INL/MIS-17-43361-Revision-0

2017 Year End Nuclear Science User Facilities (NSUF) Post-Irradiation Examination Summary Report

October 2017

Collin J. Knight

Changing the World's Energy Future

Idaho National Laboratory

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NSUF Nuclear Science User Facilities

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Acronyms

APS	Advanced Photo Source
ATR	Advanced Test Reactor
CAES	Center for Advanced Energy Studies
CINR	Consolidated Innovative Nuclear Research
DOE	U.S. Department of Energy
EML	Electron Microscopy Laboratory
EPRI	Electric Power Research Institute
FIB	focused ion beam
FIMA	fissions per initial metal atom
FY	fiscal year
HFEF	Hot Fuel Examination Facility
IMCL	Irradiated Materials Characterization Laboratory
INL	Idaho National Laboratory
MCOE	Materials Center of Excellence (Westinghouse)
MFC	Materials and Fuels Complex
MIT	Massachusetts Institute of Technology
MRCAT	Materials Research Collaborative Access Team
NSUF	Nuclear Science User Facilities
PIE	post-irradiation examination
PNNL	Pacific Northwest National Laboratory
RTE	Rapid Turnaround Experiments
SEM	scanning electron microscopy
SPT	shear punch test
TEM	transition electron microscopy
UCB	University of California, Berkeley
UCF	University of Central Florida
ZG	zirconium growth

1. INTRODUCTION

The Nuclear Science User Facilities (NSUF) are part of a growing number of U.S. Department of Energy (DOE) user facilities in the U.S., and is the only national designated nuclear energy user facility. Through a peer-reviewed proposal process, NSUF provides external research teams with cost-free access to reactor, post-irradiation examination (PIE), and beamline capabilities at Idaho National Laboratory (INL) and a diverse mix of affiliated partner institutions at universities, national laboratories, and industry facilities located across the country.

In fiscal year (FY) 2017, the NSUF sponsored planning, management, and execution of PIE activities at INL, the Center for Advanced Energy Studies (CAES), and other NSUF partner facilities. The individual projects associated with these PIE activities are a result of DOE awards under the following mechanisms: Consolidated Innovative Nuclear Research (CINR) awards that include full sample irradiation and PIE, PIE only (using samples from the NSUF Fuel and Materials Library) awards, or Rapid Turnaround Experiments (RTE). An accounting of FY 2017 activities for each project is provided in this report.

2. FISCAL YEAR 2017 AND PRIOR AWARDS

2.1 Consolidated Innovative Nuclear Research Projects (Post-Irradiation Examination Only)

2.1.1 Idaho National Laboratory MO 8418

The purpose of this project is to elucidate the microstructural evolution and the microscopic phenomena that formulate the macroscopic fuel behavior in U-Zr and U-Mo fuels through utilizing a systematically designed set of irradiated fuels as a function of composition, fluence, and temperature to: (1) assess phase stability and chemistry, (2) understand defect microstructure and conduct grain analysis, (3) determine mechanical properties, and (4) conduct multiscale modeling to complement experiments. This award used samples irradiated in the Advanced Test Reactor (ATR) as part of the University of Central Florida (UCF)-1 experiment.

The first phase of specimen testing was completed in late April 2016 in the Materials Research Collaborative Access Team (MRCAT) beam line at the Advanced Photo Source (APS). These samples were returned to the Materials and Fuels Complex (MFC) after testing was completed. New samples were shipped to APS on July 20, 2017, for Phase 2 of the testing. A total of 58 samples were sent, including nine control samples. Only samples < 5 mRem/hr @ 30 cm (gamma) and < 20 mRem/hr @ 30 cm (beta) were sent, which excluded a significant portion of the 1 dpa (displacements per atom) UCF-1 specimens. Beam time at MRCAT was expected to be completed the week of July 25, 2017; however, a safety issue at the MRCAT beam line has delayed testing in the near term. It is not expected that testing will be completed until sometime between February through April 2018. The samples are being held at Argonne National Laboratory in the near term until additional decisions can be made. A total of two tests at MRCAT are planned for this experiment.

