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Ion-Neutron Irradiated BOR60 Sample Preparation and Characterization: Nuclear Science User Facility 2017 Milestone Report

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OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

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Materials Science and Technology Division

ION-NEUTRON IRRADIATED BOR60 SAMPLE PREPARATION AND CHARACTERIZATION: NUCLEAR SCIENCE USER FACILITY 2017 MILESTONE REPORT

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ACRONYMS

APT	atom probe tomography
EBSD	electron backscatter diffraction
FIB	focused ion beam
HAADF	high angle annular dark field
LAMDA	Low Activation Materials Development and Analysis
NSUF	Nuclear Science User Facility
PI	principal investigator
RIS	radiation-induced segregation
S/TEM	scanning/transmission electron microscope (microscopy)
SEM	scanning electron microscope
TEM	transmission electron microscope (microscopy)
UM	University of Michigan

EXECUTIVE SUMMARY

This document outlines the results obtained by Oak Ridge National Laboratory (ORNL) in collaboration with the University of Michigan-led Consolidated Innovative Nuclear Research project, “Feasibility of combined ion-neutron irradiation for accessing high dose levels.” In this reporting period, neutron irradiated were prepared and shipped to the University of Michigan for subsequent ion irradiation. The specimens were returned to ORNL’s Low Activation Materials Development and Analysis facility, prepared via focused ion beam for examination using scanning/transmission electron microscopy (S/TEM), and then examined using S/TEM to measure the as-irradiated microstructure. This report briefly summarizes the S/TEM results obtained at ORNL’s Low Activation Materials Development and Analysis facility.

1. INTRODUCTION

In 2016, University of Michigan (UM) principal investigators (PIs) and an Oak Ridge National Laboratory (ORNL) co-investigator successfully proposed to study the microstructures obtained in LWRS-relevant materials by ion, neutron, and combined ion and neutron irradiation with the goal of determining the level of fidelity ion irradiation could obtain compared to neutron irradiation, all under a Nuclear Science User Facility (NSUF) access call. By studying the comparison of ion+neutron vs neutron-induced microstructures, this work will contribute to the Office of Nuclear Energy (NE) research goal to allow improved design of accelerated irradiation experiments.

This award provided funded access to an ORNL coinvestigator and, to ORNL's Low Activation Materials and Development Analysis (LAMDA) facility and its laboratory technicians to identify, prepare, survey specimens for use in the LAMDA microcopy laboratory. It also funded the user support on instruments including the focused ion beam (FIB) and transmission electron microscope (TEM).

This report documents the specimen preparation and characterization in successful completion of a Program Information Collection System Milestone M3UF-17OR0207042 "Complete Microstructural Characterization on Set 1 after Ion Irradiation."

2. SPECIMEN IDENTIFICATION AND METALLOGRAPHY

ORNL hosted Dr. Zhijie Jiao and graduated student, Samara Levine, in December 2016 to oversee the specimen preparations in the LAMDA facility. The UM PIs identified seven relevant samples, which were located and photographed by LAMDA laboratory technicians. From these samples, a total of 11 specimens were harvested. As shown in Figure 1, two coupons were cut from the broken BS13 tensile bar, and two were cut from the BS16 broken tensile bar.

