We have the computational ability to do that, what we don’t have is the experimental capability to validate each step in that process. So the next frontier for the user facility is to start focusing on providing validation data for the models people are generating now, that were impossible five or six years ago.”

The Users Meeting plays a critical role in these exciting new processes by bringing scientists and students together to discuss, dream, exchange ideas and collaborate.

“The key is not just the people who attend,” said Pasamehmetoglu, “it’s what happens to them after they attend. Some of them came to their first conference with little or no idea what nuclear fuel is, and now they are writing good, solid proposals on nuclear fuel experiments that need to be done.”

“What I learned at Users Week really helped me connect with what I’ve been hearing back at UCF. I understand what I’m hearing in the classroom a lot better now.”

Kim Westerlund, undergraduate, University of Central Florida

Over the past six years, Users Week has had more than 560 participants. Out of the 164 proposals received by ATR NSUF during that time, 90 of them came from Users Week attendees, and 36 of those were awarded, with research currently underway or completed. This is solid proof that the Users Meeting plays a huge role in the workforce development at ATR NSUF, and in catching up the lost generation of nuclear energy researchers who will help create a brighter future for the energy industry and for the world.

ATR NSUF Industry Program Forges New Path Forward

What does the nuclear industry want? The ATR NSUF Industry Program knows because it works so closely with nuclear power companies. To help ensure those needs are met, the Industry Program is spearheading efforts that:

- Help industry to generate power as efficiently as possible, as safely as possible and as inexpensively as possible.
- Forge closer relationships between nuclear power companies and university researchers.
- Help to usher in a new generation of young nuclear engineers and scientists.

Good relationships between industry and academic researchers are vital to advancing nuclear energy, according to Dr. John Jackson, ATR NSUF Industry Program lead. The program is encouraging those relationships as a means to generate power more efficiently and safely, and to build the next generation of nuclear engineers and scientists.

The formation of industry-academic relationships is an especially important part of the Industry Program's many responsibilities, because when the nuclear industry and university nuclear researchers work together, both sides win, according to Dr. John Jackson, ATR NSUF Industry Program lead.

Joint projects help nuclear energy companies to make important upgrades to the fleet of reactors that produce the power of today and tomorrow. For university researchers, partnerships with industry provide relevance for their projects while energizing their students about the contributions they can make in the real world. "Everybody comes out ahead," Jackson said. "And that includes the energy-consuming public who ultimately reap the rewards through low-cost and safe power."

Industry, EPRI and the New IASCC Test Rig

"The advent of the Industry Program is coincident with the advent of ATR NSUF as a whole," Jackson said, noting that since its inception in 2007, ATR NSUF has been offering access to cutting-edge nuclear materials and fuels research capabilities at Idaho National Laboratory as well as at a growing number of partner facilities (now totaling 11). "At the beginning, we reached out to industry and asked for their input to see what sorts of facilities they would like to have access to and which issues were most pressing to them, and that helped to build the overall direction for ATR NSUF," Jackson said.

One of the major players in the success of the Industry Program is the Electric Power Research Institute (EPRI), a nonprofit organization that conducts research and development relating to the generation, delivery and use of electricity. EPRI's members represent approximately 90 percent of the electricity generated and delivered in the United States.

"We carry out research programs on a variety of issues related to nuclear power," said Raj Pathania, EPRI technical executive. "Our research results are used by the nuclear power industry to improve the safety, reliability and efficiency of their plants. Our results also are used by other stakeholders, including regulators and engineering code groups, to inform the development and implementation of technically sound standards and regulations. These activities not only benefit the power industry, but ultimately the public as well."

One of the first industry suggestions to ATR NSUF, in fact, came from EPRI, Jackson said. "Initially when ATR NSUF was just starting out, we wanted to build confidence and to advertise our capabilities, so we invited relevant stakeholders to submit suggestions for projects. EPRI responded," he said. EPRI's proposed project would test how a certain material, a so-called superalloy called nickel-base Alloy X-750, performed in a reactor. Specifically, EPRI was interested in the growth of cracks in the material when exposed to the high-radiation environment of a reactor. The growth of cracks in materials subjected to stresses and environment is

Westinghouse became ATR NSUF's first commercial partner in 2013, adding its Materials Center of Excellence Laboratories (MCOE) Remote Testing and Metallographic Facility and accompanying laboratories to the wide variety of ATR NSUF nuclear materials and fuels research capabilities. Known as a hot cell, the facility (pictured here) allows the safe, remote-control evaluation of highly irradiated or highly contaminated materials. Photo courtesy of Westinghouse Electric Company.

ATR NSUF Industry Program Forges New Path Forward (cont.)

known as stress corrosion cracking, or irradiation-assisted stress corrosion cracking (IASCC) when the environment includes radiation.

“This project was a naturally good fit for us because it allowed us to go from beginning to end,” Jackson said. For that initial project, ATR NSUF got the material from EPRI, machined it, and then irradiated it in the ATR center flux trap. “This was the first civilian program that used the ATR center flux trap where we have installed our controlled water chemistry loop. Incidentally, the loop was another response to industry requests,” he said.

That also led to the development of the newly built and installed IASCC test rig at INL’s Materials and Fuels Complex. “There are only three or four other facilities in the world where you can do the sort of testing we can do with our IASCC test rig, and ours is unique in that it is

First ATR NSUF Commercial Partner, Westinghouse, Steps It Up

In 2013, ATR NSUF welcomed its first commercial partner, energy giant Westinghouse, which has a hand in more than 40 percent of the 440 nuclear power plants operating in the world today. “This is a tremendous asset, because Westinghouse has capabilities that are unique in the world,” said Dr. John Jackson, ATR NSUF Industry Program lead.

“Over the past five years, we’ve worked with the ATR NSUF Industry Advisory Committee, we have made presentations at Users Week (the ATR NSUF annual event for nuclear scientists and engineers), and we have formed good relationships with the staff at INL,” said Dr. Mike Burke, Westinghouse senior technical staff member. “But now as the first commercial partner at ATR NSUF, this is really breaking new ground.”

Under the enhanced partnership with ATR NSUF, Westinghouse added its Materials Center of Excellence Laboratories (MCOE) Remote Testing and Metallographic Facility and accompanying laboratories to the wide variety of ATR NSUF nuclear materials and fuels research capabilities. Known as a hot cell, the facility allows engineers and technicians to remain safely separated from highly irradiated or highly contaminated materials, but still operate the test equipment and perform the necessary microstructural and chemistry evaluations of the materials via remote control, according to Brian N. Burgos, manager of Churchill operations. “That allows us to perform hands-off, remote evaluation of materials that may be representative of 60 years of exposure in a nuclear power plant.”

Tests like these are critical to the future of the nuclear energy industry. “With reactors that were originally designed for 40 years of operation and are now being extended out beyond 60 years, you have to understand how materials are going to behave at those longer reactor exposures,” Burgos said. This data can also be used to design new materials as well

"It's a great way of getting meaningful discussions flowing back and forth, and of promoting research that is relevant to commercial applications."

as advanced nuclear reactors, such as Westinghouse’s AP1000 power plant. The only Generation III+ nuclear reactor to currently have design certification from the U.S. Nuclear Regulatory Commission, the AP1000 incorporates a “passive safety system that relies on simple things like gravity, condensation and natural circulation to provide the cooling for the system,” Burgos said.

He and Burke have high hopes for the new partnership with ATR NSUF. “By opening up our facility to the nation’s scientific investigators, we are facilitating a more effective technical information exchange between the U.S. Department of Energy, university programs, and the commercial industry,” Burgos said. “It’s a great way of getting meaningful discussions flowing back and forth, and of promoting research that is relevant to commercial applications.”

Since the announcement about its new partnership status, Westinghouse has been fielding queries about the details and is already developing a university project to measure and characterize volume changes in core-surrounding materials that were irradiated in INL's Advanced Test Reactor, Burke said. Westinghouse is also working with several different INL scientists to machine-test their specimens at the Churchill facility.

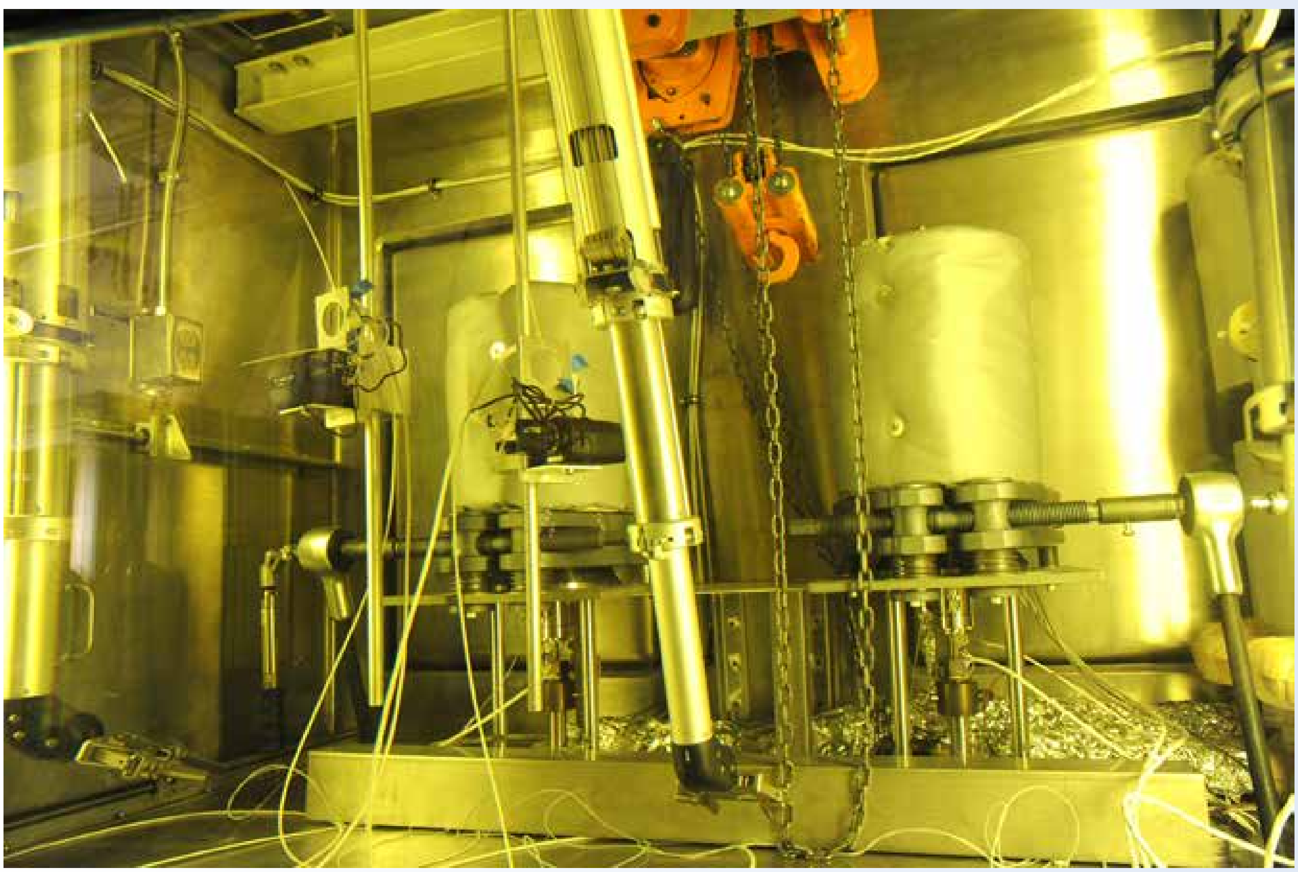
That’s just the start. “It’s clear from our discussions with different researchers that there are numerous other areas of common interest, and through the partnership, we think we will see many of them carried into projects,” Burke said.

more heavily shielded than the others and can therefore handle higher levels of radioactivity," he said. Not only can material samples be tested in the most extreme of conditions, but the INL IASCC test rig can also test higher volumes of material.

"In addition to that, our systems are capable of testing full-size specimens." That's important when testing certain classes of internal structural materials used in reactors. Austenitic stainless steels are an example of such materials. "If you tested a smaller sample, you may not get a true fracture toughness," Jackson said. Fracture toughness is essentially a measure of the energy required to induce failure in material containing a pre-existing crack. "Instead, a smaller specimen is going to give you a size-dependent toughness, which means you then have to figure out how to scale that to real-world applications," he said. By measuring a full-size specimen, the uncertainty is eliminated. "It gives you a true, usable toughness number, and for engineering applications, you really need that."

The IASCC test rig is providing important information, Jackson said. The irradiated nickel-base Alloy X-750 from the EPRI-suggested project is currently being tested using the brand-new IASCC test rigs, and the results will soon be evaluated. "In this case, EPRI is able to test materials under the conditions it is interested in and at a very significantly reduced cost, while we at the ATR NSUF have been able to prove to industry that we can do this."

EPRI has indeed been pleased with the development and installation of the IASCC test rig. "This is an example of collaboration between the U.S. Department of Energy (DOE) and industry, with DOE and ATR NSUF providing the irradiation facilities and irradiation exposures, and industry funding the testing," Pathania said, noting that the EPRI funding came from its Boiling Water Reactor Vessels and Internals Project, which has participation from the light water reactor industry in the U.S. and around the world.



The newly built and installed Irradiation-Assisted Stress Corrosion Cracking (IASCC) test rig at INL's Materials and Fuels Complex is a unique facility that not only has the capacity to conduct examinations of large volumes of materials and full-size specimens, but also can inspect materials that are highly radioactive.

ATR NSUF Industry Program Forges New Path Forward (cont.)

Further experiments in the IASCC test rig will add to databases that describe the effects of different irradiation doses on various structural materials. “There’s very little data in the world in terms of crack growth in these structural components under these kinds of controlled conditions,” Jackson said. “The hope is to use the IASCC test rig, as well as our other state-of-the-art equipment, to begin to fill in some of these major holes in the understanding of the behavior of irradiated materials, and highly irradiated materials in particular. This information is critical to the sustainably of the light water reactor fleet.”

Staying on Track

The task ahead is to add another layer of collaboration to the Industry Program, this time between industry and the university scientists and engineers who conduct most of the nuclear energy research studies. “There’s a change in the Industry Program. From now on, universities will play a much larger role,” Jackson said. “Historically, universities have had their own projects with ATR NSUF, and industry has had its own projects with ATR NSUF. Now we’ve gotten to a point where we can start to pair up university and industry participants.”

Those collaborations are critical, said Pathania, who also chairs the Industry Advisory Committee, which provides the industry’s perspective about research done at ATR NSUF. Committee members include people from EPRI, as well as major utility companies, vendors and industry labs. “It’s very important to make sure that ATR NSUF resources are used not only to do fundamental research, but also to do research that has some application. To get the most out of the facilities, university researchers should be aware of what the industry’s needs are, and industry should be aware of the latest technologies, information and ideas that are coming out of universities,” he said. “It’s really a two-way thing.”

The Industry Program is stepping in to help facilitate those partnerships. “We can act as a matchmaker in that sense and pair up an industry participant with a university participant,” Jackson said.

One ATR NSUF research project that is already underway incorporates a suite of microstructural-analysis tools at the Center for Advanced Energy Studies, which is where ATR NSUF is headquartered. The tools include transmission

“To get the most out of the facilities, university researchers should be aware of what the industry’s needs are, and industry should be aware of the latest technologies, information and ideas that are coming out of universities.”

electron microscopy, focused ion beam milling, scanning electron microscopy and atom probe tomography. Through a collaboration between Atomic Energy of Canada Limited (AECL) and Canadian scientists, the project objective is to evaluate the microstructure of certain types of springs, specifically nickel-base Alloy X-750 spring spacers, to begin to understand what causes the springs to fail in Canada’s pressurized heavy water reactors, which are known as CANDU reactors.

Projects like this one help nuclear power companies determine the lifetime of reactor components. By gaining more insight into flaws in materials and how quickly they propagate, they can make better-informed decisions about when they need to replace parts, switching them out only when needed but doing so before they fail, Jackson said.

EPRI has made other suggestions that have turned into projects and yielded results. One focuses on welds. Even when the structural materials and how they perform under irradiation are well understood, the welds that hold structural materials together are not, Pathania said. “We have data on welds that have not been irradiated, but we have pretty limited data on what happens to welds after they’ve been in the reactor for a long time.”

That idea grew into a proposal prepared by EPRI, INL, the Massachusetts Institute of Technology and the University of Florida in 2013 with the purpose of investigating the microstructure of stainless steel welds after exposure to varying thermal and irradiation levels. “It has a very broad scope, because we’re looking at their embrittlement properties and also what happens to their corrosion and cracking resistance,” he said. The project doesn’t have the green light from the DOE yet, but Pathania is optimistic. “This research is of interest to industry, includes fundamental science, and will help to train at least one student each from the University of Florida and MIT, perhaps more.”

Other current industry research projects include an EPRI-led project on irradiation-induced strain in zirconium, and one with the Nuclear Regulatory Commission to understand IASCC in austenitic stainless steel. "In addition to those projects, we have a couple in the works that also combine industry and university partners," Jackson said. "The Industry Program's main goals are to help develop research projects that are relevant and that support the U.S. Department of Energy's mission for ATR NSUF, and projects like these are definitely doing that."

Building the Next Generation

Another central mission of the Industry Program looks toward the future of the workforce. "There is a sort of gap between the older generation of nuclear scientists and engineers, and the current and coming generations that needs to be filled. When these older men and women retire, there is going to be a serious lack of scientists and engineers in the field," Jackson said. It was the recognition of that gap that was part of the impetus for ATR NSUF.

It is indeed vital to produce a new generation of researchers who understand the nuclear issues, said Pathania. "The nuclear workforce is aging, so we do need to develop a new generation of scientists and engineers who have the education, experience and motivation to carry the field into the future," he said.

One of the best ways to do that is through industry-university partnerships, Jackson asserted. Students get the opportunity to see their work used in the real world, and that is both affirming and exciting.

That's true, said Dr. Mike Burke of Westinghouse, which has been designing, manufacturing and servicing commercial nuclear power plants since the 1960s. "This is a very valuable message: Students can see that their scientific endeavors are carried out in the industry, that there is a future for them in this field."

Westinghouse became the first commercial partner at ATR NSUF in 2013 (see sidebar), and is already making plans to welcome student researchers. "We've had discussions with several different universities about having potential post-doc students actually reside here at our Churchill facility for a year's period of time," said Brian N. Burgos, manager of Churchill operations. "Those discussions are just beginning, but I think everyone on both sides – industry and academic – sees the value of having university students engaged in real-world commercial work."

The other benefit of such immersive student opportunities is that they provide an excellent bridge from college to career, Jackson said. "If you're an industrial entity with a student working at your site, it really offers a great chance to get to know these young researchers and see what they're made of. It's like an extended job interview, and in the end, you may well decide that this person would be a great fit for your company."

Pulling it Together

Overall, the ATR NSUF Industry Program has a considerable charge: to encourage industry-university collaborative research projects at ATR NSUF that are relevant to the nuclear industry, and through those projects, to provide industry access to facilities that would otherwise be out of reach; to support DOE's mission to maintain and improve the current fleet of nuclear reactors and to develop new technologies for future nuclear power production; and to help build the next generation of nuclear scientists and engineers.

Although the Industry Program has been in existence since 2008, it is well on its way to meeting those goals. "We are already working on cutting-edge, current problems and projects that are relevant to industry," Jackson said. "These will together support safer and more efficient reactor operation, and essentially maintain the sustainability of the fleet and improve nuclear production into the future."

New Instrumentation Stands and Delivers

Everybody needs a good set of tools. That is especially true for the scientists and engineers working on nuclear power research projects.

For them, however, the toolbox has been missing some key pieces, notably accurate, but exceptionally durable, instrumentation that can provide detailed information about the performance and strength of fuel and material samples inside a nuclear materials testing reactor (MTR), while themselves continuing to operate at optimum levels under the harsh conditions in an MTR. To fill that void, researchers at INL's ATR NSUF are developing new instrumentation to meet both those requirements.

Thermocouples

An example of a needed type of instrumentation is a thermocouple, a device that measures temperature via two wires that are typically made of metal. Researchers need thermocouples that can endure the high temperatures and intense bombardment of neutrons (also known as flux, or cumulatively, as fluence) in MTRs, such as the Advanced Test Reactor. This is because they often need to accelerate the radiation exposure a material would face in a commercial reactor. They irradiate materials at extremely high fluxes for short periods of time as a way of simulating the longer-term, but lower radiation exposure in operating reactors.

Back row (left to right): Christine White, Joy Rempe, Kurt Davis
Front row (left to right): Troy Unruh, Darrell Knudson, Benjamin Chase, Joshua Daw. Dr. Joy Rempe, shown here with her research team, recently moved into the new High Temperature Test Laboratory (HTTL) facility. The new facility has enhanced capabilities for conducting high-temperature material property testing and instrumentation development. The research group has already developed several specialized sensors with both the sensitivity and durability to measure the performance and strength of materials within the harsh environment of a nuclear reactor.

"It's a matter of testing as many different materials as possible in the most intensive conditions possible."

Two of the commonly used, commercially available thermocouples are type N, which is made of nickel-chromium-silicon and nickel-silicon wires, and the type K, which is made of nickel-chromium or nickel-aluminum wires. "The problem is that the commercially available type N and type K thermocouples are limited to about 1,100° C, and many tests in MTRs go to much higher temperatures," said Dr. Joy Rempe, who leads instrumentation development efforts for the ATR NSUF.

While some higher-temperature thermocouples are available, she said, they typically rely on materials for the thermoelements that cannot withstand the bombardment of neutrons they face in a test reactor. "These thermal elements will decalibrate due to transmutation – they will change their elemental composition because of the neutron exposure – so you can't rely on them either."

Rempe and her research team were able to step in and create thermocouples using alloys of the silvery metal called molybdenum and the soft, gray metal called niobium for the thermal elements. "These materials can withstand high temperatures and high fluences (neutron exposures) for long durations of irradiation without significantly decalibrating," she said. With the new thermocouples, researchers can obtain accurate temperature measurements throughout the irradiation.

This work benefits a number of U.S. Department of Energy (DOE) projects, and her lab is currently fabricating high-temperature thermocouples for a test to support the Next Generation Nuclear Plant program, which is funded by DOE.

In addition, the DOE is interested in her group's work in other areas. This includes the development of a new sensor for measuring changes in sample thermal conductivity, a new flux detector and evaluating the performance of candidate new ultrasonic transducers. The latter two efforts are funded through DOE's Nuclear Energy Enabling Technology (NEET) program.

Another component of the group's work is a melt wire library. Melt wires are wires of varying composition that melt at precise temperatures, and therefore serve as fairly simple but quite effective temperature-measuring sensors. "This and our measurements on silicon carbide temperature monitors are deployed not only in ATR NSUF tests for universities and the Electric Power Research Institute," Rempe said, "but also for other DOE programs." (See "ATR NSUF Industry Program Forges New Path Forward.")

Ultrasonic Transducers

Rempe and the instrumentation group are also collaborating with a Penn State University research group studying ultrasonic transducers as part of an ATR NSUF-funded experiment. "There are lots of different kinds of ultrasound-based sensors that are used outside the nuclear industry. We want to be able to adapt those technologies and use them 'in core' (in the reactor) because we think we'll be able to get measurements that are more accurate than those we can get right now, and also make completely new measurements that we couldn't in the past," said Joshua Daw, an INL research scientist and engineer who is part of the instrumentation group.

Through the project, they hope to characterize the survivability of candidate ultrasonic transducers when subjected to long-term radiation exposure. They are targeting two types of ultrasonic transducers: those made of piezoelectric materials, including aluminum nitride, zinc oxide, and bismuth titanate; and those made of magnetostrictive materials, including an iron-cobalt alloy called Remendur and a iron-gallium alloy called Galfenol.

After multiple discussions and reviews of the scientific literature, the researchers settled on these particular materials, which they felt had the most potential to be radiation-tolerant. Each of the materials has its pluses and minuses: some materials are very sensitive to incoming signals but don't transmit them very well, others have the opposite proficiency; some are more sensitive to neutron radiation, some less so. "It's a matter of testing as many different materials as possible in the most intensive conditions possible," Daw said. "In the long run, we have a long list of different sensors that we want to be able to deploy in-pile. Those that we will be able to pursue will depend on the results of this test."

The testing is underway in the Massachusetts Institute of Technology Research Reactor (MITR), which is a partner of the ATR NSUF. "This will be a very well-instrumented test," Rempe said. "We will have sensors to measure neutron flux in real time, sensors to measure gamma heating in real time, and thermocouples to

One of the HTTL's projects is the transient hot wire method needle probe (THWM NP), which is designed to yield measurements from inside a nuclear materials testing reactor during irradiation. Here, Benjamin Chase (left) and Joshua Daw discuss possible THWM NP testing options.

New Instrumentation Stands and Delivers (cont.)

measure temperature in real time as well as melt wires to verify peak temperatures and flux wires to verify neutron fluence," Rempe said.

The diversity and real-time aspect of these measurements are critical. Without these capabilities, the only other option is to conduct the irradiation experiment on the specimen, remove the irradiated specimen to a shielded nuclear radiation containment chamber, known as a hot cell, and then evaluate it based on data gathered before and after the experiment. In other words, the researcher can only infer exactly what happened in the reactor. "You can estimate based on calculations what the peak temperature was, for instance, but you don't have confirmation of that without data," Rempe said. "Furthermore, when you're taking the specimen out of the reactor and setting it in the hot cell, there's the potential for damaging it. For example, if there's a crack, you won't know whether it cracked on removal or cracked during the irradiation."

Ultimately, the researchers believe these tests will lead to much improved sensors for use in research projects. Daw said, "This would give us a whole new class of sensors that could potentially revolutionize the way we run tests in the ATR, at MIT or at any of the other test reactors."

NEET is funding Daw's involvement in the project. That makes good sense, he said. "The ultrasonic transducer irradiation test is, in my opinion, the definition of an enabling technology. If it's successful, we will enable the development of this entire new class of sensors," he said. These new sensors will advance many DOE programs. "Whenever we can, we try to find synergy between the different programs, and we like to think that our work on sensors will basically support every other program that has

"This would give us a whole new class of sensors that could potentially revolutionize the way we run tests in the ATR, at MIT or at any of the other test reactors."

anything to do with nuclear energy," he said. "I know that sounds like a big statement, but I think it's true."

At this point, the design work on the ultrasonic transducer irradiation test is completed and testing is underway. "We want to irradiate these things to a higher fluence than anybody has ever irradiated a piece of electric material before. Our maximum time frame is two years, although they may fail before that," he said. "Now it's just a matter of tracking the data and seeing how long everything lasts." The test is scheduled for insertion into the MITR in February 2014.

Ultrasonic Thermometry

Ultrasonic thermometry is a new type of temperature instrumentation under development in the instrumentation group at the ATR NSUF. The work is actually part of a doctoral thesis Daw is completing at the University of Idaho, and was primarily funded by the DOE's Fuel Cycle and Development program. He explained, "Basically, I take a wire, put a series of notches into it, send sound waves up the wire and listen for the reflections to come back down. From that, I can determine the temperature of the material between these reflections."

The advantage of the ultrasonic thermometer is that a single probe can take multiple temperature measurements, whereas a normal sensor can take only one. "That means

Research scientist Troy Unruh injects epoxy onto specialized micro-pocket fission detector (MPFD) components. These detectors are used to simultaneously measure thermal neutron flux, fast neutron flux and temperature.

The Next Generation of Nuclear Researchers

Where is the next generation of nuclear engineers coming from? It depends who you ask. Three members of the INL instrumentation group described their very different backgrounds and paths into the field.

Joshua Daw

Daw is a first-generation college graduate. “Although my parents both attended college, they had to quit before completing their degrees,” he said. “My mother quit in order to focus on the family; and my father was drafted into the Vietnam War, where he won several medals for valor. As such, it was important to them that I be the first in the family to earn a college degree.”

He chose engineering, because as a child growing up on an Idaho potato farm, he spent many hours watching Star Trek. “It was always the engineers who saved the day,” he said.

In order to stay close to his family, he chose Idaho State University. “I did really well. I was in a couple of honor societies and even was on the dean’s list seven semesters in a row,” he said. He was such a good student, in fact, that he was offered a University of Idaho master’s fellowship working at INL. “I’ve been at INL ever since.”

He continued his education. After being chosen as the University of Idaho, College of Engineering Outstanding Graduate Student in 2008, he earned his master’s degree, and is now on his way to completing his doctorate in May 2014. His primary thesis advisor, UI Professor and Chairman of Mechanical Engineering John C. Crepeau describes Daw as in the top 1-3 percent of the graduate students that he has had at the University of Idaho, a considerable achievement for a first-generation college student.

Joshua Daw, shown here obtaining data from ultrasonic thermometer resolution evaluations, is completing his doctorate at the University of Idaho.

At INL, Daw traveled to Halden, Norway, and completed a six-month assignment as a visiting researcher in the Fuels Division. In January 2014, he was awarded the INL Exceptional Engineering Achievement Award for his sustained contributions to the development and deployment of innovative in-pile instrumentation, in particular for research enabling deployment of ultrasound-based sensors.

the ultrasonic thermometer will be able to measure the temperature gradient along its length instead of just the temperature at one point,” Daw said. Knowing the gradient is especially critical for validating new multiscale multiphysics modeling tools, which may ultimately reduce the amount of irradiation testing required for deploying new fuels.”

Ultrasonic thermometry has a long history. INL researchers first started working on the idea back in the late 1960s, and scientists and engineers from around the world have since tried to make it into a practical device. The supporting technologies, however, just weren’t available until now. “Today, we can sample faster; we have new techniques for signal processing; we can make new materials; and we can fabricate things in ways that previously weren’t possible,” Daw said. “There have been advancements in just about every aspect involved with the ultrasonic thermometer.”

Using those advancements, Daw has been able to build a thermometer that can receive and send signals. Currently the signals require the use of an algorithm to process the signal data and obtain temperatures. By working with ultrasonic research groups at Argonne National Laboratory and Pacific Northwest National Laboratory on an upgrade,

New Instrumentation Stands and Delivers (cont.)

The Next Generation of Nuclear Researchers (cont.)

Benjamin Chase

Chase is an Idahoan, too. "I am originally from Pocatello, so while I was growing up, I was influenced by the presence of Idaho National Laboratory nearby," he said. When he graduated from high school, he enrolled at ISU and initially had an interest in applying to medical school. He was working on a degree in the biological sciences, but he was intrigued by the coursework in the physical sciences.

That interest led him to pursue an engineering degree instead. The influence of INL in the region steered him toward the nuclear engineering program. He earned his bachelor's degree, and then joined the Navy, where he wound up teaching at the Naval Nuclear Power School in Charleston, South Carolina.

As his Navy stint ended, he hoped to return to the Intermountain West. Again, he was drawn to INL. "It presented an enticing opportunity for me to come back to this area and engage in a rewarding job in the nuclear industry," he said.

He started out at INL as a nuclear reactor engineer for the ATR and has since moved to the instrumentation group at the High Temperature Test Laboratory, where he is working on the transient hot wire method needle probe. He has been awarded two INL Exceptional Contribution Awards for work on ATR NSUF experiments in the ATRC. While at INL, he returned to ISU to get his master's degree, which he is finishing up now, and is already taking classes toward his doctorate in nuclear engineering.

Benjamin Chase highlights key aspects of THWM NP response data to HTTL visitors. A Navy veteran, he is finishing his master's degree at Idaho State University and is taking courses toward his doctorate.

he hopes to enhance the signal-processing step. "We want users to be able to read temperatures directly and in real time," he said.

Added Rempe, "The ultrasonic thermometer would be very beneficial. A single UT ultrasonic thermometer can provide an axial temperature profile and can go to very high temperatures: at least 2,600° C, and perhaps higher. This would be a major step forward in irradiations in MTRs, such as the ATR, and in transient test reactors, such as the TREAT reactor that may be restarted at INL."

Transient Hot Wire Method Needle Probe

One piece of new instrumentation that is drawing international interest is the transient hot wire method needle probe (THWM NP), which is under development at INL. Like the ultrasonic thermometer, the probe is designed to yield measurements from inside an MTR during irradiation. It measures thermal conductivity and is under consideration as a replacement for other methods that are used in MTRs, such as the Halden Reactor in Norway.

"In the two-thermocouple method, there's a thermocouple in the material you're testing and a second thermocouple that's in the coolant, so many assumptions are required related to heat transfer from the fuel to the cladding and from the cladding to the coolant. These assumptions can adversely affect the accuracy of the data," said Benjamin Chase, a research and development engineer at INL and a master's student at Idaho State University (ISU). His work on the probe is part of his master's thesis.

Troy Unruh

A Kansas native, Unruh got excited about nuclear engineering during a high school trip to Kansas State University. “They showed us their nuclear reactor as part of the tour and told us that if you were in the mechanical engineering program and took a few extra classes, you could be a nuclear engineer. I thought, ‘Wow, this is really cutting-edge stuff!’” After graduating a year early from high school, he enrolled. Once he got to KSU, he found out that if he took the training, he could get a job actually working at the nuclear reactor on campus. He couldn’t resist. He became a nuclear reactor operator when he was 20 years old. “When you’re in college, you want to do something to set yourself apart. What does that better than saying you operate a nuclear reactor?” he said with a laugh. He stayed on at KSU for a master’s degree that focused on semiconductor neutron detectors. This research led to Unruh receiving an R&D 100 award.

Nearing graduation, Unruh received a recommendation from the reactor manager at KSU and landed a position as a reactor engineer at the ATR. A few years later, following a successful six-month staff exchange that sent him to France to study fission chambers, he had the opportunity to move into the INL instrumentation group. “I jumped at the chance. The research side has always been my passion, and I love it.”

Research scientist Troy Unruh, shown here inserting an MPFD prototype into the HTTL furnance for heating evaluations, is pursuing his doctorate from ISU.

Recently, Unruh helped complete flux-detector evaluations at INL’s ATRC. Like his colleague Chase, he was also awarded two INL Exceptional Contribution Awards for work on ATR NSUF experiments in the ATRC and is working toward a doctorate in nuclear engineering at ISU.

The THWM NP is designed to offer a streamlined alternative: one probe with one thermocouple and, consequently, fewer uncertainties. So far, the tests on the probe are promising. “In theory, we think it’s a better idea than the two-thermocouple method; initial testing is showing high accuracy,” Chase said, noting that much of the early work on the probe was conducted by Daw with substantial contributions from INL researcher Darrell Knudson.

The next round of tests will be done at the Halden Reactor and the Alternative Energies and Atomic Energy Commission (the nuclear research organization in France, abbreviated CEA). “CEA will test the needle probes on nuclear fuels in a furnace, and Halden will test the probes in fuel in a reactor environment,” Chase said.

He foresees this research being valuable to a number of DOE programs, including the Advanced Fuel Cycle program and to industrial organizations proposing new fuels for commercial reactors. He remarked, “If we can indeed get improved data with the needle probes, we could help increase understanding of fuel behavior, decrease margins without adversely impacting safety and improve nuclear reactor economics.”

Flux Detection

Better flux and fission detectors are in the works, too. “Reactor power is really tied to how many neutrons you have inside the reactor core, so the number of neutrons – the flux – is an important measurement to tell you how the reactor is operating,” said research scientist Troy Unruh,

New Instrumentation Stands and Delivers (cont.)

who is pursuing his doctorate from ISU and helping to facilitate the ISU-led project on flux detectors.

In the flux-detector project, researchers are testing a wide range of sensors in the Advanced Test Reactor Critical Facility (ATRC), a full-size but lower-power version of the ATR. 'The ATRC is nice because it offers a way to test the sensors and to make sure we're satisfied with how they're operating before we put them in the ATR, where fluxes are much higher," Unruh said.

By testing the sensors, they are learning which work best for different applications. "For some sensors, we want a very sensitive measurement down to counting every single neutron, and for that we are using some specialty fission chambers from the CEA," he said. Unruh has an excellent working relationship with the CEA, because he spent six months at CEA facilities as part of a researcher exchange program in 2010-2011.

They are also testing sensors known as self-powered neutron detectors, which were obtained from ISU Nuclear Engineering Professor George Imel. These detectors are not as precise as the fission chambers, but they function at very high powers and last much longer because of their simple design and operational characteristics, Unruh explained. In addition, the researchers are concurrently conducting validation experiments to test the accuracy of computer models developed by INL physicists and how well they reflect the sensor-detected fluxes inside the reactor.

Another sensor, called a back-to-back fission chamber, will undergo testing in 2014. "Just like it sounds, it's effectively two chambers in one, and it's made such that we can measure different energies of neutrons in just one detector," he said. "This is important because it allows us to calibrate the other sensors and determine which works better for different neutron energies."

"All of this gives us better information that we can then use to design more effective materials and improve reactor operation and safety."

The Big Picture

Overall, the goal of the flux-detector project and the other instrumentation work is quite straightforward. "The reason we do all of this work is to make sure we know how the various sensors will survive in proposed test conditions. Once we know that, then we can do what's most important, which is to measure what's going on inside the reactor," Unruh said.

"These high-fidelity, real-time sensors that can survive conditions in an MTR are extremely important for any DOE or other program that's testing in the ATR, because they'll be able to provide very accurate information in real time, and we couldn't do that before," he said. "With these new sensors, we will be able to see exactly when the material fails, or exactly at which temperature it reaches, or exactly how the thermal conductivity changes over the irradiation history. All of this gives us better information that we can then use to design more effective materials and improve reactor operation and safety."


Benjamin Chase, a research and development engineer at INL, obtains data from THWM NP from various material samples.

Diverse ATR NSUF Research Advances Nuclear Energy

As nuclear reactors enter their sixth decade of providing power to the United States, a new push is underway to expand nuclear energy and to improve the safety, economy and sustainability of reactors. This includes the development of advanced reactors (Generation-IV or Gen-IV reactors) that will operate under even higher temperatures and irradiation levels than current reactors.

Advanced reactors will have demands of their own, including improved and new nuclear fuels and materials that function well within those extreme conditions. To do that, researchers must get a much clearer picture of fuels and materials, and how they respond to increasingly high temperatures and radiation levels. Through ATR NSUF, much of the work is taking place at Idaho National Laboratory (INL), affiliated facilities at partner institutions and universities, other national laboratories and industry.

Dr. Yongho Sohn and his University of Central Florida research group are characterizing metallic fuels and materials, which are potential candidates for advanced reactors, to understand the specific effects of high temperatures and irradiation. Here, Sohn (left) and doctoral candidate Youngjoo Park use a transmission electron microscope to examine samples. The full research group (inset photo) includes students and scholars in front of Sohn's Laboratory for Materials and Coatings for Extreme Environment. Photos provided by Yongho Sohn's research group.

"All of these projects that we have done and are doing would simply not have been possible without the ATR NSUF program."

Three projects that highlight the range and importance of this research are:

- Metallic fuels for highly efficient energy production, which are under study by the research group of Yongho Sohn at the University of Central Florida (UCF).
- Comparison of radiation tolerance of high-tech nanocrystalline and ultrafine grain materials to conventional materials currently used in reactors, led by K. L. Murty and his team at North Carolina State University (NCSU).
- Radiation damage accumulation in steels and other metals used in or proposed for reactors, and how their microstructure affects their performance, conducted by Mitra Taheri and her research group at Drexel University.

Metallic Fuels

Metallic fuels are generating a good deal of interest as potential candidates for advanced reactors for a few reasons. "One advantage is they have a higher energy production efficiency when compared to the currently used fuels," said Dr. Sohn, UCF professor of materials science and engineering. The reason is that metallic fuels – fuels that may contain a variety of metals, such as uranium, silicon, zirconium and molybdenum – have a higher burn-up capability, higher fission density and higher thermal conductivity than typical oxide fuels. "By using metallic fuels, we can therefore produce energy with more efficiency," he said.

On the flip side, metallic fuels tend to interact with their surroundings, including the metal-alloy cladding that encases the fuel. "We want to understand the interaction completely so we can predict what's going to happen when these fuels are used in a reactor," he said. To do that, he and his research group are focusing on the two major reactor conditions that affect fuels and materials: high temperatures and irradiation.

"Our expertise is really in the process of diffusion and reaction in materials," Sohn said. Diffusion occurs when the atoms in a material move out and into another material, and reactions can occur when one material, sometimes as a result of diffusion, is transformed into another. "This is a high priority interest for us because when these materials

The research group of Dr. K. L. Murty at North Carolina State University has several ongoing projects, including a study of nanocrystalline and ultrafine-grained materials. For this work, the group utilizes both NCSU and Idaho National Laboratory facilities to study the effect of grain size on material properties under the extreme conditions inside a reactor. Pictured from left are: Hao Ping, Botros Hanna, Dr. Apu Sarkar, Murty, Boopathy Kombaiah, Ahmad Alsabbagh, and visiting scholar Dr. Linjiang Chai. Ping is a graduate student; Hanna, Kombaiah and Alsabbagh are doctoral students; and Sarkar is a post-doctoral researcher. Photos provided by the NCSU Nuclear Materials Research Group.

interactions occur, the system is basically intermixing, and this is directly related to the performance of power generation," he explained.

To determine the effects of high temperature on diffusion and reaction in materials, Sohn's group is meticulously characterizing metallic fuels and materials. "At the University of Central Florida, we are doing a portion of the characterization work 1) without irradiation, and 2) using depleted uranium rather than actual fuel," he said, explaining that depleted uranium is far more safe to handle in the laboratory than the uranium that powers a reactor.

With that knowledge in hand, they are now readying additional experiments at ATR NSUF that will add the irradiation component. "At this point, we have a small piece of the pie, and we want to understand the rest through ATR NSUF experiments. It's a summation basically: first understanding without irradiation, and then understanding with irradiation," he said. "We have samples ready for ATR testing in 2014, and once those are irradiated, we can compare them with the unirradiated samples, and that will tell us exactly how irradiation has altered the rate and mechanisms of diffusion and reactions."

Their experiments will be some of the first to provide quantitative measurements of the effects of temperature and irradiation with respect to diffusion, reaction and microstructural development. "At the end of our irradiation tests, we will be able to measure things quantitatively and identify impacts of irradiation clearly," he said. This falls into line with the U.S. Department of Energy's Reduced Enrichment for Research and Test Reactors program and its Fuel Cycle Research and Development program, which support Sohn's work. He said, "The ongoing collaboration with these programs prior to ATR NSUF, including those with Dr. Dennis D. Keiser Jr., Dr. Maria Okuniewski, Dr. Bulent Sencer and Dr. J. Rory Kennedy at Idaho National Laboratory, forms the pillars for the work at UCF. And the ATR NSUF program was devised with Dr. Maria Okuniewski for a true team effort, so that the best scientists and engineers from UCF and INL will contribute to the design, experiment, analysis and understanding."

Personally, Sohn is interested in the basic physics associated with metals and their behavior under irradiation. From a broader perspective, he hopes his group's work will "contribute to that of the many scientists and engineers who are trying to develop the next generation nuclear power systems." He added, "In the grand scheme of things, nuclear energy safety demands that our understanding of materials be complete, so we are very happy to be able to supply this piece of the puzzle, even if it is a small piece."

Diverse ATR NSUF Research Advances Nuclear Energy (cont.)

Murty (right) and Alsabbagh discuss the work at NCSU's Nuclear Materials Lab. Alsabbagh spends about half of his time at INL and the other half at NCSU.

Nanocrystalline/Ultrafine Grain Materials

Nanocrystalline and ultrafine-grained materials have become an especially hot area of research, much of it centered on their performance under high radiation levels.

Both materials are comprised of components called grains, and each grain in a nanocrystalline material is less than 100 nanometers in size, which is as small as – or smaller than – a virus. Ultrafine grains are between about 100-1000 nm in size, but still much smaller than those found in conventional materials (around 10-100 µm). Because of that, a volume of nanocrystalline or ultrafine-grained material not only has more grains compared to the same volume of a conventional material, but it also has many more spaces between the grains. These spaces are called grain boundaries.

The increased number of grain boundaries makes a difference inside a reactor during irradiation, when high-energy neutrons bombard the material. "These neutrons produce a lot of defects in the matrix of the material," said Dr. K. L. Murty, NCSU professor of nuclear engineering and director of graduate programs. Defects often involve the neutrons jarring grains from their place in the material's structure. In normal materials with larger-sized particles, these defects can accumulate over time and weaken the material to the point that it fails.

In nanocrystalline and ultrafine-grained materials, however, the dislodged grains are absorbed into the grain boundaries. "They have what can be described as a self-healing effect, and these materials are expected to be more radiation tolerant because of it," Murty said. That doesn't mean they are perfect. "The biggest problem is most of these nanostructured materials are really unstable and it's expected that their grain size will start increasing with even a slight rise in temperature in a reactor."

Before nanocrystalline or ultrafine-grained materials can be considered for advanced nuclear reactors, researchers need to gain much better comprehension of the effect of grain size on material properties under the extreme conditions inside a reactor, and that's where Murty and his research group come in.

"To get a basic understanding of the phenomenon, about six years ago, we came up with this idea of using copper and nickel," he said, explaining that the former was thought to experience an increase in grain size when exposed to reactor conditions, and the latter a decrease. They later added ultrafine-grained carbon steel, produced through a process called equal channel angular pressing (ECAP).

Using NCSU's PULSTAR nuclear research reactor, Murty's research group began irradiating samples of the materials to low neutron fluences, and then tested the samples at the much higher fluences available at INL's Advanced Test Reactor. As an example, doctoral student Ahmad Alsabbagh explained his study of ultrafine-grained materials. "We put samples of both ultrafine–grained materials and conventional materials – the same materials, but different grain sizes – under neutron irradiation at the same conditions, and then we studied them to see which one showed better radiation resistance," said Alsabbagh, who spends about half of his time at INL and the other half at NCSU. He will be finishing his doctorate in 2014.

One of the unique things about the NCSU project is the material sample itself. "With radioactive materials, the bigger the sample is, the more risky it is to handle it, so what we did is we created a new set-up so we can prepare and handle very minute samples," Alsabbagh said. The facilities ATR NSUF provides access to at INL made it easier, too, because "all of the machines we need to characterize the materials, including atom probe tomography (which provides three-dimensional, atom-by-atom imaging), are all in one place."

The ATR-irradiated nickel samples await characterization, but Murty and his research group have already been able to evaluate the copper samples. They verified that while the copper samples showed very good radiation tolerance initially when compared to the conventional materials, the grains eventually began growing.

> **“We want to understand the interaction completely so we can predict what’s going to happen when these fuels are used in a reactor.”**

The copper research was interesting, but it was the ECAP findings that got researchers especially excited: The low-dose irradiation of the ultrafine-grained ECAP steel revealed significantly fewer and much less severe radiation effects compared to conventional-grained steel. “These were very interesting results, because they showed that the ultrafine-grained ECAP steel is far, far superior to the conventional grain-sized materials,” Murty said.

“Even if ECAP carbon steel is not the exact kind of steel used in commercial reactors, work like this provides valuable information, especially for the DOE’s Nuclear Energy University Program (NEUP),” he said. NEUP focuses in part on the development of radiation-tolerant materials for the nuclear reactor fleet of today and tomorrow.

In addition, Murty’s research group is using ATR to study how irradiation can compromise welds and is also collaborating with Oak Ridge National Laboratory to understand the effects of irradiation on graphite. Graphite is a candidate material for advanced nuclear reactors and is also used as a neutron moderator in current nuclear reactors. (A neutron moderator tempers the speed of neutrons so they can sustain the nuclear chain reaction that is essential for a nuclear reactor operation.) That work is continuing.

ATR NSUF reactors and equipment have been beneficial in his group’s research, Murty said. “All of these projects that we have done and are doing would simply not have been possible without the ATR NSUF program.”

Microstructure and Irradiation

Nanomaterials are also the centerpiece in the work of Dr. Mitra Taheri, Hoeganaes assistant professor of metallurgy at Drexel University. She and her research team are interested in metal alloys that resist irradiation, high temperatures and corrosion, and are concentrating on nanocrystalline and ultra fine-grained nickel- and iron-based metals, including steels, as well as conventional stainless steels.

They conduct their research in an interesting way: They watch for structural defects in the material in situ, or as it is being irradiated. “We mostly use ion beams to mimic neutron irradiation, because then our material samples are not radioactive, and we can watch it dynamically and actually see the evolution of radiation damage,” she said. “So for instance, we can measure how much damage is taking place in a nanocrystalline material versus a conventional material at a particular radiation dose and over a certain time frame, and see where in the material the failures occur and how they occur.”

Dr. Mitra Taheri (pictured) and her research group at Drexel University are conducting experiments on radiation damage accumulation in steels and other metals used in or proposed for reactors, and learning how their microstructure affects their performance. Photo courtesy of Drexel University's College of Engineering.

Diverse ATR NSUF Research Advances Nuclear Energy (cont.)

> **"Information such as this is necessary to produce strong and resilient materials for advanced reactors."**

With the ion-irradiated results in hand, the researchers then conduct similar experiments at ATR NSUF's INL facilities, this time with neutron-irradiated materials. They also draw on the ATR NSUF sample library, which is a collection of various material samples that have already gone through neutron irradiation. From there, they use atom probe tomography equipment and a high-resolution transmission electron microscope at the Center for Advanced Energy Studies and INL's hot cell facility to compare the ion-irradiated and neutron-irradiated samples. This allows her research group to see exactly what was rearranged at the atomic scale after irradiation, and use that information to make alterations to the materials, eliminating portions that failed and enhancing those that didn't.

They are already getting good results. "Recently, we've shown that certain kinds of grain boundaries in the steels and nickel alloys prevent radiation-induced segregation, which can lead to embrittlement common to traditional materials," she said.

Information such as this is necessary to produce strong and resilient materials for advanced reactors, Taheri said, noting that DOE's Advanced Reactor Concepts program is supporting this work. In addition, DOE's Office of Basic Energy Sciences and NEUP, along with the Nuclear Regulatory Commission, are funding other of her group's projects, including a study of corrosion in zirconium alloys (Zircaloy), which are used in current reactors.

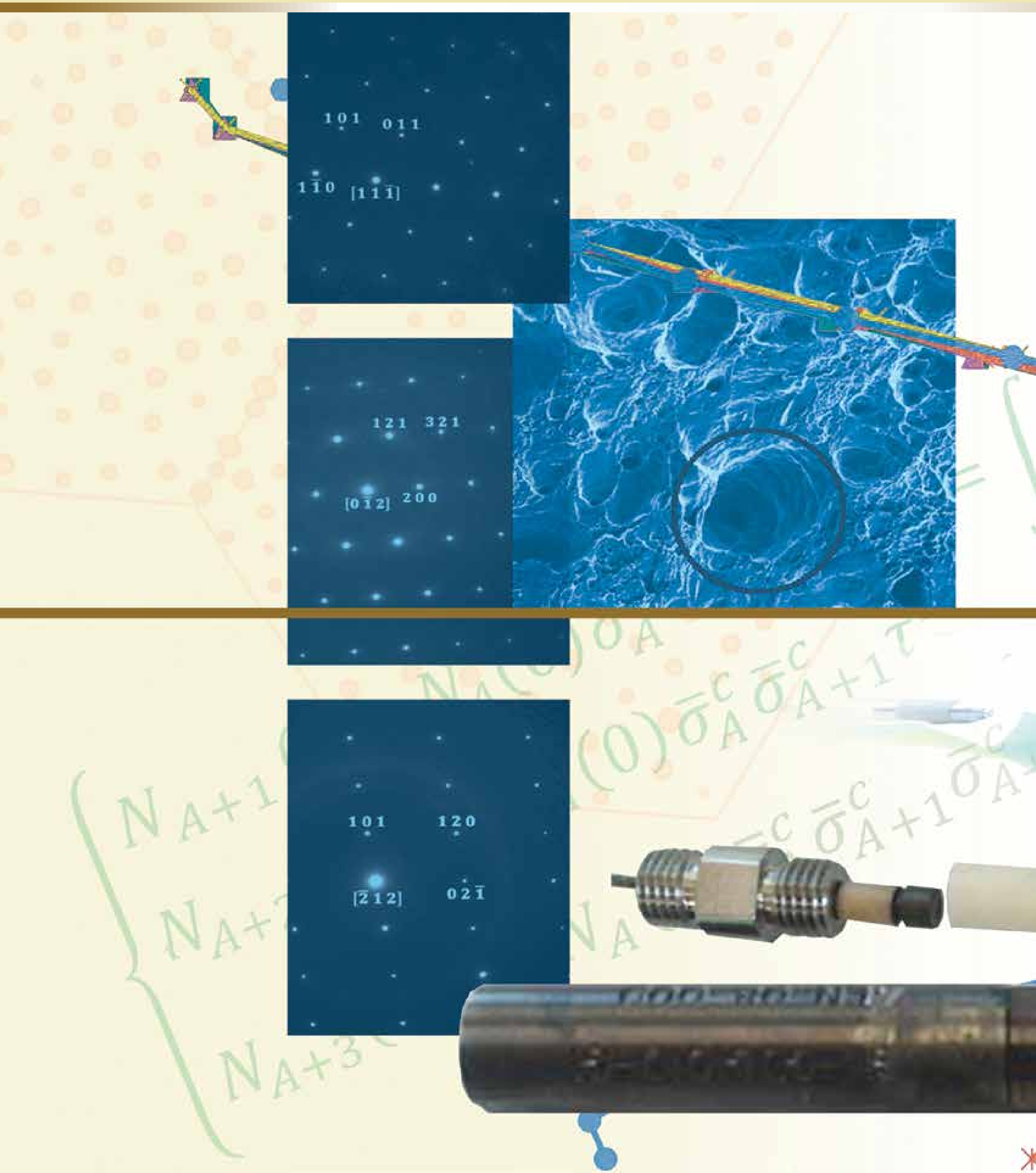
Overall, she said, "By studying materials dynamically through the ion-irradiated experiments and comparing this data with that gained from neutron-irradiated materials, we are able to use that data to lay a foundation to develop next-generation materials."

Research Impacts

The variety of collaborative projects underway at universities and ATR NSUF facilities highlight how far the field has come and also how much is left to learn. Improved and completely new nuclear fuels and materials are in the works as research groups design and run experiments to learn more about the impact of reactor conditions, and by doing so, help to ensure safe, economical and sustainable nuclear energy into the future.

"Scientific results from projects like this will guide us in designing new structural materials for current reactors as well as advanced reactors that will operate under higher temperatures and irradiation levels." Murty said, "It is a very exciting time."

ATR NSUF Program Information



Program Overview

ATR NSUF: A Model for Collaboration

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

In 2013, ATR NSUF's partnership program had eight universities, two national laboratories, and added one industry partner. The partner facility capabilities greatly expand the types of research offered to users. The avenues opened through these partnerships facilitate cooperative research across the country, matching people with capabilities, students with mentors. In 2013, ATR NSUF included INL and the following institutions:

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

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

U.S. DEPARTMENT OF ENERGY | Nuclear Energy

ATR NSUF Research Supports DOE-NE Missions

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

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

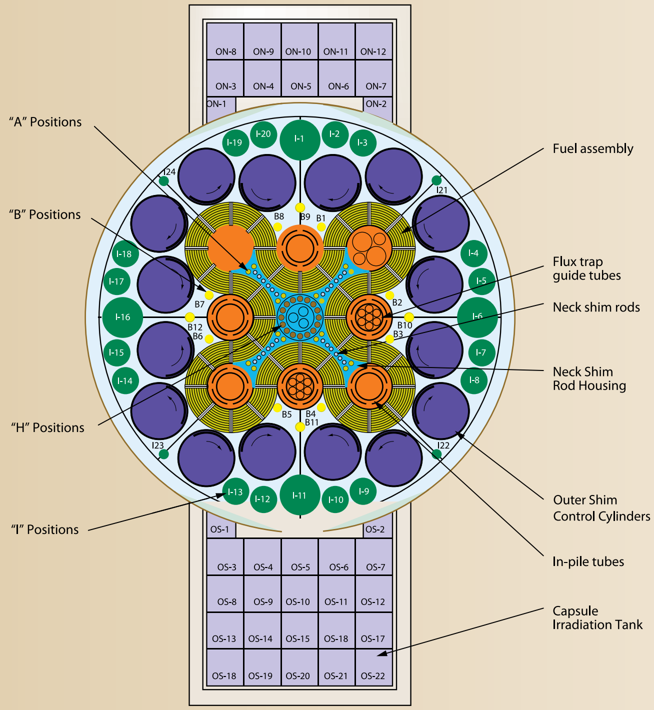
ATR NSUF research addresses a number of these mission needs. Most of the research contained in this report looks at either understanding the mechanisms of radiation on materials and fuels to address the challenges of the aging current fleet, or looks at materials and fuels for the next generation of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

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

Reactor Capabilities

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

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



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

Idaho National Laboratory Advanced Test Reactor

ATR is a water-cooled, high-flux test reactor, with a unique serpentine design that allows large power variations among its flux traps. The reactor's curved fuel arrangement places fuel closer on all sides of the flux trap positions than is possible in a rectangular grid. The reactor has nine of these high-intensity neutron flux traps and 68 additional irradiation positions inside the reactor core reflector tank, each of which can contain multiple experiments. Experiment positions vary in size from 0.5 to 5 inches in diameter (1.27 to 12.7 centimeters) and all are 48 inches (121.92 centimeters) long. The peak thermal flux is $1x10^{15}$ n/cm^2-sec and fast flux is $5x10^{14}$ n/cm -sec when operating at full power of 250 MW. There is a hydraulic shuttle irradiation system, which allows experiments to be inserted and removed during reactor operation, and pressurized water reactor (PWR) loops, which enable tests to be performed at prototypical PWR operating conditions.

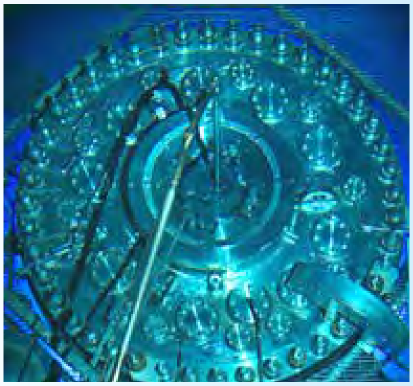
More information: http://atrnsuf.inl.gov/documents/ATRUsersGuide.pdf

Idaho National Laboratory Advanced Test Reactor Critical Facility

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

More information: http://atrnsuf.inl.gov/documents/ATR-C_UserGuide.pdf


Aerial view of the ATRC reactor core and bridge.

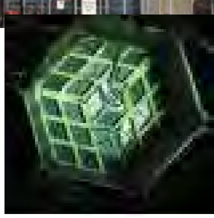


Top of the HFIR reactor.

Oak Ridge National Laboratory High Flux Isotope Reactor

HFIR is a versatile 85 MW research reactor offering the highest steady-state neutron flux in the western world. With a peak thermal flux of 2.5x10^{15} n/cm^2-s and a peak fast flux of 1.1x10^{15} n/cm^2-s, HFIR is able to quickly generate isotopes that require multiple neutron captures and perform materials irradiations that simulate lifetimes of power reactor use in a fraction of the time. HFIR typically operates 7 cycles per year, each cycle lasting between 23 and 26 days. Associated irradiation processing facilities include the Hydraulic Tube Facility, Pneumatic Tube Facilities for Neutron Activation Analysis (NAA), and Gamma Irradiation Facility.

More information: http://atrnsuf.inl.gov/documents/HFIR_UserGuide.pdf



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

Massachusetts Institute of Technology Reactor

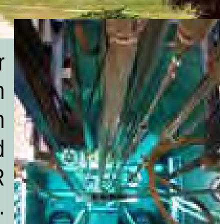
MITR is a 5 MW tank-type research reactor. It has three positions available for in-core fuel and materials experiments over a wide range of conditions. Water loops at pressurized water reactor/boiling water reactor (PWR/BWR) conditions, high-temperature gas reactor environments at temperatures up to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

More information: http://atrnsuf.inl.gov/documents/MITR_UserGuide.pdf

North Carolina State University PULSTAR Reactor

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

More information: http://atrnsuf.inl.gov/documents/PULSTARReactor.pdf

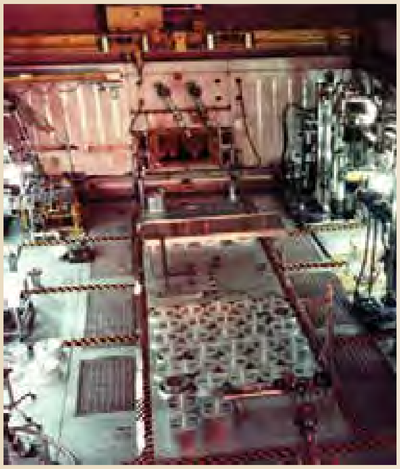


(above) PULSTAR reactor facility on the NCSU North Campus in Raleigh, North Carolina. (right) Downward view of the PULSTAR reactor pool.

Post-irradiation Examination Capabilities

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

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



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

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

HFEF is a large alpha-gamma hot cell facility dedicated to remote examination of highly irradiated fuel and structural materials. Its capabilities include nondestructive and destructive examinations. The facility also offers a 250 kWth Training Research Isotope General Atomics (TRIGA) reactor used for neutron radiography to examine internal features of fuel elements and assemblies.

The Analytical Laboratory is dedicated to analytical chemistry of irradiated and radioactive materials. It offers National Institute of Science and Technology (NIST)-traceable chemical and isotopic analysis of irradiated fuel and material via a wide range of spectrometric techniques.

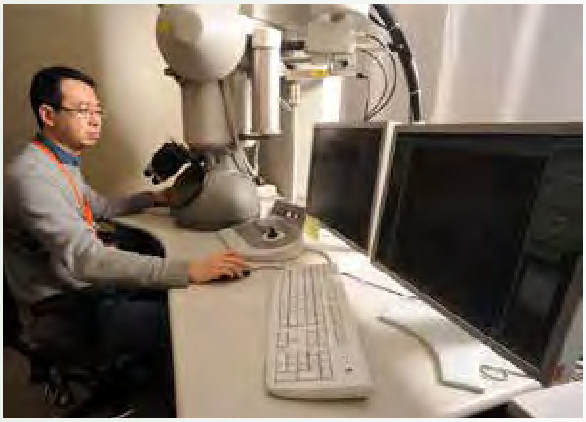
The Electron Microscopy Laboratory (EML) is dedicated to materials characterization, primarily using transmission electron, scanning electron and optical microscopy. The EML also houses a dual-beam FIB that allows examination and small-sample preparation of radioactive materials.

