ATR
National Scientific User Facility

2012 Annual Report

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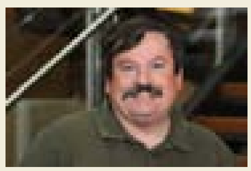

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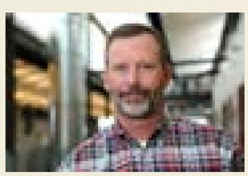

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Advanced Test Reactor National Scientific User Facility

2012 Annual Report

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For the most up-to-date information, visit the ATR NSUF website at http://atrnsuf.inl.gov
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ATR NSUF Industry Program Projects

Acronym List

AECL	Atomic Energy of Canada Limited
AES	auger electron spectroscopy
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
CVD	chemical vapor deposited
DNB	departure from nucleate boiling
DOE.	Department of Energy
dpa.	displacements per atom
EBSD	electron backscatter diffraction
ED	electro-deposition
EDS	energy dispersive X-ray spectroscopy
EFRC	Energy Frontier Research Center
EFTEM	energy-filtered transmission electron microscopy
EFPD.	effective full power days
EML	Electron Microscopy Laboratory
EPRI	Electric Power Research Institute
EUVR	extreme ultraviolet reflectometry
EXAFS	X-ray absorption fine structure
FCC	face-centered cubic
FEG	field-emission gun
FFTF	Fast Flux Test Facility
FIB	focused ion beam

FIMA........................ .... fission per initial metal atom
FSRT ....Faculty/Student Research Team
HFEF..... ....Hot Fuel Examination Facility
HFIR... High Flux Isotope Reactor
HRTEM... high resolution transmission electron microscopy
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
ISU.. .... Idaho State University
ITER.. International Thermonuclear Experimental Reactor
LAMDA..Low Activation Materials Development and Analysis
LANL...Los Alamos National Laboratory
LBE... ....lead-bismuth eutectic
LBP.. .... late blooming phases
LEAP ....local electron atom probe
LBP .... late blooming phases
LM .... liquid metal
LOCA.. .... loss-of-coolant accident
LWR... ....light water reactor
MaCS .... Microscopy and Characterization Suite
MANTRAMeasurement of Actinide Neutronic Transmutation Rates with Accelerator Mass Spectrometry
MFC... .... Materials and Fuels Complex
MIBL. .... Michigan Ion Beam Laboratory
MIT..... .... Massachusetts Institute of Technology
MITR.. .... Massachusetts Institute of Technology Reactor
MOX.. ....mixed oxide
MPC.......... .... multi-purpose coupons
MRCAT......... .... Materials Research Collaborative Access Team

Acronym List

MSTL	Materials Science and Technology Laboratory
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
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
REDC	Radiochemical Engineering Development Center
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
SEM	scanning electron microscopy
SHaRE	Shared Research Equipment Collaborative Research Center

SIMS .... secondary ion mass spectroscopy
SPT .... shear punch test
SRNL........ Savannah River National Laboratory
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
TTS .... transition temperature shift
UCB .... University of California, Berkeley
UCSB .... University of California, Santa Barbara
UF .... University of Florida
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
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

Welcome & Introduction

Dr. Todd Allen
ATR NSUF Scientific Director
and
Professor of
Engineering Physics,
University of Wisconsin

It has always been a pleasure to introduce the Advanced Test Reactor National Scientific User Facility (ATR NSUF) Annual Report, and none more than this year. As most of you know, I resigned from my position as Scientific Director of ATR NSUF in December of 2012 to become the Deputy Laboratory Director of Science and Technology for the Idaho National Laboratory (INL). In my new position, I will provide oversight to ATR NSUF activities and look forward to watching what started as a vision for collaborative research grow into a strong, vibrant network of users and facilities.

To recap a very exciting year, we'll start in October of 2011 when ATR NSUF aligned its fall call with the Department of Energy (DOE) Nuclear Energy University Programs (NEUP) call to allow university researchers to submit a single proposal for both NEUP funding and ATR NSUF capability. Twelve pre-applications for this "joint" call were submitted and out of those, three full proposals were requested. After the NEUP ranking, two proposals were sent to the ATR NSUF panel committee to be ranked along with proposals submitted only to the ATR NSUF call. After our final ranking, one proposal was awarded from the joint call. We were excited by the interest in this new process, and hope to see more researchers using this avenue for submitting proposals in coming years.

In January, ATR NSUF announced two new partner facilities and a new capability from an existing partner. We welcomed both Pacific Northwest National Laboratory (the Radiochemistry and Materials Science and Technology Laboratories) and Purdue University (IMPACT facility) as new partners and added the LAMDA facility at our existing partner, Oak Ridge National Laboratory. The addition of the two new partners brought the total number of partners to 10.

And speaking of partners and their impact, in 2012 we awarded 22 new research projects, many of them rapid turnaround experiments (RTE). 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 March of 2012, University of Florida master's student Don Moore and I traveled to Washington, DC to represent ATR NSUF at the National User Facility Organization (NUFO) Exhibit for Congress. This was the second year that NUFO was invited by Congress to DC to exhibit the capabilities offered by DOE and other federal user facilities around the country. We look forward to this event continuing since it is an effective way for Congress to learn about user facilities in general and about the amazing science being done every day at these facilities. It is worth noting here that the combined user facilities that make up NUFO had 30,000 users worldwide at the end of 2012. I would also like to encourage any of who you have not yet joined, to join the ATR NSUF Users Organization. Your membership and participation is a vital part of creating a thriving user facility. Through the Users Organization, we learn how to improve our processes and where to focus capability development.

In June, we hosted the ATR NSUF fifth annual Users Week. Different from past years, the 2012 Users Week was a joint event co-hosted with the MeV school held at Oak Ridge National Laboratory, and lasted two full weeks. Participants in this event included 31 students, 11 faculty members, and 8 representatives from industry and/or other national laboratories. This was a much smaller group of participants than in the past, but by taking this approach we were able to give participants an enhanced learning environment centered on two interrelated nuclear themes; materials and fuels. It also allowed us to offer more interactive tours of INL's unique nuclear facilities along with hands-on activities.

Also notable in 2012 were the advanced degrees received by students involved in ATR NSUF research. Walid Mohamed of North Carolina State University, and Alicia Certain and Kevin Field from the University of Wisconsin received their Ph.D.s. All three of them have found employment in the DOE laboratory system and we look forward to working with them in the future.
In addition, Don Moore from the University of Florida completed his master's degree and has gone to work for private industry. Let me offer my congratulations to each of you and wish you all the best in your future endeavors.

Another student highlight worth mentioning is the outstanding work performed by Monty Anderson of Brigham Young University-Idaho. Monty joined ATR NSUF as an intern and was given the responsibility of cataloguing every sample specimen to date that either has been or will be irradiated in the ATR as part of an ATR NSUF experiment. This catalog of samples, once the corresponding experiments have been completed, will become part of the ATR NSUF sample library. Through his efforts, we have a single place where the sample specimens can be tracked and sorted with ease. In fact, we were so pleased with his work that we sent him to Los Alamos National Laboratory during the past summer to catalog the ATR NSUF library specimens stored there.

Participants of the joint 2012 ATR NSUF Users Week and MeV Summer School.

The last item I wanted to highlight was the addition of two new staff members to ATR NSUF in 2012. Please join me in welcoming Dr. James Cole and Ms. Julie Ulrich. Jim is the ATR NSUF Chief Scientist and has spent the last 15 years at INL studying the effects of radiation on materials and nuclear fuels in current and advanced nuclear energy systems. Jim joined ATR NSUF because he wanted to use his

Welcome & Introduction (cont.)

Dr. Jim Cole
Chief Scientist

knowledge to help younger generation researchers design innovative experiments to achieve their research goals. He explained, “The resources at the laboratory are very unique and should be made available to external users who have great ideas but don’t have access to the needed facilities at their home institutions.”

Some of the goals Jim has in his role as Chief Scientist include expanding user facility capabilities, and streamlining how post-irradiation examination (PIE) is conducted to provide access to a greater number of users. On a grander scale, he would like to assist INL in developing state-of-the-art methods for conducting nanoscale characterization of highly irradiated materials on a routine basis. According to Jim, “This type of capability is lacking in the U.S. nuclear research infrastructure and will be critical to supporting the modeling and simulation efforts related to nuclear fuel performance and materials degradation in reactor environments.”

Jim also told me about some of the challenges he sees for ATR NSUF. One challenge is figuring out how to effectively prioritize a significant number of diverse research efforts. The overall goal is to maximize the impact of limited resources in a challenging budget environment. According to Jim “We want to ensure researchers are able to complete as much research as possible given the time, effort and cost required to conduct a high-quality irradiation experiment and associated post-irradiation examination.” Another challenge Jim is addressing is how to ensure effective communication between university researchers and the ATR experiment design and engineering staff.

He has learned that setting reasonable expectations early on in the experiment planning process is important. Jim explains, “It is critical to keep the lines of communication clear between the engineering staff who carry out the experiments at INL, and the university researchers.

There is a lack of knowledge on the part of the university researchers as to the level of complexity and detail required to design an irradiation experiment.” He continues, “The engineering staff understands not only how to design the experiment but also how to evaluate the safety case so the experiment can be inserted into the reactor. They also understand what it takes to conduct the PIE. The researchers have the insight and appreciation of the fundamental science behind the experiment. Bringing the two together can be a challenge.” I am pleased that Jim is working toward accomplishment of these goals. They are the kinds of issues that need addressing and I look forward to seeing great strides in our research processes in the future.

Julie Ulrich
Communications Liaison

Julie Ulrich has an MBA with a marketing emphasis and joined ATR NSUF part time as the Communications Liaison in May of 2010 to help ATR NSUF researchers and technical staff communicate with a wide variety of audiences. In 2012, Julie started working for ATR NSUF full time. Julie’s main goal as communications liaison is to make ATR NSUF more visible both inside INL and externally to researchers throughout the country and around the world. In line with that, she is working on a redesign of the ATR NSUF website, improving functionality and creating a more user-friendly format.

According to Julie, “The research we do here at ATR NSUF is exciting and the website gives me an excellent vehicle to help share that with the rest of the world.” One of her main objectives with the new website is to make both the current and past research project information more accessible to researchers. She explained, “If researchers can easily find information that helps them better understand the kinds of research opportunities we offer, it will help them be more successful in writing their proposals.”

Working for ATR NSUF as the communications liaison also provides some interesting challenges. According to Julie, “The partner facility concept in and of itself creates some unique communication opportunities. Not only does having partners make us different than other user facilities, but because new partners are always being added, ATR NSUF is constantly evolving.” In her view, the partner concept makes ATR NSUF more complicated to define and describe, but also provides her with a fun, ongoing challenge. Another item on Julie’s mind these days is how a new scientific director will envision the user facility and what kinds of changes that brings to her communication efforts. However, Julie also told me that in her mind change brings opportunity. “A new director will bring a fresh perspective, and that could drive the program toward even more success.”

Julie also told me she is enjoying working for ATR NSUF. “With a mix of designing the website, creating brochures, writing articles, and interfacing with media and researchers, there is always something new to do. Even better, the research has a real impact on the world and I get to be a part of that.” She said one of the reasons she decided to come to ATR NSUF was the dedication of the staff. Julie explains, “Everyone working for ATR NSUF truly cares about the success of the program, which makes communicating the user facility story that much easier because everyone is eager to share their contributions.” She also enjoys communicating the significance of the research being performed. “Through ATR NSUF I have the opportunity to talk with professors who are excited about the research they get to perform. I also get to meet students who are working with INL scientists and engineers on an ATR NSUF project as part of their advanced degrees. The enthusiasm of these researchers and their students is contagious.” I agree and look forward to the new website and to seeing continued improvements in our outreach activities.

In closing, it has been my pleasure serving as the Scientific Director for ATR NSUF. As the first Scientific Director, I had the privilege of molding the user facility into a network of partners and researchers, offering multi-faceted collaborations that are helping energize nuclear research in the U.S. I realize that with the help of the ATR NSUF staff, the trust and willingness of our current and future partners, and the creativity of the researchers who submit proposals, ATR NSUF has and will continue to become a user facility DOE-NE and INL can be proud to claim as their own. It’s a nice legacy to leave whoever steps in as the new scientific director and one I’m proud to pass forward.

Research without Walls

Researchers learn about ATR NSUF capabilities in a hands-on workshop in the Microscopy and Characterization Suite in CAES during Users Week.

Nuclear power provides some 20 percent of the world's energy needs today, and, as those needs grow, the demand for nuclear energy will increase right along with them. While the search for new and better nuclear fuels continues to be an ongoing issue, arguably the driving force in today's nuclear industry is finding ways to make the next generation of nuclear reactors longer-lived. Accomplishing this admittedly tall order will require the best and brightest minds across a broad range of scientific fields. But perhaps equally important, it will require those minds to come together and share the knowledge they have gained from the myriad of research being done on this complex, and often baffling, subject.

Enter Dr. Todd Allen, ATR NSUF's outgoing scientific director. "ATR became a national scientific user facility in 2007," says Allen. "When I started as the scientific director in 2008, we quickly recognized that the reactor had a lot of volume compared to the size of a typical scientific sample being analyzed. In other words, we were in a position to irradiate a lot of additional specimens. But in order to get the scientific answers we need, those specimens must then be put into whatever instrument is appropriate, depending on what you're measuring. We soon realized that, compared to the number of samples we might need to examine at any given time, the number and types of instruments at Idaho National Laboratory (INL) alone will probably never keep up with the needs of the users. So we said, why don't we see if there are other facilities across the country that are in a position to help?"

The U.S. Department of Energy (DOE) was impressed with the idea, so Allen and his staff came up with a set of parameters for potential partners, and put together a committee to review proposal applications. The process was simple by design. Those interested nominate themselves, then the committee reviews their proposals on the basis of whether the capabilities are useful, and whether they are capabilities that the ATR NSUF doesn't have, or that they may have but are likely to be oversubscribed. If the answer to those questions is yes, they are accepted as a partner.

"A university facility, for example, might be limited to a lower level of radioactivity than what we work with at INL," Allen explains, "but that's okay. They could still take some of the projects that are radiologically easier, if you will, off our plate. That way, ATR NSUF could be more responsive to all the users we're trying to serve. The final piece was making connections with the other national laboratories. To make this cooperative venture work, we had to know how much radiological material they could tolerate, and whether they were willing to allow samples that we irradiate in ATR to be looked at in their facilities."

Those questions were answered when Oak Ridge National Laboratory (ORNL) and Pacific Northwest National Laboratory (PNNL) joined the growing list of facilities that were coming on board as partners.

“Bottom line,” Allen says, “we want to link the national capabilities in a way that if somebody has an idea, but they don’t have access to the specialized equipment they need to determine if their idea is really a good one, then we should try and make that connection for them.”

As each partner joined ATR NSUF, word was beginning to spread, but before this burgeoning network of nuclear partners could really be put to use, Allen and his staff decided it would be a good idea to run a test project to help them figure out how to work with somebody from outside INL who does not have a detailed knowledge of nuclear-based testing. For that, Allen turned to an old colleague, Dr. Kumar Sridharan, a research professor at the University of Wisconsin (UW).

Drs. Kumar Sridharan and Heather MacLean Chichester look on as UW student Tyler Gerczak works on the scanning electronc microscope.

“This whole thing was very unusual,” says Sridharan, “for a university to be coming to a national lab and getting their research done. This model is not very common at all, and almost unheard of for irradiation work.” Sridharan agreed to lead the test project. He was immediately connected to INL principal investigator Heather MacLean Chichester because of their similar backgrounds in materials science. They prepared some 500 individual samples of 20 to 25 different structural materials and put them into ATR. The samples came not only from the UW, but also through connections at the University of Michigan (UM) and Los Alamos National Laboratory (LANL), among others. Once the irradiations were completed, a number of students performed post-irradiation analysis on some specimens. However, a large number of specimens were put into the ATR sample library for other researchers to use.

“The biggest part of my connection with that project now is to make sure the samples are getting analyzed,” Sridharan says. “We spent a lot of time and effort in the irradiation of these samples and we would like them to be used. Anybody who is interested in examining a certain material that’s on the list can write a proposal to ATR NSUF, access the samples, and work on them.”

The fact that Sridharan’s students have been able to research some of the samples using transmission electron microscopy (TEM) at the University of Nevada, Las Vegas, small angle neutron scattering (SANS) at the Los Alamos Neutron Science Center (LANSCE), and the local electrode atom probe (LEAP) as well as TEM at the Center for Advanced Energy Studies (CAES), is testament to the exponential growth of the partnership network.

One of these students, Alicia Certain, was involved in the pilot project almost from the beginning, and the work became a significant part of her Ph.D. thesis. In addition to writing scientific reports and papers on the project, she traveled extensively to national labs and universities performing independent research on some of the samples, most notably building a collaboration with the National Institute of Standards and Technology (NIST) Center for Neutron Research. Her work on the test project helped land her a position at PNNL after graduation.

“Her work using LEAP techniques to quantify micro-segregation due to radiation has both paved the way and set standards for future students,” says Sridharan. “She also performed some ground-breaking work on modeling the stability of oxide nano-particles in steels, thereby providing foundational work for the design of the next generation of nano-structured steels.”

But the pilot project’s connections didn’t stop there. Two students who were instrumental in the irradiation phase of the project were Yong Yang, now an assistant professor at the University of Florida (UF), and Lizhen Tan, who took some samples from the project to study when he went to work for ORNL. Tan was also one of the scientists originally selected to perform technical reviews of proposals for the fledgling ATR NSUF program. This initial knowledge spurred him to submit a proposal of his own in the spring of 2012, using the UM Ion Beam Laboratory. That project was awarded, and is underway.

Research without Walls (cont.)

Dr. Yong Yang prepares samples for analysis as part of the University of Wisconsin pilot project in the Microscopy and Characterization Suite in CAES.

"ATR NSUF provided unique capabilities that fit my project task," Tan says. "During the course of the project I've built some good collaborations through colleague recommendations and attendance at conferences, and we have since partnered with Dr. Meimei Li at Argonne National Laboratory (ANL) and Yong Yang at UF, to submit another proposal to ATR NSUF which will utilize some irradiated specimens from their sample library, along with a variety of ATR NSUF characterization instruments."

The idea of a distributed network of partners, nurtured by Todd Allen and his staff and validated by Kumar Sridharan's pilot project, has flourished in the five years since it was first created. Its web of connections now spreads across the country.

"I think it's a real success," Sridharan says. "Most people don't realize that these experiments are really difficult to do. When we talk about working with neutron irradiation, it's always time consuming, but it has come a long way. Now you can offer proposals just to do post-irradiation analysis, and they are giving out these awards three or four times a year. Numerous universities and national labs are using this on a regular basis."

One scientist who has really taken advantage of the program is Dr. Michele Manuel, an assistant professor at UF, who has submitted at least half-a-dozen proposals, the first in 2011. A few years earlier, she had worked with Todd Allen at the Energy Frontier Research Center (EFRC), a DOE Center based at INL. She was interested in studying the atomic structure of nuclear fuels, and when she learned that CAES was going to get a LEAP she simply bided her time for a couple of years until it was up and running, then submitted a proposal.

"It's a nice process," says Manuel. "They give you a pretty rapid response. They're very easy to work with and it's easy to get samples there. I actually don't go and use the instruments myself, I send my students. I've never had any problems with the students being there, and they always get the results they need. It's been beyond my expectations of a user facility."

Even though she doesn't usually travel to ATR NSUF facilities in Idaho, the results of her projects are broadening her professional connections in the field.

"ATR NSUF is allowing us to study materials that we usually can't study in LEAP instruments," Manuel says. "Most facilities won't allow nuclear fuels in their instruments because of the risk of contamination, but the fact that we can utilize these samples through ATR NSUF has allowed us to be the first to look at certain types of materials in this way. That has garnered interest from potential collaborators like Brent Heuser at the University of Illinois, and Andy Nelson at LANL, who want to do the same thing with their materials. We're starting to write our first publications based on the data we've been generating over the past two years, and that will really help get the word out about the ATR NSUF program."

Manuel, like many of her colleagues, has also been attending conferences like those sponsored by The Minerals, Metals and Materials Society (TMS) and the American Nuclear Society (ANS). And she has since submitted a number of additional proposals to ATR NSUF that will allow her to continue using more of the instruments located in Idaho.

"A lot of the national labs have this reputation that you kind of have to be inside the system to get access," Manuel says. "But I think ATR NSUF really tries to go out and specifically ask junior people to attend some of the workshops, like Users Week in the summer. It's more like they're welcoming people who are new. They really try to help you understand how everything is going to work, and constantly ask you questions, like how are you going to do this, or when are you going to that? So I've kind of learned the process now. That's very difficult for a junior person to do anyplace else."

Users Week is ATR NSUF's major educational event. Held each summer in Idaho, it offers workshops on a wide variety of topics, such as fuels and materials issues in nuclear systems, what to think about when designing a reactor experiment, and various ways to analyze samples when they come out of a reactor.

"We recognized early on that there wasn't a lot of recent experience in reactor-based testing," Allen says. "So with Users Week we're doing a couple of things. We're introducing people to the idea of the user facility, the capability that can be accessed, and how to go about that. Now, with the partnership program, we invite the partners in to be part of those presentations. Imagine showing up as a student and getting a presentation by a scientist from the MIT reactor talking about the capabilities they have to offer and some of the experiments they've done as part of the user facility program. Imagine the connections being made."

Another recent player in the program is Texas A&M University (TAMU). In 2009, just as the ATR NSUF partnership program was beginning to evolve, Lin Shao was one of the first users from the state of Texas to travel to Idaho and participate in Users Week.

"I was quite impressed on my first visit," says Shao. "We got a lot of information about the upcoming calls for proposals and the different programs there. ATR NSUF did a very meaningful thing by offering the university community access to nuclear-related research facilities. Our whole department appreciates this effort."

A short time later, Shao submitted a rapid turnaround experiment (RTE) proposal that was subsequently awarded. Based on the success of that experiment, he proposed a three-year project on simulated nuclear fuel and fuel cladding interactions.

"On our first experiment, we needed a very quick verification of the hypothesis," explains Shao, "but this current proposal is a long-term effort. We need three years of cooperation with ATR NSUF, and at least two more years of work after that."

While Shao's second RTE is ongoing, connections are being made through the data published on the first project. TerraPower, a company established by Bill Gates, took notice of Shao's work and is looking into the possibility of future collaborations. Two INL scientists, Bulent Sencer and Rory Kennedy, have already contracted with Shao and his team for other work, and one of the students Shao assigned to the initial project, Assel Aitkaliyeva, who worked at ATR NSUF right up the project's completion, was hired by INL after her graduation.

"The program is very important for students," Shao emphasizes. "I have about 20 students here at TAMU, and any time I mention that we may send somebody to work on a project at ATR NSUF they all get very excited and everyone volunteers, because they see a way to extend their work and learn what's going on outside the university. Dr. Allen and I have even talked about incorporating my lab as a partner facility as well."

Word of Shao's work with ATR NSUF also found its way across campus to the electrical engineering department, where Dr. Haiyan Wang was studying the ability of thin-film ceramic coatings on structural materials in cladding tubes to prevent diffusion and corrosion.

"I'd had this idea for a long time," says Wang, "and after two or three years of development we had gotten to the point where we needed to do some real corrosion tests. We asked around, but nobody on campus was doing that. Then Dr. Shao suggested we look into the ATR NSUF program."

Texas A&M Ph.D. student Assel Aitkaliyeva works on the Lin Shao RTE experiment using the focused ion beam in the Microscopy and Characterization Suite in CAES.

Research without Walls (cont.)

Once Dr. Wang's proposal was awarded by ATR NSUF, it was determined that the tests she needed done could best be performed by one of their partner facilities, the UM Ion Beam Laboratory.

"I was really honored to be able to work with Dr. Gary Was at the UM," says Wang. "He is very well known in the field. And it turned out the results were surprisingly good. We were all very excited. Based on that collaboration we got to know several of the scientists there, and we wrote another proposal together for a similar corrosion test, but under slightly different conditions. We also want to do some ion irradiation experiments and see how the coating performs in those severe environments. We're working on a paper right now for the first project, and we hope to publish soon."

Since UM joined the partnership program, a number of other researchers have also been connected to their Ion Beam Laboratory through ATR NSUF.

"It's a good example of a partnership right now," says Allen. "It's part of the program's national character. Scientists who want to use the UM facility send a proposal to us, we review it, and if it's approved, the samples are sent to Michigan. They do the irradiation, and then we reimburse UM for the time on their beam."

The ATR NSUF partnership program helps make a lot of connections that would not have been possible otherwise. By giving researchers access to capabilities that may be duplicative of something at ATR NSUF, but is currently oversubscribed, the connection to a partner facility allows them to get their work done faster. Likewise, some proposals that ask to use ATR don't really require its full capability. Using a partner facility like the MIT reactor, for example, is not only less expensive, it enables ATR NSUF to increase the number of experiments being done and, in turn, the number of experimenters getting access to the facilities they need.

"Let's say the experiment you wanted to perform involved putting a sample that had been irradiated in ATR into the beamline at APS," Allen explains. "In the past, you would have had to write a proposal to ATR NSUF to get permission to use the sample, and then write another proposal to APS to get their permission to use their beamline. So our staff worked with the APS folks, and now you can come in with a single proposal to APS that asks for both the sample from the ATR NSUF sample library and time on the APS beamline. We all evaluate the proposal and come back with a single answer. If it's awarded, we'll retrieve the sample, ship it to APS, they'll give you the beam time, then ship the sample back to us."

Michigan Ion Beam Laboratory

ATR NSUF piloted this streamlined process in 2012. Now they're talking with other NSUFs to see if they'll take part in this joint-solicitation concept to help make it easier on scientists proposing research that requires more than one facility. Making these kinds of connections between scientists and facilities all leads to one of Allen's ultimate goals, something he calls "research without walls".

"It's all about bringing researchers together to share their experiences," Allen explains. "Once they start talking, all kinds of new perspectives and collaborations begin to happen. That can only be a good thing."

According to Tan, "The purpose of research is to understand, and then to improve or prevent. Different opinions, or even arguments, may exist between us in our dedication to finding the truth, or a solution, but these arguments will not be a wall between us, preventing our communication, because we have the same goal. I think we are well on our way to research without walls."

The phrase conjures up a different meaning for Shao. "For me, 'walls' does not mean layers of separation," he says. "At the university, we get the freedom to do whatever research we want, and many of our projects are based on our own interpretation of needs. But the rules for ATR NSUF proposals are very specific. They are only interested in projects that will have an immediate impact. This has been a guide for me. I immediately get a sense about the direction of my future projects, and it helps me align my efforts in those directions which will have a big impact."

Now in its fifth year, the ATR NSUF partnership program has seen its initial collaborations branch out into a major national network of facilities and institutions working together to advance our knowledge of nuclear science. Where it will all lead is anyone's guess, but thanks to the efforts of Todd Allen and ATR NSUF staff, one thing is for sure; the process for getting there is infinitely smoother and exponentially faster. And that, most definitely, is a good thing for us all.

New Ceramic is Promising Alternate for Problem Material at Fukushima-Daiichi

Dr. Mujid Kazimi
Professor

Massachusetts Institute of Technology

Following the nuclear disaster at the Fukushima-Daiichi facility in Japan in 2011, the problems with a particular material – one that is typically used in many nuclear power plants around the world – became all too obvious. The material, a zirconium alloy called Zircaloy, reacted with steam and led to hydrogen explosions that released radiation into the atmosphere.

Fortunately, work on alternatives for Zircaloy is well under way. One very promising candidate is a new ceramic composite material being studied by Mujid Kazimi, professor of mechanical engineering, TEPCO professor of nuclear engineering, and director of the Center for Advanced Nuclear Energy Systems (CANES) at the Massachusetts Institute of Technology (MIT). His research group has already obtained results showing that this new ceramic composite has an "orders-of-magnitude" decrease in chemical reactivity with steam, while providing additional benefits in corrosion resistance, increased service life, and possibly even overall cost-savings. Using the MIT Research Reactor (MITR), the composite has been shown to perform well under normal reactor conditions in a pressurized water loop.

Explosive and Corrosive Issues

All light water reactors, such as those at the Fukushima-Daiichi plant, work in the same way: During nuclear fission, neutrons bombard the fuel rods, which house the fissionable material, uranium or plutonium, in the reactor core. This causes the fuel atoms to split, resulting in the production of thermal energy as well as more neutrons that can then further bombard the fuel rods in a chain reaction. The fuel rods sit in coolant, water in most cases, which circulates through the reactor. The coolant absorbs the heat and carries it away from the reactor, and that heat is used to generate steam at high pressures and temperature. The steam feeds into turbines that power electric generators, which eventually provide energy to consumers.

To prevent radioactive fission products from escaping the fuel and contaminating the coolant, tubes of cladding material encase the fuel rods, providing a barrier between the fuel and the coolant. At all commercial nuclear plants, the cladding material of choice is the zirconium alloy Zircaloy.

"We have several issues with Zircaloy," said Kazimi. "One of them is that the zirconium reacts with water while it's in service, and that produces corrosion problems. The result is that the cladding becomes thinner, and therefore we cannot allow it to stay in the core for more than a certain period of time before it has to be replaced. We need an alternative to Zircaloy that corrodes, or oxidizes, at a slower rate."

Another issue became even more pressing following the Fukushima-Daiichi accident. There, an earthquake off the coast triggered the automatic shutdown of the five reactors that were in operation at the time (a sixth reactor was already shut down). Emergency generators kicked in to provide power to the coolant system pumps. Unfortunately, an earthquake-caused tsunami rushed onshore and swamped the generators that support three of the reactors, cutting off power to the coolant pumps. Without the circulation of water, the water started to boil and the fuel temperatures rose.

At the same time, the higher temperatures also caused the coolant water to boil, which amplified the reaction between the Zircaloy and the steaming water. "This reaction can not only degrade the Zircaloy cladding, but can in fact also produce hydrogen. The hydrogen can be produced at such

Following irradiation, one of the tests performed on the ceramic composite material is a burst test, which measures how much internal pressure tubes of the material can withstand. Here, a hydraulic ram pressurizes the interior of an irradiated tube to measure the tube's strength.

Research engineer Gordon Kohse unloads a ceramic composite irradiation capsule inside a hot cell at the MIT reactor.

a rapid rate that it accumulates and becomes a potential source for ignition during an accident," Kazimi said. That is exactly what happened at Fukushima-Daiichi, he explained. "The hydrogen exploded in three reactors."

In addition, the reaction between Zircaloy and steam can also yield additional energy. "So you start having a situation where the temperature continues to increase. At those high temperatures, the Zircaloy began to melt. In the end, that can lead to the melting of the fuel, not just the cladding. Although the fuel melting point is much higher than the cladding melting point, continuous regeneration both by the decaying heat in the fuel and also by the zirconium reaction with water can exacerbate and accelerate the melting of the fuel," he said. At Fukushima-Daiichi, the fuel rods did indeed overheat to the point that they began melting.

"The accident at Fukushima-Daiichi made it more urgent to find a replacement for the cladding, so we can reduce or eliminate the potential for hydrogen explosions under severe accident conditions," Kazimi said. "From a safety perspective alone, we need to improve the performance of the cladding."

Composite Material Offers Alternative

One alternative cladding material under study is made of silicon and carbon that, when bonded together, produce a very hard ceramic called silicon carbide (SiC). The cladding material is a composite comprising three stacked layers of silicon carbide, and each of the layers serves a different purpose, said Youho Lee, a Ph.D. candidate working in Kazimi's research group.

The innermost layer, called monolithic silicon carbide, prevents the release of fission gases from the fuel rods. The second layer incorporates strands of silicon carbide that wind around the inner layer and gives the composite added strength. This is important because ceramics generally cannot withstand internal, or tensile, pressures as well as metallic materials can. "While the monolith on its own has the potential to fail under internal pressure more easily, the wound strands around the inner layer give the cladding strength so that it can expand and maintain its integrity," Kazimi said.

New Ceramic is Promising Alternate for Problem Material at Fukushima-Daiichi (cont.)

In other words, Lee added, “That means that the two layers together have a higher fracture toughness than the monolith, so it would be much more difficult for a crack to propagate.”

The final, outer layer of the triplex SiC material is an environmental barrier coating designed to protect the cladding from chemical reactions, including oxidation, that might degrade it, Lee described.

Does It Work?

Kazimi’s research group tested the SiC ceramic material in MITR to determine how well the material would perform under the harsh conditions in a nuclear power plant. “We measured the condition of the material after exposure to water and to irradiation under very similar conditions to what would exist in a commercial power reactor,” Kazimi said. “We inspected the conditions at several intervals – after three months, after six months, and after a year – and then did a variety of post-irradiation examination tests to determine what was happening to the silicon carbide as a consequence of the exposure to both elements.”

The results were quite encouraging.

“Definitely, there was better ability of the triplex composite to withstand the water,” Kazimi said. “Compared to Zircaloy, we saw far less corrosion, far less reaction with water. In fact, we saw orders of magnitude differences between the rate of reaction of silicon carbide and water, even at 300° C, which is the temperature at which the experiments were done.” When he and his research group started the research, they weren't sure about the degree of protection that the outermost layer of the composite would provide, but were pleasantly surprised with how well it functioned. “The results indicate that when manufactured in the right way, you can avoid any oxidation or corrosion to the inner layers.”

In an offshoot experiment that Lee conducted as part of his doctoral project, he found that the monolith silicon carbide also showed excellent resistance to oxidation at extreme temperatures of 1,500° C. “We actually measured the weight before and after the oxidation (loss indicates corrosion), and found that silicon carbide exhibited almost three orders of magnitude less oxidation rate than that of Zircaloy,” he said. A paper describing those findings is pending publication in the journal Nuclear Technology.

Cost savings stemming from higher burnup may be another benefit of the triplex ceramic, Kazimi said. Burnup is a measure of the amount of energy that can be extracted from the nuclear fuel during its lifespan in the reactor. Plants that can achieve higher burnup can also cut operating costs. He explained that higher burnup can be accomplished in two ways: by operating the reactor at the same power level, but for a longer period; or by operating the reactor for the same length of time, but at a higher power level. The SiC composite allows for both.

Kazimi explained, “One of the limits on burnup is the retention of the cladding's strength during irradiation. Because of the corrosion rate of Zircaloy, you might have to limit the fuel rods' residence in the core to five years or so.” The ceramic composite, on the other hand, is much less corrosive, and therefore retains its strength over an extended period of time. “The composite would open up room for higher burnup, because the fuel could remain in the core longer.”

In addition, the considerable strength of the ceramic composite also allows the reactor to run at more extreme power level. “Because of its strength, we are able to operate at a higher rate of energy extraction, which we call linear regeneration rate. So if we used the fuel rods in the reactor for the same amount of time with the Zircaloy cladding versus the ceramic cladding, we would still be able to get more energy out of the fuel with the ceramic cladding, and that would again translate to a higher burnup.”

Overall, Kazimi said, “We think we can easily get around 20 percent higher burnup with the silicon carbine cladding.”

The Next Steps

Results so far are very favorable, but more work is required, Kazimi said. “For instance, one of the challenges with silicon carbide is how to bond the end caps of the cladding to the main body of the cladding. Since this is ceramic, we can't use welding. We have to do it in a different way.” As part of its work, his research group has already irradiated and tested three potential bond materials, and is focusing on one as a possibility.

In parallel experiments, the research group is also taking a hard look at accident scenarios, such as the Fukushima-

“With these experiments and measurements, we are able to provide much more representative and applicable insight into this material, and hopefully to improve operations, especially from a safety perspective.”
Dr. Mujid Kazimi, Professor, Massachusetts Institute of Technology

Daiichi disaster, Lee said. During emergencies, standard protocol is to inject water into the reactor to cool down the fuels. “When hot fuels meet with cold water, they actually get stressed, and silicon carbide ceramics are typically brittle materials that are likely to shatter under those conditions. We are investigating whether this ceramic can maintain its integrity under those accident scenarios.”

Although Kazimi's research group still has plenty of work to do, its findings are already filling glaring knowledge gaps. “Silicon carbide is widely used in industry, so people have measured things like the reaction with water at low temperatures and high temperatures, and the strength of composites, but all of these measurements have been done on unirradiated materials. There are very few studies in the literature which have measurements for irradiation, especially under conditions representative of light water reactors.”

He added, “In the nuclear business, silicon carbide in the past was used for gas-cooled reactors in a completely different geometry than the geometry needed for the cladding in light water reactors. In gas-cooled reactors, there is no water, so the cladding didn't have the exposure to water that it does in the light water reactors that are common today.” Studies like these at MITR are providing needed understanding of possible new materials for use in nuclear power plants. He noted, “With these experiments and measurements, we are able to provide much more representative and applicable insight into this material, and hopefully to improve operations, especially from a safety perspective.”

Tubes of the new ceramic composite material are loaded into a capsule and irradiated in the MIT reactor. These specimens are subjected to the same conditions found inside the core of a full-sized power reactor.

A Unique Sample Library to Provide Needed Insight into Reactor Structural Materials

Dr. G. Robert Odette

Professor

University of California, Santa Barbara

In what can only be described as an extremely ambitious project, a West Coast scientist is leading the charge to build a unique library that will generate samples of irradiated materials, distribute them to nuclear research labs around the world, and ultimately combine the resulting information to provide new insight and understanding that is critical to current and future nuclear power plant operation.

"This kind of thing has never been done before. By that, I mean pre-planning a complex and comprehensive irradiation experiment that will have many collaborators and partners helping to carry out state-of-the-art, post-irradiation examinations of the samples," said G. Robert Odette, Ph.D., professor of mechanical engineering and materials at the University of California, Santa Barbara (UCSB). "It's like a reading club, but instead of giving our patrons books, we'll be providing various samples to the world's experts on a variety of sophisticated methods that measure what irradiation has done to the material. After they come back and tell us what they've learned, we will compile the enormous complementary body of information to create a remarkable new knowledge base."

The Need for a Library

While there have been many irradiation experiments in the past, much of the data they have produced has been narrowly focused and often fragmented. The challenge is to better understand and predict what happens to materials used in the severe conditions inside a nuclear reactor. During nuclear fission, neutrons split the nuclei of atoms in the fuel, typically plutonium or uranium, which then liberate even more neutrons that then split other nuclei, releasing a great deal of energy. However, the huge number of high-energy neutrons created, and which are measured in terms of a flux, can severely damage a material over time.

"High-energy neutrons cause radiation damage that generally degrades the properties of materials," Odette said, adding that, "A perhaps too simple analogy is that the material is being continuously peppered by little

Accelerated Tests and Late Blooming Phases: Predicting Lifespans of Reactor Materials and Components

Learning from a long experience is a luxury that utility companies don't always have, especially when it comes to nuclear power.

With a history that only goes back several decades, utility companies grapple with such questions as: What is the predicted lifespan of the components made of materials that can degrade in the severe environment in a reactor? How accurate are those predictions? Is it more cost-effective to invest in replacement components and keep the plant in operation, or to scrap the plant altogether, and perhaps switch to another form of energy production?

The UCSB ATR-1 experiment and sample library (see main article) are beginning to provide answers based on the research being led by G. Robert Odette, the University of California, Santa Barbara scientist behind the 1,400-sample library. The research is being carried out as part of the dissertation of UCSB Ph.D. student Peter Wells, who Odette says "is a sure bet to become a future world-class leader in nuclear materials science and engineering."

Odette and his research group have a long history of conducting experiments and developing models to predict the effects of neutron radiation on reactor pressure vessels that have the critical job of housing the nuclear fuel and cladding core, as well as the surrounding internal structures and coolant water. He described a typical vessel as a more-than-500,000-ton steel pressure cooker with walls up to 12" thick, that allows the coolant water to be heated up to about 300° C.

"Neutrons leak out of the core and bombard the pressure vessel, causing radiation damage," Odette said. "Although neutron exposure is relatively low, over time neutrons cause an increase in the temperature below which the steel becomes more brittle and prone to fracture. That is a high-energy neutron bullets. One bullet does not do much, but many eventually hasten an end to the useful life of a material." Other conditions in nuclear power plants, including high temperatures, stress, and corrosion can combine with radiation damage in a way that further accelerates degradation and aging of a material. As a result, a thorough understanding of radiation damage and the ability to predict the aging of reactor materials is paramount to ensuring that power plants run both safely and efficiently.

The library will tackle a wide variety of unanswered questions, Odette said. "Some are continuing questions that haven't been fully addressed, and others are new ones that, for instance, are associated with advanced developmental materials that we don't have a great deal of experience with yet."

The first step toward a clearer understanding of the materials used in a reactor service is to irradiate them in a carefully controlled manner. Few researchers, however, have access to nuclear reactors, or to the facilities to measure radioactive specimens in standard forms. That's where the library steps in. It will house the irradiated samples, and check them out to researchers around the world in both a coordinated way, and in a greatly miniaturized form, which reduces their radioactivity to such tiny levels that they can be studied in research labs without special handling facilities.

To create the library, Odette's research group used ATR at INL to irradiate 1,400 samples. Made up of a large number of structural alloys, these samples were irradiated under conditions that simulate some of the various environments the materials experience in nuclear power plants. "We call this the UCSB ATR-1 experiment. We designed this very scientifically-oriented irradiation to answer a large number and variety of fundamental questions about what happens when a material is exposed to a severe neutron irradiation environment," he said.

In planning the experiment, including which materials and samples to encompass, and under which specific conditions to irradiate them, Odette drew on guidance from current theory and modeling, and from previous experiments, as well as his own 45 years of experience in the field. He explained, "The intent is to address many major outstanding issues and key mechanisms that aren't well-understood at this point, in a way that the ultimate results will feed into developing new models that can predict the long-term behavior of materials used in reactors."

Why So Many Samples?

The range of irradiated samples is impressive. The library includes numerous specialized specimens made of more than four-dozen different materials that have been irradiated side-by-side under a wide range of conditions. Odette noted, "The effect of irradiation depends on the specific combination of a large number of variables that describe the material,

condition that obviously needs to be avoided," he said.