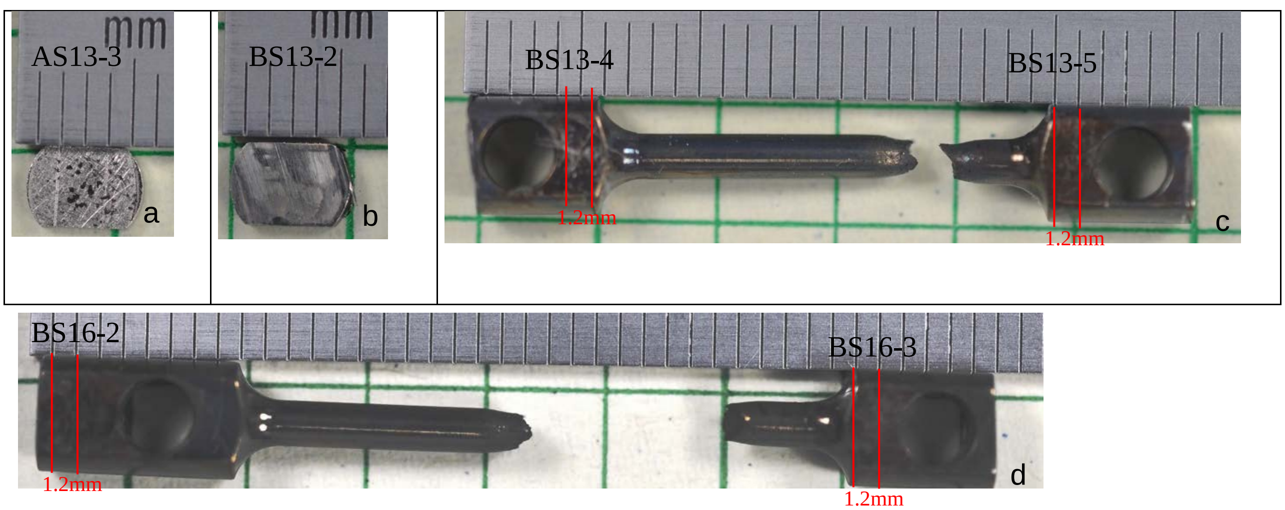


Figure 1. (a) AS13-3 coupon before polishing, (b) BS13-2 coupon before polishing, (c) BS13 tensile bar prior to cutting BS13-4 and BS13-5 as depicted, and (d) BS16 broken tensile bar prior to cutting specimens BS16-2 and BS16-3 as depicted.

After the rough-cut geometries of the final 11 samples listed in Table 1 were accepted by the PI, the specimens were mechanically polished on both sides and electropolished to provide the best specimen surface quality for ion irradiation experiments. Finally, they were surveyed and shipped to the UM Ion Beam Laboratory.

Table 1. Samples that were polished, electropolished, and shipped to UM

Sample ID	Alloy	Damage (dpa)
AS13-R1	SS304	5.5
AS13-3	SS304	5.5
AS14-3	SS304	5.5
AS17-R2	SS304	10.2
AS17-R3	SS304	10.2
AS18-R4	SS304	10.2
AS22-R6	SS304	47.5
BS13-2	SS304	4.8
BS13-3	SS304	4.8
BS13-4	SS304	4.8
BS16-2	SS304	10.2
BS16-3	SS304	10.2

Note: dpa = displacements per atom

After taking possession of the specimens, the UM PIs conducted ion irradiations and shipped four specimens back to ORNL for microstructural characterization. ORNL took possession of these four samples on July 31, 2017. During the week of August 10, ORNL hosted UM PIs, Dr. Zhijie Jiao and graduate student, Samara Levine.

3. FIB AND SPECIMEN PREPRATIONS

3.1 ELECTRON BACKSCATTER DIFFRACTION

Electron backscatter diffraction (EBSD) was performed in the ORNL LAMDA Versa3D dual-beam SEM-FIB instrument. Grain boundaries that were (1) high angle, (2) not coincident site lattice-related, and (3) made up of grains with <101> orientations nearly parallel the grain boundary plane's intersection with the sample surface, were selected for FIB preparation. This is illustrated in Figure 2, where two candidate grain boundaries are marked. This made it possible to analyze both radiation-induced dislocation structures and grain boundary radiation-induced segregation (RIS) in a single S/TEM sample, and reduced the sample preparation effort by roughly 50% compared to the need for separate RIS and dislocation measurement specimens.