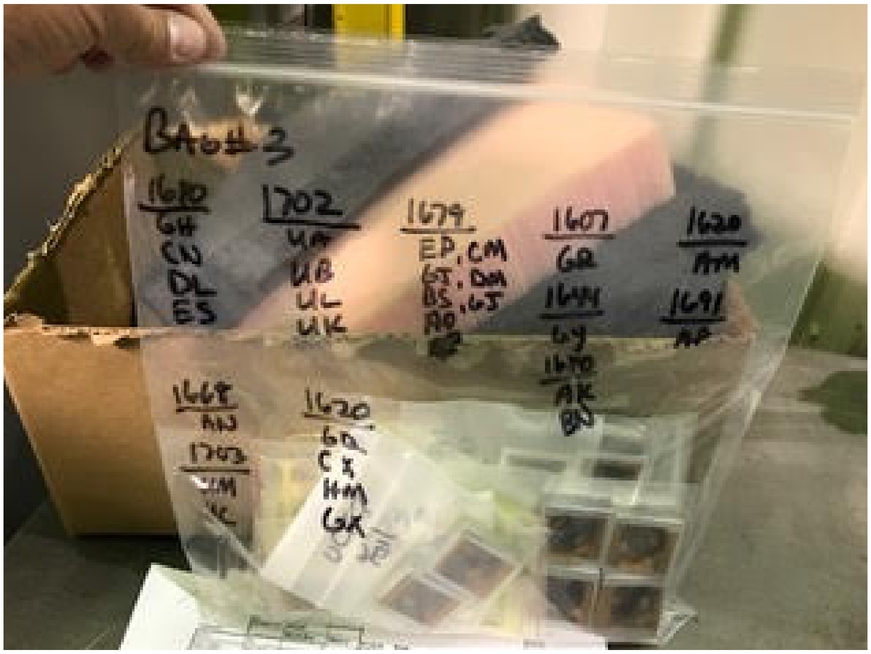

Figure 1. University of Central Florida samples prepared for shipment to the Advanced Photo Source.

2.1.2 Idaho National Laboratory DG 10639

This project performed X-ray absorption spectroscopy and Extended X-ray Absorption Fine Structure spectroscopy on Hf-Al materials at the MRCAT beamline at APS. The objective of the study was to determine the oxidation state and the coordination environment of the hafnium compounds.

The samples consisted of irradiated and unirradiated material. The material was fabricated by hot pressing various amounts of $HfAl_3$ intermetallic particles in an aluminum matrix to produce a metal matrix composite consisting of 20.0, 28.4, 36.5, or 100 vol% $HfAl_3$. The $HfAl_3$ intermetallic was produced by casting Hf and Al in a centrifugal caster. Both unirradiated (in powder and monolith form) and irradiated samples (in monolith form) were examined. Unirradiated samples consisted of 100 vol% $HfAl_3$ powder and transition electron microscopy (TEM) disks of 20.0, 28.4, and 36.5 vol% $HfAl_3$.

Nine specimens from the Utah State University experiment were successfully tested in the MRCAT beam line from March 1–6, 2017. The samples have subsequently been returned to Electron Microscopy Laboratory (EML), completing this project.

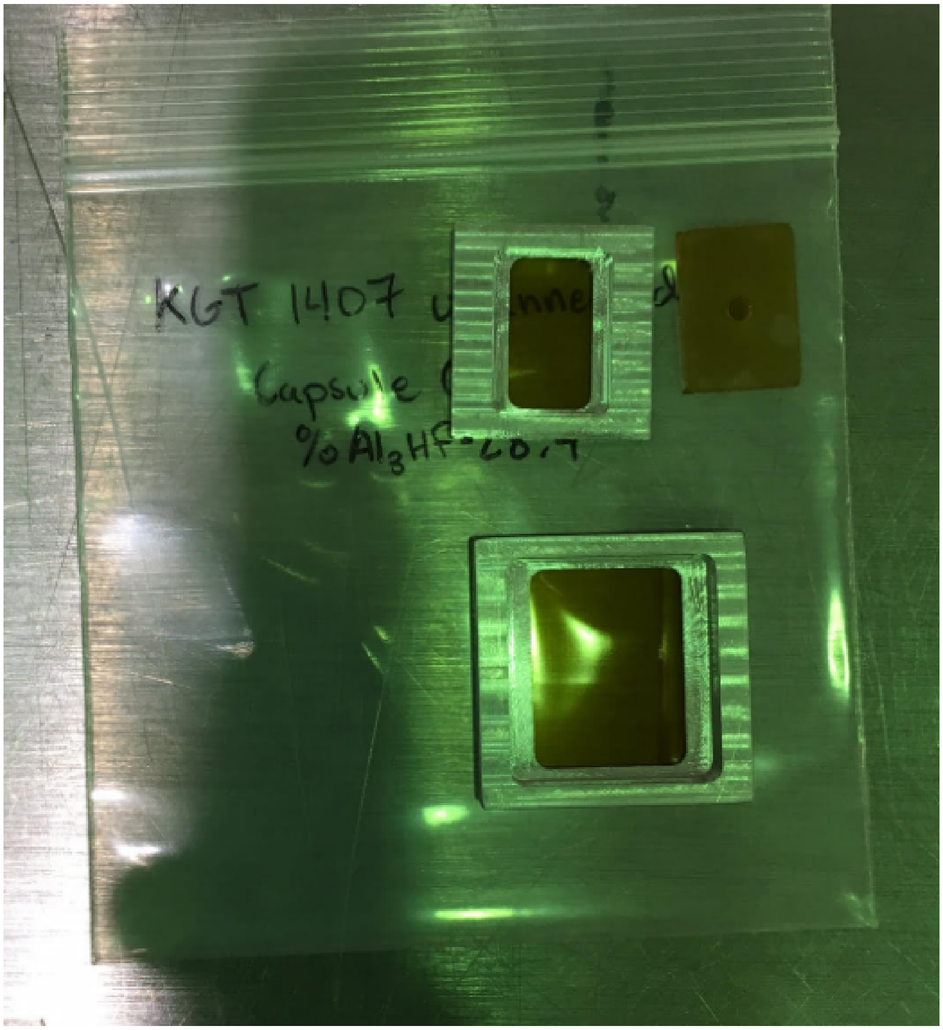

Figure 2. University of Illinois samples prepared for shipment to the Advanced Photo Source.