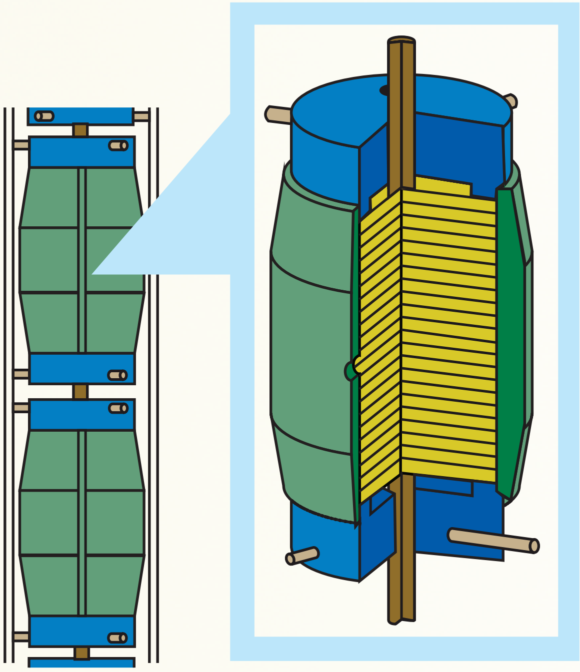
To keep tabs on the level of such "embrittlement," samples of pertinent steels are mounted in capsules on an interior wall of the pressure vessel and periodically tested. Since these "surveillance" samples are inside the vessel and closer to the core, they are irradiated a bit more quickly than the vessel itself. The resulting surveillance database is used to develop models that predict embrittlement well in advance of what is experienced by the vessel itself. (cont. next page)

G. Robert Odette, the University of California, Santa Barbara scientist behind the 1,400 sample library, points out a detail on an atom probe map to UCSB Ph.D. student Peter Wells. Wells is focusing his dissertation on so called "late blooming phases" that cause materials to become embrittled. This work has the potential to provide early insight into what could happen in operating vessels during extended service.

A Unique Sample Library to Provide Needed Insight into Reactor Structural Materials (cont.)

like its composition and processing history, and the irradiation environment including the neutron flux, energy spectrum, temperature, and exposure time. Further, measuring different properties requires various types of specimens. Thus a comprehensive experiment can get very large, very quickly, even if it is cleverly designed to be highly efficient."

One of the most important UCSB ATR-1 experimental parameters was temperature. "There are reactors that are now operating at about 300° C, and there are advanced reactors on the books that will go up to 750° C or even higher, so we need to understand what happens to a material as a function of neutron exposure over this wide range of temperatures," Odette said. He added, "And we want to compare the behavior of different materials so we can pick or develop one that meets the needs of a particular application." To that end, the UCSB ATR-1 irradiation includes sample packets at seven temperatures between 290-750° C. "If in the future a researcher needs data on materials in a reactor that runs at 650° C, he or she can go to the library and check out the specimens that were exposed under those conditions. More generally and importantly, UCSB ATR-1 provides for side-by-side comparisons of the behavior of many different materials at different temperatures and neutron exposure levels."



As part of the UCSB ATR-1 experiment, specimen packets (the yellow stack in the schematic illustration) were irradiated at precisely controlled temperatures. The experiment includes 32 such packets that cover a temperature range from about 300-750° C. This allows researchers to compare the behavior of different materials under different irradiation conditions that span a very wide range of advanced nuclear reactor applications.

Accelerated Tests and Late Blooming Phases Predicting Lifespans of Reactor Materials and Components (cont.)

"Including surveillance capsules was a very, very wise decision for assuring the safe operation of reactor pressure vessels, but the issue now is that the utility companies want to extend the plant lifetimes," Odette said. "Current models permit predictions out to about 30 years, but companies need early insight into what's going to happen at lifetimes 60, 80, even or even 100 years of operation."

Two key issues arise from this challenge, he said. "The high exposures that occur at low flux and over long times during extended vessel life can be quickly achieved at higher flux in test reactors. However, this approach begs the question: How does the higher accelerated flux affect embrittlement?" He added, "A second issue is whether new and different things – things that have not been previously experienced – may emerge from the bushes at high neutron exposures and low flux to cause severe embrittlement."

Many students and young postdocs from around the world will be involved with this research, including UCSB Ph.D. student Peter Wells, whose research under the sponsorship of ATR NSUF and the DOE Nuclear Energy University Program (NEUP) on alloys irradiated in UCSB ATR-1 is providing a glimpse of the future. The experiment involved a flux about 5,000 times higher, and a fluence (flux times time) that was about 10 times higher than the vessels would experience in a typical light water reactor. "We know that high flux requires higher fluence to produce the same amount of embrittlement, but we do not yet know how much higher," Odette said. "Nevertheless, the high flux and fluence UCSB ATR-1 irradiation is giving us early insight into what could happen in operating vessels during extended service."

To be sure they're comparing apples to apples, he and his research group are also studying how well high fluence accumulated over a short time mimics the same fluence acquired over a much longer time

UCSB ATR-1 irradiated the set of materials at neutron flux levels higher than those in current light water reactors. Proposed fast reactors – those that use enriched fuel and produce only high-energy neutrons – run at these higher flux levels. "We don't have fast reactors in the U.S. now, but the Department of Energy wants to develop alternative reactor concepts that can burn the fuel more efficiently, reduce the waste burden, and require fewer resources," he said. "The understanding we're generating in UCSB ATR-1 will play an important role in developing advanced reactors."

The UCSB ATR-1 experiment will also provide data relevant to accident-tolerant fuel cladding in current light water reactors (LWRs). "The severe consequences of the accident at the Fukushima Daiichi plant in Japan in 2011 were greatly amplified by the zirconium alloy used in the cladding. At high temperatures, following the loss-of-coolant accident, or LOCA, zirconium reacted with steam to produce hydrogen; and it was the resulting hydrogen explosions that spread radiation over a wide area of Japan," Odette said. "Consequently, there's a push to consider replacing or modifying zirconium claddings in such a way that production of hydrogen is reduced or eliminated during a severe LOCA."

The library covers a wide variety of cladding materials. These include currently used alloys, commercially available materials that are not presently used in reactors, and "completely new alloys that we're developing and believe will have a much higher degree of tolerance to radiation damage." He added, "These include what we call nanostructured ferritic alloys. These are a new class of potentially transformational materials in terms of their high-temperature strength and the amount of radiation exposure they can tolerate without experiencing severe degradation of their important engineering properties. These, and other advanced materials, might also be among the good candidates for accident-tolerant cladding materials."

Learning from the Library – Small is the New Big

Unlike a public library where the patron checks out a full book, the sample library will provide many of its users with just a small portion of any individual sample. In many cases, the small specimens will be generated through the use of a recently developed piece of equipment called a focused ion beam (FIB) microscope. The FIB can cut out samples with dimensions of only microns, or a millionth of a meter. As the size diminishes, so, too, does the radiation level. The larger samples are often fairly radioactive, but once the FIB micromachines them to extract the miniscule bits, the specimens carry so little activity (the number of decays per second) that they are no longer considered to be radioactive. In fact, he said, the radiation in one of these tiny specimens can be lower than that in a single banana.

Although small in size, the specimens are chockfull of valuable information, especially now

in actual service. This involves comparisons of the effects UCSB ATR-1 and other irradiations that span an overall flux range of more than 10,000, while using special mechanism experiments and models to splice together data that covers different fluence ranges.

This work has already identified what he called "late-blooming phases" that cause embrittlement, but form only at high fluence. "Once late-blooming phases start to form, they can grow very quickly and could cause severe embrittlement, which could be a nasty technical surprise that is not accounted for in current regulations and models," Odette said.

The late-blooming phases aren't a major safety issue in operating plants, he pointed out, because they would become evident before the material became overly degraded. "Understanding this phenomena, however, is important because the utilities need to make decisions now about whether they want to go ahead and continue to operate their reactors, which together produce about 20 percent of our electricity today, and an even larger fraction of our carbon-free electricity. These are very significant questions."

He added, "While we can't give them a direct answer right now, by learning about the mechanisms involved and by developing physical models based on a growing database, including contributions from UCSB ATR-1, we are starting to be in a position to make better predictions and provide reliable answers."

Odette also noted that UCSB ATR-1 is not the end of the story: "Our large ATR NSUF UCSB ATR-2 experiment directly addresses the issue of pressure-vessel embrittlement at high fluence, but at a much lower, although still accelerated, flux compared to UCSB ATR-1." He believes that work at ATR NSUF will bring better resolution and perhaps closure to some of these very complicated questions in a way that will support extended vessel life, while assuring his primary objective, which is an extraordinary margin of safety.

A Unique Sample Library to Provide Needed Insight into Reactor Structural Materials (cont.)

that researchers have at their fingertips "some wonderful new tools both for measuring mechanical properties of materials, and for characterizing their microstructures," Odette said. The latter is important because the microstructure determines the material's properties. "It's kind of like flour, water, sugar, and other ingredients. You can mix them all together, but it's not a cake yet. The same holds true for materials. You have to properly manipulate the 'ingredients' so they produce the correct arrangement of atoms to generate the microstructure you desire, that in turn results in the properties you're after."

Neutron exposure, however, alters the microstructure, most often degrading it and producing changes that are detrimental to the material's performance. "Consider a steel that's typically made of iron, carbon and at least three or four other alloying elements, such as nickel and chromium," Odette said. "What we need to do is to characterize the arrangement of these elements into various microstructural features, such as precipitates, down to the atomic scale. We're talking about things that are really, really small."

The new and evolving tools are providing researchers this incredible view. "The instruments include atom probes that allow you to peel off a single atom and identify what it is and where it was located in the material, and to repeat this tens of millions of times so that the atomic microstructure can be reconstructed in three-dimensions," he said. "Other important tools that can see inside a material at an incredibly small scale include advanced electron microscopes and very powerful synchrotron sources of X-rays. Post-irradiation examinations of the samples using these tools will revolutionize our understanding of reactor materials."

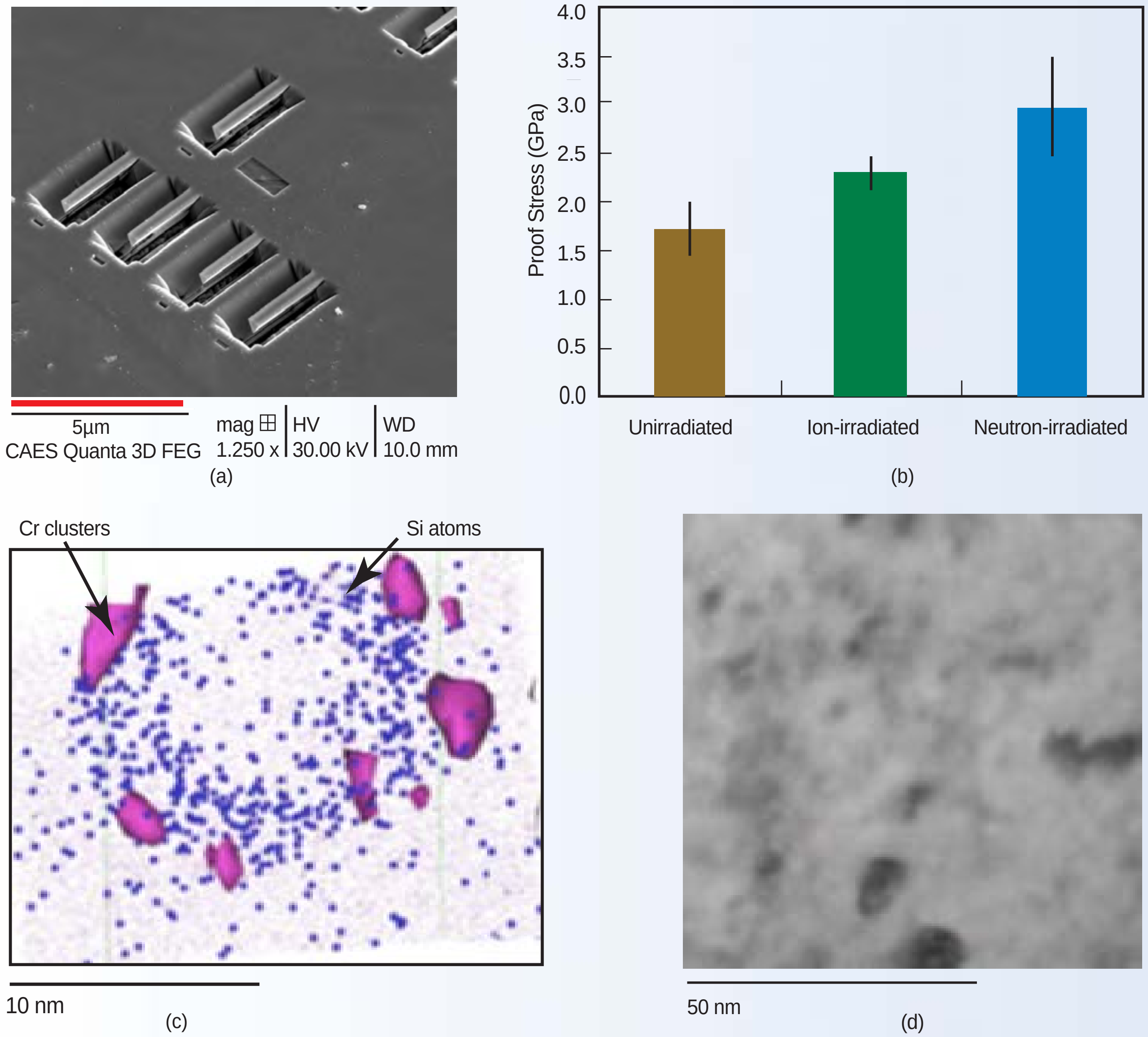
Growing Interest

With about two dozen of the 1,400 irradiated samples already available to study, and more on the way, the library is not only quickly gaining attention, but is also attracting its first patrons. "For example, we're working with Professor Emmanuelle Marquis, a world-leading expert on atom probe tomography at the University of Michigan. Emmanuelle is carrying out remarkable measurements on an iron-chromium (Fe-Cr) series of alloys irradiated in UCSB ATR-1," Odette said. "We're also working with Professor Steve Roberts at the University of Oxford, who is an international leader in microscale testing methods that can extract mechanical properties from specimens measuring about a tenth the diameter of a human hair."

Other researchers are waiting in the wings to obtain library specimens and to join the "reading club." At a side gathering Odette organized during the Annual Meeting of TMS in 2010, about 25 people from England, France, Japan, many of the U.S. national laboratories, and a numerous universities assembled to learn about the library and opportunities for collaboration. He said, "The interest is definitely there, and we're hoping to get a large number of world-class scientists involved."

The library and the researchers in the "reading club" will have a major impact on the nuclear power industry, Odette believes. "Our collaborators will check out library specimens, do their expert post-irradiation examinations, report their findings and write papers. This will put us in a position to assemble, assimilate, and integrate all this different information, thus giving us a huge lever in answering the many questions we have about nuclear reactor materials."

With an obvious enthusiasm, he concluded, "All of this research will be great. However, there is an equally important added benefit: Many students and young postdocs from around the world will be involved with the library and with meeting the goals of the network of collaborations. The work of UCSB Ph.D. student Peter Wells (see inset), for instance, illustrates the role of extremely high-quality scientific research in addressing immensely important technological issues. The quality and impact of their research and experience in a new type of international collaboration will be a great foundation for their ongoing contributions to the field of nuclear energy for many decades to come."



Through this UCSB ATR-1 experiment, doors are opening to a unique library that will ultimately generate 1,400 samples of irradiated materials, distribute them to nuclear research labs around the world, and provide new insights into current and future nuclear power plant operation. Research is already under way on the effects of irradiation on microstructure and mechanical properties on iron chromium (Fe 6%Cr) alloys: (a) micro machined beams are used for testing the effect of irradiation on the alloy strength (the red line is the average diameter of a human hair) (Oxford University); (b) the strength of the alloy in unirradiated, and ion- or neutron-irradiated condition at the same damage level (University of Oxford); (c) an atom probe tomography solute map showing a dislocation loop irradiation defect decorated with chromium and silicon atoms in the neutron-irradiated alloy (University of Michigan); (d) an electron microscopy image of decorated loops in the ion-irradiated alloy (University of California, Santa Barbara). These studies will add up to a whole that affords a greater understanding of irradiation effects on Fe-Cr structural alloys.

ATR NSUF Program Information

ATR NSUF users at work.


NEC

Program Overview

ATR NSUF: A Model for Collaboration

ATR NSUF and its partner facilities represent a prototype laboratory for the future. This unique model is best described as 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 nation-wide 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 the radiation environment of a nuclear reactor.

Since ATR NSUF established the partnership program, eight universities and two national laboratories have offered their facilities' capabilities, greatly expanding the kinds of research that can be offered. The avenues opened through these partnerships facilitate cooperative research across the country, matching people with capabilities, students with mentors.

In 2012, 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.

In the pages that follow, you will read specific details on the capabilities of ATR NSUF and its partners. You will also learn how to access these capabilities through the calls for proposals. ATR NSUF hosts a yearly Users Week designed to instruct and inform. This week 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.

We hope you take time to familiarize yourself with the many opportunities offered by ATR NSUF, and consider submitting a proposal or two!

Dr. Todd Allen welcoming participants to the Idaho portion of 2012 Users Week.

U.S. DEPARTMENT OF **ENERGY** | **Nuclear Energy**

ATR NSUF Research Supports DOE-NE Missions

The 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.

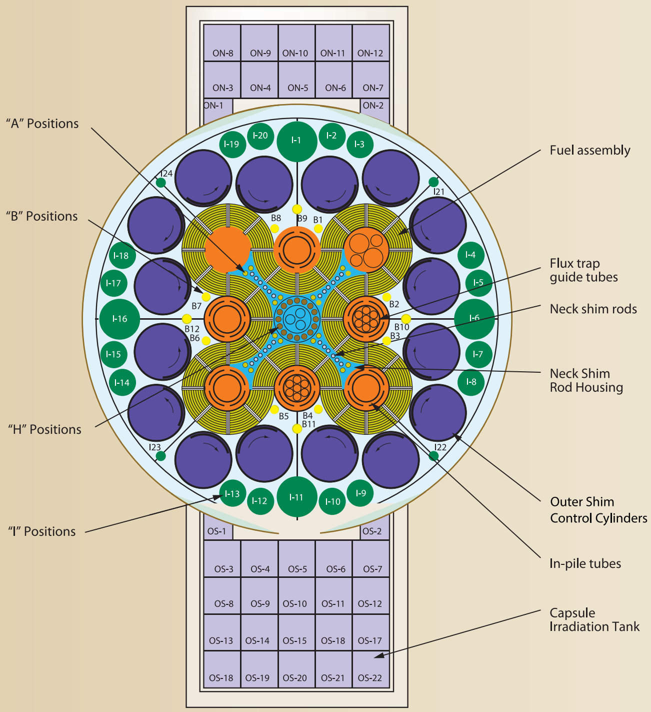
ATR NSUF research addresses a number of these mission needs. Most of the research contained in this report looks at either understanding the mechanisms of radiation on materials and fuels to address the challenges of the aging current fleet, or looks at materials and fuels for the next generation of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

To learn more about proposing a research project, please visit the ATR NSUF website at: 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

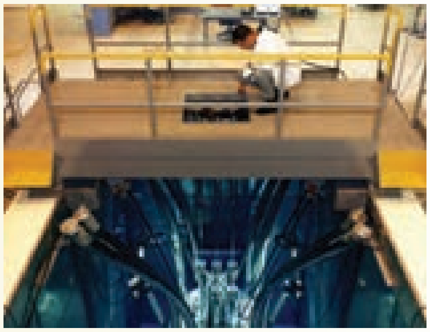
ATR is a water-cooled, high-flux test reactor, with a unique serpentine design that allows large power variations among its flux traps. The reactor's curved fuel arrangement places fuel closer on all sides of the flux trap positions than is possible in a rectangular grid. The reactor has nine of these 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 5.0" in diameter and all are 48" long. The peak thermal flux is $1x10^{15}$ n/cm^2-sec and fast flux is $5x10^{14}$ n/cm^2-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



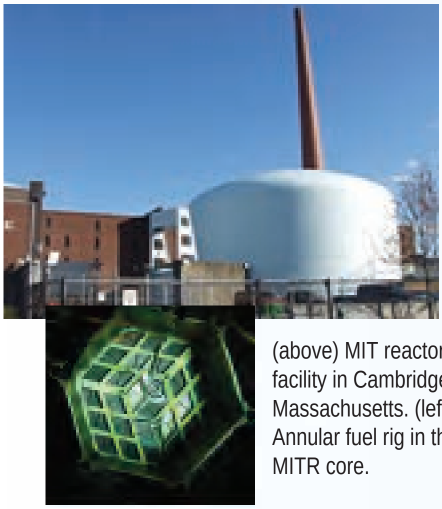
Aerial view of the ATRC reactor core and bridge.

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 2.5x1015 n/cm2-s and a peak fast flux of 1.1x1015 n/cm2-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 1014 and 5x1014 n/cm2-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%.

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% 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.

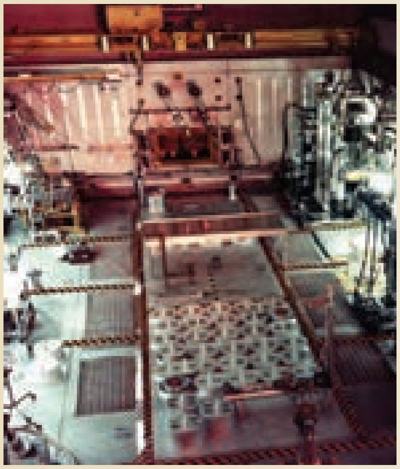
More information: http://atrnsuf.inl.gov/documents/PULSTARReactor.pdf

(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.

Included in 2012 were 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.

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

The Scanning Electron Microscope in Oak Ridge National Laboratory's 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

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

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

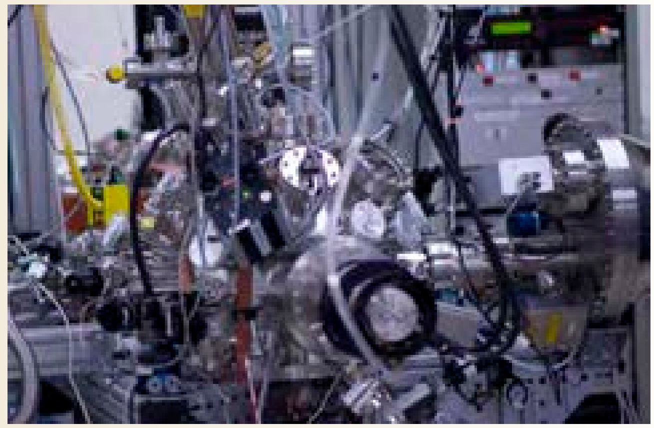
More information: http://atrnsuf.inl.gov/documents/PNNL_UserGuide.pdf

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.

UC Berkeley nano-indentation system.

University of California, Berkeley Nuclear Materials Laboratory

The Nuclear Materials Laboratory provides several capabilities for examining irradiated material samples including a 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.

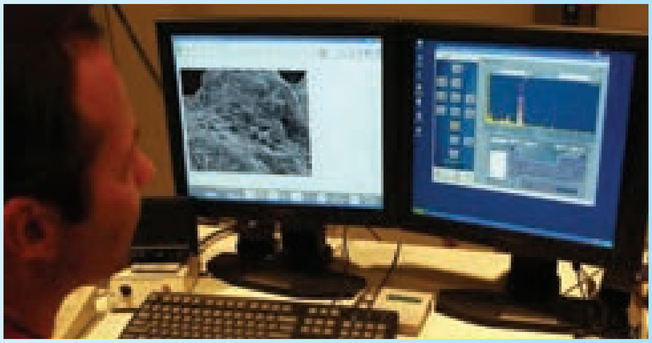
More information: http://atrnsuf.inl.gov/documents/UniversityofMichiganIMCandMIBLFacilities.pdf

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

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 imagining, as well as positron and neutron activation analysis.

In 2012, 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

Frances Marshall
Manager, Program Operations

Mary Catherine Thelen
Program Administrator

Calls for Proposals

The ATR NSUF mission is to offer access to unparalleled nuclear fuels, materials, and reactor technology capabilities to help addres the unique challenges of nuclear energy related questions. 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 two research proposal options (described in more detail below) through an online submittal system that helps prospective researchers develop, edit, review, and submit their proposals. ATR NSUF staff is available to help any researcher submit a proposal.

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

All proposals are subject to a peer-review process before selection. An accredited U.S. university or college must lead research proposals for irradiation and post-irradiation examination only experiments. Collaborations with other national laboratories, federal agencies, non-U.S. universities and industries are encouraged.

Any U.S.-based entities, including universities, national laboratories and industry can propose research that would utilize the Materials Research Collaborative Access Team (MRCAT) beamline at the Advanced Photon Source or would be conducted as a rapid turnaround experiment.

Calls for Irradiation, Post-irradiation Examination and MRCAT Experiments

ATR NSUF annually conducts two open calls for proposals. The first is the fall call, which is aligned with the Nuclear Energy University Program (NEUP) call.

This alignment allows proposers who require both NEUP funding and ATR NSUF capabilities to propose only once, to the joint ATR NSUF/NEUP call. For proposers who only want access to ATR NSUF capabilities, the call opens in July and closes in December. Awards are made at the same time as NEUP awards, and therefore the award date is subject to change. ATR NSUF also offers a spring/summer call that opens in January and closes in June. Awards for this call are typically made in late October or early November. Proposals for the open calls are accepted for:

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

All proposals submitted to the open calls undergo thorough reviews for feasibility, technical merit, relevance to DOE-NE missions, and cost. Proposals submitted to the joint ATR NSUF/NEUP call undergo reviews in accordance with NEUP guidance. The results for proposals requiring ATR NSUF capability 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 scientific director. For the joint call, proposals must rank high in both the NEUP and ATR NSUF rankings. 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.

Users Week

Participants attending a fuels course during 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 participants trying out a glove box in CAES.

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 ATR NSUF's call for proposals. Users Week supports ATR NSUF as a model for the laboratory of the future, where collaborative research and shared resources among universities and national laboratories will help prepare a new generation of nuclear energy professionals.

The week's events are free of charge for students, faculty and postdocs 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 508 participants from 30 countries and 35 U.S. universities.

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

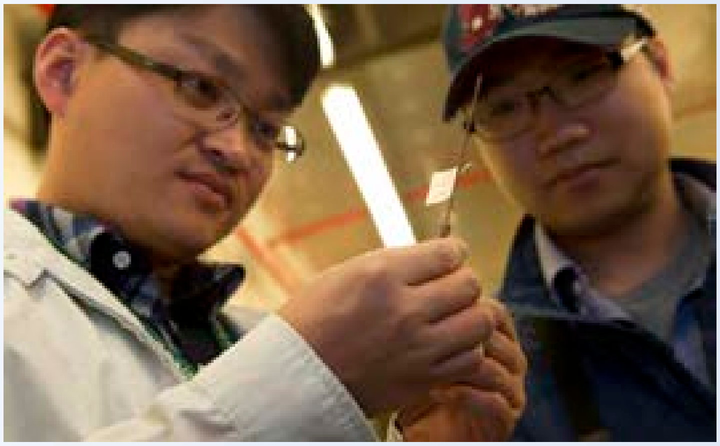
What to Expect at Users Week

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.

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 ATR as well as INL's Materials and Fuels Complex where many post-irradiation examination facilities are housed.

For more information about Users Week please visit the ATR NSUF website at: http://atrnsuf.inl.gov



Users Week participants checking flux wires for irradiation experiments.

Touring the Electron Microscopy Laboratory at the Materials and Fuels Complex during Users Week.

Jeff Benson
Education Coordinator

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 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.

Participants of the ATR NSUF Users Week Workshop.

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 December, 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

A hands-on workshop at the INL High Temperature Test Laboratory during Users Week.

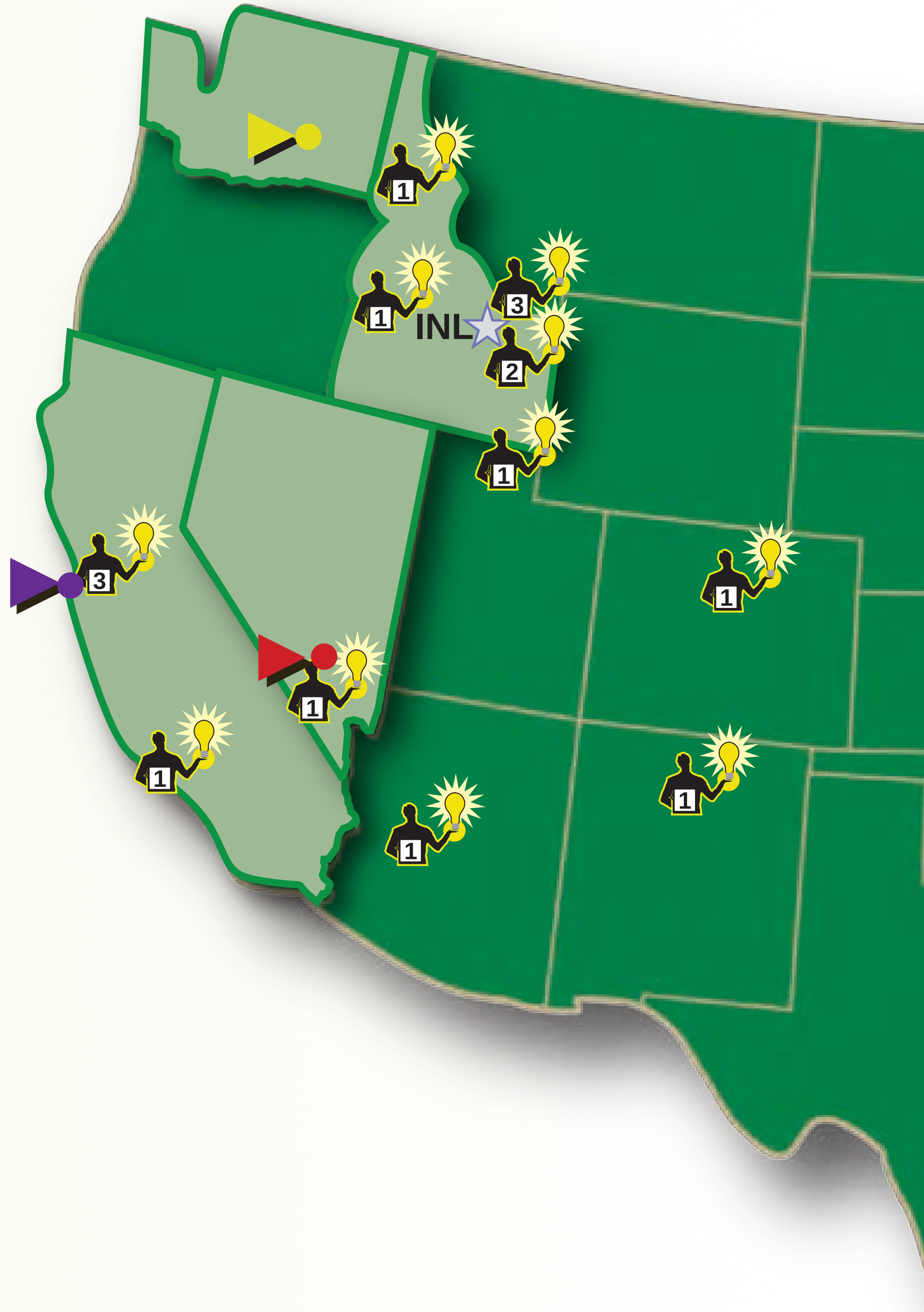
INL researcher, Dr. Assel Aitkaliyeva explaining microscopy to several students during Users Week.

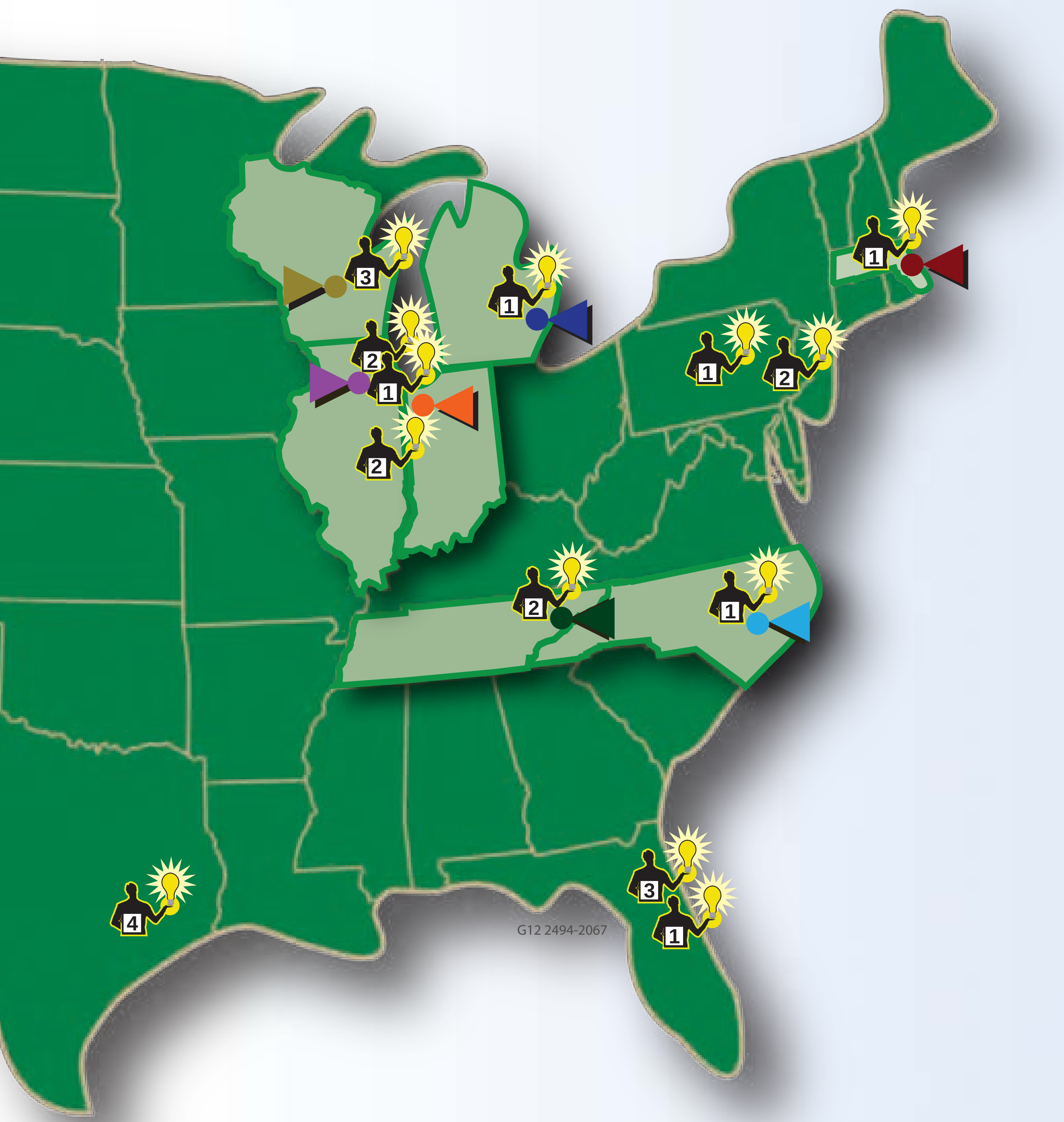
Distributed Partnership at a Glance

PIs/Users

Partners

- BERKELEY, UNIVERSITY OF CALIFORNIA
- ILLINOIS INSTITUTE OF TECHNOLOGY
- Massachusetts Institute of Technology
- University of Michigan
- NC STATE UNIVERSITY
- OAK RIDGE National Laboratory
- Pacific Northwest NATIONAL LABORATORY
- PURDUE UNIVERSITY
- UNLV
- THE UNIVERSITY WISCONSIN MADISON

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ATR NSUF Research Project Reports

Principal Investigators: Mehdi Balooch and Donald R. Olander – University of California, Berkeley
e-mail: mehdibalooch@gmail.com / fuelpr@nuc.berkeley.edu

Hydride LWR Fuel Rod Irradiation

Introduction

One of the fuels proposed for use in light water reactors (LWR) is uranium-zirconium hydride fuel pellets bonded with a lead-bismuth eutectic (LBE) liquid metal (LM) alloy.[1] This fuel has the potential to improve safety margins, allow for power uprates, and increase fuel burnup.

Liquid metal bonded hydride fuel has the potential to improve safety margins, allow for power uprates, and increase fuel burnup.

Project Description

This ATR NSUF research project set out to study the materials issues of this hydride fuel for power production in LWRs and to explore the use of an LM as a replacement for helium (He) to fill the pellet-cladding gap. The materials' aspects of as-fabricated hydride nuclear fuels have been investigated on the laboratory scale,[2] however, in order to fully evaluate the feasibility of the LWR hydride fuel concept, a shift from laboratory-scale experiments to more relevant environments was necessary.

Beginning in March 2011, mini-fuel rods with centerline and surface thermocouples attached were inserted into titanium (Ti) capsules connected to cover gas lines at the Massachusetts Institute of Technology Reactor (MITR) for irradiation. Centering pins were attached to the capsules to position them within the flow channel to ensure a uniform flow of coolant around the capsules' outer surfaces (Figure 1).

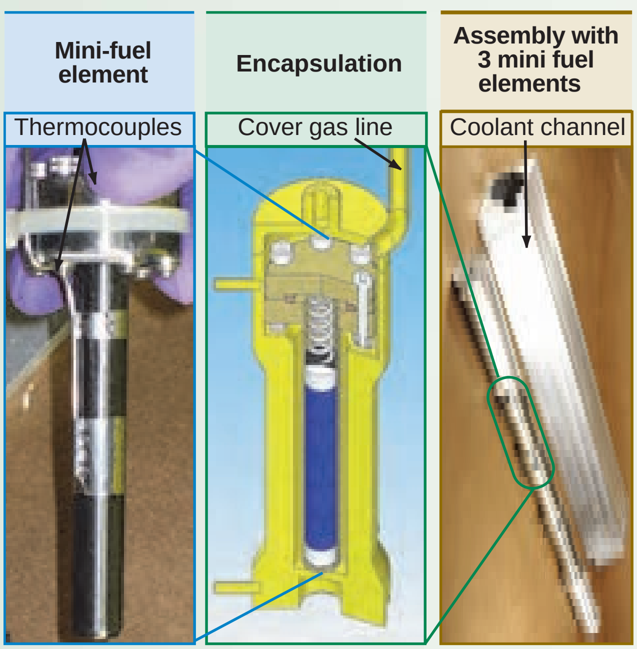

Figure 1. Mini-fuel, Ti capsule, and assembly for insertion into the MITR core for irradiation.

Two initial capsules were inserted at the lower and middle positions, while the upper position was filled with an aluminum dummy capsule. During the first phase of the experiment, reactor power was gradually raised to 4.0 MW, and fuel temperature data was collected at different reactor powers. After the temperature and cover gas measurements were confirmed to be within the approved limits, a third fuel rod capsule was added to the upper position, and the reactor power was increased to 6.0 MW. Power density was back-calculated from the reactor power monitored by neutron detectors located adjacent to the core.

Accomplishments

Calculations from the temperature gradient and water flow rate through the core showed good agreement between the neutron detector signal and thermal power. Rather than being directly measured, local power variations inside the core and within the experimental capsules were modeled by the Monte Carlo N-Particle Code, number 5 (MCNP5). The center fuel pellet and cladding temperatures were continuously monitored throughout the irradiation and shutdowns by K-type thermocouples. In order to quantify the rate of fission-gas leakage from the fuel rod into the capsule interior, the He cover gas inside the capsules was periodically purged into a sampling container and analyzed by gamma spectroscopy.

On June 26, 2011, at the highest flux and temperature, an increase in fission-gas release from capsule 2 was detected and the capsule was removed. Failure of the copper gasket sealing the upper flange of the fuel rod or, less likely, failure of the cladding, is suspected. In November 2011, capsule 1 showed a similar, though less dramatic, increase in fission-gas release. It was removed and replaced with a dummy capsule.

At the end of irradiation, the equivalent oxide burnup for capsules 1, 2, and 3 reached 4.5, 3.0, and 2.5 MWd/kgU, respectively. The extracted in-pile thermal conductivity data for U(30wt%)-$ZrH_{1.6}$ fuel as a function of time shows a slight rise at the beginning of irradiation followed by constant values on two of the capsules. A significant decline in the inferred thermal conductivity of the fuel for all three capsules was observed at higher burnup (Figure 2).

During a reactor outage in January 2012, an inspection of the capsules revealed mechanical damage to the bottom spacer in the fuel capsule stack and some missing pins on both the spacers and capsules. This was attributed to inadequate welds on the pins together with possible handling damage during capsule change-outs. As a result, all three capsules were removed from the reactor and transferred to the spent fuel pool, where they are currently awaiting transport to INL’s Hot Fuel Examination Facility (HFEF) for post-irradiation examination (PIE).

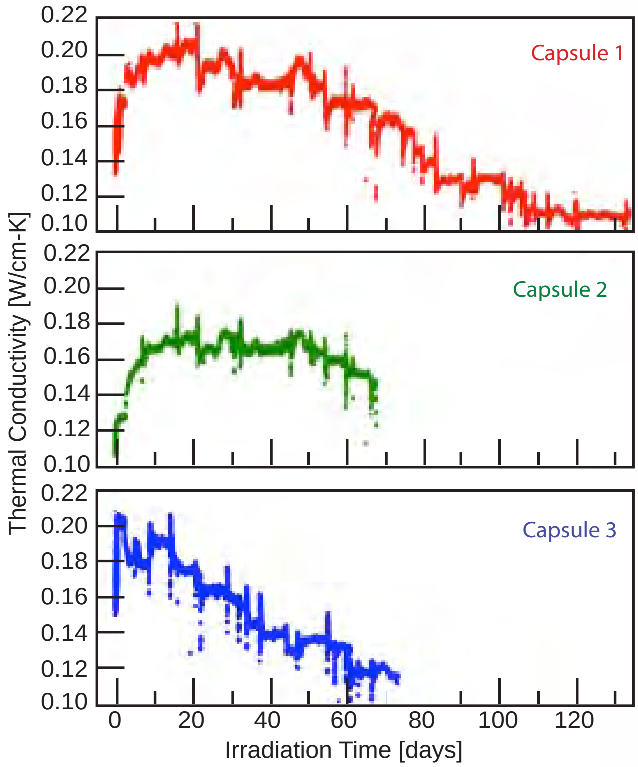

Figure 2. Thermal conductivity of U(30wt%)-$ZrH_{1.6}$ fuel measured in-pile at MITR.