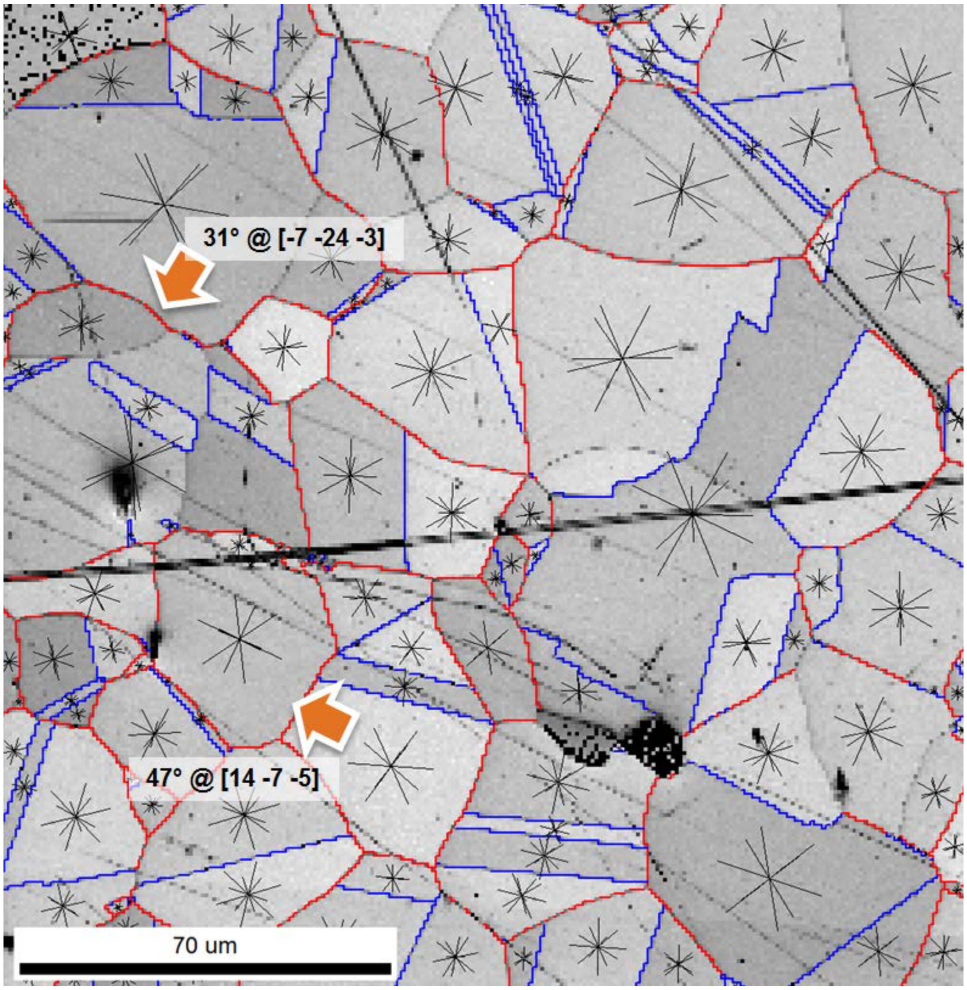


Figure 2. EBSD band contrast map (gray) with superimposed high-angle (red) and coincident-site-lattice (blue) grain boundaries. The thin black lines are projected <101> orientations for each grain. The two grain boundaries marked with orange arrows are high-angle boundaries with at least one grain having <101> nearly parallel to the boundary.

3.2 PREPARATION OF TEM SAMPLES

Samples were prepared for TEM and S/TEM measurements using standard FIB-based lift out methods. The specimens listed in Table 2 were prepared via EBSD-guided FIB for subsequent S/TEM characterization. In Figure 3, the lifting out across an identified high-angle grain boundary is illustrated.

Table 2. Samples prepared, and their irradiation conditions

Sample ID	Alloy	Neutron irradiation		Additional Ion irradiation	
		Damage (dpa)	Temperature (°C)	Damage (dpa)	Temperature (°C)
AS13-3	304	5.5	320	4.7	380
AS17-R3	304	10.2	320	—	—
AS22-R6	304	47.5	320	—	—