2.1.3 University of California, Berkeley 10-244

This experiment performed in-pile measurement of uranium-zirconium hydride fuel to determine the thermal conductivity of uranium-zirconium hydride fuel pellets clad in Zircaloy-2. Fuel pellets in light water reactor geometry were prepared by machining and center less grinding specimens from TRIGA fuel slugs (19.7% enriched fuel, U:Zr:H atomic ratio of 0.17:1:1.6) where the fuel consists of metallic uranium particles dispersed in ZrH1.6±x matrix. Fuel pellets were liquid-metal-bonded (lead-bismuth eutectic alloy) to the cladding to eliminate the large uncertainty in gap conductance. The cladding was liquid metal bonded to the titanium irradiation capsule. After irradiation in the Massachusetts Institute of Technology (MIT) reactor, PIE was performed to potentially link the evolution in fuel thermal conductivity to the microstructure.

Three irradiated capsules were shipped from MIT to Pacific Northwest National Laboratory (PNNL) for analysis. Of the three irradiated capsules, analysis of one capsule was completed in FY 2016. Upon completion of PIE activities for Capsule 1, project related material was prepared for shipment to INL where it will be stored in the NSUF Fuel and Material Library. Material from Capsule 1 and the two unexamined irradiated capsules were shipped from PNNL to the Hot Fuel Examination Facility (HFEF) on June 21, 2017. The unexamined capsules were sent in their original shipping containers (used for shipping from MIT) while the examined capsule was shipped in an 8,500 shielded cask to MFC. Sample unloading in the HFEF decontamination cell was completed in July 2017. An unirradiated capsule sent to PNNL for scoping tests and sample handling technique development has also been shipped to MFC and unloaded in the EML. This capsule will likely be held in EML or the Irradiated Materials Characterization Laboratory (IMCL) for long-term storage.

Figure 3. University of California-Berkeley experiments packaged for shipment to Idaho National Laboratory.

2.1.4 University of Illinois 8305

The objective of this project is to investigate and determine the impact of neutron irradiation-induced microstructural changes on the mechanical properties of Fe-Cr base alloys using in-situ synchrotron wide-angle X-ray scattering tensile tests. The objective of this research program is to utilize the special capabilities of the MRCAT beamline at APS to analyze the evolution of tensile properties following ATR irradiation of a series of model, commercial, and developmental Fe-Cr alloys. The results of this project, when coupled with the post-irradiation microstructure characterization data of the same samples obtained from another PIE program, will yield a family of results that can improve the understanding of the joint influences of irradiation, temperature, and microstructure on tensile deformation.

Seventy-six tensile specimens have been shipped to APS for testing in the MRCAT beam line. Only the specimens with dose rates < 5 mRem/hr @ 30 cm gamma were shipped, limiting the number of specimens available for testing in APS (over 100 additional samples have been clean and measured with 95 found to be below APS's threshold for radiological samples). Although the original plan called for testing these samples in FY 2016 and again in January 2017, testing was delayed as APS worked with MRCAT to verify sample testing could be safely completed. APS has provided approval and the first 5 days of the MRCAT beam line testing was completed the week of July 3, 2017. The second 5-day testing period is now expected to be completed between February 1 and April 30, 2018.

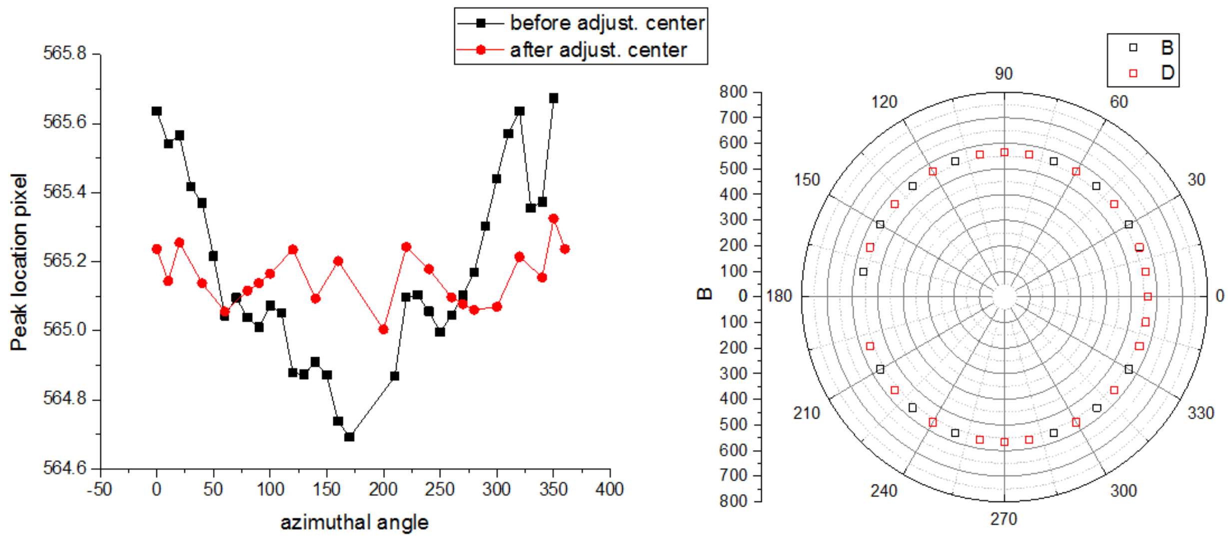

Figure 4. Test results from University of Illinois specimens tested at Materials Research Collaborative Access Team beam line at the Advanced Photo Source.