No significant recovery in thermal conductivity was observed during multiple shut-downs and ramp-ups of power. The gradual increase in thermal resistance between the center fuel and cladding during irradiation may have resulted in unexpected oxidation of the cladding or LM, or the formation of gaseous fission products within the gap. Nevertheless, the thermal conductivity of the hydride fuel is shown to be three to six times greater than that of oxide fuel (Figure 3 next page).

Conductivity reduction with burnup has also been reported for oxide fuel, but at much higher burnup values. There, the reduction could be attributed to one or more of the following:

- Structural or electrical point defects created by irradiation
- Growth of solid fission products
- Fission-gas bubbles
- Fracturing of the fuel.

Future Activities

PIE investigation of the mini-fuel elements, involving characterization techniques previously demonstrated on as-fabricated hydride fuels, should provide more concrete reasons for the sensitivity of thermal conductivity during irradiation. The extent of fission-gas release from the fuel through the LM gap filler, as well the redistribution of hydrogen in the fuel and the condition of the Zircaloy cladding, will be examined in INL’s HFEF. Fuel swelling will be measured to determine whether it is caused by solid fission products, fission-gas bubbles, or other defects. The possibility of gas bubble formation in the LM bond, which would compromise the high thermal conductivity of the gap, will also be checked.

Hydride LWR Fuel Rod Irradiation (cont.)

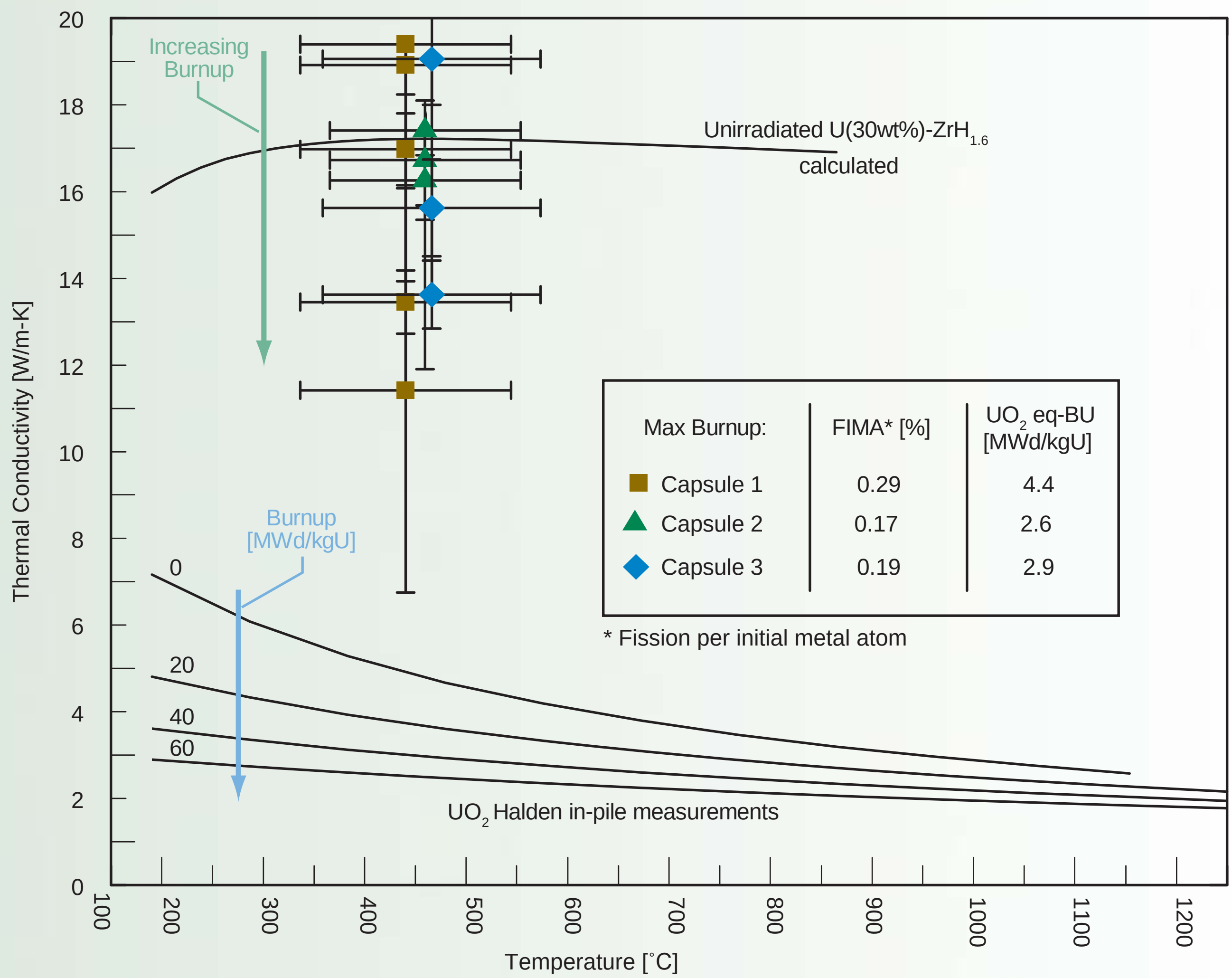

Figure 3. Comparison of in-pile thermal conductivity of U(30wt%)-$ZrH_{1.6}$ and UO_2.

References

1. Full issue, 2009, "Hydride Fuele LWRs," *Nuclear Engineering and Design*, Vol. 239, No. 8, August 2009, pp. 1373-1570.

2. Kurt Terrani, 2010, *Incorporation of Hydride Nuclear Fuels in Commercial Light Water Reactors*, Ph.D. Thesis: University of California, Berkeley, California.

Publications and Presentations

1. Sung Joong Kim, David Carpenter, Gordon Kohse, Kurt Terrani, Lin-wen Hu, 2012, "Thermal-Hydraulic Analysis of LWR Hydride Fuel Irradiation Experiment in MITR," *ANS 2012: Nuclear Fuels and Structural Materials for the Next Generation Nuclear Reactors (NFSM 2012), Chicago, Illinois, June 24–28, 2012.*

2. Mehdi Balooch, Kurt Terrani, Donald Olander, Sung Joong Kim, Thomas Newton, Gordon Kohse, Lin-wen Hu and David Carpenter, 2011, "In-pile Thermal Conductivity Measurement of Liquid-Metal-Bonded Hydride Fuel Rods," *The 2nd Workshop on Hydride Utilization in Nuclear Reactors, Tokyo, Japan, December 19-20, 2011*.

3. Mehdi Balooch, 2012, "Investigation of Feasibility of Incorporation of Hydride in Fuels" *ATR NSUF Users Week Research Forum, Idaho Falls, Idaho, June 18-22, 2012.*

4. Brendan Ensor, 2012, *Thermal Analysis of Uranium Zirconium Hydride Fuel Using a Lead-bismuth Gap at LWR Operating Temperatures,* B.S. Thesis: Massachusetts Institute of Technology, Cambridge, Massachusetts.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	PIE facilities
Massachusetts Institute of Technology	Massachusetts Institute of Technology Reactor
Team Members/Collaborators	
University of California, Berkeley Mehdi Balooch (principal investigator), Donald Olander (co-principal investigator), Kurt Terrani (Ph.D. student, presently at Oak Ridge National Laboratory)	
Idaho National Laboratory James Cole (INL principal investigator)	
Massachusetts Institute of Technology Gordon Kohse, Lin-wen Hu, David Carpenter (co-investigators), Brendan M. Ensor (student collaborator)	

Irradiation Effect on Thermophysical Properties of Hf3Al-Al Composite: A Concept for Fast Neutron Testing at ATR

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This experiment will provide necessary data for the development of a fast neutron test capability at ATR.

Introduction

Conducting fast neutron irradiation tests is essential to meet fuels and materials development requirements for future nuclear reactors. This development is currently hindered by a lack of domestic capabilities for these tests. The concept behind this project is to add a neutron filter to one of the corner lobes of INL's ATR to absorb the thermal neutrons and booster fuel in order to augment the neutron flux.

Project Description

For this project, researchers produced and characterized specimens of a new absorber material comprised of hafnium aluminide (HfAl$_3$) particles (~28.4 % by volume) in an aluminum matrix ((HfAl$_3$-Al), which can absorb thermal neutrons and transfer heat from the experiment to pressurized water cooling channels. Thermal analysis conducted on a candidate configuration confirmed that the design of the water-cooled HfAl$_3$-Al absorber block is capable of maintaining all system components below their maximum allowable temperature limits.

However, the thermophysical properties of HfAl$_3$-Al have never been measured, and the effect of radiation on these properties has never been determined. In order to proceed with the design and optimization of these new materials, it is essential to obtain data on the thermophysical and mechanical properties of both the HfAl$_3$-Al intermetallic and HfAl$_3$-Al composite, as well as corrosion behavior and radioactive decay products.

Specific objectives of the experiment are to determine the:

- Thermophysical and mechanical properties of HfAl$_3$-Al intermetallic and HfAl$_3$-Al composite at different temperatures
- Effect of irradiation on the thermophysical and material properties of the HfAl$_3$-Al intermetallic and HfAl$_3$-Al composite, and physical/morphological, metallurgical, and microstructural changes in the HfAl$_3$-Al composite after different cycles of irradiation
- Decay products of hafnium (Hf-179m1 versus Hf-179m2) and corrosive behavior of the HfAl$_3$-Al composite.

Successful completion of the project will not only provide necessary data for the development of fast neutron test capabilities at ATR, but it will also fill a knowledge gap on the basic properties of these materials and advance the scientific understanding of irradiation effects on them.

Accomplishments

The irradiation was completed in February of 2012 and the capsules were removed from the reactor to the canal for cooling. The capsules were shipped to MFC for PIE in April of 2012, and PIE is expected to be initiated in late 2013.

During 2012, mechanical property testing and microstructural evaluations of the unirradiated materials were performed in the Microscopy and Characterization Suite (MaCS) at CAES. Microhardness and tensile testing were conducted at room temperature. Local electrode atom probe (LEAP) and transmission electron microscopy (TEM) analyses were performed to investigate the microstructure of the materials (Figure 1).

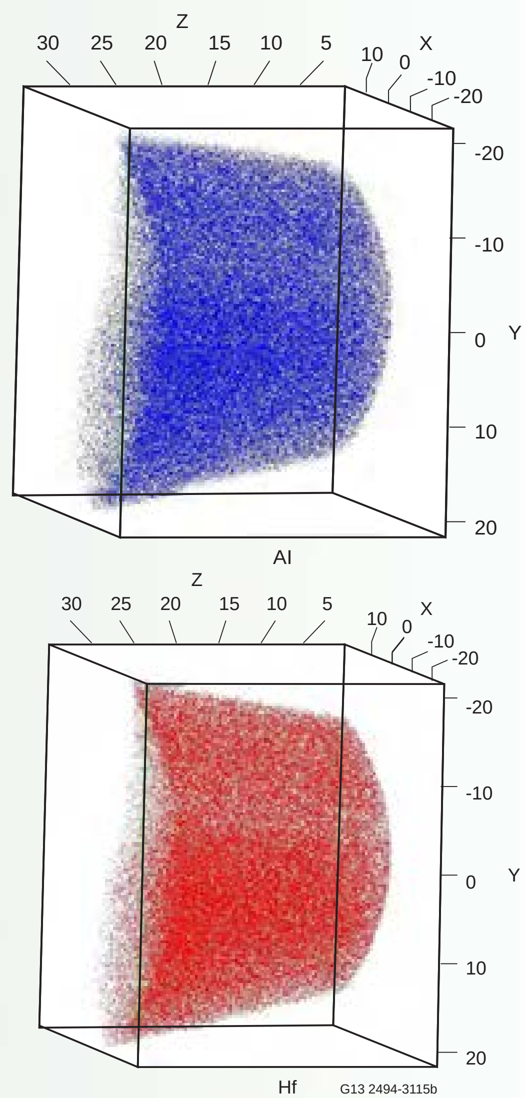
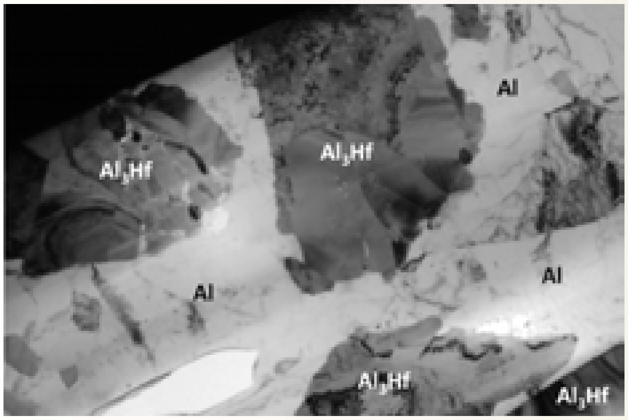


Figure 1. Elemental maps obtained from LEAP analysis.

"This project has been the most exciting and meaningful of my career! Our team developed a new reactor material, fabricated miniature specimens for irradiations in ATR, and engaged the energy and enthusiasm of a number of university students who have, due to this research, gone on to pursue advanced degrees in engineering."

Dr. Donna Post Guillen, INL Principal Investigator

Ultra-thin samples were prepared from a larger specimen using a focused ion beam (FIB) technique to capture the phase interface between a hafnium-aluminide particle and the aluminum matrix. TEM bright field and high-resolution images showed a sharp interface between the two phases (Figure 2). These studies confirmed that the particles did not interact with the matrix during hot pressing to form extra compounds at the interface.



2 µm

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Figure 2. Low magnification bright field TEM image of the sample, showing a microstructure mixed with Al and Al_3Hf.

Future Activities

Thermal diffusivity and effusivity measurements are scheduled to be performed on the unirradiated material at Utah State University (USU) in 2013. Post-irradiation examination (PIE) of the irradiated specimens has not yet begun. The four USU capsule pairs were shipped from ATR to INL's MFC, and are currently stored in the Hot Fuels Examination Facility awaiting a collimated axial gamma scan to determine whether highly radioactive metastable isotopes of hafnium are present. Depending on the results, the capsules will either be shipped to a partner facility or remain at MFC for PIE. If the PIE takes place at MFC, the capsules are scheduled to be disassembled in May 2013.

Publications and Presentations

1. Donna P. Guillen, W. Dave Swank, Adam X. Zabriskie, and Heng Ban, 2011,"Prediction of Specimen Temperatures During Irradiation of a New Composite Material," *The 14th International Topical Meeting on Nuclear Reactor Thermal Hydraulics, Toronto, Ontario, Canada, September 25-29, 2011*, NURETH14-108.

2. Donna P. Guillen, Brian P. Durtschi, Adam X. Zabriskie, and Heng Ban, 2011, "Integrated Static and Flow-Through Capsule Assembly for Irradiation Testing," *2011 Transactions of the American Nuclear Society*, 2011 *American Nuclear Society Meeting, Hollywood, Florida, June 26-30, 2011.*

3. Donna P. Guillen, Douglas L. Porter, Jim R. Parry, Heng Ban, 2010, "In-Pile Experiment of a New Hafnium-Aluminide Composite Material to Enable Fast Neutron Testing in the Advanced Test Reactor," *2010 American Nuclear Society International Congress on Advances in Nuclear Power Plants ICAPP '10, San Diego, California, June 13-17, 2010,* Paper 10115.

4. Heather Wampler, Adam Gerth, Heng Ban, Donna P. Guillen, Douglas L. Porter, Cindy Papesch, and Thomas Hartmann, 2010, "Fabrication and Characterization of a Conduction Cooled Thermal Neutron Filter," *2010 American Nuclear Society International Congress on Advances in Nuclear Power Plants ICAPP '10, San Diego, California, June 13-17, 2010,* Paper 10118.

Patents Applied For

Attorney Docket No. 2939-10350US (BA-537), Neutron Absorbers and Methods of Forming at Least a Portion of a Neutron Absorber, filed May 24, 2011. Inventors: Donna Guillen, Doug Porter, Dave Swank, and Arnold Erickson.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
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Idaho National Laboratory Donna Post Guillen (principal investigator)	
Center for Advanced Energy Studies Yaqiao Wu, Jatu Burns, Bryan Forsmann, Kerry Allahar (collaborators)	
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Advanced Damage-Tolerant Ceramics: Candidates for Nuclear Structural Applications

Introduction

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

MAX phases, which are a new class of machinable, layered ternary carbides and nitrides, are excellent candidate materials for these demanding environments, either as fuel matrices or coating materials. They show excellent potential for significantly improving material performance thanks to their superior metallic characteristics for mechanical- and thermal-related designs, including:

- High-temperature capabilities (up to 1300° C)
- High damage tolerance
- Greater chemical resistance
- Ability to be manufactured in a variety of methods from slip casting to metal-injection molding.

All MAX phases are fully machinable, even though some of them, such as Ti_3SiC_2 and Ti_3AlC_2, are similar to titanium in density but are three times as stiff. Their thermal and electrical conductivities are high and metal-like, they have high fracture toughness, some are chemically stable in corrosive environments, and in heavy ion studies, they have shown resistance to irradiation damage.

Project Description

The intent of this ATR NSUF project is to investigate the damage made to Ti_3SiC_2, Ti_3AlC_2, and chemical vapor deposited silicon carbide (CVD SiC) (for comparison) by exposure to a spectrum of neutron irradiation consistent with conditions found in light water nuclear reactors. The carbides were exposed to a series of neutron fluence levels at moderate to high irradiation temperatures (see Table 1) in ATR at INL. The 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 as they are irradiated by neutrons.

Accomplishments

Begun in 2009, this project is a collaborative effort among ATR NSUF, Drexel University, and Savannah River National Laboratory (SRNL). As noted above, the team seeks to characterize the effect of neutron irradiation of select MAX phases to evaluate their use in nuclear reactor applications. Table 2 lists the dosages and temperatures reached for each sample capsule. The last of the capsules completed irradiation in November 2011.

Table 2. Irradiation Dosages and Temperatures, MAX Phase Samples.

INL Capsule ID	Phases	Dose, dpa (E > 1 MeV)	Temp, °C
Drex-Rabbit	Ti_3SiC_2, Ti_3AlC_2, and SiC	0.1	100, 650, 1000
Drex-B	Ti_3SiC_2, Ti_3AlC_2, and SiC	1	100, 650, 1000
Drex-A	Ti_3SiC_2, Ti_3AlC_2, and SiC	10	100, 650, 1000

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During the project's second year, all samples were shipped to the Hot Fuel Examination Facility (HFEF) at INL's MFC. Unfortunately, there were extensive delays at MFC throughout 2012 due to a facility-wide stand-down where operations were suspended for several months. There were also several work stoppages at INL beyond the experimenters' control due to conflicts with unrelated

Table 1. Test matrix for sample irradiation.

	Temperature (°C)	Target Dose* (dpa)	Specimen types
Ti_3SiC_2	100, 650, 1000	0.1, 1, 10	TEM, resistivity and tensile
Ti_3AlC_2			TEM, resistivity and tensile
SiC (CVD)			TEM, resistivity

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

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"We are very excited for PIE to begin in the coming months, as the samples await capsule opening and cataloguing."

Darin Tallman, Research Assistant, Drexel University

The MAX phases show great promise for use in the next generation of nuclear reactors.

experiments. As a result, the ATR NSUF project has been significantly delayed. At this time, budget and schedule constraints are pushing the actual PIE work out to 2013.

Future Activities

The capsules are currently stored in the HFEF main cell awaiting cataloging. In May 2013, the first samples are scheduled to be cataloged and shipped to the MFC Electron Microscopy Laboratory (EML) where they will undergo cleaning and sample preparation. PIE analysis of the samples will follow, with characterization of microstructures via transmission electron microscopy (TEM), tensile samples of each composition tested at both room and irradiation temperatures, and resistivity measurements of irradiated materials taken.

Publications and Presentations

Darin Tallman, Elizabeth Hoffman, Robert Sindelar, Gordon Kohse, Michel Barsoum, 2012, "Effect of Neutron Irradiation on Select MAX Phases," *The Metals, Minerals, and Materials Society 2012 Annual Meeting, Orlando, Florida, March 11-15, 2012.*

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Team Members/Collaborators	
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Microstructural Study of Ion-Irradiated 14LMT Nanostructured Ferritic Alloys

Introduction

Nanostructured ferritic steels (NFS) show great potential for tolerance to radiation damage due to their promising high-temperature mechanical properties and microstructural stability. These properties primarily derive from the presence of a high number density of nanometric yttrium-titanium-oxygen (Y-Ti-O) precipitates that are thought to form during hot consolidation following mechanical alloying. These types of nano-features are capable of serving efficiently as dislocation pinning sites, point defect sinks, and helium trapping sites.

Traditionally, yttrium oxide (Y_2O_3), a.k.a. yttria, has been the only rare earth oxide used in NFS. However, a 1990 study, "Endo et al"[1] noted that the addition of a new rare earth oxide, lanthanum oxide (La_2O_3), to molybdenum-based oxide dispersion strengthened (ODS) alloys led to better creep resistance than that of Y_2O_3 and cerium oxide (Ce_2O_3).

If successful, this research will help in the development of longer lasting, high performance fuel cladding and matrix materials for advanced nuclear reactors.

Project Description

The goal of this ATR NSUF rapid turnaround experiment (RTE) project is to characterize the role of alloying elements in a unique NFS by adding (La_2O_3), a.k.a. lanthana. One reason for choosing lanthana is its relative abundance (25-38 wt%) in the United States compared to yttria (~0.2 wt%).

In addition, a new sintering method called pulsed electric current sintering (PECS) was applied in place of hot isostatic pressing. Commercially referred to as spark plasma sintering, this method is conventionally used to produce bulk NFS. However, the reduced sintering temperature and time required by PECS allows us to retain the nanostructured material. In addition, the texture and anisotropy of the produced alloys are reduced, along with associated fabrication costs.

The RTE project was carried out in conjunction with another project funded by the Laboratory Directed Research & Development (LDRD) program of INL. In this combined project, an alloy composition similar to commercial MA957 (Fe-14Cr-1Ti-01.3Mo-0.3Y_2O_3) was designed with La_2O_3. The composition developed (Fe-14Cr-1Ti-0.3Mo-0.5La_2O_3) was named 14LMT.

The milling parameters, such as milling time, ball-to-powder ratio, and ball size were optimized to obtain a solid solution with the smallest crystallite and particle sizes. In addition, different powder compositions (Fe-14Cr, Fe-14Cr-0.5 La_2O_3, and Fe-14Cr-1Ti-0.3Mo-0.7 La_2O_3) were ball-milled and consolidated by PECS in order to study the effects of different alloying elements. Originally, the project planned to carry out characterization of ion-irradiated materials. However, due to the limited time and funding available on an RTE project, attention was focused on the detailed characterization of the processed alloys.

Accomplishments

A) Microstructural Studies of 14LMT Powder

Due to the small size of the individual powder particles, it is difficult to prepare specimens for viewing the microstructure. General metallographic procedures do not work because high-energy ball-milling is expected to create nanocrystalline particles, which are not resolvable by standard optical microscopy.

Using focused ion beam scanning electron microscopy (FIB-SEM), 14LMT powder specimens were prepared for transmission electron microscopy (TEM) and local electrode atom probe (LEAP) studies in the Microscopy and Characterization Suite (MaCS) at CAES. In the TEM micrograph of 14LMT powder shown in Figure 1a, the microstructure has an average grain diameter of ~25 nm. Figure 1b shows the ring diffraction pattern of a randomly-oriented nanocrystalline Fe-Cr matrix. However, with the help of high-resolution TEM imaging, along with energy dispersive X-ray spectroscopy (EDS) analysis and LEAP

Figure 1. (a) A bright field TEM micrograph of 10-hour ball-milled 14LMT powder, (b) a diffraction pattern of ball-milled 14LMT powder, and (c) an HRTEM image of nanoscale features.

"Working with awesome CAES MaCS people and state-of-the-art instruments has really helped me a lot in my Ph.D. research. I have learned valuable skills for my future research career. Thank you all!"

Somayeh Pasebani, Ph.D. Student, University of Idaho

studies, it was found that the powder is not a true solid solution (Figure 1c), but contains clusters of various solute types (CrO, TiO, and La-TiO-O). Therefore, previous conjecture that nanoclusters only form in the mechanically alloyed solid solution during hot consolidation of mechanically alloyed powder is not entirely correct.

Few significantly different contrasts are observed in Figure 2a, implying a reasonably homogeneous distribution of solute elements. However, based on EDS results, some features with brighter contrast are enriched with more Fe. Figure 2b shows the concentration in atom% along a 100 nm EDS line scan. Some regions within the line show significantly increased Cr concentration, while Ti presents a marked decrease, suggesting the presence of CrO. Some regions show the opposite behavior, indicating the presence of TiO, while other regions show both Ti and Cr present in higher amounts.

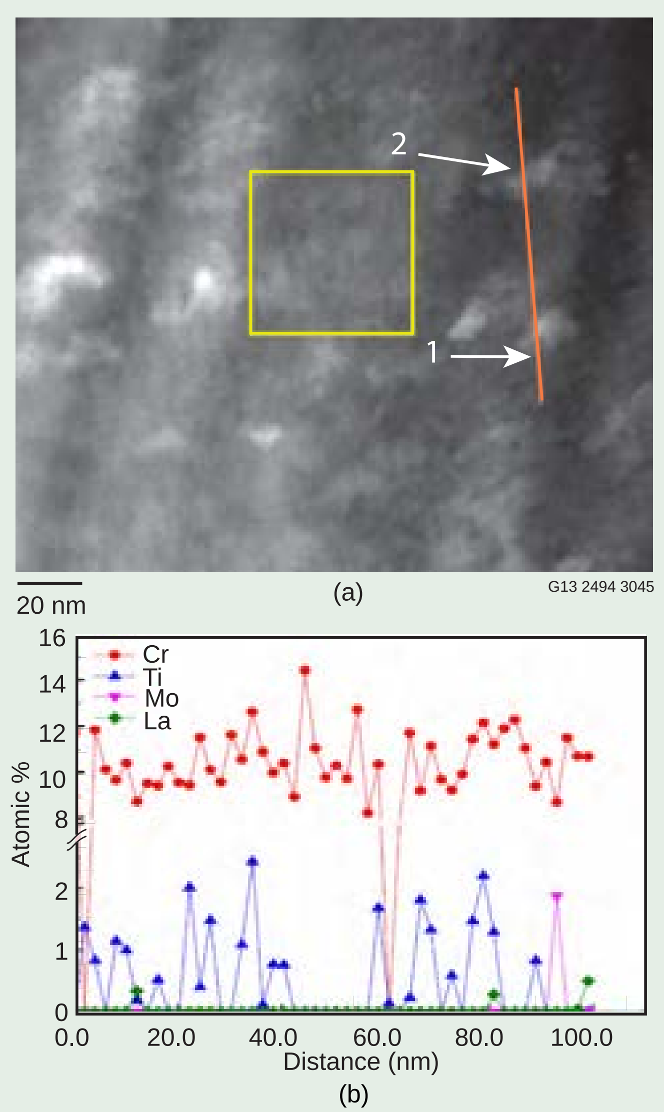

Figure 2. (a) A STEM image of 10-hour ball-milled 14LMT powder, and (b) an EDS line scan concentration profile of Cr, Ti, Mo, and La.

Also in Figure 2b, the concentration of La increases along a 2 nm scan (contrast feature #1 in Figure 2a), whereas those of Cr and Ti decrease. This indicates the presence of a La-containing oxide nanofeature with more Cr and Ti. As shown in contrast feature #2 in Figure 2a, the concentrations of Cr, Ti, and La increase from the matrix to the nanofeature, which makes it plausible that the addition of Ti changes the chemical equilibrium from the formation of CrO's towards that of TiO's because of Ti's greater affinity for O [17].

The atom maps and 1D concentration profile in Figures 3a and b show that Ti is found more as TiO due to its high oxygen affinity. In addition, nanoclusters with diameters of 2 nm – 5 nm found in the as-milled powder, with complex interfaces, are enriched with O, La, and TiO, and are expected to have significantly increased the hardness of the as-milled powder.

It is likely that a high concentration of lattice vacancies is produced during the milling process, and Ti and La

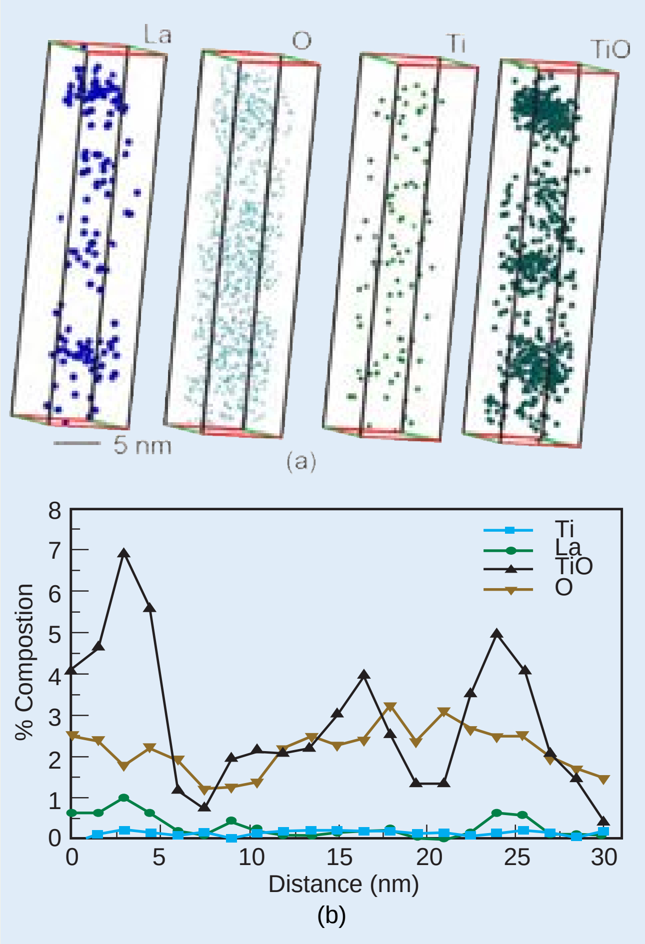

Figure 3. (a) Atom probe maps of La, O, Ti, and TiO in 10-hour ball-milled 14LMT power, and (b) 1D concentration (atom%) profile along the direction of analysis.

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Microstructural Study of Ion-Irradiated 14LMT Nanostructured Ferritic Alloy (cont.)

alloying elements in the ferritic matrix bind strongly with oxygen-vacancy complexes. Since both Ti and La can form strong bonds with these complexes, free energy of formation for La-, Ti-, and O-rich nanoclusters can become lower than that of the stable oxide phases.

B) Microscopy of PECS 14LMT Material

Bulk samples of 14LMT alloy were made by consolidating the mechanically alloyed powder via PECS at 900º C for seven minutes. The focused ion beam (FIB) can be used to prepare TEM samples in a similar way, but it takes longer and is more expensive. In addition, jet polishing can provide high quality TEM samples of metals and alloys quickly, and saves FIB-SEM instrument time. During this project, the Fischione jet polisher was utilized for the first time, and a new procedure developed by a staff member was approved by the CAES MaCS leadership team. This is likely to generate huge savings in instrument time and costs for the FIB-SEM, which can now be used entirely for preparing LEAP specimens.

Some interesting observations were made in the alloy that was produced. Bright field TEM imaging of the microstructure revealed a bi-modal grain size distribution: some regions had nanocrystalline grain sizes, along with some micron-sized grains (Figure 4a). This is due to an inhomogeneous distribution of the alloying element, possibly as a result of ball-milling. Micron- and submicron-sized grains are observed, along with nano-sized grains. A high number density of nano-precipitates is found along the grain boundaries and also inside the grains. Figure 4b shows the ring diffraction pattern of randomly-oriented grains and precipitates that need to be identified and indexed.

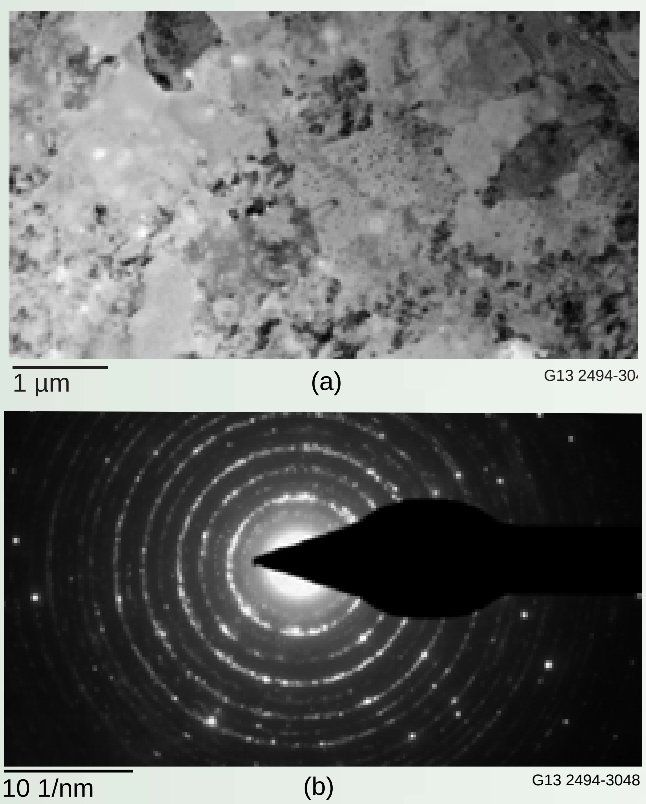

Figure 4. (a) A bright field image from the microstructure of PECS 14LMT showing a bi-modal grain size distribution, and (b) its diffraction pattern showing randomly distributed nanocrystalline and spot patterns related to CrO and La-Ti-O.

In Figure 5a, nano-precipitates of different sizes are shown, some of them pinning dislocations, while 5b is a high resolution TEM (HRTEM) image of ~2-nm precipitates that are a different phase from the matrix.

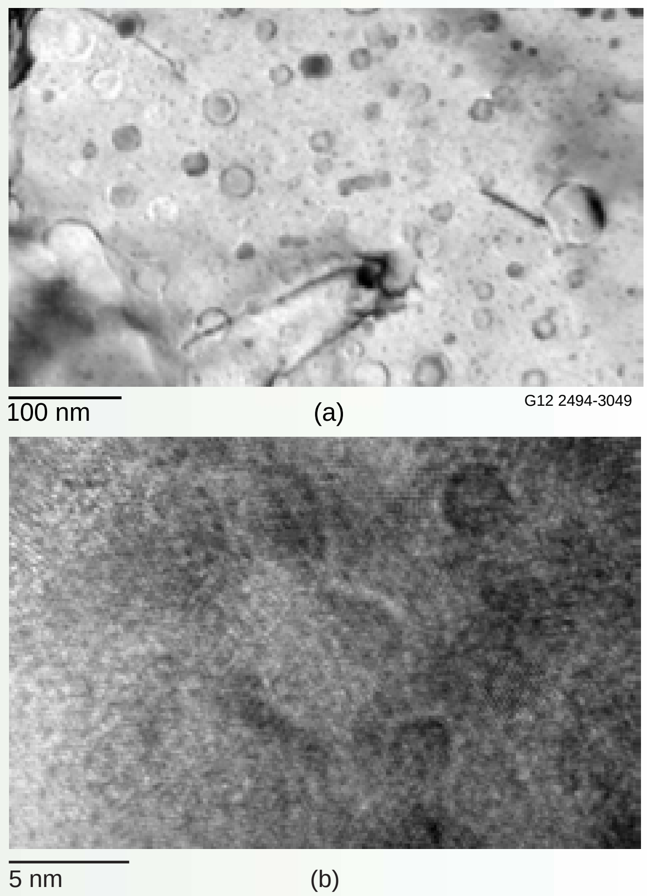

Figure 5. (a) Oxide precipitates with varying sizes pinning dislocations, and (b) HRTEM image showing precipitates smaller than 5 nm.

In Figure 6a, contrast features (darker than matrix contrast) that are different from the matrix phase can be observed. The size of each contrast feature is different, and an EDS scan line conducted along two ~15 nm precipitates shows they are composed of Cr-La-Ti-O.

The atom maps in Figure 7a represent a very small volume of the PECS sample (20x20x70 nm). Cr and Mo are evenly distributed in the volume, however, LaO, TiO, and

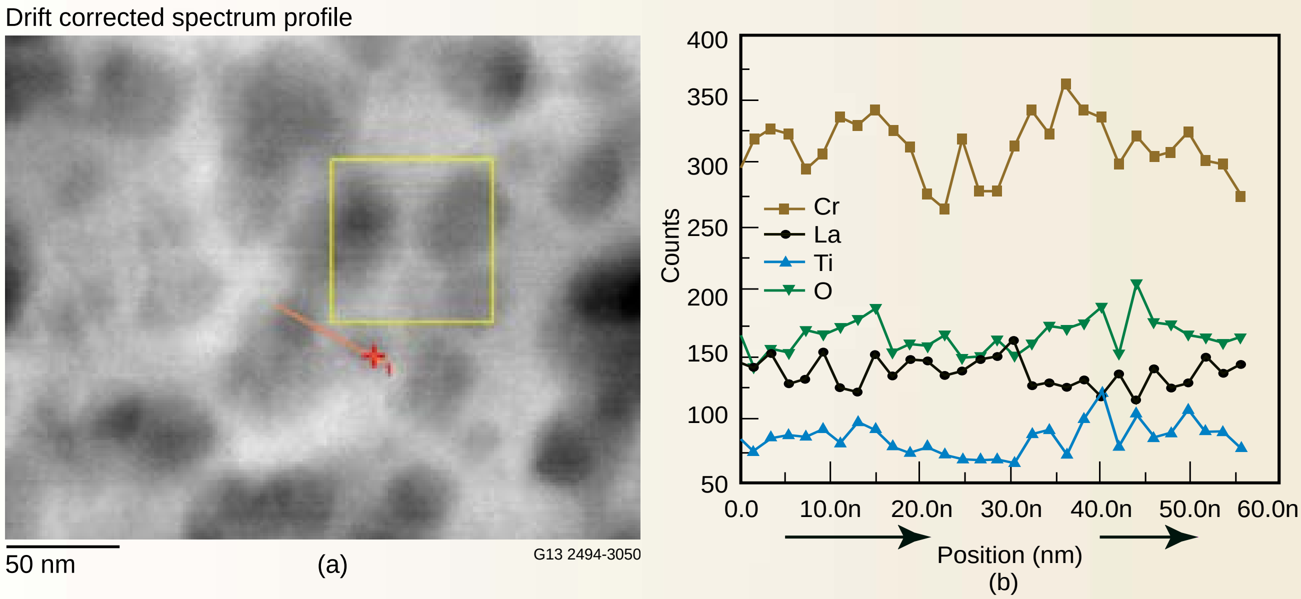

Figure 6. (a) STEM image and EDS line scan between two precipitates as small as 20 nm, and (b) EDS line scan concentration profile.

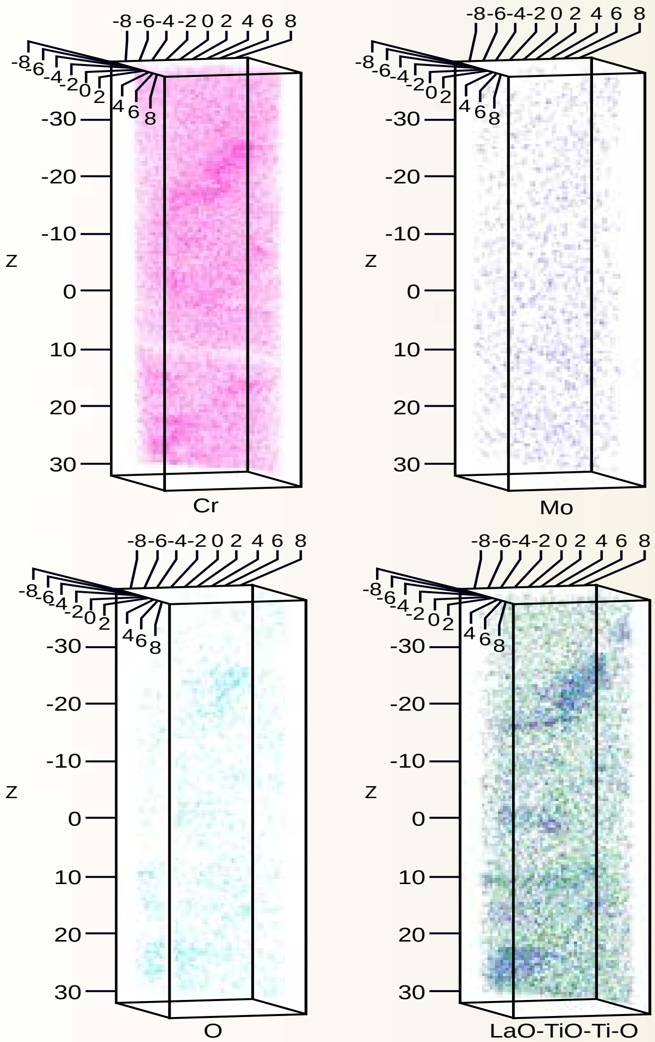

Figure 7a. 3D reconstruction of APT data showing distribution of solutes (Cr and Mo) and small precipitates of LaO-TiO-O.

Microstructural Study of Ion-Irradiated 14LMT Nanostructured Ferritic Alloy (cont.)

Principal Investigator: Indrajit Charit – University of Idaho (cont.)
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O form very small clusters (~2 nm – 5 nm in diameter). No clusters of La or Ti were found, while LaO and TiO were mainly clustered.

Figure 7b shows the atomic percentage of La and Ti in La-Ti-O clusters. For example, in one cluster, the ratio of Ti to La was ~0.5, and the ratio of La+Ti to O was ~0.6.