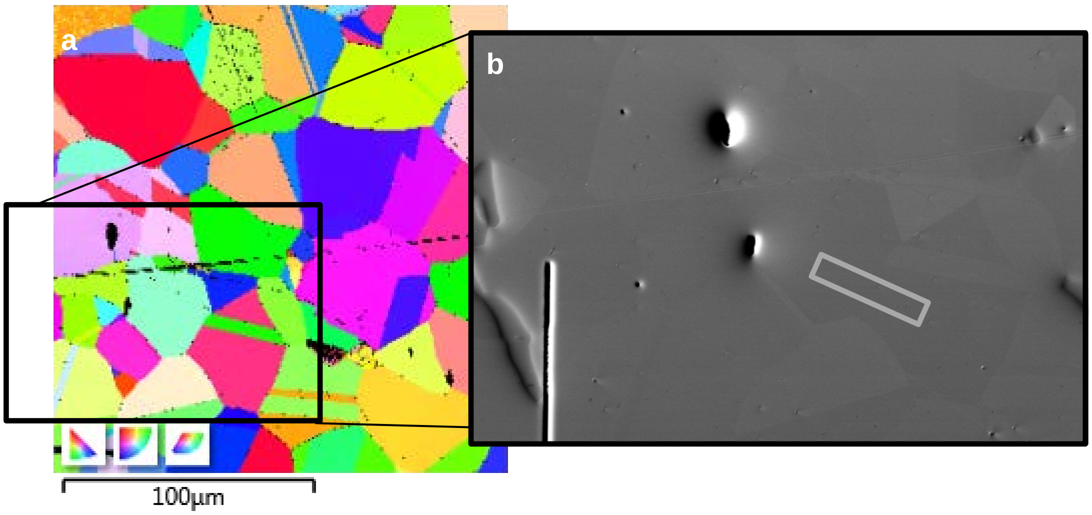


Figure 3. (a) EBSD map of AS22-R6. (b) A secondary electron SEM image of AS22-R6 high-angle grain boundary TEM lift out location. Full horizontal width of SEM image ~145 µm.

3.3 PREPARATION OF ATOM PROBE TOMOGRAPHY SPECIMENS

To characterize the ion irradiation and neutron damage to the surface of the specimens a number of tips were prepared from samples AS13-3, AS17-R2, and AS22-R6 for atom probe tomography (APT) measurements using standard FIB-based lift out methods. The APT samples were surveyed and shipped to UM where the APT experiment will be conducted.

4. S/TEM CHARACTERIZATION

The samples listed in Table 2 were examined using S/TEM, using the FEI[1] Talos F200X S/TEM tool in the ORNL LAMDA facility. TEM images utilizing two-beam conditions were acquired near the <101> zone axes to determine the dislocation structures in the materials and for subsequent offline analysis and quantification by the UM team. Examples from two of the three samples, from both the ion- and neutron-only ranges of AS13-3, are given in Figure 4. The final sample (AS22-R6) will be examined in TEM mode at UM.

[1] FEI is now Thermo Fisher Scientific.