More information: http://atrnsuf.inl.gov/documents/INL_PIE_UserGuide.pdf

Center for Advanced Energy Studies Microscopy and Characterization Suite

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

More information: https://inlportal.inl.gov/portal/server.pt/community/caes_home/281/macs_home



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

North Carolina State University Nuclear Services Laboratories

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

More information: http://atrnsuf.inl.gov/document/PULSTARReactor.pdf

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

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

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

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

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

More information: http://atrnsuf.inl.gov/documents/ORNL_PIEUserGuide.pdf

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

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

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

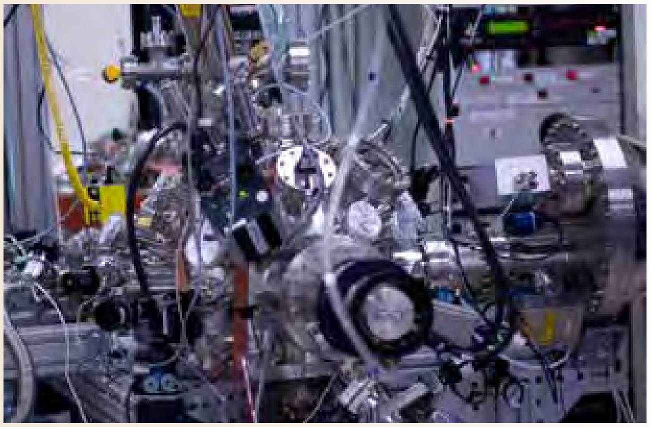
More information: http://atrnsuf.inl.gov/documents/PNNL_UserGuide.pdf

Post-irradiation Examination Capabilities (cont.)

Purdue University IMPACT Facility

The Interaction of Materials with Particles and Components Testing (IMPACT) facility offers a wide range of spectroscopy techniques to study the surface of materials. The IMPACT facility houses a variety of examination instruments including low-energy scattering spectroscopy (LEISS), X-ray photoelectron spectroscopy (XPS), auger electron spectroscopy (AES), extreme ultraviolet reflectometry (EUVR), extreme ultraviolet (EUV) photoelectron spectroscopy and mass spectrometry.

More Information: http://atrnsuf.inl.gov/documents/PURDUEIMPACTLAB.pdf



The IMPACT facility at Purdue University.

University of California, Berkeley Nuclear Materials Laboratory

UC Berkeley nano-indentation system.

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

More information: http://atrnsuf.inl.gov/documents/UCBerkeley_UserGuide.pdf

University of Michigan Irradiated Materials Complex

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

More information: http://atrnsuf.inl.gov/documents/UniversityofMichiganIMCandMIBLFacilities.pdf

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

University of Nevada, Las Vegas Harry Reid Center Radiochemistry Laboratories

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

More information: http://atrnsuf.inl.gov/documents/UNLVPartnerFacilityUserGuide.pdf

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

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

University of Wisconsin Characterization Laboratory for Irradiated Materials

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

More information: http://atrnsuf.inl.gov/documents/UniversityofWisconsinCLIMGuide.pdf

Westinghouse Materials Center of Excellence Laboratories

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

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

Beamline Capabilities

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

In 2013, the ATR NSUF program offered researchers access to four university partner beamline facilities. These include the Illinois Institute of Technology Materials Research Collaborative Access Team (MRCAT) beamline at Argonne's Advanced Photon Source, the PULSTAR reactor facility at North Carolina State University, the University of Michigan Ion Beam Laboratory, and the University of Wisconsin Tandem Accelerator Ion Beam.

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

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

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

More information: http://atrnsuf.inl.gov/documents/AdvancedPhotonSource.pdf

North Carolina State University
PULSTAR Reactor Facility

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

More information: http://atrnsuf.inl.gov/document/PULSTARReactor.pdf

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

University of Michigan
Michigan Ion Beam Laboratory

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

More information: http://atrnsuf.inl.gov/documents/UniversityofMichiganIMCandMIBLFacilities.pdf

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

University of Wisconsin
Tandem Accelerator Ion Beam

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

More information: http://atrnsuf.inl.gov/documents/UniversityofWisconsinCLIMGuide.pdf

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

Calls for Proposals

Jeff Benson
Program Administrator

Calls for Proposals

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

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

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

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

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

The ATR NSUF annually conducts two open calls for proposals: a fall call, which opens in late July and closes in December, and a spring call, which opens in late January and closes in June. Proposals are accepted for:

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

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

Calls for Rapid Turnaround Experiments

Rapid turnaround experiments are experiments that can be performed quickly — typically in two months or less — and include, but are not limited to, PIE requiring use of an instrument (FIB, TEM, SEM, etc.), irradiations in the PULSTAR reactor, ion beam irradiation and neutron scattering experiments. Proposals for rapid turnaround experiments are reviewed on a quarterly basis in January, April, July, and October and awarded based on the following rankings:

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

For more information, visit the ATR NSUF website at http://atrnsuf.inl.gov

ATR NSUF Sample Library

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

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

Note: The program may be changing in the future, see the website for the most current information on calls.

Users Week

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

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

For more information about Users Week, visit http://atrnsuf.inl.gov

The week's events are free of charge for students, faculty and post-docs as well as researchers from industry and national laboratories who are interested in materials, fuels, post-irradiation examination and reactor-based technology development. In the five years since its inception, ATR NSUF Users Week has hosted 568 participants from 30 countries and 35 U.S. universities.

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

What to Expect at Users Week

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

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

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

Educational Programs and Opportunities

Jeff Benson
Program Administrator

Faculty/Student Research Teams (FSRT)

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

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

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

Photos of educational opportunities: (above) Touring Hot Fuel Examination Facility during Users Week. (opposite top) Students at Users Week Introductory Workshop. (opposite bottom) Students touring Electron Microscopy Laboratory.

Proposals should be designed to meet the following criteria:

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

Proposals are generally submitted in January, reviewed and funded in early summer through a subcontract to the university faculty project lead.

Graduate and Undergraduate Internships

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

To learn more about educational opportunities visit the ATR NSUF website at: http://atrnsuf.inl.gov

Distributed Partnership at a Glance

Partners

BERKELEY
UNIVERSITY OF CALIFORNIA

ILLINOIS INSTITUTE OF TECHNOLOGY

MIT Massachusetts Institute of Technology

University of Michigan

NC STATE UNIVERSITY

OAK RIDGE National Laboratory

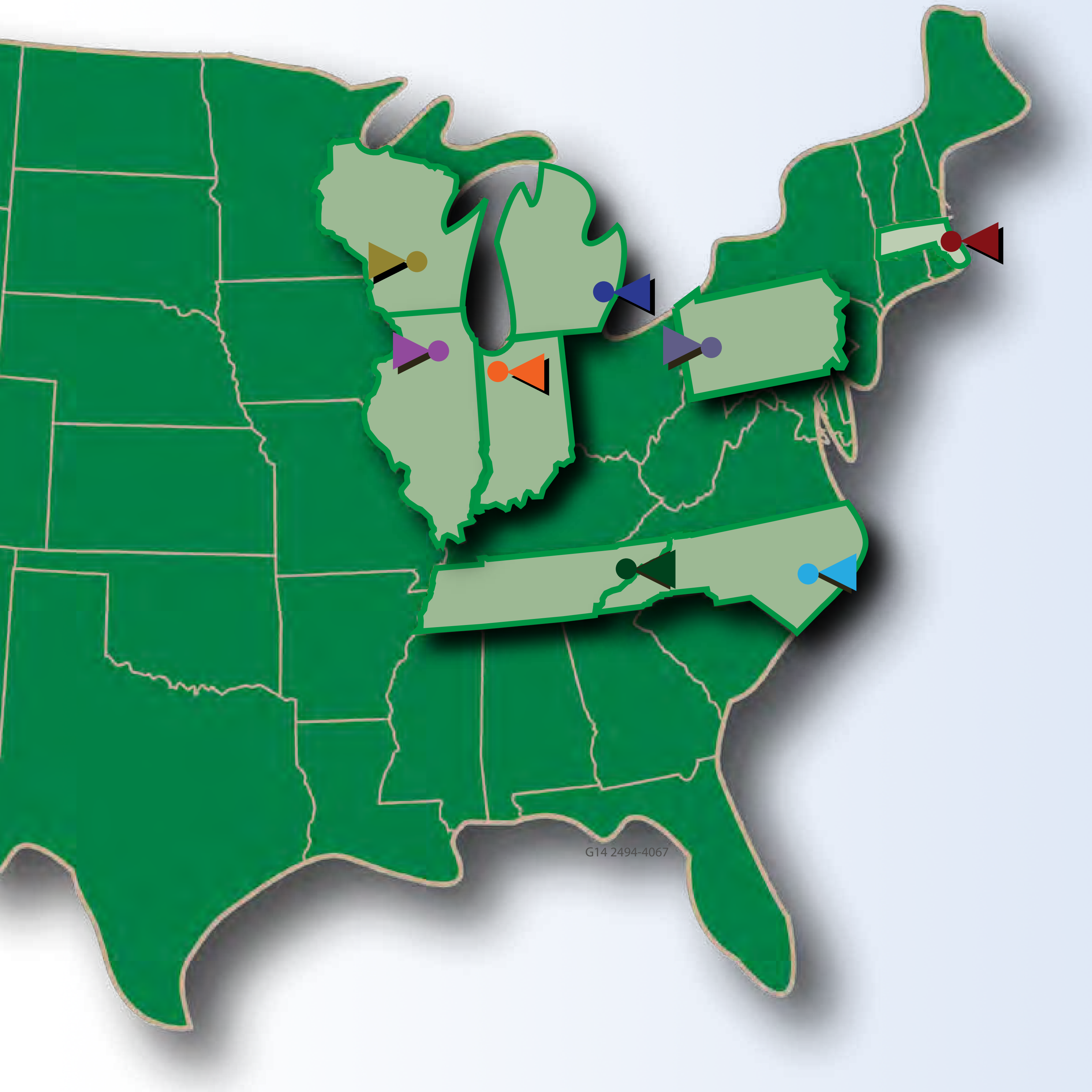
Pacific Northwest NATIONAL LABORATORY

PURDUE UNIVERSITY

UNLV

WISCONSIN
UNIVERSITY OF WISCONSIN-MADISON

Westinghouse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ATR NSUF Research Project Reports


EZ-VIEW® Microscope
CPP

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

Principal Investigator: Heng Ban–Utah State University
email: heng.ban@usu.edu

Introduction

Fast neutron irradiation tests are essential to fuels and materials development requirements for next-generation nuclear reactors. However, the lack of domestic, fast neutron testing capabilities hinders that development.

Project Description

The concept behind this project is to use one of the ATR corner lobes, with the addition of a thermal neutron filter to absorb the thermal neutrons and booster fuel, and augment the neutron flux. An absorber material composed of hafnium-aluminide (Al_3Hf) particles in an aluminum matrix (Al_3Hf-Al) can absorb thermal neutrons and transfer heat from the experiment to pressurized water cooling channels. Thermal analyses conducted on a candidate configuration of this new material confirmed that the design of the water-cooled Al_3Hf-Al absorber block is capable of maintaining all system components below their maximum allowable temperature limits.

However, the thermophysical properties of Al_3Hf have never been measured, and the effect of irradiation on these properties has never been determined. The purpose of this project is to determine the effect of irradiation on the thermophysical and mechanical properties of both the Al_3Hf intermetallic and Al_3Hf-Al composite. That data, along with other information such as corrosion behavior and radioactive decay products, are necessary in order to proceed with design and optimization of the block. Specific objectives are to determine:

- Thermophysical and mechanical properties of both the intermetallic and composite materials at different temperatures.
- The effect of irradiation on these properties, as well as the physical/morphological, metallurgical and microstructural changes of Al_3Hf-Al after different cycles of irradiation.
- Decay products of Hf-179ml versus Hf-179m^2.
- Corrosion behavior of Al_3Hf-Al.
- Successful completion of this project will provide necessary data for the development of a fast neutron test capability at ATR. It will also fill a knowledge gap on the basic properties of the Al_3Hf intermetallic and Al_3Hf-Al composite, and advance the scientific understanding of the irradiation effects on these materials. The end result, in terms of the data and fundamental understanding obtained, will directly support the Department of Energy's (DOE) mission and benefit the science community in general.

The lack of domestic, fast neutron testing capabilities hinders the development of next-generation reactors. This ATR NSUF project seeks to provide data necessary for the development of a fast neutron test capability at ATR.

Accomplishments

The irradiation test plan (ITP) was prepared in March 2010. A project review meeting was conducted and a preliminary post-irradiation examination (PIE) plan was drafted. Once a quality-approved process was established, specimen materials were fabricated at INL and inspected in accordance with INL's Quality Plan. They were then sent to an independent laboratory for independent elemental analysis. Flow testing of the irradiation capsule design was performed and compared to computational fluid dynamics (CFD) predictions. Thermophysical properties of the unirradiated material were measured at INL's Idaho Research Center (IRC). The experiment was inserted into ATR in April 2011 and remained in the reactor for four operating cycles. The irradiation campaign was completed in February 2012.

While the specimens were in ATR, Utah State University (USU) performed CFD simulations for a novel hybrid capsule design originally developed for use in this experiment. The results showed that the non-intuitive pressure drop observed in hydraulic flow experiments is due to a complex interaction of vortices and turbulence interactions. Although the hybrid capsule design was deemed too immature for this irradiation campaign, the concept could be considered for future capsule designs. Results from the CFD simulation formed the basis of USU graduate student Adam Zabriskie's master's thesis.

During 2012, mechanical property testing and microstructure evaluation of the unirradiated material were performed at CAES. Microhardness and tensile testing were conducted at room temperature. Local electron atom probe (LEAP) and transmission electron microscopy (TEM) analyses were performed to investigate the material microstructure. Ultra-thin samples were prepared from a

"I am grateful for the opportunity to work with a top researcher from INL and experienced technicians from CAES. This has provided me with hands-on experience on state-of-the-art equipment for the analysis of materials. As a recent graduate, this is an important step toward establishing scientific credibility and providing direction to my career."

Zilong Hua, Post-Doctoral Researcher, Utah State University

larger specimen using a focused ion beam (FIB) technique to capture the phase interface between a Al_3Hf particle and the aluminum matrix. TEM bright-field and high-resolution images show a sharp interface between the two phases. These studies confirmed that the particles did not interact with the matrix during hot pressing to form extra compounds at the interface.

In July 2013, the four USU capsules were disassembled at MFC's Hot Fuel Examination Facility (HFEF). A new tool was designed and fabricated to facilitate removal of the specimens from the capsules (Figure 1).

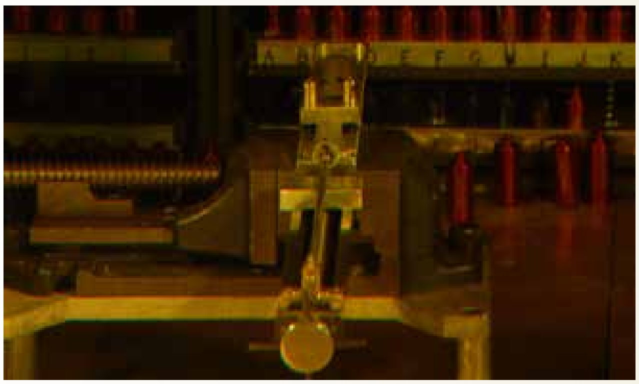

Figure 1. Fixture fabricated to facilitate disassembly of the USU capsules.

Specimens from the B, C, and D capsules were removed without damage. However, five 28.4vol% composite material specimens and two 36.5% composite material tensile specimens were sheared across one end when the capsule was cut open (Figure 2). Two of the 3 mm disks were found to be deformed—one 28.4vol% specimen from capsule A-1 and one 20.0vol% specimen from capsule A-2.

Figure 2. Photograph of tensile specimens sheared during capsule disassembly.

The neutron flux wires were removed from the capsules and shipped to Pacific Northwest National Laboratory (PNNL) for analysis. In the process of resolving apparent inconsistencies in the data, researchers discovered that the capsules had been loaded into the reactor and irradiated in a different configuration than specified in the ITP. Without this extremely valuable information, the PIE data would have been difficult to reconcile. The results of the flux wire analysis are documented in a PNNL report. Supporting calculations were performed by INL staff. Figure 3 shows the fast neutron flux for the actual capsule arrangement.

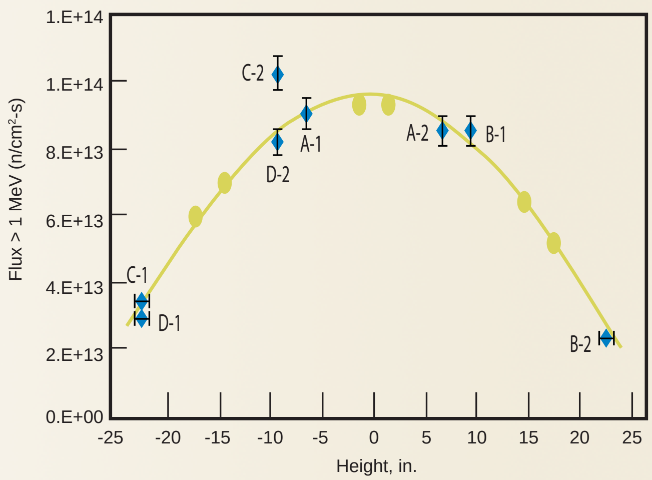

Figure 3. Fast neutron flux for actual capsule arrangement.

A set of nine 5 mm disk specimens and nine 3 mm specimens were sent from HFEF to the Analytical Lab (AL) hot cell, where density and rad measurements as well as gamma scans were completed. Thermal diffusivity measurements were made on unirradiated samples with the scanning differential thermal microscope (SDTM) at IRC to investigate the suitability of applying the measurement to our material. Based on the results, and the unavailability of the SDTM at AL, it was determined that it would be better to use the laser flash dilatometer at the Electron Microscopy Laboratory (EML). The differential scanning calorimeter (DSC) at AL was also unavailable, so the DSC at EML will be used instead.

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Irradiation Effect on Thermophysical Properties of Hafnium-Aluminide Composite: A Concept for Fast Neutron Testing at ATR (cont.)

Scanning electron microscopy was conducted at CAES on unirradiated material to determine the grain size of the matrix and the overall microstructure (Figure 4). Electron backscatter diffraction (EBSD) analysis was performed using a scanning electron microscope (SEM) with an EBSD system (Figure 5).

Figure 4. Mounted SEM samples.

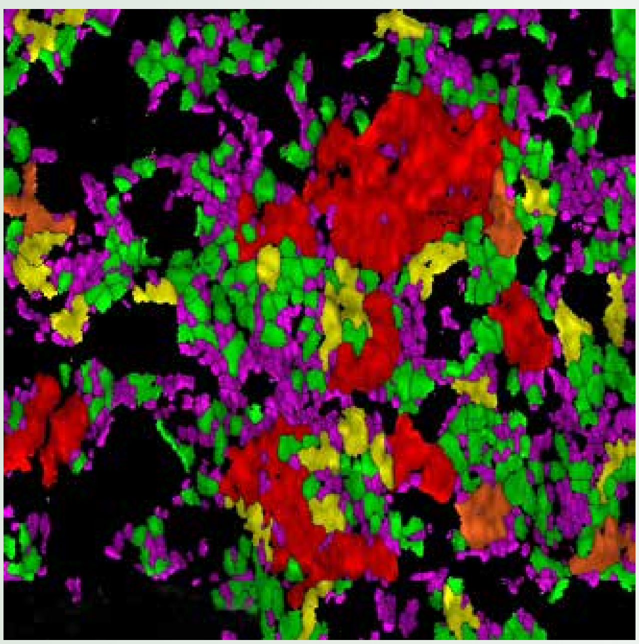

Figure 5. SEM image of 28.4vol% material colored by grain size.

Future Activities

PIE will be performed to assess the effects of irradiation on the material. The specimens are expected to be decontaminated and transferred from AL to EML in early 2014. Laser flash dilatometry and DSC will be performed on 18 irradiated specimens. Radiation-induced changes to the microstructure will be examined using SEM.

USU post-doctoral researcher Zilong Hua will perform three-dimensional (3D) microstructural reconstruction using a sample of unirradiated 28.4vol% material. A 3D field-emission gun (FEG), which is attached to the FIB, will be used to acquire image data for each cross-sectional slice. A set of two-dimensional images will be used to construct a 3D image of the material's microstructure using DREAM3D software. The 3D image will be visualized using ParaView, an open-source, multiplatform data analysis and visualization application.

Publications and Presentations*

Donna P. Guillen and Bryan L. Forsmann, "Microhardness of Hafnium Aluminide Composite Material for Nuclear Reactor Applications," *The Minerals Metals and Materials Society Meeting, San Antonio, Texas, March 3-7, 2013.*

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Pacific Northwest National Laboratory	Materials Science and Technology Laboratory
Collaborators	
Idaho National Laboratory Donna Post Guillen (INL principal investigator), Douglas Porter (materials science), James Parry (neutronics)	
Utah State University Heng Ban (principal investigator), Zilong Hua (post-doctoral researcher), Adam Zabriskie (graduate student), Kurt Harris (undergraduate student), Heather Wampler (undergraduate student)	

Principal Investigator: Michel Barsoum – Drexel University
email: barsoumw@drexel.edu

Advanced Damage-Tolerant Ceramics: Candidates for Nuclear Structural Applications

Introduction

Robust materials are critical to meeting the evolving designs of advanced, next-generation reactors and fuels. These materials must operate in extreme environments of elevated temperatures, corrosive media and high radiation fluences for lifetimes exceeding 60 years. Fully understanding a material's response to irradiation is paramount to ensuring long-term, reliable reactor service.

The layered ternary carbides and nitrides known as MAX phases have the potential to be used in next-generation nuclear reactors. All MAX phases are fully machinable, even though some of them, such as Ti_3AlC_2 and Ti_3SiC_2, are similar in density to titanium metal but are three times as stiff. Their thermal and electrical conductivities are high and metal-like, and they have high fracture toughness values. Some are chemically stable in corrosive environments, and heavy ion studies have shown they also possess a high tolerance to irradiation damage.

Project Description

This project was initiated in 2009 as a collaborative effort of INL's ATR NSUF, Savannah River National Laboratory and Drexel University. Its goal is to characterize the effect of neutron irradiation on select MAX phases—specifically, Ti_3AlC_2, Ti_3SiC_2, and chemical vapor deposited (CVD) silicon carbide (SiC) (for comparison)—in order to investigate the damage inflicted on these materials after exposure to a spectrum of neutron energies consistent with conditions found in light water nuclear reactors (LWR).

The carbides were exposed to neutron fluence levels of 0.1, 1.0, and 10.0 displacements per atom (dpa) at moderate to high irradiation temperatures (100°, 650°, 1000° C) in ATR. Damage to the microstructures and the effects of the radiation on the mechanical and electrical properties of the materials will be characterized during post-irradiation examination (PIE). The results will provide an initial database that can be used to assess the microstructural responses and mechanical performances of these ternaries after neutron irradiation.

	Temperature (°C)	Target Doses* (dpa)	Specimen Types
Ti_3SiC_2	100, 650, 1000	0.1, 1, 10	TEM, resistivity and tensile
Ti_3SiC_2			TEM, resistivity and tensile
SiC (CVD)			TEM, resistivity

* For simplicity use: 7 x 10^{20} n/cm^2 = 1 dpa (E> 1 MeV)

Table 1. Test matrix for sample irradiation.

Accomplishments

All capsules containing the irradiated samples were shipped to INL's PIE facilities at MFC. Receipt, cask unloading, experiment disassembly, and cataloguing of specimens have now commenced.

Upon opening the capsules, researchers observed that in several instances the samples had either fused together or fused to the capsule (Figure 1) making retrieval difficult.

The most troublesome capsules were those held at 100° C for the longest times. It is believed that the materials swelled more than anticipated and exceeded the respective volumes of the capsules. Researchers recovered as many samples as possible, and all samples have been separated and organized into storage vessels.

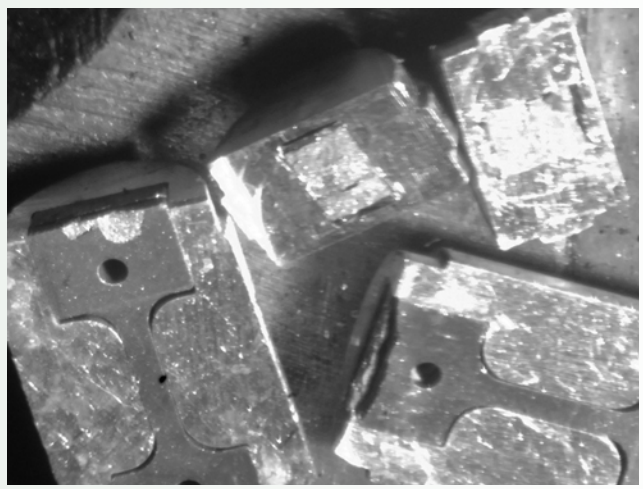

Figure 1. Tensile and transmission electron microscopy (TEM) specimens from Capsule G, irradiated to 1 dpa at 100° C. The specimens are strongly stuck together to the capsule walls. The TEM discs broke in half when the capsule was opened. Attempts to remove these specimens were unsuccessful. The capsule halves are being transported to INL's Electron Microscopy Laboratory (EML).

After opening the capsules, each temperature's 0.1 dpa samples that had already undergone transmission electron microscopy (TEM) were sent to EML for decontamination. In addition, the 1.0 dpa, 100° C resistivity specimens were also submitted to EML. Close examination of the samples at EML revealed that, with the exception of one sample that had an identifying mark on it, they could not be distinguished from one another. Apparently, irradiation had caused all of them to turn the same color.

"The capsules have been opened and catalogued. I am looking forward to coming out to INL to assist with characterization of the neutron-irradiated samples."
Darin Tallman, Research Assistant, Drexel University

Twelve specimens from Capsule F (0.1 dpa at 1000° C) were observed in a scanning electron microscope (SEM) and using electron backscatter diffraction (EBSD) to determine each sample's material type.

Two TEM lamella exposed to 0.1 dpa at 1000° C from each material were prepared using a focused ion beam (FIB). TEM micrographs of Ti_3SiC_2 (Figure 2 and Figure 3), Ti_3AlC_2 (Figure 4) and SiC (not shown) revealed evidence that dislocation loops formed in each specimen. More loops, however, formed in Ti_3SiC_2 than in Ti_3AlC_2. A denuded zone is seen near a grain boundary in Ti_3SiC_2 (Figure 2b).

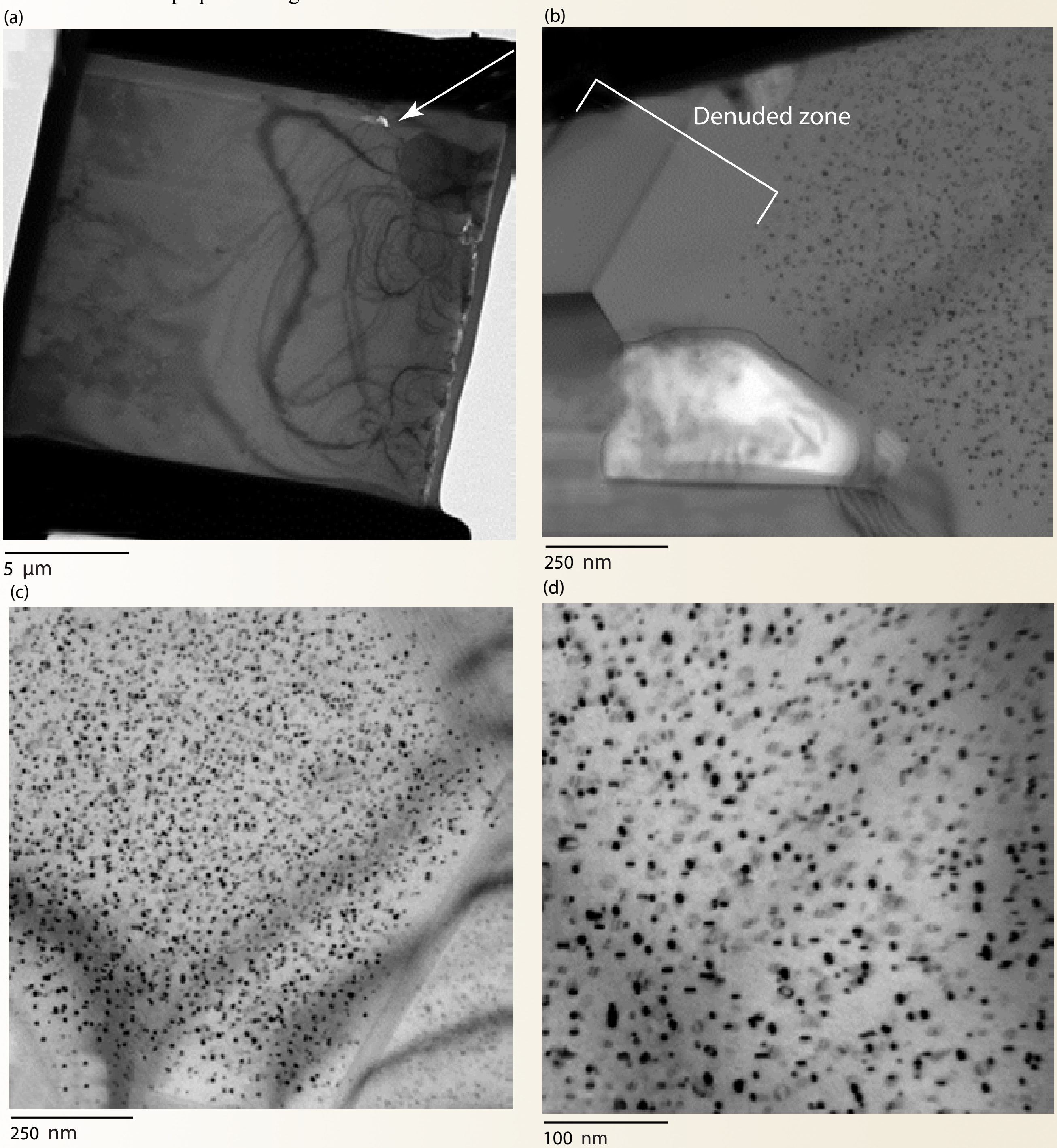

Figure 2. Preliminary TEM micrographs from specimen 1 of Ti_3SiC_2 irradiated to 0.1 dpa at 1000° C, showing: (a) a low magnification image of the entire liftout, the large black swirls are bend contours; (b) a micrograph of the triple-point boundary near the top edge of the sample containing a high density of dislocation loops and a denuded zone near the grain boundary; (c) randomly distributed and oriented dislocation loops from another region of the sample; and (d) a higher magnification micrograph of (c).

Principal Investigator: Michel Barsoum – Drexel University (cont.)
email: barsoumw@drexel.edu

Advanced Damage-Tolerant Ceramics: Candidates for Nuclear Structural Applications (cont.)

This observation notwithstanding, it is important to note that these are preliminary results, and no conclusions can be made at this point. For example, the researchers looked at two Ti_3SiC_2 lamellas. One showed many dislocations (Figure 2), while the other did not (Figure 3).

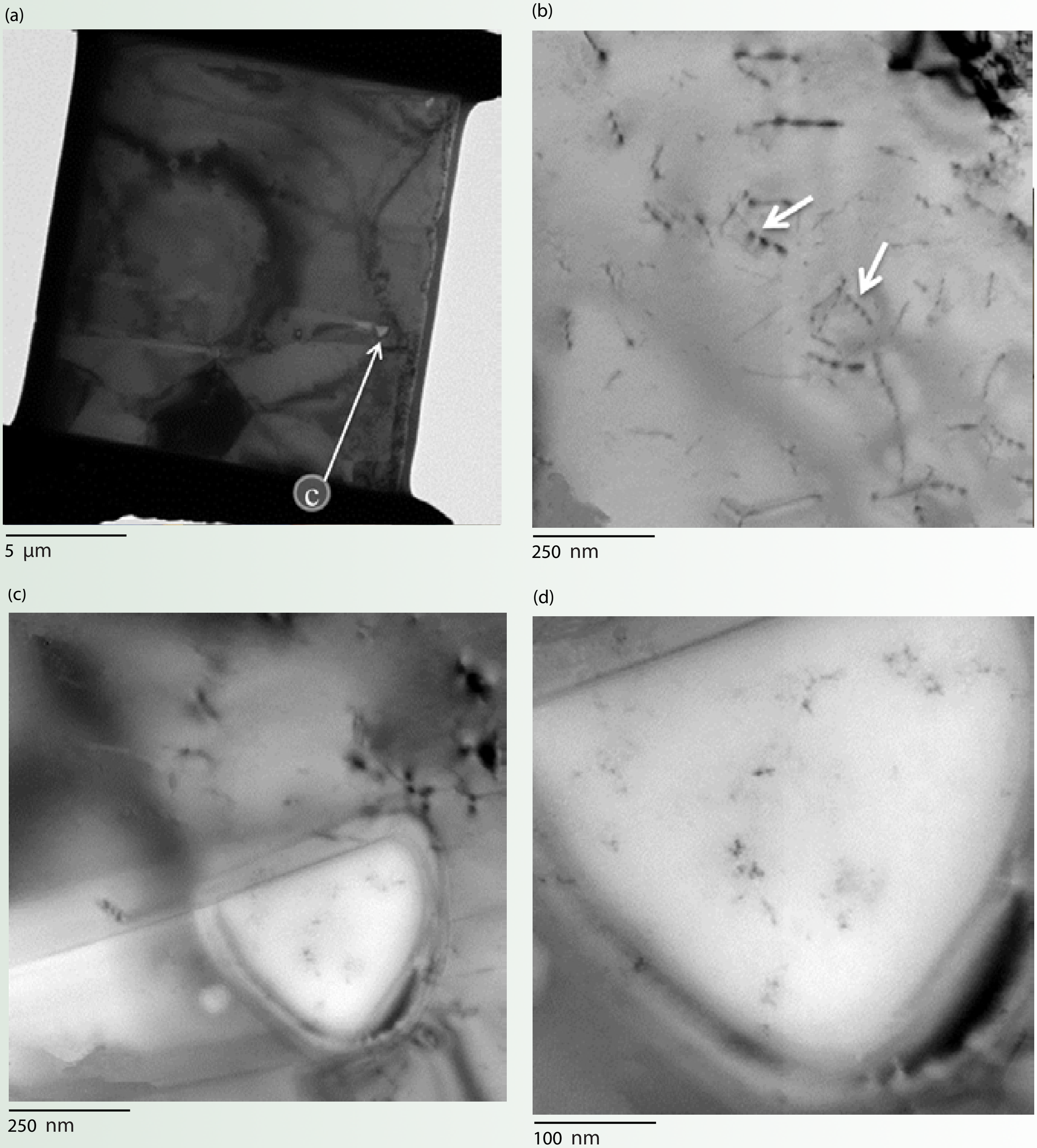

Figure 3. Preliminary TEM micrographs from specimen 2 of Ti_3SiC_2 irradiated to 0.1 dpa at 1000° C, showing: (a) a low-magnification micrograph of the entire liftout; (b) a micrograph showing disorganized dislocation arrays (white arrows) in a grain, likely present in the sample before neutron irradiation; (c) a micrograph from the thin region in the middle of the liftout, revealing connected dislocation arrays, but no loops; and (d) a higher magnification micrograph of (c).

Future Activities

More information is required before researchers can fully characterize the observed dislocation loops. It will be interesting to see how the irradiation-induced dislocation loops interacted with the dislocation arrays that were present in samples before irradiation (Figure 3b and Figures 4c and 4d). Based on the results found thus far in the MAX phase samples that were neutron-irradiated at the Massachusetts Institute of Technology Reactor (MITR),

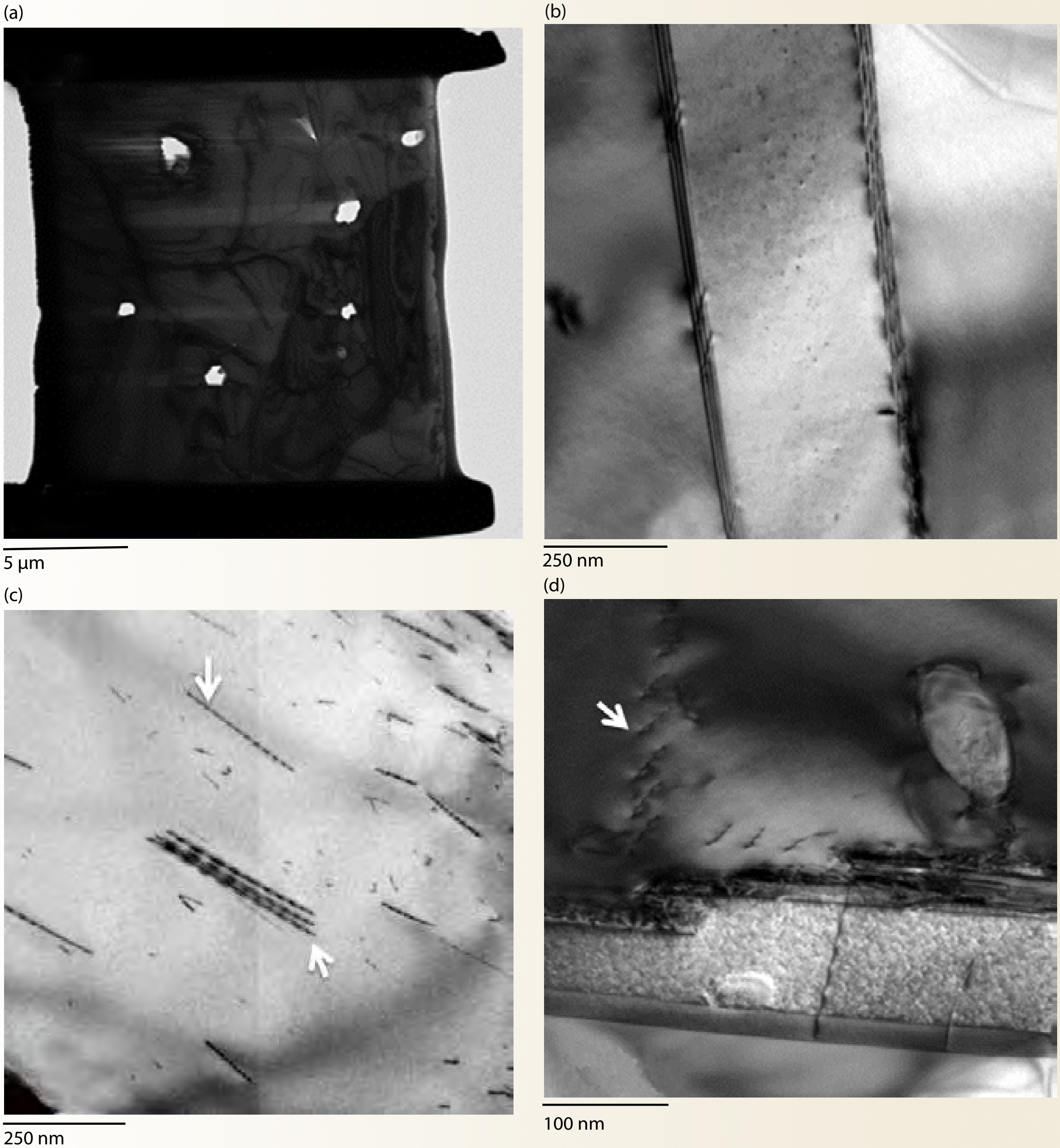

Figure 4. Preliminary TEM micrograph from specimen 1 of Ti_3AlC_2 irradiated to 0.1 dpa at 1000° C showing: (a) a low-magnification image of the entire liftout, revealing many perforations (bright holes); (b) a thin grain with a low density of small dislocation loops in the bulk, with denuded zones at both grain boundaries; (c) a region in specimen 2 where dislocation loops are not resolvable, while dislocation arrays (white arrows) are seen in bulk; and (d) a highly damaged grain in specimen 1 (bottom) with dislocation arrays throughout.

Principal Investigator: Michel Barsoum – Drexel University (cont.)
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Advanced Damage-Tolerant Ceramics: Candidates for Nuclear Structural Applications (cont.)

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

researchers anticipate that most of the irradiation damage in the MAX phases would have rapidly annealed at 1000° C.

INL staff members are arranging researcher visits to perform more in-depth TEM characterization. Resistivity jigs have been designed and are being fabricated, and resistivity measurements of the available resistivity bars are underway. X-ray diffractograms will also be obtained.

Publications and Presentations

Darin Tallman, Elizabeth Hoffman, Robert Sindelar, Gordon Kohse, Michel Barsoum, "The Effect of Neutron Irradiation on MAX Phases," *International Conference and Exposition on Advanced Ceramics and Composites (ICACC), Daytona Beach, Florida, January 31, 2013.*

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Collaborators	
Drexel University Michel Barsoum (principal investigator), Darin Tallman (collaborator) **Idaho National Laboratory** Jian Gan (principal investigator) **Savannah River National Laboratory** Elizabeth Hoffman (collaborator)	

Principal Investigator: Denis Beller – University of Nevada, Las Vegas
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Development and Validation of an Advanced Test Reactor Critical Radiation Transport Model

Introduction

Researchers at the University of Nevada, Las Vegas (UNLV) are developing and validating radiation transport models for the Advanced Test Reactor Critical (ATRC) facility at INL. These models will be used to conduct and evaluate integral and critical benchmark experiments at ATRC.

Project Description

In an earlier phase of the project, a UNLV graduate research assistant modified an existing Monte Carlo N-Particle 5 (MCNP5) model from ATRC using geometric modeling to convert drawings into three-dimensional representations of surfaces, bodies, and cells. The intent was to modify the input file of the ATRC model. The assistant also reviewed the known information, which showed that the materials, components, and configurations in the model were consistent with the actual physical conditions in ATRC. A draft ATRC benchmark evaluation was then submitted to INL for review.

In Phase II of the project, UNLV designed, fabricated, and assembled a UNLV International Critical Safety Benchmark Evaluation Project (ICSBEP) cassette. This was used in a criticality benchmark experiment to validate the ATRC model and demonstrate the ATRC's usefulness in future cross-cutting fuel-cycle research and development and additional criticality benchmark evaluations.

UNLV developed the MCNP model and validation experiments in accordance with guidelines in handbooks from the International Reactor Physics Experiment Evaluation Project and the ICSBEP.

Accomplishments

In late 2012, researchers Denis Beller and Jay Boles of UNLV and Darin Lords, Benjamin Chase, John Bess and Craig Jackson of INL prepared to conduct the benchmark experiment. INL finalized the test plan, conducted criticality modeling and safety analyses and acquired the necessary permissions. In January 2013, UNLV shipped numbered flux wires and the ICSBEP cassette (Figure 1) to INL and provided documentation and reactivity predictions for the UNLV aluminum (Al) bars (see Figure 2 for placement) based on an almost 30-year-old ATRC model.

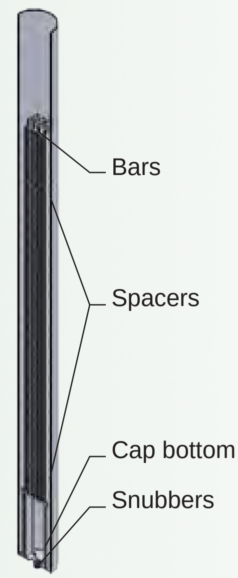

Figure 1. Artist's rendition of a cutaway of the UNLV ICSBEP cassette showing the tube, Al bars and other components.

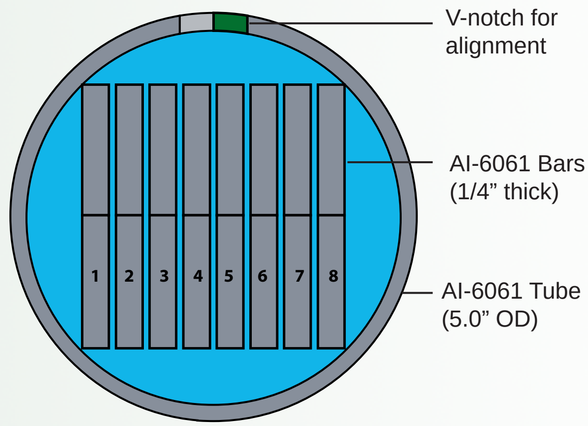

Figure 2. Artist's rendition of a cutaway of the UNLV ICSBEP cassette showing the tube, numbered Al bars and water.

ATRC operators conducted the UNLV criticality benchmark experiment in May 2013. INL subsequently delivered the data to UNLV for analysis. Preliminary examinations revealed substantial differences in the reactivity of each aluminum bar compared to the predictions (Figure 3).

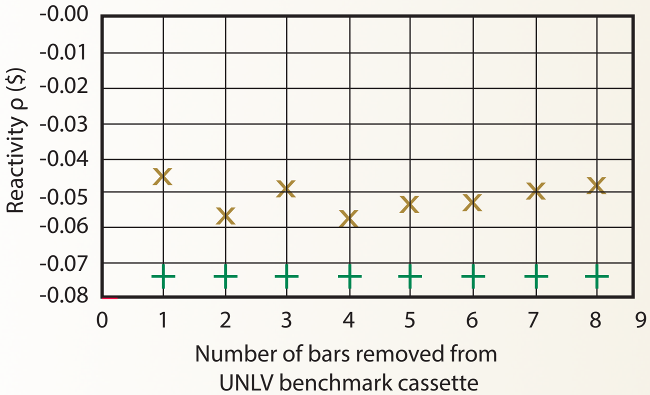

Figure 3. Reactivity versus number of Al bars removed (X). Bars were alternately removed from opposite sides of the stack. The lower symbols (+) represent the average calculated reactivity per bar based on MCNP5 modeling using the ATRC-94 core and a removal sequence progressing from left to right.

If successful, this collaborative project could lead to a new mission for ATRC in support of criticality experiments for a variety of U.S. research, production, and processing efforts.

Future Activities

The project is complete. No additional work will be performed.

Publications and Presentations

1. Denis Beller, John D. Bess and Fred Hua, "Development and Validation of a Nuclear Criticality Benchmark Capability in the Advanced Test Reactor Critical," *Transactions of the ANS,* 109 (2013).
2. Denis Beller, John D. Bess, Darin Lords, Jeremiah Boles, Kimberly Clark, Anthony Santo Domingo, Alex Lui, Jacob Mills, Larry Lakeotes, Craig Jackson, Benjamin Chase, J. Blair Briggs and Fred Hua, "The UNLV ATRC Criticality Benchmark Experiment Project," *Transactions of the ANS,* 109 (2013).

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor Critical facility, PIE facilities
Collaborators	
Idaho National Laboratory Darin Lords (principal investigator), John Bess (collaborator), Benjamin Chase (collaborator), Craig Jackson (collaborator), J. Blair Briggs (collaborator)	
University of Nevada, Las Vegas Denis Beller (principal investigator), Jeremiah (Jay) Boles (graduate student)	

Studying the Role of Alloying Elements on the Microstructure of Nanostructured Ferritic Steels Fabricated via Pulsed Electric Current Sintering

Principal Investigator: Indrajit Charit – University of Idaho
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Introduction

With nanostructured ferritic steels (NFS) gaining importance as fuel cladding materials in advanced reactors, this ATR NSUF research project is expected to result in the discovery of alternative fuel cladding/matrix materials with new steel compositions and fabrication methods.

This research is expected to result in the discovery of alternative fuel/cladding matrix materials with new steel compositions and fabrication methods.

Project Description

NFS, a subcategory of oxide dispersion strengthened (ODS) steels, have outstanding high temperature strength, creep strength [1, 2] and excellent radiation damage resistance [3]. These enhanced properties have been attributed to the high number density of yttrium-titanium-oxygen (Y-Ti-O)-enriched nanoclusters (NCs) with diameters of 1-2 nm [4], which have been found to be stable under irradiation and effective in trapping helium (He) [5]. The nanoclusters are formed by the mechanical alloying of iron-chromium-titanium (Fe-Cr-Ti) powder with Y_2O_3, followed by a hot consolidation route such as hot isostatic pressing (HIP), or hot extrusion [6, 8].

Matthew J. Alinger et al. [4] investigated the effects of alloying elements on the formation mechanism of NCs in NFS processed by HIP, and reported that both Ti and high-milling energy were necessary for the formation of NCs. Michael K. Miller and Chad M. Parrish [9] found that the excellent creep properties in NFS result from the pinning of grain boundaries by a combined effect of solute segregation and precipitation.

Although HIP and hot extrusion are commonly used to consolidate the NFS powder, their anisotropic properties and processing costs are considered challenging. Recently, both pulsed electric current sintering (PECS) and spark plasma sintering (SPS) have been used to sinter the alloy powder at a higher heating rate, lower temperature, and shorter dwell time. This can be done by simultaneously applying uniaxial pressure and direct current pulses to a powder sample contained in a graphite die [10]. Except for a few studies on consolidating simple systems, such as Fe-9-Cr-0.3/0.6Y_2O_3 [11] and Fe-14Cr-0.3Y_2O_3 [10], the SPS process has not been extensively utilized to consolidate NFS with complex compositions. Recently, the role of Ti and Y_2O_3 in processing of Fe-16Cr-3Al-1Tk-0.5Y_2O_3 (wt.%) via mechanical alloying and SPS was investigated by Kerry Allahar et al. [12], and a bimodal grain size distribution was obtained in conjunction with Y-Ti-O-enriched NCs [12, 13].

In this research, an Fe-14Cr (wt.%) alloy was designed as the base, or matrix alloy, and then Ti, lanthanum-oxide (La_2O_3) and molybdenum (Mo) were sequentially added to the ferritic matrix and ball-milled. This approach allowed researchers to study the effect of solutes, both individually and in combination, on the formation of nanoclusters and other microstructural evolutions. SPS was used to consolidate various powder batches. The mixture of Fe-Cr-Ti-Mo powder with Y_2O_3 was also mechanically alloyed and sintered via PECS for comparison.

Accomplishments

Different alloys with systematic variations in compositions were processed using a combined route of high-energy ball-milling and PECS. Microstructural characterization was carried out in the Microscopy and Characterization Suite (MaCS) at CAES using high-resolution transmission electron microscopy (HRTEM) (Figure 1), and atom probe tomography (APT) (Figure 2). MaCS staff, Yaqiao Wu and Jatuporn Burns, were very helpful in training graduate student Somayeh Pasebani and assisted with experiments whenever needed.

Density and hardness test results are illustrated in Figure 3. The Fe-14Cr alloy was found to have high hardness at room temperature due to the strain hardening. The stability of its microstructure at high temperatures was improved by adding La, forming Cr-La-O-enriched nanoclusters as characterized by the APT experiments. Adding La and Ti to the Fe-14Cr matrix significantly improved the mechanical strength and microstructural stability of the 14LMT alloy due to the high number density of Cr-Ti-La-O-enriched nanoclusters.

Future Activities

While the majority of the project is complete, the effects of thermal treatment on the stability of nanoclusters in 14LMT alloy will be investigated in 2014.

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[1] Matthew J. Alinger, G. Robert Odette, Glenn E. Lucas, "Tensile and Fracture Toughness Properties of MA957: Implications to the Development of Nanocomposited Ferritic Alloys," *Journal of Nuclear Materials*, Vol. 307-311 (2002) pp. 484-489.

"I have come to love the rapid turnaround experiment (RTE) mode of proposal submission under the ATR NSUF program. The decision is fast, experiments are targeted, and it provides convenient access to the state-of-the-art facilities and great staff."

Indrajit Charit, Associate Professor, University of Idaho

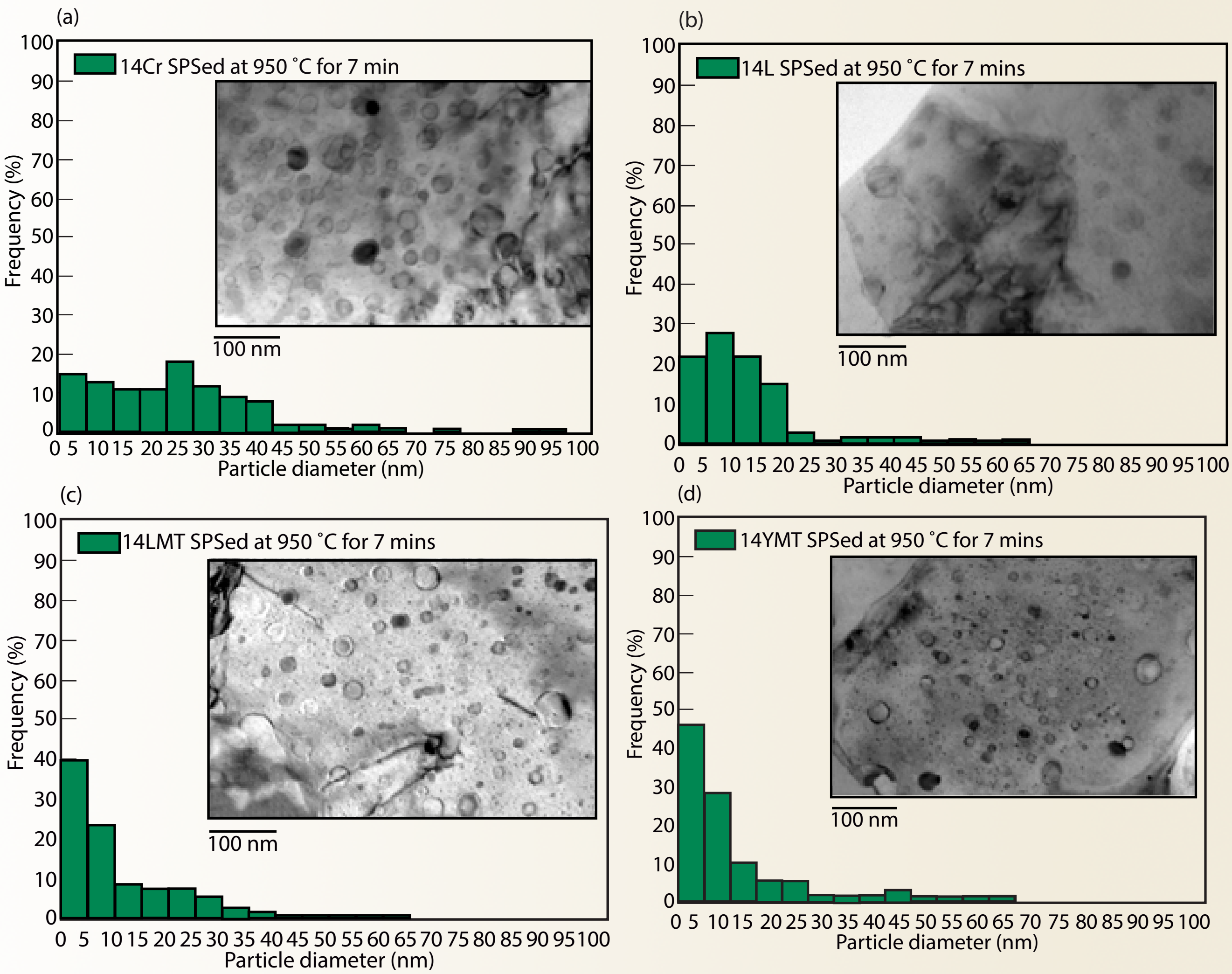

Figure 1. Particle size frequency histogram for (a) 14Cr, (b) 14L, (c) 14LMT, and (d) 14YMT alloys along with corresponding representative TEM micrographs.

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[7] Shigeharu Ukai, Masayuki Fujiwara, "Perspective of ODS Alloys Application in Nuclear Environments," Journal of Nuclear Materials, Vol. 307–311 (2002) pp. 749-757.

Studying the Role of Alloying Elements on the Microstructure of Nanostructured Ferritic Steels Fabricated via Pulsed Electric Current Sintering (cont.)

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email: icharit@uidaho.edu

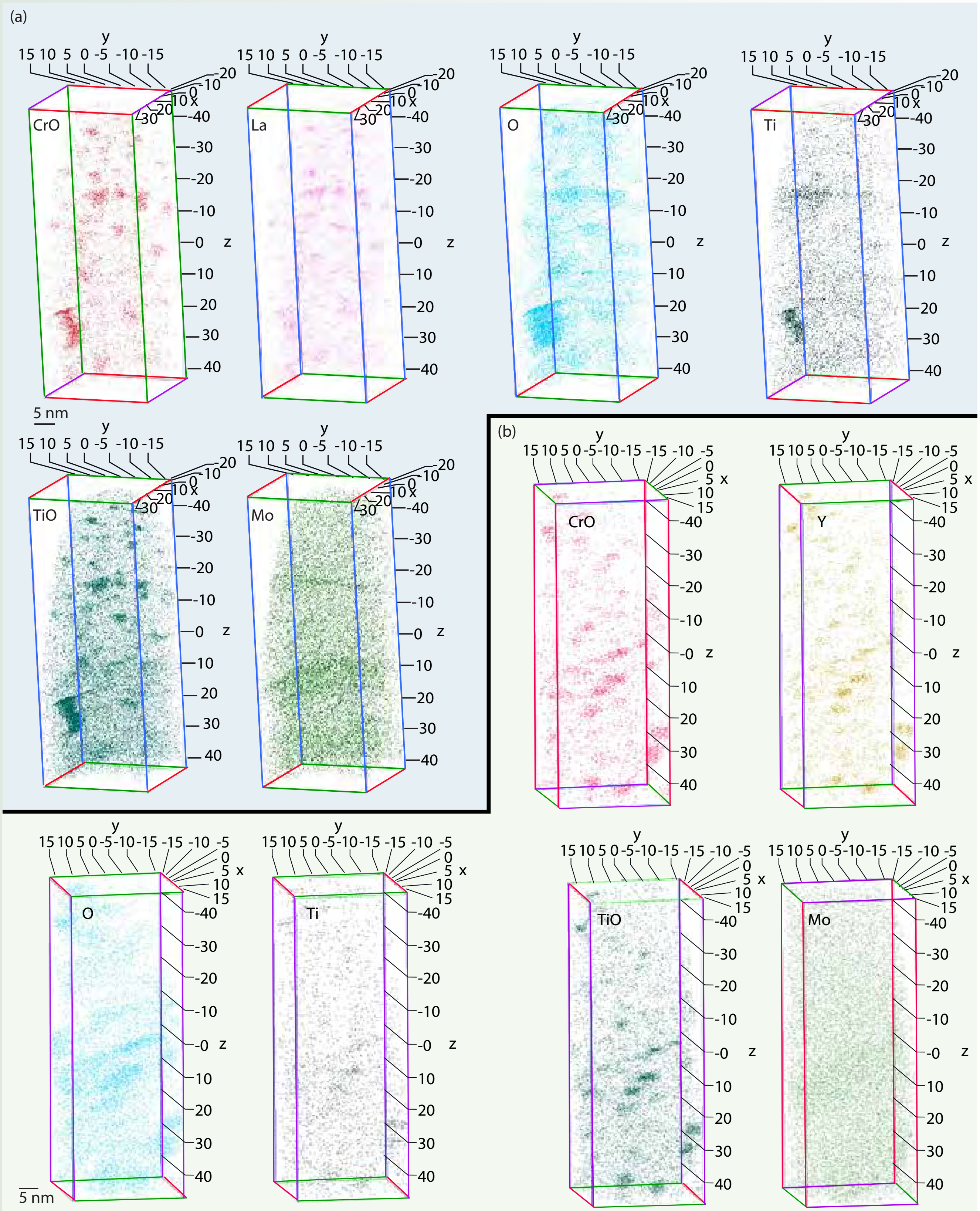

Figure 2. Three-dimensional atom maps showing NCs for (a) 14LMT-91x34x30 nm^3 and (b) 14YMT-93x30x30 nm^3.

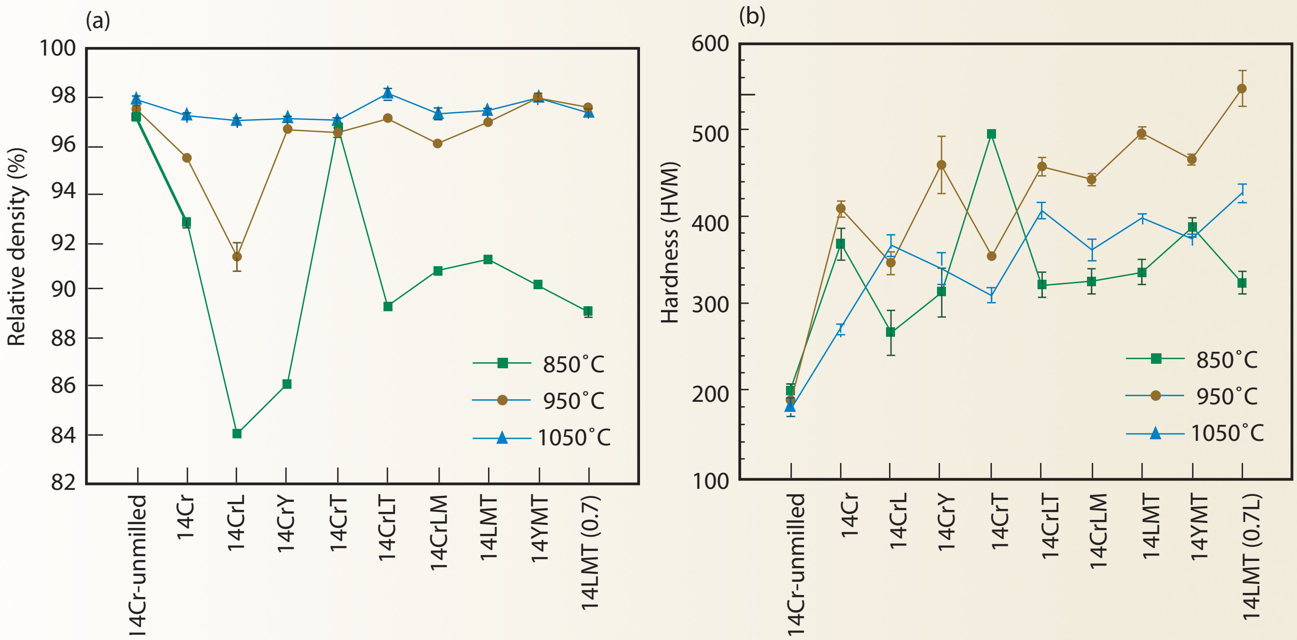

Figure 3. (a) The relative density and (b) microhardness values for different alloys at different PECS temperatures.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Materials and Characterization Suite
Collaborators	
Boise State University Darryl P. Butt (collaborator), Kerry Allahar (collaborator) **Center for Advanced Energy Studies** Yaqio Wu (collaborator), Jatuporn Burns (collaborator) **Idaho National Laboratory** James I. Cole (INL principal investigator) **University of Idaho** Indrajit Charit (principal investigator), Somayeh Pasebani (graduate student)	

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[13] Yaqio Q. Wu, Kerry N. Allahar, Jatuporn Burns, Brian Jaques, Indrajit Charit, Darryl P. Butt, James I. Cole, Fe-Cr-Mo-Based ODS Alloys via Spark Plasma Sintering: A Combinational Characterization Study by TEM and APT Crystal Research and Technology, DOI 10.1002/crat.201300173 (2013) 1-9.