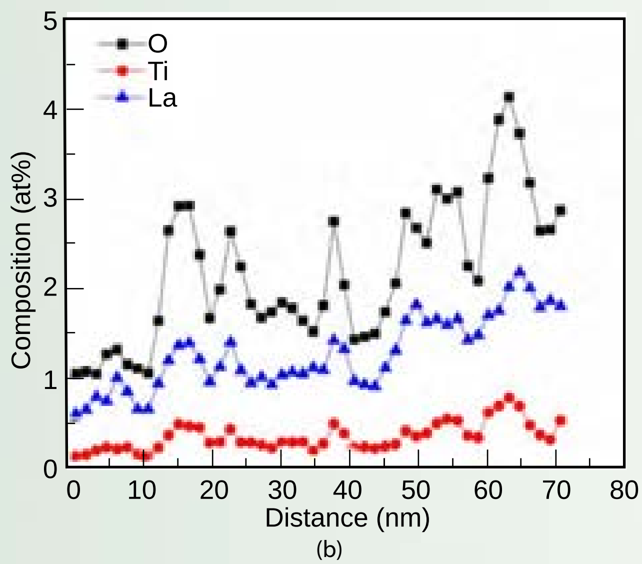

Figure 7b. 1D concentration profile along z axis showing several clusters with varying amounts of La, Ti, and O.

Future Activities

Detailed TEM studies, including collecting spot patterns from the precipitates at different zone axes, are scheduled for 2013. The patterns will be indexed in order to identify their crystal structure. It is anticipated that LEAP data analysis will also be completed for the different PECS temperatures and chemical-composition samples.

Preparing mini-tensile specimens and performing tensile tests at both room and high temperatures will complete the mechanical testing of the samples. The effects of annealing and thermal stability on microstructural characteristic and mechanical properties will also be investigated. In addition, tests will be conducted on the thermal stability of nano-precipitates in newly developed NFS. These results will be compared with NFS containing yttria.

Publications and Presentations

1. Somayeh Pasebani, Indrajit Charit, Kerry Allahar, James Cole, and Darryl P. Butt, 2012, "Oxide Dispersion Strengthened Steels via Mechanical Alloying and Spark Plasma Sintering," *Materials and Fuels for the Current and Advanced Reactors Symposium*, *TMS Annual Meeting, Orlando, Florida, March 11-15, 2012.*

2. Somayeh Pasebani, Indrajit Charit, Darryl P. Butt, and James Cole, 2012, "A Preliminary Study on the Development of La203-Bearing Nanostructured Ferritic Steels via High-Energy Ball Milling," *Journal of Nuclear Materials*, Vol. 434, No. 1-3, pp. 282-286.

References

1. Motomu Endo, Kuninari Kimura, Teiichi Udagawa, Shigemitsu Tanabe, and Seto Hiroyuki, 1990, "Effects of Doping Molybdenum Wire with Rare Earth Elements," *High Temperatures – High Pressures*, Vol. 22, pp. 129-137.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Team Members/Collaborators	
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Principal Investigator: Lingfeng He – University of Wisconsin
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Study of the Microstructure of Irradiated Cerium Dioxide by Advanced Microscopy Techniques

Understanding the behavior of microstructures is necessary to the understanding of the properties of these materials.

Introduction

Uranium oxide (UO_2), a ceramic compound with a fluorite structure, is the most widely used nuclear fuel in commercial light water reactors. Over the past few decades, a number of studies have focused on radiation damage in UO_2 fuel, resulting in large empirical databases of various properties and behaviors, such as volumetric swelling and fission gas release. Nevertheless, these studies still lack a fundamental understanding of defect generation and evolution. Therefore, studying the evolution of microstructures under irradiation combined with theoretical modeling and simulation becomes crucial to advancing the current understanding of radiation damage in UO_2.

Project Description

Initiated in 2011, this rapid turnaround experiment is a collaborative effort of INL's ATR NSUF, Argonne National Laboratory, and the University of Wisconsin.

In this work, the research team used cerium dioxide (CeO_2) as the surrogate material to study the microstructure evolution of UO_2 under ion irradiation. CeO_2 has the same crystal structure and lattice parameter as UO_2, but it does not become radioactive after ion irradiation. Due to the similarities between the structures and physical properties of CeO_2 and UO_2, the microstructure responses to ion irradiation of CeO_2 can be effectively compared and contrasted to those of UO_2. In addition, the microstructure development of CeO_2 under ion irradiation can be used to verify the theoretical modeling of ion irradiation-driven defect and microstructure change, as well as thermal transport, in irradiated fuel oxide.

Accomplishments

To simulate fission fragment damage to CeO_2, 150 keV krypton (Kr) and 1 MeV Kr were used to irradiate polycrystalline CeO_2 bulk samples having various grain sizes. To date, the research team has used advanced transmission electron microscopy (TEM) to study the defects—including bubble, dislocation loops, and dislocation segments—and their interactions with grain boundaries in Kr-irradiated CeO_2 (Figure 2).

It has been shown that Kr bubbles exhibit a smaller size and lower density in nanograin regions than in micrograin regions, indicating grain boundaries are efficient sinks for defects (Figure 3).

Future Activities

The research team will study the effects of grain boundary types (twin, low-angle and high-angle) and grain boundary chemistry on defect evolution in polycrystalline UO_2 irradiated by Kr ions. The 300 kV Tecnai TF30

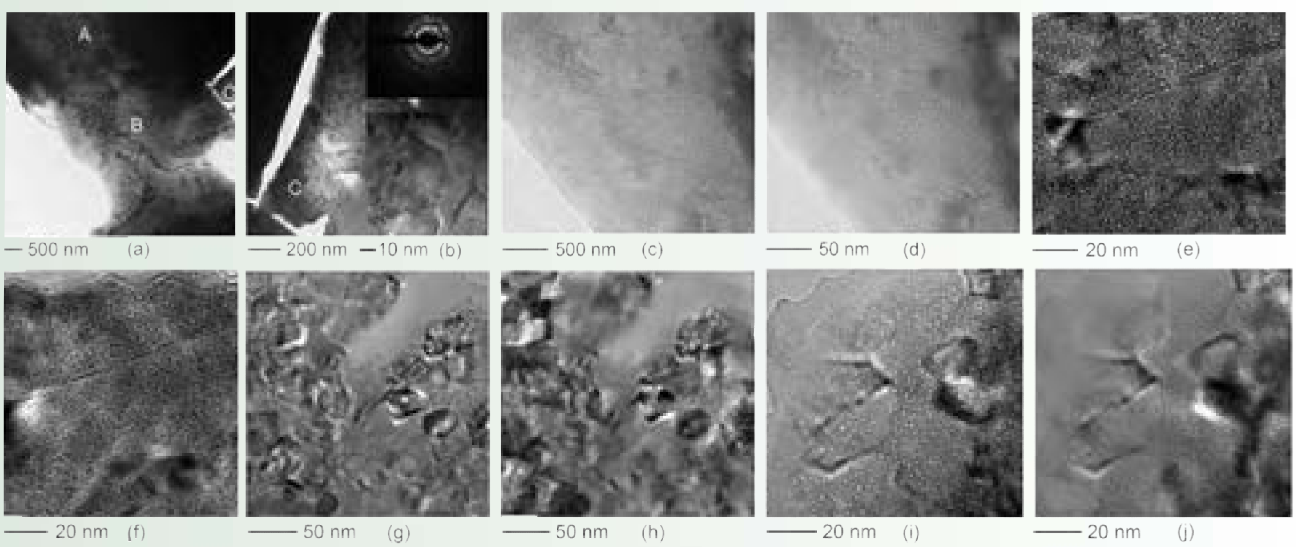

Figure 1. Microstructure of polycrystalline CeO_2 irradiated with 150 keV Kr at 600°C up to a dose of $1{\times}10^{16}$ ions/cm^2. (a) Micrograins [(A and B)] coexist with nanograins (C). (b) Enlarged nanograin region of C (insets show the diffraction pattern and high-magnification TEM image for nanograins). [(c) and (d)] Under-focused and over-focused images, respectively, of the CeO_2 micrograin region. [(e) and (f)] High-magnification, under-focused and over-focused images, respectively, of the CeO_2 micrograin region. [(g) and (h)] Under-focused and over-focused images, respectively, of the CeO_2 nanograin region. [(i) and (j)] High-magnification, under-focused and over-focused images, respectively, of the CeO_2 nanograin region.

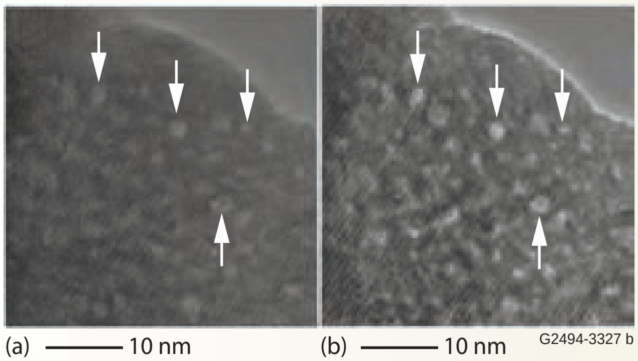

Figure 2. (a) High-resolution TEM image of a CeO_2 grain irradiated with 150 keV Kr at 600°C up to a dose of 1×10^{16} ions/cm^2 when the electron beam is parallel to the [110] zone axis, and (b) Under-focused image of the same. Some bubbles are marked by arrows.

field emission gun (FEG) TEM in the Microscopy and Characterization Suite (MaCS) at CAES will be used to view the microstructure of polycrystalline UO_2 before and after the Kr irradiation. Dislocation loops and either dislocation segments or dislocation networks, whichever is present, will be examined, and the Burgers vector will be analyzed. Cavities or bubbles in the grains and at the grain boundaries will also be investigated.

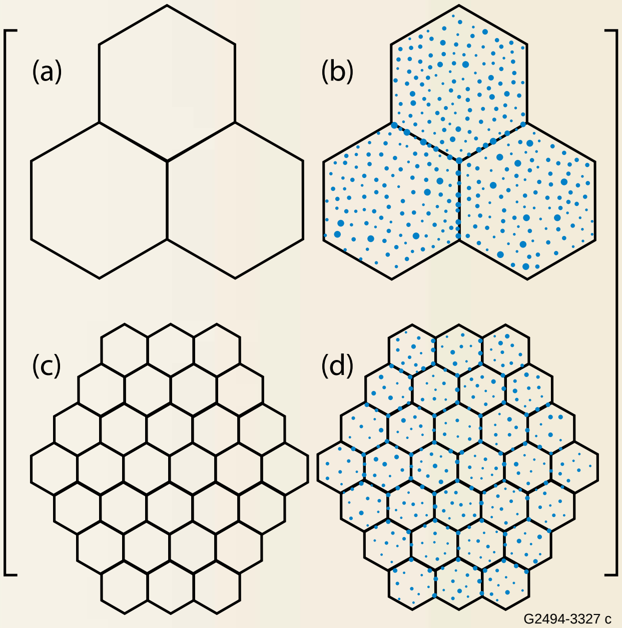

Figure 3. Schematic diagram of the microstructure of bubbles at micrograin [(a) and (b)] and nanograin regions [(c) and (d)] in polycrystalline CeO_2 before implantation [(a) and (c)], and during implantation [(b) and (d)]. For simplicity, the spherical bubbles are used instead of lenticular bubbles at the grain boundaries.

Publications and Presentations

1. Lingfeng He, Clarissa Yablinsky, Mahima Gupta, Todd Allen, Jian Gan, 2012, "Irradiation Damage of CeO_2 with Xe and Kr Implantation," *The Minerals and Materials Society 141st Annual Meeting & Exhibition, Orlando, Florida, March 11-15, 2012.*
2. Lingfeng He, Clarissa Yablinsky, Mahima Gupta, Marquis Kirk, Todd Allen, "TEM Investigation of Kr Bubbles in Polycrystalline CeO_2," *Nuclear Technology*, (in press).

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Team Members/Collaborators	
University of Wisconsin Lingfeng He (principal investigator), Mahima Gupta (Ph.D. candidate), Clarissa Yablinksy (post-doctoral student)	
Idaho National Laboratory Jian Gan (co-principal investigator)	
Argonne National Laboratory Marquis Kirk (collaborator)	

Principal Investigator: Brent Heuser – University of Illinois
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Effect of Heavy Ion Bombardment on Uranium Dioxide Thin Films Under Various Conditions of Irradiation Dose, Temperature, and Actinide Surrogates

Introduction

Begun in 2012, this ATR NSUF project is a collaborative effort among INL, the University of Illinois, and the Illinois Institute of Technology. The research team hopes to develop methods that will provide experimental data that can assist modelers in predicting the effects of radiation damage in nuclear fuels while avoiding the complications inherent in working with highly radioactive neutron-irradiated fuels.

Project Description

The goal of this project is to characterize the geometric structures of thin-film samples of both doped and nondoped uranium dioxide (UO_2), as well as UO_2 having a uniform distribution of the plutonium/actinide surrogate material, neodymium (Nd).

Researchers used X-ray absorption spectroscopy (XAS) at the Advanced Photon Source (APS) to observe changes in UO_2's local atomic structure under irradiation. In 2012, the team measured the changes in local atomic structure of nondoped UO_2 as a function of irradiation dose. This experiment was performed to help researchers understand how UO_2 behaves in Generation IV reactors.

Accomplishments

Single-crystal UO_2 thin films were grown at the University of Illinois. The thin films of UO_{2+x}, where x varies from 0.0 to 0.66, were prepared using magnetron sputtering. For this work, the films were deposited on titanium dioxide (TiO_2) substrates and then bombarded with a 1.8 MeV beam of krypton (Kr^+) ions with doses ranging from $5x10^{14}$ to $2x10^{16}$ ions/cm^2 (a unirradiated thin film was included as a control sample). This dose range is equivalent to the fission per initial metal atom (FIMA) range of 0.1% to 10.0% and represents the target burnup values in the next generation of reactors.

This project is developing methods that university researchers can use to study radiation damage in fuel samples without the complication of high activity.

Six samples of $UO_{2+0.0}$ (unirradiated and doses of 5E14, 1E15, 5E15, 1E16, and 2E16 ions/cm^2) were sent to the Materials Research Collaborative Access Team (MRCAT) beamline (Figure 1) in the APS at the Argonne National Laboratory. Using XAS on the U-L_3-edge (17166 eV), researchers measured changes in the local atomic structure around the uranium atoms. All samples were measured in fluorescence geometry using a four-channel Vortex detector.

Figure 2a shows the X-ray absorption near-edge spectroscopy (XANES) region, and Figure 2b shows the magnitude of the transformation of the extended X-ray absorption fine structure (EXAFS) data from two samples

Figure 1. Mohamed ElBakhshwan (University of Illinois) and Tim McNamee (Illinois Institute of Technology) measure X-ray absorption spectra from UO_2 samples at the MRCAT beamline.

“I enjoyed working on this project. I had worked on uranium speciation in the environment before, but it was nice to participate in an experiment in a different area of the nuclear fuel cycle."

Dr. Bhoopesh Mishra, Research Assistant Professor of Physics, Illinois Institute of Technology

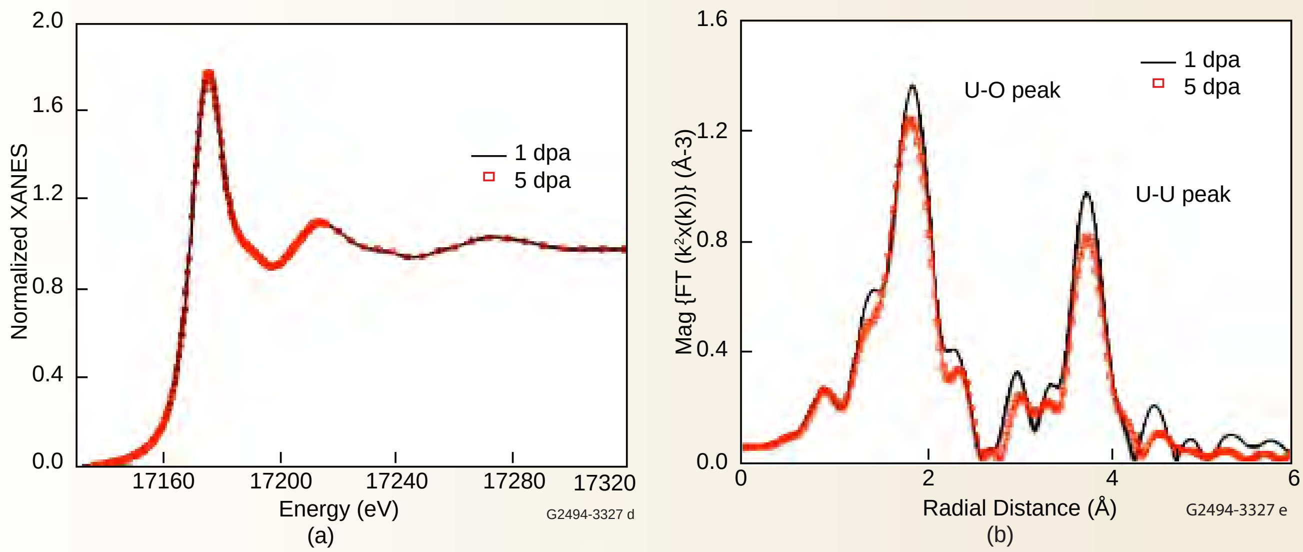

Figure 2. (a) UL3 edge XANES, and (b) Fourier transform magnitude of EXAFS data for UO_2 thin-film samples and radiation doses of 1 dpa and 5 dpa.

irradiated to 1 displacement per atom (dpa) and 5 dpa, respectively. These regions are sensitive to changes in the chemical state and geometric structure around their respective absorbing atoms.

The UO_2 samples were highly tolerant to incident ion radiation. The XANES region of the spectrum did not indicate any change in the chemical state when irradiated. However, the EXAFS region did show a loss of coordination around the uranium atoms in the UO_2 thin film after irradiation. Figure 2b shows the reduced intensity in the peaks attributed to the U-O interatomic distance and the U-U interatomic distance as the ion irradiation dose increased. The strong peaks indicate that much of the crystallinity in the film was retained.

Researchers are continuing this modeling in order to better interpret the observed changes in defect formations in the UO_2 lattice. Developing an understanding of this damage will assist in the second part of the experiment, which will examine damage around a simulated fission product (Nd).

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Illinois Institute of Technology	Materials Research Collaborative Access Team at the Advanced Photon Source
Team Members/Collaborators	
University of Illinois Brent Heuser (principal investigator), Mohamed S. ElBakhshwan (graduate student) **Illinois Institute of Technology** Jeff Terry (co-principal investigator), Bhoopesh Mishra (collaborator), Hasitha Ganegoda, Dan Olive, Tim McNamee (graduate students)	

Future Activities

The next goal of this work will be to repeat the measurements on samples doped with Nd. Under heavy ion bombardment, the Nd can provide a model for fission fragmentation of these materials in the next generation of reactor environments.

Principal Investigator: Peter Hosemann – University of California, Berkeley
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Characterization of Reactor-Irradiated Oxide Dispersion Strengthened Materials Using Local Electrode Atom Probe Tomography

Introduction

In order to build more efficient advanced nuclear reactors with higher burnup rates, new materials must be developed with which to construct them. Oxide dispersion strengthened (ODS) ferritic steels are attractive candidates for this type of construction because of their reduced activation, excellent high-temperature creep strength, and resistance to potential swelling and helium embrittlement. These properties are due in part to the fine dispersion of titanium-yttrium-oxygen (Ti-Y-O) enriched particles throughout the material. However, the exact nature of these particles is not completely understood. Their small size (<1 nm) and heterogeneity make analysis of their structures and compositions difficult.

Project Description

Using atom probe tomography (APT), this rapid turnaround experiment will investigate the oxide particle morphology and chromium (Cr) cluster formation in samples of the ODS steel MA957, which was irradiated in the Fast Flux Test Facility-Materials Open Test Assembly (FFTF-MOTA) as part of the Liquid Metal Fast Breeder Program (LMFBP) in the 1980s. APT's high, element-specific resolution, along with its ability to render atomistic data in three dimensions, make it a useful tool in the characterization of these oxide particles.

Among the components that bear the brunt of radiological and thermal effects created by advanced nuclear reactors are cladding materials. As part of ongoing irradiation experiments in support of the LMFBR program, the 14 Cr ODS alloy MA957 was irradiated in FFTF-MOTA as pressurized creep tubes to doses up to 121

The characterization of ODS steels using APT will shed light on the efficacy of a class of cladding materials suggested for use in advanced reactor systems.

displacements per atom (dpa) at temperatures ranging from ~400º C to 750º C. Selected specimens, shown in Table 1, were recently recovered from long-term storage for analysis.

Table 1. Samples investigated using APT.

Temperature (°C)	DPA	Stress (MPa)	Number of APT Specimens
750	121	15	3
670	110	60	5
670	110	0	3
550	113	60	4
412	109	60	7

Accomplishments

Using a warm FEI Quanta 3G focused ion beam (FIB) at the University of California, Berkeley (UCB), the samples were cut and sharpened into APT needles measuring approximately 50 nm in diameter and 200 nm in length. APT data was collected at CAES in Idaho Falls, Idaho, and analyzed using the local electrode atom probe (LEAP). It was found that the sizes and distribution of the particles are neither significantly affected by irradiation, nor does the particle chemistry vary with respect to particle size.

However, the irradiation temperature does appear to slightly affect the distribution of the particles. At lower temperatures, the particle number density appears to increase. Planar concentrations of alloying elements and trace elements were observed, and Cr-rich clusters, presumably alpha, can be seen, as shown in Figure 1.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
University of California, Berkeley	PIE facilities

Team Members/Collaborators

University of California, Berkeley
Peter Hosemann (principal investigator), Nathan Bailey (collaborator)

Pacific Northwest National Laboratory
Mychailo Toloczko (collaborator)

Center for Advanced Energy Studies
Erich Stergar (collaborator)

Future Activities

In order to build a more complete sample set of irradiation conditions, temperatures, and creep stresses, project researchers plan to study

“Any (nuclear) engineering design is only as good as the materials from which it is made.”
Dr. Peter Hosemann, Professor of Nuclear Engineering, University of California, Berkeley

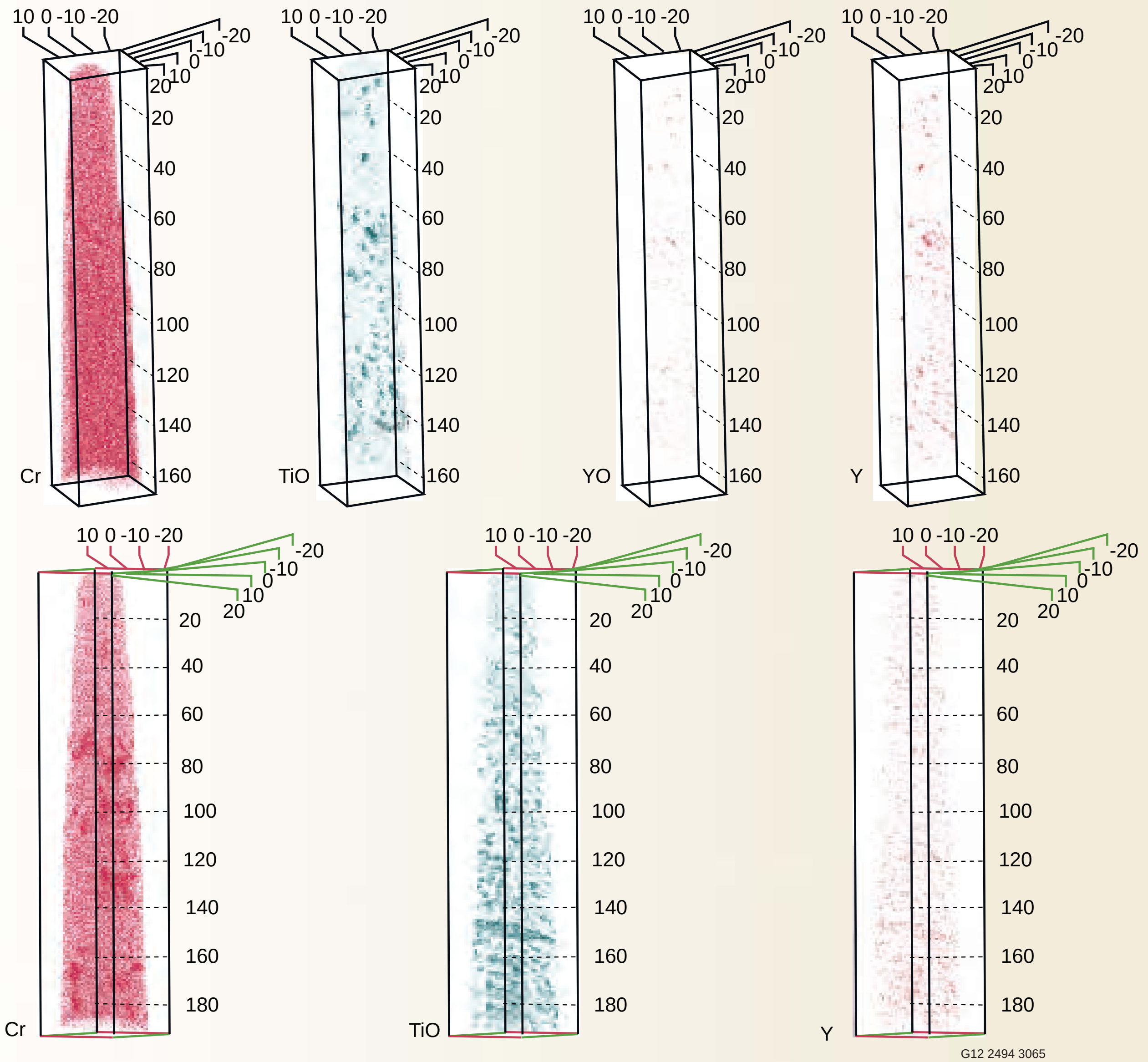

Figure 1. Top: APT result of the sample irradiated to 670º C, 110 dpa. A large number of Ti-Y-O clusters are found. Bottom: APT results of the sample irradiated to 412º C, 109 dpa. A large number of Ti-Y-O clusters are found while larger Cr-rich clusters are visible.

more irradiated samples of ODS steels utilizing APT at the CAES facility. These materials will be characterized using transmission electron microscopy (TEM) in an effort to create a three-dimensional reconstruction of an atom probe tip. The tip would then be run in the LEAP to guide reconstruction of the data, hopefully resulting in a better understanding of how an atom probe can be used to investigate these types of systems, and to provide correlative evidence for the trends suggested by an atom probe in the performance of these materials.

Publications and Presentations

1. Nathan Bailey, Erich Stergar, Mychailo Toloczke, Peter Hosemann, 2012, “Initial APT Analysis of Irradiated MA957,” *Microscopy and Microanalysis*, Vol. 18, July 2012, pp. 1418-1419.

2. Nathan Bailey, Erich Stergar, Mychailo Toloczke, Peter Hosemann, 2012, “Initial APT Analysis of Irradiated MA957,” *Transactions of the American Nuclear Society*, 106, in press, 2012.

Measurement of Actinide Neutronic Transmutation Rates with Accelerator Mass Spectroscopy (MANTRA)

Principal Investigator: George Imel – Idaho State University
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Introduction

Recent studies devoted to Generation IV reactor systems have pointed out the need for more accurate data. Since this experiment began in 2009, the very high mass actinides have been shown to play a significant role in the feasibility assessment of innovative fuel cycles. As an example, the potential build-up of Cf-252 when recycling transuranic (TRU) waste in light water reactors (LWR) can lead to increased neutron emissions that could impact the fuel fabrication process. As a consequence, the nuclear data on higher mass TRUs should be significantly improved.

Project Description

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

Three sets of actinide samples will be irradiated in order to obtain effective neutron capture cross-sections corresponding to neutron spectra ranging from fast to epithermal. The first set will be filtered with cadmium, while the other two will be filtered with boron at thicknesses of 5 mm and 10 mm. Atom density tests will be carried out before and after irradiation at INL using inductively coupled plasma mass spectrometry (ICPMS) and/or the thermal ionization mass spectrometer (TIMS) in the Analytical Lab at INL's MFC. Similar tests will be conducted at Argonne National Laboratory (ANL) using accelerator mass spectrometry (AMS) in the Argonne Tandem Linac Accelerator System (ATLAS).

Accomplishments

Work done in 2012 primarily focused on preparing the actinide and fission product samples for irradiation in ATR. As the equation (top right) demonstrates, the energy-integrated neutron capture cross-section of a nuclide of mass number A is inferred by subtracting the initial amount of nuclide of mass number A+1 from the final amount of that same nuclide. It is therefore important to minimize the initial amount of nuclide A+1 in the sample in order to decrease the uncertainty of the inferred cross-section as much as possible.

If successful, this project will generate data on minor actinide reaction rates that are presently lacking with acceptable uncertainties.

$$\sigma_A^C \sim \frac{\frac{N_{A+1}(t_f)}{N_A(t_f)} - \frac{N_{A+1}(t_i)}{N_A(t_i)}}{(t_f - t_i)\Phi} \equiv \frac{\frac{N_{A+1}(t_f)}{N_A(t_f)} - \frac{N_{A+1}(t_i)}{N_A(t_i)}}{\tau}$$

When it was deemed necessary, and when possible, the available samples were re-purified to lower the level of initial impurity of mass number A+1, A+2, etc. Figure 1 illustrates the last steps of the Cm-244 sample preparation.

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Figure 1. Loading the Cm-244 sample into a vial that will go into the ATR irradiation capsule.

"This ATR NSUF experiment has resulted in collaborations at several national laboratories."

Dr. George Imel, Professor of Nuclear Engineering, Idaho State University

Sm-149	1	Flux Wires	17
Np-237	2	U-238	18
Flux Wires	3	Am-243	19
Am-243	4	Pu-239	20
U-233	5	U-236	21
U-235	6	Pu-242	22
Cm-248	7	Flux Wires	23
Pu-242	8	Am-241	24
Pu-244	9	Eu-Cs-Rh	25
Flux Wires	10		
Pu-240	11		
Th-232	12		
Am-241	13		
Np-237	14		
Pu-244	15		
Cm-248	16		

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Figure 2. Sketch of the loading plan used for the thin-boron-filtered samples, and thin boron filters, prior to insertion in ATR.

Once the isotopic compositions were known, the following loading plans were determined for the boron-filtered irradiations (Figure 2):

- 21 capsules for samples: 19 for actinides + 2 for fission products as listed in the following: Th-232, U-233, U-235, U-236, U-238, Np-237 (2), Pu-239, Pu-240, Pu-242, Pu-244 (2), Am-241, Am-243 (2) and Cm-248 (2), Sm-149, Eu-153, Cs-133, Rh-103
- 4 extra-capsules for U-235 wires to measure the total fluence + spectrum wires (the reference boron idea was dropped).

One of the boron-filtered irradiations, scheduled for two cycles at full power, began in February 2012. The cadmium-filtered irradiation began in November 2012, and is scheduled for one cycle only. Both irradiations are expected to be completed by January 2013.

The Cm-244 sample is not being irradiated with the boron filters because preliminary calculations show that the amount of Cm-245 build-up from neutron capture on Cm-244 would be too small compared to the amount of Cm-245 initially present in the sample. With the cadmium filter, the Cm-245 build-up will be sufficient.

Table 1 (next page) presents the expected A+1, A+2, and A+3 build-up from successive neutron captures on A after 110 days of irradiation with a thin boron filter.

The analytical expressions resulting in these atom densities are solutions of the general Bateman equations, and are relatively involved. However, the simple expressions below provide excellent approximations for most cases, as well as some insight into the physics. Note that in all of the samples less than 1% of the initial isotope A is transmuted.

$$\begin{cases} N_{A+1}(t) \sim N_A(0)\bar{\sigma}_A^c \tau \ \ with\ \tau = \int \emptyset(t)dt \\ N_{A+2}(t) \sim \frac{1}{2} N_A(0)\bar{\sigma}_A^c \bar{\sigma}_{A+1}^c \tau^2 \\ N_{A+3}(t) \sim \frac{1}{6} N_A(0)\bar{\sigma}_A^c \bar{\sigma}_{A+1}^c \bar{\sigma}_{A+2}^c \tau^3 \end{cases}$$

Combining this information with the initial isotopic compositions of the samples gives definition to the sample loading plan.

Future Activities

Two of the three scheduled irradiations are expected to be completed in 2013. The irradiation schedule for the final boron-filtered samples has yet to be determined. The INL portion of the post-irradiation examination is scheduled to be completed by December 2013.

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Measurement of Actinide Neutronic Transmutation Rates with Accelerator Mass Spectroscopy (MANTRA) (cont.)

Table 1. Expected A+1, A+2, and A+3 build-up from successive neutron captures on A after 110 days of irradiation with a thin boron filter.

Th-232	U-233	U-234	U-235					
1.0E+00	5.6E-04	5.4E-08	1.2E-11					
U-233	U-234	U-235	U-236					
1.0E+00	3.7E-04	1.5E-07	3.5E-11					
U-235	U-236	Np-237	Pu-238					
1.0E+00	7.0E-04	2.3E-07	1.2E-10					
U-236	Np-237	Pu-238	Pu-239					
1.0E+00	6.6E-04	5.6E-07	1.5E-10					
U-238	Pu-239	Pu-240	Pu-241					
1.0E+00	6.3E-04	2.5E-07	1.2E-10					
Np-237	Pu-238	Pu-239	Pu-240					
1.0E+00	2.0E-03	9.4E-07	2.6E-10					
Pu-239	Pu-240	Pu-241	Pu-242					
1.0E+00	8.5E-04	6.2E-07	1.2E-10					
Pu-240	Pu-241	Pu-242	Am-243					
1.0E+00	1.4E-03	4.2E-07	1.0E-10					
Pu-242	Am-243	Cm-244	Cm-245					
1.0E+00	7.3E-04	7.2E-07	2.6E-10					
Pu-244	Cm-245	Cm-246	Cm-247					
1.0E+00	5.0E-04	1.5E-07	3.3E-11					
Am-241	Am-242m	Pu-242	Cm-242	Am-243	Cm-243	Cm-244	Pu-238	Pu-239
1.0E+00	2.5E-04	3.2E-04	1.4E-03	1.8E-07	4.4E-07	2.1E-10	3.2E-04	1.1E-07
Am-243	Cm-244	Cm-245	Cm-246					
1.0E+00	2.0E-03	1.1E-06	2.1E-10					
Cm-244	Cm-245	Cm-246	Cm-247					
9.9E-01	1.1E-03	3.1E-07	7.0E-11					
Cm-248	Bk-249	Cf-249	Cf-250	Cf-251				
1.0E+00	4.4E-04	5.2E-05	4.7E-07	2.2E-10				

Publications and Presentations

1. Gilles Youinou, Guiseppe Palmiotti, Massimo Salvatores, George Imel, Richard Pardo, Filip Kondev, Michael Paul, 2010, *Principle and Uncertainty Quantification of an Experiment Designed to Infer Actinide Neutron Capture Cross-Sections*, INL/EXT-10-17622, January 2010.

2. Gilles Youinou, Massimo Salvatores, Michael Paul, Richard Pardo, Giuseppe Palmiotti, Charles McGrath, Filip Kondev, George Imel, 2010, "MANTRA: An Integral Reactor Physics Experiment to Infer Actinide Capture Cross-Sections From Thorium To Californium With Accelerator Mass Spectrometry," *Eleventh International Conference on Nuclear Data for Science and Technology, Jeju Island, Korea, April 26-30, 2010,* published in the *Journal of the Korean Physical Society*, Vol. 59, No. 2, August 2011, pp. 1940-1944.

3. Gilles Youinou was invited to the *FCR&D Nuclear Physics Working Group meeting*, *Port Jefferson, New York, June 24-25, 2010*, to present the MANTRA experiment.

4. Giuseppe Palmiotti (INL Fellow) was invited to the *Third Meeting of the Expert Group on Integral Experiments for Minor Actinide Management*, *NEA Headquarters, Issy-les-Moulineaux, France, September 13-14, 2010,* to present the MANTRA experiment.

5. Richard C. Pardo, Filip G. Kondev, Sergey Kondrashev, Catherine Nair, Tala Palchan, R. Scott, Dariusz Seweryniak, Richard Vondrasek, Michael Paul, Phillipe Collon, C. Deibel, Gilles Youinou, Massimo Salvatores, Giuseppe Palmotti, Jeff Berg, Jaqueline Fonnesbeck, George Imel, "Toward Laser Ablation Accelerator Mass Spectrometry of Actinides with an ECRIS and Linear Acceleration and Initial Background Studies," submitted to *Nuclear Instruments and Methods* (B).

The principle and status of the MANTRA experiment was presented to an international panel during the Nuclear Science & Technology Directorate Review in June 2011.

In August 2011 the MANTRA integral reactor physics experiment was presented to the Department of Energy (DOE) during a two-day meeting between the principal investigators (with 2009 and 2010 awards in Applications of Nuclear Science and Technology supported by DOE Office of Nuclear Physics and American Recovery and Reinvestment Act funds), and nuclear physics federal program managers. The slides are available at http://science.energy.gov/np/benefits-of-np/anst/anst-exchange-meeting-08222011.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Team Members/Collaborators	
Idaho State University George Imel (principal investigator), Eric Burgett (collaborator), Jyothir Kumar (M.S. student) **Idaho National Laboratory** Gilles Youinou (principal investigator) **Argonne National Laboratory** Richard Pardo (collaborator)	

Irradiation Behavior of Triplex SiC Composite Tubing under PWR Conditions

Principal Investigator: Mujid S. Kazimi – Massachusetts Institute of Technology
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Introduction

Recently, massive amounts of debris from the Fukushima tsunami that devastated Japan in 2011 washed up on the shores of the Hawaiian Islands. For those of us in the research community, this is a reminder of the impact natural disasters can have not only on the general populace but also on the future of the nuclear power industry that serves it. Thus, there is a growing consensus that fuel behavior must be improved in loss-of-coolant accidents (LOCA) at nuclear reactors.

One exciting potential material is Triplex Silicon Carbide (Triplex SiC), which holds promise as a significant component in accident-tolerant fuel systems, especially as clad tubing. However, the existing data on the behavior of Triplex SiC composite clad tubing in light water reactor environments is very limited. Therefore, one of the major technical goals of this research is to collect as much relevant information as possible on the use of triplex tubing as a reactor fuel clad. Obtaining this data in a relatively short time will assist tubing manufacturers in improving the irradiation behavior of the material. It is also important in building sufficient confidence in the material's behavior to justify expensive, time-consuming integral fuel experiments in test reactors.

Triplex SiC composite tubing has been shown to survive under simulated PWR coolant conditions in an in-core loop for up to 850 effective full power days.

Project Description

This ATR NSUF project is part of a larger effort to develop and qualify all SiC composite materials for use as fuel cladding in pressurized water reactors (PWR). The primary focus of the research is to expose a variety of candidate tubing materials and bonding methodologies to PWR conditions in an in-core loop at the Massachusetts Institute of Technology Reactor (MITR). Post-irradiation examination (PIE) is used to characterize the corrosion behavior and mechanical property evolution of the samples. The MITR PWR loop tests play a key role in the research because they allow researchers to study the interaction between the cladding and the coolant under prototypical reactor conditions. Over the course of the ATR NSUF project, the MITR team has used loop tests to accumulate the equivalent of approximately three years of reactor exposure for the most-irradiated materials still in the test.

Other related in-reactor research, such as that conducted in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL), addressed other important areas such as fuel/cladding interactions. At MIT as well as the ORNL, out-of-pile chemical reactions with steam under potential LOCA or other off-normal conditions are being investigated.

The potential benefits of successfully developing the Triplex SiC/SiC-composite ceramic cladding as a replacement for conventional zirconium alloy (Zircaloy) fuel cladding include better fuel behavior during LOCAs and departure from nucleate boiling (DNB) transients and increased fuel burnup.

These benefits are derived from Triplex SiC's intrinsic high-temperature strength and radiation resistance as well as the following man-made features that have been engineered into it:

- An inner, monolithic SiC layer that seals the clad and prevents the release of fission product gases
- An SiC, fiber-based composite middle layer that provides mechanical strength and a "graceful" failure mode that confines the solid fission products while maintaining a coolable geometry even under LOCA conditions
- An outer layer of chemical vapor deposited (CVD) SiC and an environmental barrier coating.

Accomplishments

The major ATR NSUF experiment accomplishments at MITR in 2012 were:

- The completion of the in-core irradiation of the SiC and Zircaloy specimens in the PWR-conditions water loop
- PIE of the extracted tube and bond specimens in the hot cell and hot lab facilities.

The two irradiation periods took place between November 2011 and the end of March 2012 and from the middle of April 2012 to the end of June 2012. During these times, 20 SiC tube specimens, one Zircaloy tube specimen, and 10 SiC bonding specimens were exposed in the pressurized water loop of the MITR-II core.

During the first irradiation period, 21 tube and six bonding test specimens were exposed for 89 days at temperature for 6,890 megawatt-hours (MWh) of MITR operation. Following irradiation, the specimens were removed from the reactor and all of the bond specimens were replaced with a new bonding test set. During the second irradiation period, the 21 tube and four bonding test specimens were exposed for 73 days at temperature for 7,660 MWh of MITR operation.

The 12 SiC tube specimens contained in the first irradiation module (known as the "Round 7" specimens) were first irradiated in March 2010, and the second tube module containing eight SiC tubes and one Zircaloy tube ("Round 6" specimens) from a previous irradiation was added in December 2010. The SiC tubes were a mixture of monolithic SiC (α- and β-phase) and Triplex SiC (β-phase monoliths with a SiC-SiC-fiber composite over-braid and coating). The SiC tube specimens from these groups were exposed for 654.5 days at temperature for 53,794 MWh in the MITR core. If previously irradiated specimens are also considered, the maximum sample irradiation exposure was 952 days at temperature for 83,510 MWh of MITR operation. A summary of all the specimens currently available at MIT and their cumulative overall irradiation exposures are provided in Table 1.

"With the significant exposures achieved in the ATR NSUF-funded irradiations, we have a unique opportunity to evaluate the long-term viability of silicon carbide as an accident-tolerant cladding material for light water reactors. SiC has generated a lot of interest for its resistance to the LWR core environment, but this experiment gives insight into its behavior over the entire life of a fuel rod."

Dr. David Carpenter, Research Scientist, MIT Nuclear Reactor Laboratory

Table 1. Summary of Exposures for Specimens Irradiated in the MITR Water Loop.

