doi:10.1017/S1431927614010769
Microsc. Microanal. 20 (Suppl 3), 2014


# Microstructural Characterization of the Irradiated Nuclear Fuels

J. Gan[1], B.D. Miller[1], D.D. Keiser[1], Jr., J. Jue[1], A.B. Robinson[1], J.W. Madden[1], P.G. Medvedev[1], D.M. Wachs[1]

[1]Nuclear Fuels and Materials Division, Idaho National Laboratory, Idaho Falls, ID 83415, USA

The microstructural characterization using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) for the irradiated fuels played an important role to the understanding of fuel performance. Significant progresses have been made in recent years on SEM and TEM work for fuel development in reduced-enrichment for research and test reactors (RERTR) program [1, 2]. It is extremely challenging to prepare the samples from the highly radioactive irradiated fuel for high resolution microscopy analysis. For the complex microstructure of irradiated fuels, the traditional mechanical polishing tends to produce a smeared and disturbed surface making it difficult to reveal the original microstructure in SEM while the traditional TEM sample preparation often limits the ability to access the areas of interest for detailed analysis. The new development using the focused-ion-beam (FIB) lift-out and polishing technical at the Idaho National Laboratory (INL) demonstrated the great advantage in microstructural characterization for the irradiated nuclear fuels.

This work is based on the SEM and TEM characterization of an irradiated dispersion fuel plate (U-7Mo/Al-5Si, Plate ID: R3R050). A small piece (1.0 mm dia.) was punched from the irradiated fuel plate (~1.5 mm thick) with its local fission density and centerline irradiation temperature of $5.2 \times 10^{21}$ fissions/cm$^3$ and 130°C, respectively. SEM Met-mount sample was prepared, characterized and then loaded into a dual-beam FEI Quanta3D FIB/SEM system to (1) perform SEM analysis of the ion-trenched and undisturbed microstructure and (2) prepare TEM samples from the areas of interest identified in the SEM work. The SEM information produced from the FIB/SEM system provides information that is critical complementary to the standard SEM data. TEM characterization was carried out using a 200 keV TEM/STEM system equipped with a LaB6 filament, a 2048×2048 pixel and 16-bit digital camera for imaging and a Si drift detector for EDS composition analysis.

Figure 1 shows a comparison of mechanical polished surface vs. the FIB ion beam produced undisturbed surface for SEM imaging (middle). The detailed microstructure of fuel particle interior (left) and fuel-matrix-interaction (FMI) layer (right) are clearly revealed with large round bubbles in a fuel zone (4-8 μm) right next to the FMI. It was found that this is amorphous zone with high Si content. At the given fission density, the large bubbles in the fuel particle developed an amorphous shell-type substructure consisting of solid fission product such as Zr, Sr, Ba, Ce and Y. These informations cannot be obtained from the conventional SEM met-mount sample. Figure 2 shows an overview of a FIB-TEM lamella (left) and the high resolution view of the clean area of fuel particle (middle) at roughly 15% volume fraction containing fission gas bubble superlattice (GBS) in fcc structure imaged at zone [110] of bcc U-Mo, along with a schematic (right) showing orientation relationship between GBS and U-Mo. GBS is a key feature for the U-Mo fuel performance.

In the fuel particle interior containing large bubbles, the complex irradiated microstructure shows the heterogeneous characters as illustrated in Figure 3. It captured the process of GBS collapse and the development of large bubbles. These areas only exhibit diffused rings in selected area diffraction patterns. It is believed that these areas originally consist of GBS at lower fission density. The

development of large bubbles through bubble coarsening is evident. The process appears heterogeneous with some areas showing multiple large bubble formation and the other area is relatively clean. This work was supported by the U.S. Department of Energy (DOE), Office of Nuclear Security Administration (NNSA) [3].

References.

[1] S. Van de Berghe et al., J. Nucl. Mater., 375 (2008) 340. [2]
J. Gan et al., J. Nucl. Mater., 396 (2010) 234.
[3] Funding was supported by DOE/NNSA to the RERTR program at INL, operated by Battelle Energy Alliance, LLC, under DOE Idaho Operations Office Contract DE-AC07-05ID14517.