Studying the Role of Alloying Elements on the Microstructure of Nanostructured Ferritic Steels Fabricated via Pulsed Electric Current Sintering (cont.)

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1. Somayeh Pasebani, Indrajit Charit, “Effect of Alloying Constituents on Spark Plasma Sintering of Nanostructured Ferritic Steels,” Submitted to *Journal of Alloys and Compounds*, 2013.
2. Somayeh Pasebani, Indrajit Charit, Darryl P. Butt, James I. Cole, Yaqio Q. Wu, Jatuporn Burns, “Development of Heterogeneous Microstructures in a La2O3-Bearing Nanostructured Ferritic Steel During Spark Plasma Sintering,” Submitted to *Metallurgical and Materials Transactions A,* 2013.
3. Somayeh Pasebani, Indrajit Charit, Darryl P. Butt, Yaqio Q. Wu, James I. Cole, “Mechanical Alloying of Lanthana-Bearing Nanostructured Ferritic Steels,” *Acta Materialia,* Vol. 61 (2013) pp. 5605-5617.
4. Somayeh Pasebani, Indrajit Charit, Darryl P. Butt, James I. Cole, Yaqio Wu, Jatuporn Burns, Kerry N. Allahar, “Processing of a Novel Nanostructured Ferritic Steel via Spark Plasma Sintering and Investigation of its Mechanical and Microstructural Characteristics,” *International Workshop on Structural Materials for Innovative Nuclear Systems, Idaho Falls, Idaho, October 7-9, 2013.*
5. Somayeh Pasebani, Indrajit Charit, Darryl P. Butt, James I. Cole, Yaqio Q. Wu, Jatuporn Burns, “Spark Plasma Sintering of Lanthana-Bearing Nanostructured Ferritic Steels,” *Microscopy and Microanalysis, Indianapolis, Indiana, August 4-8,* 2013.
6. Somayeh Pasebani, Indrajit Charit, Darryl P. Butt, James I. Cole, “Effect of Alloying Elements and PECS/SPS Parameters on Nano-Dispersion Formation in Nanostructured Ferritic Steels,” *The Minerals, Metals and Materials Society, San Antonio, Texas, March 3-7, 2013.*
7. Somayeh Pasebani, Indrajit Charit, Darryl P. Butt, James I. Cole, “Mechanical Properties and Microstructure Characteristics of Nanostructured Ferritic Steel Produced by Spark Plasma Sintering of Mechanically Alloyed Powder,” *Materials Science and Technology 2012, Pittsburg, Pennsylvania, October 7-11, 2012.*

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

Principal Investigator: Mary Lou Dunzik-Gougar – Idaho State University
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Characterization and Correlation of SiC Layer Grain Size/Grain Boundary Orientation with Strength/Layer Growth Conditions

Introduction

Silicon carbide's (SiC) unique combination of thermo-mechanical and physiochemical properties provides a potential opportunity for its use in nuclear applications. One of these applications is as a very thin layer in Tri-Isotropic (TRISO)-coated fuel particles for high-temperature gas reactors (HTGRs). Produced by chemical vapor deposition (CVD), this SiC layer can withstand the pressures of fission and transmutation product gases in a high-temperature environment. Various researchers have demonstrated that macroscopic properties can be affected by changes in the distribution of grain boundary plane orientations and misorientations [1-3]. In addition, various researchers have attributed the release behavior of silver (Ag) through the SiC layer as a grain boundary diffusion phenomenon [4-6], further highlighting the importance of understanding the actual grain characteristics of the SiC layer.

Both historic HTGR fission product release studies and recent INL experiments [7] have shown that the release of ^{110m}Ag is strongly temperature dependent. Although the maximum normal operating fuel temperature range of a HTGR design is 1000° - 1250° C, the temperature may reach 1600° C under postulated accident conditions. The aim of this specific study is, therefore, to expand on initial studies by Van Rooyen *et al.*, [8, 9] so as to determine the magnitude of temperature dependence on SiC grain characteristics.

Project Description

The overarching objective of this research is to contribute to the SiC knowledge base by answering key questions about SiC properties under extreme conditions. Specifically, this work seeks to reveal correlations between coating/annealing parameters and SiC layer grain size, grain boundary orientation and strength. SiC samples previously studied at Nelson Mandela Metropolitan University were made available for these studies, which reduced the project's cost and completion time. The SiC samples were divided into five batches, differing in SiC layer thickness, deposition temperature, and deposition method. The samples have a spherical geometry and have been previously characterized via nano-indentation for compressive strength and hardness. The resulting data also are available for reference as part of the current research.

The specific research work as shown in Figure 1 requires characterization via focused ion beam (FIB) and/or electron backscatter diffraction (EBSD) in the CAES Microscopy and Characterization Suite to determine the grain size and grain boundary orientations of the SiC layers. The goal of this rapid turnaround experiment (RTE) was to analyze two specific samples, the results of which would be added to those from seven samples already characterized. The additional data sets would allow for more statistically significant comparisons. Unfortunately, as is often the case in experimental work, results were not as anticipated. In fact, it was possible to get useful grain size and boundary orientations from only one of the two samples.

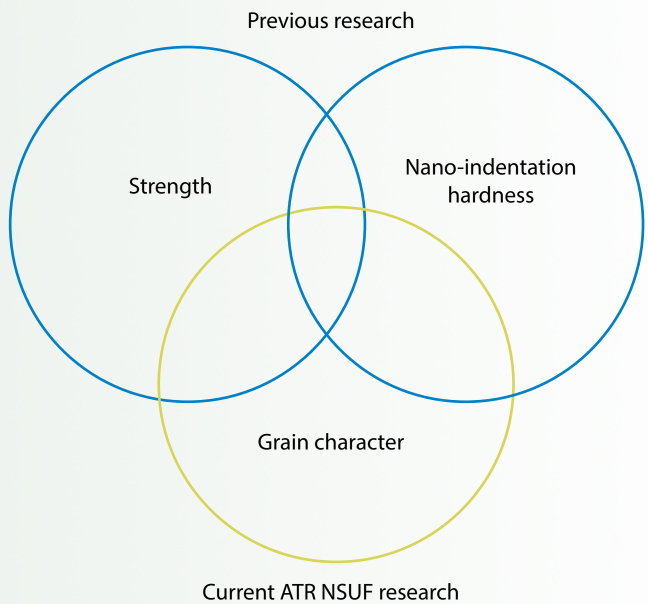

Figure 1. Schematic representation of current ATR NSUF rapid turnaround experiment shown in gold.

In support of the completion of the student researcher's thesis and to make up for the lack of meaningful data from one of the samples, the researchers decided to consider another SiC layer characteristic affected by process temperatures, namely, the crystalline polytypes of the SiC layers. While SiC forms many polytypes, the cubic (3C) structure has been identified as having the most desirable properties for fuel particle coating. Furthermore, the presence of more than one crystal structure in the layer is a source of instability, which may compromise the overall performance. The layer production temperature is a key parameter in controlling the formation of pure 3C SiC and avoiding the presence of hexagonal 6H SiC. 3C SiC obviously was dominant in all the samples that have been analyzed to this point. However, the presence of 6H SiC was neither confirmed nor denied.

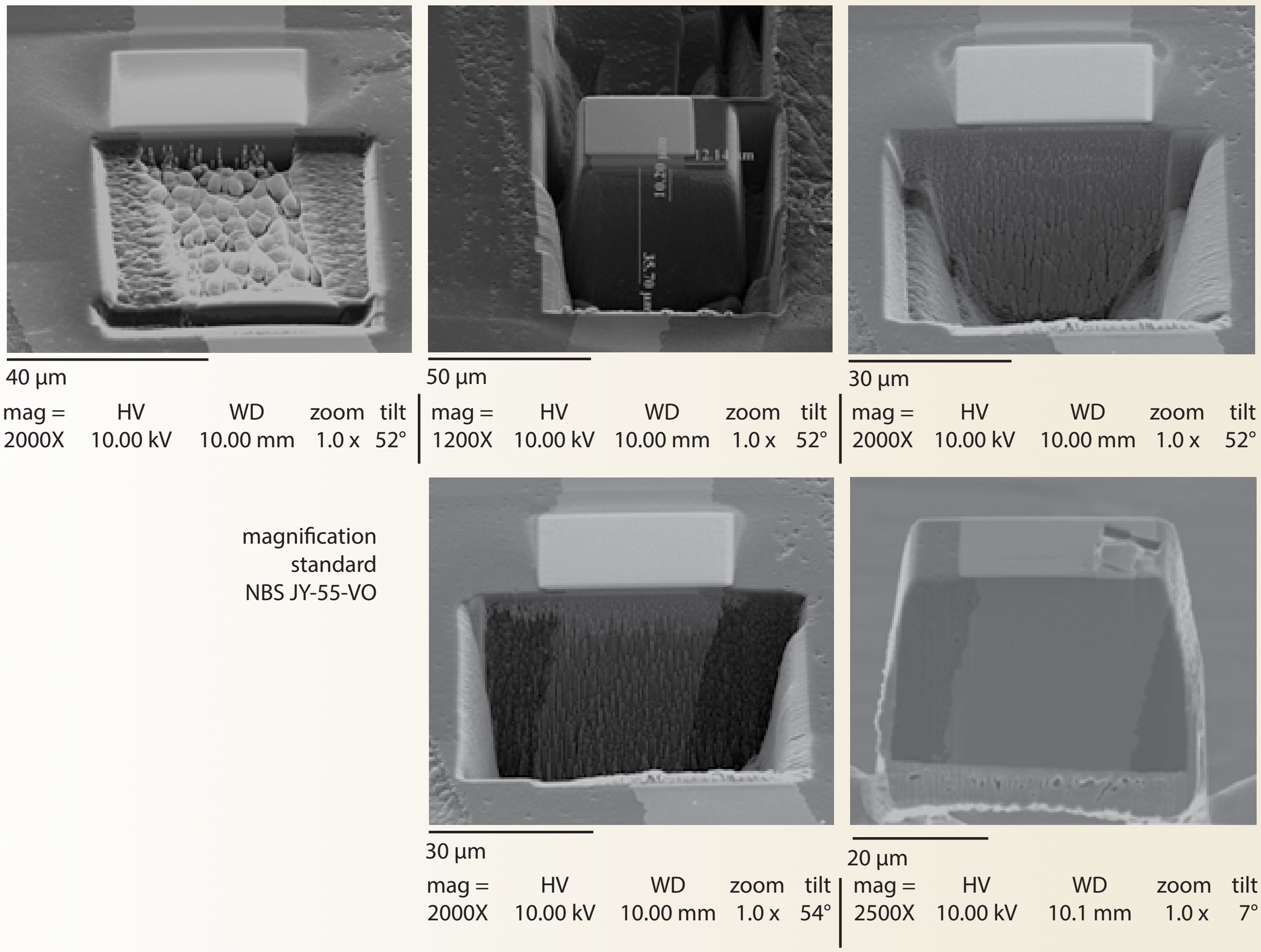

Figure 2. Examples of FIB milling techniques of a TRISO SiC layer annealed at 1600° C for one hour (sample batch B).

Accomplishments

The graduate student working to earn her Master of Science degree gained unique experience in FIB/EBSD sample preparation and characterization. Images exemplifying the FIB SiC layer milling techniques are shown in Figure 2.

In addition, she learned and practiced reduction of raw data for all of the samples analyzed via FIB/EBSD for the parent project. These data supported results reported for van Rooyen's project titled, "Correlating Silicon-Carbide Grain Size and Grain Boundary Orientation with Strength and Silicon-Carbide Layer Growth Conditions." (12-363, see page 147.)

Understanding interface stability under high-temperature irradiation will be a significant step in the development of radiation-resistant materials.

To supplement the originally planned analyses, the student re-analyzed a group of samples for the SiC layer crystalline structure, with particular emphasis on 3C and 6H SiC. Figure 3 provides a comparison of SiC crystal types identified in the SiC layers of two TRISO particle batches. Very little 6H is seen in the layers for either

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Characterization and Correlation of SiC Layer Grain Size/Grain Boundary Orientation with Strength/Layer Growth Conditions (cont.)

pre-annealed or annealed samples; however, the relative presence of 3C is enhanced by annealing (30 minutes at 2000° C) and the relative presence of 6H is decreased. These results indicate that the deposition temperature applied during the production of these particles is effective at producing the 3C SiC polytype with limited 6H and that the annealing process is effective at enhancing the 3C phase while eliminating some of the 6H.

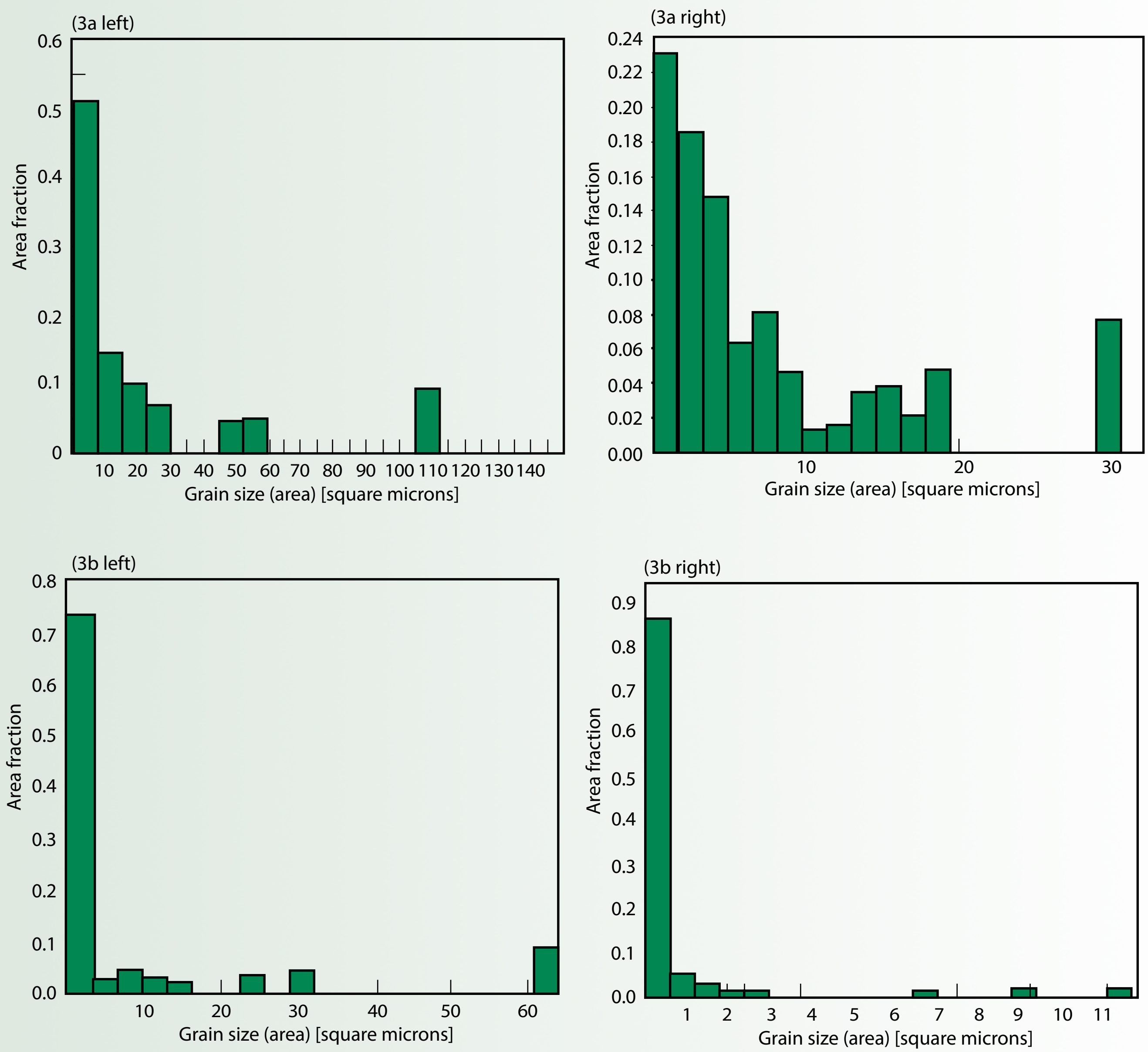

Figure 3. (a) EBSD grain-size-area fraction versus area analysis for SiC layer 3C-polytype for non-annealed (left) and annealed at 2000° C for 30 minutes (right). SiC coating occurred at 1450° C for both. (b) EBSD grain-size-area fraction versus area analysis for SiC layer 6H-polytype for non-annealed (left) and annealed at 2000° C for 30 min (right). SiC coating occurred at 1450° C for both.

Future Activities

In conjunction with sister project RTE 12-363, the goals for this continuing research are:

- Publish a journal paper on the EBSD sample preparation technique.
- Interpret and integrate the results in the larger overarching project with a follow-up journal paper.
- Continue work with the project collaborators to complete the larger project.

Publications and Presentations

Master's thesis in nuclear science and engineering at Idaho State University to be completed for Spring 2014 graduation.

References

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Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies **Idaho National Laboratory**	Microscopy and Characterization Suite Advanced Test Reactor
Collaborators	
Idaho National Laboratory Isabella van Rooyen (co-principal investigator) **Idaho State University** Mary Lou Dunzik-Gougar (principal investigator), Connie Hill (collaborator)	

Principal Investigators: George Imel/Jason Harris – Idaho State University
email: gimel@isu.edu; harrjaso@isu.edu

Real-Time Advanced Test Reactor Critical Flux Sensors

Introduction

Advanced real-time flux sensors are needed for complete characterization of the irradiation environment during irradiation testing.

Project Description

This joint Idaho State University (ISU) / French Alternative Energies and Atomic Energy Commission (CEA) /ATR NSUF project was initiated in 2010 to investigate the feasibility of using neutron sensors to provide online measurements of the neutron flux and fission reaction rates in the ATR Critical Facility (ATRC).

Accomplishments

Several real-time neutron detectors, some with specialized, self-powering capabilities, were developed by industry and research laboratories. Miniature fission chambers were developed by the CEA for measuring fast and thermal flux. Both were tested at ATRC over the three-year period of this project (Figure 1).

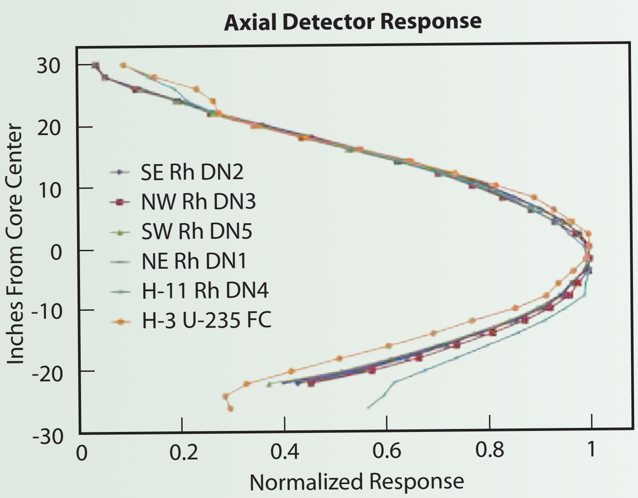

Figure 1. Axial flux profile from flux detectors.

CEA scientists traveled to ATRC in 2010 to assist ISU and INL researchers at the beginning of this testing program. Testing was completed in 2013, and all detectors were tested (Figure 2). Initial results of these tests are useful for a more complete understanding of ATRC power and flux profiles.

Future Activities

The project was completed in 2013, and reports have been published (see publications).

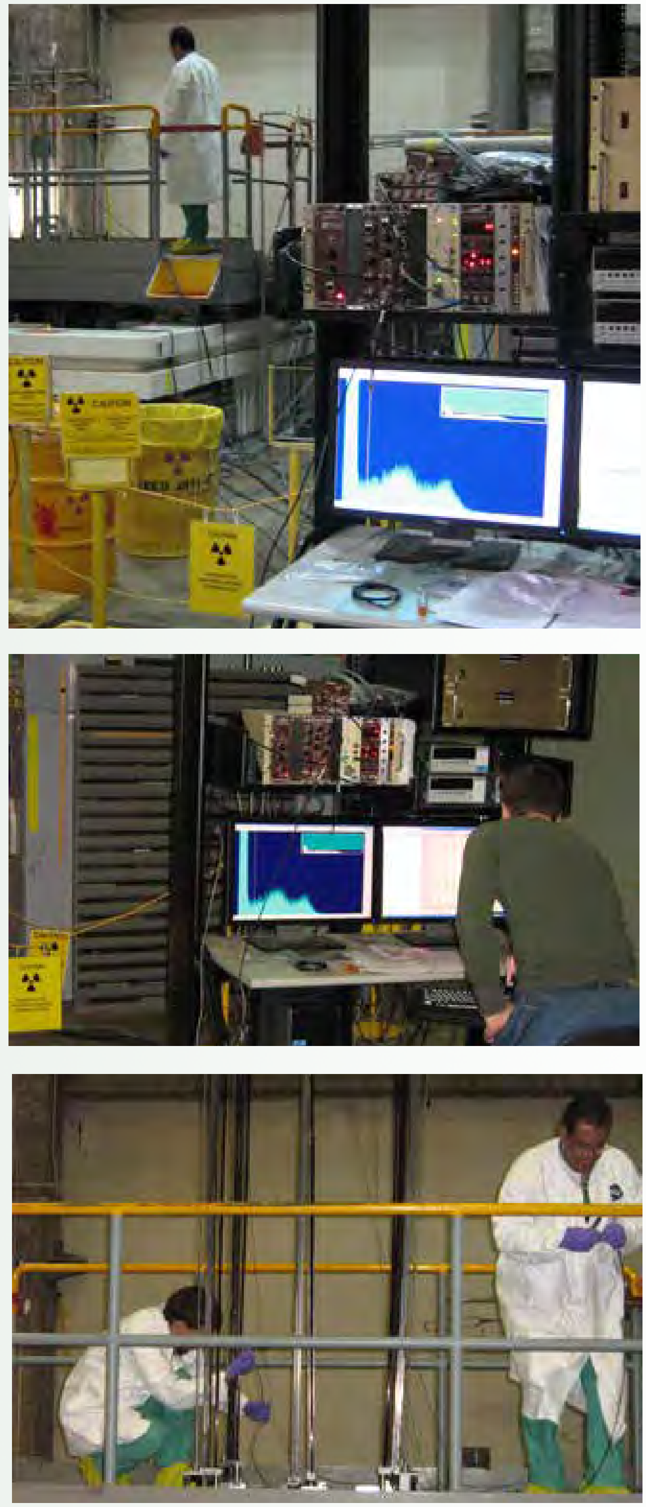

Figure 2. INL/ISU researchers performing flux detector evaluations in ATRC.

"The ATRC capability developed in this project provides researchers from ISU, INL, CEA and other organizations with a unique opportunity for evaluating real-time flux detectors."

George Imel, Idaho State University

Through this ATR NSUF project, several real-time neutron detectors, some with specialized, self-powering capabilities, were developed by industry and research laboratories.

Publications and Presentations*

Troy Unruh, Benjamin Chase, Joy Rempe, *ATRC Neutron Detector Testing Quick Look Report,* INL/EXT-13-29896, August 2013.

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor Critical
Collaborators	
French Alternative Energies and Atomic Energy Commission Jean-Francois Villard (collaborator) **Idaho National Laboratory** Joy Rempe (principal investigator), Troy Unruh (co-principal investigator), Benjamin Chase (co-principal investigator) **Idaho State University** George Imel (principal investigator), Jason Harris (principal investigator), Eric Bonebrake (graduate student), Todd Sherman (graduate student)	

Principal Investigator: George Imel – Idaho State University
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Measurement of Actinide Neutronic Transmutation Rates with Accelerator Mass Spectroscopy (MANTRA)

Introduction

Neutron cross-sections characterize the way neutrons interact with matter. They are essential to most nuclear engineering projects and even though theoretical progress has been made as far as the predictability of neutron cross-section models, measurements are still indispensable to meet tight design requirements for reduced uncertainties. Within the field of fission reactor technology, the following specializations rely on the availability of accurate neutron cross-sections:

- Fission reactor design.
- Nuclear fuel cycles.
- Nuclear safety and safeguards.
- Reactor monitoring and fluence determination.
- Waste disposal and transmutation.

In particular, the assessment of advanced fuel cycles requires an extensive knowledge of actinide cross-sections. Data on plutonium (Pu) isotopes, as well as americium (Am), curium (Cm), and up to californium (Cf) isotopes, are required with a small uncertainty in order to optimize significant features of the fuel cycle that have an impact on feasibility studies (e.g., neutron doses at fuel fabrication, decay heat in a repository etc.).

Project Description

The purpose of this experiment is to irradiate very pure actinide samples in ATR and determine the amount of different transmutation products that develop after a given irradiation time. The determination of the nuclide densities both before and after neutron irradiation will allow for the inference of energy-integrated cross-sections. This information, together with differential measurements of these cross-sections, will ultimately be used in nuclear data evaluations for the Evaluated Nuclear Data File (ENDF), among others.

In order to obtain effective neutron capture cross-sections that correspond to different neutron spectra, ranging from fast to epithermal, three sets of actinide samples will be irradiated. The first one is filtered with cadmium, and the other two are filtered with different thicknesses (5 mm and 10 mm) of boron. Determination of the atom densities before and after irradiation will be carried out using inductively coupled plasma mass spectrometry (ICPMS) at INL, and accelerator mass spectrometry (AMS) in the Argonne Tandem Linac Accelerator System (ATLAS) at Argonne National Laboratory (ANL).

The use of these two independent measurement techniques will benefit the reactor physicists interested in the neutron cross-sections by providing them with two sets of independent measurements. It will also provide the experimentalists in charge of both facilities with a consistent benchmark of their respective techniques.

This experiment will generate enough data for nuclear evaluators to validate their models and provide advanced reactor designers with better neutron cross-sections for years to come.

MANTRA became an official ATR NSUF project in January 2010, with funding from the Department of Energy (DOE) Office of Science, in addition to ATR NSUF.

Accomplishments

Irradiation of the 5 mm boron-filtered (two cycles) and the cadmium-filtered (one cycle) samples at ATR was completed in January 2013. The capsules contained the following highly pure isotopes, all meticulously prepared by INL chemist Jeff Berg: thorium (Th)-232, uranium (U)-233, U-235, U-236, U-238, neptunium (Np)-237, plutonium (Pu)-239, Pu-240, Pu-242, Pu-244, americium (Am)-241, Am-243, curium (Cm)-244, Cm-248, samarium (Sm)-149, europium (Eu)-153, cesium (Cs)-133, and rhodium (Rh)-103.

Under neutron irradiation, these isotopes transmute into other isotopes, and even though the amount of transmutation products at the end of irradiation is relatively small (a few tenths of a percent in the 4 mm boron-filtered samples), it is sufficient to infer the neutron capture cross-section if the measurements are precise enough.

Figure 1. R&D Technician James Sommers checks the test sample results within minutes of starting up the Multi-Collector.

The irradiated samples were transported to MFC in March 2013, where they were disassembled in the Hot Fuel Examination Facility (HFEF). They were then sent to the Analytical Laboratory (AL) for further analyses using the newly acquired Multi-Collector Inductively Coupled Plasma Mass Spectrometer (Multi-Collector), which provides a faster and more accurate method for

"This experiment is unique in that it will provide a consistent set of neutron cross-sections in fast and epithermal neutron spectra for most isotopes of interest to reactor physics."

Gilles Youinou, INL Principal Investigator for MANTRA

determining how much of which elements and isotopes are present in any given sample.

"The Multi-Collector gives us the ability to provide ultra-precise measurements of isotopic ratios in a given sample," said Dr. Jeffrey Giglio, head of AL's research and operations team. "Our researchers can detect almost any element on the periodic table, along with its isotopes, and have the results back in days, or even hours. This instrument gives us a cutting-edge capability that can help advance our mission to support nuclear energy and national security research."

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Collaborators	
Idaho National Laboratory Gilles Youinou (INL principal investigator), Jeff Berg (collaborator), James Sommers (R&D techhician), Tom Maddock (ATR NSUF project manager) **Idaho State University** George Imel (principal investigator)	

Figure 2. A view of all the hardware components of the Multi-Collector.

At the request of ANL collaborators, who are in charge of the AMS measurements, a few actinide samples (Np-237, Am-243, Cm-248, and U-238) were prepared and loaded into the ion source sample holder for the first tests with the laser ablation system. These tests were completed in March 2013, and showed that the system needed some further tuning before it can be used in production mode.

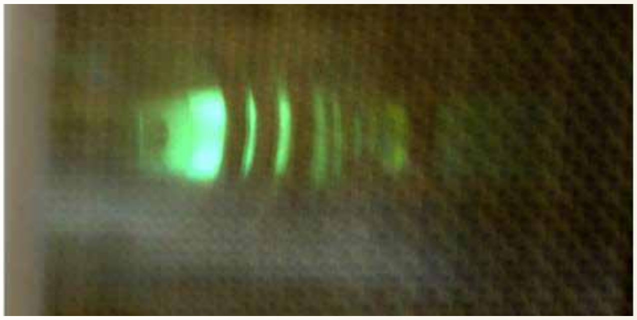

Figure 3. The plasma used to vaporize and ionize test samples for analysis is visible through a small viewport during a sample run.

A report on the status of the experiment was presented at the Nuclear Data for Science and Technology Conference, held in New York City on March 2013 [2], and a journal article was published by the ANL team [1].

"MANTRA has made so much progress thanks in great part to our project manager, Tom Maddock," said Gilles Youinou, INL principal investigator. "His commitment to the success of this experiment has been infallible."

Future Activities

Irradiation of the 10 mm boron-filtered samples is expected to take place at ATR in 2014. Measurements of all the irradiated samples will then be finalized using the Multi-Collector at INL.

Publications and Presentations*

1. Richard Pardo, Filip Kondev, Sergey Kondrashev, Catherine Nair, Tala Palchan, Robert Scott, Richard Vondrasek, Michael Paul, Gilles Youinou, Massimo Salvatores, Giuseppe Palmiotti, Jeff Bert, Jaqueline Fonnesbeck, George Imel, "Toward Laser Ablation Accelerator Mass Spectrometry of Actinides," *Nuclear Instruments and Methods in Physics Research B*, Vol. 294, January 2013, pp. 281-286.

2. Gilles Youinou, Richard Vondrasek, Harmon Veselka, Massimo Salvatores, Michael Paul, Richard Pardo, Giuseppe Palmiotti, Tala Palchan, J. Kumar Nimmagadda, Catherine Nair, Paul Murray, Tom Maddock, Sergey Kondrashev, Filip Kondev, Warren Jones, George Imel, Christopher Glass, Jaqueline Fonnesbeck, Jeff Berg, "MANTRA: An Integral Reactor Physics Experiment to Infer Neutron Capture Cross-Section of Actinides and Fission Products in Fast and Epithermal Spectra," *International Conference on Nuclear Data for Science and Technology, New York City, New York, March 4-8, 2013.*

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

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Using Atom Probe Tomography to Study the Effect of Surfaces on the Chemistry of Depleted Uranium Dioxide

Introduction

This experiment will provide the necessary understanding of the evaporation behavior in uranium dioxide (UO_2) during atom probe tomography (APT) to allow continued future studies into the irradiation-induced chemistry changes in UO_2, specifically at the surface.

Project Description

The objective of this ATR NSUF project study was to gain a fundamental understanding of the science of microstructure in nuclear fuel. Of specific interest was the effect of the dynamic nature of surface chemistry in UO_2. Defects at the fuel's surface cause chemical changes that influence lattice vibrations, which are how the fuel transfers heat—the phenomenon that drives fuel performance.

To increase fuel performance, heat transfer must be improved in current fuel materials such as UO_2, and to improve heat transfer, a better understanding of fuel chemistry dynamics at an atomic level is needed. Experimental data on the role played by changes in surface chemistry on the microstructure of the fuel at this level is currently lacking. APT is uniquely suited to investigate this important aspect of microstructural evolution.

This study proposes that a better understanding of the physics-based description will provide deeper insight into these microstructural changes. Understanding this mechanism will provide a new, fundamental understanding of stoichiometry deterioration in nuclear fuel. Mitigating such behavior can prevent the loss of thermal and mechanical performance in the fuel, thus extending its lifetime and safety in light water reactors (LWRs).

Accomplishments

This study was the first attempt to analyze oxide nuclear fuel materials with APT. Accurate nanoscale chemical analysis requires the optimization of instrument parameters to attain uniform surface evaporation. Due to the dielectric nature of UO_2, a pulsed ultraviolet laser was required to assist the evaporation, which added parameters that also need to be considered for proper analysis.

The effect of sample temperature, ion detection rate and laser energy lead on the observed stoichiometry. Mass resolution and tip uniformity were also studied. Taking all these parameters into account, laser energy had the greatest influence on evaporation behavior (Figure 1). Differences in the laser energy changed the ion evaporation mechanism present in the UO_2. Results

> **Experimental data on the role played by changes in surface chemistry on the microstructure of UO_2 at the atomic level is currently lacking. APT is uniquely suited to investigate this important aspect of microstructural evolution.**

indicated that the optimal run condition of UO_2 for APT is 50 K temperature, 100 kHz repetition rate, 10 pJ laser energy, and 0.3% detection rate.

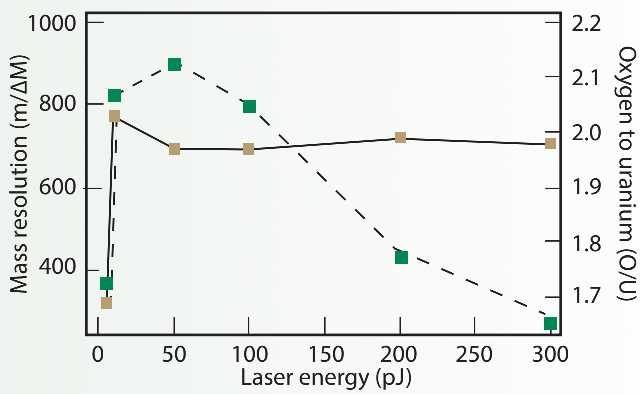

Figure 1. Effect of laser energy on the mass resolution (solid line and squares) and observed oxygen-to-uranium (O:U) ratios (dashed line and green squares) for UO_2.

Defect structures in UO_2 after stoichiometry, in turn influenced thermal conductivity, irradiation resistance and reactivity. Therefore, measuring the true surface stoichiometry is important, and APT provides this capability. Results from the unirradiated single crystal UO_2 used for this study show that the initial surface is inherently hyperstoichiometric. In other words, it has an O:U ratio greater than 2.00 (Figure 2). This indicates that the defect structure at the surface, and consequently the thermal conductivity, will differ compared to the bulk. Results further indicate that not only will thermal conductivity be affected, but also the fuel's reactivity with its surrounding atmosphere, which will also affect future ion irradiation studies.

"It was interesting to understand the role of the UV-laser on the evaporation behavior of UO_2 in laser-assisted atom probe tomography."

Billy Valderrama, Graduate Student, University of Florida

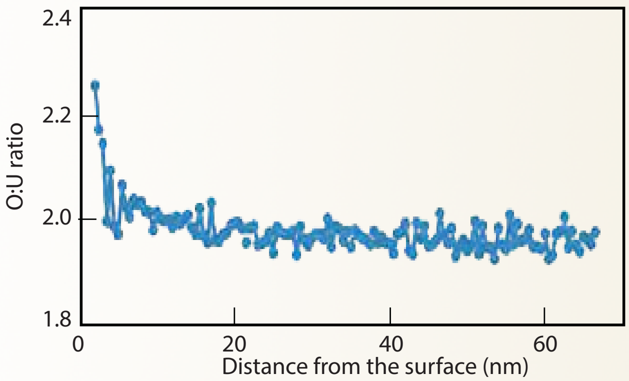

Figure 2. O:U ratio measured by APT of a surface from single-crystal UO_2 indicating hyperstoichiometry at the surface.

Future Activities

Research has been completed.

Publications and Presentations*

Billy Valderrama, Hunter B. Henderson, Jian Gan, Michele V. Manuel, "The Influence of Instrument Conditions on the Evaporation Behavior of Uranium Dioxide with UV-laser Assisted Atom Probe Tomography," Ultramiscroscopy (2013) (under review).

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
University of Florida Michele V. Manual (principal investigator), Billy Valderrama (collaborator), Hunter Henderson (collaborator)	

Principal Investigator: Michele Manuel – University of Florida
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Using Atom Probe Tomography to Study Helium Segregation and Bubble Formation in Uranium Dioxide

Introduction

Irradiation of oxide fuels, specifically uranium dioxide (UO_2), leads to the formation of insoluble fission products, such as helium (He), which alter the paths traveled by photons, thereby reducing the fuel's thermal transport behavior. These fission products tend to cluster intragranularly as the irradiation dose increases, swelling the fuel and degrading its overall performance. This ATR NSUF project represents the first atomic-level investigation that indicates a change in chemistry is present at the grain boundaries of UO_2, which presents a possible mechanism for enhancing the driving force and accommodation of fission products.

Project Description

Researchers investigated the evolution and segregation of He in UO_2 with atom probe tomography (APT). APT is uniquely suited to the project because of its nanometer-scale spatial resolution and chemical identification capabilities, as well as its ability to investigate the growth characteristics of the He clusters and their chemical composition. Changes in UO_2 near the clusters were also investigated. The results are expected to provide new insight into the spatial and chemical distribution of fission product clusters in UO_2, which could open doors for correlation with atomic-level simulations, and the ability to link with mesoscale structure-property relationships.

He-doped polycrystalline UO_2 was chosen for this project because of its documented role in fission product damage—namely fission gas release and void formation. Researchers believe that a physics-based description will provide deeper insight into the microstructural changes that occur in nuclear fuel under irradiation, specifically the migration of fission products to the grains and the formation of solute clusters. Understanding these mechanisms will provide a new fundamental understanding of void formation and grain boundary deterioration in nuclear fuel. Mitigating these behaviors can prevent the loss of thermal and mechanical performance in the fuel, thus extending its lifetime in light water reactors (LWRs).

Accomplishments

Fission products in UO_2 tend to cluster and then segregate toward interfaces such as grain boundaries. Specific grain boundary types in UO_2 can accommodate fission products such as helium (He), xenon (Xe) or krypton (Kr) more favorably due to differences in misfit orientation. In this investigation, He-irradiated UO_2 was used to study the degrees of variation in oxide chemistry at specific types of grain boundaries. Electron backscatter diffraction (EBSD) was used to map grain orientations and grain boundary types in the sample, and a focused ion beam (FIB) liftout of a high-angle grain boundary was used to make an APT tip (Figure 1). The results showed oxygen depletion at the high-angle grain boundary, indicating that it is likely to accommodate fission products.

This project represents the first atomic-level investigation that indicates a change of chemistry is present in the grain boundaries of UO_2, which presents a possible mechanism for enhancing the driving force and accommodation of fission products.

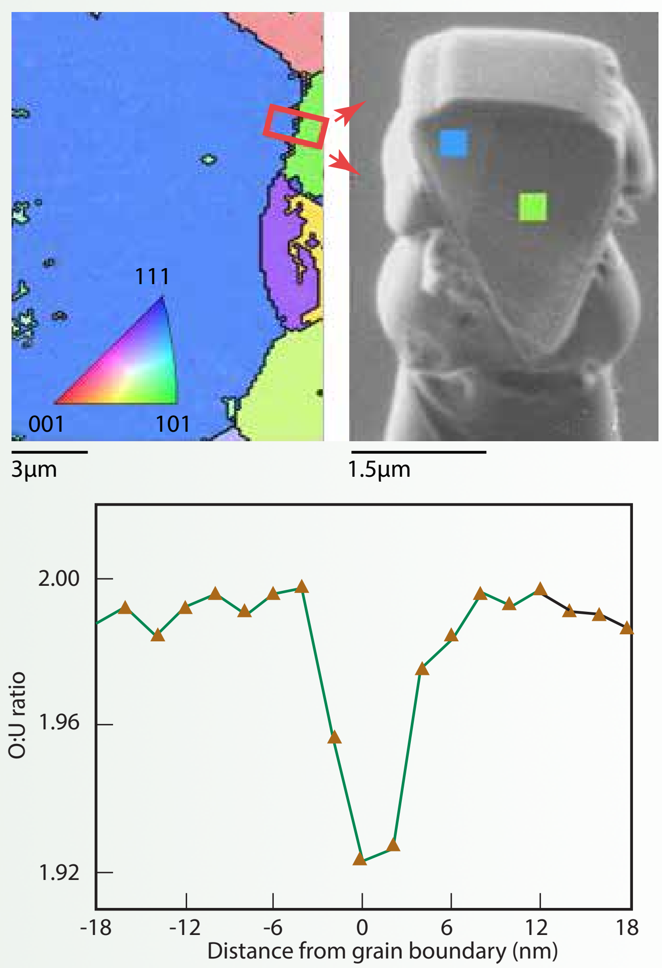

Figure 1. O:U ratio measured by APT across a high angle grain boundary in He-irradiated UO_2 for the selected region of interest from the EBSD map. Preliminary results indicate oxygen depletion at the grain boundaries, which can serve as fission product trapping sites.

"This experiment will help elucidate the role of grain-boundary chemistry on the segregation behavior of fission products in oxide fuel materials."

Billy Valderrama, Graduate Student, University of Florida

Some difficulties were encountered in these experiments. The irradiation conditions were expected to only produce point defects. The He-UO_2 samples were not implanted with enough He atoms to allow for clustering of the simulated fission product, therefore only limited clustering presented. In addition, the surfaces of the samples were not prepared well enough for successful EBSD mapping. To mitigate this issue, extensive FIB time was used to make large cube liftouts of the samples and ion milling was used to polish the surfaces for better EBSD mapping. These new samples are currently being irradiated under more appropriate conditions.

Future Activities

With a procedure now available for fabricating atom probe samples that include grain boundaries, researchers expect to accomplish the goals originally set forth in this proposal during 2014.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Idaho National Laboratory Jian Gan (collaborator) **University of Florida** Michele V. Manual (principal investigator), Billy Valderrama (collaborator), Hunter Henderson (collaborator)	

Principal Investigator: Michele Manuel – University of Florida
email: mmanuel@mse.ufl.edu

Using Atom Probe Tomography to Study the Annealing Temperature Dependence on Krypton Bubble

Introduction

During the irradiation process, insoluble fission products such as xenon (Xe) and krypton (Kr) are produced in oxide fuels, specifically uranium dioxide (UO_2). These products tend to cluster, causing the fuel to swell. This leads to changes in the thermal transport properties of the fuel, reducing its overall performance. To better understand the role played by fission product clusters, more accurate chemical information is needed about their distribution and the influence of annealing temperature on their size.

Project Description

A detailed atomic-level study was proposed to address a critical need for material-physics relationships under the Light Water Reactor Sustainability Program. These experiments focused on the influence of annealing temperature on the evolution of fission product bubbles and their chemical composition. Atom probe tomography (APT) was used to characterize the effect of fission damage processes in nuclear fuel.

The materials under investigation were Kr-doped polycrystalline UO_2. Kr was chosen because of its documented role in fission product damage—namely, fission gas release, void formation, and grain boundary modification. A physics-based description will provide deeper insight into the microstructural changes that occur in nuclear fuel under irradiation. Understanding these mechanisms will provide a new fundamental understanding of void formation and grain boundary deterioration in nuclear fuel. Mitigating these behaviors can prevent the loss of thermal and mechanical performance of the fuel, extending its lifetime in light water reactors (LWRs).

Accomplishments

In order to understand the early stages of fission gas bubble formation in UO_2, both microstructural and chemical changes brought on by irradiation need to be understood. APT offers the unique ability to observe chemical changes in both the fuel material and the fission products. Investigations into the distribution of Kr in UO_2 were successfully compared to the transport of ions in matter (TRIM) simulations (Figure 1).

Atom probe tomography provided a unique opportunity to experimentally study fission product segregation in UO_2 nuclear fuels.

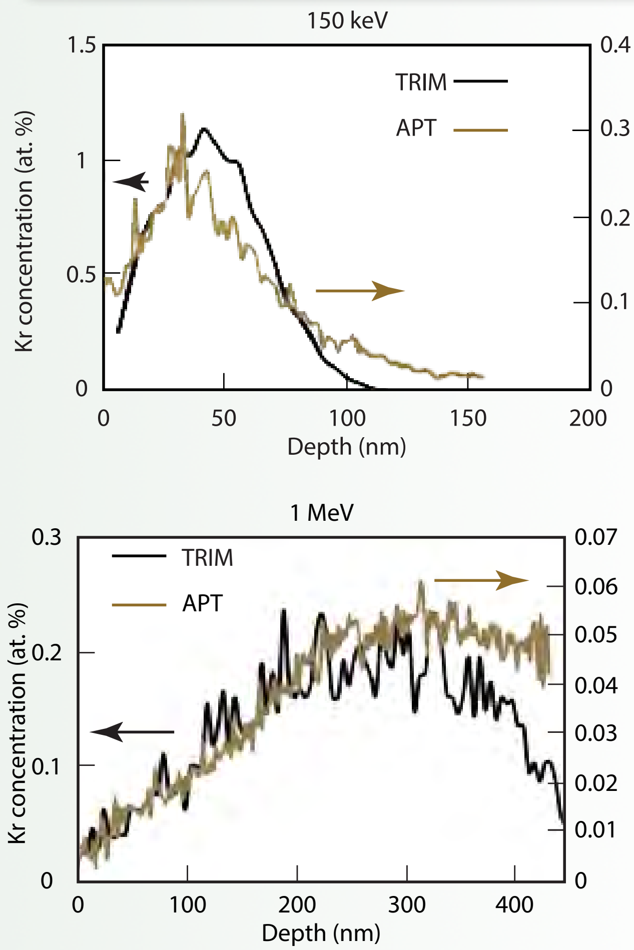

Figure 1. Comparison of Kr distribution as measured with APT to TRIM simulation for two implanted energies of 150 KeV and 1 MeV.

"These experiments provided a new avenue for investigating fission product distributions in oxide fuel materials."
Billy Valderrama, Graduate Student, University of Florida

The amount of Kr detected by the APT was much lower than that in TRIM. This drop in concentration can be attributed to a loss of gaseous Kr when the bubbles were exposed to vacuum in the atom probe instrument. The Kr detected is considered to be the amount that did not migrate toward the bubbles, but was left behind in the defect sites created during irradiation.

In the samples investigated, the Kr bubbles had an expected diameter of 2 nm, as measured by transmission electron microscopy (TEM). Unfortunately, these bubbles were too small to be observed by APT. Future investigations will include samples containing bubbles with diameters of 10 nm or greater, which will be detected more easily with APT.

Future Activities

Research was completed in 2013.

Publications and Presentations*

Billy Valderrama, Hunter B. Henderson, Lingfeng He, Clarissa Yablinsky, Jian Gan, Abdel-Raman Hassan, Anter El-Azab, Todd R. Allen, Michele V. Manuel, "Fission Products in Nuclear Fuel: Comparison of Simulated Distribution with Correlative Characterization Techniques," Microscopy and Microanalysis 2013. Indianapolis, Indiana, August 4-8, 2013.

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Idaho National Laboratory Jian Gan (collaborator) **University of Florida** Michele V. Manual (principal investigator), Billy Valderrama (collaborator), Hunter Henderson (collaborator)	

Principal Investigator: Emmanuelle Marquis – University of Michigan
email: emarq@umich.edu

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

Introduction

Radiation-induced segregation/radiation-induced depletion (RIS/RID) and its deleterious impact on properties in austenitic stainless steels have been studied extensively, particularly at high-dose rates. However, the validation of life-extension plans for light water reactors and future applications in advanced fission and fusion reactors will require additional data on the effects of high fluence at low-dose rates on the microstructural and mechanical properties of these materials.

Project Description

The objective of this study is to characterize the microstructural changes in hex blocks irradiated in the Experimental Breeder Reactor (EBR)-II at INL. The results will complement the limited amount of existing data on stainless steels irradiated at low dose rates, provide new methods of grain boundary analysis, offer further insights into the mechanisms of radiation-induced segregation and depletion in these complex systems, and support predictions of materials degradation in current fission plants.

Accomplishments

Once INL's Bulent Sencer ensured that the right specimens were available and properly prepared, University of Michigan student Yan Dong was able to collect initial data on irradiated hex block 304 steel using focused ion beam (FIB) technology, transmission electron microscopy (TEM), and atom probe tomography (APT) during her last visit to CAES. Her observations showed the presence of voids (Figure 1), nickel (Ni)- and silicon (Si)-rich precipitates, chromium (Cr) carbides with Ni and Si segregations at the interface (Figure 2), and a dense network of Ni- and Si-decorated dislocations. Larger precipitates, most likely carbides, with two main morphologies (plates and 100 nm spheres) cover most of the large-angle grain boundaries.

The results of this experiment will complement the limited amount of existing data on stainless steels irradiated at low dose rates.

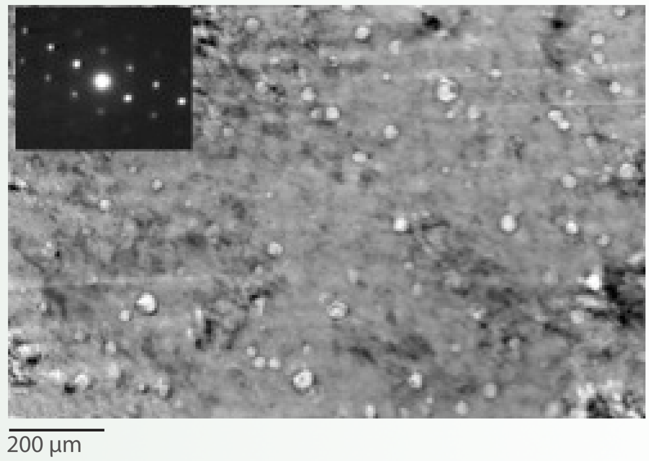

Figure 1. TEM bright field image taken near the <011> zone axis reveals the presence of faceted voids.

Future Activities

The data obtained are still preliminary and additional experiments are planned for 2014. Using specimens available through CAES, additional APT/TEM observations will focus on grain boundary analysis. The work will compare the results obtained through the two different techniques to try and establish that the formation of grain boundary precipitates and RIS may be dependent on grain boundary orientation. Comparing these results with observations on the unirradiated alloy will clarify whether the precipitates are induced by irradiation. Electron diffraction in the TEM will be used to obtain the crystal structures of the different types of precipitates, complementing the chemical information already acquired by APT.

"This research will provide new insights into the microstructural development of 304 steel and impact modeling of the long-term irradiation evolution."

Emmanuelle Marquis, Assistant Professor, University of Michigan

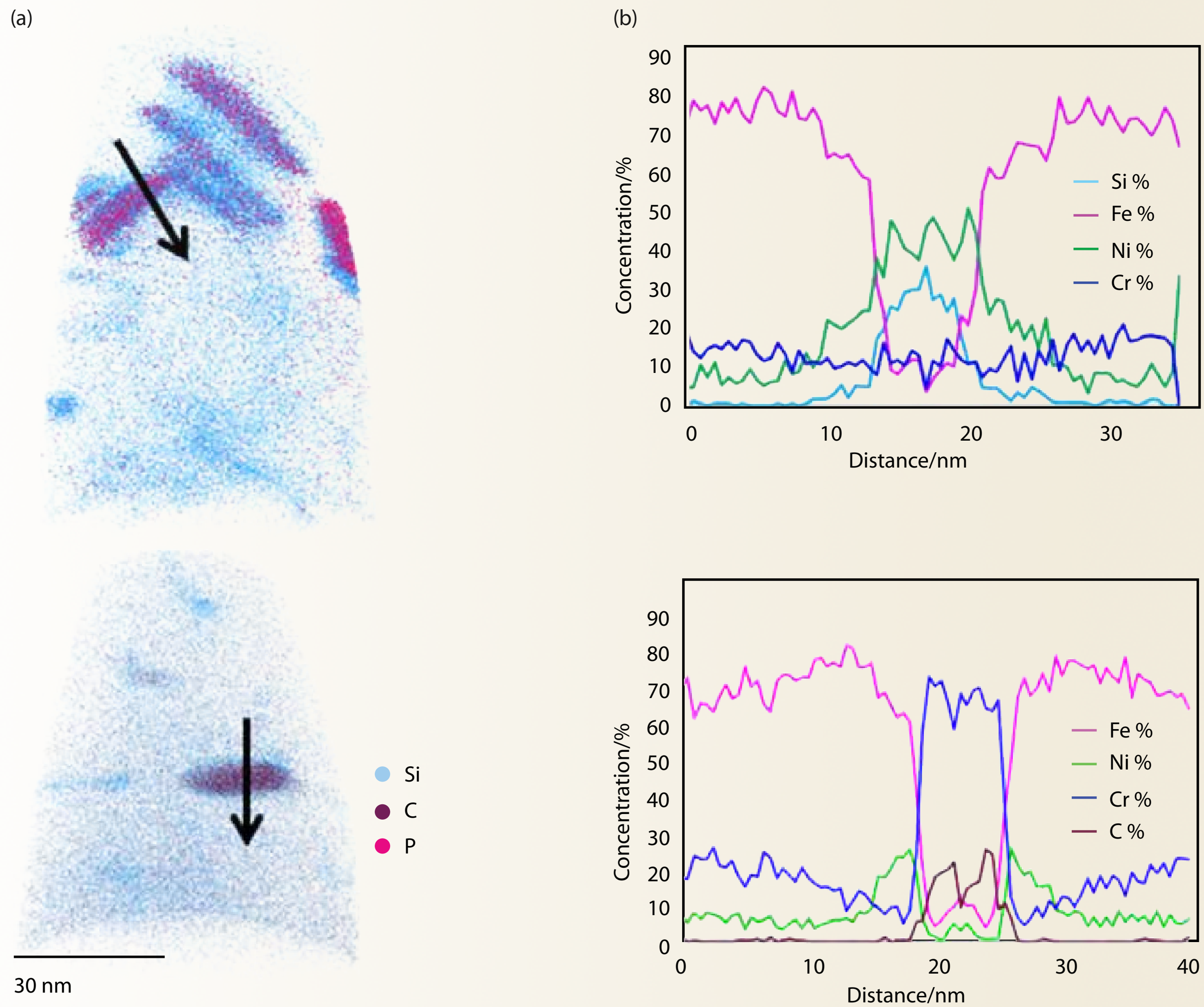

Figure 2. (a) APT reconstructions and concentration profiles showing carbides and Ni/Si-rich precipitates smaller than 30 nm in the matrix. (b) Dislocation lines/loops with Ni and Si enrichment.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Idaho National Laboratory Bulent Sencer (INL principal investigator) **University of Michigan** Emmanuelle Marquis (principal investigator), Yan Dong (graduate student)	

Effects of Neutron Irradiation on the Mechanical and Microstructural Properties of Equal Channel Angular Pressing Steel

Principal Investigator: K. L. Murty – North Carolina State University
email: murty@ncsu.edu

Introduction

This project is a collaborative effort between ATR NSUF and North Carolina State University (NCSU). Its goal is to understand the effect of grain size on the irradiation tolerance of certain materials.

Project Description

The project uses ultra-fine grained (UFG) plain carbon steel produced using equal channel angular pressing (ECAP) and the corresponding conventional grain (CG) steel to characterize the effect of neutron irradiation. The UFG, low-carbon steel used in this study has a wt% composition of 0.1% carbon (C), 0.27% silicon (Si) and 0.5% manganese (Mn). It was processed using four passes through an ECAP die whose channels intersect at 90°. In turn, the CG steel samples were produced by annealing the UFG samples to 800º C. Figure 1 shows the electron backscatter diffraction (EBSD) images for both materials prior to irradiation.

Accomplishments

Pre-irradiation studies have been completed at both NCSU and CAES. Microhardness tests, tensile tests, X-ray diffraction (XRD) and scanning electron microscopy (SEM) were performed at the NCSU Nuclear Materials Laboratory, while the EBSD and the transmission electron microscopy (TEM) were conducted at CAES.

The samples were irradiated to 0.001 displacements per atom (dpa) in the PULSTAR reactor at NCSU, and to 1 dpa and 2 dpa in ATR at INL. The effects of neutron irradiation on both the UFG and CG carbon steels were investigated by exposing the materials to high-energy neutrons in the PULSTAR reactor at 1 MW for 200 hours. Figure 2 shows the materials' irradiation position inside the PULSTAR reactor.

Preliminary results indicate that the ECAP UFG steel is more radiation-tolerant than CG steel, meaning UFG steel is a promising new structural material for future reactor applications.

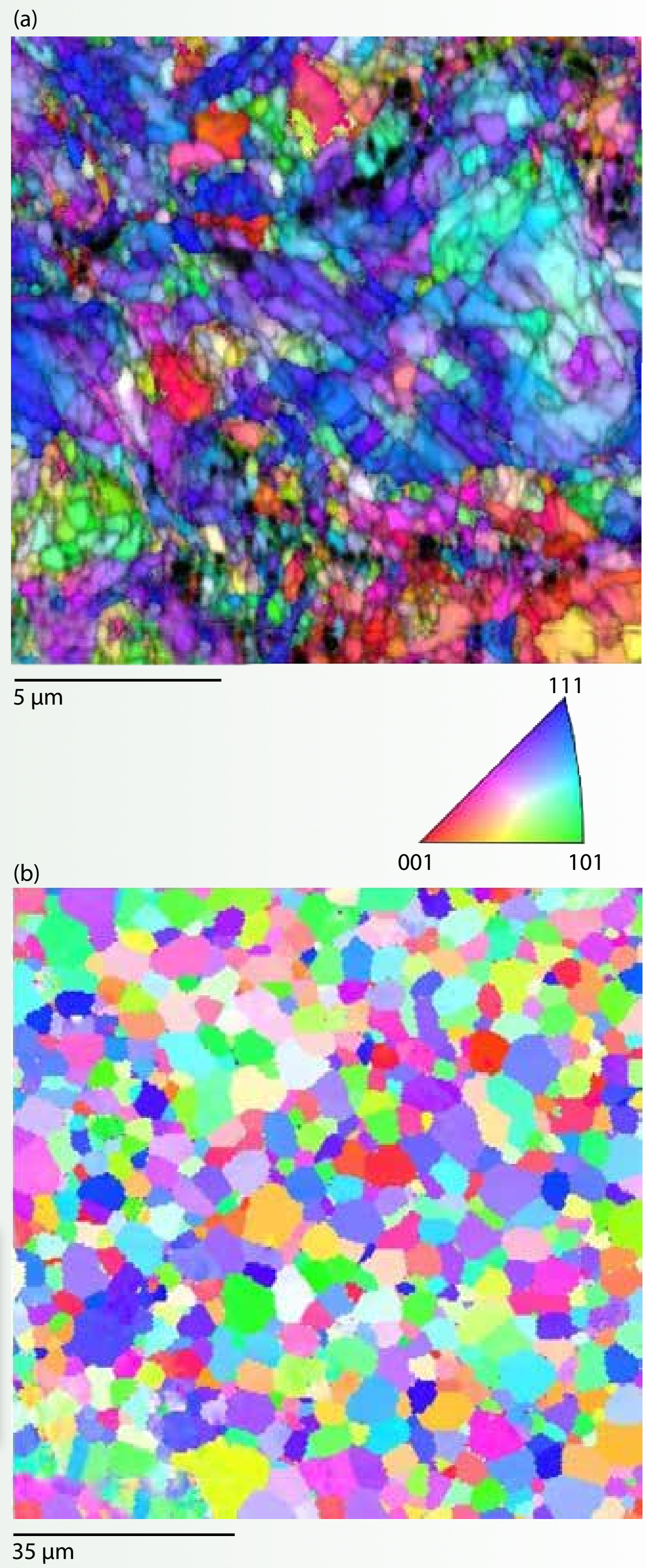

Figure 1. Pre-irradiation EBSD patterns for (a) UFG and (b) CG steel.