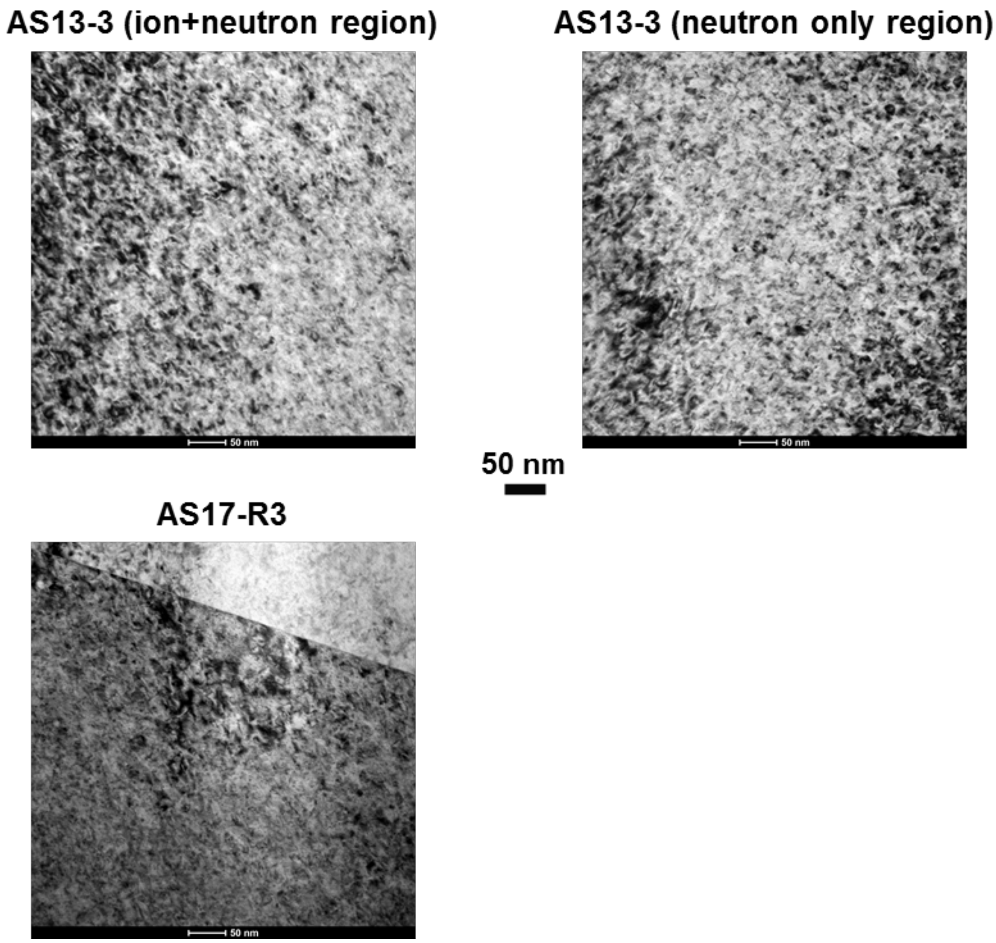


Figure 4. TEM images of the dislocation structures in two of the three samples, and both regions of sample AS13-3.

STEM images were also acquired, particularly of the FIB-prepared lift outs of grain boundary areas, and the grain boundaries were tilted to edge-on conditions using the S/TEM system sample goniometer. This is illustrated in Figure 5.