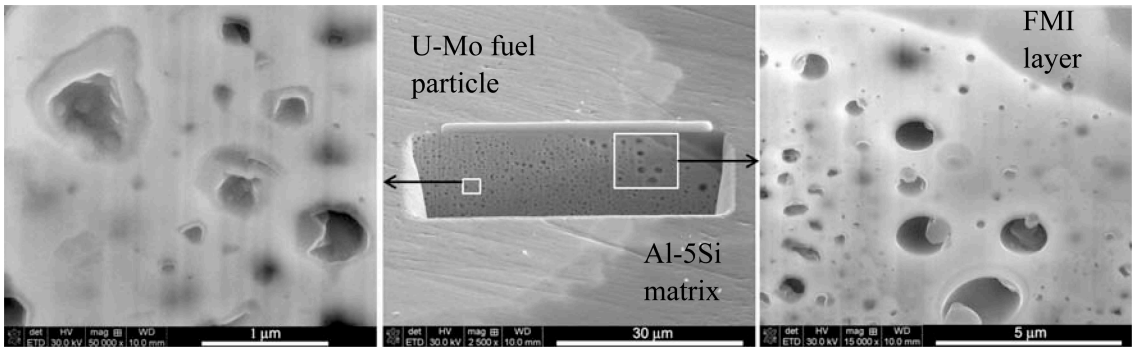


**Figure 1.** SEM images of an irradiated fuel particle from a FIB sectioned surface.

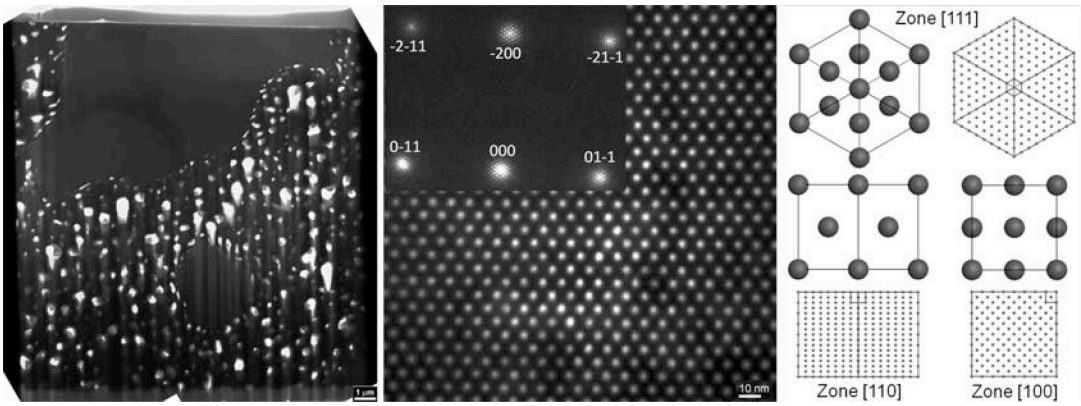


**Figure 2.** TEM images of a FIB lamella made from a fuel particle (left), fission gas bubble fcc superlattice from the clean areas shown on the left at zone [110] of bcc U-Mo (middle) and a schematic (right) showing orientation relationship between bubble superlattice and U-Mo bcc lattice.

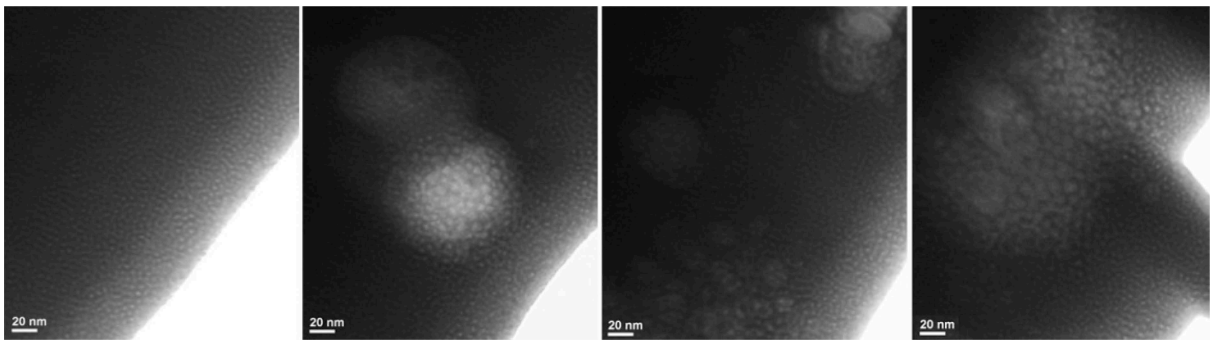


**Figure 3.** TEM images showing collapse of bubble super lattice and development of large bubbles.

https://doi.org/10.1017/S1431927614010769 Published online by Cambridge University Press