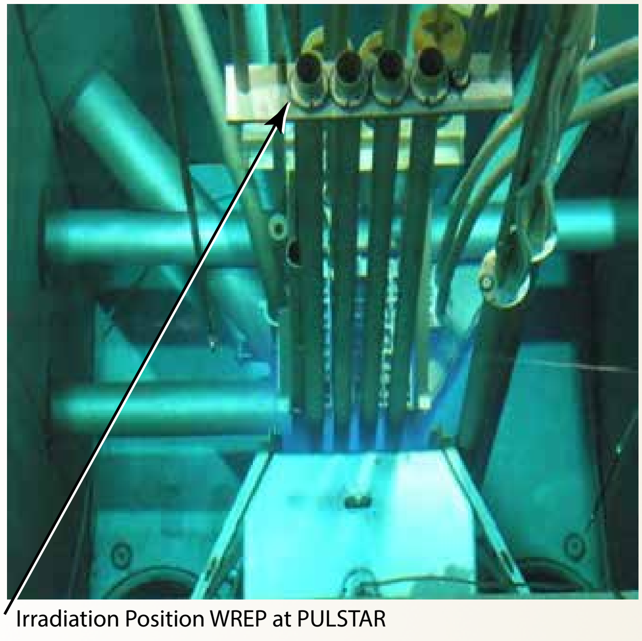

Figure 2. Irradiation position of UFG and CG carbon steels inside the PULSTAR reactor at NCSU.

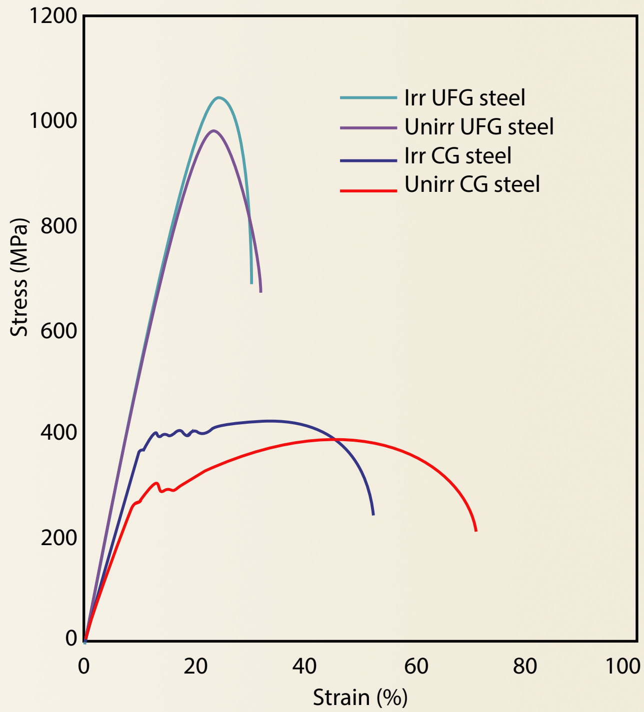

Figure 3. Stress–strain curves for both unirradiated and irradiated CG and UFG steels.

Grain size measurements were performed on UFG steel using both XRD and TEM. The XRD patterns show that the irradiation caused no phase changes. Also, the full width at half maximum (FWHM) in peaks of similar planes in the XRD patterns of both materials were very close, indicating no grain growth during irradiation. TEM micrographs of the UFG steel revealed mean grain sizes of 0.35 and 0.36 ± 0.2 µm, respectively, for unirradiated steel and steel exposed to neutron radiation.

The mechanical properties of the materials were evaluated using Vickers microhardness tests as well as tensile tests using mini-tensile samples at a strain rate of 10-3 s-1. Figure 3 shows the results of the tensile tests performed on the CG and UFG steels before and after neutron irradiation. Three tests were performed for each condition, and the resulting mean values were calculated.

The CG steel exhibited increased strength accompanied by decreased ductility, as was expected given normal radiation hardening and embrittlement. However, the UFG steel clearly exhibited less change. No yield point phenomena were observed in the UFG steel because impurity atoms (principally carbon) will migrate to the grain boundaries. Thus, they were not available for pinning the dislocations, resulting in the disappearance of the yield point.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor
North Carolina State University	PULSTAR Reactor
Collaborators	
Idaho National Laboratory Douglas Porter (co-principal investigator), Brandon Miller (co-principal investigator)	
North Carolina State University K. L. Murty (principal investigator), Ahmad Alsabbagh (collaborator)	

Principal Investigator: K. L. Murty – North Carolina State University (cont.)
email: murty@ncsu.edu

Effects of Neutron Irradiation on the Mechanical and Microstructural Properties of Equal Channel Angular Pressing Steel (cont.)

Microhardness measurements revealed increases in hardness similar to those reported in tensile tests. The increase in hardness in the UFG material was 2% compared to an 8% increase for the CG steel. The fracture surfaces of the tensile specimens were investigated using SEM for both UFG and CG materials before and after irradiation (Figure 4). While no significant differences were observed in the UFG fracture surface before and after irradiation, the CG steel shows a decrease in the diameters of the dimpled rupture depressions, indicating less time was needed for fracturing and, therefore, there was less ductility.

Preliminary results indicate that the ECAP UFG steel is more radiation-tolerant than CG steel, meaning UFG steel is a promising new structural material for future reactor applications.

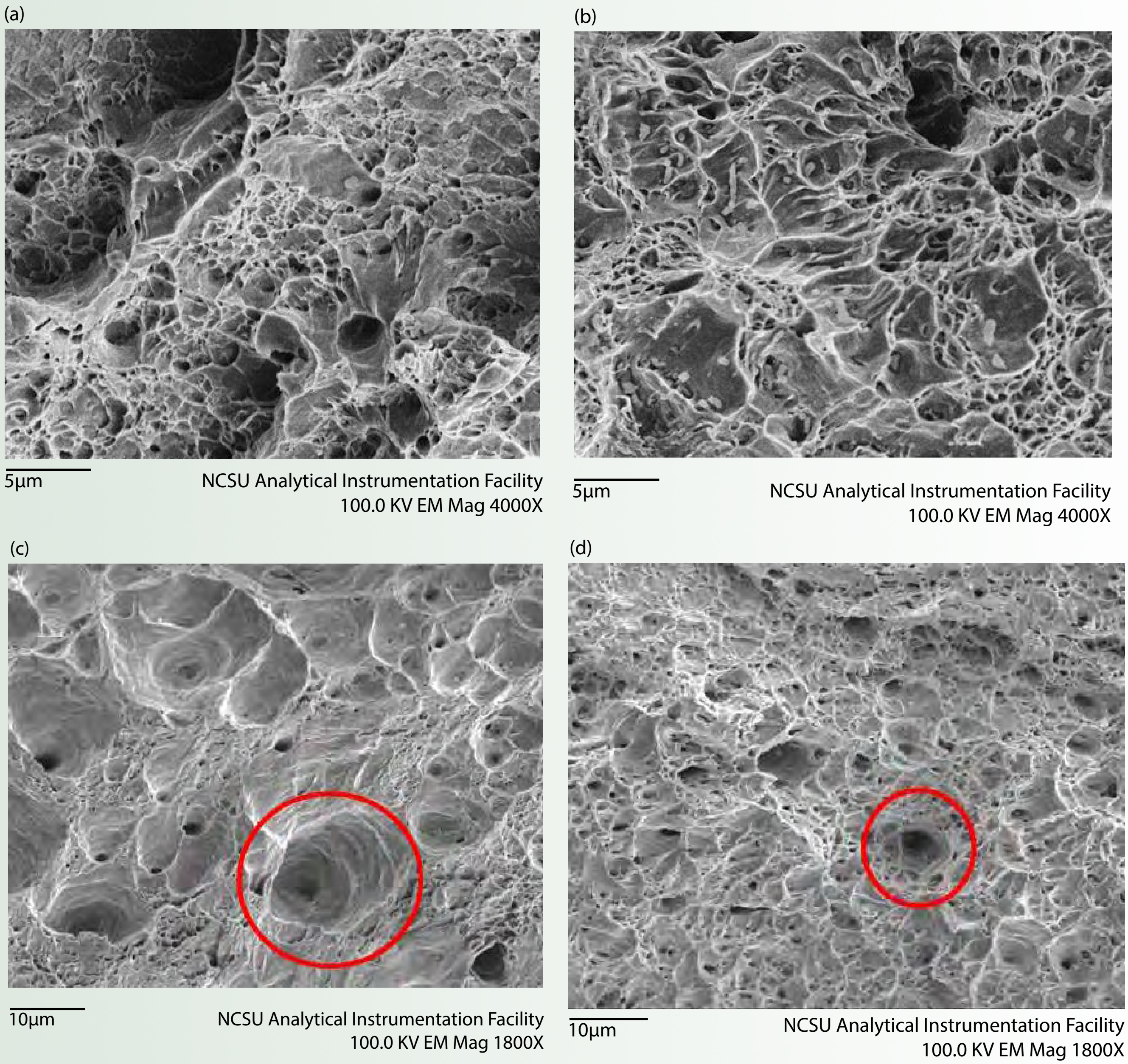

Figure 4. Fractography SEM images for (a) UFG steel before irradiation, (b) UFG steel post-irradiation, (c) CG steel before irradiation, and (d) CG steel post-irradiation.

Future Activities

The samples irradiated in the PULSTAR reactor were analyzed. Samples irradiated to 1 dpa at ATR were examined and the results are being analyzed. Evaluation of some irradiation results and testing of samples irradiated at the ATR are planned for January 2014. The 2 dpa samples will be examined at a future time.

Publications and Presentations*

Ahmad Alsabbagh, Ruslan Valiev, K. L. Murty, "Influence of Neutron Irradiation on Equal Chanel Angular Pressed Structural Steel," *Materials Science & Technology 2013, Montreal, Canada.*

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

Principal Investigator: K. L. Murty – North Carolina State University
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Ion Irradiation of Nuclear Grade NBG-18 and Highly Ordered Pyrolytic Graphites

Introduction

This ATR NSUF project helps address industry's general lack of clarity on the damage mechanisms of graphite by working to develop mechanistic, thermo mechanical models for graphite that can be applied to next-generation, high-temperature reactors.

Project Description

The ion irradiation experiments at the University of Wisconsin (UW) through the ATR NSUF aim to characterize the early-to-late stage damage mechanisms in graphite under irradiation. This follows the low-dose neutron irradiation studies in the PULSTAR reactor at North Carolina State University (NCSU) and high-dose studies at Oak Ridge National Laboratory (ORNL).

The specific objective of the work at the UW test facility was to conduct low- and high-displacements per atom (dpa) irradiation tests (1/25 dpa) at 300° K, 600° K, and 900° K with C^+ ions on NGB-18 samples. As noted in the UW ion irradiation report, the experimental parameters were:

Ions: Carbon (C^+)

Energy: 2.0 MeV

Projected range: Rp = 2 μm

Temperature: 300° K, 600° K, 900° K

Total fluence: 25 dpa-peak - $2.2\times10^{(17)}$ ions/cm^2, 1 dpa-peak – $8.8\times10^{(15)}$ ions/cm^2

Carbon displacement energy Eth = 28 eV

Average flux:

300° K-1 dpa: 1.3×10^{13} ions/(cm^2)

600° K-1 dpa: 1.1×10^{13} ions/(cm^2)

900° K-1 dpa: 1.3×10^{13} ions/(cm^2)

600° K-25 dpa: 1.4×10^{13} ions/(cm^2)

300 K-25 dpa: 1.2×10^{13} ions/(cm^2)

Time:

1 dpa ~ 12 minutes

25 dpa ~ 4.5 hours

Area: 1.32 cm^2

There are many conflicting viewpoints about what causes the disordering mechanisms in graphite used in high-temperature reactors. We show that the topological defects in graphite play an important role in its behavior under irradiation.

Accomplishments

The mechanisms of defect formation in graphite during the early stages of irradiation are still not completely understood. The traditional view postulates that a large number of interstitial and vacancy point defects are generated under irradiation. Over time, the point defects agglomerate and collapse into dislocation loops. Under continued irradiation, additional planes that are formed between the graphitic layers contribute to an expansion along the c–axis perpendicular to the basal plane. Not all experimental observations fall in line with this description. Solid-state disordering and amorphization have also been observed in graphite with neutrons, ions and electrons, particularly at low temperatures and high fluences.

In the current study, the nature of the bond and defect structures in nuclear-grade NBG-18 graphite under neutron and ion irradiation has been characterized using Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), and high resolution transmission electron microscopy (HRTEM). The magnitude of the defect D peak in the Raman spectra increases with irradiation, which indicates an increase in the number of topological defect structures that maintain the sp2-bond connectivity.

The experiments also show that the D peak does not shrink significantly in graphite, even when irradiated to several tens of dpa, indicating the persistent presence of a layered structure with topological defects. Thus, a direct condensation of the topological defects, which are initially formed during irradiation, into three-dimensional vacancy pores or amorphous pockets can be deemed highly improbable for the doses expected in high-temperature reactors. The non-vanishing D peak in the Raman spectra, together with a generous number of dislocations, even at low irradiation doses, indicates a dislocation-mediated amorphization process in graphite.

Figure 1a shows the Raman spectra of virgin and neutron-irradiated (to 0.006 dpa), nuclear-grade NBG-18 samples, while Figure 1b depicts the peak intensity ratios for different levels of radiation damage. The data for 1 dpa is

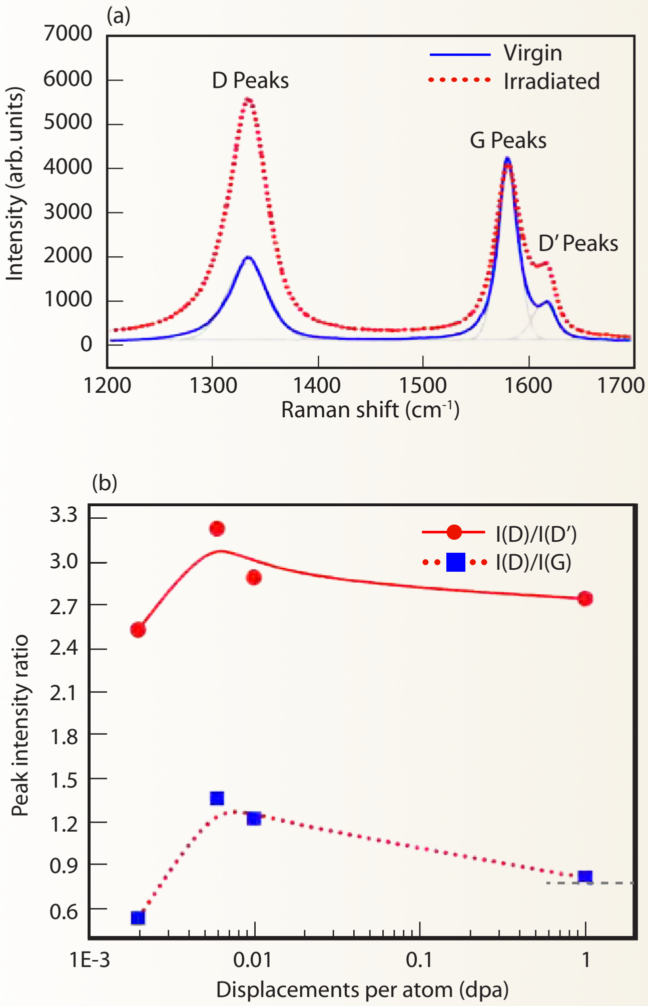

Figure 1. (a) The Raman spectra of nuclear-grade NBG-18 samples virgin and neutron-irradiated to 0.006 dpa; (b) the peak intensity ratios for different levels of radiation damage. The data for 1 dpa is based on ion irradiation at 300° K.*

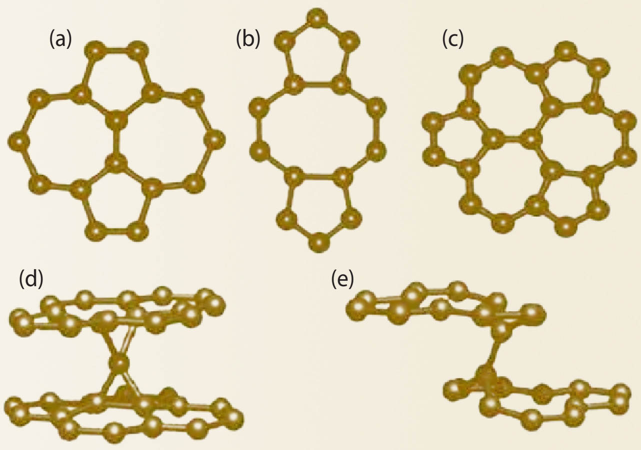

Figure 2. (a, b, c): Three topological defect configurations: Stone-Wales 55–77, 5–8–5, and 555–777; (d, e): delineations of two interplanar defect configurations: spiro and divacancy.*

based on ion irradiation at 300° K. Figures 2a, 2b, and 2c show three topological defect configurations: Stone-Wales 55–77, 5–8–5, and 555–777. Figures 2d and 2e delineate two interplanar defect configurations: spiro and divacancy.

Because each carbon atom has exactly three neighbors, the defects in the top panel maintain their sp2 connectivity. Only bonds with sp2 connectivity (topological defects) can activate the D peak in the Raman spectra.

Future Activities

All ion irradiation studies at UW have been completed.

Publications and Presentations

Jacob Eapen, Ram Krishna, Timothy D. Burchell and K. Linga Murty, *Materials Research Letters*, Vol. 2, No. 1, 2013, p. 43.

** Figures are reproduced from Jacob Eapen, Ram Krishna, Timothy D. Burchell and K. Linga Murty, Materials Research Letters, Vol. 2, No. 1, p. 43 (2013).*

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
University of Wisconsin	Tandem Accelerator Ion Beam
Collaborators	
North Carolina State University K. Linga Murty (principal investigator), Jacob Eapen (co-principal investigator), Ram Krishna (co-principal investigator)	

Principal Investigator: K. L. Murty – North Carolina State University
email: murty@ncsu.edu

Post-irradiation Examination of ATR-Irradiated Nanocrystalline Materials

Introduction

The overall objective of this project is to develop friction-stir welding (FSW) as a joining process for high-performance, oxide-dispersion-strengthened (ODS) alloys used in advanced nuclear reactors. The ODS alloys have applications as in-core materials due to their excellent high-temperature resistance and enhanced tolerance to radiation damage.

Project Description

Two ODS alloys, MA956 and MA754, were investigated. The MA956 alloy, with its high aluminum content (about 5.5 wt.%), is especially encouraging as a potential accident-tolerant material.

The objectives of these investigations are:

1. To examine the feasibility of using ODS alloys as structural and cladding materials in advanced nuclear reactors.
2. To optimize the FSW technique for joining MA956 and MA754.
3. To evaluate the mechanical properties of processed/welded materials by using microhardness and mini-tensile testing.
4. To study the microstructures and microtextures of the processed/welded materials.
5. To investigate the effect of fast-neutron radiation exposure of 1 displacement per atom (dpa) and 2 dpa through post-irradiation examination (PIE) and compare the results with the unirradiated parent and welded materials.
6. To develop appropriate structure-property correlations.

Items 1 – 4 have been completed. The research team's focus is now on its last two objectives: the characterization and related analyses of the irradiated materials.

Accomplishments

FSW of MA956 and MA754 plates was performed at the Missouri University of Science & Technology (MS&T) as part of a separate, now concluded project. These welded samples were provided to the University of Idaho (UI) for further work. Both the parent and FSW samples of MA956 and MA754 were irradiated at ATR to doses of 1 dpa and 2 dpa at temperatures between 65° C and 80° C.

Microhardness testing on unirradiated parent and FSW samples of the alloys was performed at INL's MFC by UI graduate student Ramprashad Prabhakaran. Grain sizes and the microtextures of MA956 and MA754 were measured using characterization tools such as electron backscatter diffraction (EBSD).

The ODS alloys, MA956 and MA754, hold great potential for use as structural and cladding materials in advanced nuclear reactors, and the work pursued in this project will help establish them as such.

Transmission electron microscopy (TEM) was carried out on the unirradiated samples of the alloys in the Microscopy and Characterization Suite (MaCS) at CAES in order to study fine microstructural details, such as the morphology and distribution of oxide particles and dislocation structures. Atom probe tomography (APT) work using the local electrode atom probe (LEAP) was also performed on select samples to study the fate of the particle and dislocation structure after irradiation.

The irradiated samples were prepared for further characterization at MFC. TEM and APT analyses of the irradiated samples were performed at the CAES MaCS. Figure 1 shows a TEM image of the parent MA956 alloy and Figure 2 shows the FSW MA956 alloy. Both alloys were irradiated to 1 dpa. Further analyses of other specimens are ongoing, and the collected data are being analyzed.

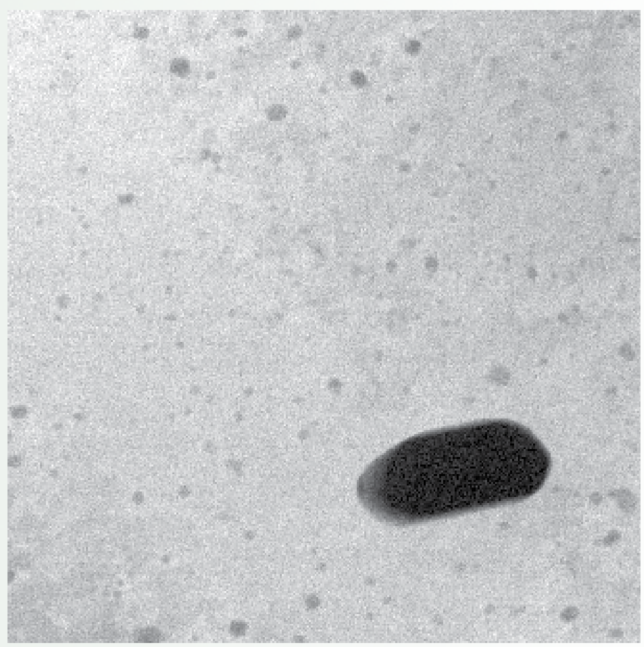

Figure 1. TEM image of the parent MA956 alloy irradiated to 1 dpa.

"We feel fortunate to have access to some of the best state-of-the-art facilities in the United States through ATR NSUF. Also, much thanks to its ever helpful staff."

Indrajit Charit, Associate Professor, College of Engineering, University of Idaho

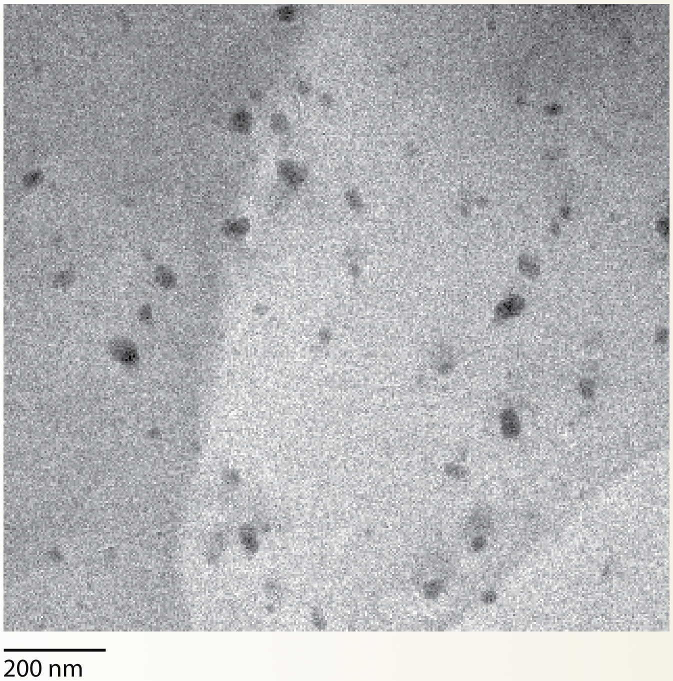

Figure 2. TEM image of the friction-stir-welded MA956 alloy irradiated to 1 dpa.

Future Activities

Microhardness testing on irradiated samples will be performed in 2014.

Publications and Presentations*

1. R. Prabhakaran, et al., "Irradiation Studies on Friction Stir Welded ODS Alloys," *The Minerals, Metals, and Materials Society (TMS) 2013 Annual Meeting, San Antonio, Texas, March 3-7, 2013.*

2. R. Prabhakaran, et al., "The Effect of Neutron Irradiation on Friction Stir Welded MA956 and MA754," *Materials Science & Technology (MS&T) 2012, Pittsburgh, Pennsylvania, October 7-11, 2012.*

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microsocopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Collaborators	
Idaho National Laboratory Doug Porter (principal investigator)	
Missouri University of Science & Technology Rajiv S. Mishra (collaborator)	
North Carolina State University K. Linga Murty (principal investigator), Walid Mohamed (collaborator)	
University of Idaho Indrajit Charit (principal investigator), Ramprashad Prabhakaran (collaborator)	

Principal Investigator: G. Robert Odette – University of California, Santa Barbara
email: Odette@engineering.uscb.edu

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

Introduction

In order to extend nuclear plant operation up to 80 years, it will be necessary to demonstrate that massive light water reactor pressure vessels (RPV) can maintain large safety margins against sudden fracture. Neutrons that leak from the reactor core bombard the RPV steels and cause embrittlement, manifested as increases in the brittle fracture temperature. Unfortunately, there are almost no transition temperature shift (TTS) plant surveillance data for high neutron fluence at the long durations experienced during extended reactor life.

Accelerated test reactor irradiations can reach high fluence levels of 10^{20} n/cm^2 in a much shorter time, but may not be reliable indicators of real-world operation due to the complex effects of neutron flux on new damage mechanisms that may emerge during extended life. This University of California, Santa Barbara (USCB) irradiation experiment (referred to as “ATR-2”) was designed to address these critical issues.

Project Description

A special test train for RPV steel specimens was designed, fabricated and inserted into INL’s ATR in June 2011. The comprehensive specimen/alloy/irradiation matrix, consisting of disc multipurpose coupons (MPC), disc compact tension fracture, subsized tensile and other specimens of 172 different RPV steels, will be studied to enable more accurate high-fluence TTS predictions.

Intermediate-flux ATR irradiations covering a temperature range of 250° C to 310° C, up to a fluence 10^{20} n/cm^2, will bridge large gaps in existing embrittlement databases. The UCSB ATR-1[1] project’s high-fluence, intermediate-flux database will be linked to other test reactor and surveillance data over a much wider range of flux. These data will be used to inform, validate and calibrate new predictive, physically-based TTS models. The objectives of the experiment include:

- Assessing the effects of flux and the synergistic interactions between all embrittlement variables.
- Using post-irradiation annealing to evaluate the effects of flux and other embrittlement variables on the various types of radiation-induced, nm-scale features that cause TTS.
- Identifying conditions that lead to the formation of so-called “late-blooming” phases that could cause the severe embrittlement but are not treated in current regulatory models.
- Conducting extensive microstructural characterization and mechanism studies.
- Evaluating annealing as a potential embrittlement mitigation strategy.
- Irradiating the new RPV alloys, including candidates for use in advanced reactors.
- Evaluating the master curve method for measuring fracture toughness at high fluence in sensitive alloys.
- Addressing issues associated with uncertainties in alloy conditions in the actual vessel.

Accomplishments

Fabrication and assembly of the UCSB ATR-2 irradiation test train was completed in late spring of 2011 and was successfully installed in ATR in May of that year. Irradiation began in June 2011. It was anticipated that the irradiation would achieve its target average fluence of 10^{20} n/cm^2 (E>1 MeV) in the fall of 2012. Thermocouple monitors (Figure 1) have shown that the specimens are generally being irradiated at or close to their target temperatures during the course of the irradiation campaign. However, the final determination of irradiation temperatures and dosimetry information (i.e., neutron flux and fluence) will be made following the test train’s removal from the reactor and disassembly.

A number of delays in ATR operation have postponed the completion of the ATR-2 irradiation campaign to the spring of 2014. As a result, the accumulated fluence at the scheduled date for a Powered Axial Locator Mechanism (PALM) cycle fell well below the target fluence. Specifically, the average neutron fluence for the ATR-2 specimens was only 6.34×10^{19} n/cm^2 at that time. PALM cycles are occasionally performed by ATR staff for complex transient testing. However, the high lobe power required in this cycle would not permit proper control of the UCSB ATR-2 specimen temperatures. Thus, a decision was made to remove the test train before a PALM cycle, and reinsert it after the cycle was complete.

This decision required designing and carrying out a mock-up experiment to load and remove the capsule. The mock-up test train was placed in the ATR tank and successfully transferred through the drop chute into the canal in February 2013. A new tool was designed to grip the bottom of the experiment without damaging the thin-walled test train tubing. The mock-up test demonstrated that the new tool (Figure 2) could maintain the correct orientation of the test train as needed to complete the transfers. Subsequently, the real test train was successfully

[1] "Characterization of the Microstructures and Mechanical Properties of Advanced Structural Alloys for Radiation Service: A Comprehensive Library of ATR Irradiated Alloys and Specimens" (referred to as "ATR-1"). Project report also included in this Annual Report.

"This UCSB irradiation experiment anchors the largest and most comprehensive international effort to develop robust models that are needed to predict nuclear reactor pressure vessel embrittlement at high fluence and low flux conditions. The long-term economic impact of this sophisticated science-based research in the service of critical energy technology is almost incalculable."

G. Robert Odette, Principal Investigator, University of California, Santa Barbara

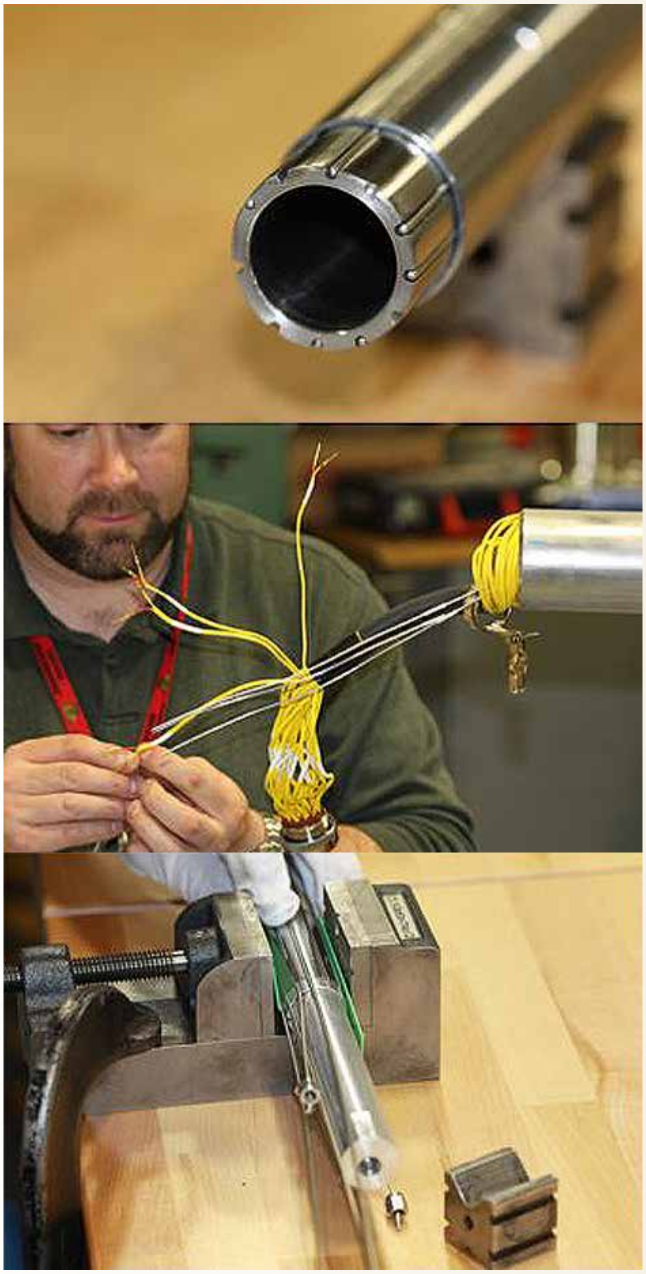

Figure 1. Thermocouple and gas feed assembly.

transferred to the canal using the same procedure. It was transferred out to the canal again in April 2013, to avoid the PALM cycle.

The experiment remained in the canal through the PALM cycle, and then was transferred back into ATR for additional irradiation cycles. UCSB fully concurs with the Experiment Manager, Tom Maddock, who reported that the INL team performing this work did an excellent job in transferring the capsule. Based on the projected cycle times, the average and peak fluences will be 0.87 and

This experiment is an absolutely critical part of the DOE effort to expand the largest C-free contribution to the U.S. electricity supply by extending the safe operation of pressurized water reactors to 80 years or more.

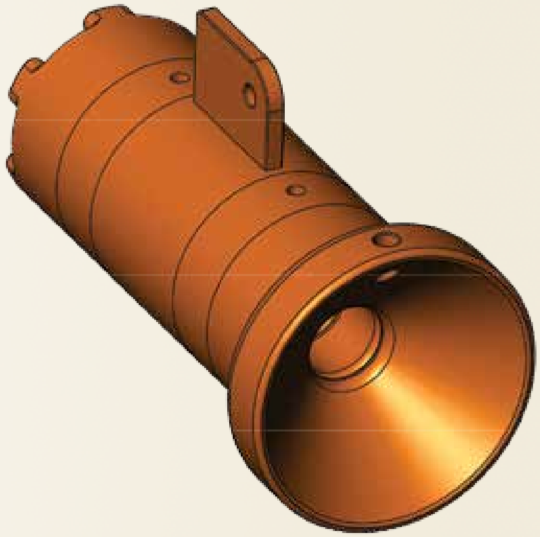

Figure 2. A mock up of the innovative tool the research team used to extract the capsule from the reactor. Drawing courtesy of Mechanical Research & Design, Inc.

1.2×10^{19} n/cm^2, respectively, after two additional cycles ending in the spring of 2014, when the ATR-2 capsule will likely be removed.

Work on developing a post-irradiation examination (PIE) test plan has continued. Initial plans for shipping the test assembly from INL to Oak Ridge National Laboratory (ORNL) were also formulated. PIE will involve extensive microstructural characterization and mechanical testing. The largest mechanical testing effort will be directed at carrying out a very large number of advanced shear punch tests (SPT). As was reported previously, we have developed procedures that provide excellent and nearly theoretically expected correlations between SPT yield (shear) stress and conventional uniaxial tensile yield stress data. In the past year the researchers have continued their effort to design a semi-automated shear punch test instrument to perform the very large number of required tests efficiently, while assuring that very high quality data is obtained. The mechanical property PIE, which will also involve testing all the tensile and compact tension fracture specimens, will be carried out at ORNL.

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High-Fluence Embrittlement Database and ATR Irradiation Facility for Light Water Reactor Vessel Life Extension (cont.)

Future Activities

Following the completion of irradiation and a four- to six-month cool-down period, the test train will be shipped to ORNL near the end of 2014. The test train and capsules will then be disassembled and the comprehensive PIE program will begin in early 2015.

Publications and Presentations

G. Robert Odette, Takuya Yamamoto, Doug Klingensmith, Mitch Meyer, M. Sprenger, Tom Maddock, P. Murrey, Joseph Nielsen, Randy K. Nanstad, William Server, "The Status of the UCSB ATR-2 RPV Irradiation Experiment," *International Group of Radiation Damage Mechanisms, 17th Semiannual Meeting, Embiez Island, France, May 19-24, 2013*.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor
Collaborators	
Bettis Laboratory Ray Stephanik (collaborator)	
Idaho National Laboratory Brandon Miller (collaborator), Collin Knight (collaborator), James Cole (collaborator)	
Japanese Central Research Institute of the Electric Power Industry Naoki Soneda (collaborator)	
Oak Ridge National Laboratory Randy Nanstad (collaborator)	
Rolls Royce Keith Wilford (collaborator)	
University of California, Santa Barbara G. Robert Odette (principal investigator), Takuya Yamamoto (co-principal investigator)	
Various segments of the U.S. nuclear industry William Server (collaborator)	

Characterization of the Microstructures and Mechanical Properties of Advanced Structural Alloys for Radiation Service: A Comprehensive Library of ATR-Irradiated Alloys and Specimens

Principal Investigator: G. Robert Odette – University of California, Santa Barbara
email: Odette@engineering.uscb.edu

Introduction

The objective of this project is to create a new, large library of irradiated alloy sample types and conditions to address many outstanding issues in the field of reactor materials for advanced fission applications. Even after testing a handful of specimens, the experiment (referred to as "ATR-1") results have produced some seminal and enormously high-impact results.

This project aims to create a new, large library of irradiated alloy sample types and conditions to address many outstanding issues in the field of reactor materials for advanced fission applications.

Project Description

The matrix for this library consists of 49 alloys including:

- Tempered martensitic steels.
- Nanostructured ferritic alloys.
- A stainless steel.
- Model alloys that include iron-chromium (Fe-Cr) binaries, manganese-molybdenum-nickel (Mn-Mo-Ni).
- Bainitic reactor pressure vessel (RPV) steels.

The irradiation conditions span a wide range of temperatures and damage levels. Various specimens target specific irradiation damage data and mechanisms. Irradiating many materials side-by-side, under many different conditions, provides a unique database that will greatly facilitate identification, understanding, modeling, optimizing, and predicting the performance of structural materials used in nuclear energy systems.

The database characterizes hardening and softening phenomena caused by irradiations from 1.5 to 6.0 displacements per atom (dpa) at temperatures from ≈ 290º C to 750º C. Mechanical property changes will be assessed by micro-hardness and instrumented shear punch tests. In addition, tensile tests will be carried out on a subset of alloys. Fracture toughness measurements using compact tension samples, in the framework of the master curve method, will also be conducted on a subset of alloys to assess irradiation embrittlement.

Model alloys will be used to study fundamental damage mechanisms. For example, Fe-3 to 18%Cr binary alloys irradiated over a wide range of temperatures will be used to probe phase boundaries and characterize solute segregation processes and mechanisms. Multi-constituent diffusion multiples, aka lab-on-a-chip specimens, will enable characterization of how various elements in complex alloys under irradiation migrate and rearrange themselves in different phases. Finally, in-situ helium implantation studies—also a lab-on-a-chip approach—will be conducted.

State-of-the-art tools will be used in post-irradiation examination (PIE) to relate the mechanical property effects to the corresponding microstructural evolutions. The majority of the approximately 1,380 specimens to be tested are disc multipurpose coupons. The list of characterization techniques includes micro-hardness, shear punch, tensile, fracture, neutron scattering, transmission electron microscopy (TEM), positron annihilation, X-ray scattering and diffraction, and atom probe tomography (APT). The library will support a very large number of national and international collaborations.

Accomplishments

This project looks at irradiation embrittlement of light water reactor pressure vessel steels. Current regulations reflect the strong effect of copper (Cu) and nickel (Ni) on embrittlement, associated with the rapid formation of Cu-rich precipitates (CRPs) that harden the steel. However, theoretical models long ago predicted a new embrittlement mechanism associated with the formation of Mn-Ni-Si at high fluence. These so-called late blooming phases (LBP) could cause severe and unexpected embrittlement even in low Cu steels. Notably, they are not addressed in current regulations, thus they need to be accounted for to ensure the safety of extending the life of reactors to 80 years or more.

Research at the University of California, Santa Barbara (UCSB) over the past decade has confirmed that LBPs do exist under some conditions. The multifaceted question now becomes: (a) at what alloy composition, irradiation temperature, fluxes and fluences do LBPs form; (b) what are their compositions, volume fractions and structures; and (c) what are the relevant mechanisms mediating LBP precipitation?

"The fact that my Ph.D. research is able to use cutting-edge science to tackle an immensely important engineering challenge of long-term, worldwide impact is highly motivating. The UCSB ATR-1 irradiation has provided the fuel for my very exciting journey."

Peter Wells, Ph.D. Candidate, University of California, Santa Barbara

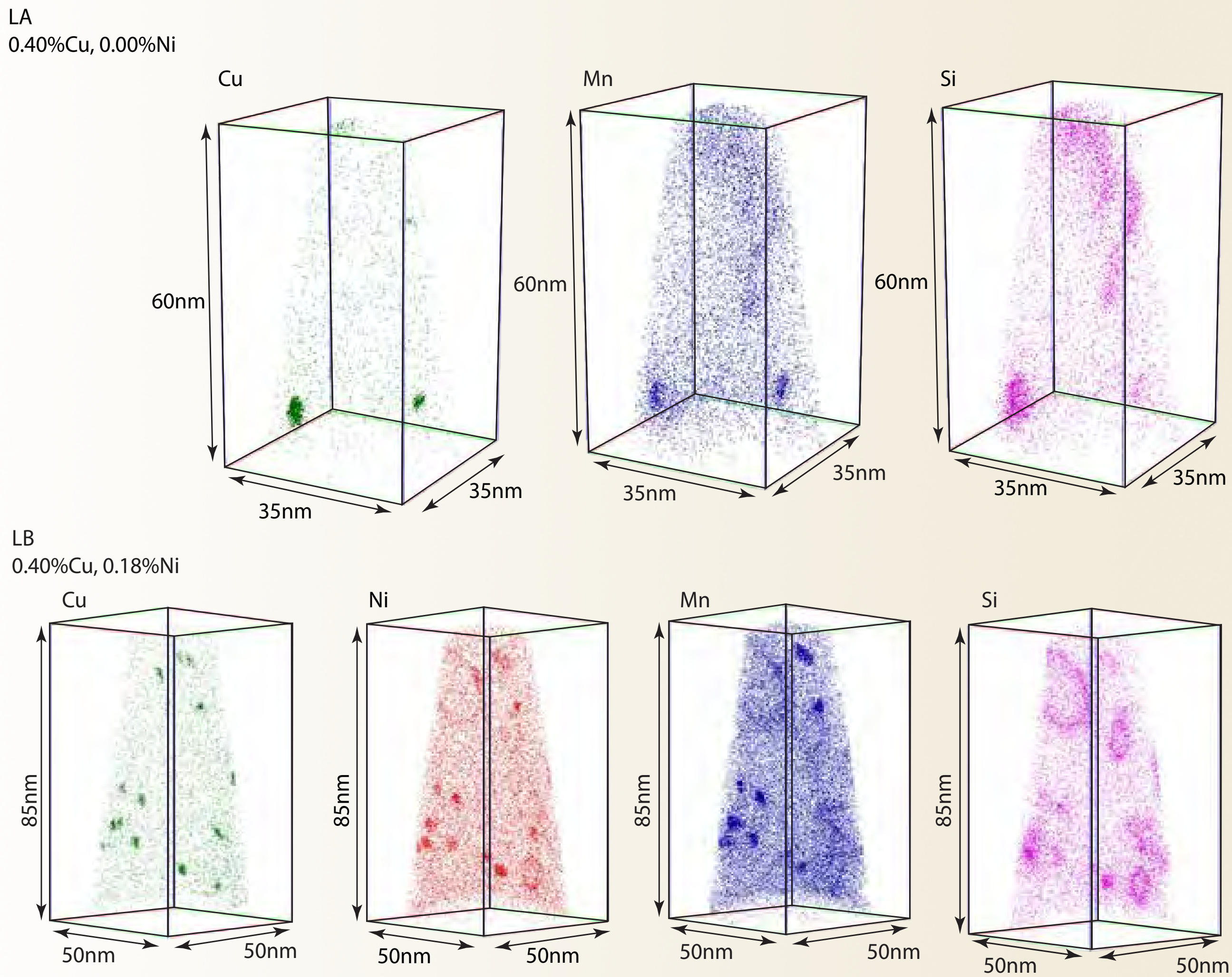

Figure 1. Atom maps of two high-Cu steels. Top is a Ni-free alloy (LA) and bottom is a low, 0.18st.%Ni (LB) alloy. The Ni-free steel contains fairly large, coarse precipitates that have a Cu-rich core surrounded by a Mn- and Si-rich "cloud." The low-Ni steel contains a higher density of smaller Cu-Ni-Mn-Si clusters.

In one of many examples, this past year researchers investigated the effect of very low Ni levels in two high-Cu (0.4 wt.%) ATR-1 steels irradiated to 1.8 dpa at 290° C. Two alloys were examined that contained 0.0wt.%Ni (LA) and 0.18wt.% Ni (LB), respectively. The resulting atom maps are shown in Figure 1. The Ni-free steel contained a low density of CRPs having some Mn and Si, although the latter appeared to form a more diffuse "cloud" around a Cu-rich core and Mn shell.

The increase in bulk Ni from 0 to 0.18wt.% resulted in a higher density of CRPs. A high density of dislocation loops were also observed, marked by significant Ni, Mn and Si segregation. In some cases, a Cu-rich cluster also

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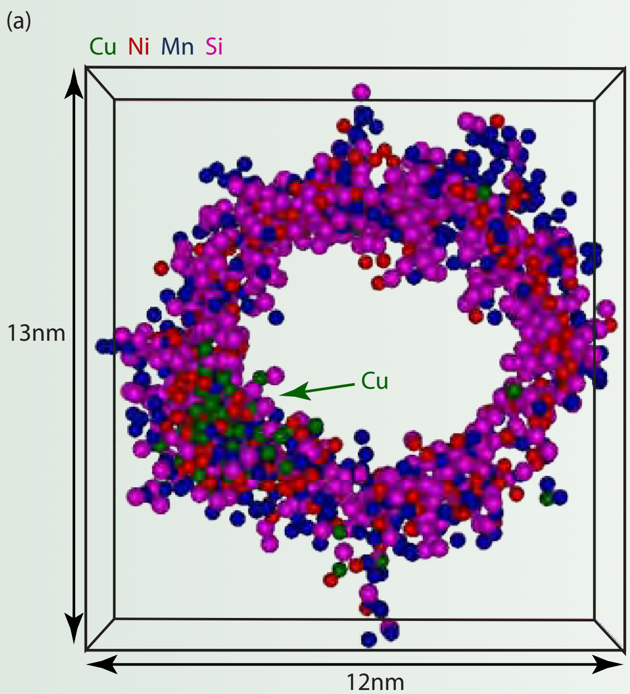
Characterization of the Microstructures and Mechanical Properties of Advanced Structural Alloys for Radiation Service: A Comprehensive Library of ATR-Irradiated Alloys and Specimens (cont.)

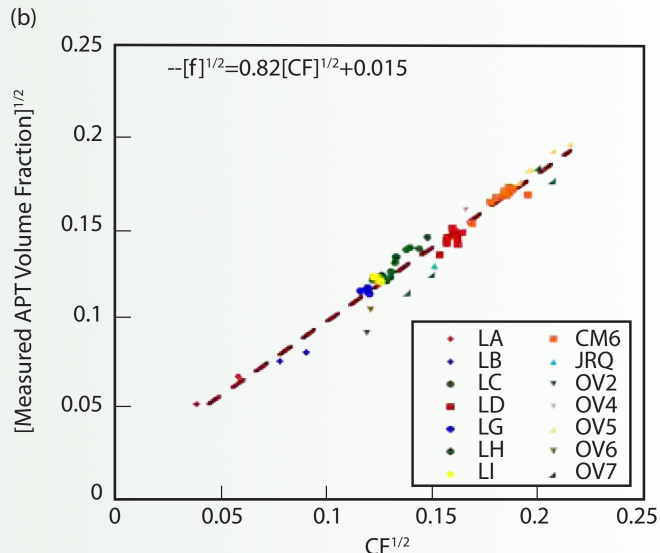
formed on one part of the Ni-, Mn- and Si-enriched loop (Figure 2a). As expected, the precipitates had a much lower concentration of Ni than was previously found in the medium- and high-Ni steels. Figure 2b shows the relation between the square root of the precipitate volume fraction and an alloy chemistry factor determined by APT.

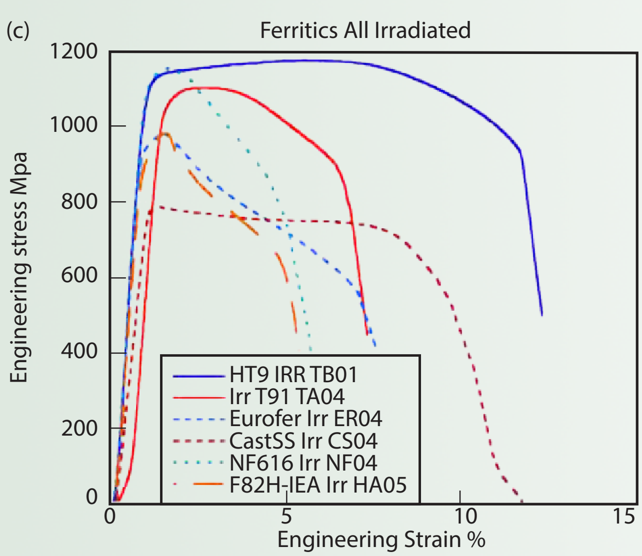
One of the major achievements during the year was the shipment of 11 packets from INL to Los Alamos National Laboratory (LANL) for disassembly and testing as part of their Fuel Cycle Research and Development (FCRD) program led by Dr. Stuart Maloy. Figure 2c shows an example of data from LANL hot cell tensile tests that reflect differences in several tempered martensitic steels and one austenitic stainless steel irradiated at 290° C under identical irradiation conditions. The opportunity to cross-compare alloys in this way is a unique feature of the UCSB ATR-1 library. Figure 2d illustrates recent study results of the Fe-3-18%Cr binary alloy series, led by Professor Emmanuelle Marquis at the University of Michigan, showing that α’ precipitation begins at 9%Cr.

Future Activities

To date, only a tiny fraction of the specimens from UCSB ATR-1 have been characterized. The main issue has been access to the specimens. That is being resolved by







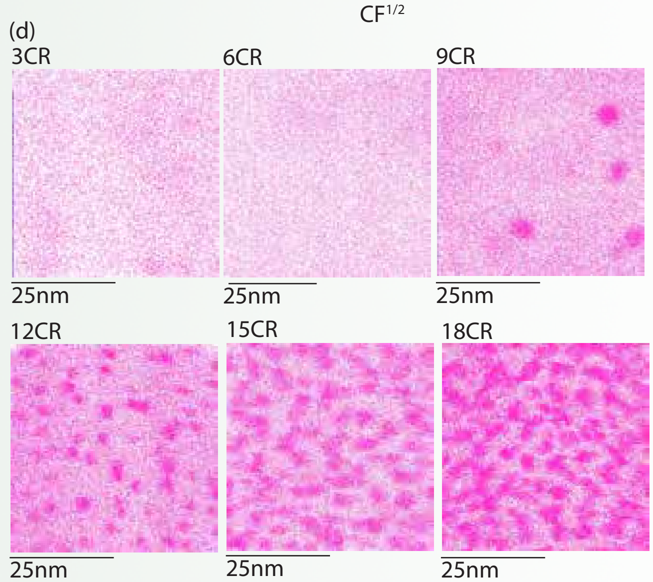


Figure 2. (a) APT reconstruction showing a solute-decorated dislocation loop, (b) √f versus √CF, (c) comparison of tensile stress strain curves for six alloys, and (d) APT reconstructions showing the initiation of α’ precipitation at 9Cr in the Cr alloy series[1].

shipping the remaining capsules from INL to LANL. One of the many RPV subtask topics will continue to address outstanding questions regarding LBPs, including their formation mechanism. This will include a series of anneals on the various alloys followed by both hardness testing and ATP. These anneals will allow us to identify different hardening features, determine their stability, and confirm that the majority of hardening is from the precipitates. However, our ultimate objective is to develop a robust, physically-based model to predict transition temperature shift (TTS) under high-fluence, low-flux conditions. The UCSB ATR-1 and ATR-2 experiments form a critical foundation for such a model.

Reference

[1] M. Bachhav, G. R. Odette and E. A. Marquis, "α' precipitation in neutron-irradiated Fe–Cr alloys," *Scripta Materialia,* 74 (2014) 48–51.

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2. G. Robert Odette, Peter Wells, Tim Milot, Nicholas Cunningham, Takuya Yamamoto, Doug Klingensmith, Kirk Fields, David Gragg, James Cole, Brandon Miller, Collin Knight, Emmanuelle Marquis, Mukesh Bachhav, Steve Roberts, Chris Hardie, Stuart Maloy, "UCSB ATR-1: Current Progress on PIE and Future Plans," *ATR NSUF Users Meeting 2013, Idaho Falls, Idaho, June 10-14, 2013*.
3. G. Robert Odette, Peter Wells, Takuya Yamamoto, Yuan Wu, Nicholas Cunningham, "On the Evolution of Late Blooming Phases in RPV Steels: Theoretical Foundations, Experimental Observations and Recent Insights," *The Minerals, Metals and Materials Society 2013 Annual Meeting, San Antonio, Texas, March 3-7, 2013*.
4. G. Robert Odette, Peter Wells, Takuya Yamamoto, Yuan Wu, Nicholas Cunningham, Huibin Ke, Wei Xiong, Leland Barnard, Dane Morgan, "The Evolution of Late Blooming Phases in RPV Steels: Theoretical Foundations, Experimental Observations Recent Insights and Implications to Life Extension," *International Group of Radiation Damage Mechanisms 17th Semiannual Meeting, Embiez Island, France, May 19-24, 2013*.
5. Peter Wells, G. Robert Odette, Yuan Wu, Takuya Yamamoto, James Cole, Brandon Miller, Collin Knight, "Late Blooming Phases in RPV Steels at High Fluence and Flux," *International Group of Radiation Damage Mechanisms 17th Semiannual Meeting, Embiez Island, France, May 19-24, 2013*.
6. Peter Wells, G. Robert Odette, Yuan Wu, Takuya Yamamoto, James Cole, Brandon Miller, Collin Knight, "The Evolution of Late Blooming Phases from High to Very High Fluence," *The Minerals, Metals and Materials Society 2013 Annual Meeting, San Antonio, Texas, March 3-7, 2013*.
7. Peter Wells, Takuya Yamamoto, Yuan Wu, Tim Milot, G. Robert Odette, James Cole, Brandon Miller, Collin Knight, Kiyohiro Yabuuchi, Akihiko Kimura, "Late Blooming Phases in RPV Steels: High Fluence Neutron and Ion Irradiations," *NuMat 2012, Osaka, Japan, October 22-25, 2012*.
8. Yuan Wu, Takuya Yamamoto, Peter Wells, G. Robert Odette, James Cole, Brandon Miller, Collin Knight, "TEM Characterization of Dislocation Loops and Precipitates in Irradiated RPV Steels," *International Group of Radiation Damage Mechanisms 17th Semiannual Meeting, Embiez Island, France, May 19-24, 2013*.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Collaborators	
Idaho National Laboratory Brandon Miller (collaborator), Collin Knight (collaborator), James Cole (collaborator)	
Los Alamos National Laboratory Stuart Maloy (collaborator)	
University of California, Berkeley Peter Hosemann (collaborator)	
University of California, Santa Barbara G. Robert Odette (principal investigator), Takuya Yamamoto (co-principal investigator), Peter Wells (collaborator), Nathan Almirall (collaborator)	
University of Michigan Emmanuelle Marquis (collaborator), Mukesh Backhak (collaborator)	
University of Oxford Steve Roberts (collaborator), Chris Hardie (collaborator)	

Principal Investigator: Janne Pakarinen – University of Wisconsin
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Microstructures of Low-Dose Helium (He^{2+}) and Hydrogen (H^+) Ion Irradiated Uranium Dioxide (UO_2)

Introduction

The development of a fundamental understanding of changes in microstructure and thermal conductivity of the ceramic nuclear fuel UO_2 during irradiation is crucial to the operation of the global fleet of light water reactors (LWRs).

Project Description

While the overall goal of the project is to resolve the link between microstructures and thermal conductivity in UO_2, the intent of this ATR NSUF sub-project is to fabricate and fully characterize a simple microstructure in ion-irradiated UO_2.

Low-dose (< 1 displacements per atom [dpa]) ion irradiation microstructures of UO_2 have previously been studied by indirect methods such as X-ray diffraction (XRD) and Raman spectroscopy. Irradiation defects have been identified as (vacancy-interstitial) Frenkel defects and/or small defect clusters. However, the link between the thermal transport properties and the irradiation damage is as yet undiscovered.

Researchers have conducted ion irradiations at the University of Wisconsin (UW), laser-based thermal conductivity measurements at INL, and advanced X-ray methods in the Stanford Synchrotron Radiation Laboratory (SSRL) at Stanford University. Combining those results with the proposed transmission electron microscopy (TEM) and scanning electron microscopy (SEM) work at CAES, state-of-the-art knowledge will be obtained from the impact of irradiation damage on the thermal transport properties of UO_2.

Accomplishments

The main emphasis of this ATR NSUF rapid turnaround project in 2013 was to characterize irradiation damage in He^{2+} and H^+ ion irradiated UO_2. The ion irradiations were carried out at UW as part of the Center for Materials Science of Nuclear Fuels, one of the DOE-funded Energy Frontier Research Centers. The work began in early 2013, with 3.9 (MeV) He^{2+} ion irradiations. Polycrystalline UO_2 samples were irradiated ($T < 200^\circ$ C) up to two fluencies (9×10^{15} and 5×10^{16} He^{2+}/cm^2). XRD measurements of the irradiated samples showed a characteristic lattice expansion (Figure 1a).

Laser-based thermal transport measurements at INL revealed that the thermal conductivity for the samples was reduced by a remarkable 50%. TEM examination at CAES provided evidence that the microstructures were fairly unrefined and contained a small density of dislocation loops, accompanied by dislocation segments in the plateau region (Figure 1b).

The link between the thermal properties and specific irradiation damage in the ceramic nuclear fuel UO_2 is as yet undiscovered.

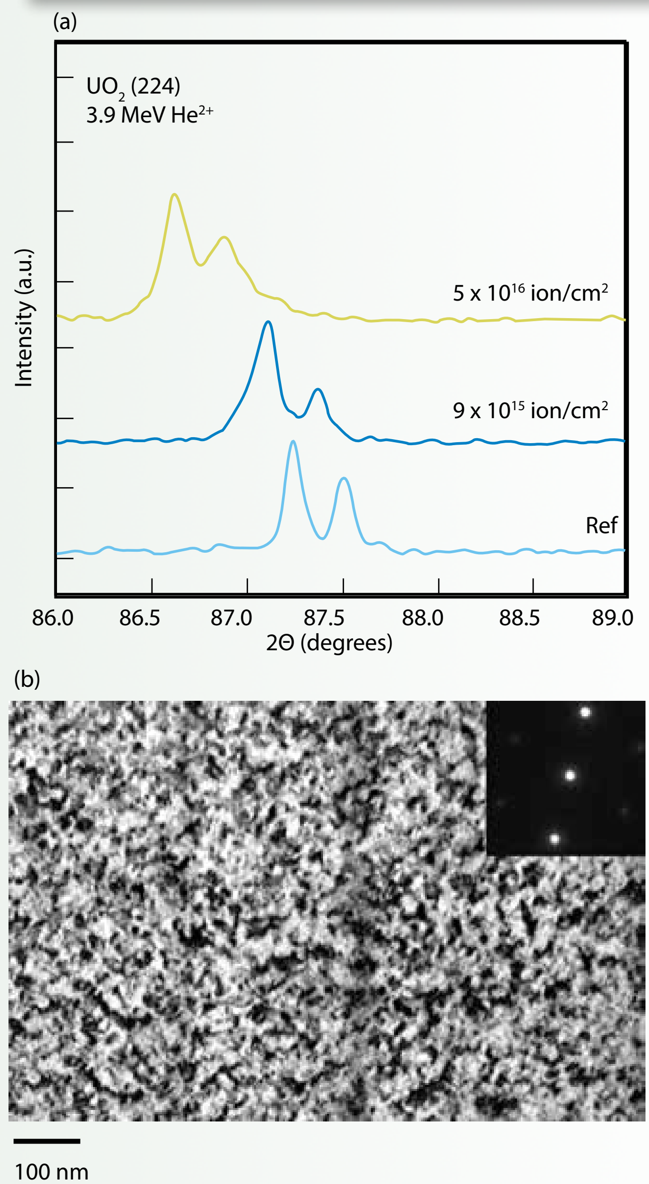

Figure 1. (a) XRD showed a clear peak shift for the He^{2+}-irradiated samples, (b) Irradiation damage consisted of small dislocation loops and dislocation segments. [1]

Large He^{2+}-induced blisters were seen in the peak damage region of the irradiated samples (Figures 2a and 2b). The blisters were more prominent in the 5×10^{16} He^{2+}/cm^2 sample, indicating that the He^+, as well as the displacement damage, influenced the blister formation. [1]

"It certainly felt bad to destroy about 50% of our precious samples in one irradiation run, but now we know the blistering limits for single-crystal UO_2 with protons."