Specimen	Type	Exposure (d)	MWh	EFPD (5 MW)
08-1021-23-1	R7 Triplex	654.5	53,794	448.3
08-1021-08-1	R7 Triplex	654.5	53,794	448.3
08-1021-08-2	R7 Triplex	654.5	53,794	448.3
08-1021-09-1	R7 Triplex	654.5	53,794	448.3
08-1021-09-2	R7 Triplex	654.5	53,794	448.3
08-1021-10-1	R7 Triplex	654.5	53,794	448.3
08-1021-10-2	R7 Triplex	654.5	53,794	448.3
08-1021-24-1	R7 Triplex	654.5	53,794	448.3
R7M-1	R7 Monolth	418.5	33,870	282.3
R7M-2	R7 Monolth	418.5	33,870	282.3
R7M-3	R7 Monolth	418.5	33,870	282.3
R7M-4	R7 Monolth	418.5	33,870	282.3
Alpha-1	R6 Monolith	849.9	73,334	611.1
F1-5	R6 Triplex	849.9	73,334	611.1
F4-5	R6 Triplex	849.9	73,334	611.1
H2-2	R6 Triplex	849.9	73,334	611.1
H3-1	R6 Triplex	849.9	73,334	611.1
H3-2	R6 Triplex	849.9	73,334	611.1
H3-5	R6 Triplex	608.0	50,513	420.9
M1-2	R6 Monolith	951.9	83,510	695.9
N1-1	R6 Zircaloy	711.9	30,590	254.9
PNL-1	Bond blocks	175.0	12,730	106.1
PNL-4	Bond blocks	46.5	3,281	27.3
Torino-2	Bond blocks	46.5	3,281	27.3
Torino-5	Bond blocks	46.5	3,281	27.3
StGobain-3	Bond blocks	46.5	3,281	27.3
StGobain-6	Bond blocks	46.5	3,281	27.3
PNL-new	Bond blocks	128.5	9,449	78.7
EWIP1-1	Bond blocks	89.3	6,890	57.4
EWIP4-1	Bond blocks	89.3	6,890	57.4
EWIP4-2	Bond blocks	89.3	6,890	57.4
Toshiba-2	Bond blocks	89.3	6890	57.4
Toshiba-3	Bond blocks	89.3	6890	57.4
StGobain-3 (2)	Bond blocks	89.3	6890	57.4
Zr4 CMC	Overbraided	72.7	7656	63.8
SiC-1	End cap	72.7	7656	63.8
SiC-2	End cap	72.7	7656	63.8
Zr4 poly	Overcoated	72.7	7656	63.8

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Irradiation Behavior of Triplex SiC Composite Tubing under PWR Conditions (cont.)

PIE included physical examination and documentation of the materials in these tubes, calculation of their weight changes, and measurement of any irradiation-induced swelling. Images of the Round 6 tube specimen designated H2-2 and a monolith specimen pre- and post-irradiation are shown in Figure 1. Weight losses, attributable to oxidation of the silicon in the exposed surfaces, were lowest—less than 0.1% per month—in the monolithic SiC tubes. The tubes containing the best-performing Triplex SiC had weight losses of around 0.5% per month. Note that some of this weight loss is attributable to enhanced corrosion at the unsealed ends of the specimens. This loss would not be observed in a fully assembled fuel rod with sealed end caps.

The bond specimens during the irradiation period beginning in November 2011 consisted of six solid α-SiC block pairs representing three different bonding processes. The blocks were bonded together either flush or with a step offset. When the bond module was opened after irradiation, four of the six pairs remained together (EWIP1-1 and StGobain-3 were unbonded).

A typical bond pair is shown in Figure 2. PIE for these pairs included measuring weight changes and swelling, photographic documentation of the bond, and shear testing if the bond showed good performance during irradiation.

Figure 1. (A Triplex SiC tube specimen (a and b) and a monolithic SiC tube specimen (c and d) before and after irradiation in the MITR water loop (> 800 effective full-power days exposure).

Figure 2. (a) An SiC bond block specimen EWIP4-1 before, and (b) after irradiation in the MITR water loop.

The second set of bond specimens was inserted into four tubes. Each tube was sealed at one end with an end cap, while the other end was left open. Two tubes held SiC monoliths; the other two tubes had a base layer of Zircaloy-4. One tube of this second set had a braided SiC outer layer, and the other had a thin polymer on the outer surface.

After irradiation, both end caps had separated from the SiC tubes. The Zircaloy tubes require additional decay time before they can be removed from the hot cell for examination, but they have no gross physical changes.

The MIT researchers also demonstrated that measurements of xenon flash thermal diffusivity can be taken on segments cut from the Triplex SiC tubes. Comparing the thermal diffusivities and conductivities of a variety of exposed and unexposed materials, they concluded that, under irradiation, degradation of the triplex tubing is similar to that observed in monolithic SiC, with saturation of the decrease occurring at about 1 displacement per atom (dpa). It is also likely that corrosion films play a role in the observed differences between the exposed and unexposed samples.

Future activities

The ATR NSUF portion of this project was completed in 2012. Work on the SiC composite clad tubing will continue under other funding. Future plans include more detailed PIE on the surviving bond samples and possibly on some of the Round 6 samples. In addition, some Round 6 samples may be selected for further exposure in the in-core loop. Although these samples do not represent the current state-of-the-art, they are unique in that during these experiments, they have accumulated irradition exposures almost equivalent to the full lifetime of a PWR fuel pin.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Massachusetts Institute of Technology	Massachusetts Institute of Technology Reactor
Team Members/Collaborators	
Massachusetts Institute of Technology Mujid Kazimi (principal investigator), David Carpenter, Edward Pilat (collaborators)	
Idaho National Laboratory Mitchell Meyer (principal investigator)	
Westinghouse Electric Company Edward Lahoda (collaborator)	
Ceramic Tubular Products Herbert Feinroth (collaborator)	

Publications

John Stempien, David Carpenter, Gordon Kohse, Mujid Kazimi, 2012, “Characteristics of Composite Silicon Carbide Fuel Cladding After Irradiation Under Simulated PWR Conditions,” *Nuclear Technology*, accepted October 4, 2012.

Principal Investigator: Michele Manuel – University of Florida
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Atom Probe Tomography to Study Fission Product Damage in Model Nuclear Fuel

Introduction

It is widely known that microstructural changes in nuclear fuels during irradiation can lead to the deterioration of thermal performance. Damage processes associated with the formation and migration of fission products play a pivotal role in the microstructural evolution of these fuels. This, in turn, can lead to detrimental micro-structural features such as solute clustering, void formation, and grain boundary segregation, all of which can promote integrity loss.

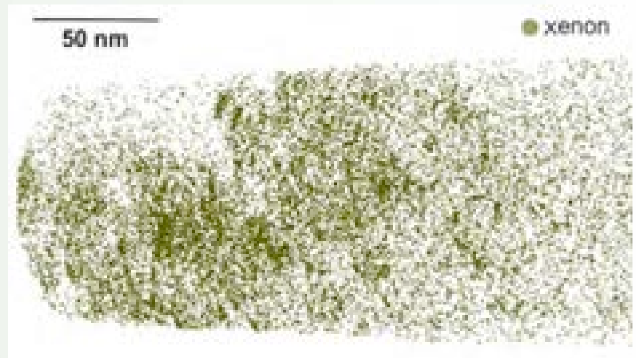
Nanoscale characterization of the changes in microstructure of fuel materials under irradiation is of great interest in understanding the connection between fission product migration and microstructural evolution.

Project Description

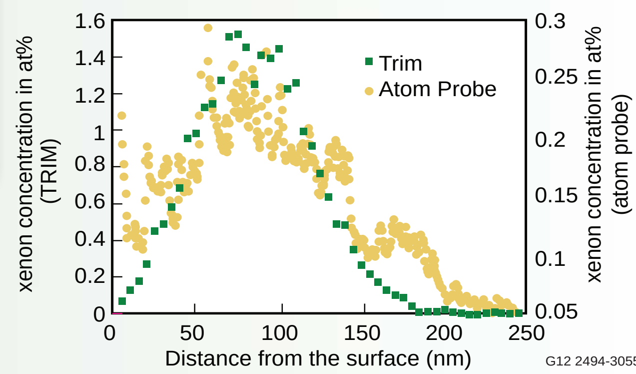
The objective of this rapid turnaround experiment is to develop a fundamental understanding of the science of nuclear fuel microstructure under irradiation. Specifically of interest is the effect of microstructural evolution on the thermal transport behavior of these fuels. Because of their documented roles in fission product damage, namely fission gas release, void formation, and grain boundary modification, this project investigated the segregation behavior of ion-implanted xenon (Xe) and lanthanum (La) as fission products in the surrogate oxide fuel, ceria (CeO_2).

Atom probe tomography (APT) was used to characterize fission product distribution, not only because of the high quality spatial compositional information it provides for the quantification of grain boundary segregation, but also because of its statistical methodology for determining the presence and location of solute clusters (Figure 1). The goal is to elucidate the fundamental material-physics underlying the connection between fission product migration and microstructural evolution.

Xenon Ceria



CeO_2 Atom Probe_TRIM Comparison 1



Ceria Atom Probe SIMS-1

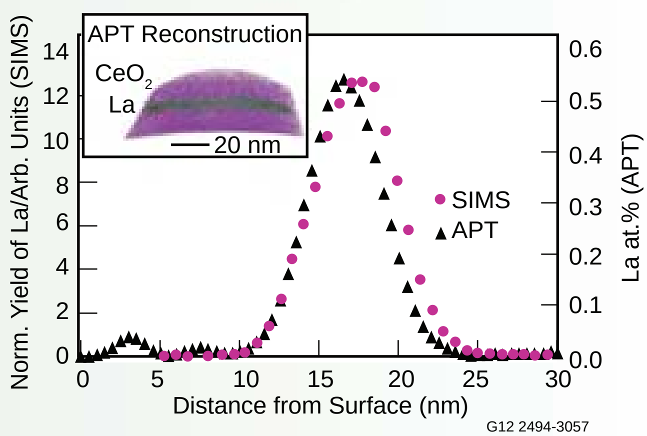


Figure 1. Atom probe reconstruction of the distribution of implanted xenon in bulk CeO_2.

"This is one of the few places where we can study radioactive materials at the atomic scale."
Dr. Michele V. Manuel, Assistant Professor, University of Florida

Accomplishments

During 2012, an APT study was performed on both La- and Xe-doped ceria. The heat treatment applied to the Xe-implanted ceria was insufficient to induce segregation of the Xe toward grain boundaries in the ceria. However, detection of implanted fission products in both sample types corresponded well with transport of ions in matter (TRIM) simulations for Xe-doped ceria and experimental secondary ion mass spectroscopy (SIMS) data for La-doped ceria.[1]

Future Activities

This project is complete, but the ability to detect low concentrations of fission products in surrogate fuel materials using APT will aid researchers moving forward to study fission product evolution in uranium oxide (UO_2).

Publications and Presentations

Billy Valderrama, Hunter Henderson, Jianliang Lin, In-wook Park, John Moore, Clarissa Yablinsky, Todd Allen, Michele V. Manuel, 2012, "Atom Probe Tomography of Simulated Fission Product Segregation in CeO2," S*MD 2012 Technical Division Student Poster, TMS Annual Meeting, March 11-15, 2012.*

References

1. Harrison K. Pappas, Brent J. Heuser, Melissa M. Strehle, 2010, "Measurement of Radiation-Enhanced Diffusion of La in Single Crystal Thin Film CeO_2," *Journal of Nuclear Materials*, Vol. 405, No. 2, pp. 118-125.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Team Members/Collaborators	
University of Florida Michele Manuel (principal investigator), Hunter Henderson, Billy Valderrama (Ph.D. students) **University of Wisconsin** Todd Allen (collaborator) **Colorado School of Mines** Jianliang Lin (collaborator) **Idaho National Laboratory** Jian Gan (collaborator) **University of Illinois** Brent Heuser (collaborator)	

Principal Investigator: K. L. Murty – North Carolina State University
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Influence of Fast Neutron Irradiation on the Mechanical Properties and Microstructure of Nanostructured Metals and Alloys

The core structural components and fuel cladding in advanced nuclear systems are exposed to very high radiation levels, requiring the materials they are made of to have superior radiation-resistant properties. In order to meet these demands, the development of new materials that can withstand these severe conditions has become a major goal of the nuclear materials community.

Bulk nanograined (NG) metals and alloys having a relatively large fraction of interfaces are expected to be more radiation-resistant than conventional grain (CG) metals. This is due to the fact that the point and line defects produced by neutron radiation migrate to these interfaces, where they are absorbed and, therefore, are not available for radiation hardening and embrittlement.

Project Description

This project is a collaborative effort of North Carolina State University (NCSU) and ATR NSUF. The goal is to understand the effect of grain size on a material's irradiation tolerance. To accomplish this, the project uses NG-copper produced using electro-deposition (ED) and the corresponding CG-copper to characterize the effects of neutron irradiation on both materials.

The NG-copper (99.999% copper) was procured from the 3M Corporation, and the CG-copper came from the nuclear materials laboratory at NCSU, both in sheet forms. Figure 1 shows bright field (BF) transmission electron microscopy (TEM) micrographs for both materials prior to irradiation. Using different microstructure characterization techniques—TEM, atomic force microscopy (AFM), X-ray diffraction (XRD), and optical microscopy—the initial average grain size of NG-copper was found to be ~ 34.4 nm and the average grain size of CG-copper was 38 µm.

We expect bulk nanograined materials having large grain boundary areas per unit volume to exhibit enhanced resistance to neutron irradiation damage.

Accomplishments

Post-irradiation examination (PIE) of NG- and CG-copper irradiated at 0.0034, 1, and 2 displacements per atom (dpa) was successfully accomplished. The responses of the developed microstructures and measured mechanical properties of both materials to neutron irradiation at

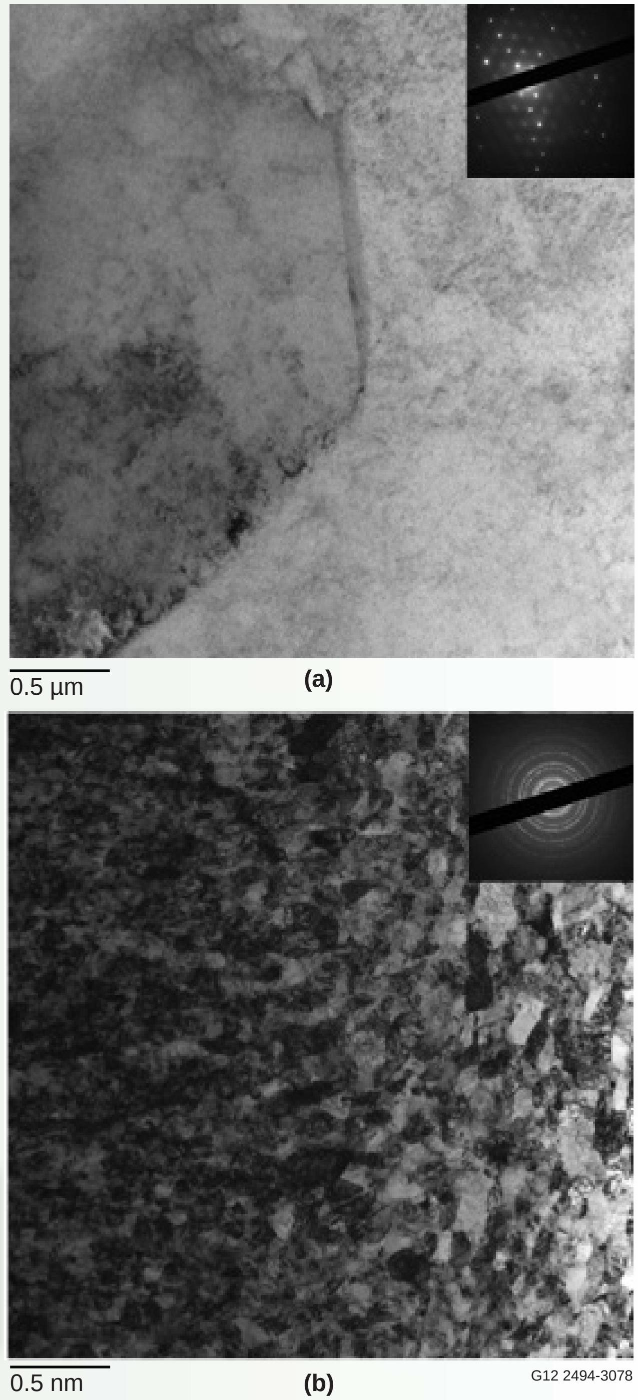

Figure 1. (a) Pre-irradiation TEM micrographs of CG-copper, and (b) NG-copper.

the different exposure levels were investigated. PIE characterization of samples irradiated at the PULSTAR reactor at 1 MW for 200 hours (0.0034 dpa) was conducted at the Nuclear Materials Laboratory at NCSU, while samples irradiated in ATR for one and two reactor cycles in the east flux traps irradiation position (1 and 2 dpa) were characterized at MFC at INL.

Irradiation response of CG-copper was found to follow the general trend of other face-centered cubic (FCC) metals after irradiation. Figure 2 shows stress-strain curves of irradiated CG-copper at all exposure levels. At a glance, the response of irradiated CG-copper to tensile loading compared to the as-received materials can be summarized as a notable increase in strength (both yield and ultimate) accompanied by a loss of ductility in terms of uniform and total elongations. These resulting properties are jointly described as irradiation hardening (describing the increase of strength) and embrittlement (the loss of ductility). Interestingly, the yield-drop phenomenon was observed in CG-copper at 2 dpa and was followed by the formation of distinct Luders bands.

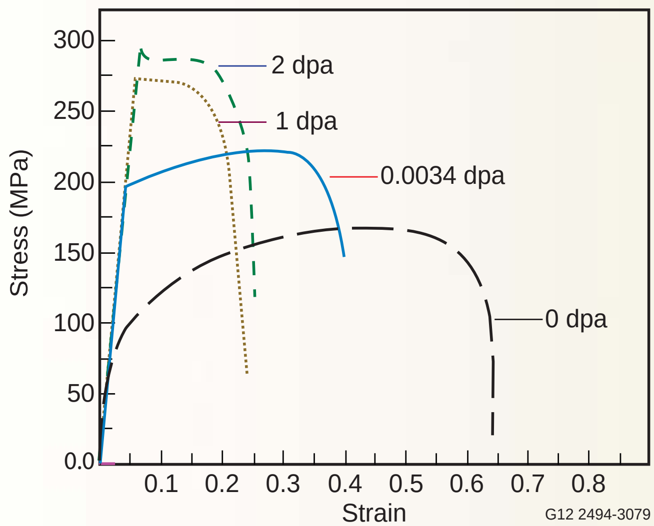

Figure 2. Stress-strain curves of irradiated CG-copper at all exposure levels.

Microstructural characterization of the irradiated CG-copper at exposure levels up to 1 dpa showed an increase in the dislocation density (Figure 3). As the density of mobile dislocations increases, the movement of those dislocations, as well as other types of defects, becomes more difficult. This explains the observed increase in strength and loss of ductility of irradiated CG-copper.

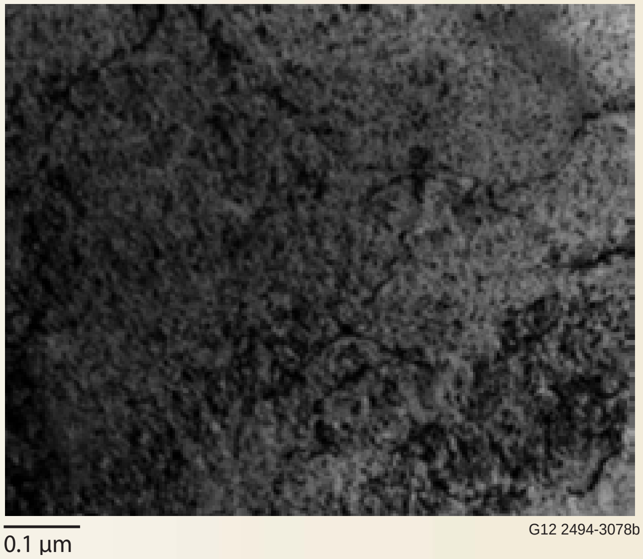

Figure 3. High dislocation density in CG-copper at 1 dpa.

Exposure of the microstructure of CG-copper at 2 dpa revealed the formation of twin structures in addition to the expected dislocation loops (Figure 4).

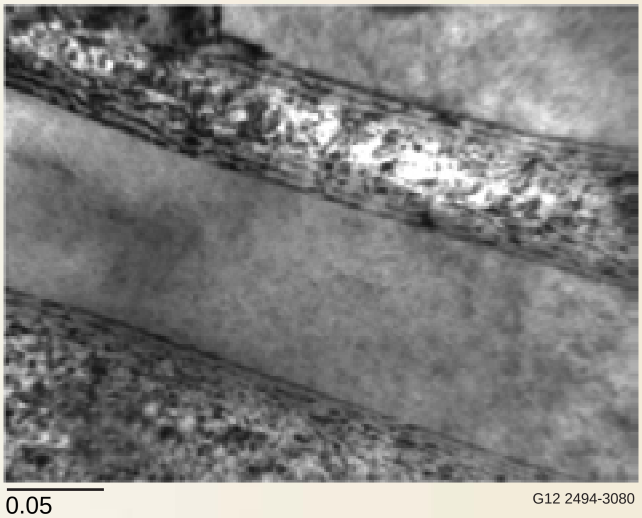

Figure 4. BF TEM micrograph of CG-copper at 2 dpa showing formation of twins structure.

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Influence of Fast Neutron Irradiation on the Mechanical Properties and Microstructure of Nanostructured Metals and Alloys (cont.)

On the other hand, the response of NG-copper to neutron irradiation was found to be highly dependent on the exposure level. Figure 5 shows the response of the material to tensile loading at all exposure levels.

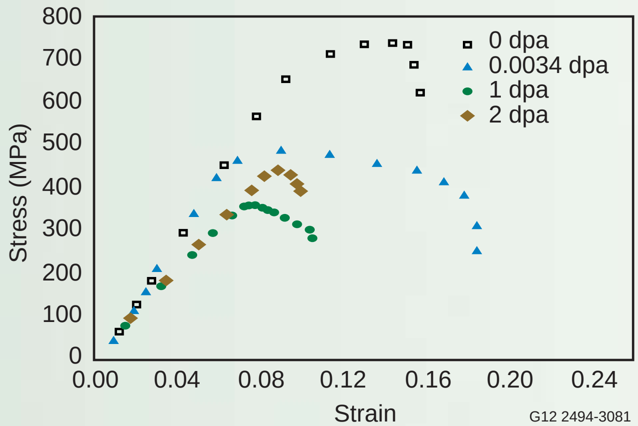

Figure 5. Stress-strain curves of NG-copper at all exposure levels.

At low exposure levels (0.0034 to 1 dpa), grain growth was observed to be the controlling mechanism in a dramatic decrease in strength and an accompanying increase in ductility. Both observations can be related to an increase in grain size, based on the well-known Hall-Petch relation.

At relatively higher exposure levels (1 to 2 dpa), the common radiation hardening started to develop as grain growth approached saturation at the submicron level.

The average grain size of NG-copper following exposure at 0.0034 dpa was estimated to be ~ 86.5 nm, with the grain size distribution shown in Figure 6. The presence of

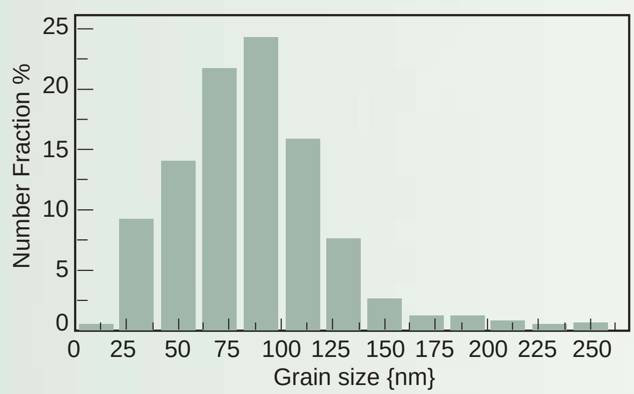

Figure 6. Grain-size distribution of NG-copper at 0.0034 dpa showing an average grain size of 86.5 nm; whereas the initial average grain size of the as-received material was ~ 34.4 nm, which indicates grain growth at this damage level.

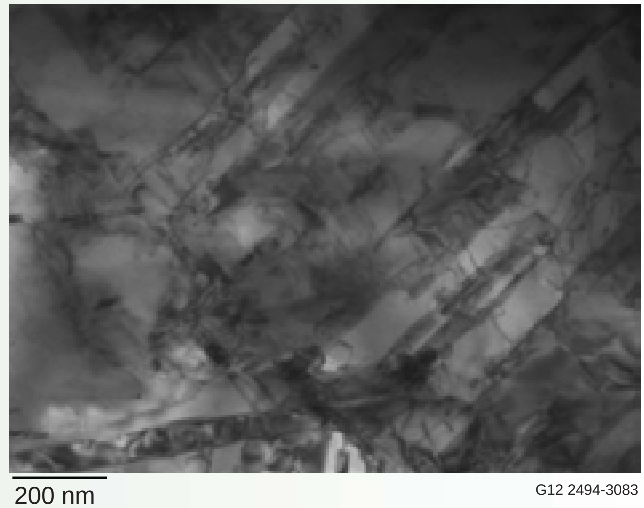

Figure 7. Bright field TEM of NG-copper at 0.0034 dpa showing grains in the sub-micron regime accommodating twins and dislocation loops.

grains in the submicron regime allowed the formation of dislocations and twin structures, which were observed in some TEM foils (Figure 7).

At higher exposure levels (1 to 2 dpa), the average grain size saturated at about 0.8 µm. Even so, the majority of the grains had grown enough to accommodate higher densities of dislocation loops and twins similar to those observed in CG-copper at the same exposure levels (Figure 8).

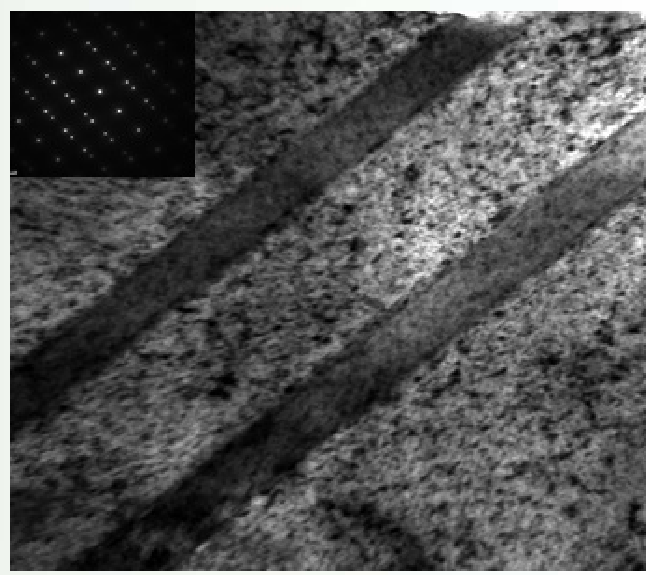

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Figure 8. High density of dislocations formed in NG-copper at 1 dpa with diffraction pattern revealing formation of twin structure.

"It was a great experience to collaborate with researchers at INL during my Ph.D. research work. This experience paved my way to become a post-doctoral appointee at one of the national laboratories upon my graduation."

Dr. Walid Mohamed, Post-doctoral Fellow, Argonne National Laboratory

It is concluded that the response of NG-copper to neutron irradiation is controlled by radiation-induced grain growth at relatively low exposure levels; whereas at relatively higher exposure levels, the predominant mechanism is radiation hardening.

Future Activities

Investigation of ultra-fine-grained steel and NG-nickel samples irradiated along with the copper samples is planned. While the PULSTAR-irradiated, ultra-fine-grained steel samples can now be handled, the NG-nickel samples are still too highly radioactive for testing in the university laboratory. Irradiated steel samples will be tested at MFC at INL in the near future.

Publications and Presentations

1. Walid Mohamed, 2012, *Influence of Fast Neutron Irradiation on Mechanical Properties and Microstructure of Nanocrystalline Copper*, Ph.D. Dissertation: North Carolina State University, Raleigh, North Carolina.
2. K.L. Murty, Walid Mohamed, Jacob Eapen, Douglas Porter, 2011, "Effect of Neutron Irradiation on Mechanical Behavior of Nanograin Structured Cu," *Material Science and Technology (MS&T '11), Columbus, Ohio, October 2011*.
3. Walid Mohamed, Jacob Eapen, K. L. Murty, "Mechanical Behavior of Nanograin Structured Metals – Effect of Neutron Irradiation," *International Conference on Nanoscience, Nanotechnology & Advanced Materials (NANOS 2010), Rushikonda, Visakhapatnam Andhra Pradesh, India, December 2010*.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
North Carolina State University	PULSTAR Reactor, PIE Facilities

Team Members/Collaborators
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Idaho National Laboratory Doug Porter (co-principal investigator), James Cole, Brandon Miller (collaborators)
University of Idaho Indrajit Charit (co-principal investigator)

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Irradiation Damage of $Ba(Zn_{1/3}Ta_{2/3})O_3$ Ceramic Materials

The data obtained will help researchers understand the influence of native defects and irradiation damage on the performance of practical devices and systems utilizing these materials.

Introduction

The development of high dielectric-constant materials with diminished microwave loss and near-zero temperature coefficients of resonant frequency will enable the production of smaller and higher-performance microwave devices that can operate over a wider range of frequencies. In order to develop a fundamental understanding of the microscopic mechanism that causes loss in practical dielectric materials, a systematic investigation of their performance resilience to high-energy irradiation will help determine whether they can be used in "radiation hard" applications.

Project Description

This rapid turnaround experiment investigated the fundamental nature of irradiation damage in technologically relevant ceramics by using a combination of experimental and theoretical methods to determine the nature and concentration of defects generated by neutron damage. Neutron irradiation was used to systematically introduce defects in the materials. The loss-tangent, along with the nature and concentration of the defects, was determined using a variety of electrical, optical, and magnetic probes. The data obtained will help researchers understand the influence of native defects and irradiation damage on the performance of practical devices and systems utilizing these materials.

Accomplishments

Using nickel (Ni) foils as flux monitors, six samples of $Ba(Zn_{1/3}Ta_{2/3})O_3$ (BZT) underwent fast neutron irradiation at fluences up to $1x10^{15}$ NV (fast neutrons) in rotating exposure ports in the PULSTAR reactor at North Carolina State University. At the end of 2012, researchers had just begun to correlate the neutron-induced changes in microwave loss with the physical properties and defect characteristics of the BZT microwave ceramics. The initial measurements are illustrated in Figures 1-3.

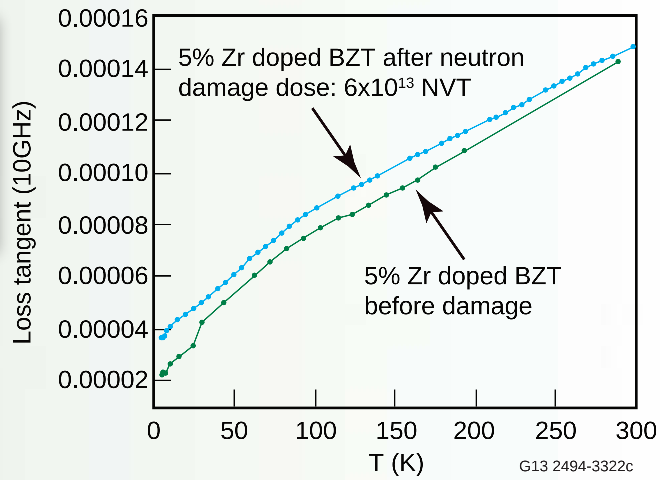

Figure 1. Microwave loss as a function of temperature for unirradiated and irradiated BZT samples. This figure indicates that native defects produced during neutron irradiation contribute to microwave loss.

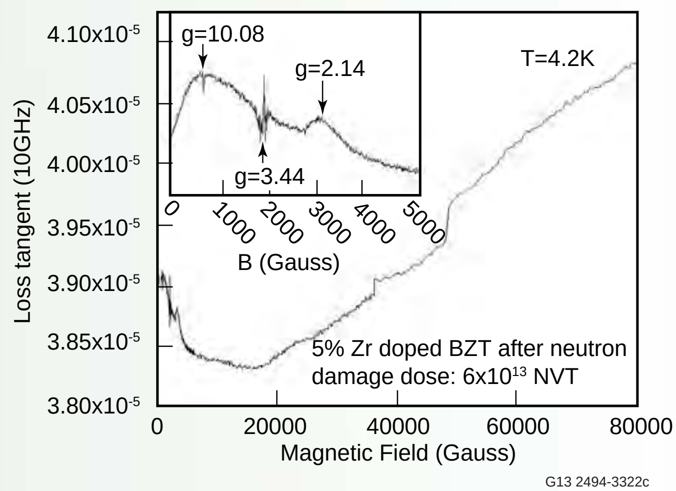

Figure 2. Microwave loss as a function of magnetic field for irradiated BZT samples measured at 4.2 K. The designated peaks in the spectra are associated with electron resonance spin flips at paramagnetic defects.

"This project has made important strides in understanding the mechanism of microwave loss in high-performance dielectrics. This fundamental knowledge will allow us to improve the performance of these materials and ultimately make smaller and higher-performance microwave communication and radar systems."

Dr. Nathan Newman, Professor of Materials Engineering, Arizona State University

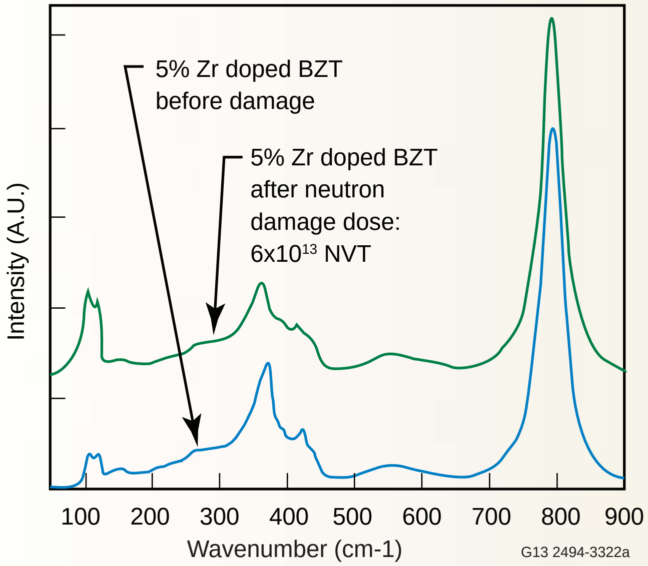

Figure 3. Comparison of Raman spectra of unirradiated and irradiated BZT samples.

Future Activities

Characterization of the materials and defect properties of the neutron-irradiated samples is scheduled to be completed in 2013.

Publications and Presentations

This work is anticipated to culminate in the submission of a manuscript to an archival journal such as *Applied Physics Letters*, or the *Journal of Applied Physics*.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
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Nonstoichiometric Spinel as Inert Matrix

Introduction

The worldwide inventory of radiotoxic weapons- and reactor-grade nuclear waste, such as plutonium (Pu), neptunium (Np), americium (Am), and curium (Cm) is growing rapidly. Disposing of this waste in safe, secure, ecologically responsible, and economically sensible ways is a national and international priority.

One way to decrease the amounts and hazards associated with radiotoxic waste is through transmutation in nuclear reactors, which converts the radioactive constituents of the waste material into more stable elements - with one important exception. Transmutation of mixed oxide (MOX)-based fuel leads to the generation of new transuranium actinides. A more promising approach is to burn Pu and other transuranic elements in an inert matrix fuel. This method generates less radioactive waste.

Understanding the behavior of materials under irradiation is important for developing inert-matrix fuels that help decrease radiotoxic waste while instituting a sustainable nuclear fuel cycle.

Project Description

In a recent Nuclear Energy Research Initiative (NERI) project at the University of Florida (UF), the INL's efforts to develop magnesium oxide (MgO)-based ceramic-ceramic (cercer) inert matrix materials were expanded. The UF investigators developed and investigated the synthesis and thermophysical properties of two potential inert matrix materials: magnesium oxide-neodymium zirconate cercer composites (MgO-$Nd_2Zr_2O_7$) and single-phase, magnesium-based spinel compounds (see Table 1).

Building on INL's previous work, this ATR NSUF post-irradiation examination (PIE) project had three objectives:

- Investigate the behavior of MgO-$Nd_2Zr_2O_7$ cercer composites when used as inert matrices in irradiated environments
- Investigate the behavior of single-phase, Mg-based spinel compounds as inert matrices in irradiated environments
- Characterize the effects of irradiation on the microstructure and thermophysical properties of the irradiated materials.

Ceramic disc samples were irradiated at approximately 350° C and 700° C to dose accumulations of 2 displacements per atom (dpa) and 4 dpa (Table 1). PIE was then performed on each material at INL's MFC.

Table 1. Irradiated materials and capsule irradiation conditions.

Materials	Temperature (°C)		
MgO $MgAl_2O_4$ Mg_2SnO_4 MgO•1.5Al_2O_4 $Nd_2Zr_2O_7$ 0.7MgO-0.3$Nd_2Zr_2O_7$	350	700	
	Capsule Identification		Dose (dpa)
	A2 or I1	B2	2
	C2 or I2	C1 or I3	4

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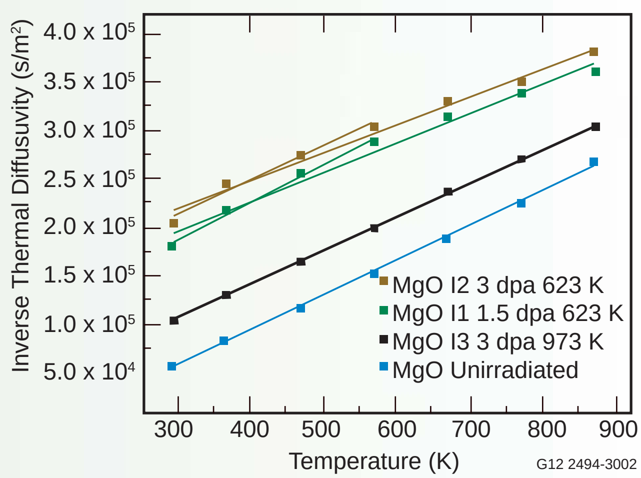
Accomplishments

During the first three years of the project, material samples were prepared, irradiated, and then subjected to initial PIE, using transmission electron microscopy (TEM), scanning electron microscopy (SEM), and X-ray diffraction (XRD). In 2012, PIE continued, mainly on the $MgAl_2O_4$ samples from capsules A2, C1, and C2.

In the last year, linear extrapolation of the thermal diffusivity of MgO samples from the previous years produced a slope that was determined by the lattice and the intercept at 0 K by defects. For the I1 and I2 slopes in Figure 1, when using diffusivity values above the irradiation temperature, fitting produced an apparent decrease in the slope, which represents the intrinsic lattice diffusivity, and an apparent increase in the intercept, which is affected by the number of defects.



G12 2494-3002

Figure 1. Inverse thermal diffusivity with linear fit to 573 and 873 K. Fitting measured diffusivity values above the irradiation temperature results in an apparent decrease in the intrinsic lattice diffusivity.

"The knowledge I gained working at ATR NSUF was invaluable. It's an experience that is rarely available at a university. Performing hands-on work at a national laboratory broadened my intellectual and experimental abilities while providing a 'big picture' view of the nuclear fuel cycle and a unique insight into the critical engineering issues surrounding nuclear reactors."

Donald Moore, Graduate Student, University of Florida

When fitting up to the irradiation temperature, the slope was unaffected by irradiation, indicating no change occurred in phonon-phonon scattering. The intercept increased when irradiation dosages were raised and decreased under higher irradiation temperatures. This is due to the fact that point defects are more effective as scattering agents than aggregates. On the other hand, higher irradiation temperatures resulted in greater aggregation and thus less scattering.

The thermal diffusivity graphs in Figure 2 show that irradiation damage caused the diffusivity to decrease. Specifically, $MgAl_2O_4$ shows a significant reduction in thermal diffusivity under low irradiation temperatures.

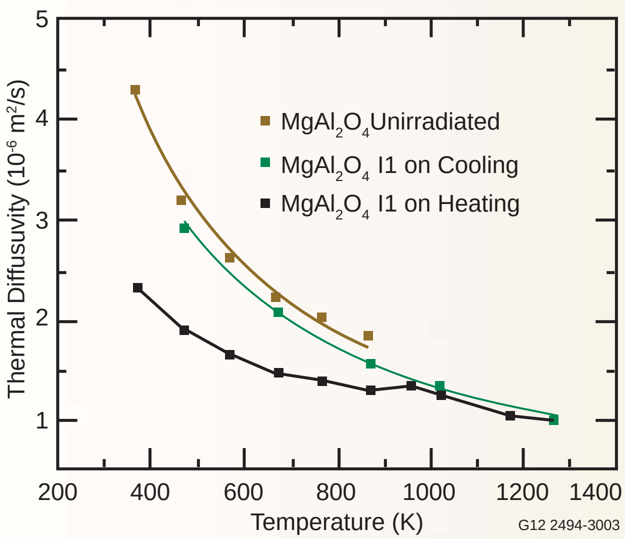


Figure 2. Thermal diffusivity of $MgAl_2O_4$ I1 on heating and cooling showing the effect of thermal annealing during the measurement.

To understand the effect of thermal annealing, thermal diffusivity was also measured on heating, and again on cooling. It was found that defects begin annealing once the measurement temperature exceeds the irradiation temperature. For example, when the measurement temperature reaches 959 K, a noticeable increase in thermal diffusivity occurs. This is notable because 959 K is close to the I3 capsule's irradiation temperature. Vacancy-interstitial recombination results in the material's recovery from the thermal diffusivity. Even as the measurement temperature increases to 1273 K, no significant annealing of the defects occurs, resulting in further recovery of the thermal diffusivity. This is shown by the measurement on cooling where the 1000 K value is relatively unchanged.

TEM images of $MgAl_2O_4$ in Figure 3 show that $MgAl_2O_4$ has bending contours due to the deformation of the TEM lamella (Figure 3-N). The $MgAl_2O_4$ sample in Capsule I1 (Figure 3-I1) has a high density of black spot damage due to neutron irradiation. However, the $MgAl_2O_4$ sample from Capsule I2 Figure (3-I2) has a higher density of black spot damage than the sample in I1 because of the larger irradiation dose I2 received. TEM of the I3 sample of $MgAl_2O_4$ (Figure 3-I3) shows areas with high densities of larger dislocations as well as areas with few dislocations. This was due to stacking faults caused by the higher irradiation temperature. TEM was also performed on Mg_2SnO_4 and compared to $MgAl_2O_4$. Those results are still being evaluated.

