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SEM CHARACTERIZATION OF AN IRRADIATED MONOLITHIC U-10MO FUEL PLATE

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ABSTRACT

Results of scanning electron microscopy (SEM) characterization of irradiated U-7Mo dispersion fuel plates with differing amounts of matrix Si have been reported. However, to date, no results of SEM analysis of irradiated U-Mo monolithic fuel plates have been reported. This paper describes the first SEM characterization results for an irradiated monolithic U-10Mo fuel plate. Two samples from this fuel plate were characterized. One sample was produced from the low-flux side of the fuel plate, and another was produced at the high-flux side of the fuel plate. This characterization focused on the microstructural features present at the U-10Mo foil/AA6061 cladding interface, particularly the interaction zone that had developed during fabrication and any continued development during irradiation. In addition, the microstructure of the foil itself was investigated, along with the morphology of the observed fission gas bubbles. It was observed that a Si-rich interaction layer was present at the U-10Mo foil/cladding interface that exhibited relatively good irradiation behavior, and within the U-10Mo foil the microstructural features differed in some respects from what is typically seen in the U-7Mo powders of an irradiated dispersion fuel.

1. Introduction

To assess the irradiation performance of U-10Mo monolithic fuel plates, reactor experiments have been conducted using the Advanced Test Reactor (ATR). To determine the microstructural development that occurred in these fuel plates during irradiation, optical metallography (OM) is performed in the Hot Fuel Examination Facility (HFEF), located at the Idaho National Laboratory in Idaho Falls, Idaho. To complement the optical metallography characterization, it is of interest to characterize the microstructure of selected fuel plates using scanning electron microscopy with energy dispersive and wavelength dispersive spectroscopy (SEM/EDS/WDS). This type of analysis allows for the resolution of smaller microstructural features and for the qualitative determination of composition in different regions of the fuel plate. This paper describes the first SEM characterization results produced for an irradiated monolithic fuel plate. The analyzed fuel plate was labeled L1F100 from the RERTR-6 experiment and was fabricated using the friction bonding (FB) process. The FB monolithic plates that were tested as part of the RERTR-6 experiment were irradiated at moderate powers (surface heat flux ~140-175 W/cm²), at high temperatures (centerline temperature at BOL ~116-180°C) and at moderate burnups (~50% LEU) [1].

2. Experimental
2.1 Fuel Plate Fabrication and Characterization

The FB process used to fabricate RERTR-6 monolithic fuel plates is a solid-state process where a rotating tool is rastered across both sides of a fuel plate to produce good bonding at the U-
10Mo foil/AA6061 cladding interface [2]. After completion of the FB process, two additional fabrication steps were applied to the RERTR-6 fuel plates that are not typically used to fabricate current generation fuel plates. The first step was called the “flattening step” and the second step was named the “homogenization anneal.” Both of these steps involved exposure of the fuel plates to high temperatures. The “flattening step” was employed due to warpage of the fuel plates as a result of FB, which was too severe to allow for final processing (thinning, surface finishing, shearing etc.). During this step, the plates were loaded into a heated platen hydraulic press heated to 385 °C and pressed at ~200 psi for 3-4 minutes. After this step, the plates were determined to be flat enough for the subsequent processing steps. The “homogenization anneal” step was employed to try and eliminate heterogeneous microstructures in the fuel plates that could be present after FB and potentially cause enhanced corrosion of the fuel plates. The homogenization anneal was performed at 500˚C for 30 minutes in the heated plan press under light force (typically 500 lb) to ensure good contact between the platens and the plate surface. For fuel plates tested after RERTR-6, neither the flattening step nor the homogenization annealing step has been employed since FB can now be routinely employed to produce flat plates, and the FB process has been found to not produce the same inhomogeneities that have been observed for another process that exposes materials to a rotating tool, viz: friction stir welding.