Janne Pakarinen, Visiting Scientist, University of Wisconsin

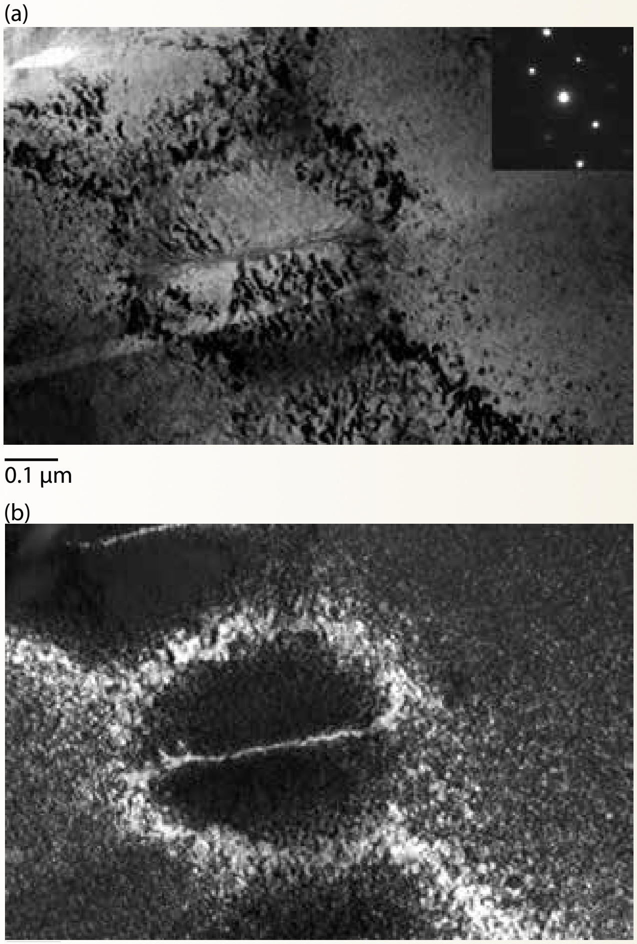

Figure 2. Bright field (a) and dark field (b) TEM showed the presence of large blisters in the peak damage region of the He^{2+}-irradiated samples. [1]

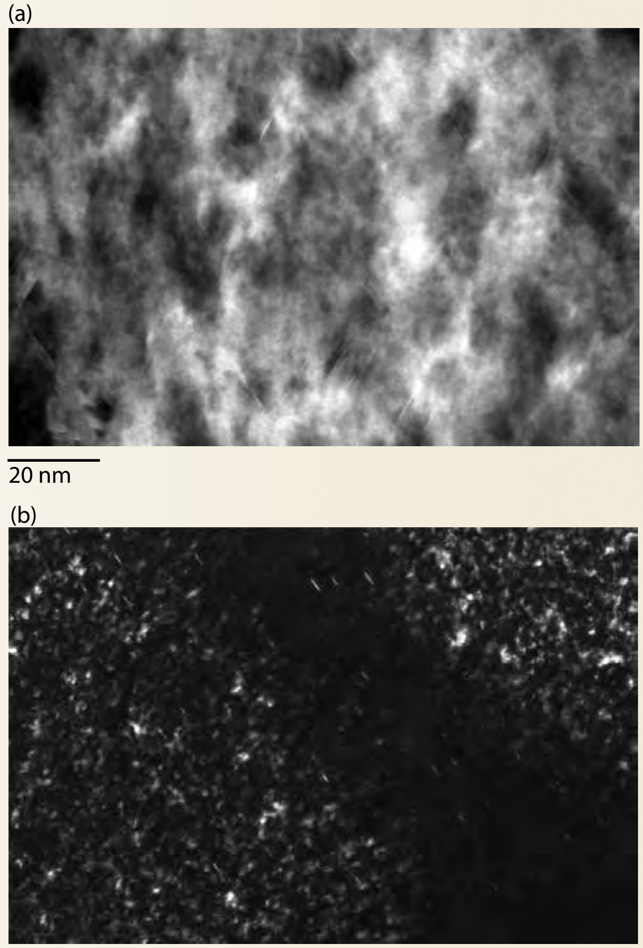

Figure 3. (a) Dislocation loops were seen edge-on at the (111) planes of UO_2. (b) Rel-rod contrast in dark field imaging conditions indicates that the loops confine a stacking fault. [4]

After the He^{2+} irradiations, a systematic study for 2.6 MeV H^+ ions was done at T< 300º C. Blistering limits for single and polycrystalline UO_2 samples were determined as a function of H^+ ion fluence [2]. Most notably, the threshold for sample degradation was found to be much smaller than anticipated, which caused the loss of eight single crystalline samples. The displacement dose limit for these samples was only < 0.05 dpa (7.05×10^{17} H/cm^2), while polycrystalline samples survived up to 0.1 dpa plateau value (1.41×10^{18} H/cm^2).

TEM examination of irradiation microstructures on both single and polycrystalline UO_2 samples was scheduled for the end of 2013 at CAES, but due to TEM down-time, only a brief examination of the polycrystalline samples was performed. Compared to the irradiated He^{2+} samples, the H^+ irradiation produced more clear microstructures.

Most notably, dislocation loops with stacking fault natures were detected on materials irradiated to the highest fluence. (Figure 3a and 3b.)

In addition to the work described above, all the single and polycrystalline samples were measured using synchrotron-based X-ray absorption fine structure (EXAFS) at SSRL.

Future Activities

Work related to this rapid turnaround proposal that is scheduled to be completed in 2014 includes:

- Finalizing TEM work for H^+- and He^{2+}-irradiated UO_2 single- and polycrystalline samples.
- Submitting manuscripts [1] and [4], which both combine TEM and thermal conductivity measurements conducted at INL.

Microstructures of Low-Dose Helium (He^{2+}) and Hydrogen (H^{+}) Ion Irradiated Uranium Dioxide (UO_2) (cont.)

- Analyzing EXAFS data for the irradiated He^{2+} and H^{+} samples and presenting them to The Minerals, Metals and Materials Society (TMS) [3].
- New H^{+} irradiation at 600º C, with varying ion energies, followed by focused ion beam (FIB) sample preparation and microstructure characterization at CAES.

Publications and Presentations

[1] Janne Pakarinen, Lingfeng He, Marat Khafizov, Chris Wetteland, Jian Gan, Andrew Nelson, Anter El-Azab, Todd R. Allen, "Microstructure Changes and Thermal Conductivity Reduction in UO_2 Following 3.9 MeV He^{2+} Ion Irradiation " accepted with changes in *Journal of Nuclear Materials* (2014).

[2] Janne Pakarinen, Lingfeng He, Mahima Gupta, Jian Gan, Andrew Nelson, Anter El-Azab, Todd R. Allen, *Nuclear Instruments and Methods in Physics Research* 319, 100 (2014).

[3] Mahima Gupta, Janne Pakarinen, Steven Conradson, Jeff Terry, Lingfeng He, Jian Gan, Andrew Nelson, Todd R. Allen, *The Minerals Metals and Materials Society* (2014).

[4] Janne Pakarinen et. al., in preparation (2014).

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	PIE facilities
University of Wisconsin	Tandem Accelerator Ion Beam; Characterization Laboratory for Irradiated Materials
Collaborators	
Idaho National Laboratory Todd Allen (co-principal investigator), Jian Gan (collaborator), Marat Khafizov (collaborator)	
University of Wisconsin Janne Pakarinen (principal investigator), Lingfeng He (collaborator), Mahima Gupta (collaborator)	

Principal Investigator: Lin Shao – Texas A&M University
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Intercompound Formation and Radiation Responses of Diffusion Couples Made of Depleted Uranium and Metals

Introduction

A better understanding of the interaction between nuclear fuel and fuel cladding under extreme conditions will greatly enhance the operations of our current fleet of reactors, as well as next-generation reactors.

Project Description

This project is aimed at understanding intercompound formation and radiation responses of diffusion couples made of depleted uranium (U) and other metals, such as iron (Fe), zirconium (Zr), nickel (Ni), and chromium (Cr). The results will have a significant impact in several areas:

- Provide validation on phase diagram and phase field theories concerning U.
- Reveal radiation-induced structural changes of different phases.
- Accelerate our understanding of fuel-cladding interactions.

Accomplishments

U-bearing diffusion couples were successfully fabricated at Texas A&M University (TAMU), and the samples were shipped to INL for characterization (Figure 1). All intermetallic phases between U-Fe, U-Zr, U-Ni, and U-Cr were identified and compared with the existing phase diagrams. Agreements and discrepancies have all been documented in reports and manuscripts.

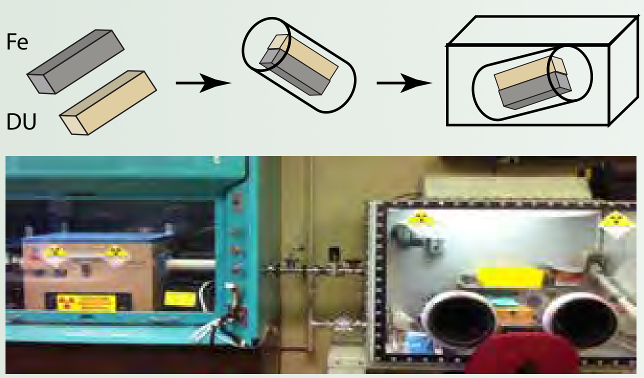
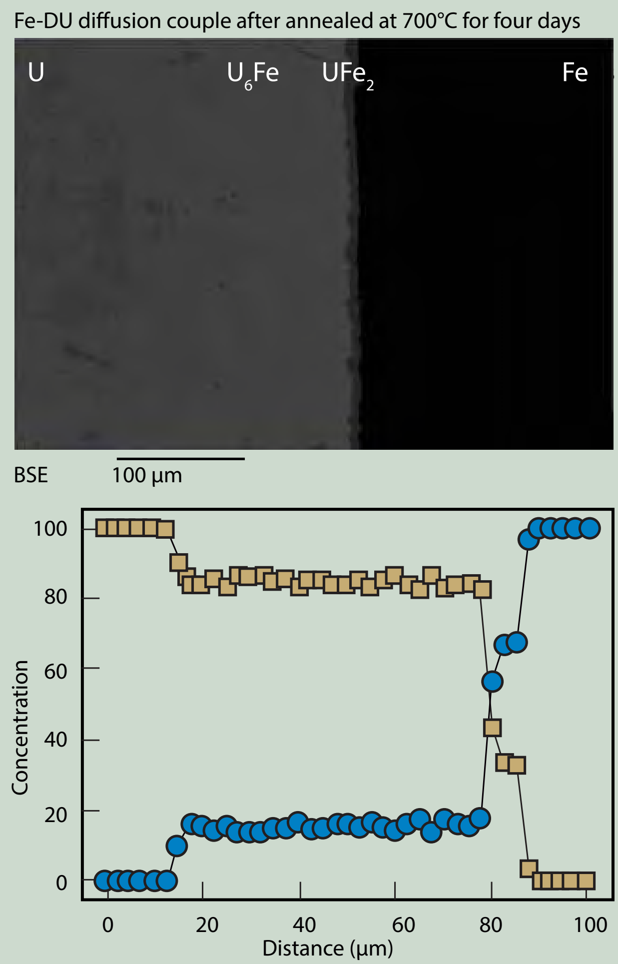


Figure 1. Fabrication of uranium-bearing diffusion couples at TAMU.

Using U-Fe as one example, U and Fe distributions were obtained across the interface and their interdiffusion coefficients were extracted (Figure 2). Fixed ion beam (FIB) lift-off technique was used to prepare the transmission electron microscopy (TEM) specimens for atomic scale characterization of crystalline structures and lattice parameters of the formed intermetallic phases U6Fe and UFe2 (Figure 3).

A better understanding of the interaction between nuclear fuel and fuel cladding under extreme conditions will greatly enhance the operations of our current fleet of reactors, as well as next-generation reactors.



Integrated interdiffusion coefficients of U

D^{U6Fe} (x 10-16 at .frac. m/s)	D^{UFe2} (x 10-16 at .frac. m/s)
3.31	1.89

Figure 2. Scanning electron microscopy (SEM) image and element distributions of U-Fe diffusion couple.

"This project would not be possible without access to the CAES facilities. The overall idea of user proposals, open access and a transparent selection process greatly helps university researchers and student training."

Lin Shao, Associate Professor of Nuclear Engineering, Texas A&M University

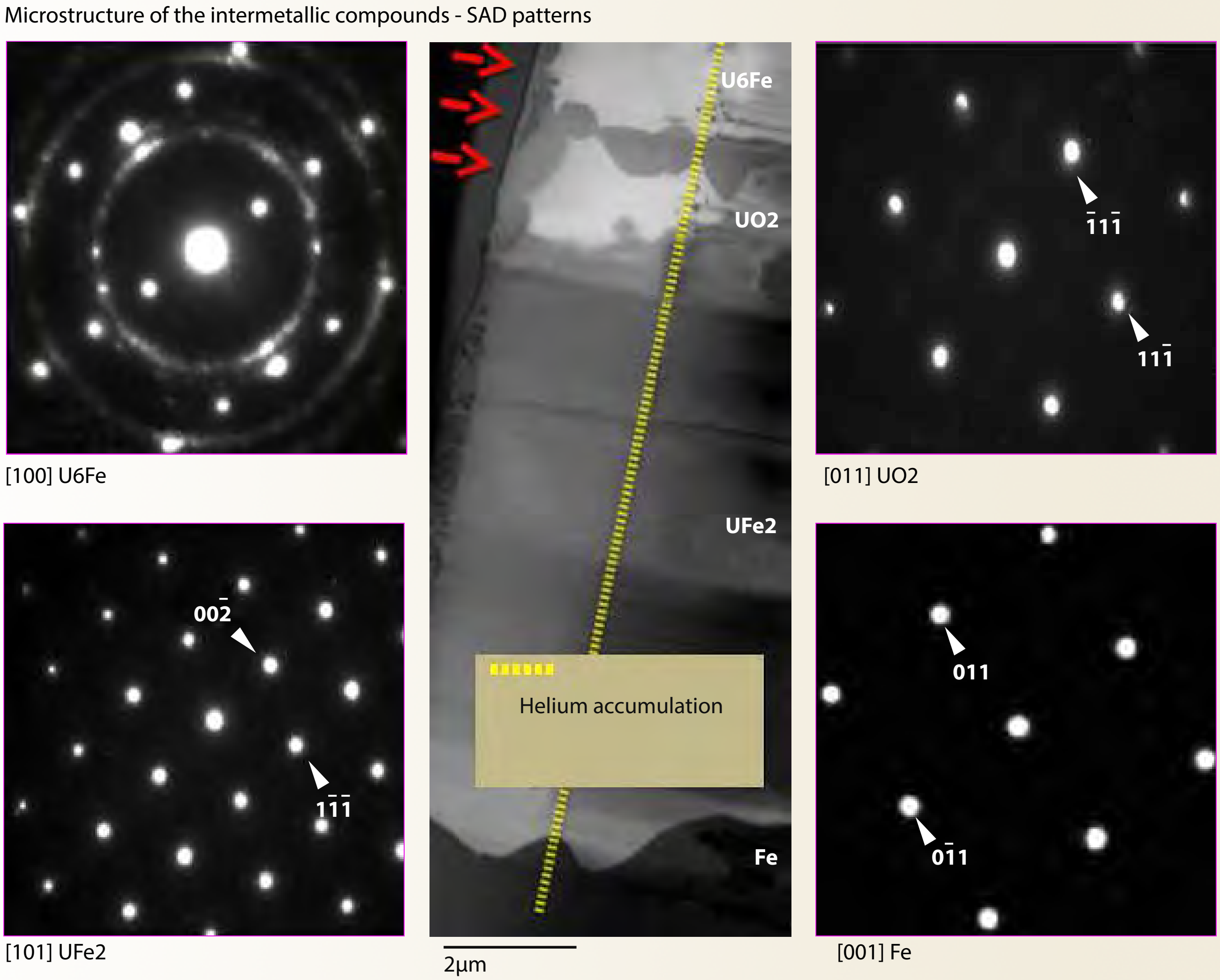


Figure 3. Cross-sectional TEM images and localized diffraction patterns of U-Fe diffusion couples.

The interface compounds and widths of each compound were measured as a function of annealing temperatures for each diffusion couple system, and the activation energies associated with interdiffusion coefficients, intrinsic growth constants, and extrinsic growth constants were extracted. A comparison study was performed between U-single-crystal metal and U-polycrystalline metal diffusion couples in order to understand the roles of grain boundaries in diffusion and interface phase formation. For example, we have shown that the formed U6Fe and UFe2 phases are narrower for U bonded with single-crystal Fe than those for U bonded with polycrystal Fe. Furthermore, the activation energies for interdiffusion are higher in single-crystal Fe (Figure 4). The findings suggest that grain boundaries act as quick diffusion paths to promote interface reactions.

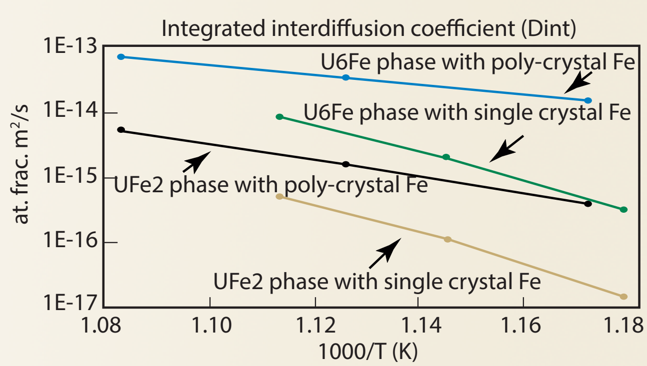


Figure 4. Interdiffusion coefficients as a function of temperature for diffusion couples bonded between U and single-crystal Fe, and bonded between U and polycrystal Fe. The difference shows that activation energy for interdiffusion is higher in single-crystal Fe due to a lack of grain boundaries that can act as quick diffusion paths.

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Intercompound Formation and Radiation Responses of Diffusion Couples Made of Depleted Uranium and Metals (cont.)

Helium (He) ion irradiations were performed for selected diffusion couple systems, and compound width changes were measured to evaluate the effects on interdiffusion and interface phase formation. Results showed that interface reactions were significantly increased (Figure 5).

FIB and TEM were performed on selected diffusion couple systems both before and after ion irradiation to characterize the void nucleation in different interface phases. Results showed that in different intermetallic phases, radiation tolerance and swelling resistance are significantly different. For example, He ion irradiation creates smaller voids with higher densities, while in UFe2 phase the same irradiation leads to larger voids with lower densities (Figure 6).

For selected diffusion couple systems the nanoindentation technique was used to induce both hardness and Young's modulus changes across the interface. The mechanical properties of each phase were characterized and compared with literature data.

Future Activities

In 2014 the project will extend into diffusion couples formed between single-crystal U and single-crystal Fe, Ni, and Cr, with fission products loaded in the U side. The key is to compare the difference in diffusion kinetics with or without influences from grain boundaries, and to understand the complexity caused by fission products.

Publications and Presentations

Chao-Chen Wei, Assel Aitkaliyeva, Zhiping Luo, Ashley Ewh, Yongho Sohn, J. Rory Kennedy, Butlent H. Sencer, Michael T. Myers, Michael Martin, Joseph Wallace, Michael J. General, Lin Shao, "Understanding the Phase Equilibrium and Irradiation Effects in Fe-Zr Diffusion Couples," Journal of Nuclear Materials, Vol. 432, No. 1-3, January 2013, pp. 205-211.

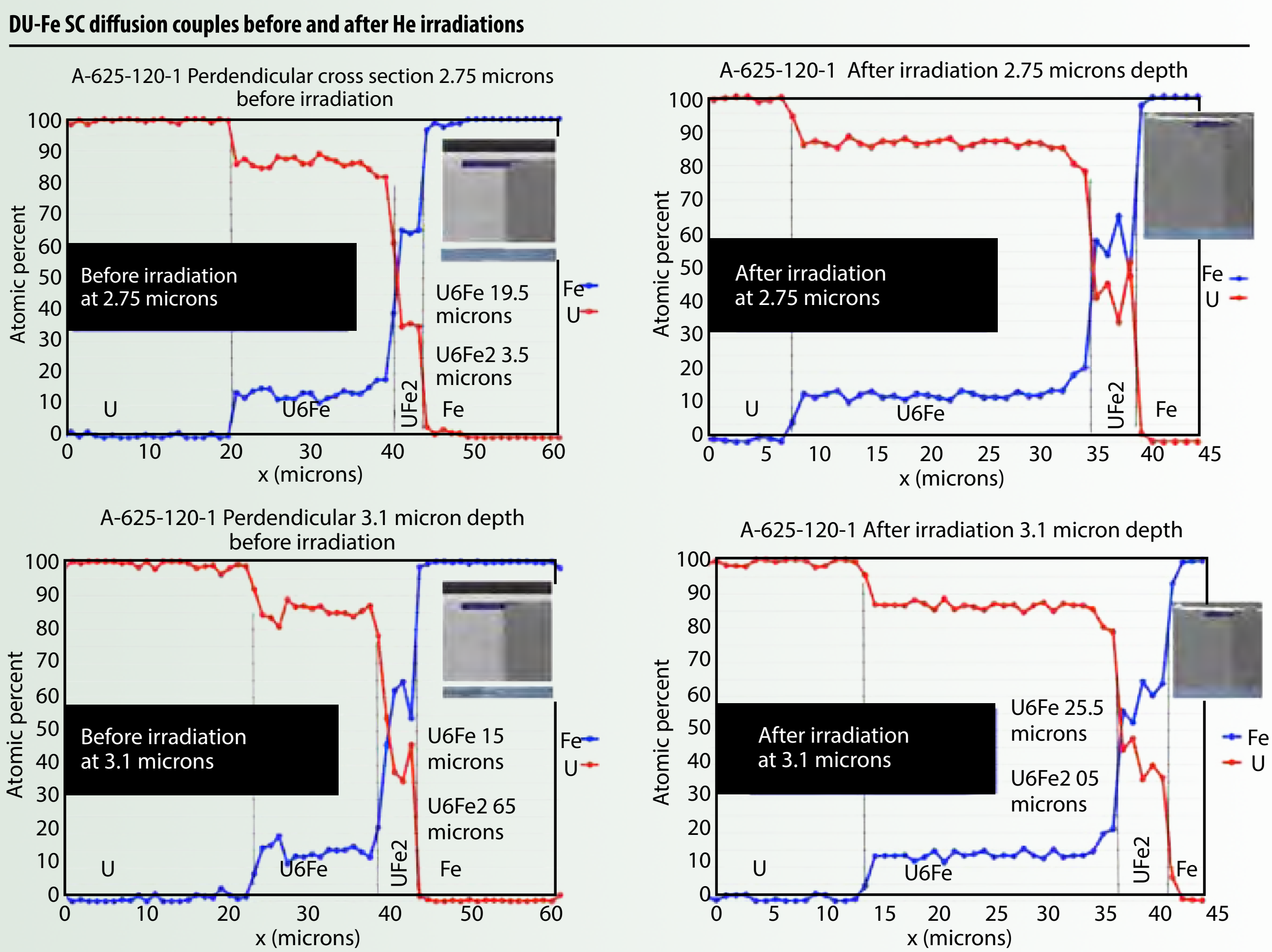

Figure 5. Cross-sectional SEM images and interface compound formation at different depths before and after 2 MeV He ion irradiation. The wider compound zones that appear after ion irradiation show the radiation-enhanced diffusion.

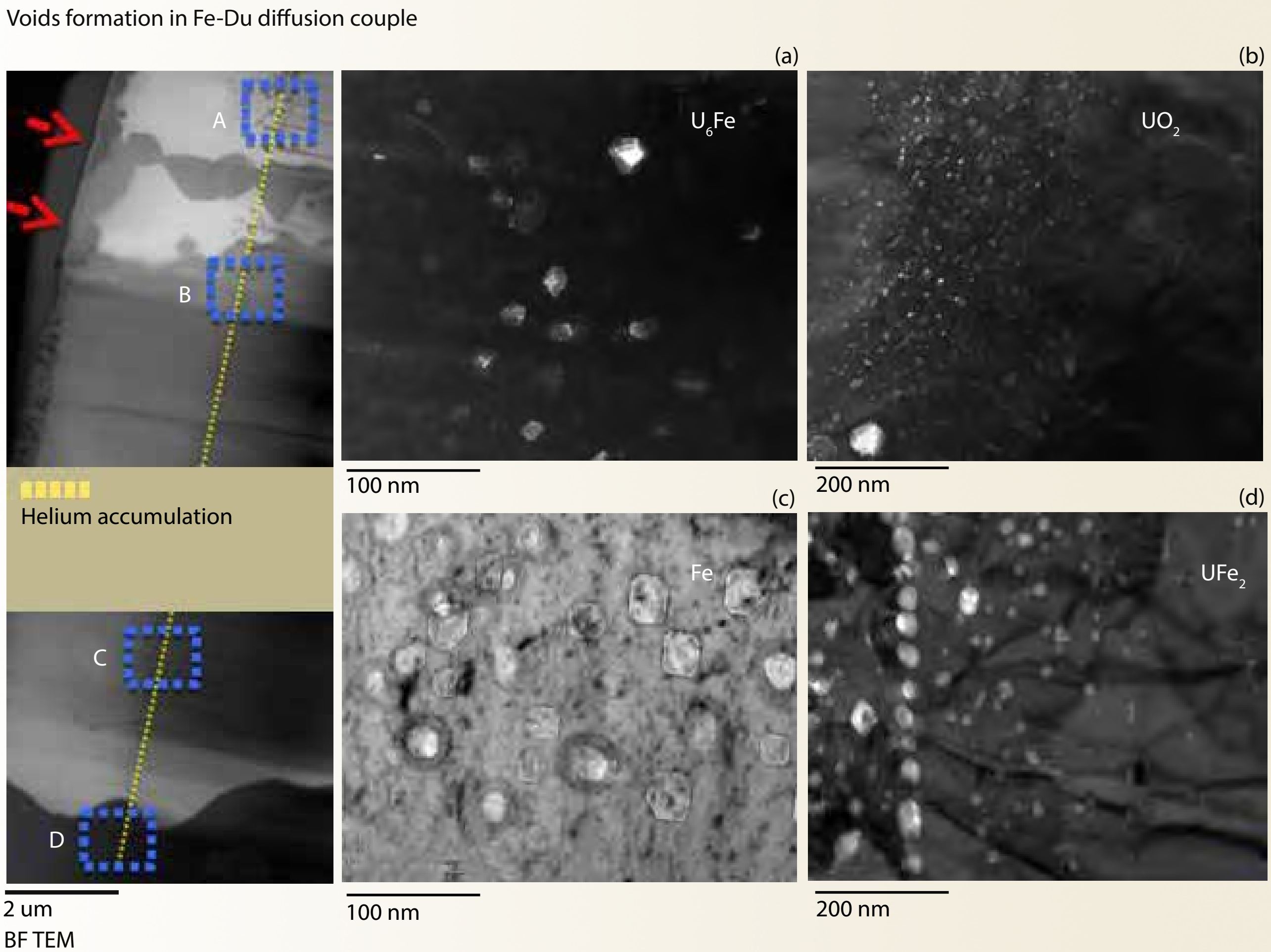

Figure 6. Cross-sectional TEM images of void formation in different intermetallic phases of U-Fe diffusion couples.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Collaborators	
Idaho National Laboratory Bulent H. Sencer (co-principal investigator), Assel Aitkaliyeva (collaborator, previously a Ph.D. student at Texas A&M University) **Texas A&M University** Lin Shao (principal investigator), Tianyi Chen (collaborator)	

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Low-Fluence Behavior of Metallic Fuels

Introduction

Quantified findings for low-fluence behavior of metallic fuels will increase our understanding of fuel performance under thermal and irradiation conditions. This systematically designed ATR NSUF research project provides critical low-fluence data for fuels where none currently exists.

Project Description

The overall objectives of the program are to understand (1) the microstructural evolution of these fuels as a function of temperature, fluence and composition, and (2) the diffusion-related phenomena in the fuels and cladding, also as a function of temperature, fluence and composition. Low-fluence experiments in metallic fuels, specifically uranium-zirconium (U-Zr) and uranium-molybdenum (U-Mo) types, have a cross-cutting relevance to both the Advanced Fuel Cycle Initiative (AFCI) and the Reduced Enrichment for Research and Test Reactors (RERTR) program. Findings in these experiments will explain early microstructural development and mechanisms in detail, as well as provide critical data for models under development in both programs.

Near term, critical results from this project will support AFCI modeling work on constituent redistribution in irradiated uranium-prasecoymium-zirconium (U-Pr-Zr) fuels that is currently being undertaken by collaborators on this team. These results will also be used in part to improve the overall accuracy of the computer models in the RERTR program that predict the overall swelling behavior of the fuel.

The purpose of characterizing the irradiation-induced microstructural evolution in U-Zr or U-Mo is to understand the effects of temperature, fluence and alloying constituents on the thermal and mechanical properties within the fuels. The emphasis of the characterization is on defect formation and microstructure (e.g., constituent redistribution, fission gas bubbles, pore development, dislocations and phases). Low-fluence testing of prefabricated diffusion couples is carried out to develop a fundamental understanding of constituent redistribution in fuels and fuel-cladding interactions that are accelerated by irradiation.

Accomplishments

For the investigation of constituent redistribution, the homogeneous fuel alloys U-Zr and U-Mo were sectioned into thin disks and diffusion-bonded with inert markers in order to determine the relevant thermokinetic parameters on intrinsic frames of reference. Irradiation-enhanced fuel-cladding interaction was also examined using intrinsic

Findings from the tested specimens will provide valuable knowledge to the fundamental science of nuclear materials, as well as properties of materials that will be critical to the development of predictive fuel performance modeling.

frames of reference, with U-Zr versus iron (Fe), iron-nickel (Fe-Ni), and iron-chromium (Fe-Cr) diffusion couples, as well as U-6Mo, U-8Mo, U-10Mo, U-12Mo versus aluminum (Al) diffusion couples. All experiments will be compared to out-of-pile testing. Findings from the tested specimens will provide valuable knowledge to the fundamental science of nuclear materials, as well as the properties of materials that will be critical to the development of predictive fuel performance modeling.

To date researchers have completed the preparation and processing of materials according to the specifications required for irradiation testing, including transmission electron microscopy (TEM)-ready samples and diffusion couples. The documentation of out-of-pile testing, including materials characterization and diffusion couples, is also complete (Figures 1-3).

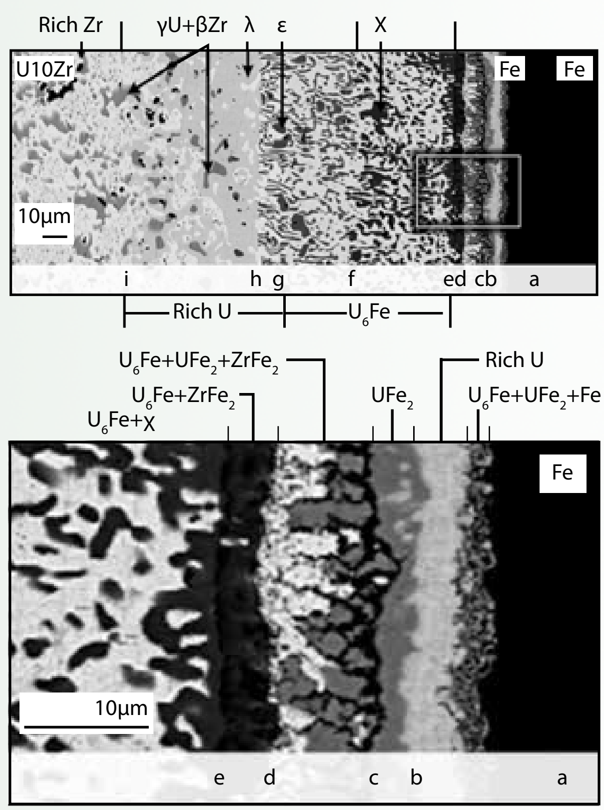

Figure 1. Backscattered electron micrographs from U-10wt.% Zr versus Fe diffusion couple annealed at 953K for 96 hours.

"My interaction as a graduate student with ATR NSUF has been truly rewarding. It gave me an opportunity to strengthen my science fundamentals, hone my hands-on laboratory skills, and gain a greater perspective on energy research with respect to application and societal impact. It is a great program in which graduate research assistants can cover the entire spectrum of R&D."

Ashley Paz y Puente (formerly Ashley Ewh), M.S. Graduate Research Assistant, University of Central Florida

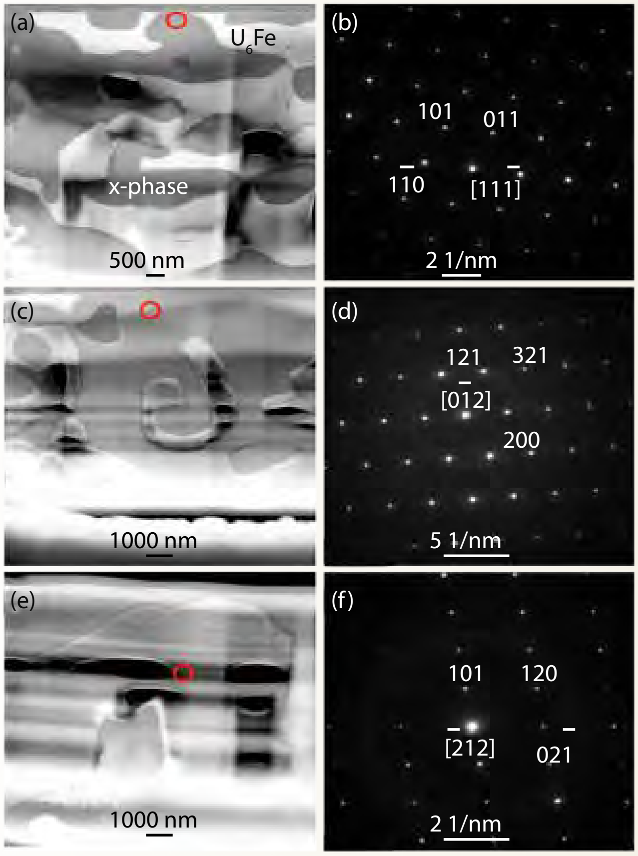

Figure 2. The complex ternary phases, Fe(Zr,U)-χ (oP8), Fe(Zr, U) $_2$-ε (tI12) and U_3(Zr,Fe))-λ (tI12) phases developed in the U-10wt.% Zr versus Fe diffusion couple annealed at 953K. TEM analysis via sample preparation with FIB in-situ lift-out (INLO) has identified their crystal structures for the first time.

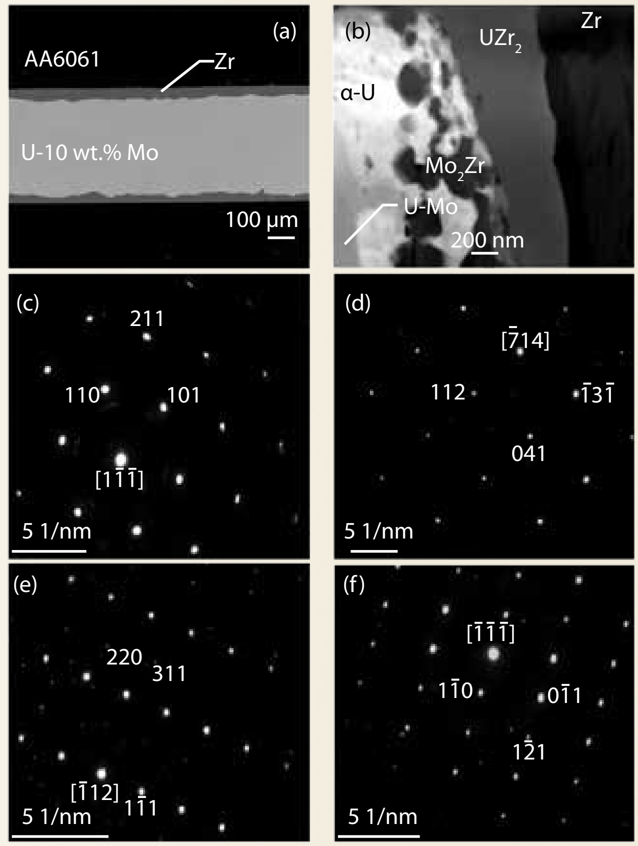

Figure 3. (a) and (b) Typical backscatter electron micrograph of the U-10wt.%Mo monolithic fuel interaction with the Zr diffusion barrier, with reaction kinetics (e.g., thickness). (c-f) Phase identification by TEM analysis via sample preparation with FIB-INLO. Selected area electron diffraction patterns from (c) γ-U(Mo) solid solution, (d) α-U, (e) Mo_2Zr, and (f) UZr_2 phases at the interface between U-10wt.%Mo monolithic fuel and the Zr diffusion barrier.

Future Activities

Complete materials processing of all samples for irradiation testing at ATR NSUF is expected to be finished in 2014, as is the characterization of alloys and diffusion couples, at which time Phase I irradiation testing will begin.

Publications and Presentations*

1. Ke Huang, Dennis D. Keiser, Jr., Yongho H. Sohn, "Interdiffusion, Intrinsic Diffusion, Atomic Mobility, and Vacancy Wind Effects in γ (bcc) Uranium-Molybdenum Alloy," *Metallurgical and Materials Transactions A,* Vol. 44A, No. 2, February 2013, pp. 738-746.
2. Ke Huang, Helga Heinrich, Dennis D. Keiser, Jr., Yongho H. Sohn, "Fuel-Matrix Chemical Interaction Between U-7wt.%Mo Alloy and Mg," *Defects and Diffusion Forum,* Vol. 333, 2013, pp. 199-206.
3. Ke Huang, Youngjoo Park, Dennis D. Keiser, Jr., Yongho H. Sohn, "Interdiffusion between Zirconium Diffusion Barrier and Uranium-Molybdenum Alloy," *Journal of Phase Equilibria and Diffusion,* Vol. 33, No. 6, December 2012, pp. 443-449.
4. Ke Huang, Youngjoo Park, Dennis D. Keiser, Jr., Yongho H. Sohn, "Interdiffusion between Potential Diffusion Barrier Mo and U-Mo Metallic Fuel Alloy for RERTR Applications," *Journal of Phase Equilibria and Diffusion,* Vol. 34, No. 4, August 2013, pp. 307-312.
5. Emmanuel Perez, Yongho H. Sohn, Dennis D. Keiser, Jr., "Role of Si on Diffusional Interaction between U-Mo and Al-Si Alloys at 823K (550 C)," *Metallurgical and Materials Transactions A*, Vol. 44A, No. 1, January 2013, pp. 584-595.

Low-Fluence Behavior of Metallic Fuels (cont.)

6. Chao-Chen Wei, Assel Aitkaliyeva, Zhiping Luo, Ashley Ewh, Yongho H. Sohn, J. Rory Kennedy, Bulent H. Sencer, Michael T. Myers, Michael Martin, Joseph Wallace, Michael J. General, Lin Shao, "Understanding the Phase Equilibrium and Irradiation Effects in Fe-Zr Diffusion Couples," *Journal of Nuclear Materials,* Vol. 432, No. 1-3, January 2013, pp. 205–211.

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor
Collaborators	
Idaho National Laboratory Maria A. Okuniewski (co-principal investigator), Dennis D. Keiser, Jr. (co-principal investigator) **Seoul University of Science and Technology** Jungwon Kang (collaborator) **University of Central Florida** Yongho Sohn (principal investigator), William Sprowes (collaborator), Nicholas Eriksson (collaborator), Youngjoo Park (collaborator), Ke Huang (collaborator), Ashley Paz Puente (formerly Ashley Ewh) (collaborator), Mian Fu (collaborator)	

Principal Investigator: Kumar Sridharan – University of Wisconsin–Madison
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Study of Interfacial Interactions using Thin Film Surface Modification: Radiation and Oxidation Effects in Materials

Introduction

Interfaces such as grain boundaries and precipitate-matrix boundaries play a profound role in the stability of material under irradiation by influencing elemental segregation and acting as sites where recombination of radiation-induced defects and high-diffusivity paths can occur. Therefore, the development of radiation-resistant materials is crucial to enhancing the safety, reliability and life-extension of nuclear reactors.

In this regard, nanograined materials and oxide dispersion-strengthened (ODS) steels are being actively investigated.

Project Description

The goal of this ATR NSUF research project is to investigate the chemical and morphological stability of interfaces under radiation.

Improved performance of nanograined materials and ODS steels in radiation fields is predicated on a large number of interfaces being present in these materials. Therefore, it is imperative that these interfaces remain stable under radiation.

For this project, researchers are investigating the stability of interfaces in a fundamental way: by irradiating thin films on substrate systems and then examining the chemical and morpohological changes at the film substrate interface. This heavy ion irradiation is used as a surrogate for in-reactor neutron radiation.

Because radiation-induced changes occur on nanometer length scales, advanced transmission electron microscopy (TEM) and scanning TEM (STEM) techniques, coupled with energy dispersive X-ray spectroscopy (EDS), were used to examine the interfaces and arrive at quantifiable metrics for radiation-induced changes at the interfaces. Thin films investigated in the study include yttrium (Y), titanium (Ti), and their respective oxides (Y_2O_3 and TiO_2), while the substrate was a binary Fe-12%Cr ferritic alloy. This means the research is relevant to nanostructured ODS ferritic steels that are strengthened by (Y, Ti) oxide nanoparticles.

Understanding the stability of interfaces under high-temperature irradiation will be a significant step in the development of radiation-resistant materials.

Accomplishments

The film-substrate system was irradiated with 5 MeV nickel (Ni) ions at 300°, 500° and 700° C. During irradiation, a small fraction of the sample surface was masked from the ion beam. This thermally exposed region was not irradiated but still experiences the same thermal history as the irradiated area. This allowed researchers to isolate thermal and irradiation-induced effects at the interfaces

Detailed examinations of the film-substrate interfaces were performed using advanced TEM and STEM facilities at CAES. A previous report presented the results of work with Y and Ti films, and this study has extended the work to Y_2O_3 and TiO_2 films.

The oxide-coated samples exhibited different behaviors than what was observed in those coated with metallic films. For the pure metal-coated films, severe intermixing between the substrate and the thin films was observed following 500° C and 700° C irradiations. However, for oxide films irradiated under the same conditions, a sharp interface devoid of intermixing effects was observed.

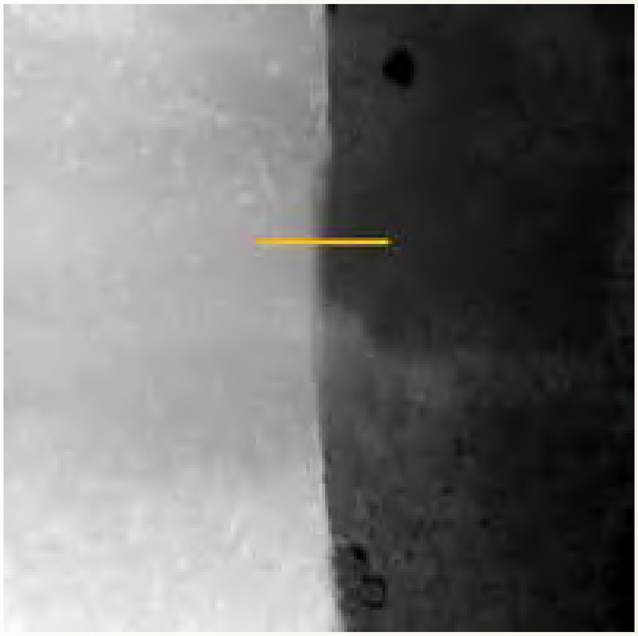
TiO_2-substrate interfaces were seen to be more stable under thermal and radiation exposure than Y_2O_3-substrate interfaces, although for both oxides the external surface exhibited a propensity to sputter during 500° C and 700° C irradiations.

Despite the formation of some chromium (Cr)-rich nonprecipitates, the TiO_2 substrate interface was still sharp. In the Y_2O_3 substrate samples, the formation of a Cr-rich layer at the interface was relatively suppressed. As an example, Figure 1 shows the STEM-EDS analysis performed for the TiO_2 and Y_2O_3 samples irradiated and thermally exposed at 500° C.

“Advanced high-resolution, electron microscopy techniques, such as those used in this study, display the physical and chemical changes at interfaces in unprecedented detail, which allows us to tailor materials with enhanced resistance to radiation damage.”

Kumar Sridharan, Distinguished Research Professor, University of Wisconsin – Madison

(a)



100 nm

(b)

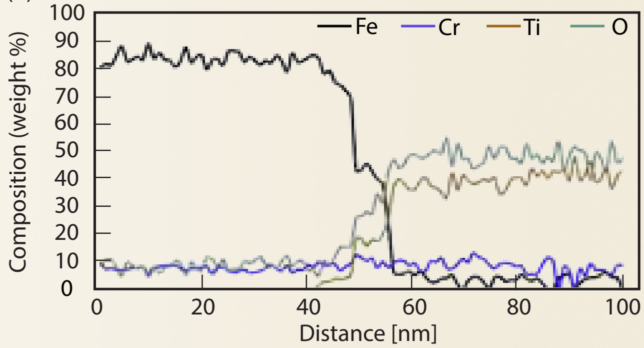
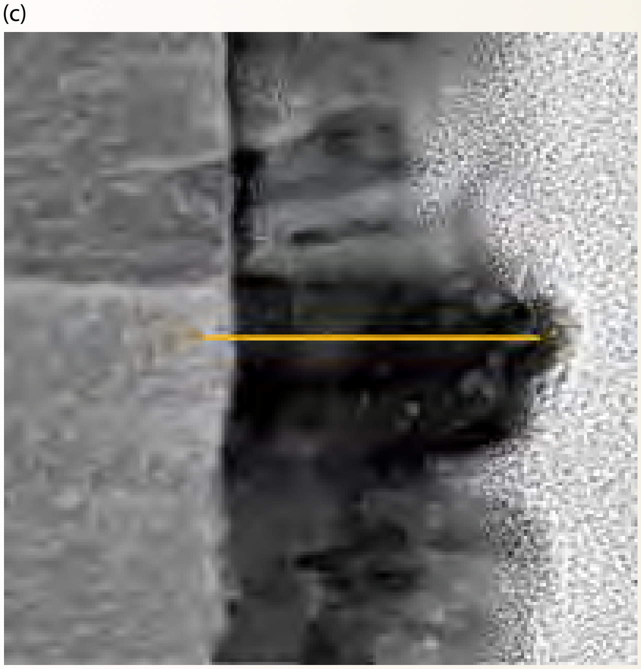


Figure 1. STEM images and EDS elemental line scans of the TiO_2 and Y_2O_3 samples irradiated with 5 MeV Ni ions at 500° C for:

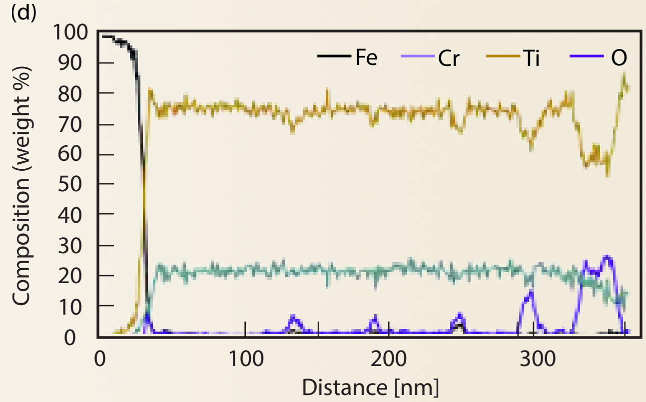
(a and b) thermally exposed TiO_2 -coated samples;

(c)



200 nm

(d)



(c and d) irradiated TiO_2-coated samples;

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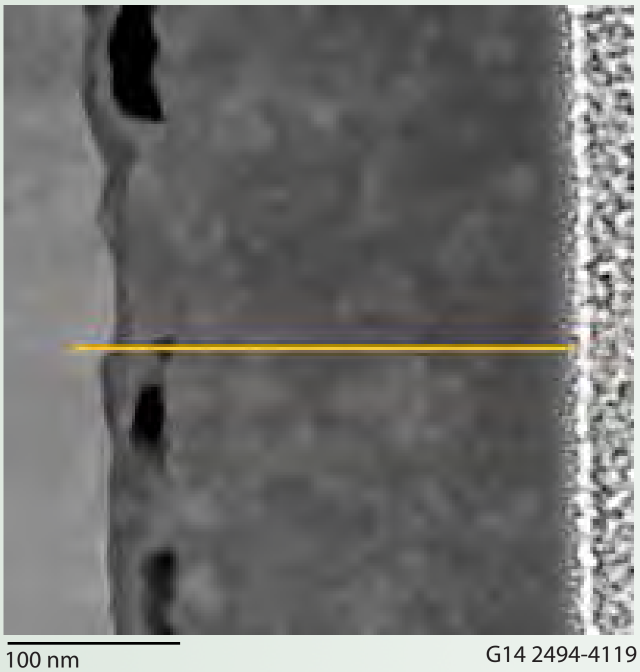
Study of Interfacial Interactions using Thin Film Surface Modification: Radiation and Oxidation Effects in Materials (cont.)

(e)



(f)

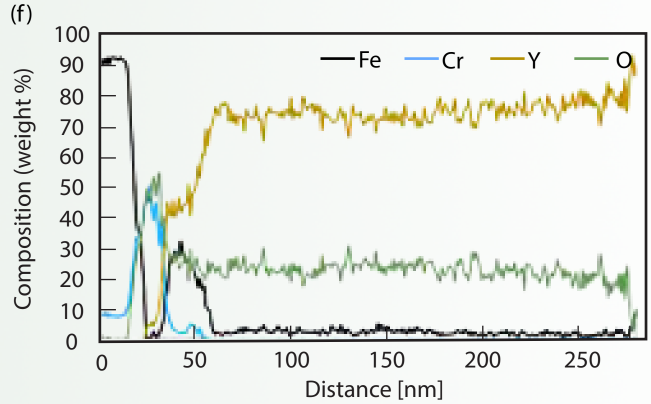
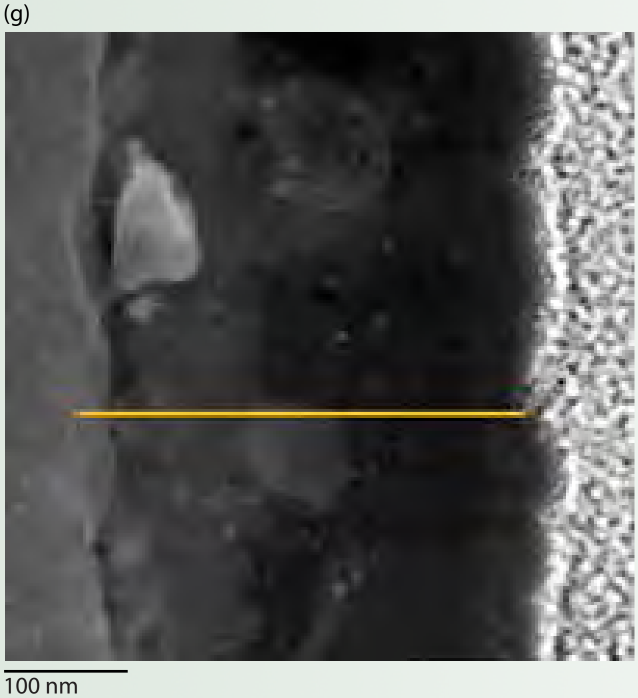
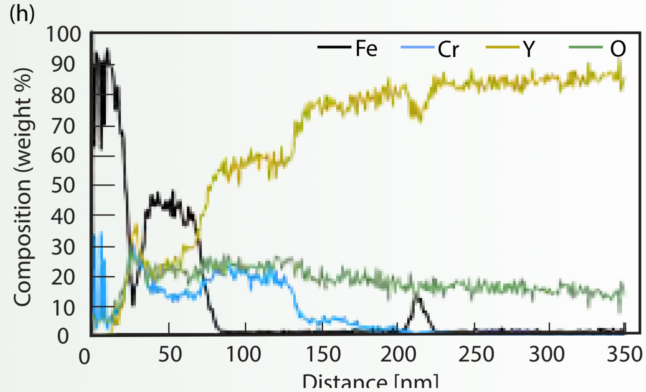


Figure 1. (cont.) STEM images and EDS elemental line scans of the TiO2 (e and f) thermally exposed Y2O3-coated samples; and

(g)



(h)



(g and h) irradiated Y_2O_3 samples. Horizontal lines in the images represent the EDS line-scan distances in the interface regions (the region on the left side of the images is the substrate).

Future Activities

Future work will focus on determining the composition of the precipitates that form as a result of ion irradiation. Additionally, we will study the effects of dose and dose rate on the evolution phases at the interfaces in these film-substrate systems. These studies will be performed using noble metal ions, such as gold or platinum, at an irradiation temperature of 300° C and three different doses and dose rates. Irradiation with heavy noble metal ions will allow researchers to achieve very high doses in short times and confine the irradiated ions to regions closer to the interface.

Publications and Presentations*

1. Alexander Mairov, Benjamin Hauch, Clarissa Yablinsky, and Kumar Sridharan, "Study of Interfacial Interactions Using Thin Film Surface Modification," *Transactions of The American Nuclear Society* 106, pp. 1270 (2012).

2. Alexander Mairov, Benjamin Hauch, Kumar Sridharan, Todd Allen, and Jinsuo Zhang, "Study of Interfacial Interactions Using Thin Film Surface Modification," oral presentation, *Materials Science & Technology (MS&T) Conference, Pittsburgh, Pennsylvania, October 2012.*

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor
Collaborators	
Los Alamos National University Jinsuo Zhang (collaborator)	
University of Wisconsin Kumar Sridharan (principal investigator), Alexander Mairov (post-doctoral candidate), Benjamin Hauch (research intern), Clarissa Yablinsky (post-doctoral candidate)	

Principal Investigator: Kumar Sridharan – University of Wisconsin–Madison
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Effect of Radiation and Temperature on the Stability of Oxide Nanoclusters in Oxide-Dispersion-Strengthened Steel

Introduction

Oxide-dispersion-strengthened (ODS) steels are being actively considered as fuel cladding for the next generation of fission reactors. ODS steels have fine, nanoscale-sized yttrium-titanium (Y-Ti) oxide particles dispersed in a ferritic matrix.

These steels are produced by mechanical alloying of the initial elements followed by consolidation using hot isostatic pressing or hot extrusion, ODS steels have superior creep rupture strength compared to conventional ferritic steels due to the presence of the (Y-Ti)-O nanoparticles, which impede dislocation movement and provide the same higher swelling resistance that is found in ferritic steels.

Furthermore, the nanoparticles in ODS steels improve resistance to radiation damage by acting as sinks for helium (He) and recombination sites for point defects that form during irradiation. The chemical and morphological stability of the oxide nanoparticles under radiation is crucial to achieving the superior performance of ODS steels.

Project Description

In the present study, atom probe tomography (APT) is used to systematically study the synergistic effects of He implantation, irradiation and temperature on the stability of the nanoclusters in 14YWT ODS ferritic steel. The use of APT enables the successful imaging of nanoclusters smaller than 2 nm and provides high-spatial resolution and excellent elemental identification. More importantly, APT provides a direct three-dimensional (3D) reconstruction of nanoclusters on an atomic scale, allowing for the accurate determination of the composition of both nanoclusters and the matrix.

Accomplishments

A realistic assessment of radiation damage in ODS steels can be made by creating defects in the presence of He. To that end, researchers working at the University of Michigan's Ion Beam Laboratory implanted He in the ODS steel at room temperature using 270 KeV and 200 KeV energies to achieve a uniform concentration of 2,000 atomic parts per million (appm) of He below the steel's surface. The He implanted samples were irradiated with 5 MeV nickel (Ni) ions at 300°, 450° and 600° C to a high dose of 100 displacements per atom (dpa). The samples were analyzed using the APT technique at CAES.

A fundamental understanding of the stability of nanostructures in ODS steels under irradiation is critically important for the application of these steels in nuclear reactors.

A high density of nanoclusters with enrichments of titanium, oxygen and yttrium are observed in 300°, 450° and 600° C samples as shown in Figure 1. The average size distribution and density of nanoclusters for each condition are shown in Figure 2. The average Guinier radii <Rg> of samples irradiated at 300°, 450° and 600° C were 1.02, 0.96 and 1.03 nm, respectively, and the number densities (Nv) were 10×10^{23}, 10×10^{23}/cu.m and 6.7×10^{23}, respectively, indicating that irradiation did not significantly affect the nanoclusters.

Future Activities

The results of this study will be compared to the stability of nanoclusters in ODS steel samples that have been neutron irradiated at ATR.

Publications and Presentations

1. Jianchao He, Farong Wan, Kumar Sridharan, Todd Allen, Alicia Certain, Vaithiyalingam Shutthanandan, Yaqiao Wu, "Stability of Nanoclusters in 14YWT Oxide Dispersion Strengthened Steel under Heavy Ion Irradiation by Atom Probe Tomography," submitted to Journal of Nuclear Materials, 2013.
2. Jianchao He, Farong Wan, Kumar Sridharan, Todd Allen, Alicia Certain, Yaqiao Wu "Response of 9Cr-ODS Steel to Proton Irradiation at 400° C," submitted to *Journal of Nuclear Materials,* 2013.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
University of Michigan	Michigan Ion Beam Laboratory
Collaborators	
University of Wisconsin Kumar Sridharan (principal investigator), Jianchao He (collaborator)	

"ODS steels possess a good combination of radiation resistance and creep-rupture strength, and therefore show excellent potential as fuel-cladding material for the next generation of nuclear reactors."

Kumar Sridharan, Distinguished Research Professor, University of Wisconsin – Madison

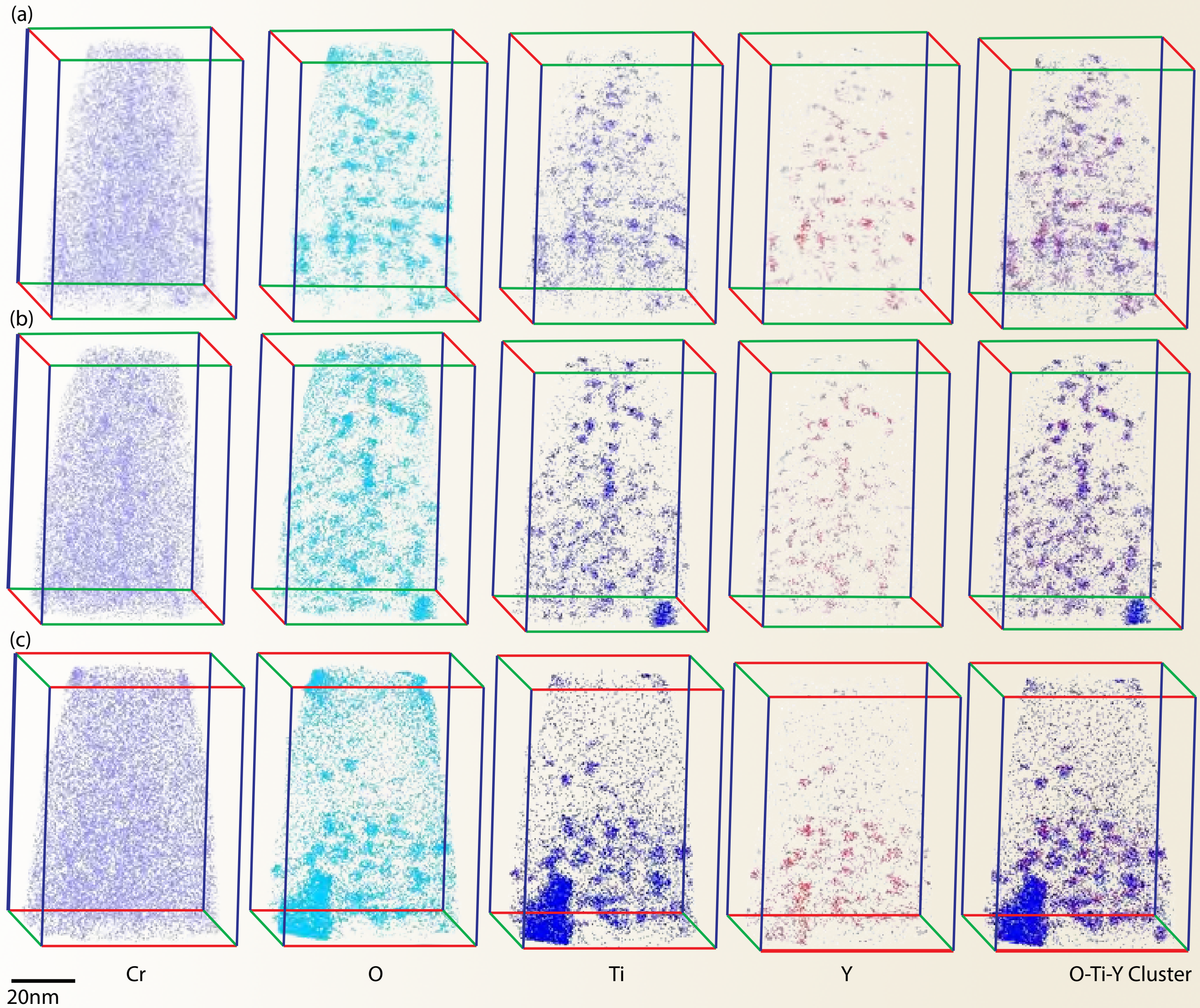

Figure 1. APT 3D reconstructions of 14YWT samples pre-implanted with He and Ni2+ irradiated up to 100 dpa at (a) 300° C, (b) 450° C, and (c) 600° C.

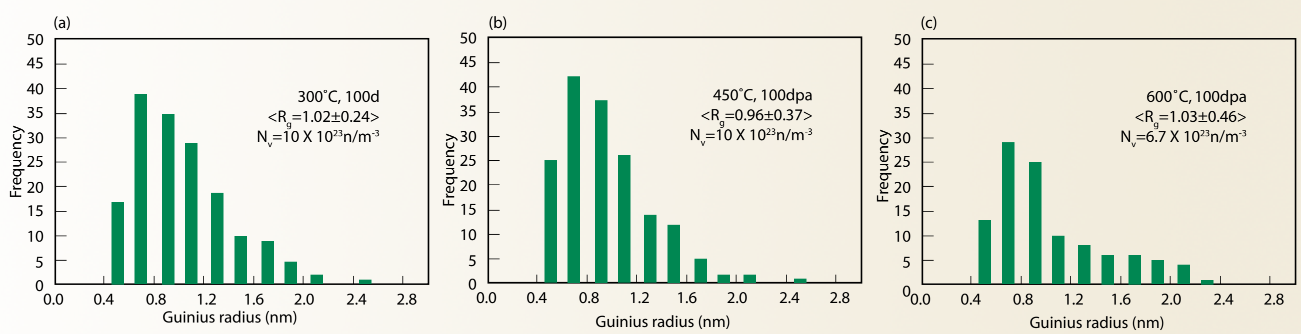

Figure 2. Charts showing nanocluster size distributions in samples irradiated at (a) 300° C, (b) 450° C, and (c) 600° C, respectively.

Transmission Electron Microscopy Investigation of Ion-Irradiated Uranium Oxide

Principal Investigator: Kumar Sridharan – University of Wisconsin–Madison
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Introduction

Krypton (Kr) is one of the primary fission gases found in UO_2. The crystal defects (i.e. dislocations) and precipitates (i.e. gas bubbles) induced in UO_2 by Kr directly affect the structure and thermal transport properties of UO_2 during both reactor operation and long-term storage. It is therefore important to understand and model the behavior of these dislocations and gas bubbles if we are to develop optimized fuel microstructures that produce improved fuel performance.

Project Description

Initiated in 2012, this ATR NSUF rapid turnaround experiment is a collaborative effort of Idaho National Laboratory, Argonne National Laboratory and the University of Wisconsin. It is intended to clarify the microstructure and stoichiometry of the standard nuclear fuel uranium oxide (UO_2) before and after irradiation.