2.1.5 University of Illinois 8312

The objective of this research is to conduct a coordinated set of experiments with PIE and analyses that will provide significant new insight into the irradiation performance of ferritic alloys for advanced reactor applications. The research is based on neutron irradiation experiments on a matched set of ferritic alloys in a high flux test reactor. With the irradiation exposures completed, the objective of this work is to perform post-irradiation analysis two types of materials (HT-9 and T-91).

A total of 24 sample conditions for T-91 and HT-9 alloys were prepared and sent to CAES for TEM, atom probe tomography, and nano-indentation testing. University of Illinois student principal investigators (Huan Yan and Xiang Liu) traveled to CAES numerous times over the past 2 years analyzing these samples. At the close of September 2017, nano-indentation, atom probe tomography, and TEM testing on 20 conditions will be completed.

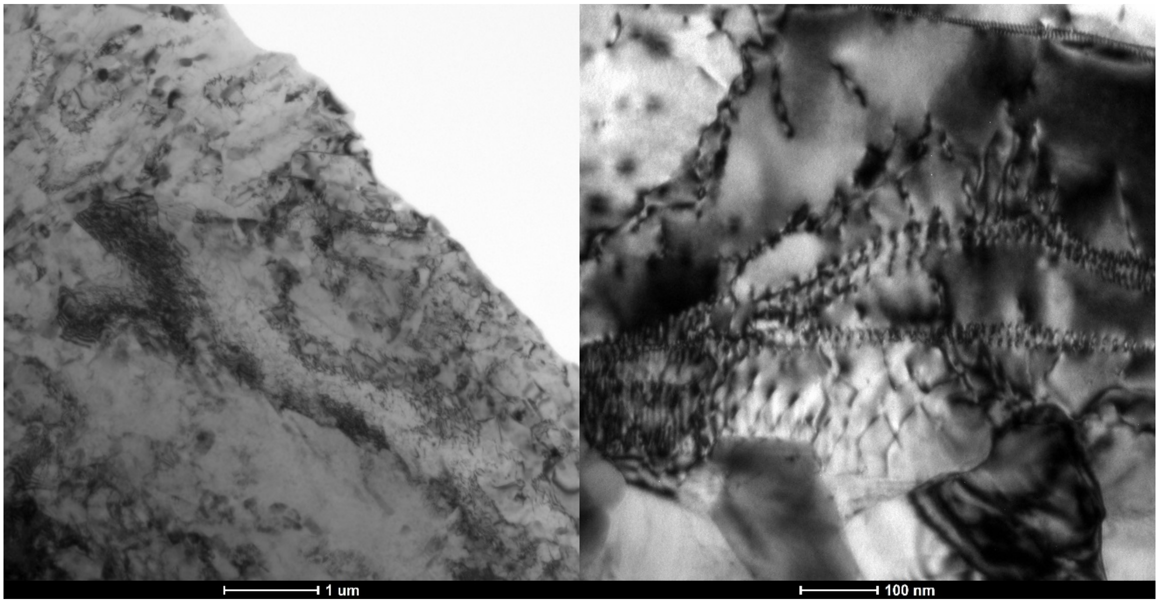

Figure 5. Transition electron microscopy images of a T-91 specimen in the Microscopy and Characterization Suite.

2.1.6 Boise State University/Purdue University 10181

This study will utilize stainless steel hexagonal blocks (EBR-II reflector material) for weld studies. The hex blocks are stored in the Westinghouse Materials Center of Excellence (MCOE) hot cells, a NSUF partner facility. This project will make laser welds on the hex blocks over a range of helium and swelling conditions, without the need for extensive pre-characterization. Weld cross-sections will be examined for cracking. Since the welds must maintain integrity under further irradiation, weld cross-sections will be subjected to additional irradiation at pressurized water reactor relevant temperatures using self-ions to doses as high as 200 displacements per atom (dpa). Subsequently, welds will be reexamined for microstructure, mechanical properties, and cracking.

The samples will be irradiated to 50, 100, and 200 dpa conditions at Texas A&M University. As the samples complete irradiation, they will be sent to CAES for PIE analysis. Testing at CAES will include X-ray diffractometry, nanoindentation, micro-cantilevers and micro-compression pillar testing, focused ion beam (FIB) lamella preparation, and TEM analysis.