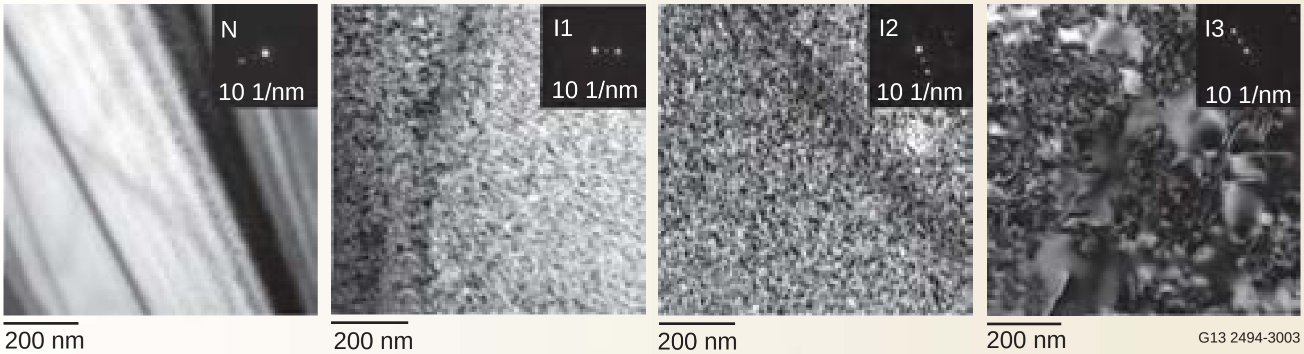


Figure 3. TEM of (N) unirradiated $MgAl_2O_4$ has bending contours due to deformation of the lamella and (I1) $MgAl_2O_4$ has a high density of black spot damage to neutron irradiation. TEM of (I2) $MgAl_2O_4$ has a higher density of dislocations compared to (I1) because of the larger irradiation dose. TEM of (I3) $MgAl_2O_4$ has areas of high density of larger dislocations and areas of few dislocations caused by stacking faults of due to the higher irradiation temperature. Each TEM image is in the g = [400] two-beam condition.

Nonstoichiometric Spinel as Inert Matrix (cont.)

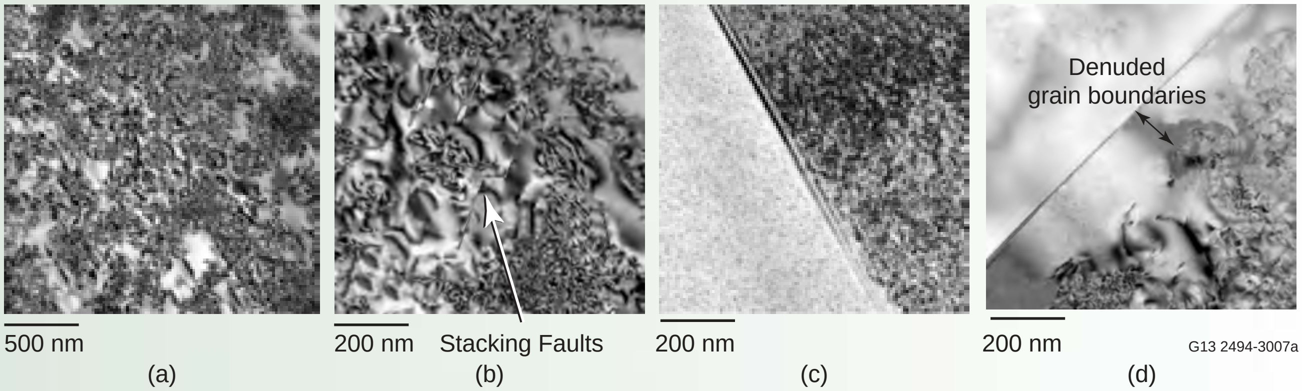


Figure 4. (a) TEM of $MgAl_2O_4$ I3 at lower magnification shows areas with lesser damage and areas with high density of large dislocations, (b) higher magnification of same sample shows that the areas of lesser damage are caused by stacking faults, (c) TEM of $MgAl_2O_4$ I2 shows damage at the grain boundaries, and (d) TEM of $MgAl_2O_4$ I3 shows denuded areas at the grain boundaries for the higher irradiation temperature.

Figure 4a shows the TEM of Capsule I3's $MgAl_2O_4$ sample at a lower magnification. Regions of lesser damage as well as regions with high densities of large dislocations are evident. At higher magnification (Figure 4b), it is apparent that the areas of lesser damage are caused by stacking faults. In Figure 4c, the $MgAl_2O_4$ from Capsule I2 has damage at the grain boundaries, whereas, in Figure 4d, the $MgAl_2O_4$ from I3 has denuded areas at the grain boundaries due to the higher irradiation temperature.

The denuded areas, which are approximately 200 nm wide, do not contain any dislocations. This is because the grain boundaries act as sinks, reducing damage to the area.

At higher irradiation temperatures, a higher diffusion of dislocations and grain boundaries occurs. As with the stacking faults, these diffusions and grain boundaries act as sinks that reduce damage to the nearby regions.

In 2012, radiation work at MFC was suspended for several months, which slowed progress on the project. Nonetheless, the research team completed characterization of the effects of irradiation on the microstructure and thermophysical properties of $MgAl_2O_4$.

Future Activities

Although this project has ended, additional PIE is possible, particularly since all the samples synthesized and utilized in the project are now part of the ATR NSUF sample library. For example, completing PIE of Mg_2SnO_4 from Capsules C1 and C2 will allow researchers to compare the effects of irradiation dose and temperature on the material's neutron irradiation stability with that of irradiation-resistant $MgAl_2O_4$. Comparing the irradiation damage between the two materials will help determine if the atomic number and inverse spinel crystal structure are key parameters to the irradiation resistance of the spinel.

Publications

1. Donald Moore, Cynthia Papesch, Brandon Miller, Pavel Medvedev, and Juan Nino, 2011, "Irradiation Behavior of Oxide Ceramics for Inert Matrices," *Materials Research Society* presentation, *November 28, 2011*.

2. Todd Allen and Donald Moore, 2012, "Advanced Test Reactor National Scientific User Facility," *National User Facility Organization - 2012 User Science Exhibition on Capitol Hill*, *Washington, D.C., March 28-29, 2012*.

3. Donald Moore, Cynthia Papesch, Brandon Miller, Pavel Medvedev, and Juan Nino, 2012, "Irradiation Behavior of Oxide Ceramics for Inert Matrices," *ATR NSUF Workshop, American Nuclear Society Student Conference*, *May 10, 2012*.

4. Pavel Medvedev, Donald Moore, Cynthia Papesch, Brandon Miller, and Juan Nino, 2012, "Irradiation Behavior of Oxide Ceramics for Inert Matrices," *2012 American Nuclear Society Annual Meeting, Embedded Topical Meeting on Nuclear Fuels and Structural*

Materials for the Next-Generation Nuclear Reactors, *Chicago, Illinois, June 28, 2012*.

5. Donald Moore, Cynthia Papesch, Brandon Miller, Pavel Medvedev, and Juan Nino, "In-Pile Irradiation Induced Defects and the Effect on Thermal Diffusivity of MgO," submitted to *Journal of Nuclear Materials*.

6. Donald Moore, 2012, "*Thermal Diffusivity of Advanced Ceramics Under Harsh Environments,*" M.S. Thesis: University of Florida, Gainesville, Florida.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
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Characterization of the Microstructures and Mechanical Properties of Advanced Structural Alloys for Radiation Service

Introduction

One of the many challenges associated with developing advanced sources of nuclear energy is overcoming the deleterious effects of radiation damage on advanced fission and fusion structural materials. In order to accomplish this, a better understanding is needed of the radiation-induced degradation of the microstructures and mechanical properties of these materials.

The University of California, Santa Barbara (UCSB) is addressing this issue in collaboration with ATR NSUF at INL. The irradiation phase of the project was completed in 2010, and researchers began developing the equipment and procedures necessary for post-irradiation examination (PIE) of the specimens in 2011. The initial PIE will provide a comprehensive database that will lead to a greater understanding of radiation damage in structural alloys. It will also support the development of models that can predict the combined effects of unirradiated materials, as well as the changes in the microstructures and constitutive properties brought on by irradiation.

Project Description

The objective of this project is to create a large library of irradiated alloy sample types and conditions that has not existed before. It will consist of some 49 structural steels, including:

- Tempered martensitic steels
- Nanostructured ferritic alloys
- Stainless steel
- Model alloys, including iron-chromium (Fe-Cr) binary alloys and manganese-molybdenum-nickel (Mn-Mo-Ni) bainitic reactor pressure vessel (RPV) steels
- Specimens targeting specific irradiation damage mechanisms.

Irradiating 49 separate materials side-by-side, under a many different conditions, presents a unique opportunity that will greatly facilitate the identification, understanding, and modeling of materials used in nuclear energy systems, and will ultimately aid in predicting and improving the behavior of these materials.

The database will contain hardening and softening phenomena caused by irradiations from 1.5 to 6 displacements per atom (dpa) at temperatures from ≈ 290º C to 750º C. Mechanical property changes will be assessed through microhardness measurements and instrumented shear punch tests. In addition, tensile tests will be carried out on a subset of alloys. Fracture studies using compact tension samples, in the framework of the Master Curve method, will also be conducted on a subset

Contemplating plant life extension for reactor pressure vessels of up to 80 years requires rigorous proof that these vessels can maintain very large safety margins.

of alloys to measure irradiation embrittlement. These tests will be further supplemented by mini-bend bar fracture tests.

Model alloys will be used to study fundamental damage mechanisms, such as 0%-to-18% chromium (Cr). Iron-chromium (Fe-Cr) binary alloys, irradiated over a wide range of temperatures, will be used to study alloying effects on microstructures and hardness. Multi-constituent diffusion multiples, a.k.a. “lab-on-a-chip” specimens, will enable characterization of how various elements in complex alloys under irradiation migrate and arrange themselves in different phases. Finally, in-situ helium implantation studies will be conducted. Based on detailed characterization studies, state-of-the-art tools will be used to relate the mechanical property tests of the irradiated materials to microstructural evolutions.

The majority of the approximately 1,380 specimens to be tested are disc multi-purpose coupons. The list of experiments includes:

- Microhardness
- Shear punch
- Neutron scattering
- Transmission electron microscopy
- Positron annihilation
- X-ray scattering and diffraction
- Atom probe tomography.

The specimen matrix also includes the following specimen types:

- Sub-sized tensile
- Disc compact tension fracture
- Deformation and fracture mini-beam
- Chevron notch wedge fracture
- Cylindrical compression.

Accomplishments

The ATR-1 irradiation began at INL in 2009, and was completed in June 2010. Thirty-two isothermal temperature packets were irradiated at seven temperatures ranging from ≈ 290º C to 750º C. Thirty-one packets were irradiated for four cycles. One packet (6A) was removed after one cycle of irradiation to 1.7 dpa at 290º C. It was

“We included the RPV alloys in ATR-1 as model materials since the fluence is much higher than in service. This serendipitous decision turned out to be the source of a major contribution by ATR-1 to both the science and technology of nuclear energy.”

Dr. G. Robert Odette, Nuclear Engineering Professor, University of California, Santa Barbara

opened and sorted at INL’s hot cell facility. Most work in 2012 focused on the RPV steel matrix it contained. The motivation for the RPV work is outlined here, along with some very exciting and important new results.

RPV’s in nuclear power reactors are exposed to a low flux of neutrons that cause irradiation hardening and embrittlement. Contemplating a plant life extension of up to 80 years requires rigorous proof, that these vessels can maintain very large safety margins to protect against this type of damage, which increases with extended service time, or fluence (flux times time). RPV embrittlement is reasonably well understood and predicted up to the current licensed plant life of 40 years. But there are no low-flux, power-reactor data to evaluate embrittlement at 80 year extended life conditions. Accelerated (shorter time) test reactor irradiations at higher flux are one way to obtain high fluence data, but the results must be properly interpreted, based on rigorous physical models that account for the effects of the higher flux. And it is particularly important to explore the possibility of completely new embrittlement mechanisms that may only emerge at high fluence.

Current regulations reflect the strong effect of copper (Cu) and nickel (Ni) on embrittlement, associated with the rapid formation of Cu-rich precipitates (CRP) that harden the steel. However, theoretical models long ago predicted a new embrittlement mechanism associated with the formation of so-called “late blooming phases” (LBP) at high fluence, which could cause severe and unexpected embrittlement even in low Cu steels. Notably, LBP are not treated in current regulations. Experimental research by UCSB over the past decade has confirmed that LBP do exist under some conditions. The multi-faceted question now becomes: at what fluence, for what alloy compositions, at what irradiation temperatures and fluxes do they form, as well as what are their compositions and structures, and what are the relevant mechanisms of LBP precipitation?

The most notable characteristic of LBP is that they are primarily composed of nickel-silicon-manganese (Ni-Si-Mn). They are enhanced by, but do not require Cu. Since Ni, Si, and Mn are all important, purposely added alloying elements in RPV steels, and since Cu is a lower concentration impurity element, LBP present the potential for even more severe embrittlement than CRP.

The UCSB ATR-1 experiment is already playing a critical role in addressing these questions. Most significantly, doctoral research by Peter Wells found that a large volume fraction of LBP form in alloys, even with no Cu (Figure 1a). Ni-Si-Mn LBP also occur in Cu-bearing alloys after this element has essentially fully precipitated (Figure 1b).

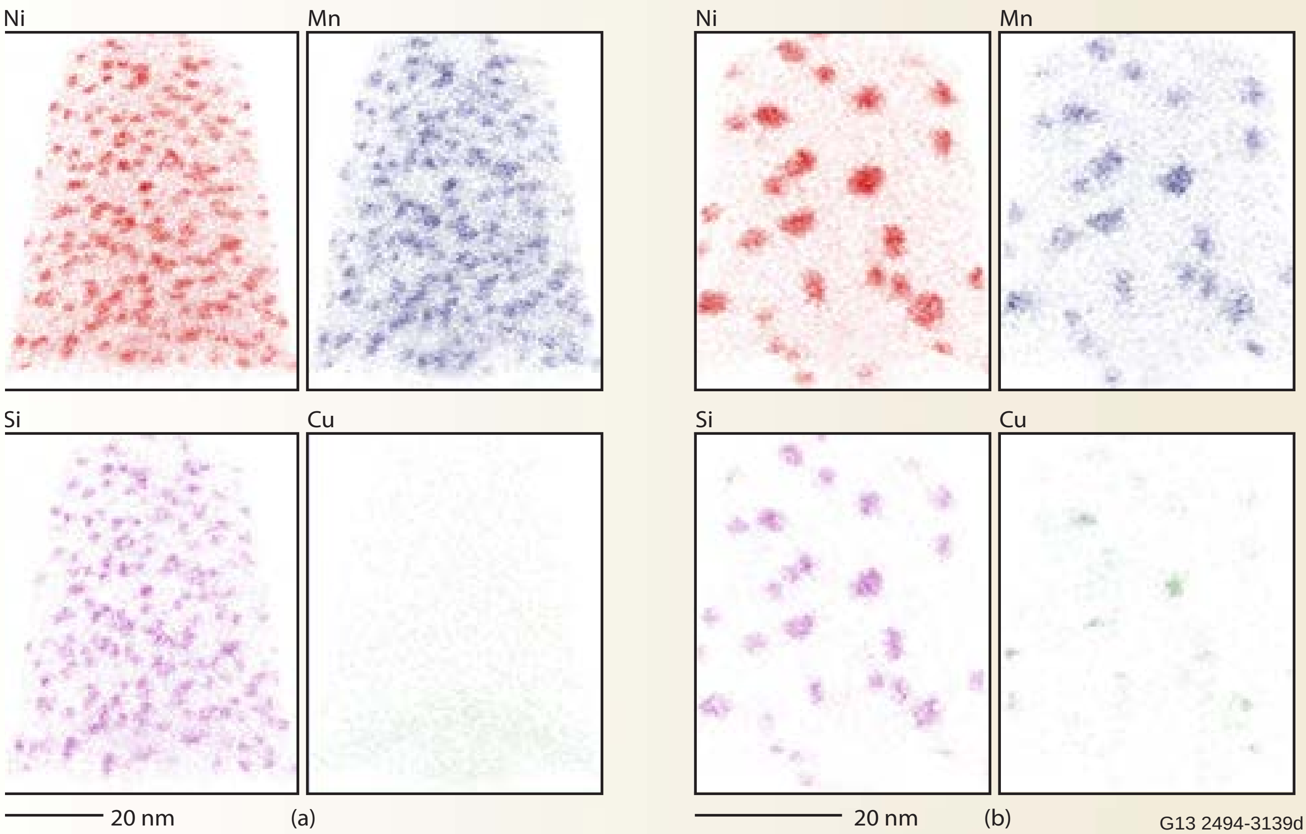

Figure 1. (a) LBP in RPV steels with nominal compositions 1.4% Mn, 0.22% Si, plus other standard alloying elements. 0.0% Cu and 0.74% Ni, and (b) 0.4% Cu and 1.25% Ni.

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Characterization of the Microstructures and Mechanical Properties of Advanced Structural Alloys for Radiation Service (cont.)

Figure 2 shows that the volume fraction of LBP increase with the alloy Cu+Mn+Ni+Si solute content.

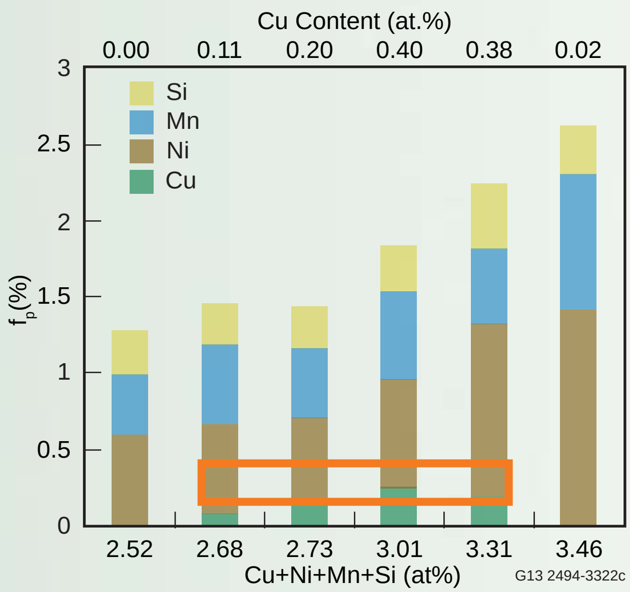

Figure 2. The volume fraction of the LBP precipitates (f_p) as a function of the total alloy Ni+Si-Cu content. The individual solute contents of the LBP are also shown. The bulk Cu composition for each alloy is shown above the graph. The orange box shows the typical CRP content of Cu-bearing steels at lower fluence, prior to the onset of LBP formation. Note that the Cu-free steels did not contain CRP.

Figure 3 shows that the large volume fraction of LBP precipitates result in up to ≈ 800 MPa hardening. The corresponding increase in the ductile-to-brittle temperature (embrittlement) would exceed an unacceptable level of more than 500º C. Notably, Figure 4 is a high-resolution transmission electron microscopy (HRTEM) diffraction pattern for a precipitate in the 1.25% Ni, 0.4% Cu alloy shown in Figure 1, that is consistent with G-phase.

These results are very significant. The UCSB ATR-1 fluence is approximately ≈ 20 times that at the end of the extended life of a typical RPV, but the ATR flux is ≈ 7,000 times higher than at the RPV. The current best estimate adjustment for the high ATR flux, compared to RPV service conditions, suggests that the low flux adjusted effective ATR fluence is only about two times that experienced by the vessel at the end of its extended life. In this case, the estimated end-of-life hardening at low RPV flux would be

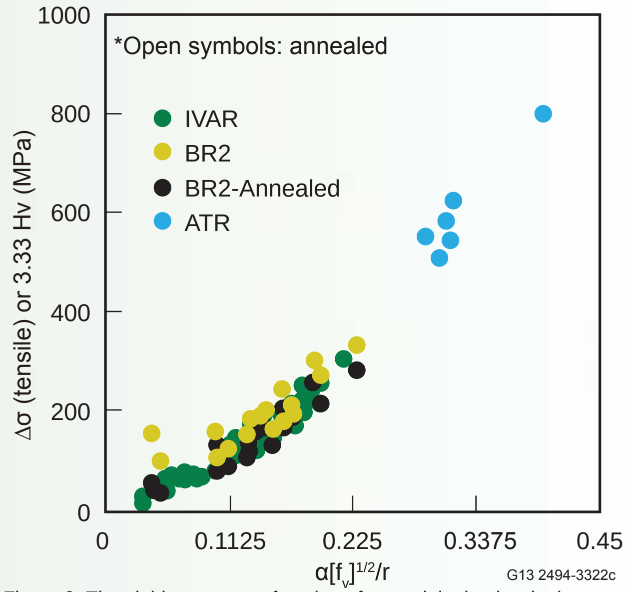

Figure 3. The yield stress as a function of a precipitation hardening parameter that scales with the $\sqrt{f_p}/r_p$ where f_p and r_p are the precipitate volume fraction and radius.

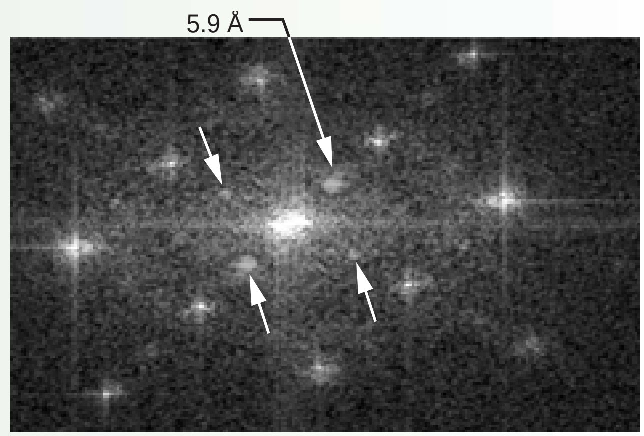

Figure 4. HRTEM FFT power spectrum diffraction pattern for a precipitate in the 1.25% Ni, 0.4% Cu alloy that is consistent with G-phase.

≈200 and 425 MPa in the low and high Cu alloys shown in Figure 1, respectively. Thus, researchers believe that the UCSB ATR-1 experiment has already provided proof in principle that LBP must be considered in developing new low flux-high fluence embrittlement models.

Future Activities

There are still many questions about LBP and how to treat flux effects. The UCSB ATR-2 irradiation, which specifically focuses on RPV embrittlement at lower, less accelerated flux than UCSB ATR-1, will help answer these questions. Meanwhile, UCSB ATR-1 is proving to be an invaluable resource for RPV life extension studies. PIE and mechanical testing on the RPV alloys are scheduled to continue in 2013.

It is also important to emphasize that the UCSB ATR-1 experiment has many objectives, and will address a very large number of unanswered questions about irradiation effects on materials. At the end of 2012, a small number of the nearly 1,400 specimens in the irradiation were available for PIE. One notable accomplishment during the year was the development of a conceptual plan to increase access to the irradiated UCSB ATR-1 alloys in 2013.

As an example of other opportunities, the 2011 UCSB ATR-1 Annual Report showed the initial results of hardness measurements on a series of binary Fe-Cr alloys. During 2012, micro-cantilever beams of one of these alloys were prepared by focused ion beam milling at CAES. The mechanical behavior of these beams will be studied in early 2013, and compared to data for charged particle irradiations. In addition, atom probe tomography (APT) samples of two of the Fe-Cr binary alloy series were also prepared at CAES. The APT studies have generated, and will continue to generate, important new scientific insight on Cr precipitation and segregation under irradiation.

Publications and Presentations

G. Robert Odette, Takuya Yamamoto, Doug Klingensmith, et al., 2011, "The Status of the UCSB ATR2 RPV Irradiation Experiments," *16th International Group-Radiation Damage Mechanisms (IGRDM), Santa Barbara, California, December 4-9, 2011.*

Distributed Partnership at a Glance	
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Los Alamos National Laboratory Stuart Maloy (collaborator)	
Oak Ridge National Laboratory Randy Nanstad, Jeremy Busby (collaborators)	
Pacific Northwest National Laboratory Richard Kurtz, Mychailo Toloczko (collaborators)	
Oxford University Chris Hardies (student collaborator)	
University of Michigan Mukesh Bachav (student collaborator)	

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A High Fluence Embrittlement Database and Advanced Test Reactor 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 plant surveillance transition temperature shift (TTS) data for the high neutron fluence and 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 and new damage mechanisms that may emerge during extended life. The USCB ATR-2 irradiation experiment was designed to address these critical issues.

Ensuring the safety of light water reactor pressure vessels.

Project Description

A special test rig for RPV steels was designed, fabricated, and inserted into INL's ATR in June 2011. The comprehensive specimen/alloy/irradiation matrix, consisting of disc multi-purpose coupons (MPC), disc compact tension fracture specimens, and sub-sized tensile specimens of 172 different RPV steels, will enable more accurate high fluence TTS predictions.

Intermediate flux ATR irradiations covering a temperature range of 250º C to 310º C, up to a fluence of 10^{20} n/cm^2, will bridge large gaps in the existing embrittlement database. The high fluence, intermediate flux database will be linked to other test reactor and surveillance data over a much wider range of flux, but generally at lower fluence. These data will be used to inform, validate, and calibrate 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 "late-blooming" phases which could cause the severe embrittlement that is 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

Irradiation will continue into early 2013, at which time the test rig will be removed, prior to a high power operating cycle. Because of unplanned power outages, the average fluence will be somewhat lower than initially planned, so a decision will be made at that time whether or not to re-insert the experiment for one to four additional reactor cycles.

In the meantime, work has continued preparing for post-irradiation examination (PIE) of the specimens in USCB ATR-2. MPC's are the predominant specimen type, and are included for all of the alloys and irradiation conditions in the experiment. Microhardness (H_v) and advanced shear punch tests (SPT) are the primary mechanical property measurements that will be conducted. In order for the results to be most useful, it is important to establish the relationship between these tests and standard tensile tests.

This subject was the topic of a UCSB M.S. Thesis completed in the fall of 2012 by Timothy Milot, entitled, "Establishing Correlations for Predicting Tensile Properties Based on the Shear Punch Test and Vickers Microhardness Data." Figure 1a shows the results of combined SPT (optimized) and tensile tests on 55 RPV alloys provided by Rolls Royce, with a wide range of strengths, plotted as the shear (τy) versus tensile (σy) yield stresses. The least square fit line has a slope of 1.77, compared to the von Mises value of $\sqrt{3}$ ($\approx$ 1.734). The measured (τy) predicts (σy) with a standard deviation of ±16 MPa. Similar intercorrelations were also developed between other SPT, tensile, and H_v properties.

The corresponding relation between H_v and the average flow stress between 0 and 10% plastic strain (σfl) is shown in Figure 1b. This is a prediction of a previously published model, shown for comparison, and the agreement is very good. Figure 1c shows the corresponding relation between the maximum load stresses from the SPT (Sm) and the ultimate tensile stress (UTS). In all of these cases, the correlations show that the H and SPT data can provide excellent predictions of the tensile test parameters.

"The multifaceted USCB ATR-2 RPV irradiation embrittlement experiment is the world's most comprehensive effort to address critical issues related to extending the life of LWR pressure vessels."

Dr. G. Robert Odette, Nuclear Engineering Professor, University of California, Santa Barbara

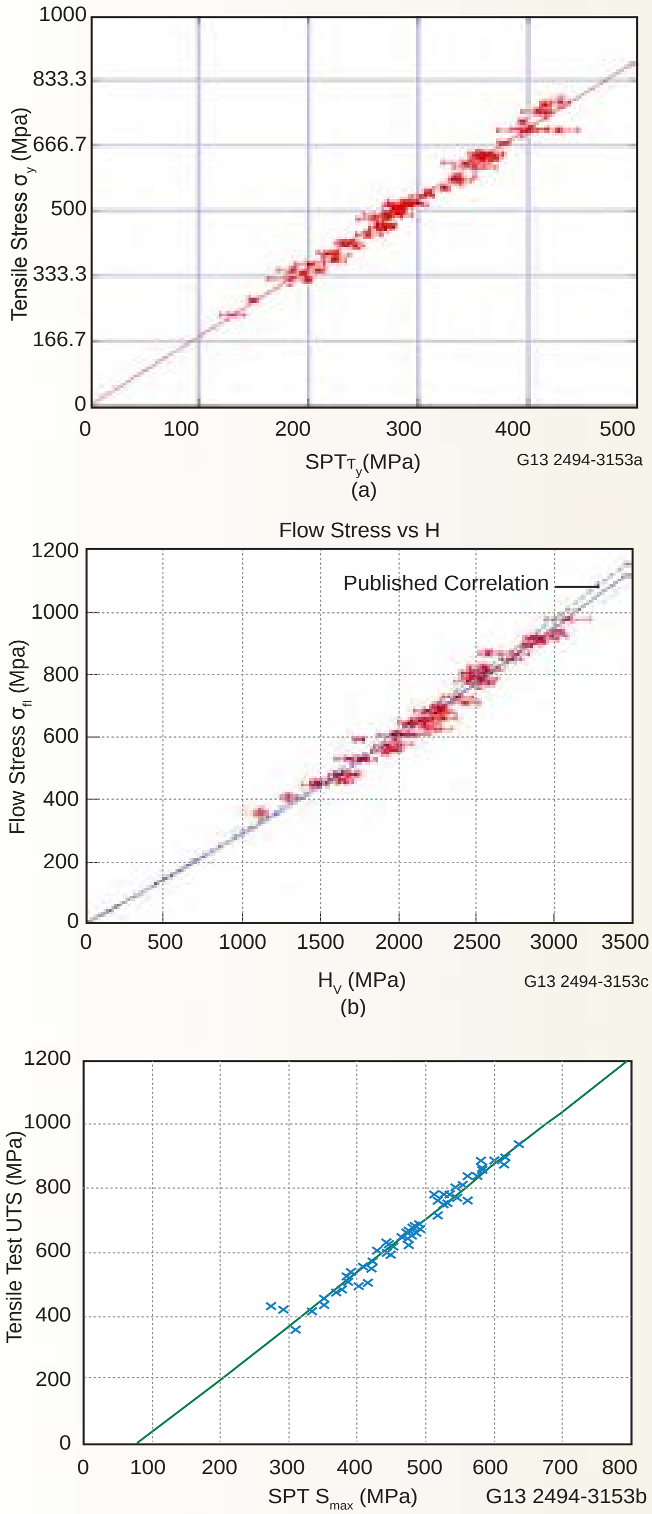

Figure 1. Plots of (a) the SPT τ_y versus the tensile test σ_y, (b) H_v versus the tensile σ_{fl}, and (c) the SPT S_m versus the tensile test UTS.

Milot's work also showed that SPT load-displacement curves can be converted to corresponding equivalent true stress-strain constitutive laws using finite element methods, as illustrated in Figure 2.

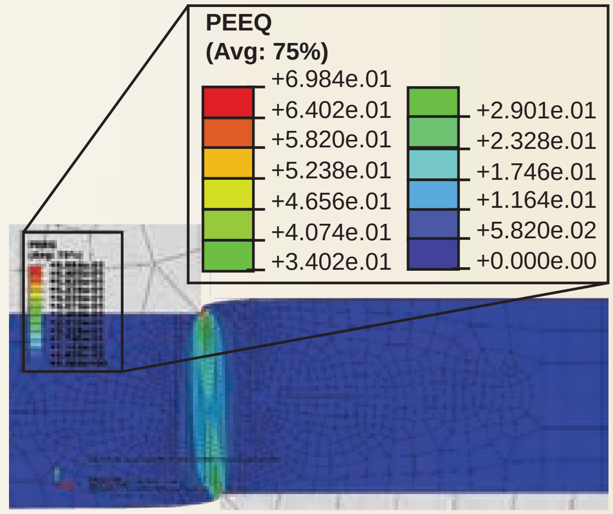

Figure 2. A finite element simulation of the shear punch tests.

Future Activities

Depending on the decision about adding cycles, the irradiation is scheduled to continue, with ongoing temperature monitoring, in 2013. PIE is expected to begin in 2014. A report on the irradiation phase of the program will be prepared at that time.

Publications and Presentations

1. Peter Wells, G. Robert Odette, Yuan Wu et al., 2012, "The Search for Late Blooming Phases: Atom Probe Tomography of Irradiated RPV Steels and Model Alloys," *2012 MRS Spring Meeting, San Francisco, California, April 9-13, 2012.*

2. G. Robert Odette, Randy K. Nanstad, Peter Wells et al., 2012, "A Brief History and Current Status of Insight on Late Blooming Ni-Mn-Si Phases in Irradiated RPV Steels," *Soteria – PERFORM60 meeting, Seville, Spain, September 17, 2012.*

3. Peter Wells, Takuya Yamamoto, Yuan Wu et al., 2012, "Late Blooming Phases in RPV Steels: High Fluence Neutron and Ion Irradiations," *NuMat 2012, Osaka, Japan, October 21-25, 2012.*

4. G. Robert Odette, Randy K. Nanstad, Peter Wells et al., 2012, "Late Blooming Phases in RPV Steels," *DOE-EPRI Meeting, October 30-31, 2012.*

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A High Fluence Embrittlement Database and Advanced Test Reactor Irradiation Facility for Light Water Reactor Vessel Life Extension (cont.)

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Team Members/Collaborators	
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Idaho National Laboratory Brandon Miller (principal investigator), Collin Knight (collaborator),	
Oak Ridge National Laboratory Randy Nanstad (collaborator)	
Bettis Laboratory Ray Stephanik (collaborator)	
Rolls Royce Marine Keith Wilford (collaborator)	
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Pilot Project for the Advanced Test Reactor National Scientific User Facility

Introduction

This ATR NSUF project consists of neutron irradiations and associated post-irradiation examinations (PIE) of a broad spectrum of structural materials that relate directly to present and future nuclear energy systems.

Irradiation samples were provided by the University of Michigan, Los Alamos National Laboratory (LANL), the University of Wisconsin (UW), Pennsylvania State University, Westinghouse Electric Company, Alabama A & M University, Oak Ridge National Laboratory (ORNL), and the Japan Atomic Energy Agency. Samples of various geometries were prepared and documented at UW and loaded into irradiation capsules at ATR in June 2008. All irradiations have been completed and post-irradiation characterization is currently in progress.

The work on proton-irradiated samples has provided students with valuable hands-on experience with advanced characterization methods.

Project Description

The project is an ambitious effort to irradiate more than 500 individual samples of a wide range of materials, including:

- The ferritic steels T91, NF616, and HCM12A
- 9%Cr oxide dispersion strengthened (ODS) steels
- Fe-9%Cr and Fe-12%Cr binary compositions (to get more fundamental insights into the radiation response of ferritic steels)
- Austenitic steels and alloys, such as IN800H, D9 and NF709
- Advanced concept alloys developed at ORNL, including super 304 stainless steel and HT-UPS-AX-6
- Grain-boundary-engineered HCM12A and IN800H *(to understand the effects of grain boundary character distribution in mitigating radiation damage)*
- The ceramics ZrO_2-MgO and silicon carbide (SiC)
- The pure metals tungsten (W) and silver (Ag) *(supplied by Westinghouse)*
- Molybdenum (Mo) ODS.

Irradiations were performed at 300, 400, 500, and 700° C to doses of 3 displacements per atom (dpa) and 6 dpa. These irradiation temperatures were established through thermal modeling, by adjusting the gas-gap distance, and, experimentally, by placing SiC electrical resistivity samples in select capsules. Samples consisted of 3 mm diameter transmission electron microscopy (TEM) disks, miniature 16 mm tensile samples, and SiC rods.

Researchers are using an array of PIE techniques including: high-resolution transmission electron microscopy (HRTEM), scanning transmission electron microscopy (STEM), atom probe tomography (APT), small angle neutron scattering (SANS), local electrode atom probe (LEAP) microhardness testing, shear punch testing, and tensile testing. Mechanical properties of irradiated materials will be evaluated using microhardness, shear punch, and tensile testing.

The PIE work is being performed at the Hot Fuel Examination Facility and Electron Microscopy Laboratory at INL; CAES in Idaho Falls, Idaho; the Radiochemistry Laboratories at the University of Nevada, Las Vegas (UNLV); ORNL; SANS facilities at the National Institute of Standards and Technology; Los Alamos Neutron Science Center at LANL; and the Characterization Laboratory for Irradiated Materials at UW.

Accomplishments

Sample irradiations at INL's ATR began in September 2008. All planned irradiations have been completed, and PIE work is now in progress. Many project samples have been consigned to the ATR NSUF sample library for the benefit of other national researchers.

The Fe-9%Cr model binary alloy samples that were irradiated at 500° C to a dose of 3 dpa were investigated for radiation-induced segregation (RIS) of chromium (Cr) to the grain boundaries. Focused ion beam (FIB) lift-outs were fabricated for TEM on the CAES FEI-Quanta 3D field-emission gun (FEG) FIB. FIB lift-out samples minimize the specimen volume size, reduce the activity of the samples, and simplify the logistics of transporting and handling the samples.

The FIB lift-out specimens were examined using the CAES FEI Tecnai F30 FEG-S/TEM, which provided two-dimensional images of the Cr distribution near and on the grain boundaries. Eleven grain boundaries were investigated for RIS following neutron irradiation. The results showed Cr enrichment increased significantly when compared to the as-received condition of all the grain boundary types. As shown in Figure 1, Cr enrichment varied depending on the grain boundary type. The investigation also found the RIS response was dependent on the local structure of the grain boundary. RIS behavior at different grain boundary types can be attributed to how point defects created by radiation damage migrate to and annihilate at grain boundaries. The results correlate with

"The ATR NSUF program is an excellent national resource for the study of reactor materials and fuels, state-of-the-art advanced structural characterization of materials, and educational activities."

Dr. Kumar Sridharan, Distinguished Research Professor, University of Wisconsin

the trends in RIS observed in ion irradiation experiments conducted at UW.

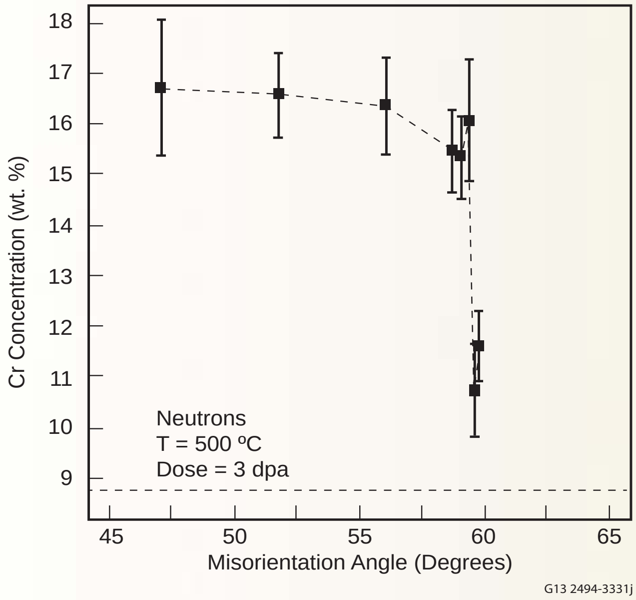

Figure 1. On-boundary Cr concentration as a function of misorientation for high-angle grain boundaries near the Σ3 orientation when neutron-irradiated to 3 dpa at 500° C.

PIE work on NF616 ferritic steel samples that were neutron-irradiated to 3 dpa at 500° C was performed using STEM-EDS and APT techniques, and the results were compared to their as-received counterparts. Data from both PIE methods showed no Cr enrichment at the grain boundaries of as-received NF616, as shown in Figure 2. However, consistent with the Fe-9%Cr model alloy, both STEM line-scan analysis and APT showed evidence of Cr segregation at the grain boundaries after neutron irradiation, as shown in Figure 3.

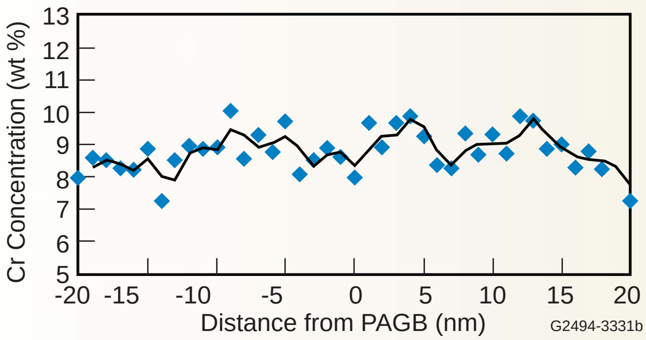

Figure 2. Concentration of Cr across a prior austenitic grain boundary (PAGB) for the as-received NF616 Cr ferritic steel sample as measured by STEM.

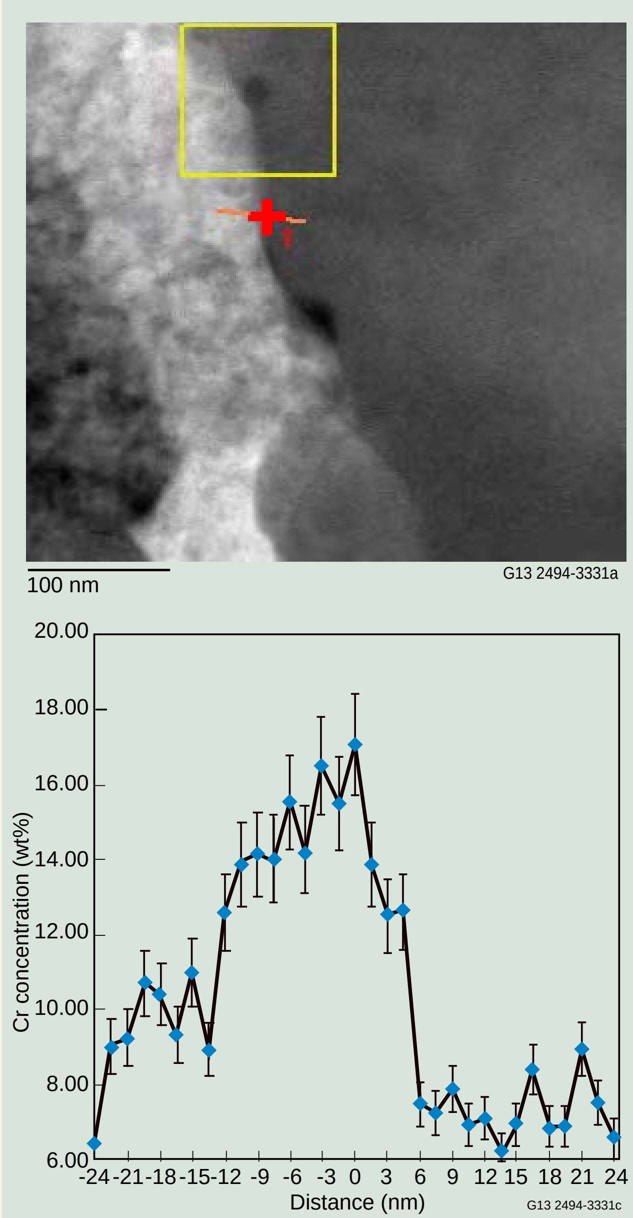

Figure 3. STEM image of a PAGB of NF616 steel irradiated at 3 dpa and 500° C (top) and a corresponding line scan for Cr composition across PAGB (bottom).