Accomplishments

To simulate fission fragment damage to UO_2, single-grain and polycrystalline UO_2 samples were irradiated with 150-keV and 1-MeV Kr. The research team used a focused ion beam (FIB) to prepare cross-section lamina of the irradiated samples followed by post-irradiation examination (PIE) using transmission electron microscopy (TEM) to study the extended defects, including bubble dislocation loops, dislocation lines and stoichiometry.

PIE showed that the irradiated specimens were relatively denuded of dislocations within 20 – 30 nm of the surface, although the average damage to this region was still above 10 displacements per atom (dpa), indicative of the sink effect of the surface on defects (Figure 1).

The nature of the dislocation loops can be determined through a Burgers vector analysis (Figure 2). Gas bubble size depends largely on the irradiation temperature rather

The extended defects induced by fission gases have a great impact on the thermal transport capabilities and the lifetimes of UO_2 fuels in light water reactors.

than ion dose or energy (Figure 3). There are no zones denuded of bubbles along the grain boundaries (Figure 3). The branching ratio of the M-edge found through electron energy loss spectroscopy (EELS) ranges between 0.691 – 0.715:1, which, for the most part, overlaps the range of U^{4+}, implying that stoichiometry is unaffected in UO_2 under Kr irradiation (Figure 4).

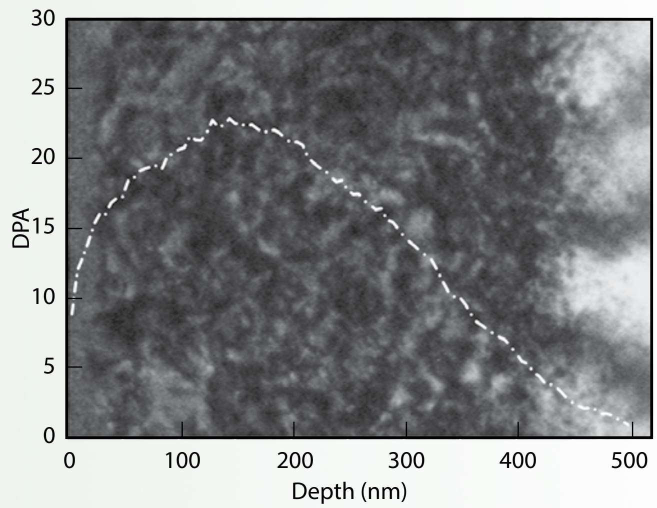

Figure 1. Bright field TEM image showing the cross section of UO_2 single crystal irradiated with 1-MeV Kr ions at 800° C to a dose of $5{\times}10^{15}$ ions/cm^2. The plot shows the Stopping and Range of Ions in Matter (SRIM) calculation of the damage profile.

"During these rapid turnaround experiments, we have made many exciting scientific discoveries about nuclear fuels using the cutting-edge facilities in the Microscopy and Characterization Suite at CAES."

Lingfeng He, Research Associate, University of Wisconsin – Madison

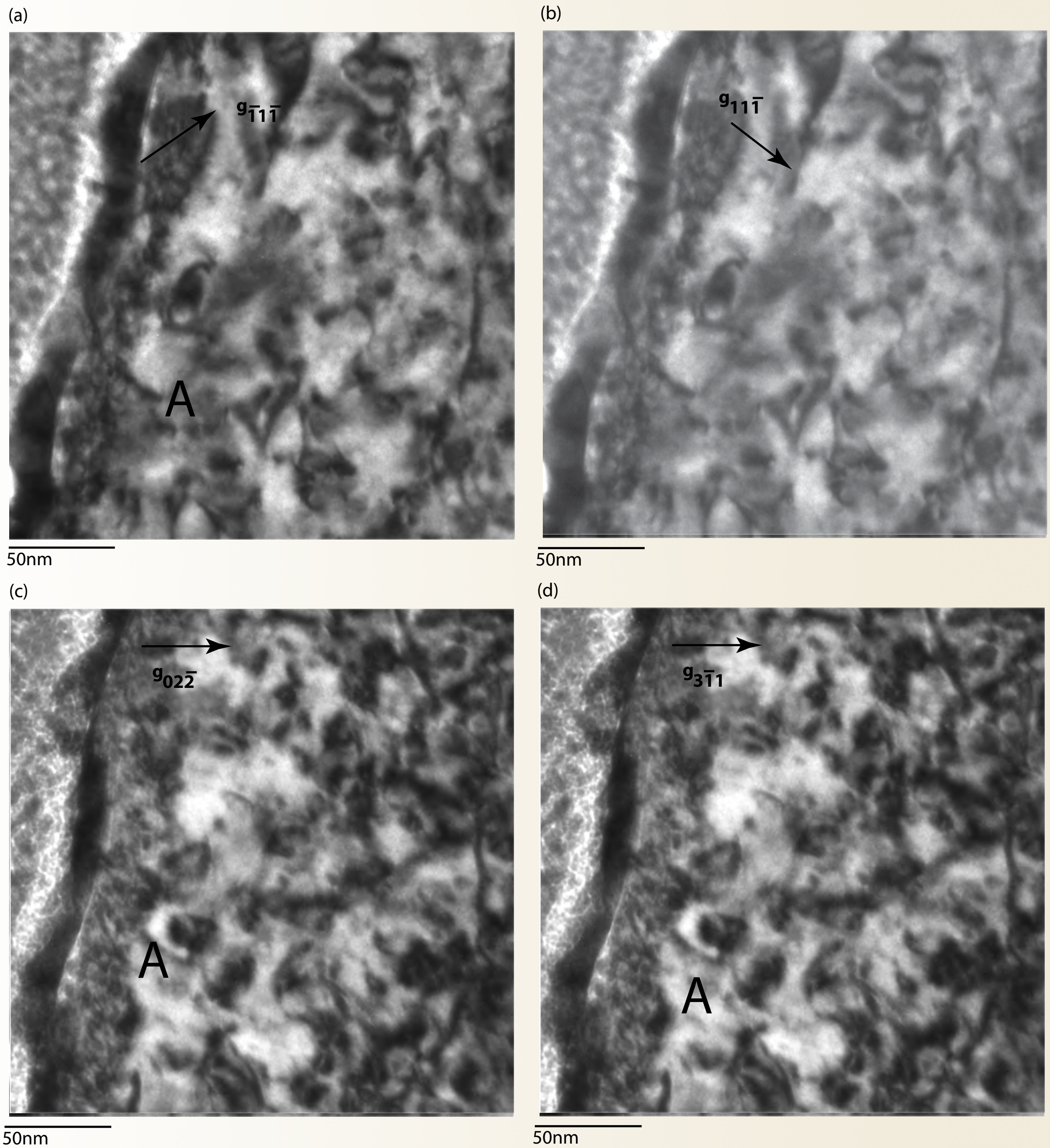

Figure 2. Series of TEM images used for part of the dislocation loop analysis in the UO_2 single crystal irradiated with 1-MeV Kr ions at 800° C to a dose of 5×10^{15} ions/cm^2. The arrows denote the direction of g: (a) g = $[\bar{1}1\bar{1}]$; (b) g = $[11\bar{1}]$; (c) g = $[02\bar{2}]$; and (d) g = $[3\bar{1}1]$. In each analysis, the beam direction is close to [001].

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Transmission Electron Microscopy Investigation of Ion-Irradiated Uranium Oxide (cont.)

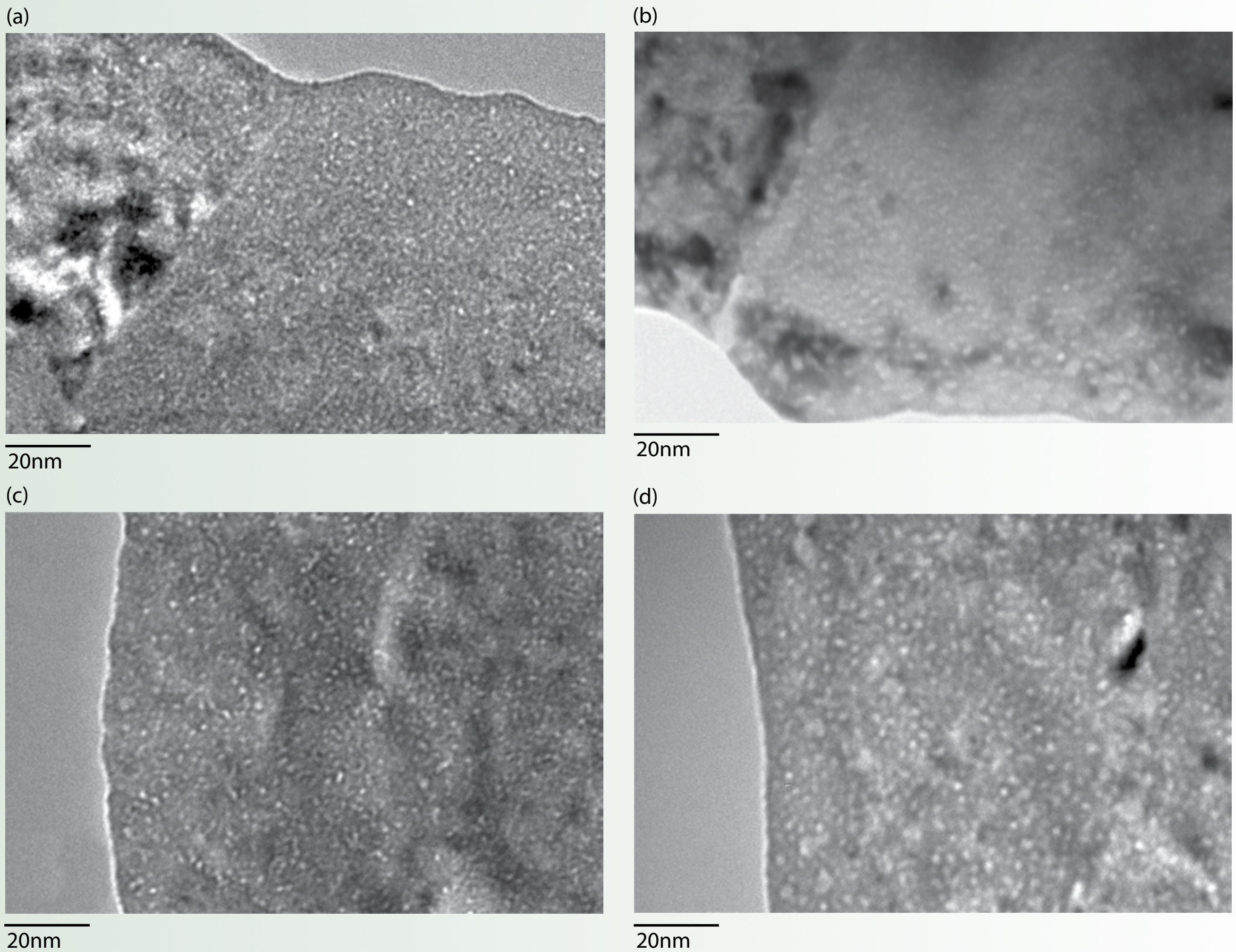

Figure 3. TEM images showing bubbles in UO_2 irradiated up to a final dose of $5{\times}10^{15}$ ions/cm^2 with 150-keV Kr at (a) 25° C and (c) 600° C, and (b and d) 1-MeV Kr at 800° C. (a) and (b) are polycrystalline UO_2; (c) and (d) are single-crystal UO_2.

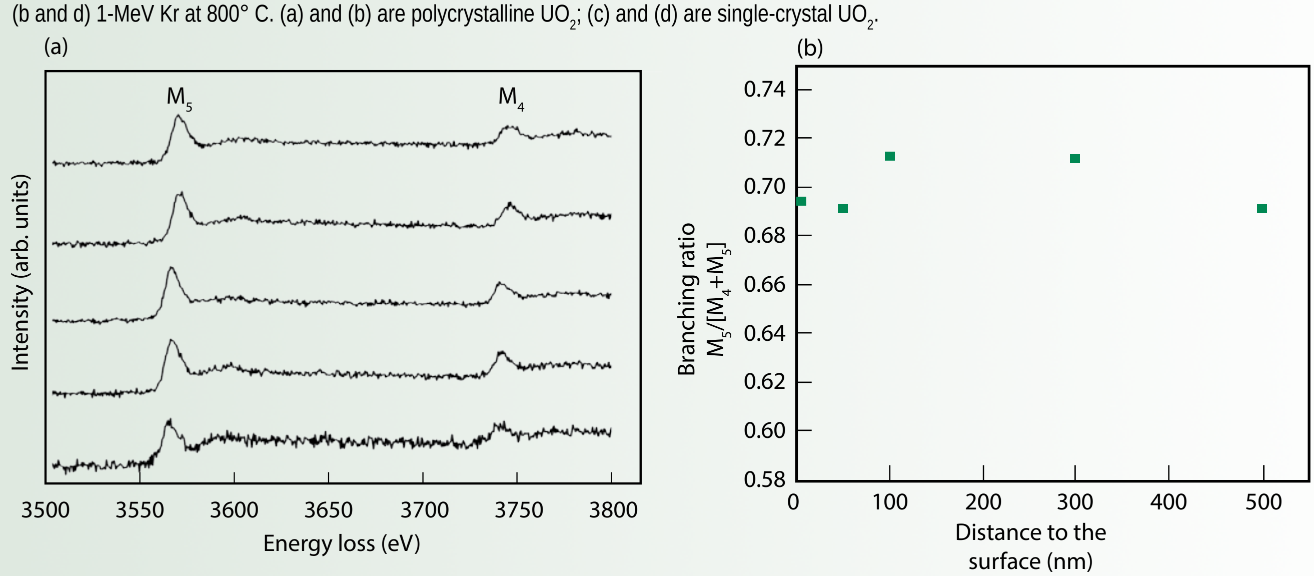

Figure 4. (a) EELS spectra as a function of the distance to the surface showing the M_4 and M_5 edges of U in 1 MeV Kr-irradiated UO_2 up to a dose of $5{\times}10^{15}$ ions/cm^2 and (b) the branching ratio, $M_5/(M_5+M_4)$, profile as a function of the distance to the surface.

Future Activities

The research team will study the effects of temperature on microstructure evolution. The samples that were irradiated at room temperature will be annealed at elevated temperatures. The 300-keV FEG-STEM at CAES will be used to image the microstructural changes in the materials after annealing. Dislocation loops, dislocation lines or dislocation networks, where present, will be studied. Cavities or bubbles in the grains and at the grain boundaries will also be investigated. The sizes and densities of these extended defects will be determined and connected to the thermal transport capability as measured by collaborators at INL.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor
Collaborators	
Idaho National Laboratory Jian Gan (principal investigator)	
University of Wisconsin Kumar Sridharan (principal investigator), Lingfeng He (collaborator), Janne Pakarina (collaborator), Clarissa Yablinsky (collaborator), Mahima Gupta (collaborator)	

Publications and Presentations*

1. Lingfeng He, Mahima Gupta, Clarissa Yablinsky, Jian Gan, Mark Kirk, Todd Allen, "Microstructural Investigation of Kr Irradiated UO_2," *The Minerals, Materials, and Metals Society, 2013 Annual Meeting & Exhibition, March 3-7, 2013, San Antonio, Texas.*

2. Lingfeng He, Mahima Gupta, Billy Valderrama, Hunter Henderson, Abdel-Rahman Hassan, Janne Pakarinen, Jian Gan, Mark Kirk, Michele Manuel, Anter El-Azab, Todd Allen, "Microstructural Investigations of Kr and Xe Irradiated UO_2," *Energy Frontier Research Centers Principal Investigators Meeting. July 18-19, 2013, Washington, D.C.*

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

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Irradiation Performance of Iron-Chromium Base Alloys

Introduction

In recent years, ferritic alloys (FeCr) have come to be considered the leading alloy system for a variety of advanced reactor components and applications due to their excellent resistance to void swelling, better thermal conductivity, lower thermal expansion and acceptable high-temperature mechanical strength compared to austenitic stainless steels.

This ATR NSUF project is a coordinated set of experiments complete with post-irradiation examination (PIE) and analysis that will provide significant new insight into the radiation performance of ferritic alloys intended for next-generation nuclear reactors.

Project Description

The main objective of this research is to subject FeCr alloys, ranging from model alloys to commercial and developmental alloys, to a set of irradiation conditions that will provide the basis for the development of materials and material-modeling with which researchers can assess radiation performance.

In addition to giving researchers a new level of understanding of the radiation performance of the FeCr alloy system, this effort will serve as a mechanism for more accurately predicting future alloy performance and development. A list of the materials and experimental parameters is provided in Table 1.

Table 1. Test Matrix (12 materials, three irradiation temperatures, six doses.)

Alloy	Temperature (°C)	Dose (dpa)	Specimen Types
Model alloy: Fe, Fe-9Cr, Fe-9Cr-0.1C, Fe-9Cr-0.5C, Fe-12Cr, Fe-12Cr-0.2C, Fe-12Cr-0.5C, Fe-14Cr*, Fe-19Cr*	300, 450, 550	0.01, 0.1, 0.5, 1.0, 5.0, 10	TEM, miniature tensile
Commercial alloys: T91, HT-9	300, 450, 550	0.01, 0.1, 0.5, 1.0, 5.0, 10	TEM, miniature tensile
Developmental alloys: MA-957	300, 450, 550	0.01, 0.1, 0.5, 1.0, 5.0, 10	TEM, miniature tensile

* Single-crystal materials, no miniature tensile specimens

There are still many open questions about phase stability, alloying element segregation and mechanical properties under irradiation in Fe-Cr, which is currently the primary structural material for nuclear reactors, that are being addressed by our ATR NSUF project.

Accomplishments

The project accomplishments are composed of three parts: transmission electron microscopy (TEM) examinations, atom probe tomography (APT) examinations and mechanical property measurements.

TEM examinations

Researchers have completed examinations of Fe-14Cr and Fe specimens irradiated at 300° C and 450° C to the doses of 0.01, 0.1, and 1 displacements per atom (dpa). The TEM specimen preparation was performed at INL's MFC and in the Low Activation Materials Development and Analysis (LAMDA) Laboratory at Oak Ridge National Laboratory. The TEM examinations were carried out in several facilities including MFC, LAMDA and CAES.

Quantitative measurements show that the evolution of the dislocation loops follows a typical process of irradiation damage: loop formation, loop growth, loop coalescence and the formation of dislocation networks. Researchers observed that the lower temperature (300° C) has substantially reduced the kinetics of loop evolution. For example, the average loop size of the 1 dpa Fe-14Cr specimen at 300° C is about 4.7 nm, while it is 20 nm for its 450° C counterpart. In addition, a well-developed dislocation network formed in the 1 dpa Fe-14Cr specimen irradiated at 450° C, but not in the 1 dpa Fe-14Cr specimen irradiated at 300 °C.

The grain boundary analysis shows that <100> loops dominate most of the Fe-14Cr specimens, except for the 1 dpa 450° C specimen, where more 1/2<111> loops than <100> loops were found. This observation contradicts the current knowledge that <100> loops are more stable than 1/2<111> loops at 450° C. Further investigation on this matter is required. Examples of the loop structures are shown in Figure 1.

For Fe specimens, the loops are smaller than they are in Fe-14Cr under the same irradiation conditions. For instance, the mean size of dislocation loops in 1 dpa Fe at 450° C is 13.6 nm, while it is 20 nm for its Fe14Cr counterpart. In addition, no dislocation networks have formed in any of the Fe specimens that are being examined. This is somewhat surprising because loop evolution is expected to occur faster in Fe specimens than

"The ATR experimental facility has been the first real opportunity for university programs to plan and lead reactor irradiation experiments in the U.S. Our work there is producing a new understanding of radiation effects as well as the next generation of nuclear materials researchers."

James F. Stubbins, Ph.D., Willet Professor and Department Head, Nuclear, Plasma and Radiological Engineering, University of Illinois

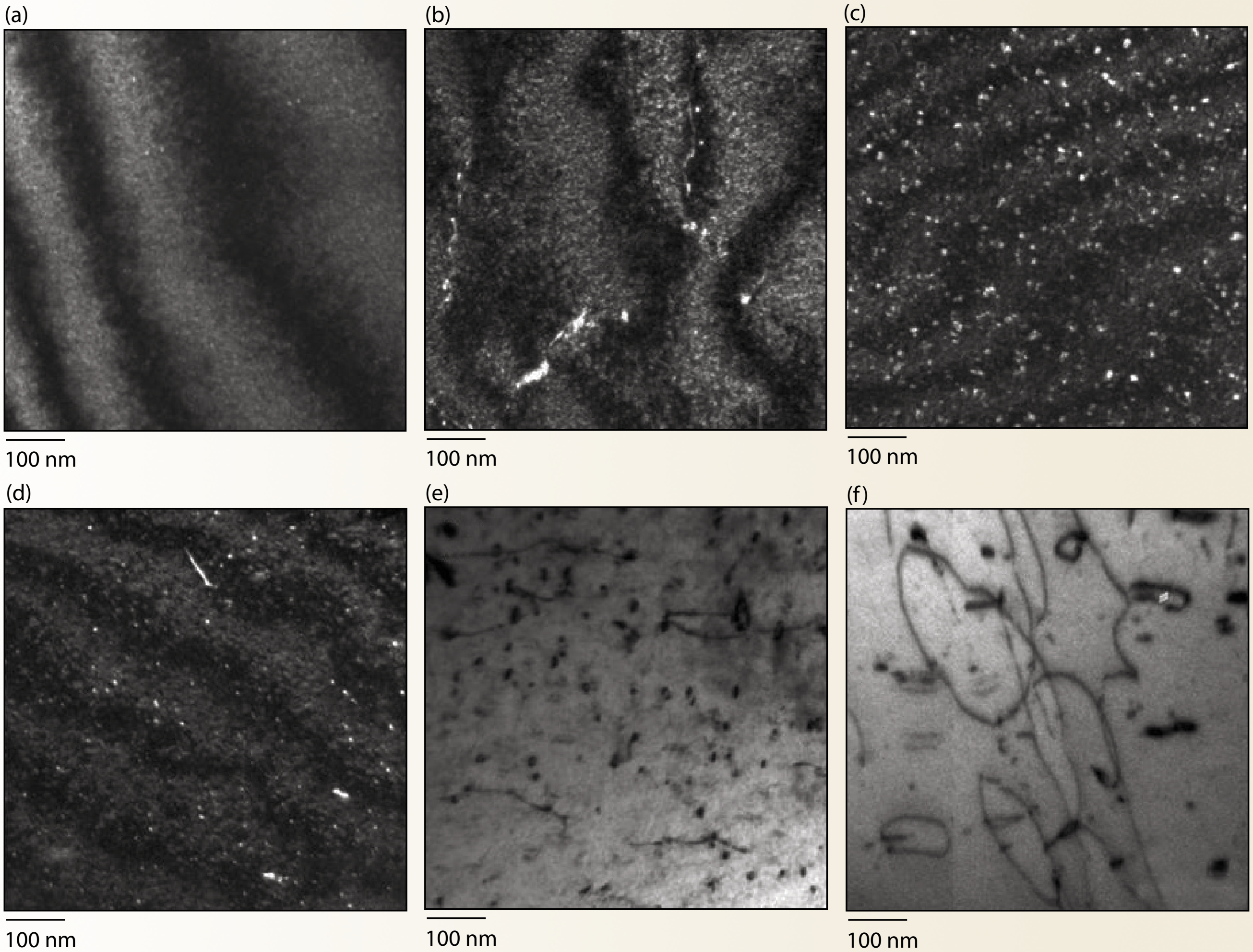

Figure 1. TEM images of Fe-14Cr specimens irradiated at 300° C to doses of (a) 0.01 dpa, (b) 0.1 dpa, and (c) 1 dpa; and 450° C to doses of (d) 0.01 dpa, (e) 0.1 dpa, and (f) 1 dpa.

in Fe-14Cr specimens. One possible explanation for this might be the effect of the respective grain boundaries (polycrystalline in Fe and single-crystal in Fe-14Cr). Electron backscatter diffraction examinations of the Fe specimens show that their average grain size is around 100 µm. Each grain comprises textured sub-grains several microns in size and bounded by small-angle grain boundaries. In addition to the smaller grain size, the Fe specimens contained a considerable number of pre-irradiation dislocations.

Researchers believe that the small grain sizes and high density of grain boundaries and dislocations acting as sinks suppress the development of point defect clusters in Fe specimens.

Finally, researchers observed voids in Fe specimens exposed to doses of 0.1 dpa and larger, but not in Fe-14Cr specimens. This result is consistent with current knowledge of swelling properties in Fe-Cr alloys. For 1 dpa Fe specimen irradiated at 300° C, the voids were found to be associated with the raft of dislocation loops. The dislocation loops themselves were frequently associated with dislocation lines. This is a new discovery, and more experiments were required to confirm it.

APT examinations

The atom probe specimens were prepared using the focused ion beam in CAES. Five to six atom probe tips were cut from each of the four sample discs and then welded to the silicon coupon. After that, the entire coupon was installed into the local electrode atom probe (LEAP).

The α' phase segregation was found in Fe-14Cr specimens irradiated at both 300° C and 450° C. For 1 dpa Fe-14Cr specimens, the α' precipitates having a size of around 1 – 2 nm and a peak Cr concentration of around 60%

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could be easily observed having isoconcentration surfaces. In the 0.01 dpa and 0.1 dpa Fe-14Cr specimens, the Cr segregation is much less limited, but is still detectable with frequency distribution analysis. For Fe-10Cr specimens, α’ precipitates of similar sizes and Cr concentrations were observed in both temperatures. However, no or very limited Cr segregations could be found in 0.1 dpa and 0.01 dpa Fe-10Cr specimens. Examples of the APT results are shown in Figure 2.

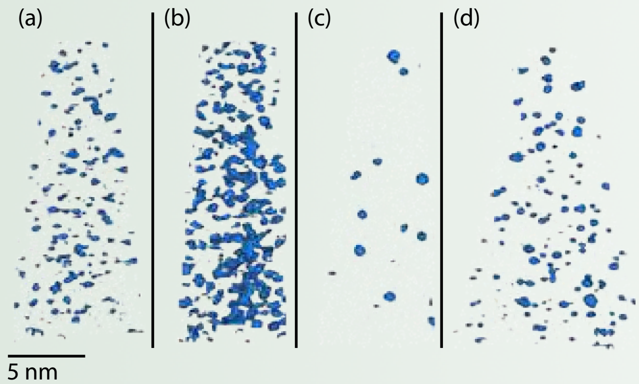

Figure 2. Isoconcentration surface analysis of α’ precipitates in 1 dpa Fe-Cr samples: (a) Fe-14Cr at 450° C, (b) Fe-14Cr at 300° C, (c) Fe-10Cr at 450° C, (d) Fe-10Cr at 300° C.

Frequency distribution analysis was performed on all of the Fe-14Cr and Fe-10Cr specimens to gather statistical information about Cr segregation. The frequency distributions of irradiated specimens were compared with binomial distribution to reveal the degree of Cr-Fe separation. Researchers used the μ-index to quantitatively indicate the deviation between the measured distribution and the binomial distribution. When $\mu = 0$, there is no Cr segregation and the alloy is randomly mixed (i.e. binomial distribution). When $\mu = 1$, the Cr and Fe are entirely separated.

The results of the frequency distribution analysis are consistent with the isoconcentration surfaces data. For example, the 1 dpa Fe-14Cr specimens have higher μ values of around 0.9, whereas the hydraulic shuttle irradiation system (also known as “rabbit”) specimens have lower μ values of around 0.2 – 0.4.

We have elaborated on the frequency analysis by first acquiring more data on each condition to achieve better statistical significance. Also, the acquisition modes used were limited to just the voltage mode because it is considered more accurate over the laser mode. In addition, the analysis was performed in a carefully chosen region of interest that does not include the artifacts from the three-dimensional reconstruction. Including the artifacts in the analysis results in unreasonably high values of μ. After implementing these elaborations, the new results show that the μ values of Fe-14 Cr specimens increase consistently with increases in irradiation doses.

For Fe-10Cr specimens, the μ value for 1 dpa at 300° C is above 0.9, whereas the value of the 1 dpa 450° C specimen is about 0.4. For the lower doses, the μ values are 0.1 – 0.2 with no observed trends in relation to irradiation doses.

Mechanical property testing

The hardness measurements were completed using nanoindentation in CAES on all of the Fe, Fe-10Cr, and Fe-14Cr specimens irradiated at 300° and 450° C to 0.01 and 1 dpa. Before indentation, specimens were jet-polished in order to minimize the effect of surface roughness. Although the hardness increases consistently as a function of irradiation doses in all of the three categories (Fe, Fe-9Cr, and Fe-14Cr), the rate at which it increases is much higher in Fe-14Cr and Fe-10Cr specimens than in Fe.

Since α’ precipitates are found in Fe-Cr specimens but not in Fe, it appears that Cr segregations play an important role in irradiation-induced hardening in Fe-Cr alloys. In addition, the lower density of dislocation loops in Fe specimens compared to Fe-14Cr specimens might be another cause for the lower increment in hardness. Researchers have conducted microhardness measurements on the lower-dose Fe and Fe-14Cr specimens in the LAMDA Laboratory. The results were consistent with the nanoindentation measurements. The comparisons of hardness measurements are shown in Figure 3.

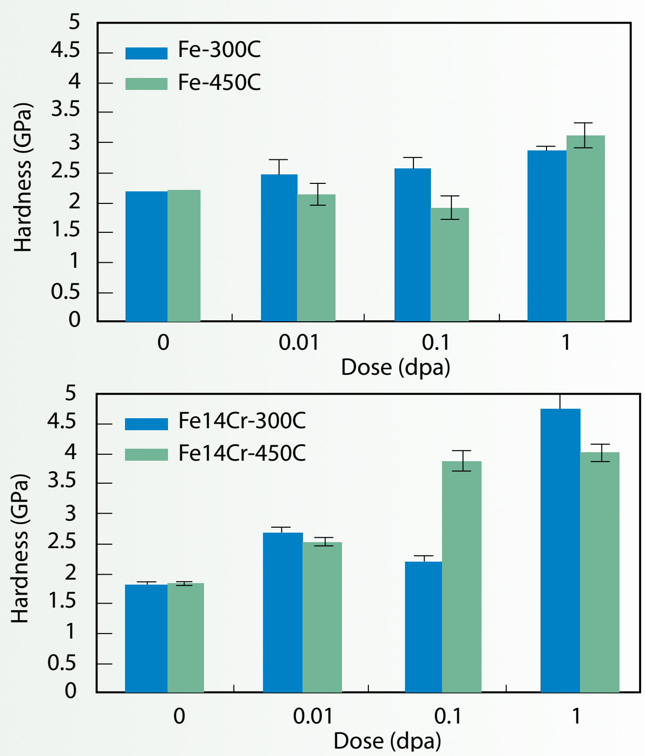

Figure 3. Hardness measurements using nanoindentations (fixed displacement of 5 μm) on Fe and Fe14-Cr specimens.

Future Activities

TEM examinations of Fe, Fe-10Cr, and more complex Fe-Cr alloy system specimens

TEM examinations of nine Fe and Fe-10Cr specimens are scheduled at MFC and CAES in late February and early March. When these are finished, a complete matrix of data from TEM, APT and hardness tests could provide valuable information on the effects of Cr concentration and irradiation temperature on the microstructure evolution in irradiated Fe-Cr alloys. Examinations of the other complex commercial alloys will follow.

Other examinations of Fe-10Cr-C and more complex Fe-Cr alloy specimens

Examinations of Fe-10Cr-C specimens will begin when the TEM tasks described above are completed. TEM, APT and hardness tests will be performed. Researchers will focus on the effects of carbon concentrations on microstructure evolution irradiated to 300° C and 450° C to doses of 0.01, 0.1 and 1.0 dpa. The results will be compared to those of similar experiments performed in 2013. APT and other examination techniques will be used to investigate the more complex microstructures of advanced Fe-Cr alloys.

In-situ synchrotron experiments

For the in-situ synchrotron experiments, we have tested the tensile machine on a few of the archived, unirradiated specimens in the Advanced Photo Source (APS) at Argonne National Laboratory. Twelve tensile specimens of selected model commercial alloys have been measured in MFC to ensure they meet the dose limits at APS. Researchers expect to perform the experiments in APS in early 2014. Other tensile testing of the more complex alloy systems will follow.

Publications*

Wei-Ying Chen, Yinbin Miao, Carolyn Tomchik, Kun Mo, Yaqiao Wu, Jian Gan, Stuart Maloy, James Stubbins, “Atom probe analysis of a neutron irradiated Fe-14Cr model alloy,” *International Conference on Fusion Reactor Materials, Beijing, China, 2013*.

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Oak Ridge National Laboratory	Low Activation Materials Development and Analysis Laboratory
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Idaho National Laboratory Jian Gan (principal investigator), Maria Okuniewski (co-investigator), Dan Ogden (project manager), Collin Knight (collaborator)	
Los Alamos National Laboratory Stuart Maloy (co-principal investigator)	
Oak Ridge National Laboratory Keith Leonard (collaborator), Lizhen Tan (collaborator)	
University of California, Berkeley Peter Hoseman (co-principal investigator)	
University of Illinois James Stubbins (principal investigator), Carolyn Tomchik (collaborator), Aaron Oaks (collaborator), Weiying Chen (collaborator), Yinbin Miao (collaborator)	

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Scanning Transmission Electron Microscopy/Local Electrode Atom Probe Study of Fission Product Transportation in Neutron-Irradiated Tristructural-Isotropic Fuel Particles

Introduction

Tristructural-isotropic (TRISO)-coated particle fuel is currently used in high-temperature, gas-cooled reactors and will be the fuel for the Generation IV Very High Temperature Reactor (VHTR). One of its primary benefits is its silicon-carbide (SiC) layer, which is designed to act as the main barrier to the release of radioactive metallic fission products (FP). However, the release of FPs, especially 110m silver (^{110m}Ag), from seemingly intact TRISO particles has been observed. Therefore, understanding the FP's transportation mechanism in SiC is necessary to limiting the release of radioactive FPs and improving reactor operations.

Project Description

Despite many research efforts in this area, it remains unclear how FPs are created and then transported through the SiC layer of the TRISO particle. It was not until March 2013 that Ag was first identified in the SiC layer of a neutron-irradiated TRISO fuel particle by a research team from INL and Boise State University using scanning transmission electron microscopy (STEM) [1].

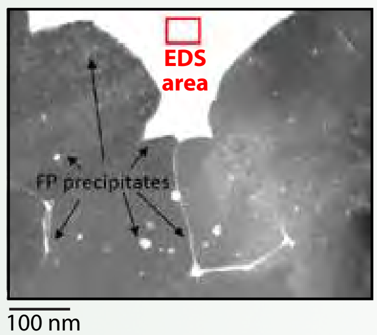
As a continuation of that initial STEM study, this project uses STEM and the local electrode atom probe (LEAP) to further investigate the morphology, composition and distribution of FPs in the SiC layer of the same TRISO fuel particle. The goal is to gain insight into the FP's transportation mechanism, particularly that of Ag.

Accomplishments

STEM/EDS Analysis

Two transmission electron microscopy (TEM) lamellae lifted from the SiC layer close to the SiC/IPyC interface were examined using STEM and energy dispersive spectroscopy (EDS). The excellent Z contrast observed under STEM clearly showed the existence of various types of FP precipitates (Figure 1).

The EDS spectrum revealed large precipitates (>100 nm), mainly palladium (Pd), silicon (Si), and uranium (U), as well as small peaks of other FPs, such as cesium (Cs), europium (Eu), cerium (Ce), and plutonium (Pu). Quantitative analysis showed the stoichiometry of the large precipitates is close to $Pd_{1.8}Si_{1.6}U$, but the assumption of low concentrations of elements such as Ag was inaccurate.



Element	Atomic %
Si(K)	36.8
Pd(K)	39.7
U(L)	22.5
Ag(K)	0.4
Cs(L)	0.6
Ce(L)	0.0
Eu(L)	0.0

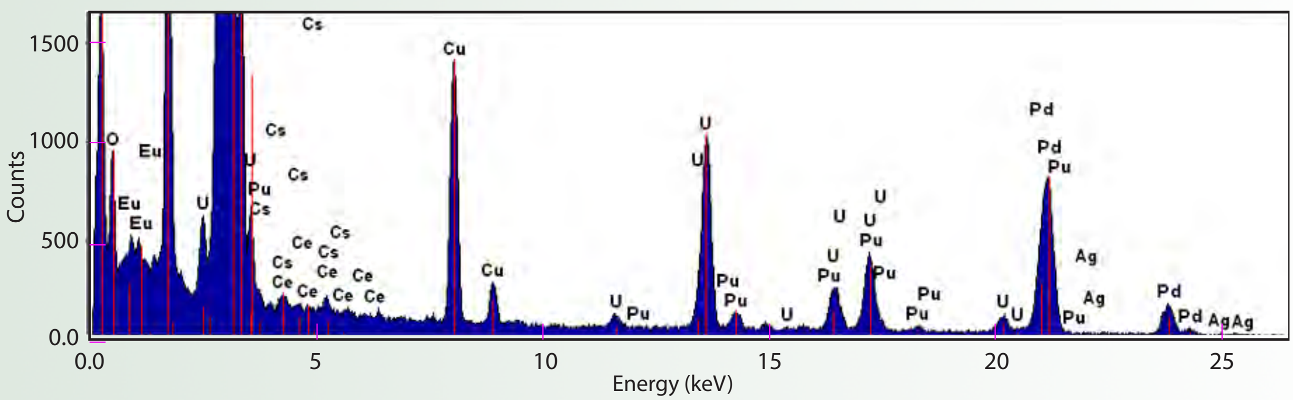


Figure 1. Distribution of FP precipitates in the SiC layer (~5 μm from the SiC/IPyC interface) of a neutron-irradiated TRISO fuel particle from ATR-1.

The various fission products found in the SiC layer of a neutron-irradiated TRISO fuel particle have improved our understanding of the fission product release mechanism in TRISO fuels.

Triple-junction precipitates and hairline grain boundary precipitates were found to be rich in either Pd or Ag (Figure 2). The presence of these Ag-rich, triple-junction and grain-boundary precipitates may indicate that the grain boundaries act as fast transportation paths for Ag. Different types of small-sphere precipitates (<15 nm) were found inside the SiC grain (Figure 3). Most of these nano-precipitates were Pd-rich (no Ag was identified), but several Au-Pd-Ag precipitates were also found. A honeycomb-like network of small U-rich precipitates was also found near the large FP precipitates.

LEAP test

Fifteen LEAP tips were prepared near the location where the TEM lamellae were made. Since this work was the first attempt to use LEAP for a neutron-irradiated SiC sample, the research team had to try several

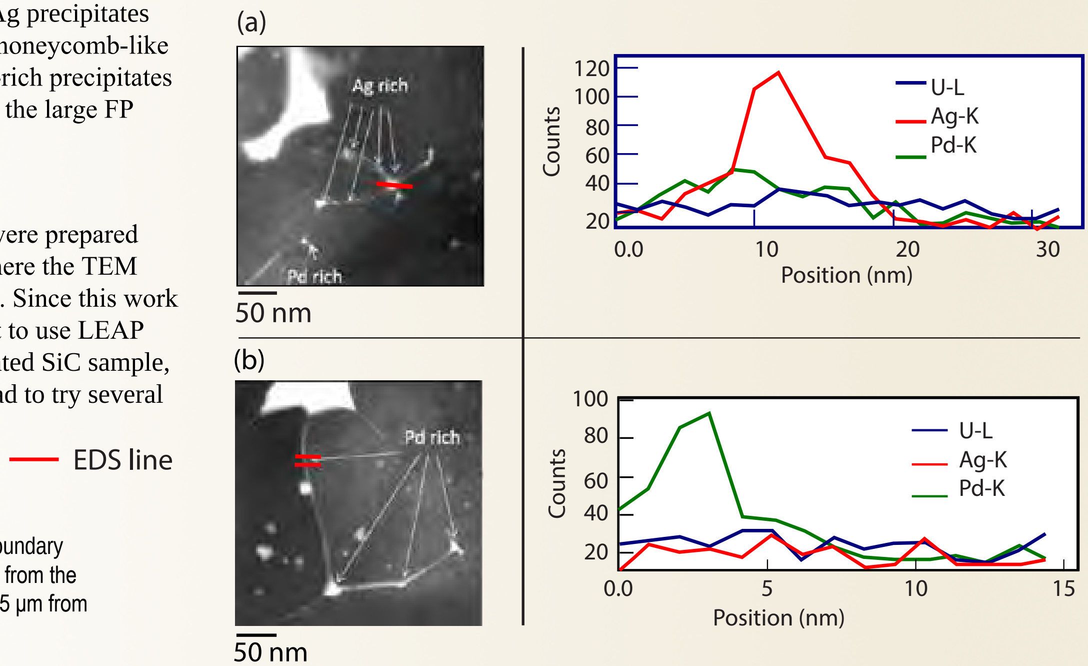


Figure 2. Triple-junction precipitates and grain-boundary precipitates at (a) ~2 μm from the SiC/IPyC interface, (b) ~5 μm from the SiC/IPyC interface.

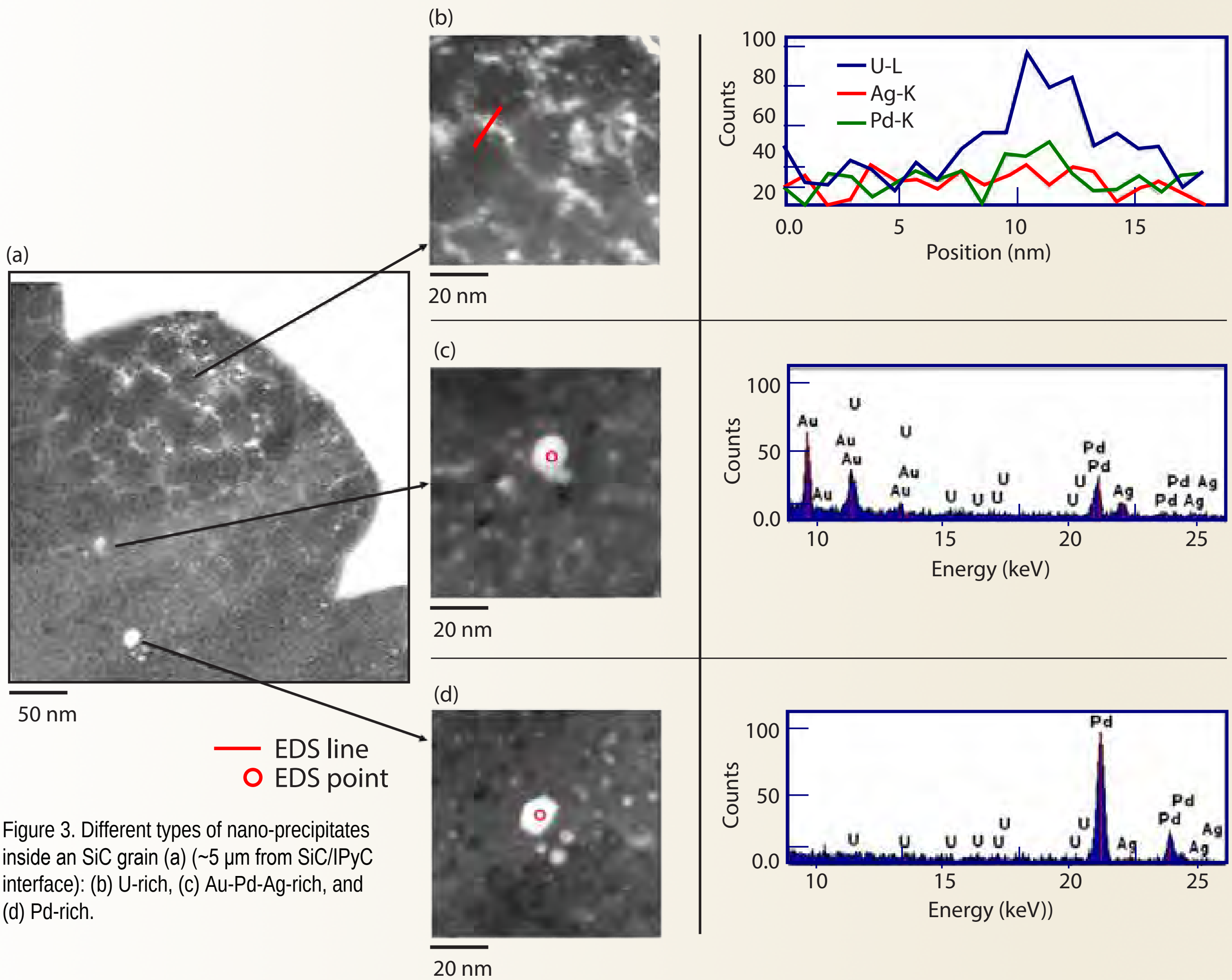


Figure 3. Different types of nano-precipitates inside an SiC grain (a) (~5 μm from SiC/IPyC interface): (b) U-rich, (c) Au-Pd-Ag-rich, and (d) Pd-rich.

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Scanning Transmission Electron Microscopy/Local Electrode Atom Probe Study of Fission Product Transportation in Neutron-Irradiated Tristructural-Isotropic Fuel Particles (cont.)

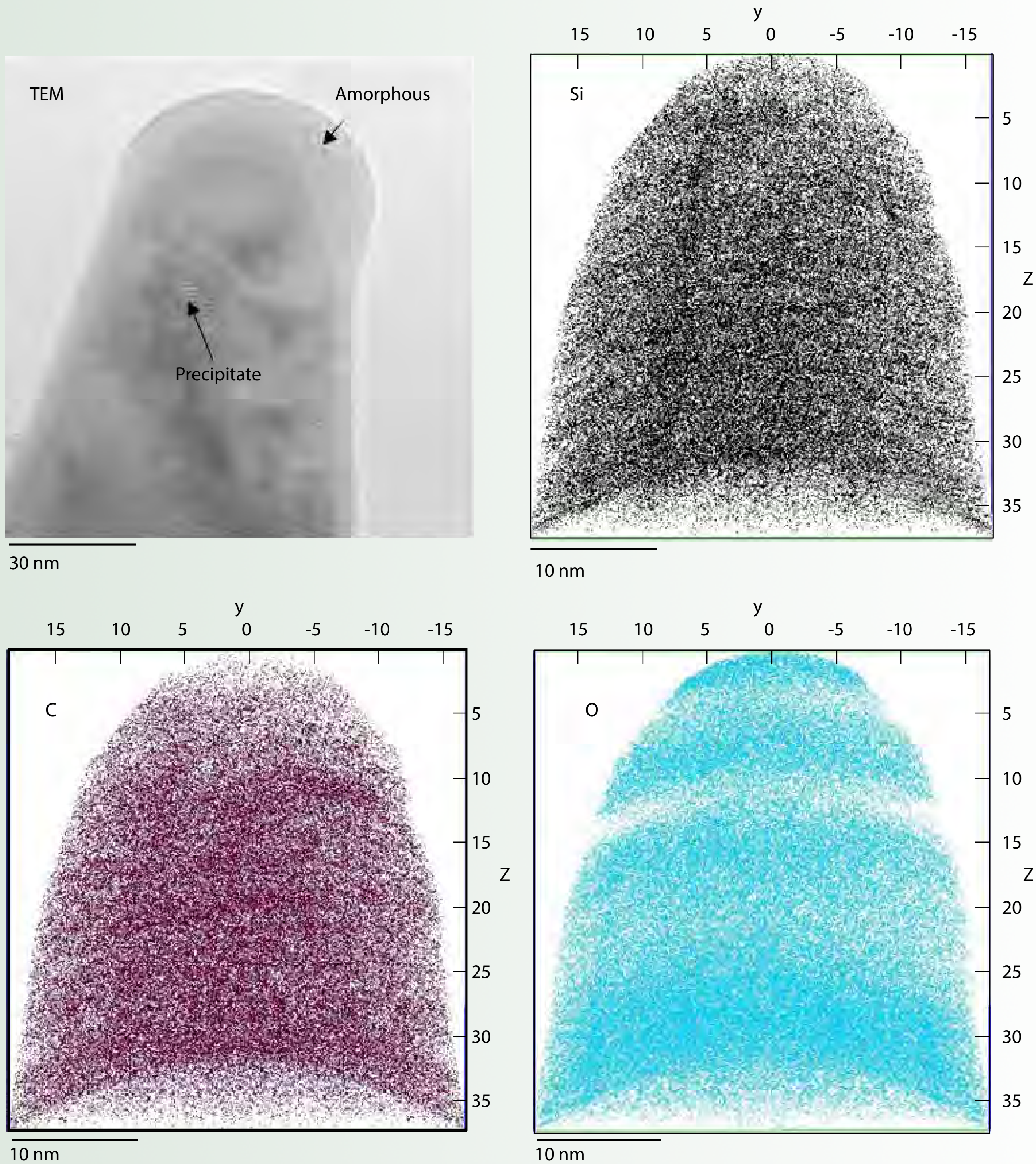

Figure 4. LEAP results showing the distribution of elements in a sample ~5 μm from SiC/IPyC.

different equipment settings before the optimal ones were found. As a result, many problems occurred during testing.

The large number of defects in the sample caused by the neutron irradiation and surface oxidation that occurred while transporting it from the Electron Microscopy Laboratory at MFC to CAES made it difficult to collect data from deep inside the tips. At the conclusion of the testing, all the tips had been consumed, either by being fractured during testing or destroyed during the testing of the adjacent tip. As a result, the largest data set researchers were able to collect came from a depth of about 30 nm (Figure 4). Although TEM images show that the tip contains FP precipitates, the LEAP test was not able to penetrate the sample far enough to detect them. Therefore, the LEAP test has not yet identified the existence of FPs.

Future Activities

In order to investigate the possible influence of irradiation, LEAP tips of Ag-implanted SiC surrogate samples will be prepared at the University of Wisconsin and sent to CAES for additional LEAP testing.

References

[1] Isabela van Rooyen, Yaqiao Wu, Tom Lillo, "Identification of Silver and Palladium in Irradiated TRISO Coated Particles of the AGR1 Experiment," *Journal of Nuclear Materials* 446 (2014) 178-186.

Publications and Presentations

Two papers from this work are being prepared. They focus on the project team's STEM and LEAP efforts, respectively.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Collaborators	
Center for Advanced Energy Studies/Boise State University Yaqiao Wu (collaborator)	
Idaho National Laboratory Isabella van Rooyen (co-principal investigator), James Madden (collaborator), Tom Lillo (collaborator)	
University of Wisconsin Izabela Szlufarska (principal investigator), Tyler Gerczak (collaborator), Bin Leng (collaborator), Kumar Sridharan (collaborator)	

Multiscale Investigation of the Influence of Grain Boundary Character on RIS and Mechanical Behavior in Steels Used in Light Water Reactors

Principal Investigator: Mitra Taheri – Drexel University
email: mtaheri@coe.drexel.edu

Project Description

Drexel University's ATR NSUF project is centered on understanding the behavior of stainless steel in light water reactors (LWRs). The research provides a foundation we can use to mitigate some of the behavior we are seeing in stainless steels that are exposed to the temperatures and doses found in LWRs over extended times.

Specifically, the objective is to determine if any trends exist regarding thermal- or radiation-induced failures, which will aid the future development of stainless steels for LWRs and other types of reactors. The project focuses on understanding defect structures at grain boundaries as well as any other associated conditions. Thus, it could help extend the life and improve the maintenance of existing reactors as well as improve the development of materials used in next-generation reactors.

Accomplishments

Drexel's ATR NSUF-funded project consists of investigating the role of grain-boundary character of radiation-induced segregation and precipitation in face-centered cubic austenitic stainless steel.

Researchers received grade 304 stainless steel (304SS) (U1302) from the outer blanket assembly of the Experimental Breeder Reactor II (EBR-II) that had been irradiated to ~4.5×10^{21} n/cm^2 at a high operating temperature (~450° – 460° C). Using atom probe tomography (APT) coupled with scanning transmission electron microscopy-energy-dispersive spectroscopy (STEM-EDS), researchers analyzed the samples with a multi-length scale that is grain boundary site-specific and process-optimized to obtain detailed chemical and structural information.

Researchers also performed electron backscatter diffraction (EBSD) (Figure 1) to determine grain boundary misorientations and used a focused ion beam (FIB) to extract specific grain boundaries. The FIB-prepared samples were then analyzed using transmission electron microscopy (TEM) and APT.

Figure 2 highlights the methodology used to examine a coherent Σ3 grain boundary APT tip in the TEM using a Hummingbird half-grid sample holder. This method enables the team to transfer samples directly from the TEM to the local electrode atom probe (LEAP) to obtain characterization of the grain boundary plane at an atomic scale.

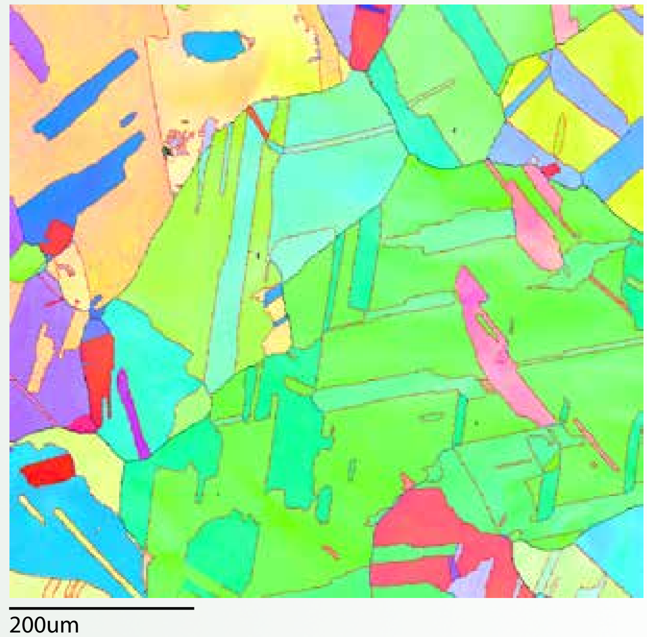

Figure 1. SEM-EBSD inverse-pole image highlighting the grain boundary character distribution in the 304SS sample (U1302), which indicates high densities of Σ3, Σ9, and random high-angle grain boundaries.

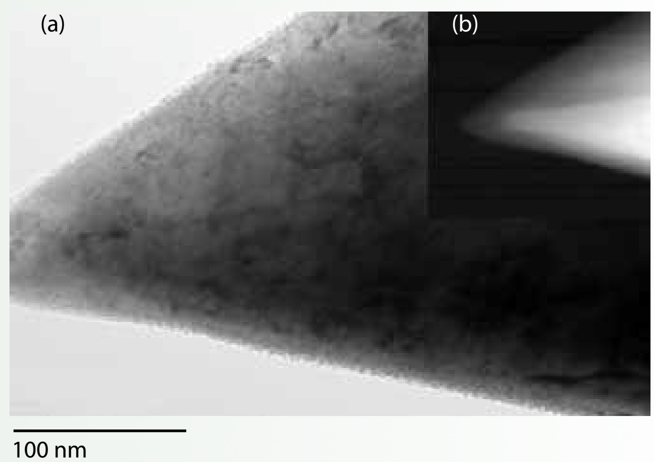

Figure 2. Illustration of coupled STEM and APT using a Hummingbird half-grid TEM holder, where (a) is a TEM bright field image of Σ3 coherent grain boundary APT tip and (b) is the dark field STEM image of the same grain boundary showing that the coherent twin grain boundary does not have any carbide growth.

Figure 3 and Figure 4 show that M23C6 carbides have grown randomly on a high-angle grain boundary in the 304SS. The presence of particularly large carbides could indicate long-term, irradiation-assisted-temperature, thermal-stability issues in the EBR-II. We also observed that coherent Σ3 twin boundaries did not have any carbides, unlike the random, high-angle grain boundaries. These differences can be linked to variations in energy between the high-angle grain boundaries and special grain boundaries such as the coherent Σ3.

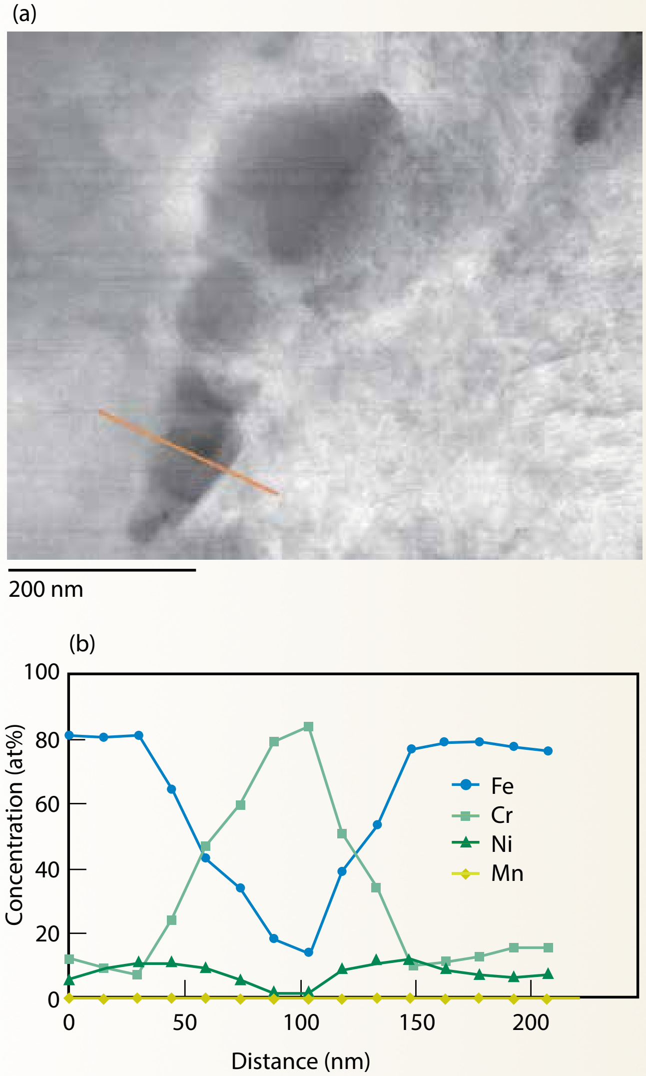

Figure 3. (a) STEM image illustrating M23C6 Chromium (Cr)-rich carbides along a 49° [2 3 9] grain boundary. (b) Corresponding STEM-EDS line profile from Cr-rich carbide.

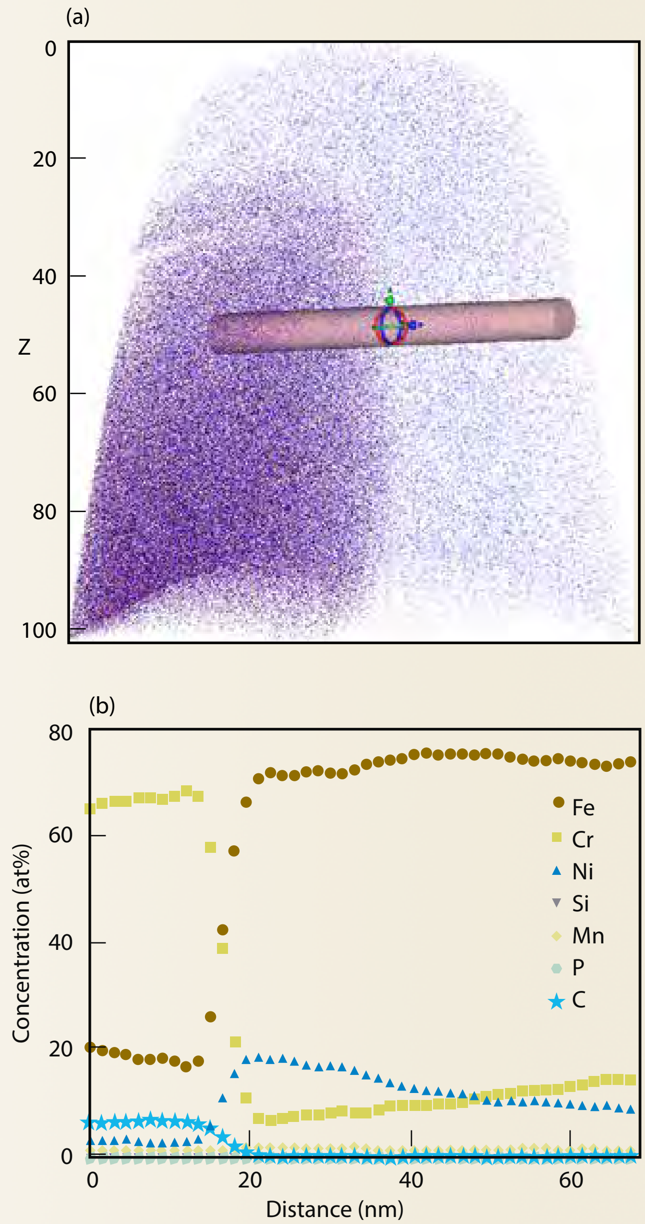

Figure 4. (a) Atom probe reconstruction of a 40° [8 9 4], carbide-grain boundary interface in 304SS, irradiated in the EBR-II to 4.5×10^{21} n/cm^2. (b) Corresponding concentration profile of an interface highlighting Cr-rich carbide and adjacent Cr-depleted region.

Multiscale Investigation of the Influence of Grain Boundary Character on RIS and Mechanical Behavior in Steels Used in Light Water Reactors (cont.)

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Future Activities

Future work on this project will focus on examining additional low-energy grain boundaries, such as $\Sigma 3$ and $\Sigma 11$, as a function of the grain boundary plane, the results of which will be compared to the initial observations. Specifically, researchers will determine whether or not the carbide growth was widespread or limited to specific grain boundaries (and if so, which ones—low-energy or high-energy).

The data we obtain during this project is significant in the development of future LWRs as it provides insight into unexpected behavior. The facilities at INL have allowed us to delve deeply into the results of our work, helping us explain anomalies in these important steels that occur during operation.

