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Post-Irradiation Examination of Low-Neutron-Dose Metal Fuel Parallelepipeds

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Reactor and Nuclear Systems Division

POST-IRRADIATION EXAMINATION OF LOW-NEUTRON-DOSE METAL FUEL PARALLELEPIPEDS

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1. INTRODUCTION AND SUMMARY

Metal fuel samples were subjected to series of low-dose irradiations at the High Flux Isotope Reactor (HFIR) Hydraulic Tube (HT) Facility in FY 2012. The objective of these irradiations was to further fundamental scientific understanding of phenomena involved in the evolution of metal fuels microstructures under irradiation based on separate effects testing of various factors including elemental composition, temperature, and neutron fluence. These data would then be used to inform theoretical models for in-service performance of metallic fuels. Because metallic fuels are generally considered for fast spectrum reactor designs, specialized irradiation capsules, referred to as "rabbits," were designed with gadolinium thermal neutron shielding surrounding the samples. This design resulted in a fast-neutron spectrum comparable with the nominal in-reactor conditions for metal fuels.

Two sample geometries were included in these irradiations. The FCT irradiation series featured disk-shaped transmission electron microscopy (TEM)–style specimens, while the FCRP irradiation series featured parallelepiped specimens. Previous reports have detailed issues with capsule/sample interactions in the FCT irradiation (Edmondson, 2016) and attempts to scavenge useful data from the compromised samples (Helmreich, 2018). Lessons learned from the FCT irradiation series were successfully applied to the FCRP irradiation series through modifications to the capsule design to prevent interactions between the samples and the gadolinium shielding. The FCRP irradiation series was completed in FY 2017, and after a holding period in the HFIR cooling pond, the capsules were transferred to the Oak Ridge National Laboratory (ORNL) Irradiated Materials Examination and Testing (IMET) Facility for disassembly and then to the Low-Activation Materials Development and Analysis (LAMDA) Facility for materialographic preparation and analysis.

Microscopy and analysis of the depleted uranium (DU) and depleted uranium-zirconium (DUZr) FCRP samples have now been completed. These results revealed microstructural features present in the samples as a result of the fabrication process but no signs of irradiation damage at the length scales imaged. Further TEM analysis will be required to investigate early onset radiation damage effects such as dislocation loops.

2. FCRP IRRADIATION SERIES

The FCRP irradiation series specimens were inserted into HFIR using capsules, referred to as "rabbits," which could be inserted and ejected from the core during full-power operation. The ability to insert or eject capsules on demand was coupled with internal design features which modified the temperature and neutron spectrum. In concert, these design features enabled quasi-independent control of neutron fluence, neutron spectrum, and irradiation temperature. Control over neutron fluence was provided by a gadolinium outer capsule which shielded the metal fuel specimens from thermal neutrons to simulate a fast-reactor spectrum. As described in Section 1, a major issue identified in a preceding irradiation series was interaction between the metal fuel samples and the gadolinium shielding despite the inclusion of zirconia diffusion barriers. This issue was resolved in the FCRP capsule design by completely separating the shielding from the samples in a dual-capsule design with a more substantial diffusion barrier, as shown in Figure 1.

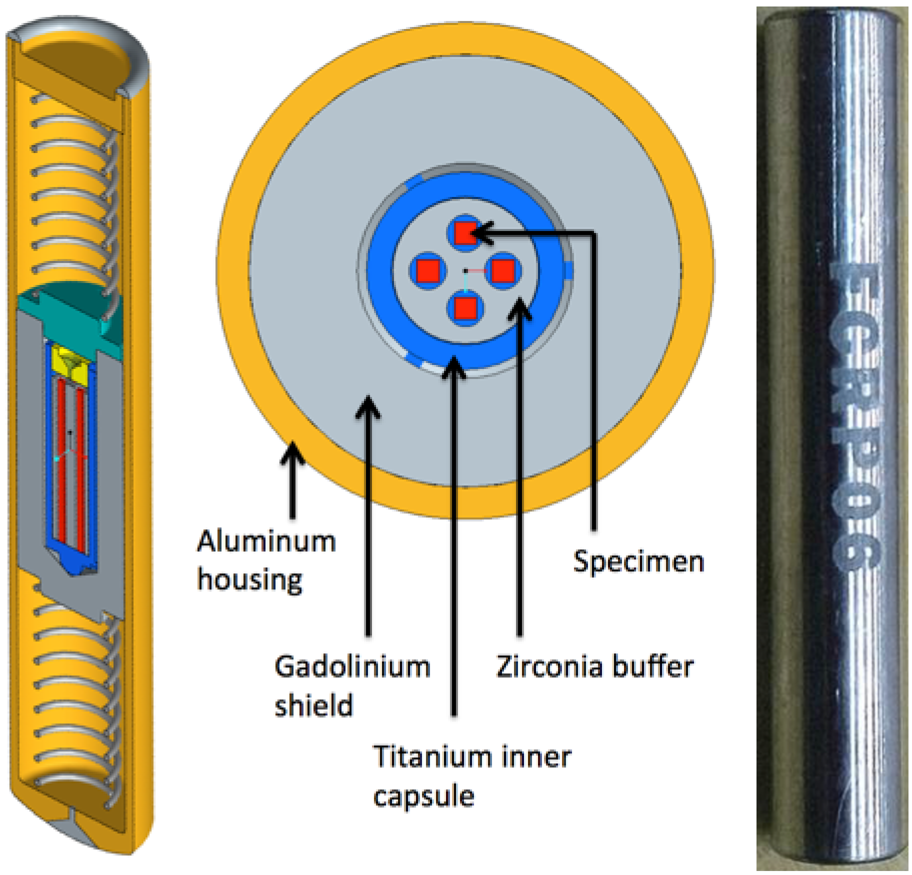

Figure 1. Schematic of FCRP irradiation capsule featuring gadolinium thermal neutron shield in a dual-capsule design (left) and a picture of a loaded FCRP irradiation capsule (right).