In addition, both techniques showed Cr enrichment at the grain boundaries on the order of 7% to 8% wt% Cr. Proton irradiations under similar conditions (3 dpa and 500° C) also showed some evidence of segregation at the grain boundaries, but to a lesser extent (0.5%) than the neutron irradiation.

In PIE studies performed on HCM12A ferritic steel that was neutron-irradiated to 3 dpa at 500° C using the APT

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Pilot Project for the Advanced Test Reactor National Scientific User Facility (cont.)

technique, nickel/silicon/manganese-rich (Ni/Si/Mn), copper-rich (Cu) and Cr-rich precipitates were observed. Similar precipitates were also observed in HCM12A steel samples that were proton-irradiated at 500° C. However, precipitate size, density, and volume fraction in the neutron-irradiated samples were considerably different from those irradiated by protons at the same temperature (500° C). Rather, their properties were similar to those samples proton-irradiated at 400° C. Figure 4 shows the precipitate size, volume fraction, and density of various precipitates found in proton-and neutron-irradiated samples.

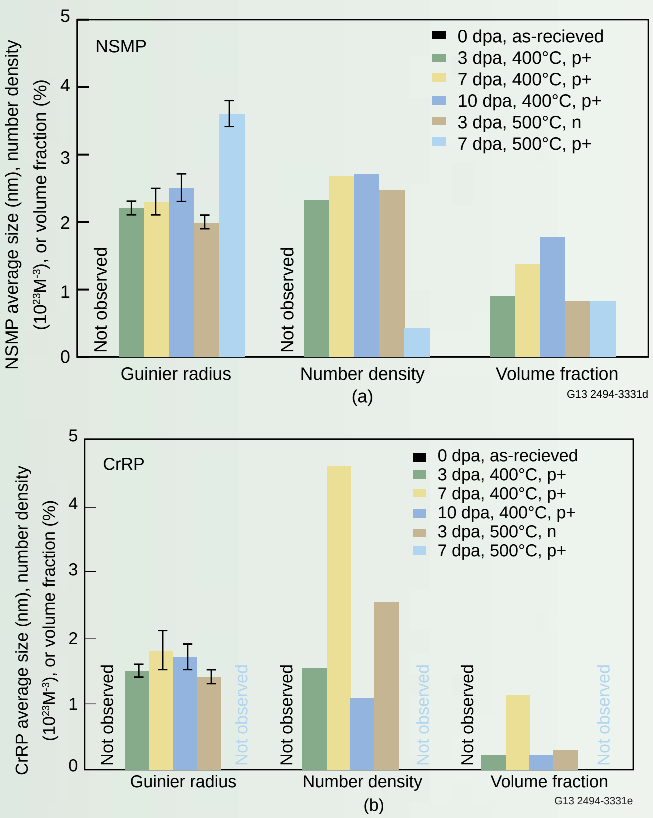

Figure 4. Average precipitate size, number density, and volume fraction of (a) Ni/Si/Mn-rich precipitates, and (b) Cr-rich precipitates in HCM12A ferritic steels formed under various irradiation conditions.

Work has continued successfully on neutron-irradiated ODS 9%Cr steels irradiated at ATR. Energy-filtered transmission electron microscopy (EFTEM) was used successfully to image clusters 2 nm or larger, while APT was required for imaging clusters smaller than 2 nm. ODS steel samples previously irradiated in ATR to a dose of 3 dpa at 500° C were prepared for TEM and APT analysis. Results from APT on the ODS steel specimen neutron-irradiated to 3 dpa at 500° C showed yttrium-titanium-oxygen (Y-Ti-O) cluster stability with no change in the average radius or number density of the clusters before and after irradiation. Figure 5 shows a typical region of nanoclusters after neutron irradiation.

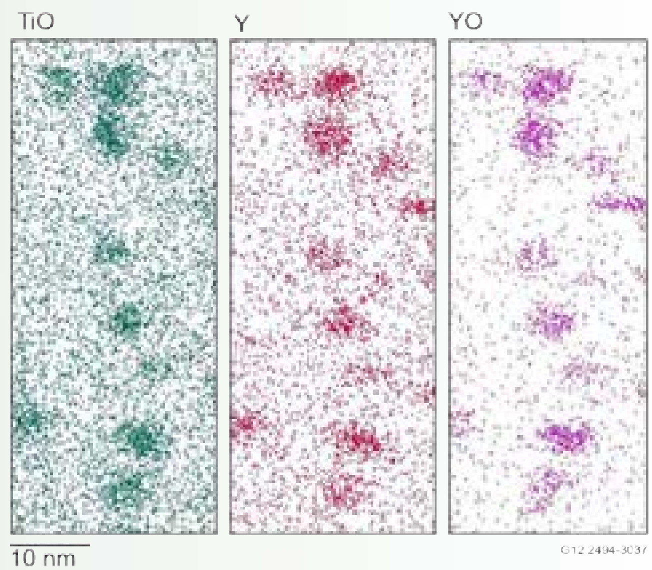

Figure 5. 2D atom probe map of 9CrODS steel neutron-irradiated to 3 dpa at 500° C.

The TEM specimens prepared at CAES were shipped to UNLV for analysis. EFTEM element maps revealed the Y-Ti-O clusters and, in agreement with the APT results, showed cluster stability after irradiation. An example of the primary EFTEM imaging of neutron-irradiated ODS steel is shown in Figure 6.

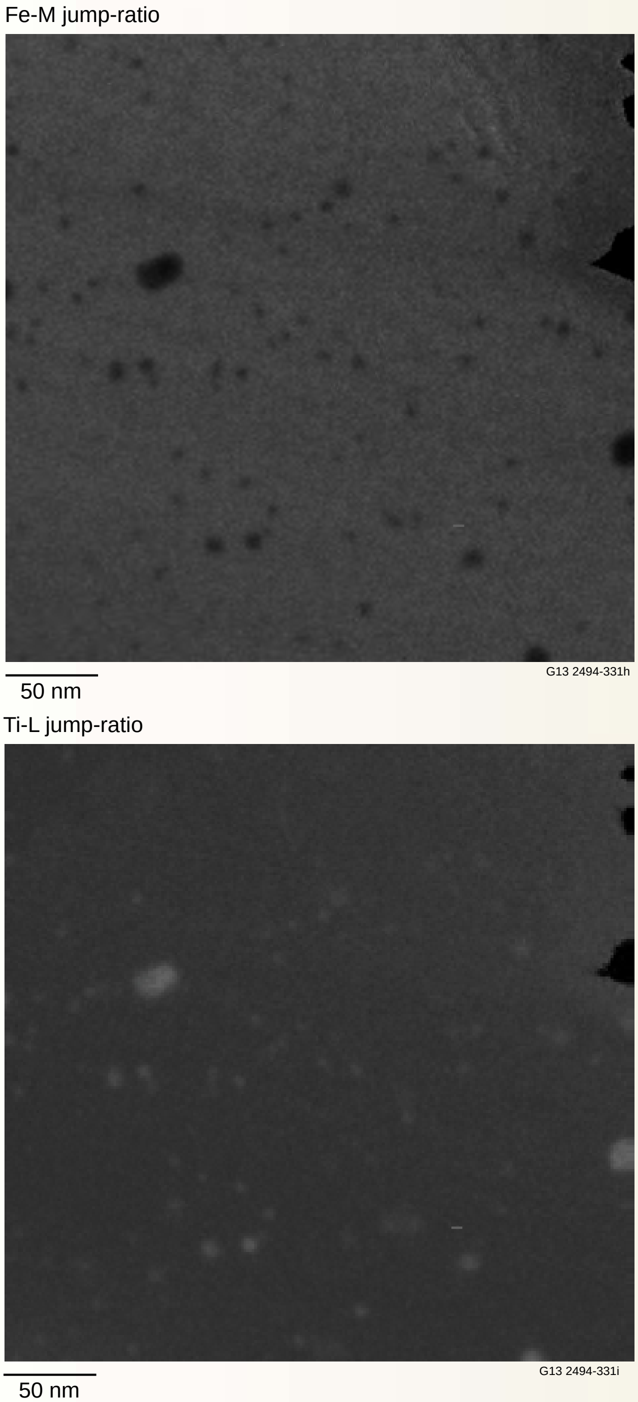

Figure 6. F/M jump-ratio and Ti-L23 jump-ratio images of 9CrODS steel neutron-irradiated to 3 dpa at 500° C. These types of images were primarily used for the nanocluster measurements.

Thermo-mechanical treatment (TMT) is a popular approach to improving the properties of metals and alloys, so researchers included the TMT-processed alloy 800H austenitic samples in the pilot project. Microstructural evolution of these materials, which were irradiated at 500° C to 3 dpa, have been characterized using TEM.

The rel-rod technique was used to image the radiation-induced Frank loops. In alloy 800H samples, the number and size of the loops in the as-received, solution-annealed (SA) and TMT samples were statistically counted and measured. The results are summarized in Figure 7.

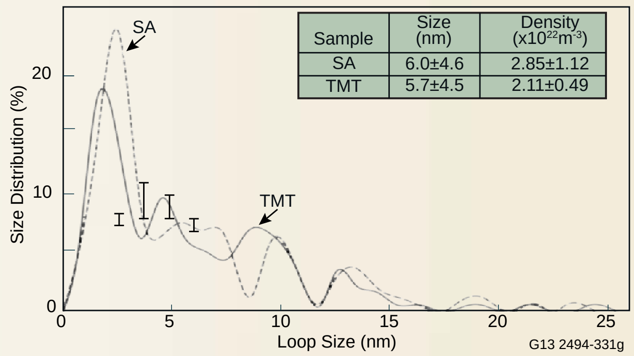

Figure 7. Statistically quantified size distribution of Frank loops in the alloy 800H under SA and TMT conditions.

Since the TMT samples have smaller loop sizes than the SA samples, the size distribution profile of the TMT samples shifts slightly to the left. The average loop sizes of the samples were fairly similar—5.7±4.5 nm and 6.0±4.6 nm for the TMT and SA samples, respectively. The TMT samples had a slightly lower volumetric number density [$(2.11\pm0.49)\times10^{22}$ m^{-3}] compared to the SA samples [$(2.85\pm1.12)\times10^{22}$ m^{-3}]. Some voids were observed in the alloy 800H samples under both SA and TMT conditions. The average size of the voids in the TMT samples (4.0±1.3 nm) was slightly larger than that in the SA samples (3.1±1.8 nm). However, the density of the voids in the SA samples, [$(1.9\pm1.1)\times10^{22}$ m^{-3}] was about 16 times greater than that in the TMT samples, [$(1.2\pm0.9)\times10^{21}$ m^{-3}].

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Pilot Project for the Advanced Test Reactor National Scientific User Facility (cont.)

Many nanoprecipitates like Ti(C, N) were introduced in-matrix by the TMT. These precipitates became stable after the 3 dpa irradiation at 500° C. Figure 8 shows an example of a Ti(C, N) nanoprecipitate that is still crystalline and coherent with the matrix after irradiation. These types of precipitates were not observed in the SA samples after the 3 dpa irradiation. Type $M_{23}C_6$ precipitates were commonly observed in the unirradiated samples. They usually had an oval shape with an aspect ratio of ~0.4 at their general boundaries. However, twin boundaries had an aspect ratio of ~0.1.

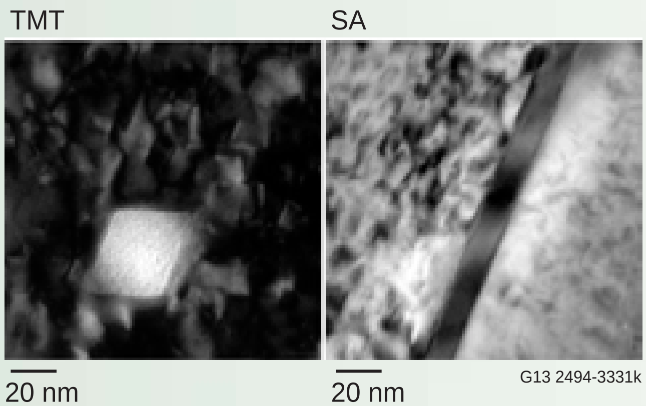

Figure 8. Bright field images of Ti(C,N) (left) and grain boundary $M_{23}C_6$ (right) observed in alloy 800H samples in TMT and SA conditions, respectively, irradiated to 3 dpa at 500° C.

Several irregularly shaped $M_{23}C_6$ precipitates were also observed in the irradiated SA samples (Figure 8), while a thin (~13 nm) $M_{23}C_6$ precipitate continuously covered a general boundary. These $M_{23}C_6$ precipitates were not observed in the unirradiated samples, thus, the radiation may have spurred the growth or formation of $M_{23}C_6$ at the grain boundaries.

Dark field images using g = 020 of the near-zone [001] axis condition were used to reveal γ'-type—e.g., Ni_3(Ti, Al)—precipitates in both the SA and TMT samples. Figure 9 shows that a larger population of γ' precipitated in the irradiated SA samples [$(1.46\pm0.23)\times10^{22}$ m^{-3}] than in the irradiated TMT samples [$(4.9\pm0.3)\times10^{21}$ m^{-3}]. However, the sizes of the γ' was similar in both the SA and TMT samples.

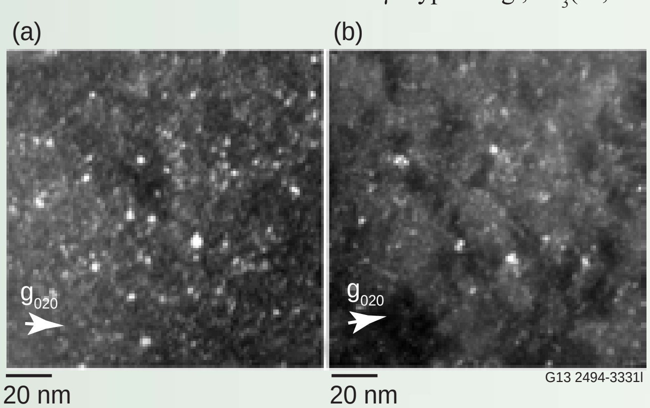

Figure 9. (a) Dark field images of radiation-induced γ' precipitates in alloy 800H after irradiation to 3 dpa at 500° C in SA, and TMT (b) samples.

In summary, TMT significantly reduced the formation of γ' precipitates and voids in the irradiated samples, leading to a ~40.9% reduction in radiation hardening compared to the SA samples. TMT also slightly reduced the formation of Frank loops. The irradiation did not alter the nanocrystalline nature of Ti(C,N) in the TMT samples, but may have encouraged the growth or formation of $M_{23}C_6$ at the grain boundaries of both the SA and TMT samples.

Future Activities

Work in 2013 will focus on detailed characterization of T91 ferritic samples irradiated at 500° C to a dose of 3 dpa. In the longer term, characterization techniques such as EFTEM, APT, and others described in this report will be used to study samples of ODS steels, 9%Cr Fe-Cr ODS and 12%Cr Fe-Cr binary model alloys, NF616, and HCM12A ferritic steels irradiated up to 6 dpa at 500° C. This will result in a trend line demonstrating how radiation damage is a function of dose.

Mechanical testing of neutron-irradiated samples and their as-received counterparts will begin at MFC at INL and will include microhardness, shear punch, and tensile tests.

Publications and Presentations

1. Kevin Field, 2012, "Understanding the Microchemical Response of Irradiated Interfaces in Ferritic/Martensitic Steels for Advanced Steel Development," *Weinberg Fellowship Seminar, Oak Ridge, Tennessee, October 9, 2012.*

2. Kevin Field, Chad Parish, Jeremy Busby, Todd Allen, 2012, "Relationship Between Grain Boundary Structure and Radiation Induced Segregation in Ferritic/Martensitic Steels," *American Nuclear Society Annual Meeting – Nuclear Fuels and Structural Materials, Chicago, Illinois, U.S.A, June 2012.*

3. Kevin Field, Leland Barnard, Chad Parish, Jeremy Busby, Dane Morgan, Todd Allen, 2012, "Role of Grain Boundary Structure on Radiation Induced Segregation in a Model Ferritic/Martensitic Steel," *Materials Research Society Meeting, San Francisco, California, Spring 2012.*

4. Alicia Certain, Satyanarayana Kuchibhatla, Vaithiyalingam Shutthanandan, Todd Allen, David Hoelzer, 2012, "Study of Nanocluster Stability Nano-featured ODS Steels Under High Dose Ion Irradiation and Low Dose Neutron Irradiation," *Transactions of the Metallurgical Society, Orlando, Florida, 2012.*

5. Lizhen Tan, Jeremy Busby, Heather MacLean Chichester, Kumar Sridharan, Todd Allen, 2012, "Microstructural Effect on Neutron Irradiation Response of Austenitic Alloy (Fe-21Cr-32Ni)," submitted to *Journal of Nuclear Materials*.

6. Lizhen Tan, Jeremy Busby, Heather MacLean Chichester, Kumar Sridharan, Todd Allen, 2012 "Microstructural Effect on Neutron Irradiation Response of Alloy 800H," *Transactions of the American Nuclear Society,* 106 (2012) 1331-1332.

7. Zhijie Jiao, Gary Was, Todd Allen, Dane Morgan, Arthur Motta, Brian Wirth, 2012, "Microstructure and Property Evolution in Advanced Cladding and Duct: Materials under Long-Term and Elevated Temperature Conditions," *Transactions of the American Nuclear Society, Chicago, Illinois, June 2012.*

8. Zhijie Jiao, 2012, "High Dose Microstructures in Ferritic-Martensitic Alloys," "Materials and Fuels for Current and Advanced Nuclear Reactors," *The Metallurgical Society Conference, Orlando, Florida, March 2012.*

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor
Center for Advanced Energy Studies	Microscopy and Characterization Suite
University of Wisconsin	Characterization Laboratory for Irradiated Materials
University of Nevada, Las Vegas	Radiochemistry Laboratories

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University of Michigan
Gary Was, George Jiao (collaborators)

Pennsylvania State University
Arthur Motta (collaborator)

University of Nevada, Las Vegas
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Japan Atomic Energy Agency
Shigeharu Ukai (collaborator, presently at Hokkaido University)

National Institutes of Standards and Technology
John Barker (collaborator)

9. Yina Huang, 2012, "RIS Study for 9Cr Model Alloy," *Consortium on Cladding and Structural Materials, Pennsylvania State University, University Park, Pennsylvania, 2012.*

10. Yong Yang, 2012, "Proton vs. Neutron Irradiations for Metals and Ceramics," *Nuclear and Radiological Engineering Seminar*, *Ohio State University, Columbus, Ohio, October 2012.*

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Nanocluster Stability of ODS Steel Under Irradiation as Determined by Atom Probe Tomography

Oxide dispersion strengthened steels are promising materials for nuclear systems. As it is with all material research in the field, the question is, can they withstand the intense environment?

Introduction

Ferritic/martensitic (F/M) steels generally provide better swelling resistance under irradiation than austenitic steels but have poor creep strength at temperatures over 600° C. To resolve this issue, oxide dispersion strengthened (ODS) F/M steels have been developed as candidate materials for cladding and structural components in fission and fusion reactors. Their nanoscale yttrium-titanium-oxide (Y-Ti-O) clusters act as pinning points for dislocations, improving the steel's high-temperature creep strength. The nanoclusters are also expected to promote the recombination of irradiation-produced point defects and trap transmutation-produced helium (He) in small, high-pressure bubbles, further strengthening the material.

Since the nanoclusters contribute to the strength of ODS steel, understanding the stability of the nanoclusters under irradiation is an important issue.

Project Description

The overall objective of the rapid turnaround experiment is to assess the stability of the Y-Ti-O nanoclusters under irradiation. Because the relatively small size of the nanoclusters (2–5 nm) makes analysis difficult, a multifaceted approach is necessary for a complete evaluation. For imaging clusters larger than 2 nm, researchers use energy-filtered transmission electron microscopy (EFTEM), while clusters smaller than 2 nm require atom probe tomography (APT).

The Microscopy and Characterization Suite (MaCS) at the CAES was an essential component of this project. Researchers used its APT and TEM facilities to prepare and analyze samples of neutron-irradiated ODS steel through focused ion beam (FIB) milling and the local electrode atom probe (LEAP), respectively.

Accomplishments

Samples of 9CrODS steel previously irradiated in ATR to a dose of 3 displacements per atom (dpa) at 500° C were prepared for TEM and APT analysis using an FEI Helios FIB instrument in MaCS. The small size of the available FIB samples drastically reduced the effects of magnetism on the TEM analysis. It also significantly reduced the activity of the samples, making handling easier and safer.

The APT specimens were characterized by an Imago Scientific Instruments LEAP 4000X HR at INL. Samples were run in laser mode at a temperature of -65° C and a laser power of 50 petajoules (pJ). Results of the APT on the 9CrODS steel specimen that had been neutron-irradiated to 3 dpa at 500° C in ATR nanocluster showed Y-Ti-O cluster stability with no change in the average radius or number density of the nanoclusters before and after irradiation. Figures 1 and 2 show a typical area of

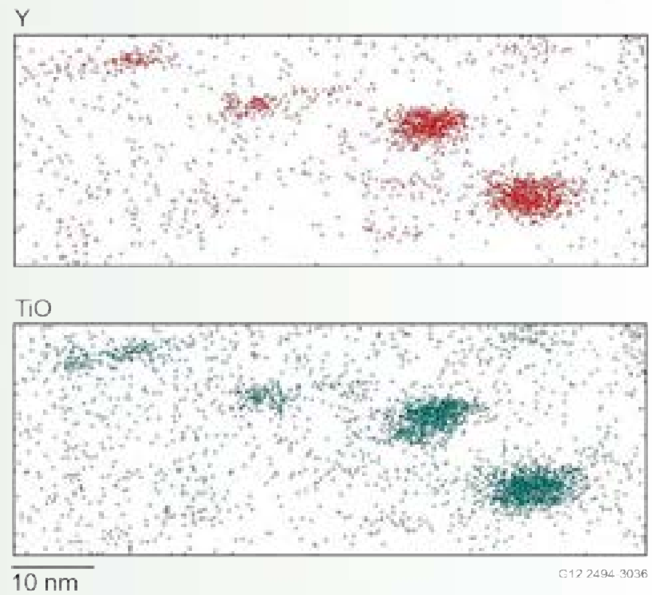

Figure 1. 2D APT map of unirradiated 9CrODS. steel.

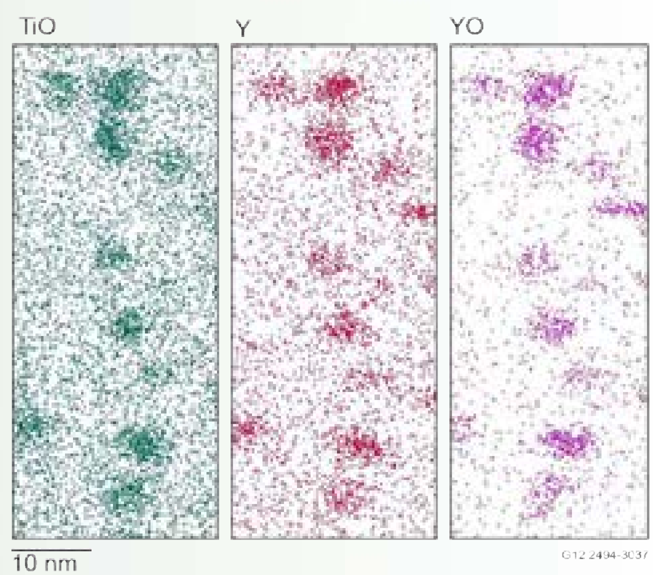

Figure 2. 2D APT map of 9CrODS steel neutron-irradiated to 3 dpa at 500° C.

"Advanced high resolution electron microscopy coupled with nano-scale chemical and grain boundary character analyses capabilities are allowing for an unprecedented fundamental understanding of irradiation-induced elemental segregation at grain boundaries which can have a profound effect on the mechanical and corrosion properties of materials."

Dr. Kumar Sridharan, Distinguished Research Professor, University of Wisconsin

nanoclusters pre- and post-irradiation, respectively. The specimens prepared at CAES were shipped to the University of Nevada, Las Vegas for TEM. EFTEM element maps revealed that the Y-Ti-O showed cluster stability after irradiation, confirming the APT results. Figure 3 shows an example of the types of element maps used.

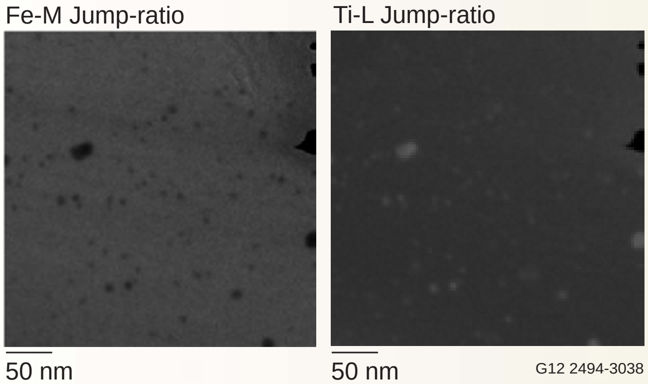

Figure 3. Fe-M jump-ratio and Ti-L23 jump-ratio images of 9CrODS steel neutron-irradiated to 3 dpa at 500° C. These types of images were primarily used for the nanocluster measurements.

Future Activities

This project's future research includes continuing work on similar alloys irradiated in ATR to different doses and different temperatures.

Publications and Presentations

Alicia Certain, Satyanarayana Kuchibhatla, Vaithiyalingam Shutthanandan, Todd Allen, David Hoelzer, 2012, "Study of Nanocluster Stability Nano-featured ODS Steels Under High Dose Ion Irradiation and Low Dose Neutron Irradiation," *Transactions of TMS 2012, Orlando, Florida.*

Distributed Partnership at a Glance	
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Team Members/Collaborators	
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Grain Boundary Microchemistry of Ion-irradiated Ferritic/Martensitic Steels as Determined by Advanced Microscopy Techniques

Introduction

Due to their excellent thermal properties and resistance to swelling under irradiation, ferritic/martensitic (F/M) steels with a 9 wt. % chromium (Cr) addition show potential as structural and cladding materials in nuclear reactors. However, F/M steels also have shown possible radiation-induced segregation (RIS) of Cr to grain boundaries. This could hamper their in-service performance, even though current studies show no conclusive trends in Cr behavior at the grain boundaries themselves of F/M steels. To facilitate the implementation of F/M steels in future nuclear reactor applications, the RIS behavior in F/M steels must be better understood.

To facilitate the implementation of F/M steels in future nuclear reactor applications, the RIS behavior of these materials must be better understood.

Project Description

The objective of this rapid turnaround experiment (RTE) is to increase the current state of knowledge of RIS in F/M steels. This will be accomplished by examining the RIS behavior of a 9 wt. % Cr model steel at varying degrees of damage (dose) at the grain boundaries of different structures. This project also serves as a precursor to a rigorous post-irradiation examination (PIE) of neutron-irradiated specimens that were part of the University of Wisconsin's initial project at ATR NSUF.

It has been predicted that grain boundaries with specific types of structure will be stronger sinks for point defects created during irradiation than other types of boundary. This results in different RIS responses from one boundary to another. This was confirmed through studies of RIS responses at different grain boundary structures using scanning transmission electron microscopy (STEM), energy dispersive X-ray spectroscopy (EDS), and Kikuchi pattern analysis.

Low-dose proton irradiations were performed at the University of Wisconsin and the University of Michigan at 1, 2, and 3 displacements per atom (dpa) at temperatures of 400° C and 500° C. Transmission electron microscopy (TEM) specimens of irradiated and as-received specimens were fabricated in the Microscopy and Characterization Suite (MaCS) at CAES using focused ion beam (FIB) techniques. The FIB reduces the volume size of TEM specimens, which, in turn, reduces the magnetic aberrations during STEM investigations. Several specimens were evaluated at CAES using its TEM/STEM equipment.

Accomplishments

Cross-sectional FIB lift-out specimens from the irradiated surface were fabricated for TEM on the FEI-Quanta 3D FEG-FIB at CAES. To achieve a relatively even dose throughout the sample, extractions of the FIB lift-outs were limited to depths of 20 µm or less. Two as-received samples and one irradiated sample were created. A STEM image of the 1-dpa-at-400° C FIB lift-out specimen is provided in Figure 1.

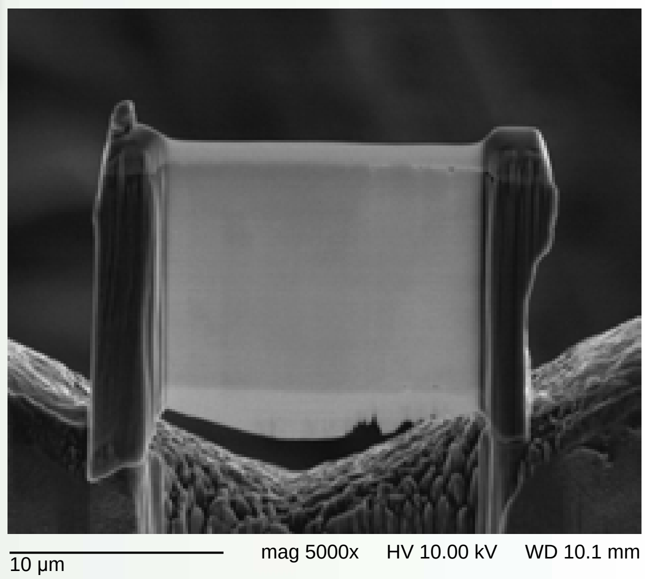

Figure 1. STEM imaging of FIB lift-out of proton-irradiated model steel.

The as-received FIB lift-outs were examined for variations in the Cr distribution (Cr is the alloying component of interest in these steels) at the grain boundaries using the scanning transmission electron microscopy coupled with energy dispersive X-ray spectroscopy (STEM-EDS) spectrum imaging technique. Spectrum imaging provides more spatial information than traditional 1D line-profile

"ATR NSUF serves as a nexus for neutron-induced radiation effects studies in the U.S."

Kevin Field, Ph.D. Student, University of Wisconsin

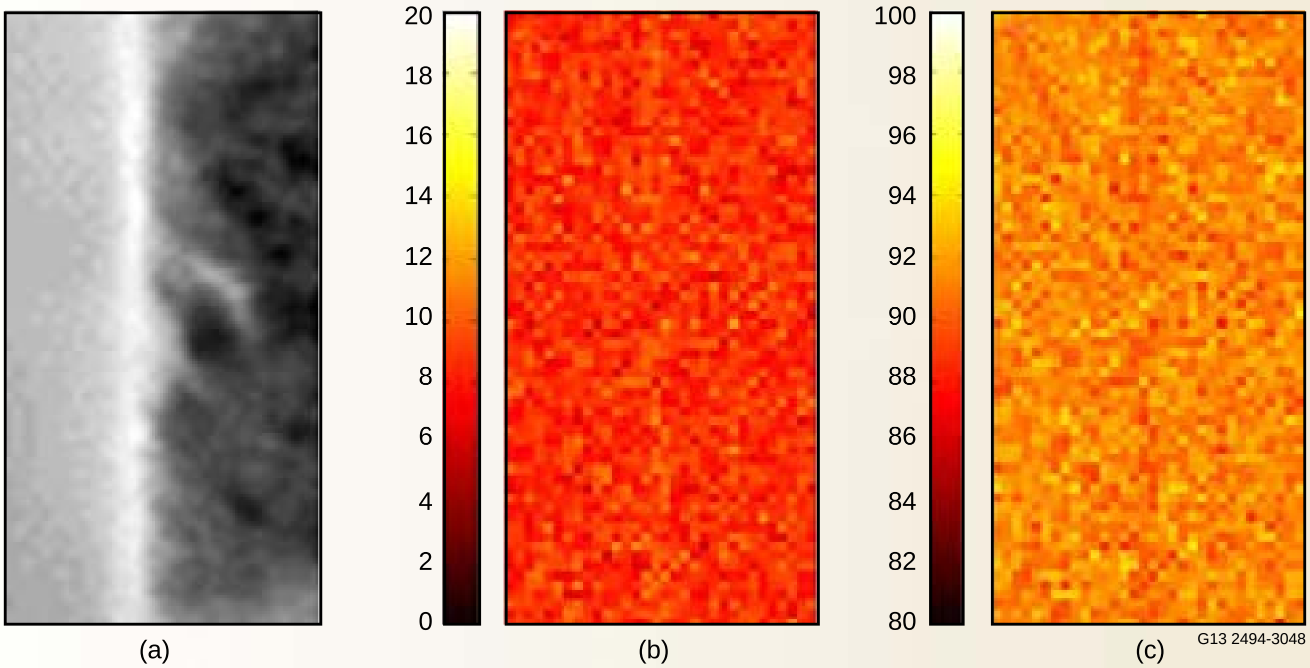

Figure 2. (a) Annular dark field image of lath boundary in as-received model steel, (b) weight percent chromium map (bulk concentration: ~8.68 wt.% Cr), and (c) weight percent iron map.

scans. For this project, the technique produced a 2D image of the Cr distribution and the near- and on-grain boundaries (Figure 2).

Spectrum images contained 32 x 64 pixels, an incident probe size of 1.5 at full width at half-maximum, a 48 x 96 nm region of interest, a 1 s/pixel dwell time, and ~1 nA incident probe current. The images were also drift-corrected and took ~40 minutes per acquisition and were post-processed using custom software that applied the Cliff-Lorimer ratio technique to create 2D concentration maps of iron (Fe) and Cr in the region of interest. Grain boundary misorientation analysis was completed using Kikuchi pattern analysis and custom indexing software.

Publications and Presentations

1. Kevin Field, C.M. Parish, J.T. Busby, Todd Allen, 2012, "Relationship Between Grain Boundary Structure and Radiation Induced Segregation in Ferritic/Martensitic Steels," *American Nuclear Society Annual Meeting-NFSM*, *Chicago, Illinois, June 24-28, 2012*.
2. Kevin Field, Leland Barnard, Chad Parish, Jeremy Busby, Dane Morgan, Todd Allen, 2012, "Role of Grain Boundary Structure on Radiation Induced Segregation in a Model Ferritic/Martensitic Steel," *Materials Research Society*, *San Francisco, California, Spring 2012*.

Future Activities

This RTE project is complete. Future research includes writing additional RTE proposals to investigate specimens irradiated at ATR NSUF using the same techniques described in this report.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Team Members/Collaborators	
University of Wisconsin Kumar Sridharan (principal investigator), Kevin Field (Ph.D. student, presently at Oak Ridge National Laboratory)	
Center for Advanced Energy Studies Jatu Burns (collaborator)	

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

Interfaces will play a significant role in the development of radiation-resistant materials with high creep strengths.

Project Description

In order to investigate interface stability under heavy ion irradiation, researchers recreated a two-dimensional interface system, emulating the interface between nanoparticles and the steel matrix of an oxide dispersion strengthened (ODS) steel. This rapid turnaround experiment (RTE) research strategy involved coating substrates of a binary alloy, Fe-12%Cr, which has the nominal composition characteristics of a widely used class of ferritic steels, with titanium (Ti) and yttrium (Y) thin films. The coated samples were then irradiated with nickel ions, and the resulting evolution of the interface between the substrate and the coating was analyzed.

Accomplishments

The Ti and Y thin films have been deposited on a Fe-12%Cr substrate and ion-irradiated using the Pelletron ion accelerator at the University of Wisconsin (UW) with 5 MeV Ni^{2+} ions to 45 displacements per atom (dpa) at two different temperatures, 300° C and 500° C. Transmission electron microcopy (TEM), scanning transmission electron microcopy (STEM), and energy dispersive X-ray spectroscopy (EDS) were conducted on the as-deposited films as well as the Ti-coated and irradiated film samples.

Diffusion and intermixing of the elements were observed across the interfaces of the as-deposited samples. Researchers observed that irradiation at 300° C enhanced diffusion, while irradiation at 500° C resulted in a significantly larger intermixed region that was characterized by an intermediate composition between the substrate and the coating. Researchers also noted that the region contained nanoprecipitates that were enriched in Ti but depleted in iron (Figure 1).

Future Activities

This RTE is complete. Future activities could include TEM and STEM-EDS analyses at UW on the Y-as-deposited, the Y-coated, and the irradiated samples. In addition, the Fe-12%Cr substrates will be coated with titanium and yttrium oxides (TiO_2 and Y_2O_3) and ion-irradiated under the same conditions. The interface region of these samples will be analyzed with TEM and STEM-EDS techniques.

Publications and Presentations

1. Alexander Mairov, Benjamin Hauch, Clarissa Yablinsky, and Kumar Sridharan, 2012, “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, 2012, “Study of Interfacial Interactions Using Thin Film Surface Modification,” presentation, *Materials Science & Technology (MS&T) Conference, Pittsburgh, Pennsylvania, October 2012.*

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor
Center for Advanced Energy Studies	Microscopy and Characterization Suite
University of Wisconsin	Characterization Laboratory for Irradiated Materials
Team Members/Collaborators	
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Los Alamos National Laboratory Jinsuo Zhang (collaborator), Benjamin Hauch (research intern)	

"Advanced high-resolution, electron microscopy techniques, such as those used in this study, provide the unprecedented detail of the physical and chemical changes at interfaces that allow us to tailor materials with enhanced resistance to radiation damage."

Dr. Kumar Sridharan, Distinguished Research Professor, University of Wisconsin

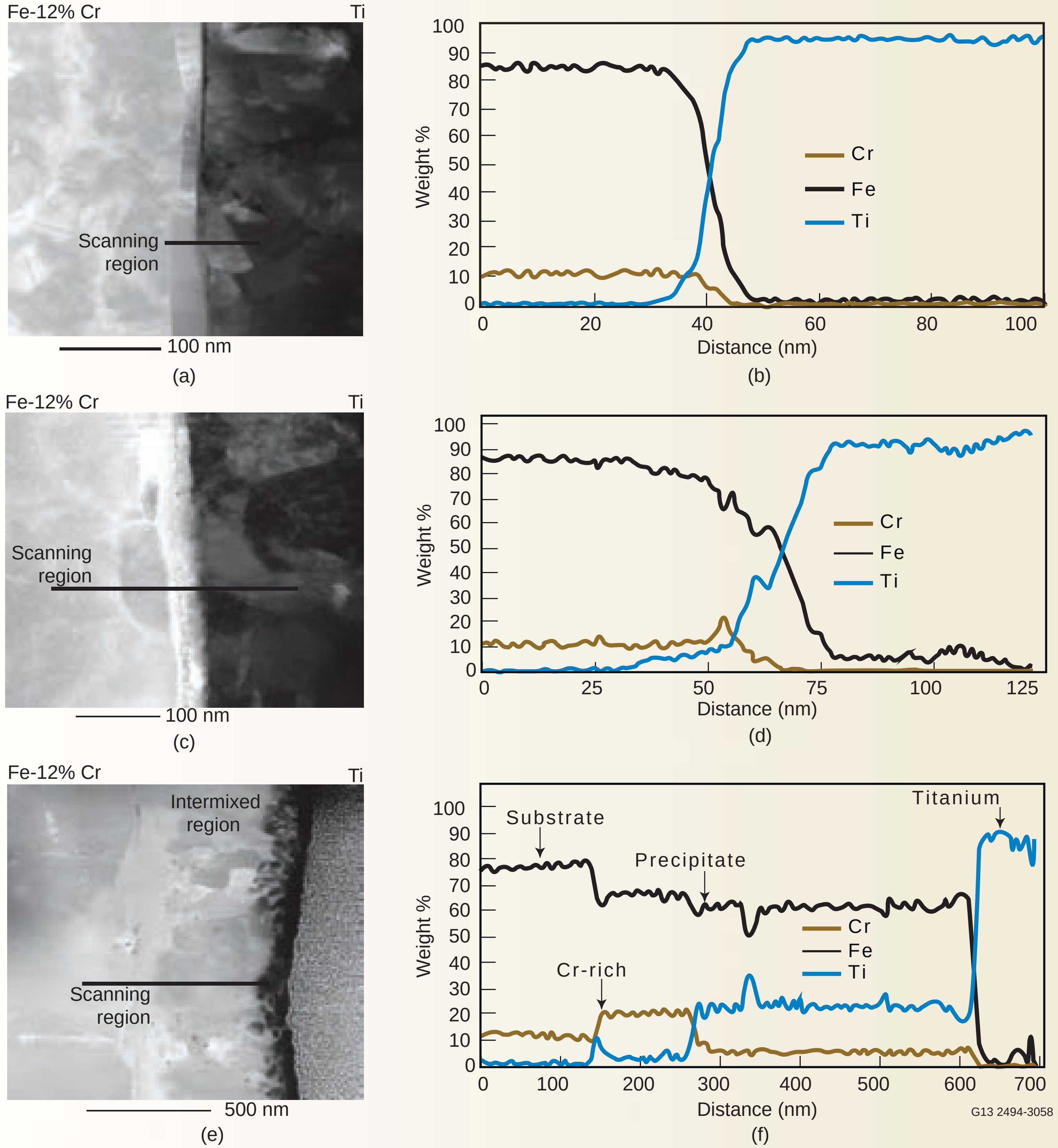

Figure 1. STEM-EDS analysis of the titanium coated samples. (a and b) slight intermixing of elements across the interface is observed in the as deposited condition, (c and d) nickel irradiation at 300° C, and (e and f) nickel irradiation at 500° C results in a wide (~600 nm) intermixed region and the formation of titanium enriched nanoprecipitates

Principal Investigator: Erich Stergar – University of California, Berkeley
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Atom Probe Tomography on Highly Irradiated Ferritic/Martensitic Steels

Introduction

One of the primary goals of the next generation of nuclear reactors is achieving high fuel burnup rates and, therefore, higher efficiencies in the reactors. Reaching this goal will require the development of materials that can resist high doses of radiation. Ferritic/martensitic (F/M) steels show considerable promise when used in cladding, ducting, and construction applications for fast reactors in the U.S. However, it is known that phase instability under neutron irradiation strongly influences the material properties of F/M steels, and can cause low-temperature embrittlement. Identifying the behavior of second-phase precipitation under the combined influences of neutron irradiation and temperature is important in understanding this characteristic.