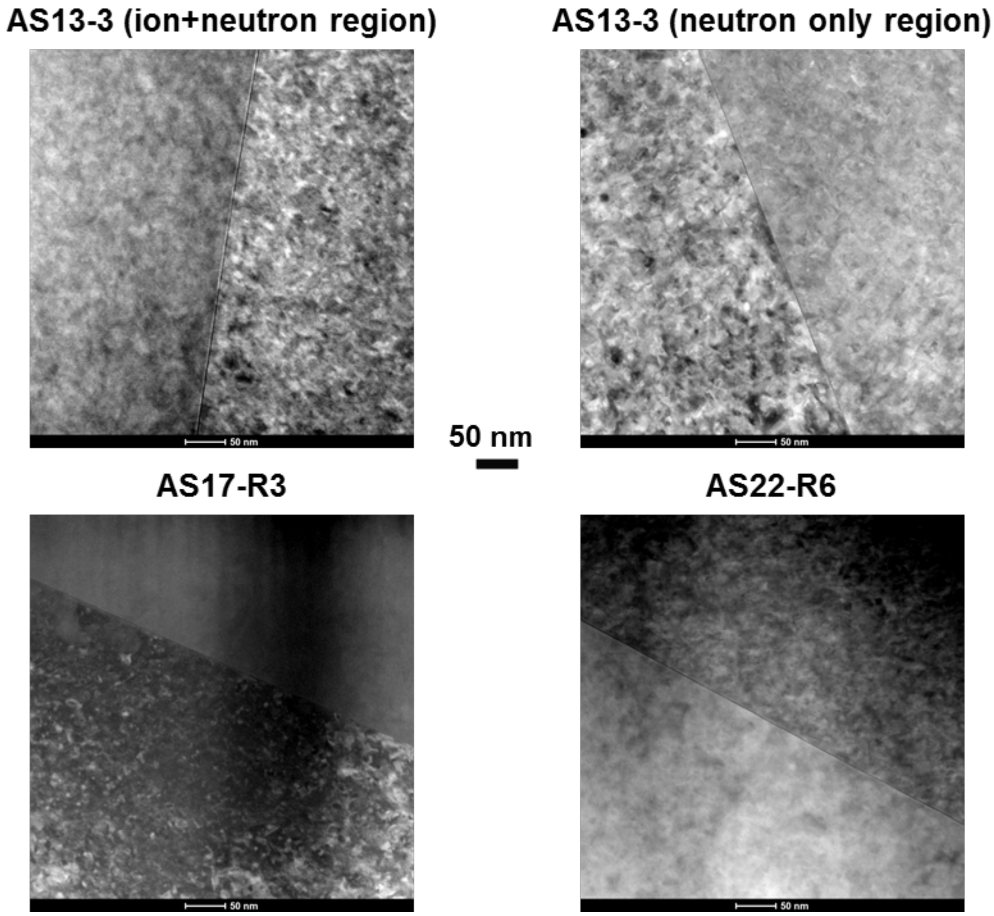
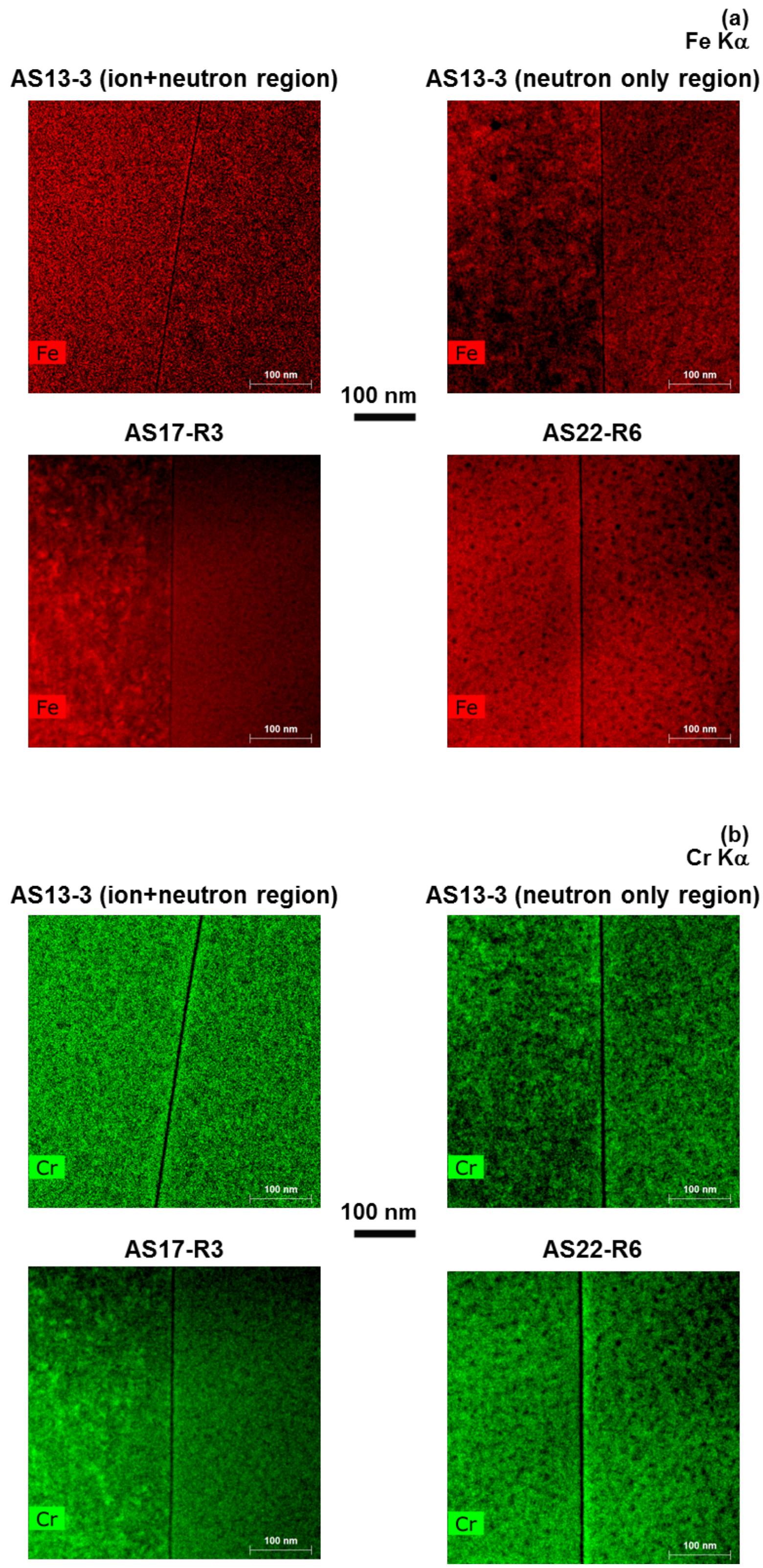
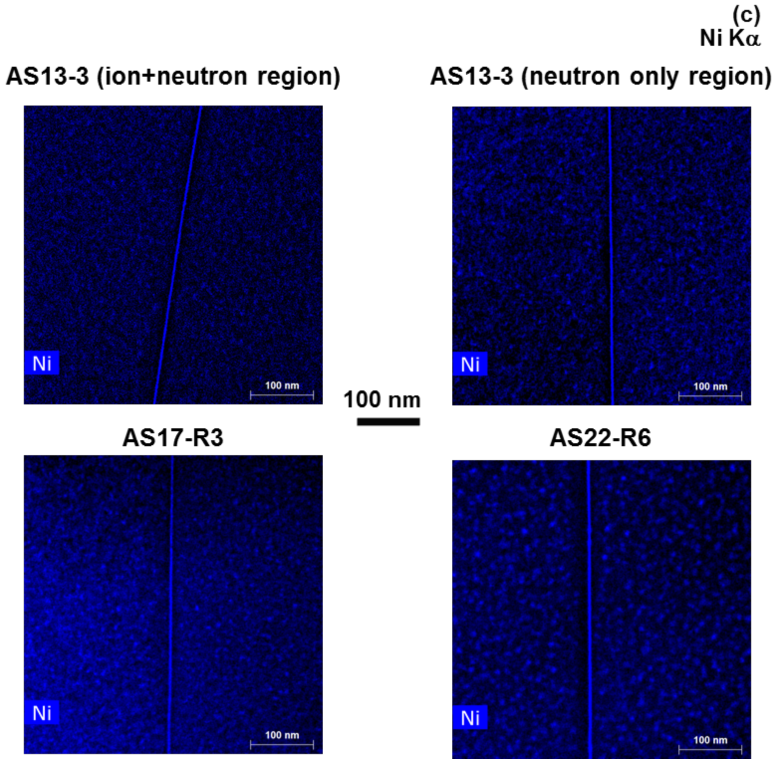