A series of six FCRP capsules were irradiated at HFIR under the irradiation conditions summarized in Table 1. This report describes the disassembly of capsules FCRP-04, FCRP-05, and FCRP-06 and the characterization of their respective DU-20Zr, DU, and DU-10Zr specimens.

Table 1. Summary of irradiation conditions for the FCRP series irradiations.

Capsule	Specimen Material	Irradiation Time (hours)	Fluence ($\times 10^{22}$ n/m^2)	Dose (dpa)	Irradiation Cycle
FCRP-01	Stainless Steel	1.3	0.29	-	462
FCRP-02	Stainless Steel	13	2.9	-	462
FCRP-03	Stainless Steel	130	28	-	462
FCRP-04	DU-20Zr	13	2.9	0.012	468
FCRP-05	DU	130	28	0.12	468
FCRP-06	DU-10Zr	180	39	0.17	471

3. CAPSULE DISSASEMBLY AND SAMPLE PREPARATION

After irradiation in HFIR and the requisite cooling, capsules FCRP-04, FCRP-05, and FCRP-06 were transferred to hot cells within the ORNL IMET facility for disassembly. The capsule/specimen interactions endemic in the FCT irradiation series did not occur in the FCRP samples, and removal from the capsules proceeded smoothly (as shown in Figure 2) with the exception of the SiC temperature monitor in FCRP-04, which stuck in the capsule. Metal fuel specimens, along with silicon carbide (SiC) thermometry monitors, were transferred to the ORNL LAMDA facility for analysis. Materialographic mounts were prepared using three sections from each of the three parallelepiped samples: one each from the top, bottom, and center.

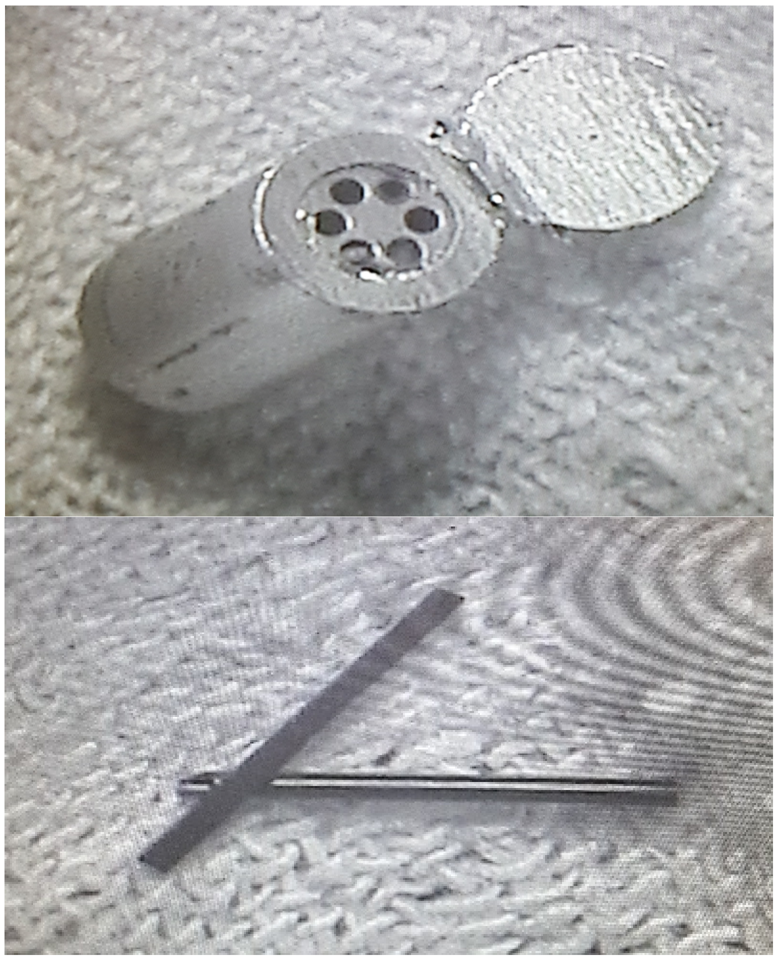

Figure 2. Opened FCRP capsule (top) and SiC temperature monitor and uranium rod after removal (bottom).

Silicon carbide temperature monitors were extracted from FCRP-05 and FCRP-06 and evaluated in LAMDA using dilatometry measurements. These measurements, shown in Figure 3, indicate a nominal irradiation temperature of 800°C for both capsules. No thermometry was included in FCRP 04; however, its position in the same irradiation cycle as FCRP-05 should have resulted in the same irradiation temperature.