It is important to investigate material irradiated under real reactor conditions in order to gain a deeper understanding of the underlying mechanisms.

Project Description

HT-9, a fully-tempered F/M steel with a chromium content of 12 wt%Cr, was used in the construction of a fuel test assembly known as the ACO-3 duct. The duct was previously irradiated in the Fast Flux Test Facility (FFTF) up to 155 displacements per atom (dpa) at a temperature range of 380º - 504º C. In cooperation with the Los Alamos National Laboratory (LANL) and the University of California, Berkeley (UCB), atom probe samples were extracted from three different zones along the face of the irradiated ACO-3 duct using focused ion beam microscopes. The prepared samples were shipped to CAES in Idaho Falls, Idaho, where they were analyzed in the Microscopy and Characterization Suite (MaCS) laboratory at the nanometer scale. Atom probe measurements were taken using the LEAP 4000 system (Figure 1).

The goal of this rapid turnaround experiment (RTE) was to investigate any secondary precipitates formed under the given conditions, and to identify their chemical compositions, sizes, and distribution. The data gained was combined with results from other high-resolution methods, such as transmission electron microscopy (TEM), energy filtered transmission electron microscope (EFTEM), and small angle neutron scattering (SANS).

Accomplishments

The results of this project show that second-phase precipitation is more sensitive to the temperature history than to the exposed neutron dose. It was also found that, in general, the density of both precipitates decreases with

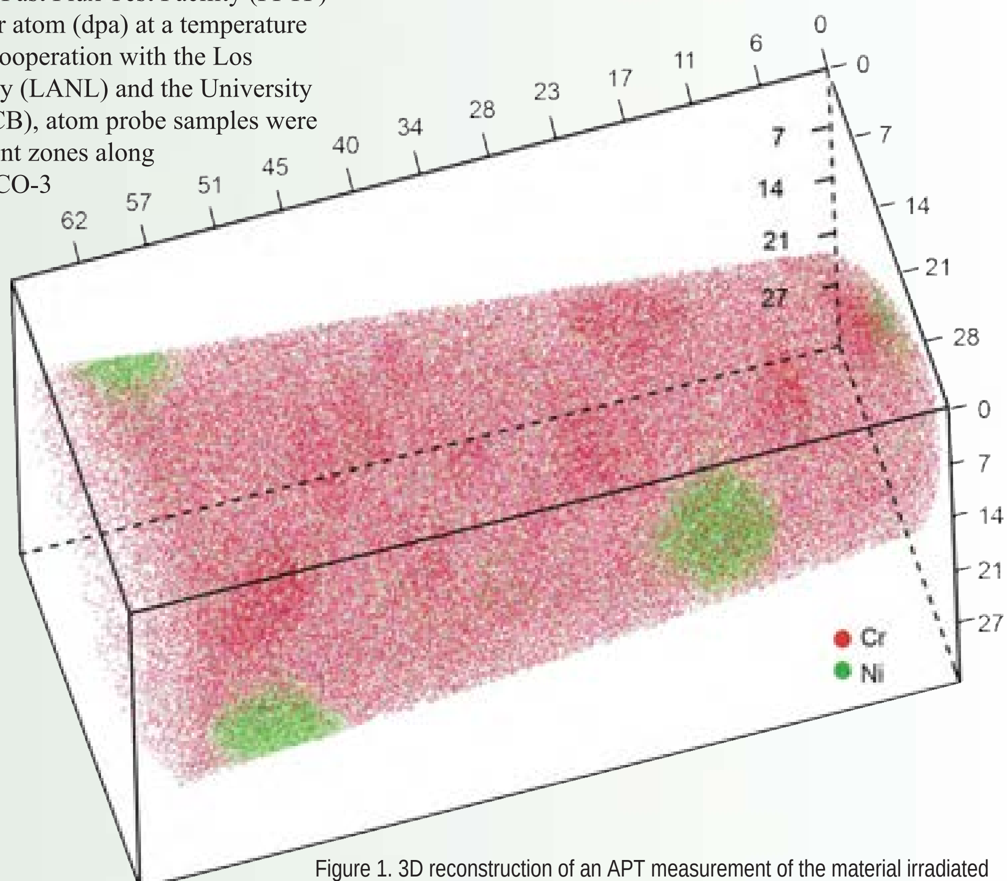

Figure 1. 3D reconstruction of an APT measurement of the material irradiated at 380º C to a dose of 20 dpa showing Cr and Ni enrichments.

"Understanding the combined influence of temperature and neutron irradiation on ferritic/martensitic steels is crucial for future reactors."

Erich Stergar, Post-doctoral Student, University of California, Berkeley

increasing irradiation temperature. Further, no significant change was observed in the average size of alpha prime, while the average size of G-phase precipitates increased at 440º C. In contrast to information previously reported in the literature, no Laves, M_6C, or Chi phases were found during this investigation.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Team Members/Collaborators	
University of California, Berkeley Erich Stergar (principal investigator), Peter Hosemann (co-principal investigator) **Los Alamos National Laboratory** Stuart A. Maloy (collaborator)	

Future Activities

The project is complete.

Publications and Presentations

Osman Anderoglu, Joris Van den Bosch, Peter Hosemann, Erich Stergar, Bulent H. Sencer, Dhriti Bhattacharyya, Rob Dickerson, Pat Dickerson, Monika Hartl and Stuart A. Maloy, 2012, "Phase Stability of an HT-9 Duct Irradiated in FFTF," *Journal of Nuclear Materials*, Vol. 430, No. 1-3, November 2012, pp. 194-204.

Principal Investigator: James F. Stubbins – University of Illinois
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Irradiation Performance of Iron-Chromium (Fe-Cr) Base Alloys

Introduction

In recent years, 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, ferritic (FeCr) alloys have become the lead candidate alloy system for a variety of advanced reactor components and applications.

Project Description

This ATR NSUF project is comprised of a coordinated set of irradiation experiments on select FeCr alloys, ranging from model alloys to commercial and developmental alloys, followed by post-irradiation examination (PIE) and analysis. The main objective of this research is to expose these alloys to a range of irradiation conditions that will provide significant new insights into their radiation performance in advanced reactor applications. It will also serve as a mechanism for developing materials modeling capabilities that will allow researchers to better predict future alloy performance and development.

Two sample geometries are included in the study: samples for transmission electron microscopy (TEM) analysis and samples for tensile tests. These specimens can also be used for atom probe and other types of materials properties experiments. The test matrix of irradiation conditions in the ATR for this project is presented in Table 1.

Accomplishments

Irradiations with low target doses of 0.01 displacements per atom (dpa) and 0.1 dpa have been performed in the hydraulic shuttle in ATR. In addition, the high-dose irradiations (0.5 dpa to 10 dpa) done in ATR were completed in June of 2011. All irradiated capsules have been transferred to the Hot Fuel Examination Facility (HFEF) at INL's MFC for unloading. Due to an MFC facility-wide stand-down where operations were suspended for several months, the unloading process in HFEF was postponed. However, by the end of 2012, four 2.3 mm discs were punched and polished from the model alloys Fe and Fe-14Cr. Those four specimens were then transferred to the CAES Microscopy and Characterization Suite (MaCS) for atom probe examinations.

Developing materials for future reactor applications.

Future Activities

PIE and analysis will start in early 2013 with the high-purity Fe and Fe-Cr alloys considered to be the highest priorities. Due to restrictions in the program budget for PIE, the investigations may initially be limited to a few lower-dose exposure conditions on the high-purity materials.

Various characterization methods will be used to investigate both microstructure and macroscopic properties, with the TEM examination being the most important technique used in this project. The TEM specimen preparations will be performed, for the most part, at MFC, and the TEM itself will be performed at MFC and at Oak Ridge National Laboratory beginning in early 2013.

Atom probe studies and nano-indentations are expected to start in October 2012 and February 2013, respectively. The atom probe offers several advantages for studying impurity segregations in atomic resolutions, while nano-indentations record the materials' mechanical properties. Both examinations will be performed in MaCS on all the specimens available.

Table 1. Text Matrix (12 materials, 3 irradiation temperatures, 6 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	TEM, Miniature tensile
Developmental Alloys: MA-957	300, 450, 550	0.01, 0.1, 0.5, 1.0, 5.0	TEM, Miniature tensile

*Single crystal materials, no minature tensile specimens

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"The ATR NSUF program provides the first real opportunity for university programs to plan and lead reactor irradiation experiments in the U.S. This adds a whole new dimension to the understanding of radiation effects in reactor materials and serves as the training ground for the students who will become the next generation of researchers in this area."

Dr. James F. Stubbins, Professor and Department Head, Nuclear, Plasma and Radiological Engineering, University of Illinois

Due to funding constraints, only a small fraction of the total number of specimens can be examined. Discussions have begun on the best, most cost-effective method to ship the tensile specimens to alternate locations for testing. Other avenues are also being explored to conduct these experiments, which will keep the full complement of planned experiments intact.

Publications and Presentations

1. Carolyn Tomchik, Jian Gan, James Stubbins, Mark Kirk, 2011, "Low Dose Neutro Irradiations of Iron and Iron-chromium Model Alloys in the ATR-NSUF," presented at the *15th International Conference on Fusion Reactor Materials*, *Charleston, South Carolina, October 2011*.

2. Weiying Chen, Yinbin Miao, Carolyn Tomchik, James Stubbins, 2013, "Low Dose Neutron Irradiations of Iron and Iron-chromium Model Alloys in the ATR-NSUF from 300," planned for presentation at the *TMS Spring 2013 meeting*, at *ICFRM* in Fall 2013*, and others*.

3. Chaitanya Deo., Srivilliputhur Srinivasan, Michael Baskes, Stuart Maloy, Michael James, Maria Okuniewski, and James Stubbins, 2008, "Kinetics of the Migration and Clustering of Extrinsic Gas in bcc Metals," *ASTM Special Technical Publication*, 1492, November 2008, pp. 177-189.

4. Xiaoqing Pan, Xiaodong Wu, Xi Chen, K. Mo, Jonathan Almer, Dean Haeffner, and James Stubbins, 2010, "Temperature and particle size effects on flow localization of 9-12%Cr ferritic/martensitic steel by in situ X-ray diffraction and small angle scattering," *Journal Nuclear Materials*, Vol. 398, No. 1-3, March 2010, pp. 220-226.

5. Xiaoqing Pan, Xiaodong Wu, Xi Chen, K. Mo, Jonathan Almer, Jan Ilavsky, Dean Haeffner, and James Stubbins, 2010, "Lattice strain and damage evolution of 9-12%Cr ferritic/martensitic steel during in situ tensile test by X-ray diffraction and small angle scattering," *Journal Nuclear Materials*, Vol. 407, No. 1, December 2010, pp. 10-15.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Team Members/Collaborators	
University of Illinois James Stubbins (principal investigator), Carolyn Tomchik (graduate student) **Idaho National Laboratory** Jian Gan (principal investigator), Maria Okuniewski (co-investigator), Greg Wachs (project engineer), Collin Knight (collaborator) **Georgia Institute of Technology** Chaitanya Deo (collaborator) **General Electric Company** Eric Loewen (collaborator) **Los Alamos National Laboratory** Stuart Maloy (collaborator)	

Correlating Silicon Carbide (SiC) Grain Size and Grain Boundary Orientation with Strength and SiC Layer Growth Conditions

Principal Investigator: Isabella J. van Rooyen – Idaho National Laboratory
e-mail: isabella.vanrooyen@inl.gov

Introduction

The importance of this work lies in the many applications in which SiC can be used. Data on the material's new properties and in-reactor relationships will be developed, along with a new characterization method that is better suited for very small, thin SiC samples. Although SiC grain size and orientation have previously been measured using the electron backscatter diffraction (EBSD) technique, no reports to date have indicated any correlations between these factors and strength/layer growth conditions.

In addition, no EBSD results, which provide knowledge on the effect of high-temperature annealing on the grain boundary characteristics of chemical vapor deposition in SiC layers, are published internationally. Demonstrating these correlations will provide new scientific information linking mechanical properties to grain boundary properties on the atomic level. At present, no mathematical model exists for such correlations. The data from this experiment will contribute to the development of such a model and the associated capability to predict SiC behavior in a variety of conditions and applications.

Demonstrating these correlations will provide new scientific information linking mechanical properties to grain boundary properties on the atomic level.

Project Description

The primary objective of this rapid turnaround experiment (RTE) is to contribute to the SiC knowledge base by answering key questions about SiC properties under extreme conditions. Specifically, the project seeks to reveal correlations between SiC layer grain size and grain boundary orientation, as well as the strength of SiC layers grown under different conditions and subsequently annealed at different temperatures.

The SiC samples used were previously characterized for compressive strength and hardness using nano-indentation at Nelson Mandela Metropolitan University (NMMU) in South Africa. All have spherical geometries and are arranged in five batches, with each batch differing in layer thickness, deposition temperature, and deposition method. Further characterization to determine grain size and grain boundary orientations of the SiC layers was done in the CAES Microscopy and Characterization Suite (MaCS) using the focused ion beam (FIB) and/or EBSD. To accomplish this, a new EBSD technique for small samples with nonstandard geometries was developed.

This research is a collaborative effort between INL and Idaho State University (ISU), and is part of a larger project which seeks to reveal correlations between SiC layer grain size, grain boundary orientation, and strength/layer growth conditions related to temperature and time.

Accomplishments

Two specific technical goals were addressed in this project. The first was to establish an EBSD technique for small samples with different geometries. To date, two baseline EBSD sample preparation methods have been developed: a conventional polishing technique and a first FIB-prepared prototype.

INL researchers measured the grain boundary characteristics on polished, mounted SiC samples using the EBSD technique to determine how well the areas chosen for these measurements were represented. The first-of-a-kind prototype FIB sample (Figure 1) was prepared at

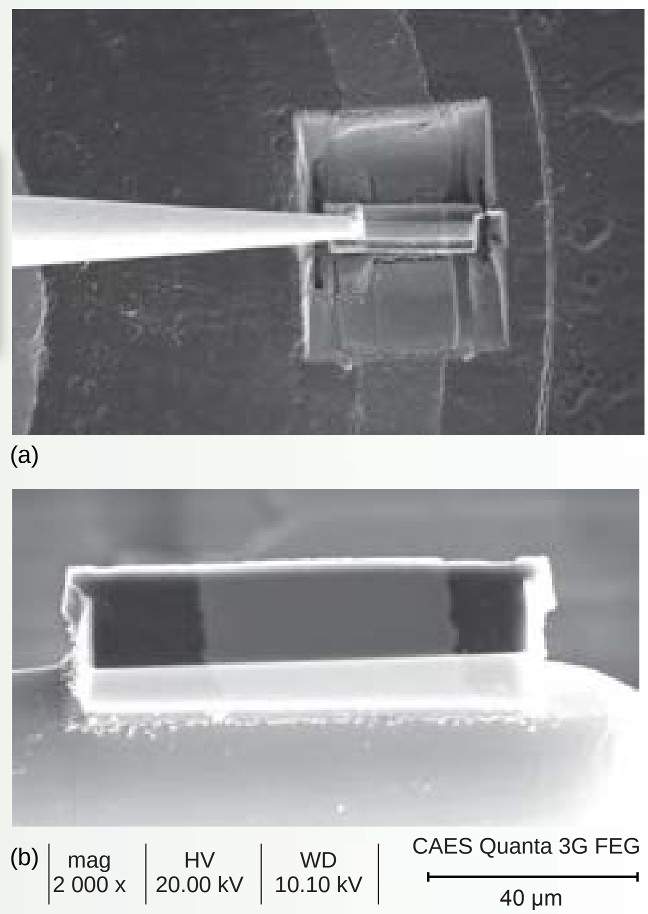

Figure 1. (a) Micrograph showing a prepared FIB lamella located in the machined wedge, and (b) Micrograph showing the completed sample after final FIB machining and polishing, ready for EBSD analysis.

"Although the project just recently started, it has already showcased the vision of INL to provide leading-edge research in new areas of nuclear engineering."

Dr. Isabella van Rooyen, Nuclear Materials Scientist, Idaho National Laboratory

INL, and then handed over to ISU and CAES to continue the first EBSD measurements. The preparation technique and sample holder fixture (Figure 2) was further optimized to provide even finer surface smoothness and enable the EBSD measurements on submicron grains (Figure 3, right).

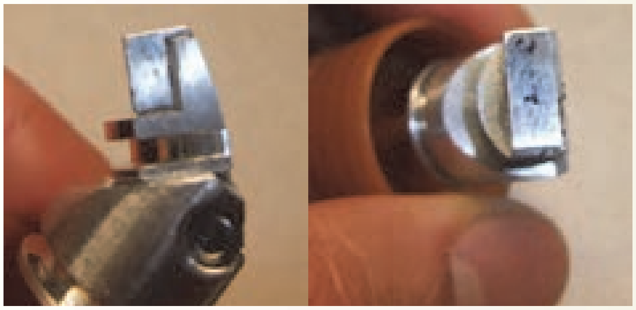

Figure 2. Modified fixture used for the EBSD analysis.

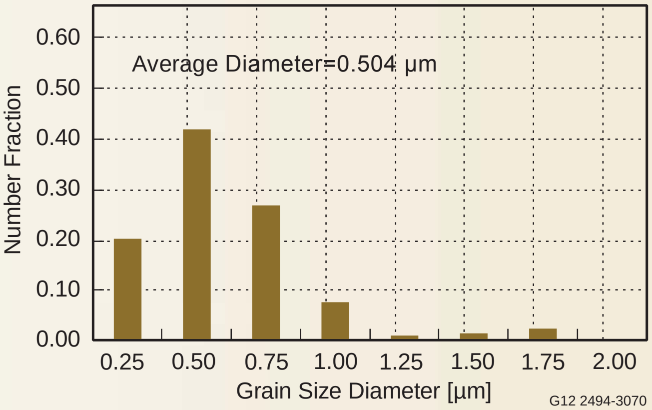

Figure 3. SiC layer grain size (diameter) distribution via EBSD analysis of FIB-prepared sample of coated particle B, annealed for 100 hours at 1600° C. The newly developed FIB preparation technique provided a tool for taking EBSD measurements of submicron size samples, which cannot be achieved using conventional polishing techniques.

The second technical goal was the actual measurement and interpretation of the results to determine the influence of annealing temperature on the SiC grain boundary characteristics. Between May 2012 and September 2012, researchers completed work on four of seven FIB-prepared samples with corresponding EBSD measurements. The remaining three samples are scheduled to undergo the same process in 2013. The original proposal called for analysis of a larger number of samples, however, that number was later decreased to only seven in order to accommodate the four-day FIB preparation process needed for each sample. Despite this reduction, the results eventually obtained from all seven samples will provide initial proof of principle. It is the intent of the principal investigators to have a nuclear engineering student complete part of the remaining work as a master's degree project. Although full interpretation is still in process, some selected results are provided in Figure 4 and Figure 5 (next page).

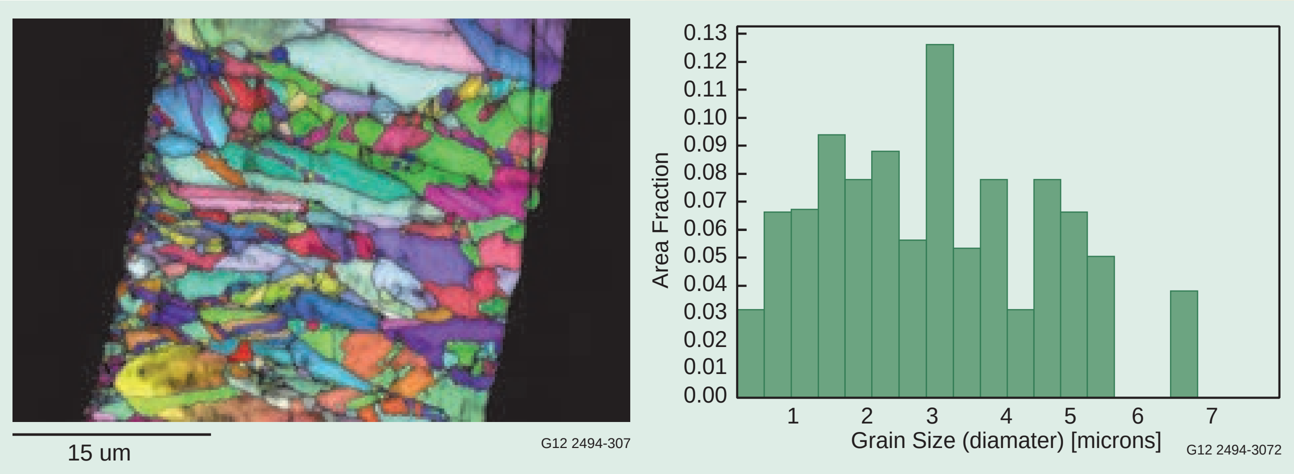

Figure 4. EBSD overlay map and grain size (diameter) distribution for the SiC layer of a FIB-prepared sample of a coated particle E reference sample (no annealing).

Principal Investigator: Isabella J. van Rooyen – Idaho National Laboratory (cont.)
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Correlating Silicon Carbide (SiC) Grain Size and Grain Boundary Orientation with Strength and SiC Layer Growth Conditions (cont.)

This project made very good progress during the first five months. The first technical goal was fully achieved, and the second is 57% complete. The value of this research to the larger, overarching project is expected to be significant and visible by the end of 2013.

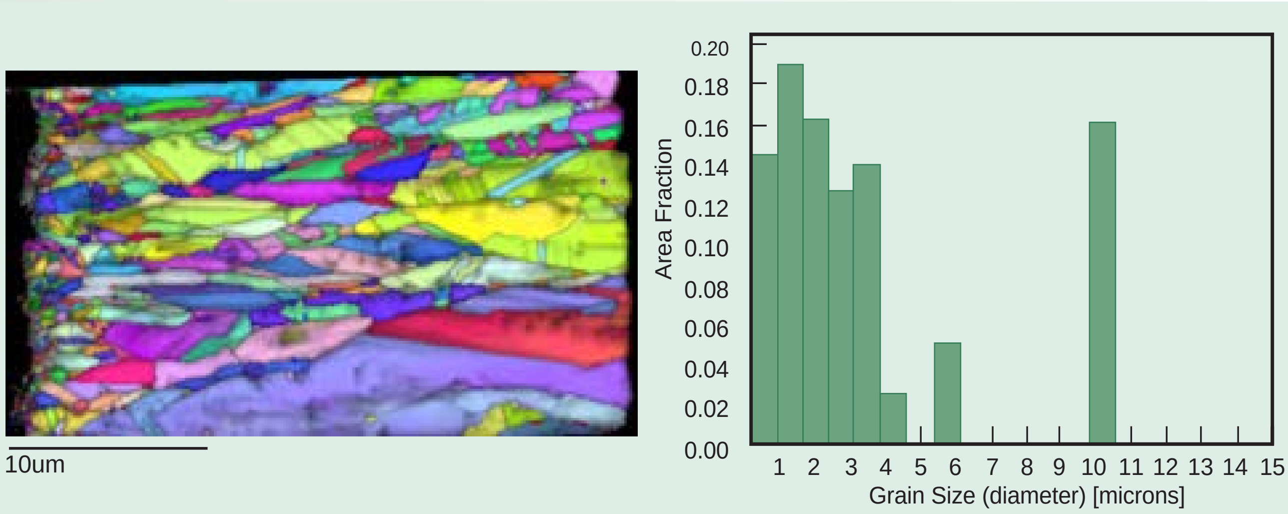

Figure 5. EBSD overlay map and grain size (diameter) distribution for the SiC layer of a FIB-prepared sample of a coated particle E, annealed for 15 minutes at 2000º C.

Future Activities

The goals for this research in 2013 include:

- Complete the remaining three FIB sample preparations and EBSD measurements
- Publish a journal paper on the EBSD sample preparation technique
- Train a nuclear engineering student from ISU to execute the FIB preparation, as well as the EBSD measurements
- Interpret and integrate the results into the larger project, and write a follow-up journal paper
- Continue work with the project collaborators to complete the larger project, pending further funding sources.

Publications and Presentations

Isabella J. van Rooyen, Mary Lou Dunzik-Gouger, Phillipus M. van Rooyen, Tammy Trowbridge, 2012, "On Techniques to Characterize and Correlate Grain Size, Grain Boundary Orientation, and the Strength of the SiC Layer of TRISO Coated Particles: A Preliminary Study," *HTR 2012 Conference, Tokyo, Japan, October 28 – November 1, 2012,* Paper HTR2012-3-024.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	PIE facilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Team Members/Collaborators	
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Idaho State University Mary Lou Dunzik-Gougar (ISU principal investigator)	
Center for Advanced Energy Studies Jatu Burns (collaborator)	
Nelson Mandela Metropolitan University, South Africa Jan Neethling, Japie Engelbrecht (collaborators)	
Phillip M. van Rooyen Network Consultants, South Africa Philippus M. van Rooyen (collaborator)	

Principal Investigator: John H. Jackson – Idaho National Laboratory
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Industry Program Project

EPRI Pilot Project for Irradiation and PIE of Alloys X-750 and XM-19

Introduction

In order to establish a basis for the development and execution of joint programs between ATR NSUF and the private nuclear power industry, a pilot project involving shared costs and responsibilities has been developed by the Electric Power Research Institute (EPRI) and INL. In addition to providing data from the experiment, the project is designed to:

- Develop administrative protocols for cooperative research, such as cooperative agreements and funding
- Develop the capabilities and staffing required to address future research and development needs
- Develop a protocol for validating project data, particularly data related to stress corrosion crack growth rates.

This project is important for three reasons. First, it is the inaugural private industry project for ATR NSUF and, as such, establishes protocols for these types of projects in the future. Second it is the first INL project to perform a full cradle-to-grave characterization of these internal reactor materials, including baseline characterization, irradiation, and post-irradiation examination (PIE). Third, it is the first project to utilize the newly reactivated Loop 2A in the ATR center flux trap and the newly installed irradiation-assisted stress corrosion cracking (IASCC) test systems.

Project Description

An investigation by the principal parties identified the fracture toughness and IASCC growth rates of irradiated high-strength alloys used for boiling water reactor (BWR) repair hardware (Figure 1) as an area of interest for the project. Very little data on these conditions exist for the Ni-based alloy X-750 and the nitrogen-strengthened austenitic stainless steel XM-19 at exposure levels up to $1x10^{21}$ n/cm^2. Therefore, the focus of this EPRI pilot project is on the irradiation and characterization of these alloys in both unirradiated (baseline) and irradiated states.

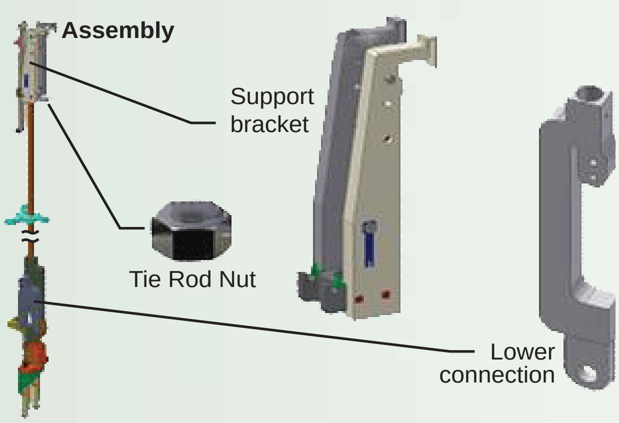

Figure 1. BWR repair support bracket comprised of X-750 or XM-19

Very little data exist on X-750 and XM-19 at exposure levels up to $1x10^{21}$ n/cm^2.

Accomplishments

The first two phases of the project were completed during 2012, and reports containing the results were issued from both INL and EPRI.

Phase I involved the fabrication of test specimens from materials provided by EPRI, and the measurement of baseline fracture toughness and crack growth rates prior to irradiation. Several baseline stress corrosion cracking (SCC) tests were completed on both alloys (Figures 2 and 3 next page). Cold-worked (19.3%) XM-19 was utilized to take advantage of the opportunity to study the material property effects of embrittlement phenomena produced during neutron irradiation and potentially similar embrittlement induced by cold-working the material. In addition to SCC tests, tensile and fracture toughness tests were conducted at temperature (not in environment).

The results of the SCC tests were compared to data produced at GE-Global Research Company as a way of benchmarking INL's capability for performing these highly specialized experiments. In all cases, the measured crack growth rates compared favorably with those produced by the benchmark laboratory.

In Phase II, specimen holders were designed and fabricated for Loop 2A in the center flux trap of ATR. A safety analysis was performed on a test train (Figure 4 next page) to meet EPRI objectives for irradiation of tensile and compact tension specimens. The Phase II report, issued in February 2012, contains descriptions of the train design and results of the analyses.

"This is an exciting project for ATR NSUF because it allows us to prove ourselves to private industry and, at the same time, provides relevant data to address an industry concern."
Dr. John H. Jackson, Industry Program Lead, Idaho National Laboratory

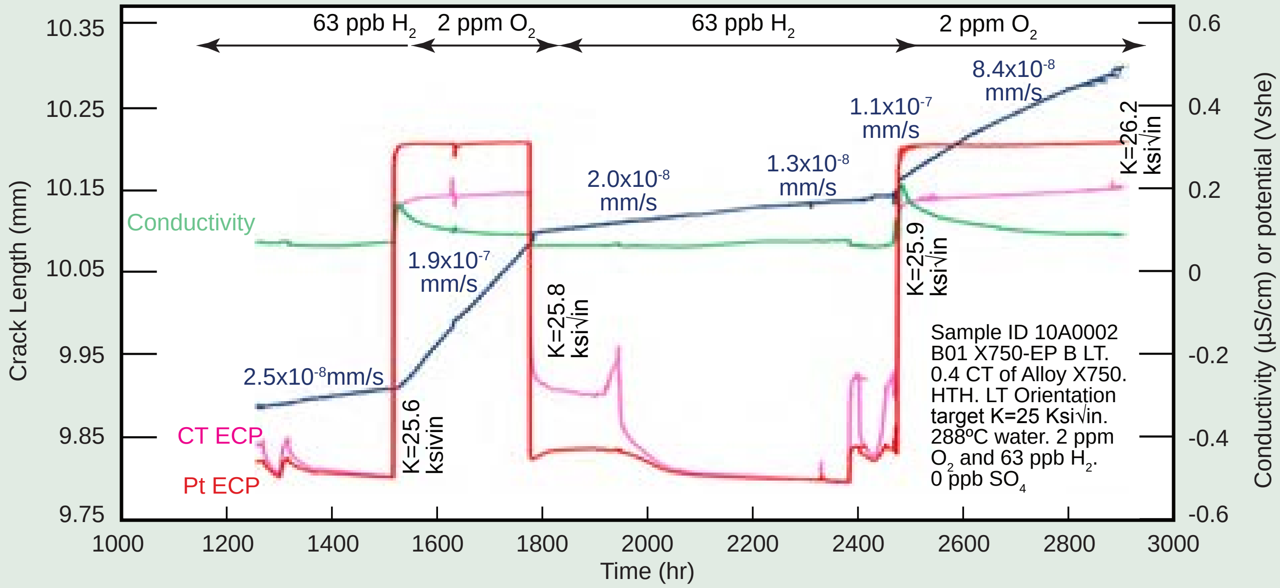

Figure 2. Typical SCC test results showing crack growth in alloy X-750 (blue line).

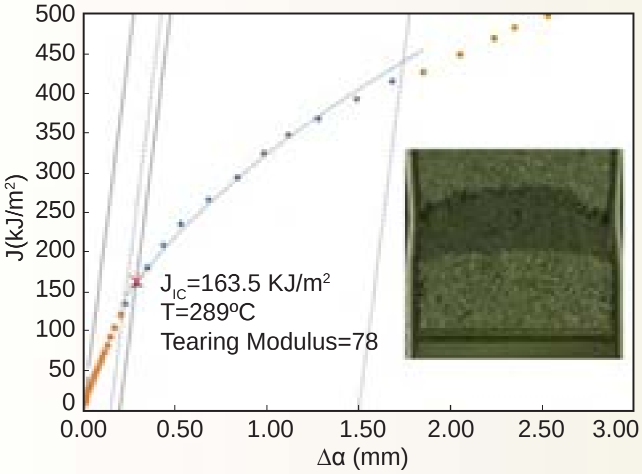

Figure 3. Typical fracture toughness test results for alloy X-750.

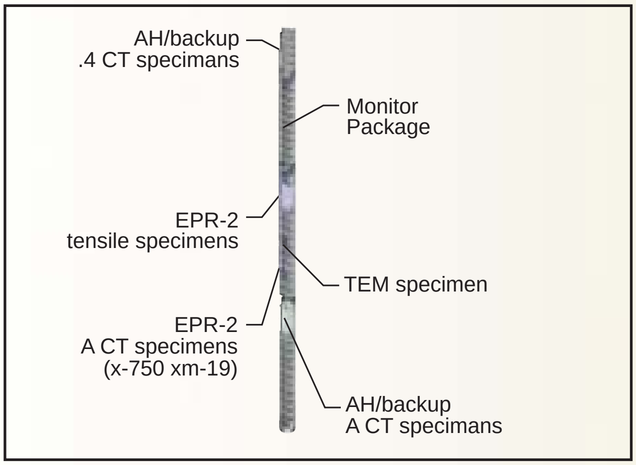

Figure 4. Typical EPRI pilot project test train.

Future Activities

The first two experiment capsules will be irradiated in 2013. These represent the first two experiments to utilize the newly reactivated Loop 2A in the center flux trap of ATR. After irradiation, the first capsule will be shipped to the Hot Fuels Examination Facility (HFEF) at MFC, where it will be disassembled in preparation for PIE. The specimens will be sent in batches to the newly installed IASCC test systems, where they will undergo fracture toughness testing using . The tensile specimens will remain at HFEF for tensile strength characterization. Additional TEM specimens, which were pre-fabricated prior to irradiation and encapsulated in CT specimen blanks, will be analyzed at the Electron Microscopy Laboratory (EML). The third capsule is scheduled to go into the reactor in 2014.

Publications and Presentations

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). EPRI, Palo Alto, CA: 2012. 1025135.

Principal Investigator: John H. Jackson – Idaho National Laboratory
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Industry Program Project

Other Industry Project Work in 2012

In addition to the EPRI Pilot project, there were three other industry projects for which significant progress was made during 2012. These three projects are briefly described in the following pages.

EPRI Zirconium Growth Pilot Project

The EPRI Zirconium Growth pilot project is a project in which zirconium alloy specimens are being irradiated in the INL's ATR to study the mechanisms of irradiation-induced growth and its dependence on hydrogen content and neutron fluence. Two hundred specimens of various zirconium alloys with various hydrogen concentrations are being irradiated at a temperature of 285° C ±10° C and to four targeted neutron damage levels (7, 10, 20, and 30 displacements per atom [dpa]) at completion. Four sets of fifty identical specimens are being irradiated in an inert environment in four separate irradiation capsules identified as capsule A (7 dpa), capsule B (10 dpa), capsule C (20 dpa), and capsule D (30 dpa). The hydrogen produced by corrosion and dissolved in zirconium alloys during service in light water reactors can form hydrides that may promote irradiation growth. Differential strain caused by hydrogen-assisted irradiation growth is postulated to be partly responsible for fuel channel bowing observed in operating boiling water reactors. Post-irradiation length measurement and transmission electron microscopy will be used to investigate the mechanisms of irradiation-induced and hydrogen-assisted growth. Figure 1 shows typical 35 mm x 6.5 mm x 0.8 mm zirconium alloy specimens prior to irradiation.

Figure 1. EPRI Zirconium specimens prepared for irradiation.

The irradiation for this pilot project began in April 2011. In 2012 the test capsules continued irradiation while flux and temperature were monitored. Additionally, the research team designed an in-cell measurement device to allow measurement of the specimens once they come out of ATR for comparison to unirradiated dimensions and subsequent calculation of irradiation-induced strain.

The first test capsule (capsule A, 7 dpa) is scheduled to be removed from ATR and shipped to the INL Hot Fuel Examination Facility in mid 2013. Capsule B (10 dpa) will follow capsule A with a scheduled completion date in late 2013. Capsules C (20 dpa), and D (30 dpa) will take quite a bit longer to irradiate and will finish irradiation in the 2015-2017 time period. It is anticipated that in-cell measurement of the first specimens will occur in late 2013.

INL-AECL Joint Project for Active FIB and TEM Analysis of Irradiated X-750

The purpose of this joint project is to examine microstructural changes of alloy X-750 as a function of neutron irradiation and temperature. Several garter spring sections (Figure 2) that had been used in service as fuel channel spacers in CANDU reactors were provided to INL by the Atomic Energy of Canada Limited's (AECL) Chalk River Laboratory. These spring sections had exhibited extreme loss of ductility during service inspections and have become the subject of fitness for service investigations by the CANDU reactor industry. Preliminary field inspections indicated a potential relationship between loss of ductility and location of spring sections; a thorough microstructural analysis was deemed necessary to understand the behavior of these spring materials.

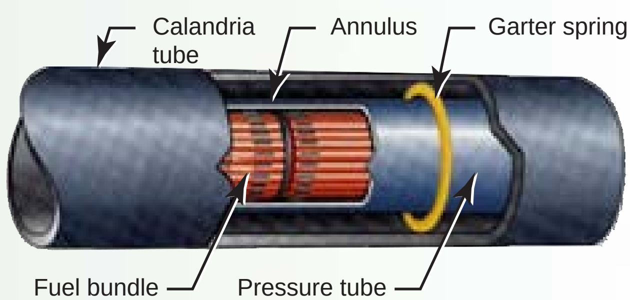

Figure 2. Depiction of fuel channel showing X-750 garter spring spacer in a CANDU reactor.

In order to achieve the goals of this project, transmission electron microscopy (TEM) foils were produced using the active focused ion beam (FIB) at the Electron Microscopy Laboratory at MFC, as well as the TEM in the CAES Microscopy and Characterization Suite (MaCS). In addition to TEM analysis, a secondary objective is to characterize the hardness of the irradiated spring sections. For this purpose, a shielded microhardness tester at MFC will be used. TEM foils will be prepared at CAES and MFC as needed using the nine spring sections from the material provided by AECL.

During 2012, the material was received at INL and the active FIB at MFC was utilized to produce TEM foils for analysis by a visiting AECL scientist. Initial TEM analyses were performed at the MaCS laboratory, along with microhardness measurements. Final TEM analysis remains to be completed.

Nuclear Regulatory Commission (NRC) Irradiation and Testing of Austenitic Stainless Steel in BWR Conditions

The core internals of a commercial light water reactor (LWR) are designed to provide structural support to maintain the core fuel assemblies in a coolable geometry. During power operation, the materials comprising the internals' structure are exposed to neutron irradiation. Neutron irradiation affects the materials' properties by causing embrittlement and creep because of the introduction of dislocations, vacancies, and segregation precipitates into the microstructure. Neutron irradiation can also cause dimensional changes from void swelling due to the production of helium bubbles within the microstructure, which may impact the structural integrity of internals components.

The NRC staff is conducting confirmatory research to address the void swelling behavior of LWR core internal materials due to neutron irradiation. The NRC staff intends to use this research to evaluate data to be submitted by the industry to quantify the expected dimensional changes in LWR internals as a function of fluence and the potential impact of void swelling on the structural integrity of LWR internals in support of an assessment of inspections related to the NRC Materials Reliability Program. 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 staff needs to ascertain that gamma heating effects can be controlled. The NRC staff has conducted irradiations to 1.2-1.4 dpa in the Halden research reactor in Norway. The Halden specimens were irradiated at prototypical boiling water reactor (BWR) temperatures, and were exposed to limited gamma heating thereby limiting migration of defects during irradiation. The proposed test program will produce samples that can be compared to the Halden samples in order to establish the ability of ATR to simulate prototypical LWR conditions.

As a first step toward full scale utilization of ATR and associated post-irradiation examination (PIE) equipment, the NRC and ATR NSUF staff have formulated a test program that is designed to establish INL as a viable destination for irradiation and PIE of reactor structural materials. This initial research program will utilize two materials that have previously been irradiated at the Halden reactor and tested at Argonne National Laboratory so that test results may be compared, thereby establishing a measure of comparability between Halden and ATR irradiations. A secondary objective of this test program will be to characterize the quality of data produced using INL's newly constructed irradiation-assisted stress corrosion cracking (IASCC) test cells.

This project between ATR NSUF and NRC was initiated in the fall of 2012. During 2013, of the two materials of choice (a sensitized 304L stainless steel and a 304L weld heat affected zone material) will undergo baseline, unirradiated crack growth rate, and fracture toughness testing. In addition, the irradiation of these materials will be started in one of the EPRI X-750 and XM-19 pilot project test capsule backup locations. PIE on this project will begin in 2014.

Distributed Partnership at a Glance	
ATR NSUF & Partners	**Facilities & Capabilities**
Idaho National Laboratory	Advanced Test Reactor, PIE facilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Team Members/Collaborators	
Idaho National Laboratory John H. Jackson (principal investigator), Paul Murray (co-investigator), James Cole, Sebastien Teysseyre (collaborators)	
Electric Power Research Institute Bob Carter (program manager), Peter Chou (technical contact), Suresh Yagnik (collaborator)	
Nuclear Regulatory Commission Appajosula (Sri) Rao (co-investigator)	

Idaho National Laboratory