Figure 5. High-angle annular dark field STEM images of the grain boundaries in the three samples, and both regions of sample AS13-3.

Multiple STEM x-ray maps of the grain boundaries were obtained as well, from the three samples and both regions of sample AS13-3. Representative x-ray maps for the major alloying elements (Fe, Cr, Ni, and Si) are given in Figure 6, illustrating that significant RIS occurred on the boundaries during the irradiations. Because a hyperspectral imaging modality was used, all elements (e.g., P, Mo, etc.) can be analyzed from the stored data sets, not just the major elements illustrated here.



(a)
Fe Kα
AS13-3 (ion+neutron region)
AS13-3 (neutron only region)
Fe
100 nm
100 nm
AS17-R3
AS22-R6
(b)
Cr Kα
AS13-3 (ion+neutron region)
AS13-3 (neutron only region)
Cr
100 nm
100 nm
AS17-R3
AS22-R6



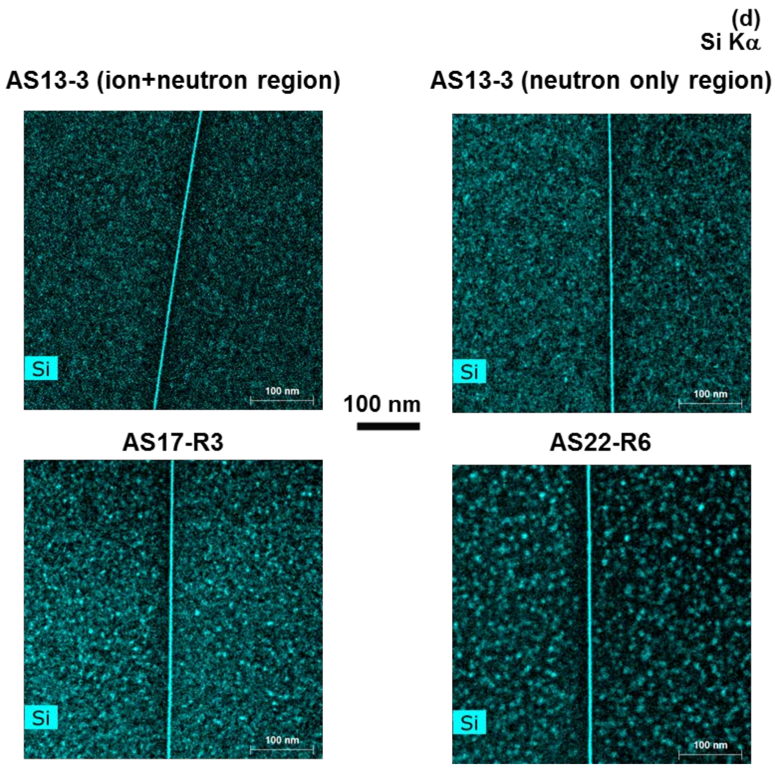


Figure 6. STEM x-ray maps of the grain boundaries in the three samples, and both regions of sample AS13-3. (a) Fe (b) Cr (c) Ni (d) Si. A 3 × 3 pixel-smoothing kernel is applied to each.