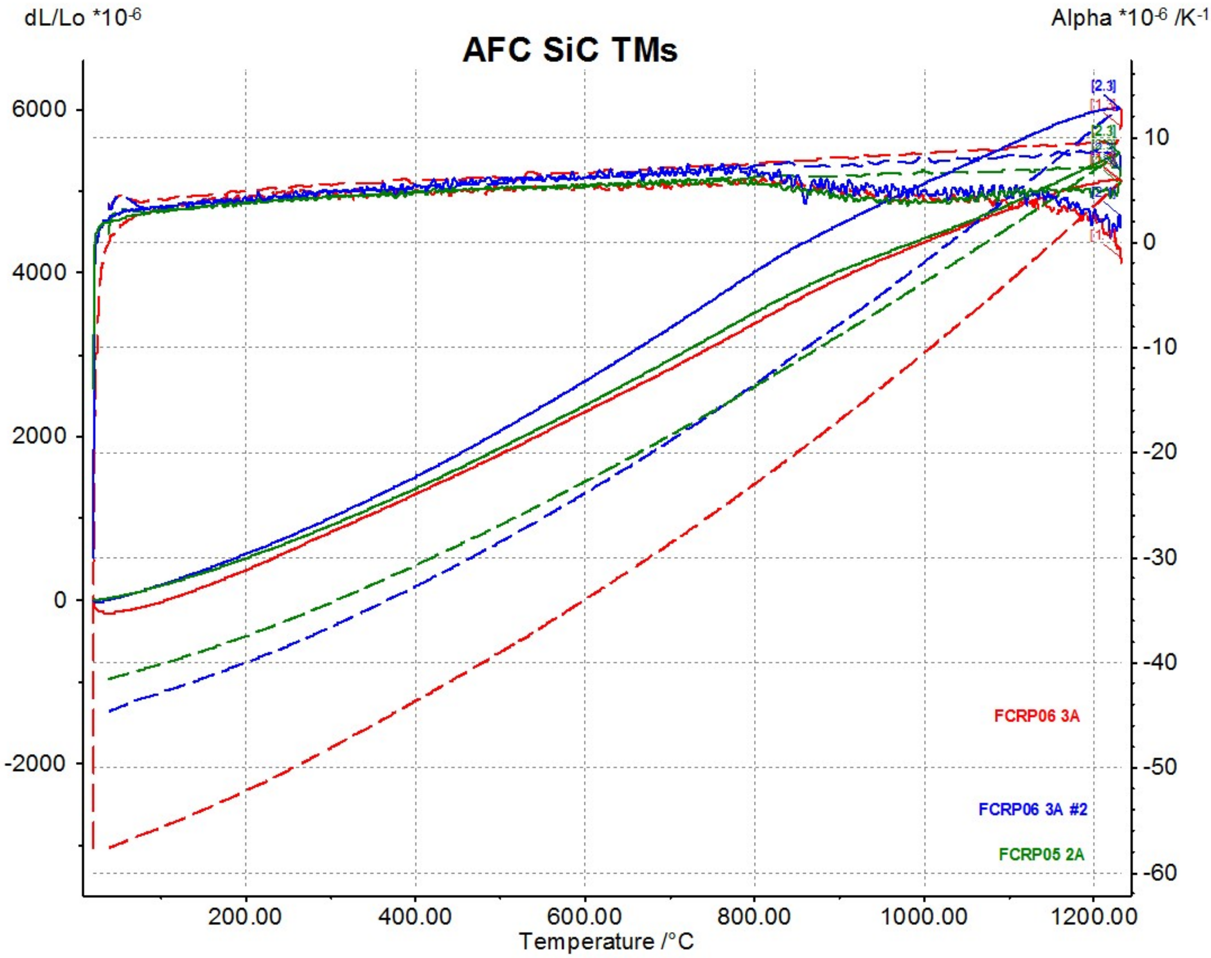

Figure 3. Dilatometry results for SiC thermometry from FCRP-05 and FCRP-06 showing an irradiation temperature of ~800°C.

4. SEM AND EDS ANALYSIS OF SAMPLES

The exploratory post-irradiation examination (PIE) analysis completed on the cross-sectioned FCRP samples consisted of scanning electron microscopy (SEM) imaging and energy dispersive x-ray spectroscopy (EDS) elemental mapping using LAMDA's FEI Versa 3D dual-beam focused ion beam (FIB) system, equipped with a Schottky field emitter for high-resolution imaging, and an Oxford Instrument XMax^{N}-150 EDS collimated detector for EDS measurements.

Images of the top, middle, and bottom cross sections of FCRP-05 are shown in Figure 4–Figure 6. There is significant tearing within the sample and a large population of bubbles. Considering the exceptionally low dose to which this sample was irradiated (0.12 dpa), these microstructural features are certainly a by-product of the original sample fabrication, which was completed using gravity casting.