In FY 2017, the 50 dpa coupon has been welded and TEM disks prepared. From six disks prepared from the coupon, one specimen was selected for the preparation of TEM lamella using Westinghouse's MCOE FIB. Four TEM disks have been shipped to Texas A&M University for ion-irradiation and the TEM lamella have been shipped to CAES for analysis. Student principal investigator, Keyou Mao (Purdue University), has completed TEM analysis in the CAES Microscopy and Characterization Suite for these six specimen lift-outs (lamella). It is expected that the 50 dpa specimens will shipped from Texas A&M University to CAES in the first quarter of FY 2018.

Figure 6. Weld coupon prepared in the Westinghouse Materials Center of Excellence hot cell.

2.2 Rapid Turnaround Experiments

2.2.1 Rapid Turnaround Experiment 16-682

This project performed destructive examination, including fission gas analysis and optical microscopy, on three elements. Elements DP-55 (U-19Pu-10Zr, 75% smear density, 11.1% BU, 2.1 P/F ratio), DP-49 (U-19Pu-10Zr, 85% smear density, 9.8% BU, 1.5 P/F ratio), and J559 (U-10Zr, 75% smear density, 9.2% BU, 1.5 P/F ratio) were selected for further analysis. The data collected in this project will be used to establish a one-of-a-kind fuel performance benchmark case for fast reactor metallic fuel designs under operational transient conditions. Benchmark cases like this are required to validate advanced modeling and simulation codes for fuel performance.

Project objectives including visual inspection, neutron radiography imaging (using the Neutron Radiography Reactor [NRAD] in HFEF), and gas sampling of fuel rods X512-DP55, -DP49, and -J559 for RTE 16-682 has been completed (also known as gas assay sample and recharge). Fission gas analysis reports have been obtained from PNNL. Sectioning of selected rod specimens (three per rod) as well as metallography of the mounts has also been completed.

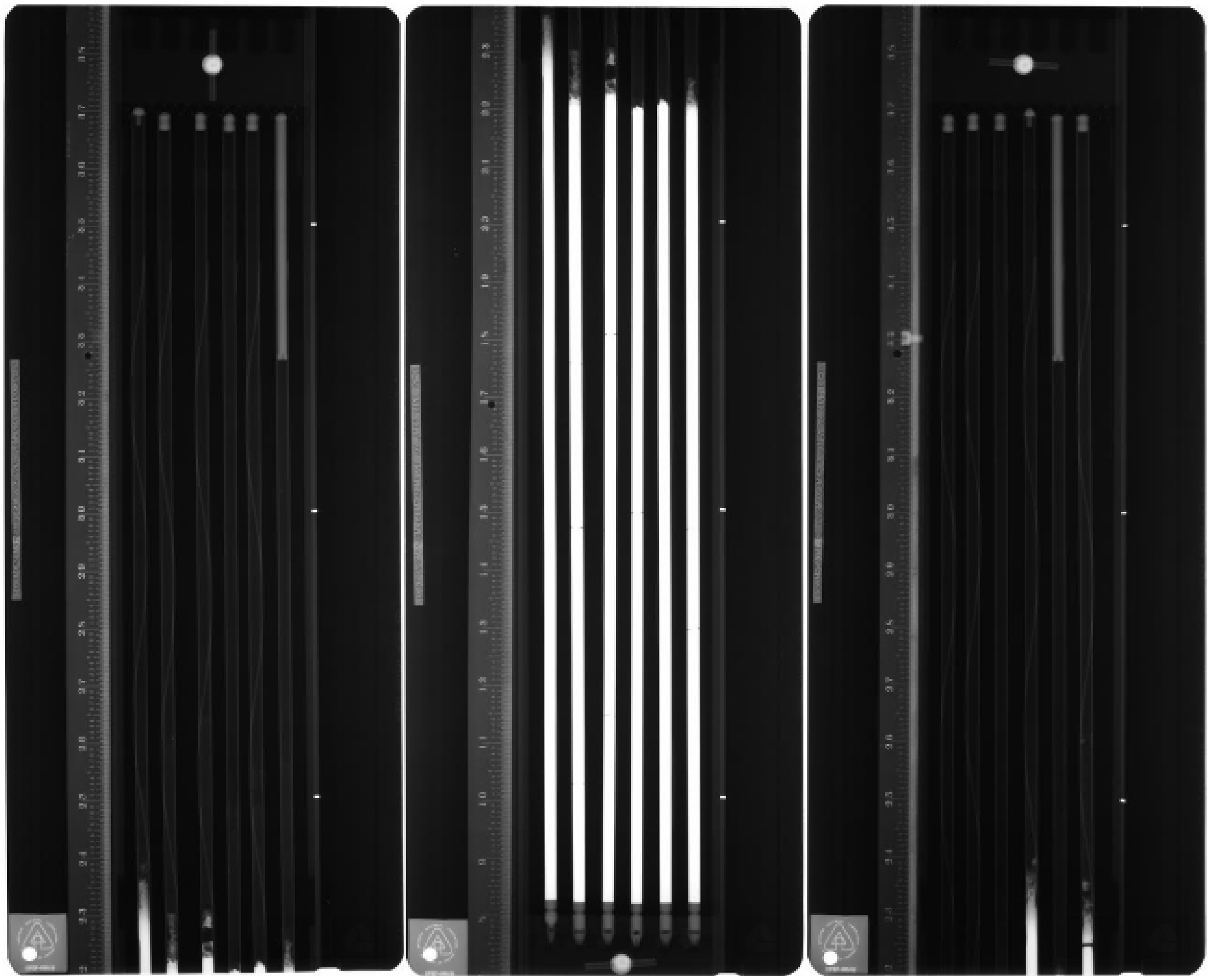

Figure 7. Radiographs of fuel rods X512-DP55, -DP49, and -J559.