Publications and Presentations

Christopher Barr, Jim Cole, Mitra Taheri, "Effects of grain boundary character on thermal aging and radiation induced segregation in neutron-irradiated 304 stainless steel EBR-II duct," being prepared for *Journal Nuclear Materials*, 2014.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	PIE facilities
Collaborators	
Drexel University Mitra Taheri (principal investigator), Christopher Barr (Ph.D. graduate student)	
Idaho National Laboratory Jim Cole (principal investigator)	

Principal Investigator: Lizhen Tan – Oak Ridge National Laboratory
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Stability of Precipitates under Ion Irradiation

Introduction

The identification of more stable MX (M = metal, X = metalloid) nanoprecipitates will not only improve our understanding of alloy degradation mechanisms, but will also help in developing advanced radiation-resistant alloys that have superior performance at elevated temperatures.

Project Description

This ATR NSUF project used the irradiation of iron (Fe^{2+}) ions to evaluate the stability of a variety of MX-type precipitates as well as iron-tungsten (Fe_2W)-type Laves phase under irradiation. Three model ferritic alloys were fabricated to promote the formation of vanadium-nitride (VN), tantalum-carbide (TaC), and tantalum-nitride (TaN) nanoprecipitates. The evolution of these alloys under Fe^{2+} irradiation at 500º C was investigated for projected damages of 20 and 200 displacements per atom (dpa).

The project outcome increases our understanding of the degradation mechanisms in current reduced-activation ferritic-martensitic (RAFM) and conventional FM steels. Additionally, it will help in the development of advanced radiation-resistant alloys that will increase safety margins and design flexibility for replacement components scheduled to be installed in current reactors, and those to be employed in next-generation reactors.

Accomplishments

One of the longitudinal surfaces on mini-bars (2x2x20 mm^3) of the model ferritic alloys was polished to a mirror finish and irradiated with 5 MeV Fe^{2+} ions at 500º C, using the ion beam facility at the University of Michigan through ATR NSUF. Professor Gary Was advised the ion irradiation experiment and Ovidiu Toader conducted the irradiation of the mini-bars. The irradiated mini-bars were shipped to Oak Ridge National Laboratory where Dorothy Coffey helped prepare transmission electron microscopy (TEM) specimens using the focused ion beam (FIB).

Microstructure characterization was performed using TEM as well as in scanning transmission electron microscopy (STEM) mode. Examining the FIB lift-out specimens parallel to the irradiation direction provides an overall picture of the evolution of MX as a function of the irradiation depth (Figure 1).

This ATR NSUF project's outcome will help in the development of advanced radiation-resistant alloys that will increase safety margins and design flexibility for replacement components scheduled to be installed in current reactors and those to be employed in next-generation reactors.

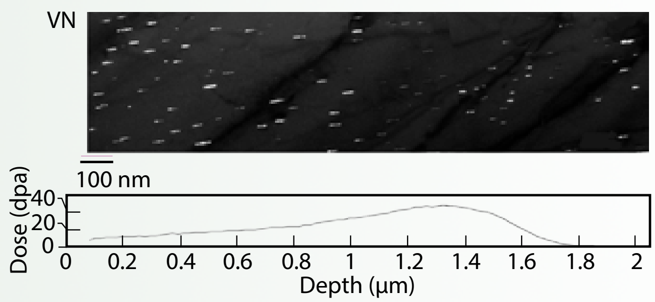

Figure 1. Dark field image showing the evolution of VN precipitates (white) under Fe^{2+} irradiation at 500º C starting from the irradiated surface (left) to bulk matrix without irradiation (right). The dose increases are shown in the Stopping and Range of Ions in Matter (SRIM) simulated depth-dependent irradiation dose profile beneath the image.

The characterized and analyzed results indicate that the nanoprecipitates are non-stoichiometric. The irradiation did not alter their crystallinity, but may have altered the structure of their defects.

Irradiation resulted in moderate reprecipitation and a slight dissolution of TaC while, in contrast, significant dissolution was observed in TaN. VN had moderate growth. Irradiation facilitated the formation of Fe_2W-type Laves phase in VN and TaN, but not in alloys containing TaC, suggesting that nitrogen promoted its formation.

These results suggest that if nitrogen levels are kept to a minimum in alloys, greater radiation resistance to the MX-type precipitates may be gained at similar temperatures. This may also postpone the formation and subsequent coarsening of Laves phase.

"Ion irradiation provides a fast and economic route to evaluate the stability of MX precipitates which is critical to retaining the properties of some materials."

Lizhen Tan, Research Staff, Oak Ridge National Laboratory

Future Activities

The ion irradiation experiment has been completed. A parallel neutron irradiation experiment, independent from this ATR NSUF project but using the same samples, was also recently completed. Post-irradiation examination (PIE) of these samples will be conducted and the results will be compared to the ion irradiation data obtained in this project.

Publications and Presentations*

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
University of Michigan	Ion Beam Laboratory
Collaborators	
Oak Ridge National Laboratory Lizhen Tan (principal investigator) **University of Michigan** Gary S. Was (co-principal investigator), Ovidiu F. Toader (collaborator)	

Principal Investigator: Bernhard Tittmann – Pennsylvania State University
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Transducers for In-Pile Ultrasonic Measurements of the Evolution of Fuels and Materials

Introduction

These experiments will enable researchers to design ultrasonic sensors that can characterize the operational evolution of fuel materials and other nuclear reactor components and will lead to the development of tools that can monitor a reactor's structural health.

Project Description

Pennsylvania State University (PSU) was awarded an ATR NSUF project in which both magnetostrictive and piezoelectric transducers are inserted into the Massachusetts Institute of Technology Research Reactor (MITR) at fluences of up to 10^{21} n/cm². Ultrasonic measurements of each sensor's material properties will be conducted in-situ while it is subjected to the neutron flux.

Accomplishments

During 2013, the following actions were performed:

- Suitable piezoelectric and magnetostrictive transducer materials were selected.
- Transducer assemblies for both piezoelectric and magnetostrictive transducers were designed, built and tested (Figures 1 and 2).

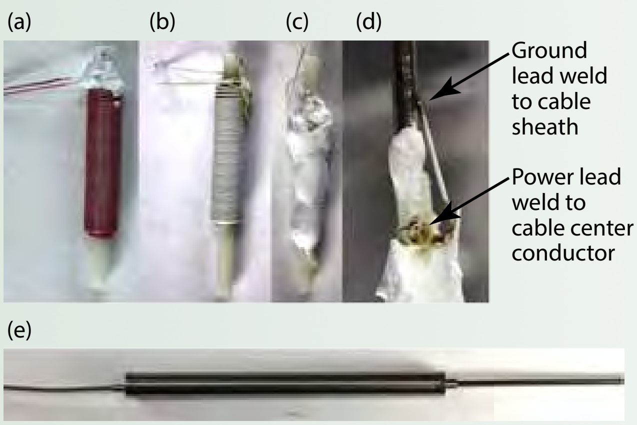

Figure 1. Magnetostrictive transducer fabrication. (a) Silver-palladium is wrapped around an alumina bobbin; (b) the wire is coated with a standoff insulator and heat treated; (c) the assembly is coated with alumina cement and heat-treated a second time; (d) a laser-welded coaxial cable is attached; and (e) the finished tranducer is welded into a prefabricated housing.

Figure 2. Piezoelectric transducer fabrication. (Top) Transducer components from left to right: stainless steel cap, alumina insulation, nickel plunger, alumina insulation, carbon-carbon backing, Kovar waveguide with zinc oxide sensor on top, stainless steel outer casing. (Bottom) Fully assembled transducer housing with cable and strain-relief sleeve to support the cable connection.

Transducer performance was characterized at design temperatures (Figures 3, 4 and 5). The zinc oxide (ZnO) transducer did not perform as expected and only operated successfully during reactor shutdowns (Figure 5). One of the ZnO transducers was replaced with an aluminum nitride (AlN) transducer, which operated successfully at the design temperature.

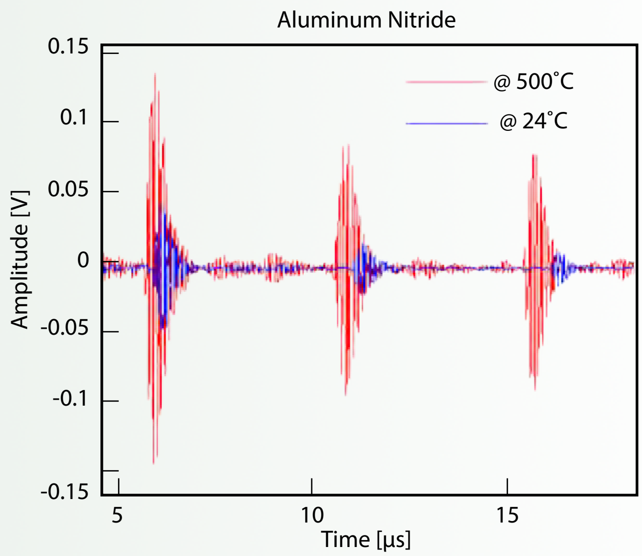

Figure 3. AlN ultrasonic pulse-echo amplitudes recorded at room temperature and the irradiation temperature.

"The in-pile use of ultrasonic transducers during materials test reactor irradiations is extremely important because they could provide users higher accuracy and higher resolution data related to the performance of candidate fuels and materials when exposed to the harsh conditions associated with irradiation testing."

Joy Rempe, Laboratory Fellow and Group Leader, Idaho National Laboratory

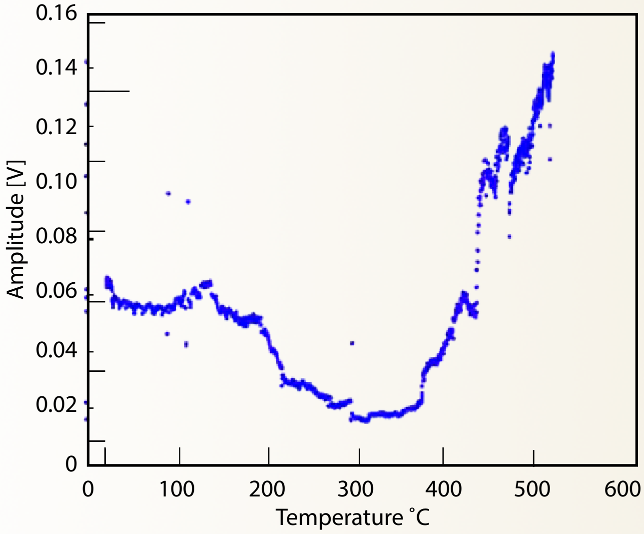

Figure 4. Temperature-dependent performance of AlN piezoelectric transducer. The transducers operated at highest efficiency at the design temperature.

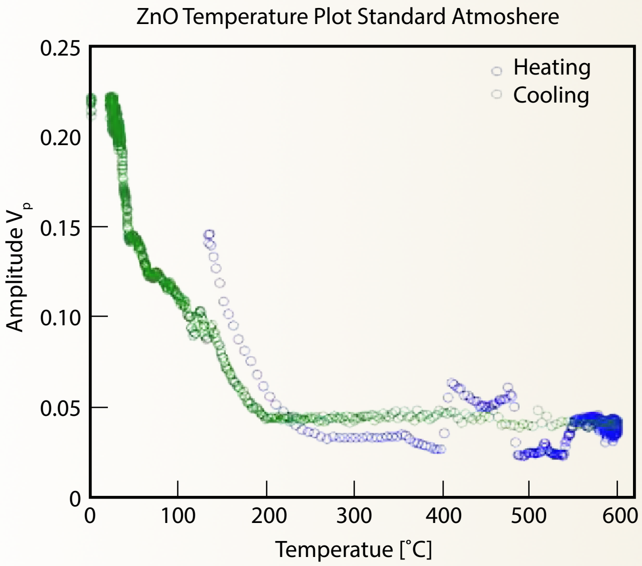

Figure 5. Temperature-dependent performance of the ZnO transducer. The transducer did not perform well at the design temperature; however, it recovered its original amplitude upon cooling. This indicates that the sensor will operate satisfactorily during reactor shutdown, enabling researchers to measure performance degradation during those times.

This project will ascertain which transducer materials are most resistant to neutron radiation, thereby enabling a suite of ultrasonic inspection techniques that can be inserted into reactors as in-pile irradiation sensors.

- Graduate students Brian Reinhardt (Ph.D.) and Andy Suprock (M.S.) joined the laboratory and have been trained on high-temperature, ultrasonic, nondestructive testing, transducer fabrication principles, and designing high-temperature and radiation-tolerant transducers.

Future Activities

The remaining work on the project consists of:

- Endurance testing of transducers.
- Inserting the transducers into MITR (anticipated to occur in February 2014).
- Irradiation of the transducers.
- In-situ monitoring.
- Post-irradiation examinations.

Publications and Presentations*

1. Joshua Daw, Joy Rempe, Joseph Palmer, Pradeep Ramuhalli, R. Montgomery, H. T. Chien, Bernhard Tittmann, Brian Reinhardt, Gordon Kohse, "Irradiation Testing of Ultrasonic Transducers," *invited paper for ANIMMA 2013 Special Edition, IEEE Transactions on Nuclear Science, submitted July 2013.*

2. Joshua Daw, Joy Rempe, Joseph Palmer, Pradeep Ramuhalli, R. Montgomery, H. T. Chien, B. Tittmann, B. Reinhardt, and G. Kohse, "Irradiation Testing of Ultrasonic Transducers," *2013 Conference on Advancements in Nuclear Instrumentation, Measurements Methods (ANIMMA 2013), Marseilles, France, June 23-27, 2013.*

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Transducers for In-Pile Ultrasonic Measurements of the Evolution of Fuels and Materials (cont.)

3. Joshua Daw, Bernhard Tittmann, "The Purpose, Experimental Design, and Expected Impact of the ATR-NSUF Ultrasonic Transducer Irradiation Experiment," *ATR-NSUF User's Week 2013, Idaho Falls, Idaho, April 2013.*

4. David A. Parks, Shujun Zhang, Bernhard Tittmann, "High Temperature (>500°C) Ultrasonic Transducers: An Experimental Comparison Among Three Candidate Piezoelectric Materials" IEEE Transactions on Ultrasonics, Ferroelectrics and Frequency Control, Vol. 60, No. 5, May 2013, pp. 1010-1015.

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	PIE facilities
Massachusetts Institute of Technology	Massachusetts Institute of Technology Technology Reactor
Collaborators	
Argonne National Laboratory H.T. Chien (collaborator)	
Bettis Atomic Power Laboratory Ben Wernsman (collaborator)	
Idaho National Laboratory Joy Rempe (co-principal investigator), Joshua Daw (collaborator), Joseph Palmer (collaborator)	
Massachusetts Institute of Technology Gordon Kohse (collaborator)	
Pacific Northweest National Laboratory Pradeep Ramuhalli (collaborator)	
Pennsylvania State University Bernhard Tittmann (principal investigator), Brian Reinhardt (graduate student), Andy Suprock (graduate student)	

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Correlating Silicon-Carbide Grain Size and Grain Boundary Orientation with Strength and Silicon-Carbide Layer Growth Conditions

Introduction

The unique combination of thermomechanical and physiochemical properties in silicon-carbide (SiC) provides an opportunity to use it in nuclear applications. One example is a very thin layer in tri-isotopic (TRISO)-coated fuel particles for high-temperature gas reactors (HTGRs). This layer, produced by chemical vapor deposition (CVD), is designed to withstand the pressures of fission and transmutation product gases in a high-temperature radiation environment.

Various researchers have demonstrated that macroscopic properties can be affected by changes in the distribution of grain boundary plane orientations and misorientations [1-3]. Additionally, researchers have attributed the release behavior of silver (Ag) through the SiC layer as a grain boundary diffusion phenomenon [4-6]. This further highlights the importance of understanding the actual grain characteristics of the SiC layer.

Historic HTGR fission product release studies, as well as recent experiments at INL [7], have shown that the release of Ag-110m is strongly temperature-dependent. Although the normal maximum operating fuel temperature of an HTGR design is in the range of 1000º-1250º C, the temperature may reach 1600º C under postulated accident conditions. The aim of this study is to determine the magnitude of temperature dependence on SiC grain characteristics, expanding upon initial studies by Isabella J. Van Rooyen et al. [8-9]. The impact of this work will showcase the effect of nanostructures on fuel performance.

Project Description

The objective of the overarching research project is to contribute to the SiC knowledge base by answering key questions about SiC properties under extreme conditions. Specifically, this project seeks to reveal correlations between SiC layer grain size and grain boundary orientations, as well as the strength of SiC layers grown, and subsequently annealed, at different temperatures.

To lessen the project cost and time for these experiments, five batches of SiC samples previously studied at Nelson Mandela Metropolitan University (NMMU) in Port Elizabeth, South Africa, were made available by Professor Jan Neethling. The batches differ with respect to SiC layer thickness, deposition temperature, and deposition method. They have a spherical geometry and have been characterized for hardness (via nano-indentation) and compressive strength. The data compiled from these studies was also made available for reference as part of this project.

The specific research for this ATR NSUF project requires characterization via focused ion beam (FIB) and/or electron backscatter diffraction (EBSD) at CAES.

This work will showcase the effect of nanostructures on fuel performance.

These tests will determine grain size and grain boundary orientations of the SiC layers (Figure 1). An associated deliverable is an EBSD technique for small samples with non-standard geometries.

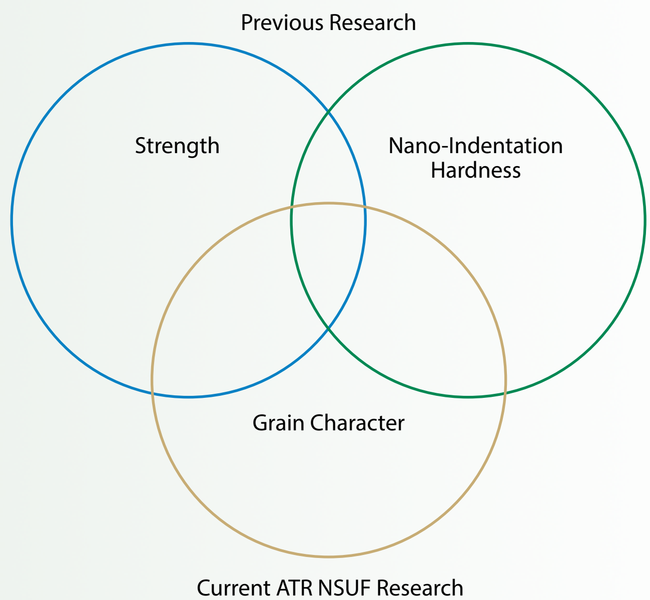

Figure 1. Schematic presentation of current ATR NSUF rapid turnaround experiment (RTE) shown in gold.

Accomplishments

The first technical goal, preparation and measurement of four experimental samples, began in April 2012 and established an EBSD technique for small samples with different geometries. The second technical goal was the actual measurement and interpretation of these results to determine the influence of annealing temperature on the SiC grain boundary characteristics. The EBSD measurements were performed during 2012 and 2013 by Boise State University researcher Jatu Burns, INL researcher Tammy Trowbridge, and Idaho State University nuclear engineering student Connie Hill. These measurements are now being interpreted and analyzed to answer the following research questions:

- Sample and location representativeness of EBSD measurement.
- Effect of sample preparation technique on grain characteristics.
- Effect of annealing time on grain characteristics.

- Effect of coater volume on grain characteristics.
- Effect of temperature on grain characteristics.
- Effect of SiC deposition temperature on grain characteristics.

Although interpretation is still in progress, some results have already been achieved. The effect of the annealing temperature on grain boundary characteristics of batches D and E is shown in Figure 2a. Although a slight increase in high-angle boundaries is observed with increased annealing temperature for batch E (black arrow), no significant trend is observed in the coincidence site lattice (CSL) boundaries with regard to temperature. Recent work by Eddie Lopez-Honorato et al. [5] showed that high-angle boundaries do enhance the diffusion of Ag. This leads to speculation that, upon annealing, the increase in high-angle boundaries in batch E will result in increased Ag release at 1600º C.

The behavior of batch D (blue arrow) is significantly different from that of batch E. A considerably higher fraction of high-angle boundaries was found in the initial reference sample, which decreased to approximately 50% after annealing at 2000º C. Consistent with batch E, no changes with regard to annealing temperature were observed in the CSL fractions in batch D.

In Figure 2b, a slight decrease of Σ3 grain boundaries for batch E is observed with increased annealing temperature. Khalil et al. [10] found by formation energy calculations that a strong segregation of Ag to the Σ3 grain boundaries are implied. Batch E showed about double the fraction of Σ3 CSL compared to those of batch D in the reference samples. It is therefore implied that batch E will initially have a lower diffusion of species compared with coated particles of batch D. The lower SiC deposition temperature of batch D is speculated to be the reason for this behavior.

Using the lineal intercept method average grain sizes as reported by Van Rooyen et al. [8], good correlation is achieved between the EBSD measurements and previously determined average grain sizes. These values showed an increase in average grain size with increased annealing temperature. The results obtained from the two methods for batch D did not show the same behavior, and remain under investigation. Although interesting, it was found in previous studies by Van Rooyen et al. [9] that the average grain size may not be the critical parameter to control for Ag transport behavioral prediction. Final conclusions on the grain characteristic trends with temperature need to be considered once a larger sample set is investigated. Additionally, these preliminary predictions of Ag transport will need to be confirmed in actual irradiated SiC studies.

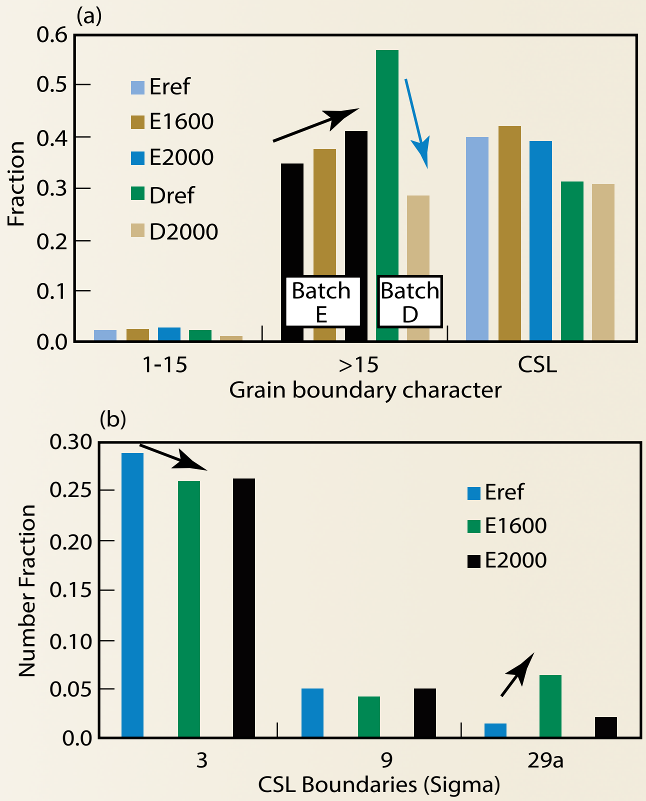

Figure 2. Effect of annealing temperature on (a) grain boundary character of batches D and E, and (b) CSL boundaries of batch E.

Future Activities

The experimental measurements were completed for this project and preliminary analyses showed interesting results. Goals for continuing research include:

- Publish a journal paper on the EBSD sample preparation technique.
- Interpret and integrate the results in the larger overarching project with a follow-up journal paper.
- Continue work with the project collaborators to complete the larger project.

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[2.] G.S. Roherer et al., Deriving Grain Boundary Character Distributions and Relative Grain Boundary Energies from Three-dimensional EBSD Data, *Materials Science and Technology,* Vol. 26, Issue 6 (2010), pp. 661-669.

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Correlating Silicon-Carbide Grain Size and Grain Boundary Orientation with Strength and Silicon-Carbide Layer Growth Conditions (cont.)

[3.] S. H. Kim et al., Grain Boundary Character Distribution and Intergranular Corrosion Behavior in High Purity Aluminum, *Scripta Materialia,* Vol. 44 (2001), pp. 835-839.

[4.] Takashi Fujita et al., Using Grain Boundary Engineering to Evaluate the Diffusion Characteristics in Ultrafine-Grained Al–Mg and Al–Zn Alloys, *Materials Science and Technology,* Vol. A 371 (2004), pp. 241-250.

[5.] Eddie Lopes-Honorato et al., Silver Diffusion in Coated Fuel Particles, *Journal of American Ceramic Society,* Vol. 93, (2010), pp. 3076-3079 [10].

[6.] Rita Kirchhofer et al., Microstructure of TRISO Coated Particles from the AGR-1 Experiment: SiC Grain Size and Grain Boundary Character, *Journal of Nuclear Materials*, Vol. 432, (2013), pp. 127-134.

[7.] Paul A. Demokowicz et al., Preliminary Results of Post-Irradiation Examination of the AGR-1 TRISO Fuel Compacts, *High Temperature Reactor Conference 2012*, Paper HTR2012-3-021.

[8.] Isabella J. van Rooyen et al., Effects of Phosphorous-Doping and High Temperature Annealing on CVD-Grown 3C-SiC, *Nuclear Engineering and Design*, Vol. 251, (2012), pp.191-202.

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[10.] Sarah Khalil et al., Diffusion of Ag Along Σ3 Grain Boundaries in 3C-SiC, *Physical Review,* Vol. B 84, Issue 214104 (2011), pp. 1-13.

Publications and Presentations

1. Isabella J. van Rooyen, Philip M. van Rooyen, Mary Lou Dunzik-Gougar, "The Effect of High-Temperature Annealing on the Grain Characteristics of a Thin Chemical Vapor Deposition Silicon Carbide Layer," *Microscopy and Microanalysis, 19 (Supplement 2),* August 2013, pp. 1948-1949. Published online: October 9, 2013: DOI: 10.1017/S1431927613011732.
2. Isabella J. van Rooyen, Philip M. van Rooyen, Mary Lou Dunzik-Gougar, "The Effect of High-Temperature Annealing on the Grain Characteristics of a Thin Chemical Vapor Deposition Silicon Carbide Layer," *Microscopy & Microanalysis Conference,* Indianapolis, Indiana, August 4-8, 2013.

<table>
<tr><th colspan="2">Distributed Partnership at a Glance</th></tr>
<tr><th>ATR NSUF & Partners</th><th>Facilities & Capabilities</th></tr>
<tr><td>Center for Advanced Energy Studies</td><td>Microscopy and Characterization Suite</td></tr>
<tr><th colspan="2">Collaborators</th></tr>
<tr><td colspan="2">Boise State University
Jatu Burns (collaborator)

Idaho National Laboratory
Isabella van Rooyen (principal investigator),
Tammy Trowbridge (collaborator)

Idaho State University
Mary Lou Dunzik-Gougar (co-principal investigator),
Connie Hill (MSc Nuclear Engineering student)

Nelson Mandela Metropolitan University
Jan Neethling (collaborator), Japie Engelbrecht (collaborator)

Network Consultants, South Africa
Philip M. van Rooyen (collaborator)</td></tr>
</table>

Principal Investigator: Di Yun – Argonne National Laboratory
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X-ray Characterization of Fission Gas Bubble Pressure in Ion-Irradiated Metallic Alloy Fuels

Introduction

The swelling of metallic fuels has been shown to directly affect the safety margins in operating nuclear reactors using these fuels. Understanding the behavior of fission gas bubbles is a key factor in the development of modeling standards for metallic fuels. The results of this research will also contribute to the advancement of nuclear fuel modeling in general and may provide significant insights into improving overall fuel designs for next-generation advanced reactors.

Project Description

The objectives of this research project are to determine the state of fission gas bubbles in metallic nuclear fuels and measure their internal pressures, particularly if they are in a solid or near-solid state. The project will provide researchers with a fundamental understanding of fission gas behavior under steady-state conditions, including an accurate reading of the internal pressure in bubbles that form in irradiated nuclear fuels.

Collecting this data will help clarify the understanding of the key mechanisms through which gas bubbles nucleate and grow, which causes the fuel itself to swell. It will also help eliminate one of the major uncertainties in the two types of mesoscale simulations of fission gas behavior in nuclear fuels: kinetic rate theory and the phase field approach.

As a result, this work provides critical information that fills one of the many gaps researchers face in their efforts to achieve a science-driven, multiscale simulation approach to understanding the behavior of materials in nuclear reactors.

Accomplishments

Project researchers at Argonne National Laboratory (ANL) conducted the ion implantation experiments. Implantation of Xenon (Xe) ions was performed at room temperature using a 250 keV Xe+ beam from ANL's Intermediate Voltage Electron Microscope (IVEM). Fission gas was inserted into a transmission electron microscopy (TEM) specimen of the metallic fuel alloy U-10Mo (10 wt.% Mo), which was then annealed in-situ to nucleate Xe bubbles. Figure 1 shows a bright field, under-focused micrograph showing the presence of Xe bubbles in the specimen.

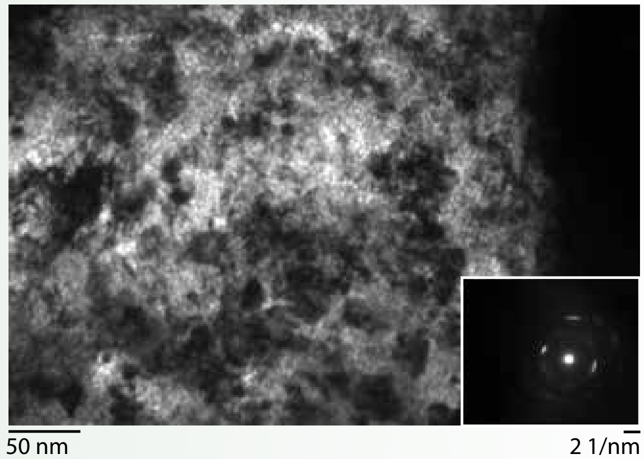

Figure 1. Under-focused TEM showing clear existence of gas bubbles, despite complex contrast after annealing at 600K for 20 – 30 minutes.

Researchers at ANL and the Illinois Institute of Technology (IIT) conducted extended X-ray absorption fine structure (EXAFS) measurements at room temperature on the IVEM in-situ TEM specimen. To meet the strict requirements of the Advanced Photon Source (APS) regarding the handling of radioactive materials, a triple-sealed container was used. In order to accurately calibrate the examination of the Xe, a target tank was filled with Xe gas and EXAFS data for gaseous state Xe was measured. Xe K-edge EXAFS data for the implanted and annealed TEM specimen were then taken.

It should be noted that performing these measurements was very difficult as the atomic concentration of Xe is estimated to be around only 1 atom %. Extremely long data-acquisition times (nearly 36 hours) were required in order to gather sufficient information.

Figure 2 shows the EXAFS data from these measurements. It is evident that even with 36 hours of data acquisition, the quality of the information collected is less than satisfactory. However, a preliminary conclusion can still be reached that the Xe bubbles in the implanted and annealed TEM specimen were in a gaseous state. There is no significant wiggling at the K-edge, indicating that the Xe atoms do not form a crystal lattice. Therefore, when conducting any future EXAFS examinations, much higher concentrations of Xe will be required to increase the effectiveness of the measurements.

"Through our work on this project, we have learned a great deal about the properties of fission gas bubbles in ion-irradiated nuclear fuel materials and would like to thank ATR NSUF for funding this project."

Di Yun, Nuclear Engineer, Argonne National Laboratory

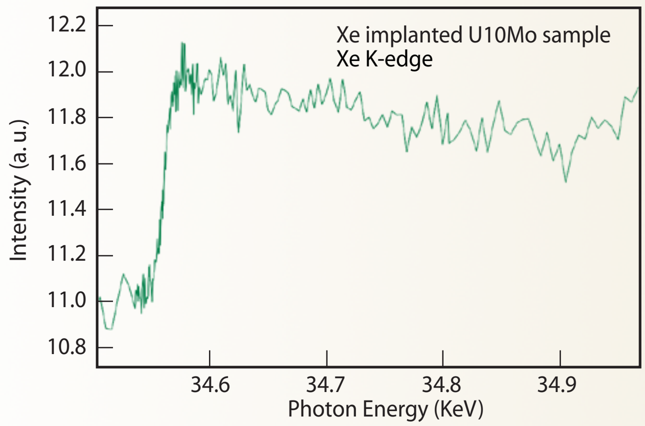

Figure 2. K-edge EXAFS data for the implanted and annealed TEM specimen.

Increased knowledge of the fission bubble state is critical for any model development of fission gas behaviors in nuclear fuels.

Future Activities

No future activities are planned after the completion of these APS experiments.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Illinois Institute of Technology	Materials Research Collaborative Access Team at the Advanced Photon Source
Collaborators	
Argonne National University Di Yun (principal investigator) **Illinois Institute of Technology** Jeff Terry (co-principal investigator), Kevin Logan (collaborator)	

Principal Investigator: Xinghang Zhang – Texas A&M University
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Critical Evaluation of Radiation Tolerance of Nanocrystalline Austenitic Stainless Steels

Introduction

Using nanocrystalline (NC) Fe-14Cr-16Ni (wt%), 304L and 316L stainless steels (SS) as candidate materials, researchers will examine the radiation tolerances of NC austenitic SS under proton and heavy ion irradiations. The team will investigate the following hypotheses:

- High-angle grain boundaries can effectively reduce void swelling in these SS.
- Radiation hardening in austenitic SS can be significantly suppressed at a moderate temperature of ~ 350° C.
- High-angle grain boundaries (or small-grain sizes) remain stable when irradiated at elevated temperatures (up to 500° C).

The rationale behind these hypotheses is that the large volume fraction of high-grain boundaries can act as effective sinks for point defects, while mitigating radiation damage by promoting the annihilation of vacancies and interstitials [1, 2].

The long-term goal of this experiment is to design and fabricate bulk nanostructured austenitic SS that obtain the similar magnitude of void swelling resistance as ferritic/martensitic (F/M) steels, such as HT-9, and exhibit significantly reduced radiation hardening.

This ATR NSUF project also ties into the Nuclear Energy University Programs' research and development objectives to develop technologies that can improve the reliability, sustain the safety, and extend the life of current reactors, and to develop sustainable nuclear fuel cycles, as well as the Fuel Cycle 2 (advanced fuels) key university need: fuel-related core materials tolerant to light water reactor (LWR) beyond-design-basis events.

Nanostructured austenitic stainless steels having superior radiation resistance to void swelling could be candidates as structural material in advanced nuclear reactors.

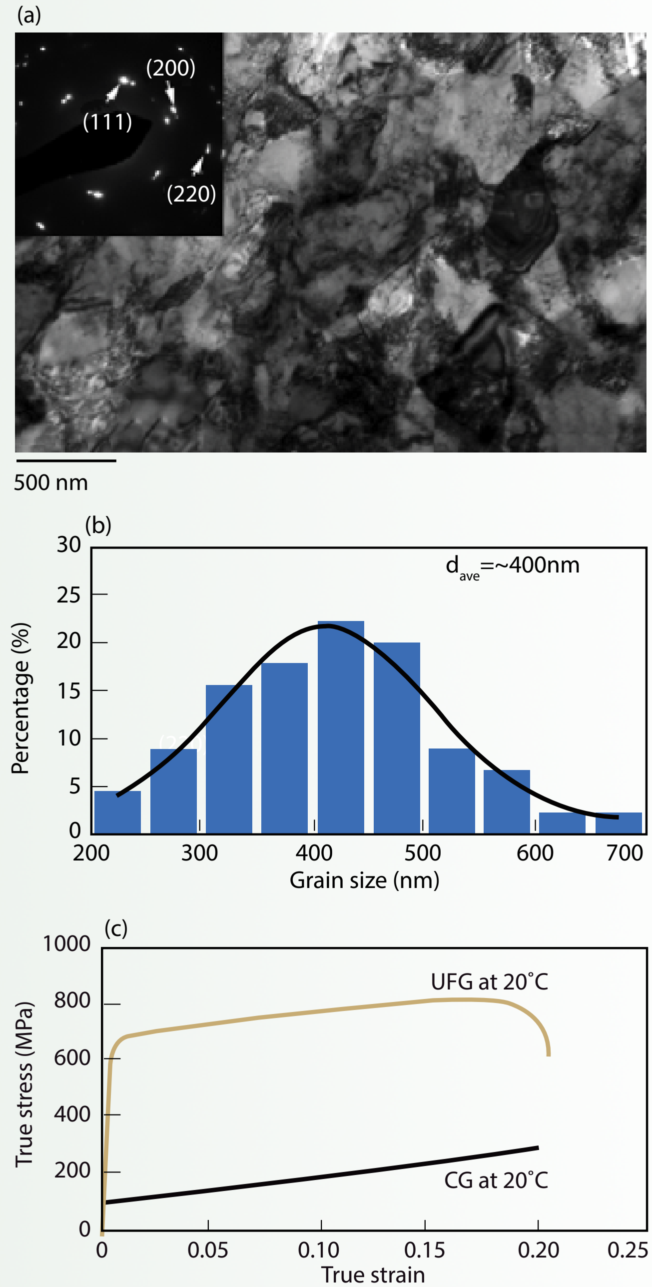

Figure 1. (a) TEM micrograph of UFG Fe-14Cr-16Ni. (b) Chart showing the average grain size in the samples is ~400 nm. (c) Graph showing the yield strength has increased from 100 megapascals (MPa) for a CG sample to over 600 MPa for a UFC sample with good ductility.

"The benefits of this ATR NSUF project include gaining insights into the physics of ion-solid and defect-grain boundary interactions, probing the responses of nanocrystalline austenitic stainless steels subjected to high-dose radiation, and enriching the database of radiation responses in nuclear reactor steels."

Xinghang Zhang, Associate Professor of Mechanical Engineering, Texas A&M University

Project Description

Researchers have successfully reduced the grain sizes of austenitic SS from several hundred µm down to ~100–400 nm. These nanograins are thermally stable up to 600° C, and show enhanced tolerance against helium (He) ion irradiation. These studies pave the way for future investigations that will involve much more rigorous examinations of radiation and thermal stability in NC SS under neutron, proton, and heavy ion irradiations, as well as post-irradiation examinations.

Compared to F/M steels, austenitic SS possess good creep resistance at elevated temperatures and increased toughness at low temperatures [3, 4]. However, a major disadvantage of austenitic SS is a vulnerability to significant void swelling in nuclear reactors, especially at the temperatures and doses that are anticipated in an Advanced Burner Reactor [3, 4]. This lack of resistance to void swelling in austenitic alloys led to the switch to F/M steels as the preferred material for fast reactor cladding applications. In addition, SS are also susceptible to significant radiation hardening at ~350° C.

Accomplishments

The following are the experiments that have been performed and their results:

- Developed a refined ultra-fine-grain (UFG) Fe-14Cr-16Ni (wt%) alloy with a grain diameter of ~400 nm, and NC 304L with a diameter of 100 – 250 nm. A tensile test experiment showed the UFG FeCrNi had a much higher yield strength than coarse-grained (CG) FeCrNi with good ductivity (Figure 1).
- He ion irradiation shows that irradiated, CG Fe-14Cr-16Ni has a high density of He bubbles and dislocation loops, while the bubble density in the UFG alloy was two times lower than that in its CG counterpart, and no dislocation loops were observed (Figure 2).

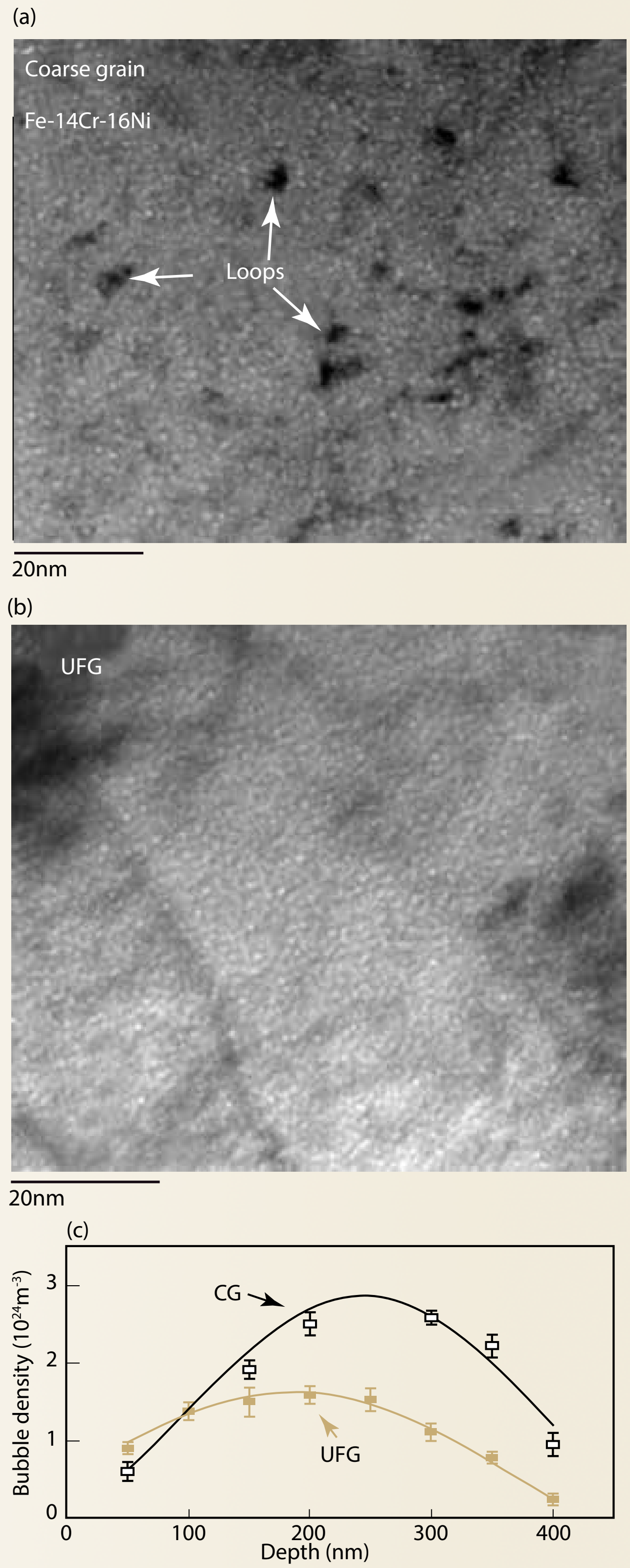

Figure 2. TEM micrograph of He ion irradiated Fe-Cr-Ni alloy (100 keV/6 x 10^{16}/cm^2 at peak damage (~5 displacements per atom [dpa]). (a) CG alloys have a high density of He bubbles and dislocation loops. (b) In the UFG alloy, no dislocation loops were observed. (c) Graph showing He bubble density in the UFG alloy was two times lower than that of the CG alloys.

Principal Investigator: Xinghang Zhang – Texas A&M University (cont.)
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Critical Evaluation of Radiation Tolerance of Nanocrystalline Austenitic Stainless Steels (cont.)

- A thermal stability study of NC 304L SS shows no significant change in hardness up to 600° C/10h annealing (Figure 3).
- Proton irradiation experiments were performed on FeCrNi and 304L SS using the Tandem Accelerator Ion Beam at the University of Wisconsin. These studies pave the way for much more rigorous examinations of radiation and the thermal stability of NC SS under neutron, proton and heavy ion irradiations.
- Heavy ion irradiation was performed on NC 304L SS in the Michigan Ion Beam Laboratory at the University of Michigan.

Future Activities

Goals for the project in 2014 are:

- Characterize the structures of proton-irradiated NC and CG SS, including NC Fe-14Cr-16Ni (wt%) and 304L SS, by X-ray diffraction, cross-section transmission electron microscopy (TEM), and scanning transmission electron microscopy (STEM), and examine the mechanical properties of the materials after irradiation.
- Begin the neutron irradiation experiment on specimens loaded in ATR (eight to 12 month radiation).
- Perform heavy ion irradiation of austenitic SS at the University of Michigan's user facility.
- Examine void swelling and radiation hardening in nanostructured steels.

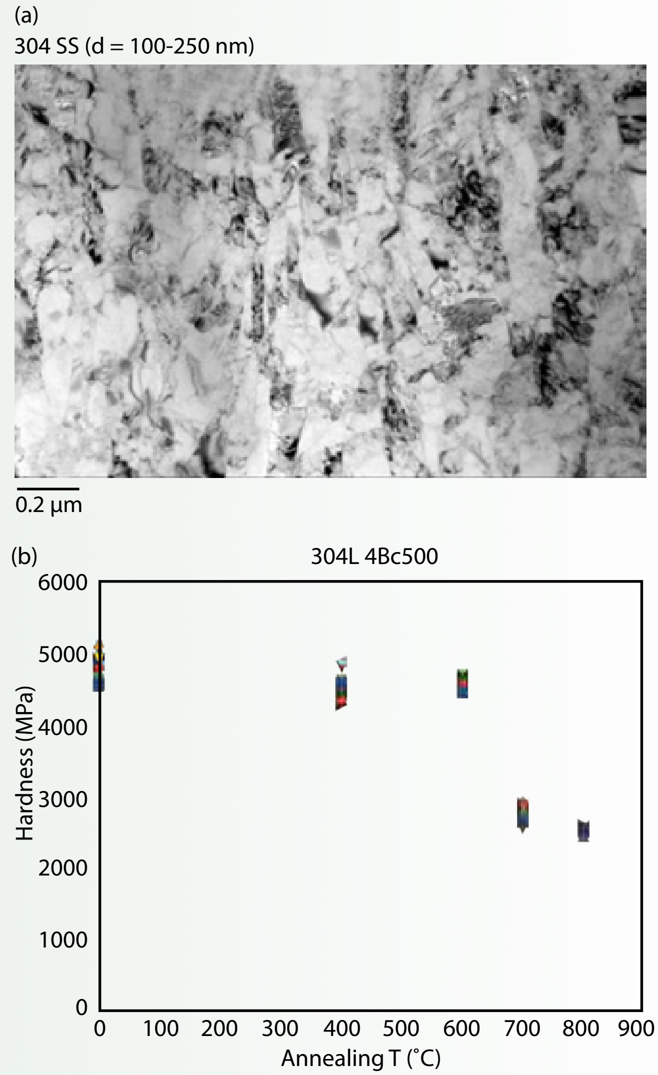

Figure 3. (a) TEM micrograph of the equal channel angular processed NC 304L SS sample. The average grain size is ~100 nm by 250 nm. (b) Graph showing the hardness of the sample remained the same after annealing up to 600° C/10h.

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[1.] Amit Misra, Michael Demkowicz, Xinghang Zhang, Richard Hoagland, "The Radiation Damage Tolerance of Ultra-High Strength Nanolayered Composites," JOM, 59 (2007) 62.

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[3.] Gary S. Was, Fundamentals of Radiation Materials Science, Springer, New York, 2007.

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Publications and Presentations*

Cheng Sun, Kaiyuan Yu, Youxing Chen, Miao Song, Marquis Kirk, Haiyang Wang, Meimei Li, Xinghang Zhang, "In-situ Evidence of Defect Cluster Absorption by Grain Boundaries in Kr Ion-Irradiated Nanocrystalline Ni," *Metallurgical and Materials Transactions A,* Vol. 44, No. 4, April 2013, pp. 1966-1974. DOI 10.1007/s11661-013-1635-9.

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

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor
University of Michigan	Michigan Ion Beam Laboratory
University of Wisconson	Tandem Accelerator Ion Beam
Collaborators	
Idaho National Laboratory Todd Allen (co-principal investigator)	
Los Alamos National Laboratory Stuart Maloy (collaborator)	
Texas A&M University Xinghang Zhang (principal investigator), Ted Hartwig (co-principal investigator), Yong Yang (co-principal investigator), Kaiyuan Yu (collaborator), Cheng Sun (collaborator), Youxing Chen (collaborator)	
University of Wisconsin Gary Was (collaborator)	

Principal Investigator: John H. Jackson – Idaho National Laboratory
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Irradiation and Post-Irradiation Examination of Alloys X-750 and XM-19: Electric Power Research Institute Pilot Program, Phase III

Introduction

As a means of establishing a basis for development and execution of a joint ATR NSUF/private industry program, the Electric Power Research Institute (EPRI) and Battelle Energy Alliance (BEA) have developed a pilot program (referred to as the EPRI Pilot Project) involving shared costs and responsibilities. Through a Cooperative Research and Development Agreement (CRADA), in addition to providing data, the pilot program is designed to:

- Develop the administrative protocols for joint research, such as cooperative agreements and funding.
- Develop portions of the capability and staffing required to address future research and development needs.
- Develop a protocol for validation of data with industry; particularly that associated with stress corrosion cracking (SCC) growth rates.

This project is important for three reasons: first, as the inaugural industry pilot project for ATR NSUF it establishes a protocol for these types of projects going forward. Second, it is a full cradle-to-grave characterization of reactor internal materials, including baseline characterization, irradiation and post-irradiation examination (PIE). Third, it is the first project to utilize the newly installed loop in the ATR center flux trap, and the newly installed irradiation-assisted stress corrosion cracking (IASCC) test systems.

Project Description

Discussions between ATR NSUF and EPRI regarding an area of interest for this initial project resulted in a decision to focus on an investigation of the fracture toughness and IASCC growth rates of irradiated high-strength alloys used for Boiling Water Reactor (BWR) repair hardware. (fig. 1) Very little data exist on these phenomena for the nickel (Ni)-based alloy X-750, and the nitrogen (N)-strengthened austenitic stainless steel XM-19 at the exposure levels of interest—up to $1x10^{21}$ n/cm^2. This three-phase project will characterize these alloys in both unirradiated (baseline) and irradiated states. The first two phases have been completed.

During Phase I (CRADA 09-CR-02) researchers fabricated specimens for the pilot project from materials provided by EPRI and established the baseline fracture toughness and crack growth rates of the unirradiated material. In Phase II (CRADA 10-CR-13) they designed and fabricated the specimen holders and performed a safety analysis on a test train to meet EPRI objectives for irradiation, tensile, and compact tension specimens in the ATR center flux trap, a pressurized water loop.

In Phase III (CRADA 12-CR-06) researchers are currently performing irradiation and PIE of the EPRI specimens delivered to BEA following phases I and II.

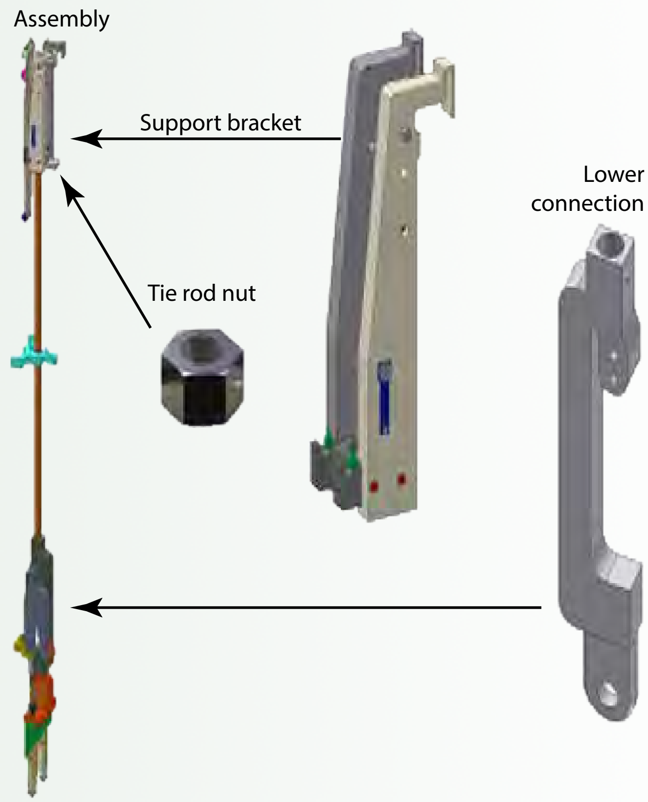

Figure 1. Repair hardware of interest for this study.

Accomplishments

Phases I and II of this pilot project were completed in 2012. Reports containing the results were issued within INL and by EPRI.

Test specimens of both X-750 and 19.3% cold-worked XM-19 alloys were fabricated and measured for baseline material properties prior to irradiation in Phase I. At the beginning of Phase III, these specimens underwent several baseline SCC tests. Utilizing the cold-worked XM-19 presented an opportunity to study potential similar material property effects between neutron embrittlement and embrittlement induced by cold-working the material. Fracture toughness and tensile tests were also performed at temperature (not in the environment).

Results of the SCC tests were compared to data produced at General Electric Global Research Company as a means of benchmarking INL's ability to perform these highly specialized experiments. In all cases, the measured crack growth rates from the tests performed at INL compared favorably with those produced by the benchmark laboratory (Figures 2 and 3).

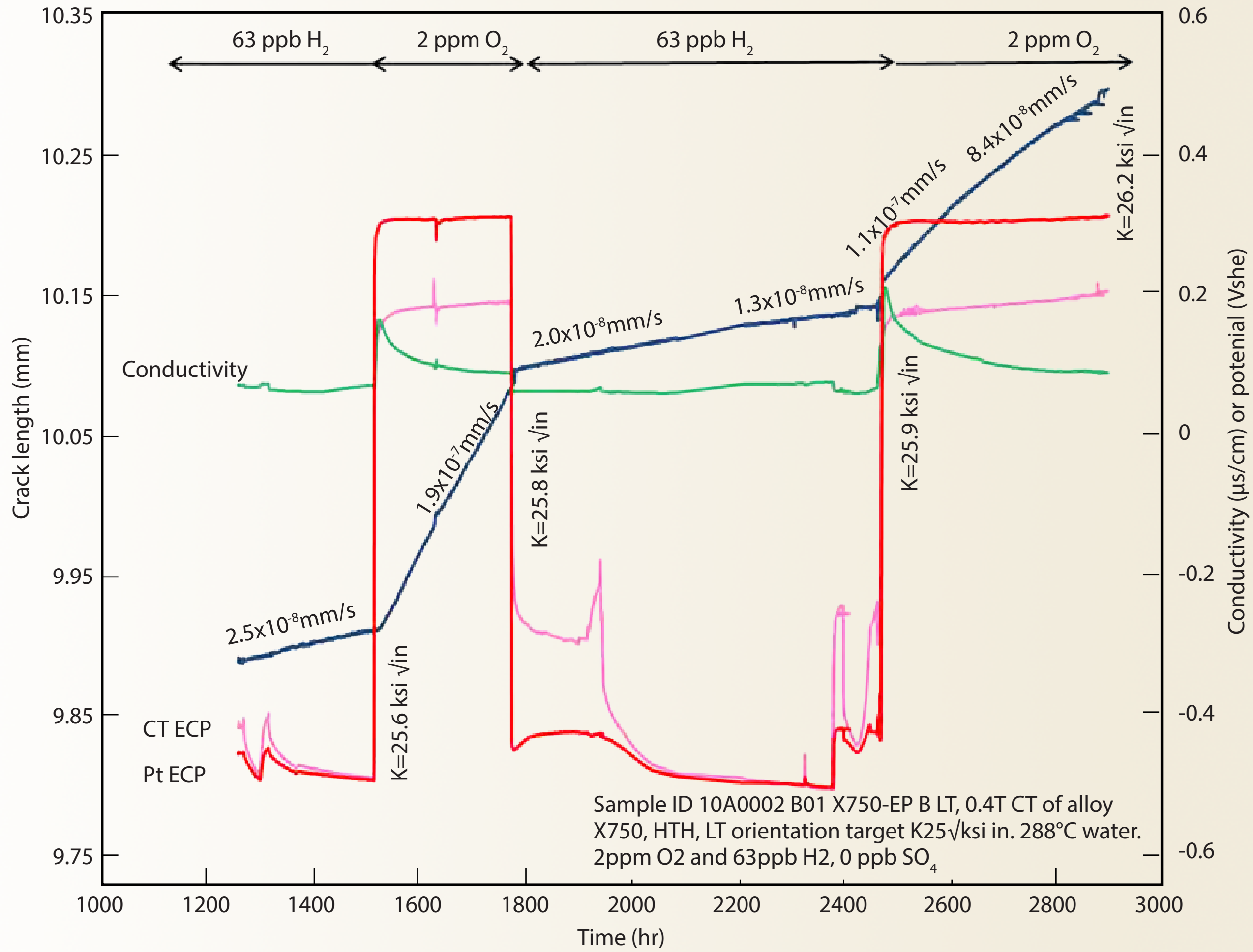

Figure 2. Typical baseline SCC test results for alloy X-750. Crack growth rate is the dark blue line.

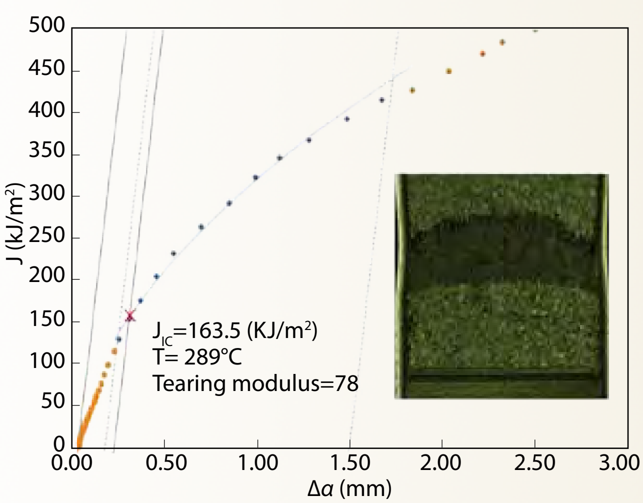

Figure 3. Typical fracture toughness test results for alloy X-750 at elevated (289º C) temperature.

The EPRI Pilot Project is ATR NSUF's first project with private industry, and will establish protocol for these types of programs going forward.

The Phase II report, issued in February 2012, contained results of the physics and safety analysis performed on the specimens, along with the test train design for irradiating materials in the ATR center flux trap.

Irradiation and Post-Irradiation Examination of Alloys X-750 and XM-19: Electric Power Research Institute Pilot Program, Phase III (cont.)

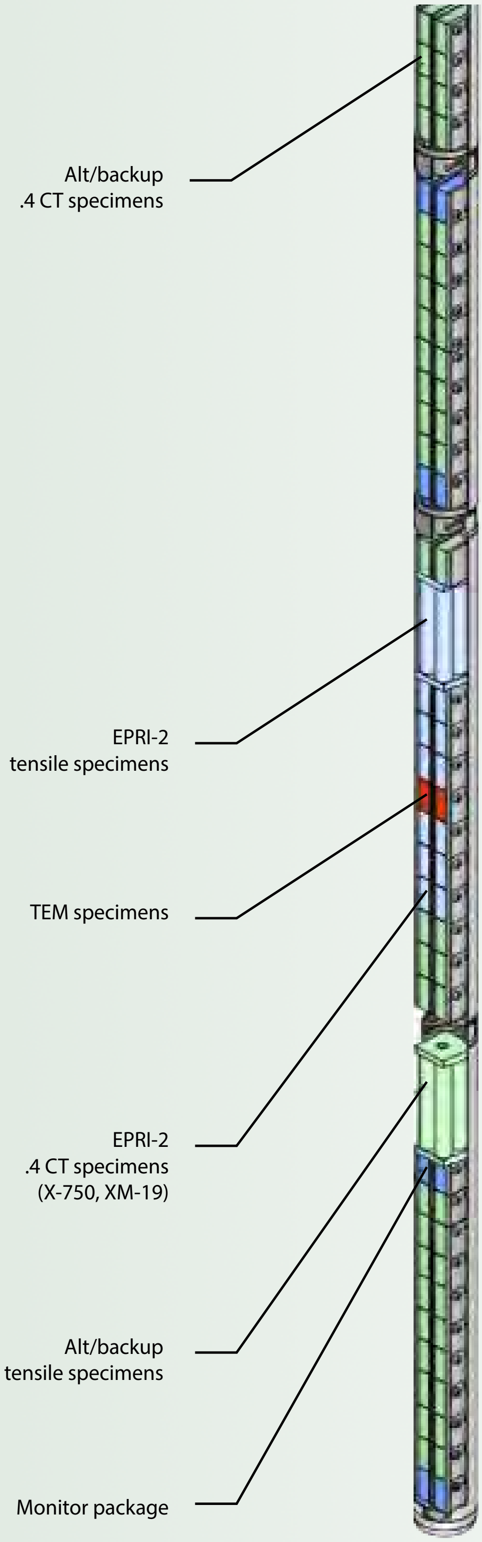

Figure 4. Typical irradiation test train containing compact tension, tensile, and transmission electron microscopy specimens.

Irradiation of the EPRI-2 capsule was completed in 2013 in the ATR center flux trap. Researchers utilized the newly installed, controlled water chemistry loop with a target fluence of 2.0×10^{20} n/cm^2 (E>1MeV). Following the irradiation and a cool-down, the capsule was shipped to the Hot Fuels Examination Facility (HFEF) where it was disassembled in preparation for PIE.

Future Activities

The newly installed IASCC test systems are expected to be fully functional in January 2014. Following readiness activities, IASCC and fracture toughness testing will commence on the EPRI-2 specimens. Concurrently, two additional test capsules will be irradiated in the ATR center flux trap: EPRI-1 (5.0×10^{19} n/cm^2) and EPRI-3 (1.0×10^{21} n/cm^2). Both are expected to be ready for shipment to HFEF in early 2015, with testing of those specimens continuing into 2016.

Publications and Presentations

EPRI, 2012, *BWRVIP-262NP: BWR Vessel and Internals Project, Baseline Fracture Toughness and Crack Growth Rate Testing of Alloys X-750 and XM-19 (Idaho National Laboratory Phase I),* 1025135, August 6, 2012, Palo Alto, CA.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Collaborators	
Electric Power Research Institute Robert Carter (program manager), Peter Chou (collaborator), Raj Pathania (collaborator)	
Idaho National Laboratory John H. Jackson (principal investigator), Sebastien Teysseyre (co-principal investigator)	

Principal Investigator: John H. Jackson – Idaho National Laboratory
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Idaho National Laboratory-Atomic Energy of Canada Limited Joint Project for Active Focused Ion Beam and Transmission Electron Microscopy Analysis of Irradiated X-750

Introduction

Relicensing efforts for light water reactors (LWR) are directly related to a number of issues surrounding their internal components. Alloy X-750 is a nickel-based super alloy that is commonly used in these components, as well as in the Canada Deuterium Uranium (CANDU) reactors developed in the late 1950s and 1960s. Embrittlement in X-750 caused by exposure to a neutron environment is of interest in predicting the lifetimes of the current fleet of reactors around the world.