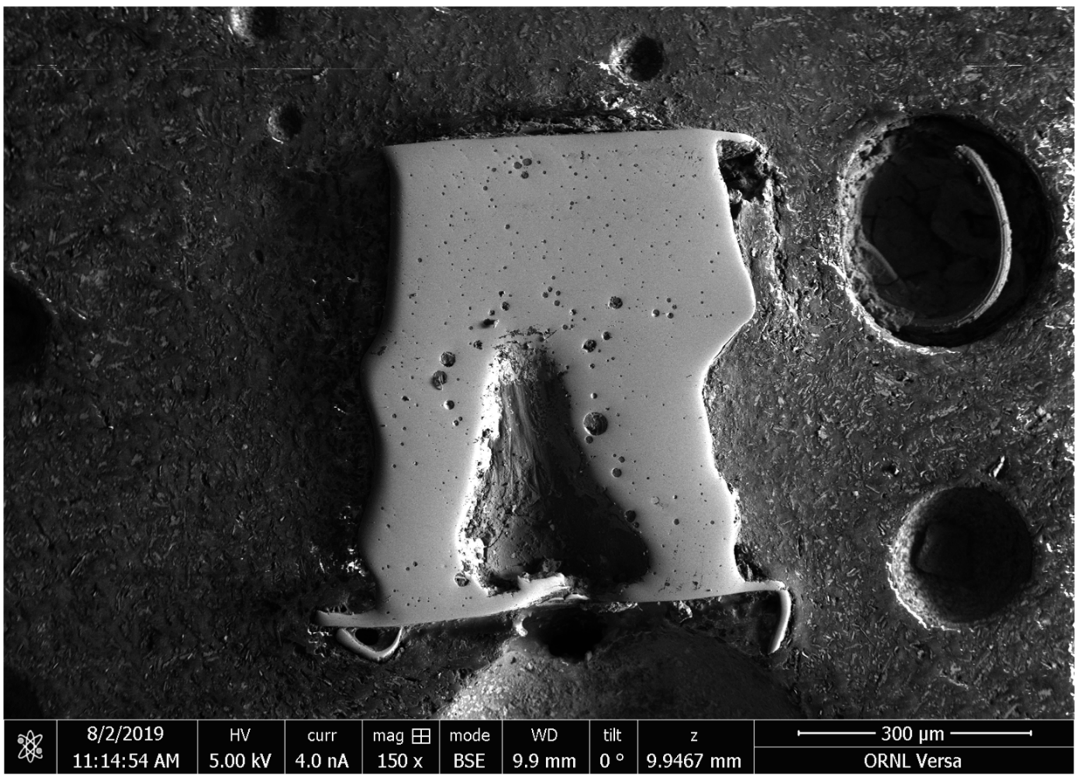

Figure 4. Top cross section from FCRP-05 DU sample irradiated to 0.12 dpa at 800°C.

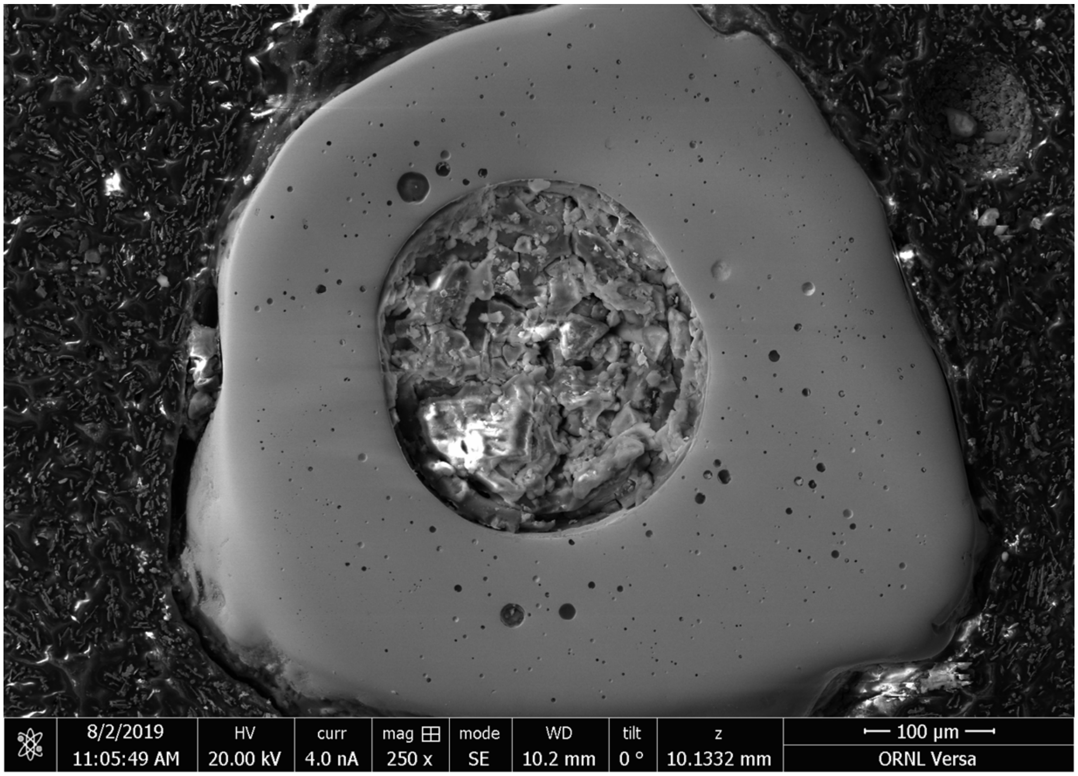

Figure 5. Middle cross section from FCRP-05 DU sample irradiated to 0.12 dpa at 800°C.