2.2.2 Rapid Turnaround Experiment 17-694

The objective of this research was to investigate the neutron irradiation effects on the mechanical properties of Fe-Cr-C ternary model alloys. The selected model alloys for this research was Fe-9Cr-0.1C and Fe-12Cr-0.2C, the composition of which are similar to commercial alloys T-91 and HT-9, respectively. Nanoindentation was carried out to characterize the mechanical properties such as hardness and Young's modulus of the irradiated specimens. In total, eight samples (four Fe-9Cr-0.1C and four Fe-12Cr-0.2C) were examined. The nominal irradiation temperature for all samples are 300°C and the doses are 0.01 dpa, 0.1 dpa, 0.5 dpa, and 1.0 dpa. Testing activities were carried out at the University of California, Berkeley (UCB).

Requested samples from RTE 17-694 were shipped to UCB in March 2017. Research activities were subsequently completed at UCB as outlined in the proposal. This completes RTE 17-694.

2.2.3 Rapid Turnaround Experiment 17-728

The objective of this experiment was to better understand radiation resistance of NF709, by studying the radiation-hardening and microstructural evolution of NF709 irradiated to 6 dpa at 400, 500, and 700°C. The outcome of this study will provide key insights to understand radiation resistance of Alloy 709 that is optimized from NF709 under the Advanced Reactor Technologies program.

Samples for RTE 17-728 (KGT 1718, KGT 1775, and KGT 1804) were sent to CAES to support FIB, atom probe and nano/micro hardness testing. Testing was completed in March and April 2017 by ORNL researcher Tianyi Chen. TEM analysis by Lingfeng He on three additional lamella using the Titian-200 instrument in IMCL was also completed. This completes RTE 17-728.

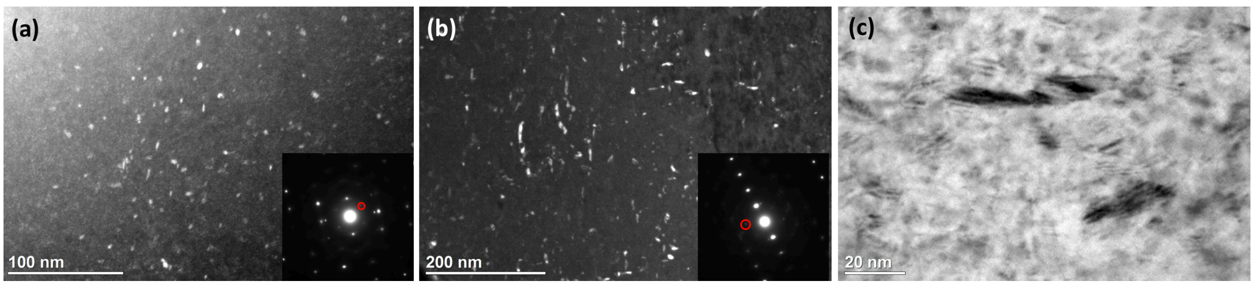

Figure 8. Images from NF709 specimen produced in the Irradiated Materials Characterization Laboratory with transition electron microscopy.

2.2.4 Rapid Turnaround Experiment 17-812

The objective of this research was to conduct detailed microstructural examination of a fuel pin with one of the highest burn-ups ever achieved in prototypic fast reactor fuel pins. The microstructure of high burn-up mixed oxide fuel with local burn-ups of 3.4–23.7% fissions per initial metal atom (FIMA) was examined. The wide range of burn-ups present in these samples provides a unique opportunity for studying fuel performance throughout life. The high burn-up structure formed in irradiated fuel was examined in addition to the radial examination of the fuel pellet, which was the focus on understanding the effects of irradiation conditions on the local microstructure of the fuel. This RTE focused on ACO-3 fuel subassembly, which achieved a peak burn-up for ~23% FIMA and a peak fast fluence of 38.9×10^22 n/cm^2 (E>0.1 MeV). The proposal conducted microstructural characterization of the fuel using TEM and FIB tomography.

As outlined in the proposal, scanning electron microscopy (SEM), FIB, and TEM analysis has been completed in EML for the prepared samples from fuel rod L01A. This completes activities for RTE 17-812.

2.2.5 Rapid Turnaround Experiment 17-813

This RTE used innovative in-situ approaches to simultaneously observe the deformation-induced microstructure evolution of neutron irradiated Fe-9Cr and 9Cr2WYT ODS alloys using SEM and TEM analysis. The TEM was used to examine the irradiation-induced microstructure, especially the nature of dislocation loops, of irradiated samples, and use atom probe tomography to study the evolution of the chemical composition of the oxide particles after neutron irradiation.