Project Description

Several garter spring sections that had been used as fuel channel spacers in CANDU reactors (Figure 1) were provided to INL by the Atomic Energy of Canada Limited's (AECL) Chalk River Laboratory for examination of microstructural changes in X-750 caused by neutron irradiation and temperature. These spring sections exhibited extreme loss of ductility during service inspections and have become the subject of fitness-for-service investigations by the CANDU reactor industry. Preliminary field inspections indicated a potential relationship between loss of ductility and the location of the spring sections in the reactor, and a thorough microstructural analysis was deemed necessary in order to understand this behavior.

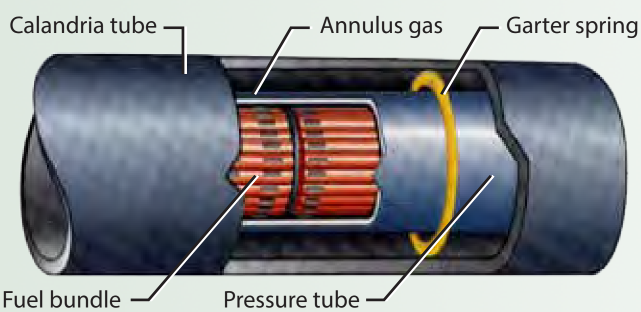

Figure 1. Depiction of a CANDU reactor fuel channel showing an X-750 garter spring spacer.

Accomplishments

In late 2011, AECL shipped INL a number of 1 mm x 1 mm cubes of irradiated X-750 taken from garter springs that had been removed from several regions of interest. Figure 2 is a representation of a typical garter spring with the calculated temperature profile during service.

It was noted that some regions of the springs exhibited ductility, while others were severely embrittled. Since temperature can contribute to embrittlement, specimens were selected from spacer regions near the 6 o'clock position (lower temperature due to pinching against the cooler calandria tube) and the 12 o'clock position (higher temperature). Additional specimens were extracted from

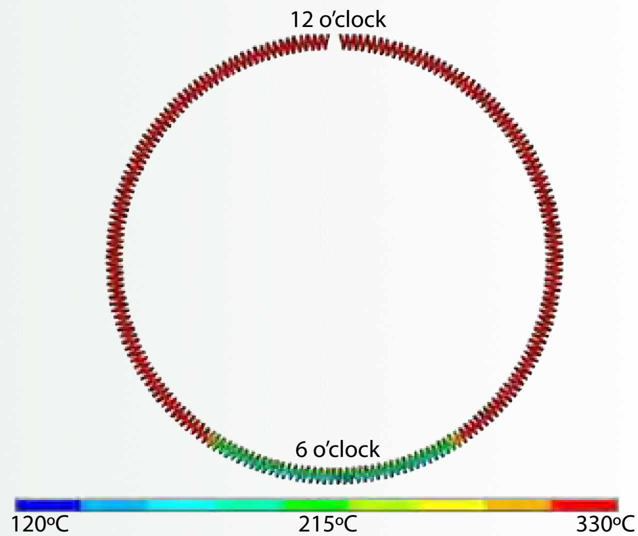

Figure 2. Temperature profile in an X-750 spacer spring.

springs that had accumulated displacement per atom (dpa) levels between 6 and 55, and that contained differing levels of helium (He) between 1,500 and 18,000 atomic parts per million (appm).

During 2012 and 2013, transmission electron microscopy (TEM) foils (Figure 3) were produced using the active focused ion beam (FIB) at INL's Electron Microscopy Laboratory (EML). Analyses were performed by a visiting AECL scientist in the Microscopy and Characterization Suite (MaCS) at CAES. In addition, hardness of the specimen sections was measured using a shielded microhardness tester in EML.

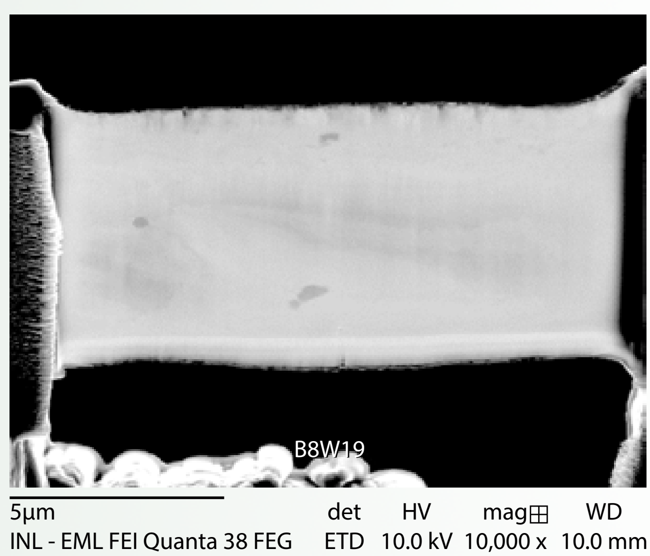

Figure 3. SEM image of a FIB-fabricated TEM foil.

"This collaboration between AECL and INL's ATR NSUF has been instrumental in determining and understanding the degradation mechanisms of heavily irradiated Inconel X-750 CANDU spacers."

Colin D. Judge, R&D Scientist, Deformation Technologies Branch, Fuel Channel Division, Chalk River Laboratories

After conducting scanning electron microscopy (SEM) fractography studies, the AECL scientist noted that there was a definite difference in ductility between spring specimens from the hot and cold regions of the reactor (Figure 4). Specimens from the hotter 12 o'clock region exhibited brittle fracture features at lower dpa levels, while at higher dpa levels and increased He content, there was no difference due to changes in temperature (Figure 5).

This work will provide much needed information toward understanding the evolution of material microstructure as a function of temperature and neutron fluence.

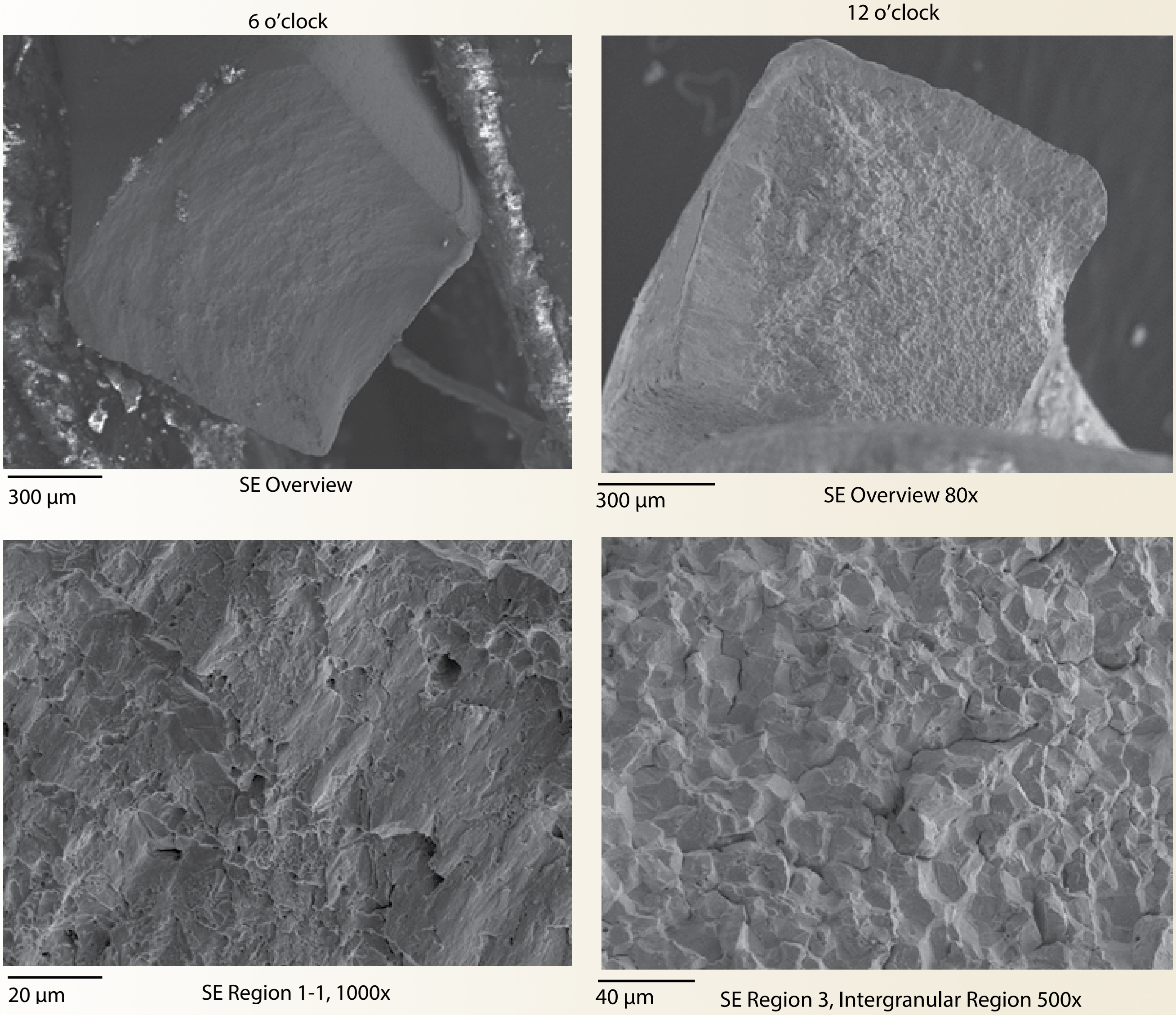

Figure 4. Fractography of 6 o'clock and 12 o'clock specimens at ~6 dpa and 1,000 appm He.

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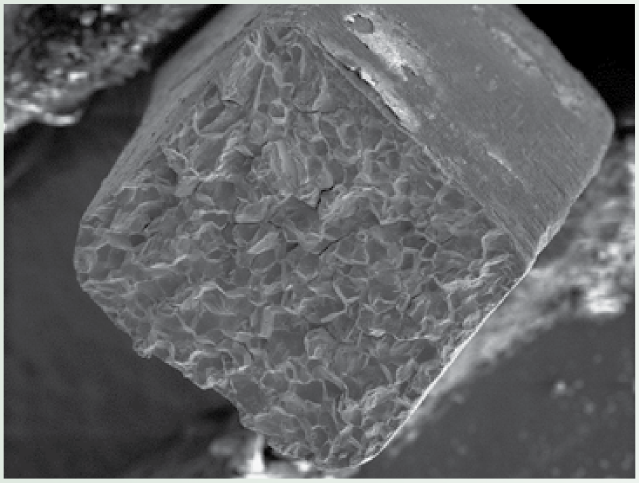
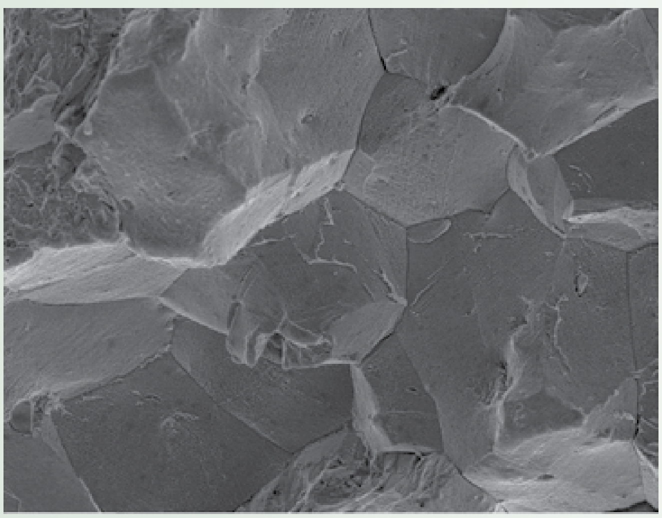
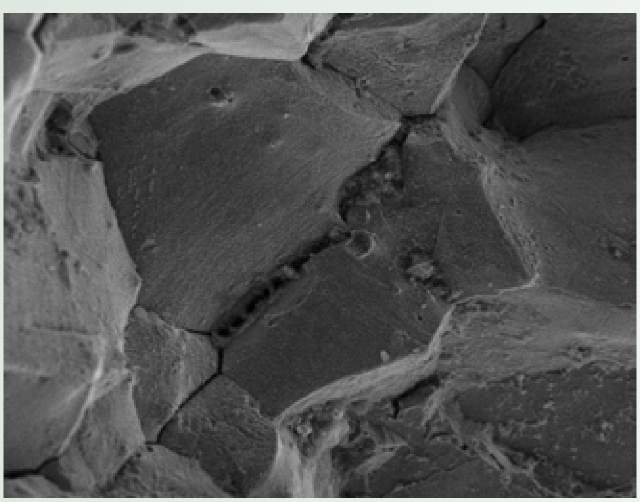
Idaho National Laboratory-Atomic Energy of Canada Limited Joint Project for Active Focused Ion Beam and Transmission Electron Microscopy Analysis of Irradiated X-750 (cont.)



200 µm SE Overview 100x



20 µm SE Region 2-1 1000x

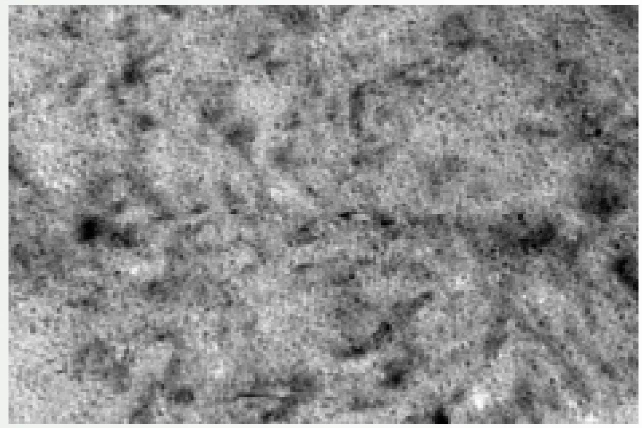


10 µm SE Region 1-2 1300x

Figure 5. Fractography of 6 o'clock and 12 o'clock specimens at ~55 dpa and 18,000 appm He showing no appreciable difference.

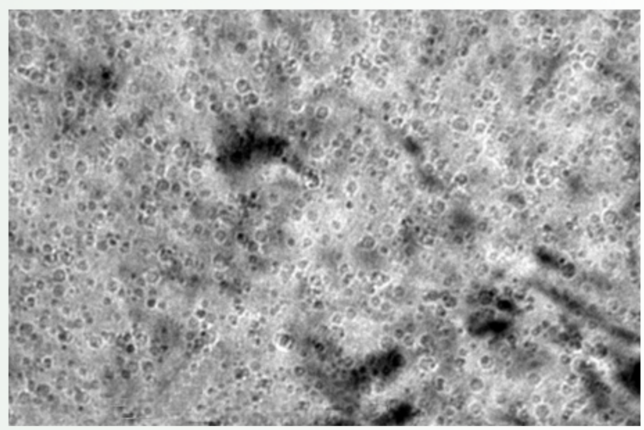
In correlation with these results, the TEM studies performed during the same time period show a difference in cavity/bubble characteristics (Figure 6) between high- and low-temperature regions, as well as cavity/bubble coalescence at higher dpa levels.

6 o'clock



100 nm

12 o'clock



100 nm

Figure 6. TEM images showing higher density of smaller cavities/bubbles in cooler region.

Future Activities

In 2014, additional TEM foil specimens will be produced using EML's active FIB. These specimens will be examined to verify and enhance some of the original findings.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	PIE facilities
Collaborators	
Atomic Energy of Canada Limited Colin Judge (co-principal investigator), Malcolm Griffiths (manager, Deformation Technologies Branch, Chalk River Laboratories)	
Idaho National Laboratory John H. Jackson (principal investigator), James Cole (collaborator), James Madden (collaborator), Brandon Miller (collaborator)	

Principal Investigator: John H. Jackson – Idaho National Laboratory
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Nuclear Regulatory Commission Irradiation and Testing of Austenitic Stainless Steel in Boiling Water Reactor Conditions

Introduction

The core internals of a commercial light water reactor (LWR) are designed to provide the structural support needed to maintain the core fuel assemblies in a coolable geometry. When exposed to neutron irradiation during power operation, the introduction of dislocations, vacancies and segregation precipitates into the microstructure causes embrittlement and creep. It can also cause dimensional changes from void swelling due to the production of helium (He) bubbles within the microstructure. All of these may impact the structural integrity of these core components.

In support of an assessment of inspections related to Materials Reliability Program (MRP) 227, members of the Nuclear Regulatory Commission (NRC) are conducting confirmatory research to address the void swelling behavior of light water reactor (LWR) core internal materials due to neutron irradiation. The intent is to quantify the expected dimensional changes in LWR internals as a function of fluence, and the potential impact of these changes on their structural integrity.

Void swelling requires exposure to very high neutron fluences. Thus it would be desirable to use a high-flux irradiation facility such as ATR for the preparation of specimens. However, high-neutron fluxes are accompanied by high-gamma doses, which can affect the evolution and growth dynamics of irradiation damage. To consider high-neutron dose irradiations in ATR for void swelling investigations, the NRC needs to ascertain that gamma heating effects can be controlled.

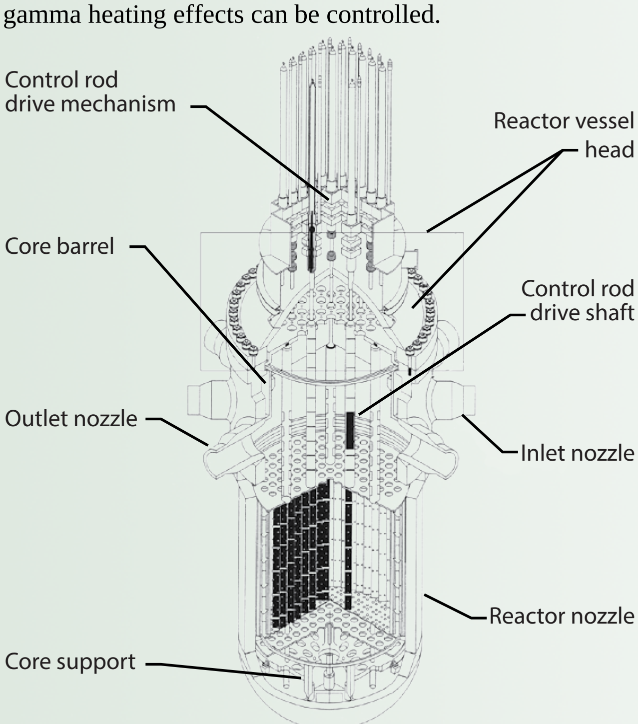

Figure 1. Cutaway of pressurized light water reactor.

This joint ATR NSUF/NRC project will enable ATR to significantly contribute to the determination of lifetime extension for the nation's light water reactor fleet.

Project Description

As a first step toward full-scale utilization of the ATR and associated post-irradiation examination (PIE) equipment, the NRC and ATR NSUF have formulated a test program that is designed to establish INL as a viable destination for irradiation and PIE of reactor structural materials.

The NRC staff has already conducted irradiations of austenitic stainless steel specimens to 1.2-1.4 displacements per atom (dpa) in the Halden research reactor in Norway. These specimens were irradiated at prototypical boiling water reactor (BWR) temperatures, and were exposed to limited gamma heating, thereby limiting migration of defects during irradiation. In order to establish the ability of ATR to simulate these conditions, this project will produce samples irradiated to approximately 1.3 dpa that can be compared to the Halden specimens.

This initial program will utilize two materials – a sensitized 304 stainless steel and a 304L stainless steel weld-heat-affected zone – to supplement test results from specimens that were previously irradiated at the Halden reactor and tested at Argonne National Laboratory (ANL). Baseline fracture toughness and stress corrosion cracking (SCC) growth rates will also be characterized, as well as irradiated fracture toughness and irradiation-assisted stress corrosion cracking (IASCC) growth rates. A secondary objective of this test program will be to characterize the quality of data produced using INL's newly constructed IASCC test cells.

This project is directly related to the DOE's LWR Sustainability Program as it seeks to enhance the currently sparse data set regarding IASCC of LWR internal materials. Results of this study will be used to enable remaining life predictions of components subjected to the harsh reactor internal environment.

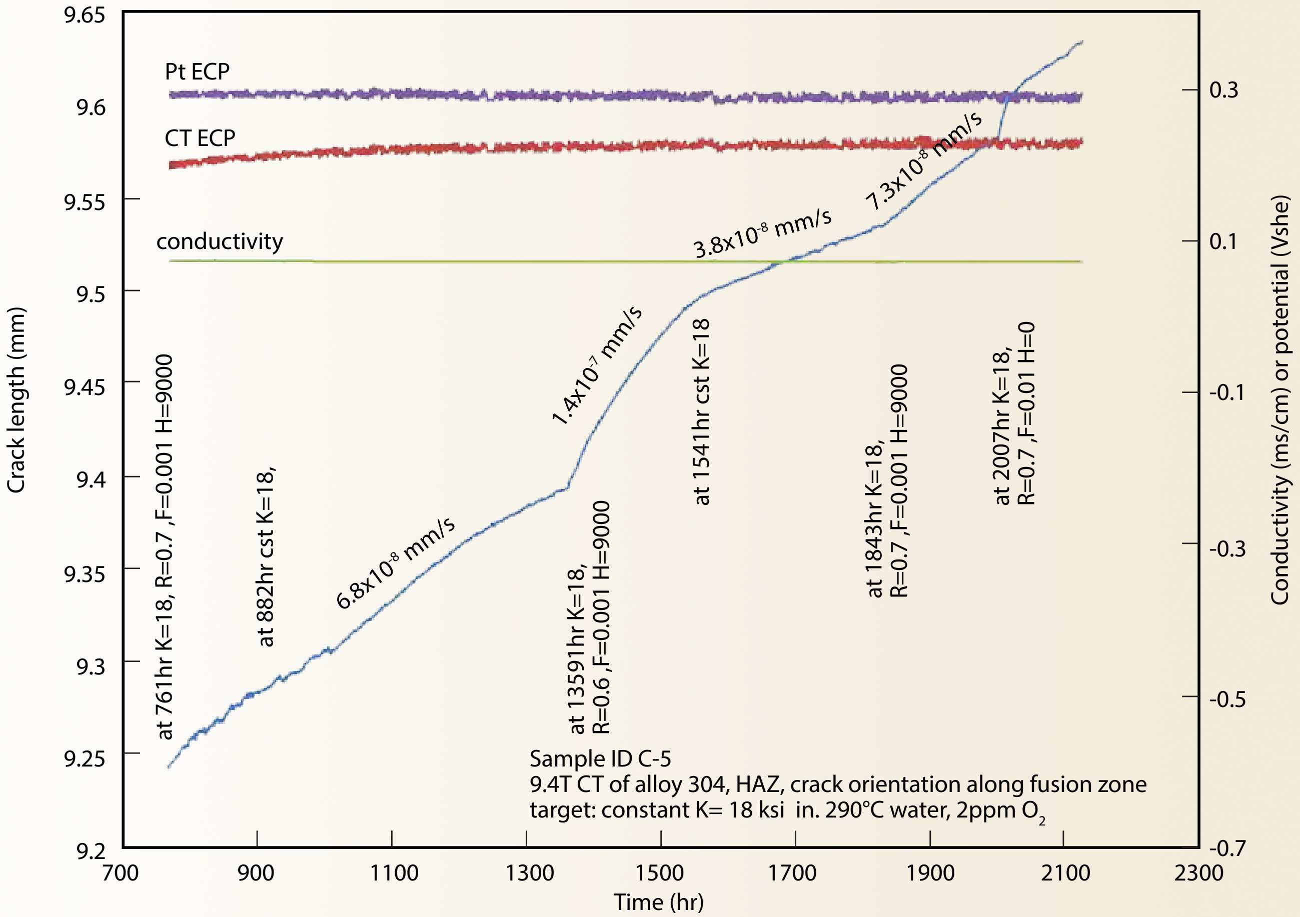

Figure 2. Crack growth rate in 304L weld heat-affected zone under BWR oxidizing conditions and subjected to constant K=18 ksi√in.

Accomplishments

In 2013, baseline characterizations of unirradiated materials were completed. This included SCC crack growth testing at a nominal constant applied stress intensity factor (K), as well as fracture toughness testing of both materials while in a BWR oxidizing environment (normal water chemistry). Figure 2 shows a portion of the SCC crack growth rate curve measured for the 304L weld heat-affected zone material. While held at a constant K =18 ksi-in$^{1/2}$, the growth rate ranged between 3.8 and 6.8x10^{-8} mm/s.

Immediately following the crack growth rate test, the specimens were subjected to a fracture toughness test while in the same environment, with the initial crack front in SCC (intergranular) crack growth mode. No significant effect was detected in either case, although the sensitized 304 stainless steel specimen contained multiple out-of-plane cracks that complicated the measurement. The material behaved in a ductile manner, and since the blunting line followed the J-R trend very closely in the vicinity where this would typically be calculated, an accurate value for J_{IC} (fracture toughness) is difficult to ascertain.

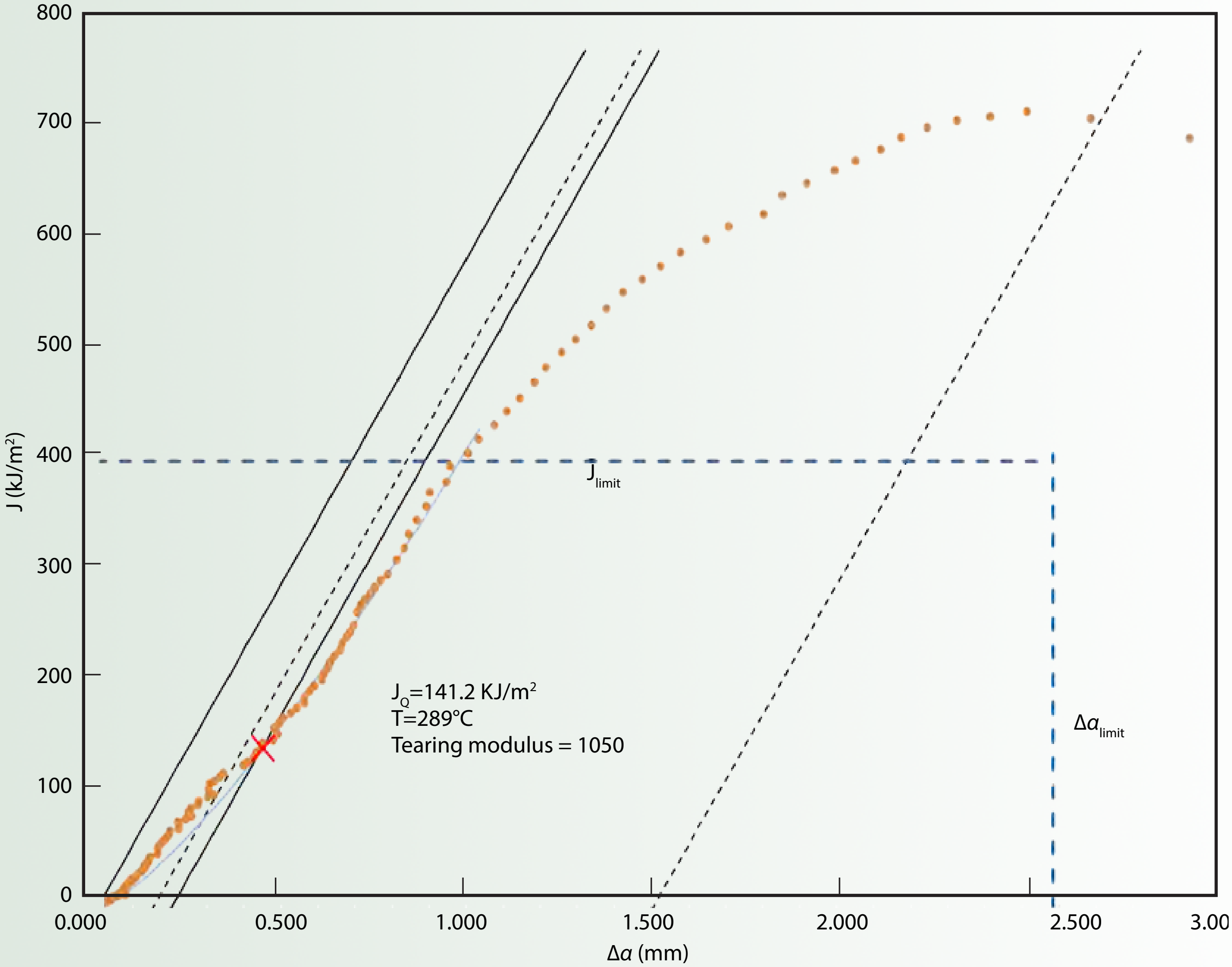

Figure 3. J-R curve for 304L weld heat-affected zone specimen in a BWR oxidizing environment.

Principal Investigator: John H. Jackson – Idaho National Laboratory (cont.)
email: john.jackson@inl.gov

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Collaborators	
Idaho National Laboratory John H. Jackson (principal investigator), Sebastian Teysseyre (co-principle investigator) **Nuclear Regulatory Commission** Matthew Hiser (NRC program manager), Appajousula (Sri) Rao (technical program monitor)	

Future Activities

Irradiation of the material for this project is expected to be completed in early summer 2014. The material will be shipped from ATR to the Hot Fuels Examination Facility (HFEF) in late 2014. It is anticipated that IASCC and fracture toughness testing of these materials will commence in early 2015 and will be completed by the end of 2016.

Principal Investigator: Paul Murray – Idaho National Laboratory
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Irradiation and Post-Irradiation Examination to Investigate Hydrogen-Assisted Anomalous Growth in Zirconium Alloys

Introduction

Within the last five years, several incidences of anomalous fuel channel bowing have been observed in boiling water reactors (BWR) using zircaloy-2 (Zry-2). This anomaly has been associated with shadow corrosion that occurs on the fuel channel surface opposite a stainless steel control blade, but only when the blade is deeply inserted into an assembly early in life. The bowing correlates with higher hydrogen content on one side of the fuel channel box. However, the extent of the bowing cannot be explained by the differential hydrogen content alone, which suggests an additional growth mechanism may be operative.

Project Description

This joint Battelle Energy Alliance (BEA)/Electric Power Research Institute (EPRI) experiment was developed to obtain data on zirconium alloy growth rates associated with channel bowing under operating conditions relevant to BWRs. Specifically, it will provide data on the stress-free irradiation growth rate of these alloys as a function of hydrogen content and neutron fluence.

Unirradiated zirconium alloy specimens supplied by nuclear fuel vendors were uniformly pre-hydrided under the auspices of the Nuclear Fuel Industry Research (NFIR) Program, led by EPRI. Along with several reference NFIR specimens, they are being irradiated to four fluence levels in a static capsule, with no active instrumentation, under near-uniform sample temperature conditions. Figure 1 shows the experiment's capsule design concept with the top and bottom areas containing temperature buffering specimens. Four capsules each contain 50 specimens in an array of five columns by 10 rows (Figure 2).

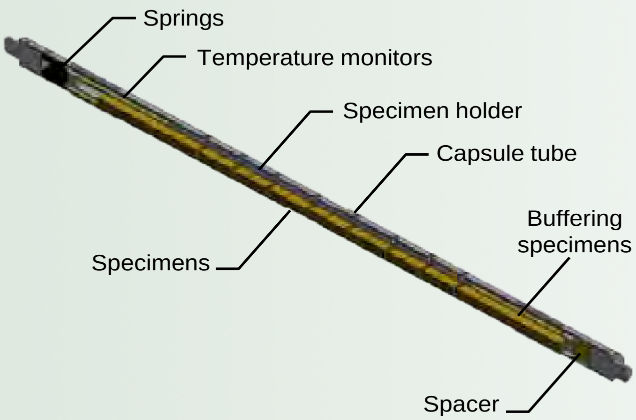

Figure 1. Test capsule assembly for irradiation.

Figure 2. Zirconium specimens used for irradiation and post-irradiation examination.

The capsules are filled with an inert gas mixture (specified to provide the correct gap conductivity) and welded inside a glovebox. Gas composition is verified online and by sampling the glovebox atmosphere. Passive silicon-carbide (SiC) temperature monitors and melt wires are installed in each sample holder for post-irradiation verification

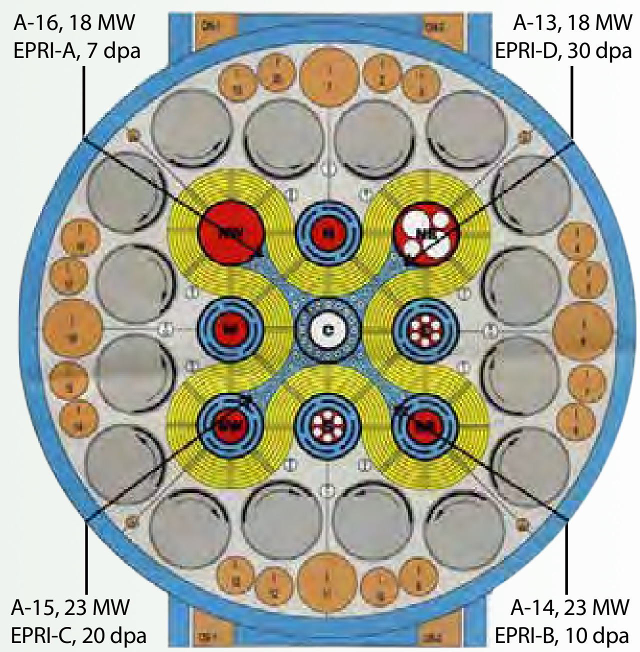

Figure 3. Cross-section of ATR showing the locations of small "A" positions where zirconium specimens will be irradiated.

"The collaborative work between ATR NSUF and EPRI on this project has developed protocols that will ensure the success of ATR NSUF industry projects for many years to come."

John Jackson, ATR NSUF Industry Program Lead

of as-run thermal analysis. Flux wires are installed in proximity to the test capsules and removed after each cycle for evaluation. Post-irradiation dimensional measurements will be used to characterize zirconium growth rates, and transmission electron microscopy (TEM) analysis will provide data on the irradiation growth mechanism. Figure 3 shows the irradiation locations within ATR.

Accomplishments

Irradiation of the test capsules began in June 2011, and continued through 2012, at which time monitoring of flux and temperature was initiated. In late 2012, the research team began designing an in-cell device to perform precision measurements of the specimens once they come out of ATR. These measurements will be compared to unirradiated specimen dimensions and will aid in subsequent calculation of irradiation-induced strain.

The removal of the first test capsule (Capsule A, 7 displacements per atom [dpa]) from ATR in January 2013, marked a major milestone for ATR NSUF, as it represented the first completed irradiation of a private industry experiment. The capsule was shipped to INL's Hot Fuel Examination Facility (HFEF) in April 2013. The final estimated fluences for the specimens are listed in Table 1. The specimens of interest (rows 1-8) had an average accumulated dpa equal to 6.995.

Position	Specimen Set	Cumulative DPA
A-16 (EPRI-A)	0 (bottom)	6.687
	1	6.871
	2	6.966
	3	7.023
	4	7.030
	5	7.063
	6	7.057
	7	7.022
	8	6.929
	9 (top)	6.680

Average (All Specimens): 6.933 DPA
Average (Specimen Sets 1-8): 6.995 DPA

Table 1. Estimated dpa levels for specimens in Capsule A.

This project directly addresses reactor safety from a fuels reliability standpoint and will provide much needed data on the boiling water reactor channel bowing issue.

The in-cell precision measurement device designed in 2012 was constructed in 2013 and installed in the HFEF hot cell, allowing remote, precision measurements of the irradiated zirconium specimens (Figure 4). The device utilizes linear variable displacement transducers (LVDTs) and an actuated stage to measure the length of the zirconium specimens with an accuracy greater than five micrometers. The device underwent extensive validation both before and after insertion into the hot cell to ensure that the measurements used to calculate strain following irradiation would be as accurate as possible. Measurements were completed on all Capsule A specimens in September 2013, as were initial strain calculations, which showed behavior consistent with expectations for these materials.

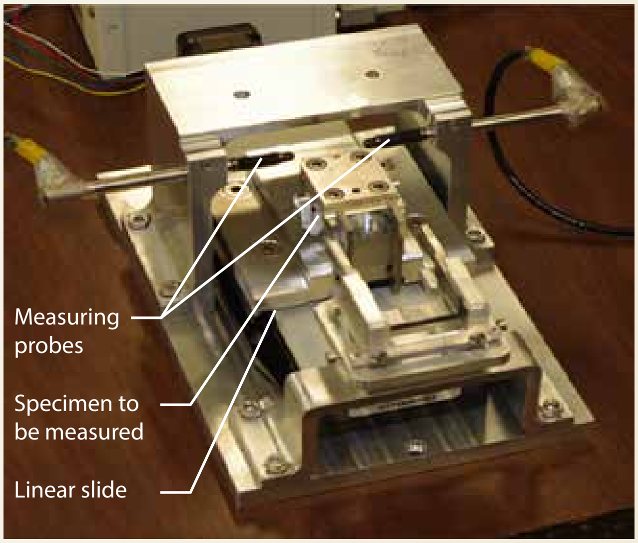

Figure 4. Remote precision measurement device designed and built by INL.

Principal Investigator: Paul Murray – Idaho National Laboratory
email: Paul.Murray@inl.gov

Irradiation and Post-Irradiation Examination to Investigate Hydrogen-Assisted Anomalous Growth in Zirconium Alloys (cont.)

Future Activities

Strain calculations are expected to be completed for Capsule A in 2014, and released in a report to EPRI and associated stakeholders. This report will also contain actual flux calculations from monitors contained in the experiment, as well as irradiation temperatures obtained by an analysis of the SiC monitors utilized. The strain data from Capsule A specimens will eventually be compared to those from the remaining three capsules using measurements that determine strain as a function of neutron fluence.

Based on a decision by the research team, the irradiation of Capsule B (10 dpa) has been extended slightly in order to increase the difference between it and Capsule A. Capsule B is expected to be discharged in January 2014, with an estimated dpa level of 12.37. Capsule C (20 dpa) and Capsule D (30 dpa) will remain in the reactor to continue accumulating fluence.

Once discharged and cooled, Capsule B will be shipped to HFEF, disassembled, and measurements will be completed. Based on the calculated strain results in comparison to Capsule A, several specimens will be selected for TEM study at INL’s Electron Microscopy Laboratory (EML) in late 2014. Microstructural analysis is expected to begin in late 2014.

<table>
<tr><th colspan="2">Distributed Partnership at a Glance</th></tr>
<tr><th>ATR NSUF & Partners</th><th>Facilities & Capabilities</th></tr>
<tr><td>Idaho National Laboratory</td><td>Advanced Test Reactor, PIE facilities</td></tr>
<tr><th colspan="2">Collaborators</th></tr>
<tr><td colspan="2">Electric Power Research Institute
Suresh Yagnik (program manager)

Idaho National Laboratory
Paul Murray (principal investigator), John Jackson (co-principal investigator), James Cole (collaborator)</td></tr>
</table>

Principal Investigator: Meimei Li – Argonne National Laboratory
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Characterization of Irradiation-Induced Defects and Precipitation in Advanced Steels

Introduction

The purpose of this project is two-fold: (1) use high-energy, wide-angle, and small-angle X-ray scattering to characterize the defects induced by neutron irradiation and radiation-induced precipitation; and (2) demonstrate that advanced steels, such as ferritic-martensitic steel, Grade 92 steel, and austenitic stainless steel NF709, are more resistant to irradiation than conventional steels.

The proposed research is directly relevant to the Department of Energy's missions in advanced reactor concepts, light water reactor sustainability, and nuclear energy-enabling technologies. The insights gained from this work will help advance the design and development of new, innovative alloys that will improve the radiation resistance of materials used to build future nuclear reactors.

The outcome of the project will also provide important information that can be used to improve the synchrotron X-ray techniques needed to characterize radiation defects.

Synchrotron X-rays can readily reveal irradiation-induced microstructural changes in complex steel alloys.

Project Description

Argonne National Laboratory (ANL) received several irradiated advanced steel specimens from the ATR NSUF Pre-irradiated Sample Library. These were archive specimens that had been irradiated to different doses and irradiation temperatures and were available for post-irradiation examination. They included ferritic-martensitic steels, a Fe-9Cr model alloy, Grade 91 and Grade 92 steels, and the austenitic stainless steels HTUPT and NF709. These are key structural and cladding materials used in advanced fast reactors, very-high-temperature reactors, and advanced light water reactors. All of the samples were examined using synchrotron X-rays in the beamline 1-ID-C at ANL's Advanced Photon Source (APS).

The radiation damage in these specimens was characterized using high-energy X-ray diffraction, small-angle X-ray scattering, and diffraction tomography. The examinations focused on studying the evolution of irradiation-induced defects, phase transformation and phase stability under irradiation.

Accomplishments

Irradiation can cause precipitate dissolution and reprecipitation, the formation of new phases, morphology changes of existing phases, amorphization, and order-disorder transformation in metallic materials.

X-ray diffraction is one of the most powerful tools for phase identification and quantitative phase analysis. As shown in Figure 1, a number of diffraction peaks of M23C6 were detected in the unirradiated Grade 91 steel, nearly all of which disappeared after irradiation to 1 displacement per atom (dpa) at ~50° C, which indicates irradiation-induced precipitate dissolution/amorphorization. Examination of the specimen irradiated under the same conditions using a transmission electron microscope (TEM) shows M23C6 became amorphous [1]. X-ray diffraction also detected several small peaks in M23C6 after irradiation to 10 dpa, while the TEM revealed no further changes in M23C6 when irradiated with this higher dose.

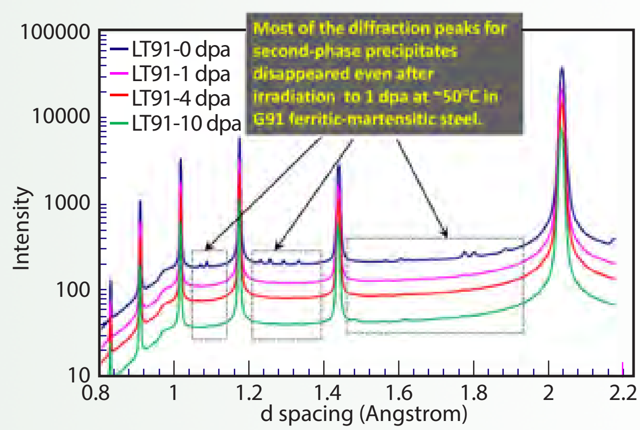

Figure 1. Diffraction peaks in irradiated G91.

"The ATR NSUF/APS partnership provides nuclear scientists with a great opportunity to study reactor materials and fuels using state-of-the art synchrotron radiation techniques."

Meimei Li, Argonne National Laboratory, Nuclear Engineering Division

The ability of synchrotron X-rays to detect minor phase changes is clearly demonstrated.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Argonne National Laboratory	Advanced Photon Source
Collaborators	
Argonne National Laboratory Meimei Li (principal investigator), Jonathan Almer (collaborator), Leyun Wang (collaborator) **University of Illinois** Yinbin Miao (graduate student)	

Future Activities

To fully understand the effects of irradiation on microstructural evolution, the specimens measured at the APS beamline will be examined further using electron microscopy and atom probe tomography.

A related proposal has been submitted to the ATR NSUF call for rapid turnaround experiments. While the proposal has not yet been selected for an award, we will continue to pursue this research.

Reference

[1] B. H. Spencer, F. A. Garner, D. S. Gelles, G. M. Bond, and S. A. Maloy, *Journal of Nuclear Materials*. 307-311 (2002) 266.

Principal Investigator: Maria A. Okuniewski – Idaho National Laboratory
email: Maria.Okuniewski@inl.gov

Grain Orientation Mapping of Irradiated Austenitic Steels

Nondestructive, three-dimensional grain and defect mapping in irradiated materials is key for understanding irradiation effects and for the development of accurate microstructural simulations of nuclear fuels and materials.

Introduction

Radiation damage in nuclear fuels and materials introduces a variety of defects ranging from point defects to bubbles and grain morphology changes. Nondestructive, three-dimensional grain and defect mapping in irradiated materials is key to understanding these irradiation-induced effects as well as for the development of accurate microstructural simulations of nuclear fuels and materials.

Project Description

The objectives of this pilot project between the ATR NSUF sample library and the Advanced Photon Source (APS) at Argonne National Laboratory (ANL) were to non-destructively examine the three-dimensional (3D) grain structures and defect morphologies in neutron-irradiated austenitic steels. Two types of experiments were conducted at APS on austenitic alloys utilizing both unirradiated and neutron-irradiated specimens. The first experiment used high-energy diffraction microscopy (HEDM) to determine changes in the 3D grain structure. The second used high-energy X-ray tomography to analyze voids, bubbles, cracks and grains.

Austenitic steels were selected because they are under consideration for core components in advanced nuclear reactors. This class of steels can withstand higher operating temperatures and has better corrosion resistance than ferritic-martensitic (FM) steels. Historically, austenitic steels were prone to void swelling following neutron irradiation, particularly in fast breeder environments. More recently; however, advanced austenitic steels have been created for both nuclear and fossil power plant applications.

The alloys of interest for this project include both high-temperature, ultrafine-precipitation-strengthened (HT-UPS) and Super 304H steels. The HT-UPS austenitic steel was developed for fast breeder reactors with carbide precipitates acting as defect sinks. However, the radiation effects on HT-UPS are not well known since the fast breeder material development program ended before these alloys could be evaluated. Super 304 H was developed to address the needs of power plants possessing super-critical (temperature and pressure) conditions. This alloy has superior creep resistance when compared to that of the 12% chromium (Cr) martensitic steels.

Two other alloys of interest are the historical austenitic alloy D9, which has previously been used for cladding applications, and NF709, a high-temperature, creep-resistant austenitic alloy, which was also created for fossil-fuel plant applications. This project will not only help researchers understand the irradiation behavior of these austenitic steels, but will also provide input and validation data in three dimensions for nuclear fuel performance models that are currently under development at INL.

Accomplishments

Nondestructive, 3D characterization was carried out on unirradiated and neutron-irradiated austenitic steels using APS at ANL (Figure 1). HEDM was used to determine changes in 3D grain structures, and high energy X-ray tomography was used to analyze voids, cracks and grains. The near-field configuration was used for the HEDM to conduct grain mapping. By utilizing this technique, the diffraction spots imaged on the detector were those of the grain cross-sections (Figure 2). High-energy X-ray tomography was also carried out to visualize voids, crack and grains in three dimensions (Figure 3).

Both of these techniques will help in understanding the effects of irradiation dose and temperature on grain morphology in four austenitic steels. Information regarding the grain boundary misorientation is important when considering irradiation effects since the degree of misorientation, or tilt angle, has been found to relate to the ability of the grain boundary to absorb point defects. Moreover, both of these techniques provide the capability to examine these specimens in a 3D manner, as opposed to most previous experiments which only had two-dimensional (2D) capabilities (e.g., electron microscopy).

Figure 1. Specimen and detector configuration for HEDM measurements.

"This pilot project for the APS and ATR NSUF provided critical access to neutron-irradiated specimens and brought together a number of experts in various fields across the DOE complex and from academia to help develop and apply new 3D, nondestructive techniques to understand irradiated material characteristics."

Maria Okuniewski, Principal Investigator, Idaho National Laboratory

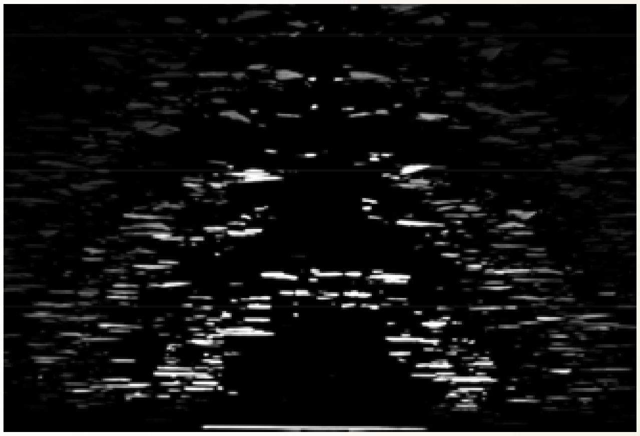

Figure 2. Example of typical HEDM results, which indicate the cross-sectional grain sizes via the diffraction spots.

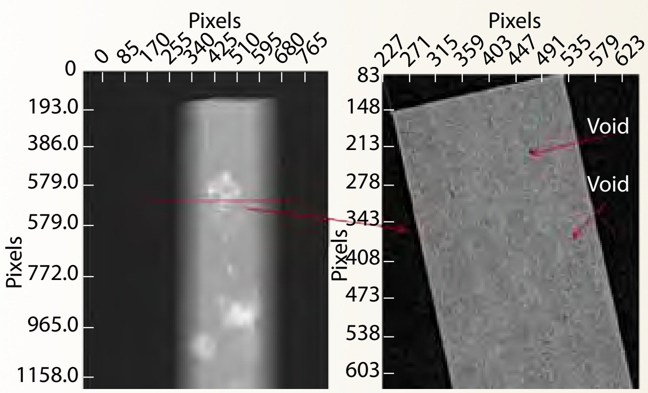

Figure 3. High energy X-ray tomography of an austenitic steel. Note that each pixel is 0.75 µm. The image on the left shows extinction regions of grains (light color). The image on the right shows a 2D slice taken from the left tomograph (denoted by the red line), which indicates the presence of voids.

Figure 4. Research team at the APS beamline (left to right) – Maria Okuniewski, James Hunter, Stephen Niezgoda and Peter Kenesei.

Acknowledgements

This project was made possible through the use of the ATR NSUF sample library, using specimens that were part of the University of Wisconsin pilot project. Kumar Sridharan and Guoping Cao are gratefully acknowledged for providing these specimens as well as the control specimens.

Future Activities

The goals for this research in 2014 are:

- Continue the data analysis, including code development, for the HEDM and high energy X-ray tomography results to produce quantitative data.
- Correlate the 3D data from the two techniques utilized.
- Provide data to the modeling and simulation efforts for nuclear fuel performance models.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Argonne National Laboratory	Advanced Photon Source
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	PIE facilities
Collaborators	
Argonne National Laboratory Peter Kenesei (co-investigator), Jonathan Almer (collaborator)	
Idaho National Laboratory Maria Okuniewski (principal investigator), Brandon Miller (collaborator), Emmanuel Perez (collaborator), Jakeob Maupin (collaborator), Jameson Root (collaborator, currently at Purdue University)	
Los Alamos National Laboratory James Hunter (co-investigator), Stephen Niezgoda (co-investigator, currently at Ohio State University), Don Brown (collaborator)	

Principal Investigator: Wade Marcum – Oregon State University
email: wade.marcum@oregonstate.edu

Development of a Validation Method for Fluid Structure Interactions

Introduction

Access to sophisticated component design and safety analysis tools traditionally has been limited to only those researchers who have supercomputers or clusters at their disposal. However, continuing advancements in computational methods and tools have made these resources available through common personal desktop workstations.

As a result, an expanding community of users is able to leverage advanced multiphysics software packages (for example, computational tools that can produce results relating to both fluid and solid domains or to fluid-structure interactions [FSI]) to produce reliable and accurate results. Although the advancements in these software tools, in particular multiphysics packages, have leaped ahead in recent years, the efforts to perform corresponding rigorous validation and verification (V&V) of multiphysics packages have fallen short. This has been due to limited V&V resources and direction, problematic complexities and a lack of available experimental data.

Project Description

Currently, no vetted standard reference material exists with which to V&V a computational multiphysics program. This can result in plausible inconsistencies in a variety of computational tools. One possible solution is to use the standards for tools that have already been developed for their respective domains (e.g., fluids, solids, etc.) to create a comprehensive V&V process.

This process would begin with unit cell (or problem) tests within a given area of physics (e.g., thermal mechanics), continue into benchmark tests and then morph into integrated tests that incorporate additional physical characteristics (e.g., thermal mechanics *and* structural mechanics; thermal mechanics *and* fluid mechanics, etc.). When this suite of tests is complete, the simulation tool will have been vetted.

The objective of this Faculty/Student Research Team (FSRT) study is to synthesize this hypothesis.

The outcome(s) of this project will enable the use of new analysis methods that will enhance nuclear reactor safety.

Accomplishments

A number of goals were accomplished during the summer of 2013 that align with the ongoing effort to develop an FSI V&V methodology. These include:

- A fundamental first principles-based analysis of the physics that drive FSI between fluids and solids.
- A literature review that quantifies the state-of-the-art computational methods currently used to simulate the physics that are actually or potentially available through the Fuels Development (FD) program.
- A qualitative assessment of the readily available and widely used commercial codes needed to synthesize this computational effort.

This qualitative assessment led to the establishment of a computational benchmark for several existing vibrational problems, as well as a novel alternative method for conducting numerical analyses.

Future Activities

This experiment, and other closely related activities, is expected to contribute to the creation of a generalized method for validating a computational tool or a suite of coupled computational tools to characterize FSI phenomena. This method is anticipated to be used at INL to validate multiphysics computational analyses in the FD program of the Global Threat Reduction Initiative (GTRI). It will also be used by ATR NSUF to supplement the experiment design process prior to qualification testing and irradiation.

Publications

Trevor K. Howard, Wade R. Marcum, Warren F. Jones, "Characterizing Virtual Mass Effects of a Submersed Body using Pseudo-Fluid Elements," *Proceedings of the American Nuclear Society Advances in Thermal Hydraulics, Reno, Nevada, June 15-19, 2014,* pp 1-10.

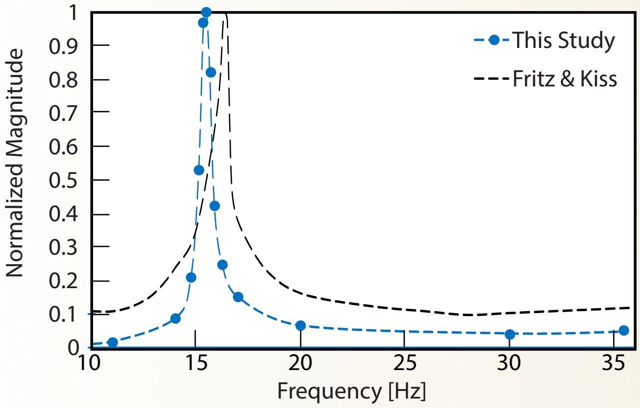

Figure 1. Computed natural frequency of cylinder compared against base study.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Multiphysics Software Tools
Collaborators	
Idaho National University Warren Jones (INL principal investigator) **Oregon State University** Wade Marcum (principal investigator), Trevor Howard (collaborator)	

Principal Investigator: Wade Marcum – Oregon State University
email: wade.marcum@oregonstate.edu

Development of Heat Transfer Module(s) within the MOOSE/BISON Platform for Application in the Transient Reactor Test Facility

Introduction

Safety must be designed into every facet of the nuclear power industry. While nuclear fuel is designed to be safe under standard operating and accident conditions, the behavior of fuel under extreme conditions must be understood to ensure the safe use of nuclear fuels. For example, one scenario in which fuel might have to be especially stable and safe is a reactivity insertion accident (RIA), in which excess reactivity is inserted into a nuclear reactor, causing the reactor's power to grow exponentially.

In the past, RIAs have been studied by exposing a test fuel element to a controlled transient inside a test facility. The Transient Reactor Test (TREAT) facility at INL is one resource for performing these tests. It has been out of operation for several years, but researchers are pursuing an effort to restart the TREAT facility for this experiment in order to perform new fuel testing under transient conditions. INL's Multiphysics Object-Oriented Simulation Environment, or MOOSE, is a computer-simulation framework that advances the process for predicting the behavior of complex systems. MOOSE makes it easier to create simulations for complex mathematical models such as BISON, which enables the study of fuel behavior at scales ranging from a grain to a full pin.

Project Description

Researchers can choose between two different methods for simulating the transients in the TREAT facility: using a homogeneous fuel or using a heterogeneous fuel. The complex physics of nuclear transients requires an engineer's experienced judgment to select the correct method to simulate the transient.

The TREAT fuel is made by mixing uranium dioxide (UO_2) with a graphic moderator until the fuel-to-moderator ratio reaches the desired value. The heterogeneous fuel accurately represents the TREAT fuel by modeling the fuel particles inside the graphic moderator. The nuclear power and heat are uniformly generated inside the particle, and heat conduction moves the heat energy from the fuel particle into the moderator.

Homogeneous fuel does not model the fuel particles, just the moderator. The power and heat are uniformly generated throughout the moderator, and heat conduction then, once again, is allowed to move the heat energy.

Both the heterogeneous and homogeneous fuels have advantages and disadvantages for simulating the performance of nuclear fuel during a transient power curve. The heterogeneous fuel accurately models a fuel's real geometry as well as the physics of the delayed heat movement to the moderator that is caused by conduction through the heat particle.

However, the fuel particle temperature in the heterogeneous fuel rises faster than the increase in moderator temperature. The heterogeneous fuel model also requires more computational resources due to the mesh refinement necessary for including small fuel particles inside a much larger fuel block. This requirement might also limit the researcher's ability to include more components in a single mesh because of scales of differing lengths.

The homogeneous fuel, on the other hand, decreases the need for computational resources by eliminating small-sized fuel particles and using uniform power generation in the moderator. The increase in the moderator temperature experiences no time lag since the heat energy no longer needs to diffuse out from the fuel particle, as is the case with heterogeneous fuel. The one disadvantage to the homogeneous fuel is it does not represent the physical TREAT fuel precisely, but only approximates it. The question then becomes, "Which simulated fuel model should be used to optimize the use of resources and the fidelity of the results?"

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Multiphysics Object-Oriented Simulation Environment
Collaborators	
Idaho National Laboratory Dan Wachs (INL principal investigator) **Oregon State University** Wade Marcum (principal investigator), Adam Zabriskie (collaborator)	

"Leveraging existing knowledge from the MOOSE/BISON computational tools along with archived documentation from the TREAT facility has facilitated this research project. The project would not have been possible without these interactions with INL through the ATR NSUF Faculty Student Research Team."

Wade Marcum, Assistant Professor of Nuclear Engineering & Radiation Health Physics, Oregon State University

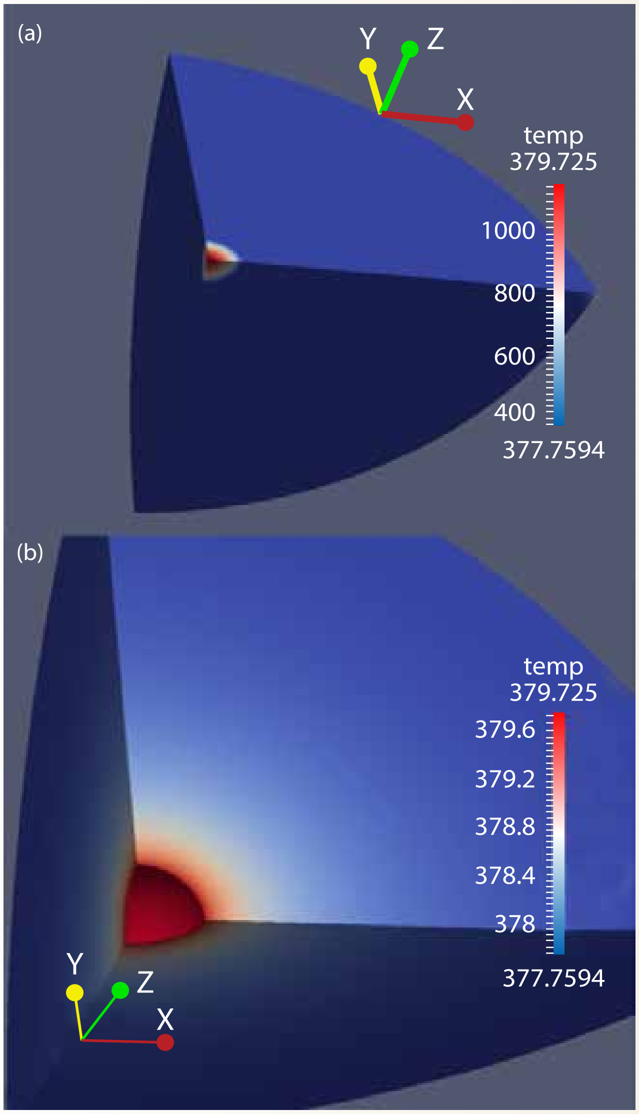

Figure 1. (a) 100 micron fuel particle and (b) graphite moderator temperature [K] during a power transient at 0.55 seconds.

This project demonstrates a new and unique understanding of a fuel's kinetic response during a reactivity insertion accident.

Accomplishments

A baseline MOOSE/BISON model was developed and run in the TREAT facility. The results demonstrated an agreement between the baseline and an analytical solution within the numerical uncertainty of the code. The model was then extrapolated to identify the overall impact of fuel particle size on the temperature lag within the fuel matrix. The resulting data demonstrated that the heterogeneous modeling of fuel (if the particle size is sufficiently large) produces the most accurate representation of reactor kinetics.

Future Activities

Work continues to identify the influence of nonuniformly mixed fuel particles as well as the effects of nonspherical particles within the fuel matrix. The preliminary results of these efforts show promise for developing a new understanding of a fuel's prompt reactor kinetic response during an RIA.

Publications and Presentations

Adam Zabriskie, Wade Marcum, Dan Wachs. "Modeling Transient Power Time-Lag in Heterogeneity with the TREAT Fuel Matrix Using MOOSE/BISON," *Proceedings of the American Nuclear Society Advances in Thermal Hydraulics, Reno, Nevada, June 15-19, 2014*, pp 1-10.