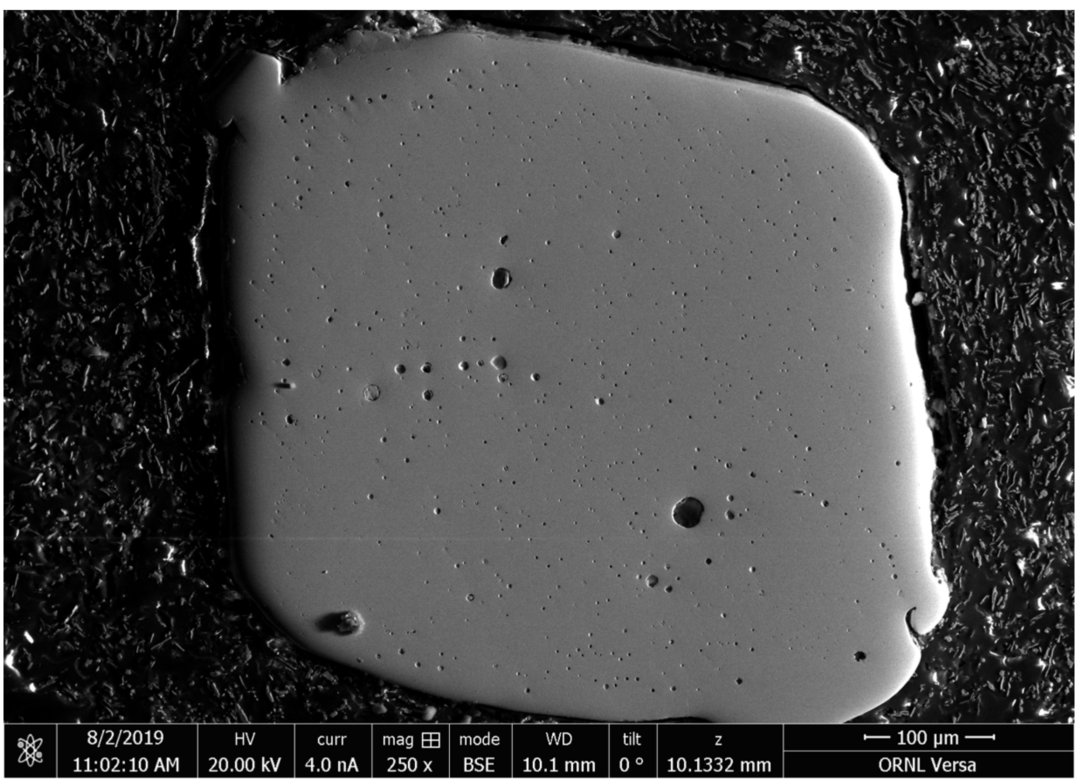

Figure 6. Bottom cross section from FCRP-05 DU sample irradiated to 0.12 dpa at 800°C.

Images of the top, middle, and bottom cross sections of FCRP-05 are shown in Figure 7–Figure 9. As with FCRP-04, there is some internal porosity which is likely due to fabrication defects; however, there are also fine dark features interspersed throughout the material (shown at higher magnification in Figure 10). EDS mapping showed that these small dark features were zirconium-rich precipitates.

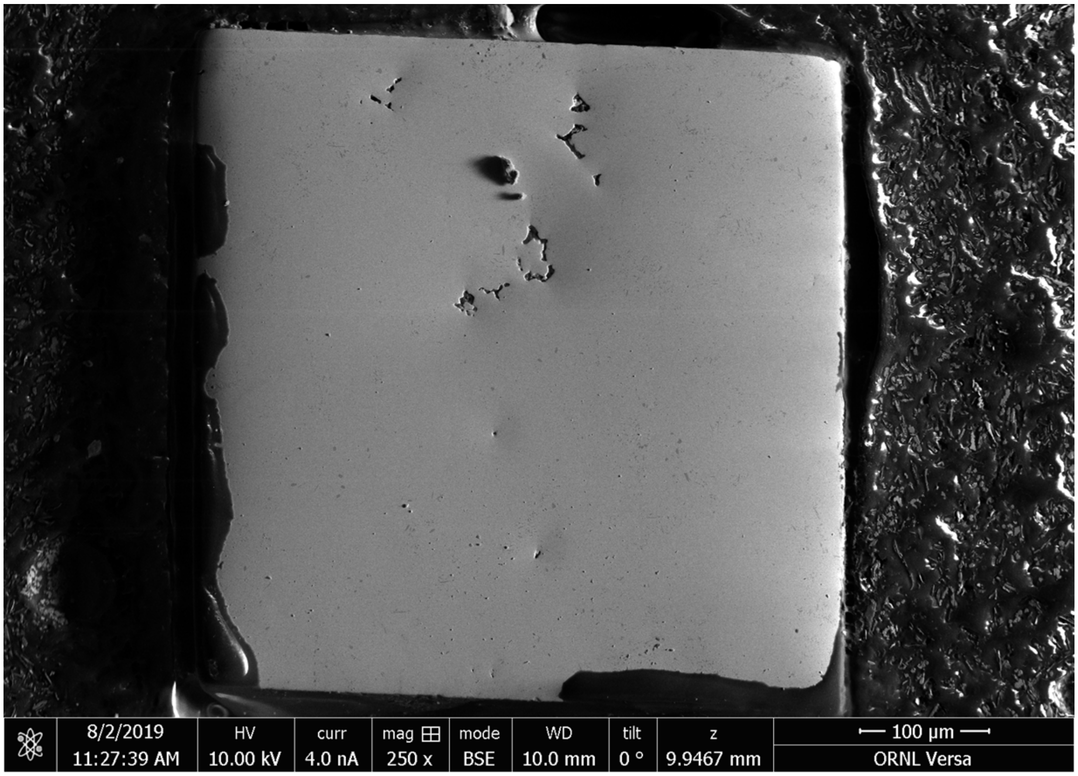

Figure 7. Top cross section from FCRP-04 DU-20Zr sample irradiated to 0.012 dpa at 800°C.