As detailed in the proposal, samples for RTE 17-813 (KGT 592 and KGT 601) have been analyzed in the CAES Microscopy and Characterization Suite using the SEM, TEM, and atom probe testing. This completes activities for this RTE.

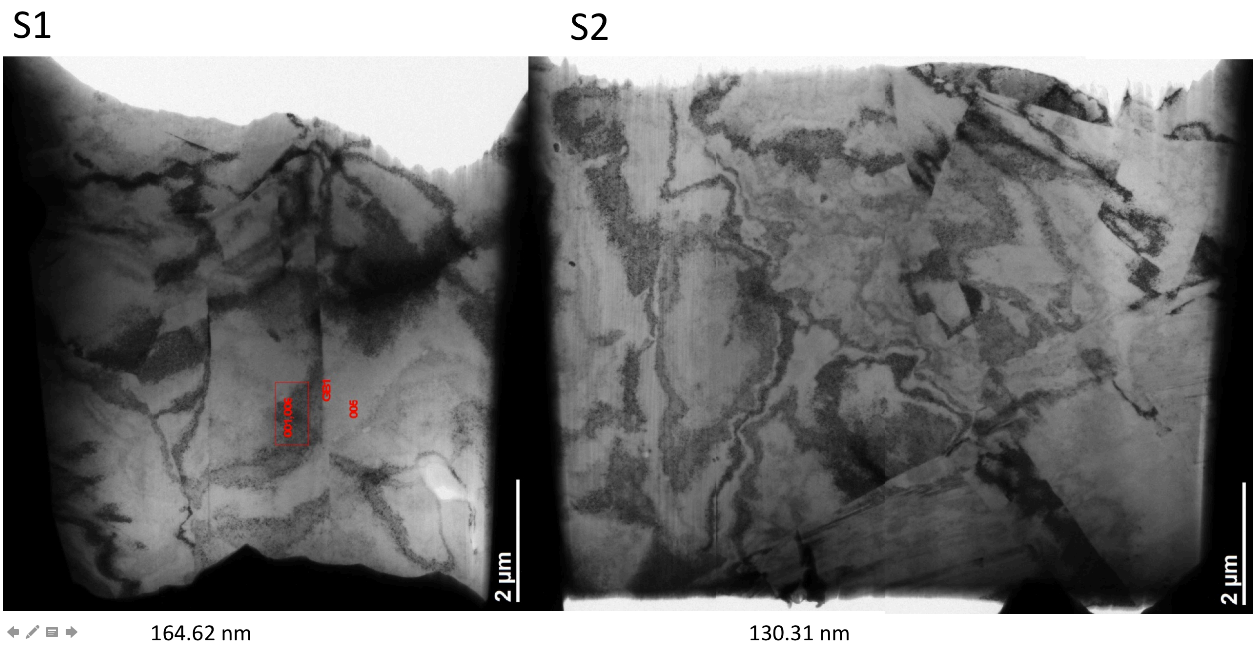

Figure 9. Images from Fe-Cr specimen produced in the Center for Advanced Energy Studies with transition electron microscopy.

2.2.6 Rapid Turnaround Experiment 17-880

This project aimed to evaluate the mechanical properties at room temperature and 320°C (reactor core materials relevant temperature) to understand the effects of radiation damage (1 dpa) on friction stir welding oxide dispersion strengthened alloys and successfully develop appropriate structure-property correlations. Thus, the proposed work was to test and analyze 16 irradiated (1 dpa) shear punch test (SPT) specimens (number of specimens to be tested: 8 microhardness, 16 SPT) present in the library.

Sixteen samples for RTE 17-880 (KGT-121 and -122) have been shipped from EML to PNNL. These include eight magnetic and eight non-magnetic specimens from MA956 and MA754, respectively. Research at PNNL continues; however, this completes INL activities for this RTE.

2.2.7 Rapid Turnaround Experiment 17-909

The objective of this research was to conduct detailed microstructural examination of a fuel pin with one of the highest burn-ups ever achieved in prototypic fast reactor fuel pins. The microstructure of high burn-up mixed oxide fuel with local burn-ups of 3.4–23.7% FIMA will be examined. The wide range of burn-ups present in these samples provides a unique opportunity for studying fuel performance throughout life. The high burn-up structure formed in irradiated fuel were examined in addition to the radial examination of the fuel pellet, which will be focused on understanding the effects of irradiation conditions on the local microstructure of the fuel. This RTE focused on the FO-2 fuel subassembly, which achieved a peak burn-up for ~6% FIMA and a peak fast fluence of 9.9×1022 n/cm^2 (E>0.1 MeV). The proposal conducted microstructural characterization of the fuel using TEM and FIB tomography. The acquired data will be reconstructed to produce three-dimensional volume, which will then be used as an input to computer models such as MARMOT.