For conducting microstructural characterization of as-fabricated FB fuel plates, transverse cross sections were used. OM analysis was employed to characterize the U-10Mo foil and AA6061 cladding, and SEM/EDS/WDS was employed to interrogate the U-10Mo foil/AA6061 cladding interface.

2.2 Irradiated Fuel Plate Characterization

In HFEF, OM was performed on a transverse cross section taken from the mid-plane of the fuel plate. SEM analysis was performed on two one-mm-diameter cylindrical samples that were generated from the fuel plate in HFEF using a punching process. These samples were transferred to the Electron Microscopy Laboratory (EML) where they were mounted, polished, and examined using SEM/EDS/WDS. Focus was given to the interface between the fuel foil and cladding and the microstructural features within the U-10Mo alloy.

3. Results and Discussion

3.1 Archive Fuel Plate Characterization

Optical images of the U-10Mo foil microstructures that were present after FB and after the homogenization anneal are presented in Fig. 1. The U-10Mo foils after FB were highly textured, but the homogenization anneal produced an equiaxed microstructure in the U-10Mo foil.

![Fig. 1. Optical Images of a U-10Mo foil microstructure after (a) friction bonding and (b) the exposure to 500˚C for 30 minutes during the homogenization anneal. The grains in (b) are around 10 μm in size.](image-url)
The SEM/EDS/WDS analysis results showed that interaction between the U-10Mo foils and AA6061 cladding had occurred during the fuel fabrication steps. Fig. 2 shows the two types of interaction zones that were observed at the foil/cladding interface for archive fuel plate L1F110. One layer (Layer 1) was very thin and the other layer (Layer 2) was thicker. Composition analysis indicated that Layer 1 was enriched in Si and Layer 2 contained varying amounts of a Si-rich phase (bright contrast) and Si-deficient phase (dark contrast). Si-rich precipitates were observed in the U-10Mo foil due to the presence of Si impurity in the original U-10Mo alloy. More detailed descriptions of the characterization that was performed on RERTR-6 archive FB fuel plates with U-10Mo foils can be found in Refs. 3 and 4.

**Fig. 2.** An SEM backscattered electron image of a cross section of fuel plate L1F110, where a thin (Layer 1) and thick (Layer 2) interaction layer could be observed at the interface between the U-10Mo (bright) and AA6061 cladding (black). In Layer 2 the brighter contrast phase is enriched in Si and the darker phase contains negligible Si.

### 3.2 Irradiated Fuel Plate Characterization
#### 3.2.1 Optical Metallography

The optical micrographs in Figs 3a and 3b identify two different interaction layer morphologies observed at the U-10Mo/AA6061 interface for fuel plate L1F100. One layer is relatively thick (Fig. 3a) and seems to be comprised of two different types of phases, and the other other is relatively thin and uniform (Fig. 3b). Both of these layers are very similar in morphology to those observed in the as-fabricated fuel plate L1F110 (Fig. 2). This indicates that these layers, which were present in the fuel plates after fabrication, did not change much in thickness or morphology during irradiation.

**Fig. 3.** Optical micrographs (a,b) showing thick and thin interaction layers observed at the foil/cladding interface of fuel plate L1F100.
3.2.2 Scanning Electron Microscopy

Fig. 4 shows SEM images of the fuel plate samples that were contained in the punchings produced at the low and high-flux sides of fuel plate L1F100. They indicate that only very small pieces of the fuel plate were present in the punchings and that the overall punching process, which has proven effective for generating samples from irradiated RERTR-6 dispersion fuel plates, is not ideal for sampling irradiated monolithic fuel plates. Apparently, the U-10Mo foil, which is brittle after irradiation, breaks into many pieces as the punch penetrates through the fuel plate and only a few small pieces of U-10Mo remain in the sample. Alternative techniques (e.g., core drilling) are being investigated for producing samples from irradiated monolithic fuel plates in HFEF. It was only in the high-flux sample (Fig. 4a) that a piece of the fuel plate could be found where the U-10Mo fuel/AA6061 cladding interface was still intact, and in the low-flux sample (Fig. 4b) only a piece of the U-10Mo fuel could be identified. Figure 5 shows SEM images of the interaction zone at different locations along the U-10Mo/AA6061 cladding interface in the sample taken at the high-flux side of L1F100.