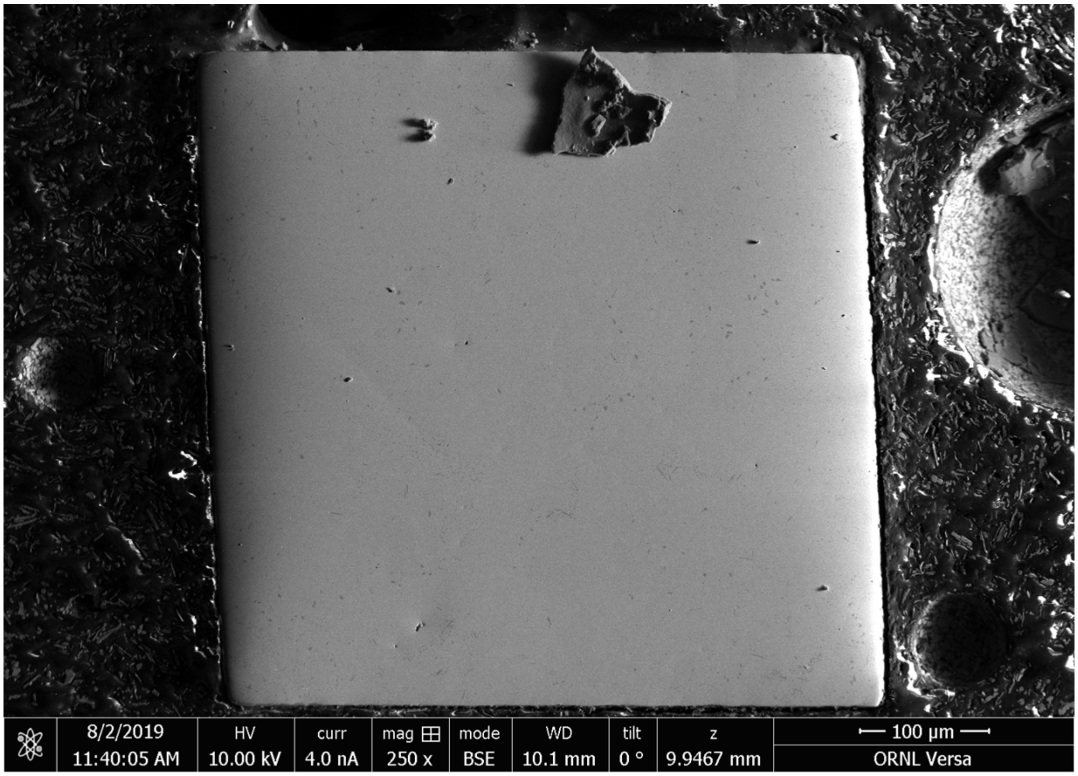

Figure 8. Middle cross section from FCRP-04 DU-20Zr sample irradiated to 0.012 dpa at 800°C.

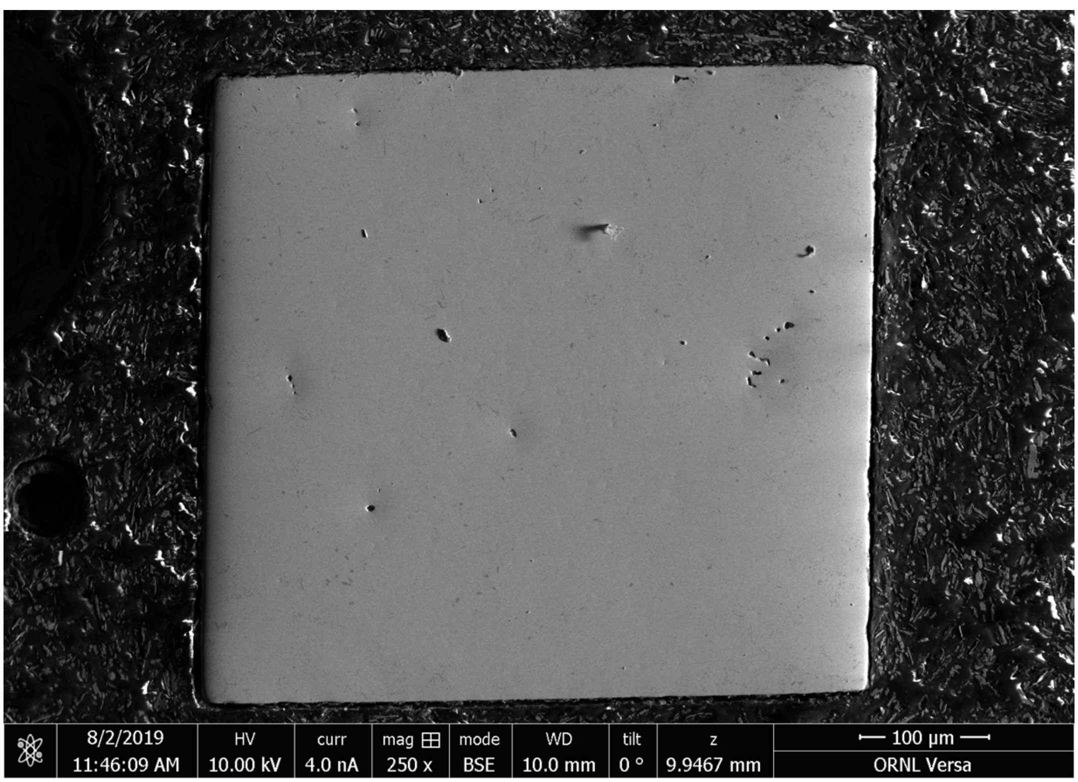

Figure 9. Bottom cross section from FCRP-04 DU-20Zr sample irradiated to 0.012 dpa at 800°C.