Samples for RTE 17-909 (MNT-71T, MNT-75T, and MNT-117T from fuel rod FO-2) have been shipped from HFEF to EML. SEM, FIB, and TEM analysis has been completed as outlined in the proposal. This completes activities for this RTE.

2.2.8 Rapid Turnaround Experiment 17-953

The objective of this study was to investigate the radiation stability of fiber optical temperature sensors for in-pile temperature monitor and control for the ATR. Eighteen sensors were fabricated at the University of Houston and have been irradiated at different temperatures and doses. Post-irradiation study at the University of Houston will find out any potential damages to the optical cavities of the sensors.

Eighteen capsules from SAM-1 (RTE 17-953) were shipped to the University of Houston in July 2017 for retrieval of the fiber-optic sensors. The sensors have been removed and analysis is underway. The holders have been returned to EML for disposition (graphite and vanadium). Receipt of the sample holders completes RTE 17-953.

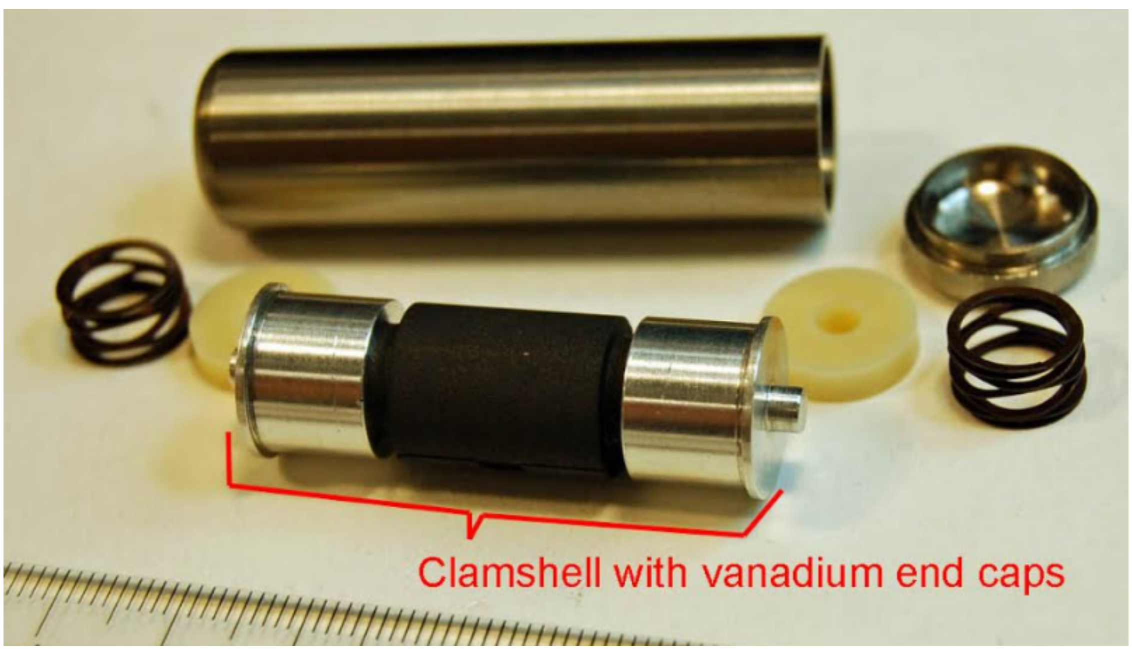

Figure 10. Photo of SAM-1 capsule prior to irradiation.

2.3 Cooperative Research and Development Agreement Support

2.3.1 Electric Power Research Institute-Zirconium Growth Experiments

The Electric Power Research Institute (EPRI)-zirconium growth (ZG) experiments are designed as drop-in static capsules and are being performed to examine the irradiation growth rate of several zirconium alloys as a function of fluence and hydrogen content and to evaluate the post-irradiation microstructure using TEM to evaluate the growth mechanism.

Specimens from the EPRI-ZG-A and EPRI-ZG-B continued to undergo TEM evaluation in EML. This work will continue for several years as test capsules reach their desired irradiation exposure and are removed from the reactor for analysis.

3. SHIPPING

Two significant shipments were made in FY 2017 in support of NSUF PIE activities. First, on June 21, 2017, a Level 3 milestone was met to receive irradiated UCB specimens at HFEF from PNNL. Second, receipt of Ultra and Drexel MAX specimens from the MIT reactor were received at MFC (HFEF and IMCL, respectively) on September 13, 2017, also meeting a Level 3 milestone.

4. MILESTONE SUMMARY

Milestone #	Description	Completion Date
M3UF-17IN0202023	Submit year end PIE summary report	9/27/17
M3UF-17IN0206052	Ship second sample set to Illinois Institute of Technology MRCAT	5/31/17
M3UF-17IN0206062	Complete PIE on second set of samples for the University of Illinois 8312	9/28/17
M3UF-17IN0206072	Ship second sample set to Illinois Institute of Technology MRCAT	7/20/17
M3UF-17IN0207022	Place contract with Westinghouse Electric Company	2/2/2017
M3UF-17IN0207212	Complete beam time at Illinois Institute of Technology MRCAT	3/6/2017