![SEM images of the samples from the (a) high-flux and (b) low-flux sides of the fuel plate.](image1)

Fig. 4. SEM images of the samples from the (a) high-flux and (b) low-flux sides of the fuel plate.

![SEM images of a (a) thin and (b) thicker interaction layer in the high-flux sample.](image2)

Fig. 5. SEM images of a (a) thin and (b) thicker interaction layer in the high-flux sample.

Figure 6 shows X-ray maps for U, Mo, Al, Si, and Xe that were produced where a relatively thick interaction layer had developed at the U-10Mo/AA6061 cladding interface in the sample from the high-flux side of the fuel plate. It can be observed that two types of phases were present where one phase was Si-rich and the other Si-deficient. The fission gas Xe was observed in both the U-10Mo fuel and the AA6061 cladding, and the Si-rich precipitates that were present in the U-10Mo in the as-fabricated fuel (Fig. 2) are also present in the U-10Mo in the irradiated fuel plate. In Figure 7, a Si X-ray map is presented for a location where a relatively thin interaction layer was observed. Overall, the layer contained appreciable Si. Linescan analysis that was
performed in the U-10Mo fuel indicated that there was some variation in the Mo content, where the Mo varied between about 9 and 12 wt%.

In some areas of the samples, fracture surfaces were observed in the U-10Mo alloy that revealed the fission gas bubbles that were present in the microstructure (see Fig. 8). Generally, fission gas bubbles could not be seen in the intragranular regions, but they could be resolved on the grain boundaries. In some regions of the microstructure, isolated areas with relatively large fission gas bubbles could be observed. Due to their morphologies, they were called “honeycomb” structures. So far, this type of structure has not been observed when performing SEM characterization on irradiated RERTR-6 dispersion fuels that were exposed to similar irradiation conditions as L1F100. In RERTR-6 dispersion fuels, any fission gas agglomeration features were about 1 to 2 \( \mu \text{m} \) in size, not the up to 6 \( \mu \text{m} \) size as for fuel plate L1F100. The presence of the honeycomb structures in monolithic fuel plates and not in dispersion fuel plates appears to be linked to the different U-10Mo alloy microstructure that is present in a FB monolithic fuel plate (with the flattening and homogenization anneal steps) compared to what is observed in the U-7Mo alloy in rolled dispersion fuel plates.

Fig. 6. Backscattered electron image (a) and WDS X-ray maps for (b) U, (c) Mo, (d) Al, (e) Si, and (f) Xe taken at the foil/cladding interface of the L1F100 high-flux sample.

Fig. 7. Backscattered electron image (a) and Si WDS X-ray map at the foil/cladding interface.
4. Conclusions

Based on the SEM characterization of samples taken from the irradiated fuel plate L1F100, it has been determined that: (1) Utilization of a punching process for generating SEM characterization samples from irradiated monolithic fuel plates process is not an effective way to generate ideal samples. Another technique will need to be developed for obtaining a sample comprised of a full transverse cross section of a fuel plate that can be used for SEM analysis. (2) Both the thin and thick interaction layers that developed at the U-10Mo/AA6061 cladding interface during the RERTR-6 monolithic fuel plate manufacturing process changed very little in composition or morphology during irradiation. (3) The fission gas bubbles that developed in the U-10Mo microstructure during irradiation of the FB monolithic fuel plate could not be resolved with SEM in the intragranular regions. However, fission gas bubbles could be resolved at the grain boundaries. In some isolated, non-grain boundary regions of the microstructure, relatively large fission gas bubbles could be observed. These gas bubbles formed features that resembled a honeycomb structure.

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References