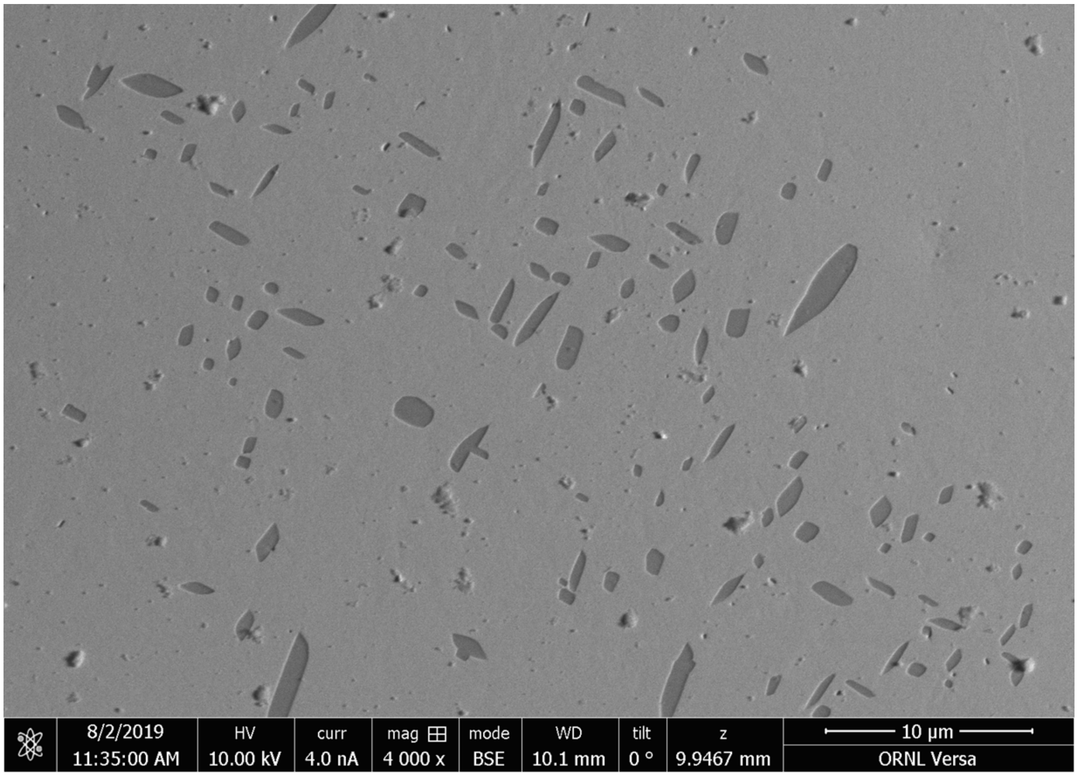

Figure 10. Dark inclusions identified in FCRP-04 DU-20Zr sample irradiated to 0.012 dpa at 800°C.

The final sample, FCRP-06, contained 10 wt% of zirconium. As with FCRP-04, high-zirconium inclusions are dispersed throughout the cross sections, as shown in Figure 11–Figure 13.

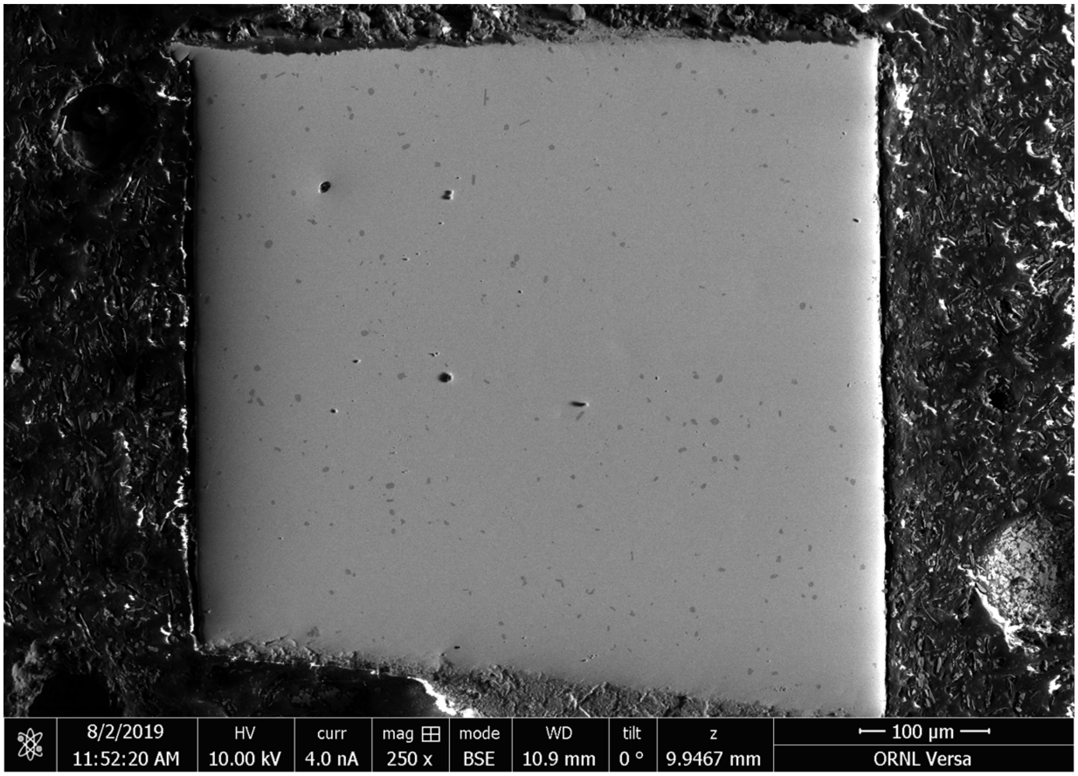

Figure 11. Top cross section from FCRP-06 DU-10Zr sample irradiated to 0.17 dpa at 800°C.