From Figure 6, the increasing irradiation doses (AS13-3 to AS17-R3 to AS22-R6) are seen to qualitatively provide increased densities and sizes of the Ni-Si rich precipitates. Further analyses of the maps are underway at UM.

Preliminary quantitative analyses of the boundary x-ray profiles for the four sample regions are given in Figure 7, indicating quantitatively large RIS values for the major elements. Further analysis and higher-fidelity quantification calculations are presently underway at UM.

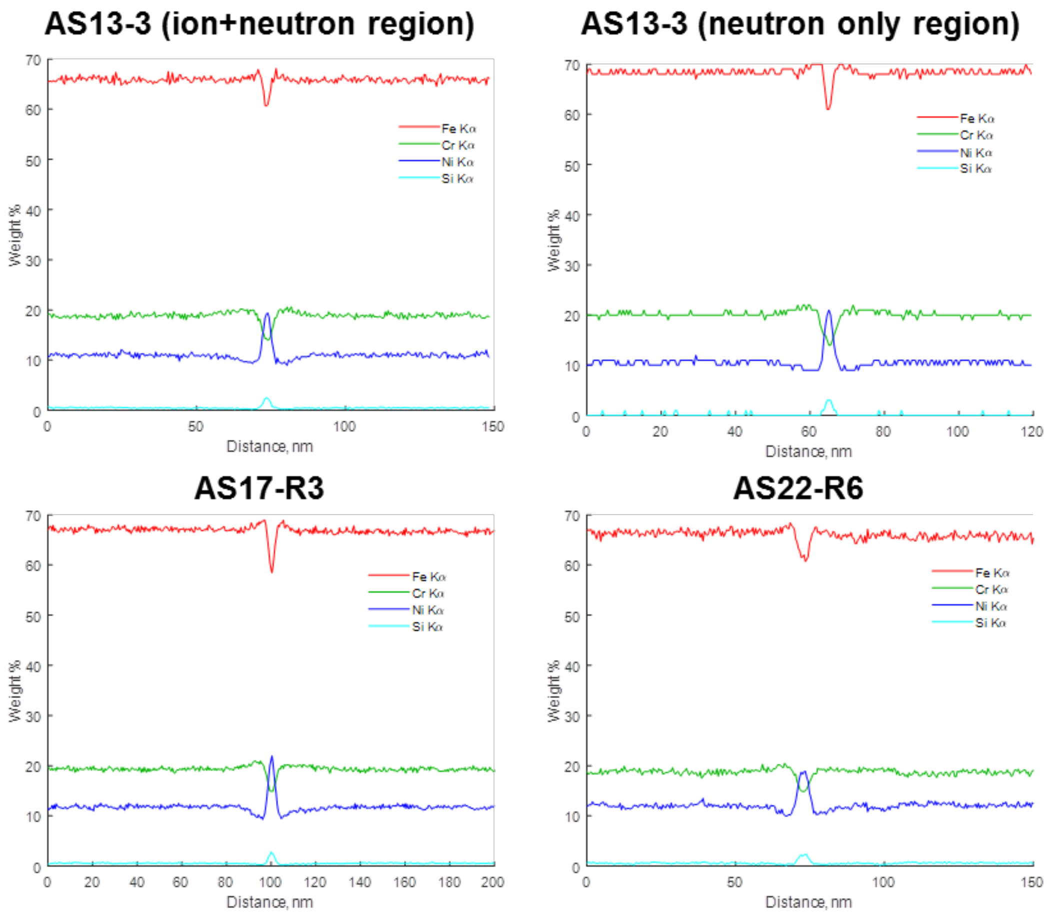


Figure 7. STEM x-ray map quantifications of the grain boundaries in the three samples, and both regions of sample AS13-3. Results are preliminary, not final.

5. CONCLUSION AND PATH FORWARD

The successful completion of 2017 NSUF milestone "Complete Microstructural Characterization on Set 1 after Ion Irradiation" included user-supported specimen identification, preparation, and shipment to UM for ion irradiation. The project continued in summer 2017 with EBSD, FIB specimen preparations and TEM. ORNL hosted UM visitors in both December 2016 and August 2017. The next round of user-supported characterization includes FIB specimen preparation and TEM on specimens in the LAMDA facility after additional ion irradiation at UM.