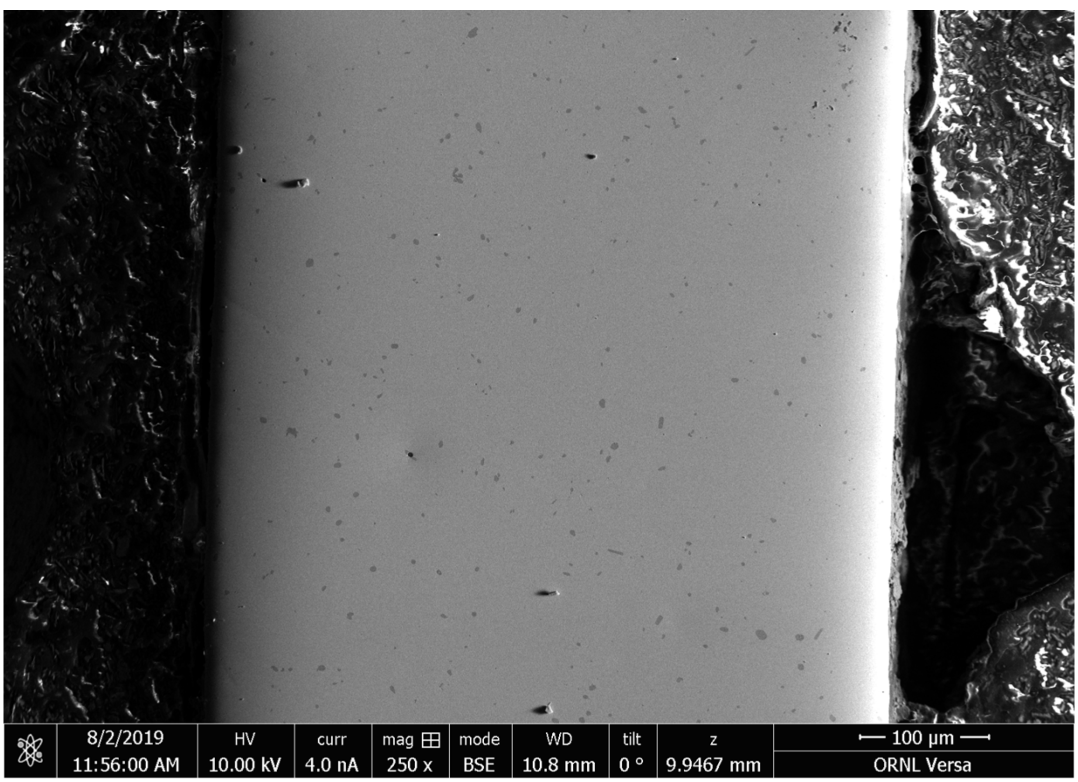

Figure 12. Middle cross section from FCRP-06 DU-10Zr sample irradiated to 0.17 dpa at 800°C.

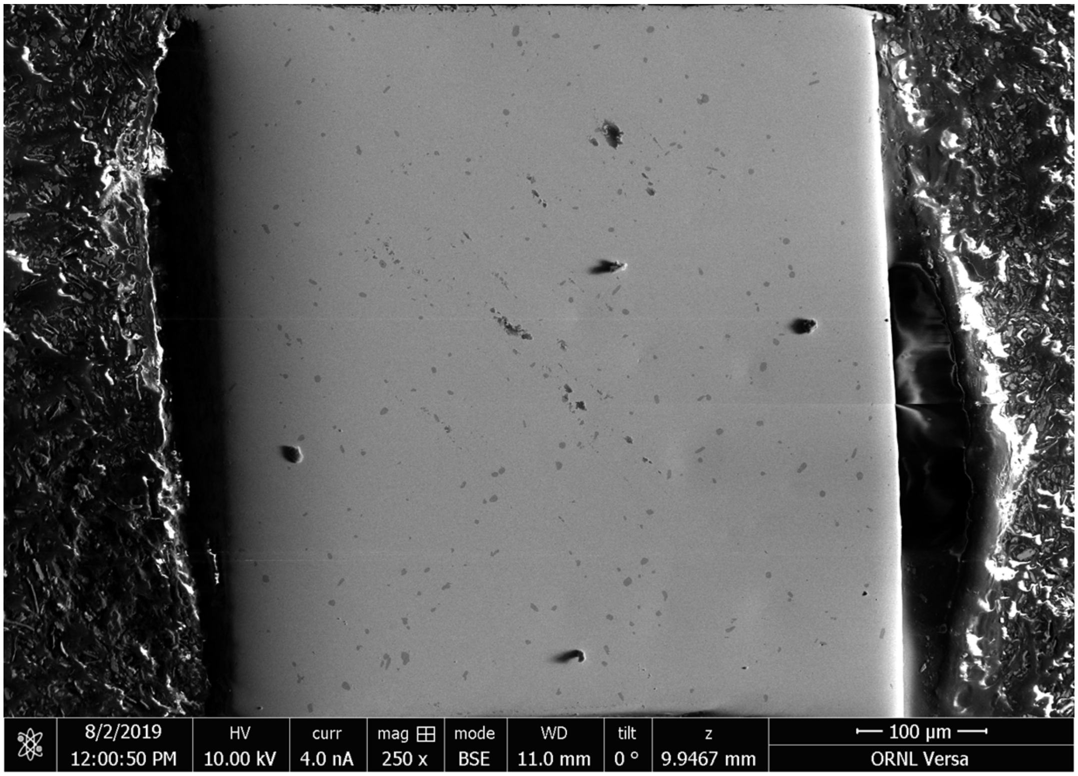

Figure 13. Bottom cross section from FCRP-06 DU-10Zr sample irradiated to 0.17 dpa at 800°C.

5. CONCLUSIONS

The FCRP irradiation series was intended to provide data on the development of irradiation microstructures in metal fuels based on low-dose irradiations of parallelepiped specimens in HFIR. Capsule design updates based on issues encountered during the preceding FCT irradiation series resulted in a successful experiment with intact samples. Analysis of SiC thermometry included within the FCRP capsules indicates an irradiation temperature of 800°C. Initial analysis of cross sections from each of the FCRP samples by SEM was sufficient to show general microstructural characteristics, primarily porosity due to fabrication and zirconium-rich inclusions in alloyed samples. Given the exceptionally low dose levels these samples received, it is likely that further analysis by TEM of features such as dislocation loops will be necessary to observe any irradiation effects.

6. REFERENCES

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