Title

Effects of heat treatment on U–Mo fuel foils with a zirconium diffusion barrier

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Abstract

A monolith fuel design based on U–Mo alloy has been selected as the fuel type for conversion of the United States' high performance research reactors (HPRRs) from highly enriched uranium (HEU) to low-enriched uranium (LEU). In this fuel design, a thin layer of zirconium is used to eliminate the direct interaction between the U–Mo fuel meat and the aluminum-alloy cladding during irradiation. The co-rolling process used to bond the Zr barrier layer to the U–Mo foil during fabrication alters the microstructure of both the U–10Mo fuel meat and the U–Mo/Zr interface. This work studied the effects of post-rolling annealing treatment on the microstructure of the co-rolled U–Mo fuel meat and the U–Mo/Zr interaction layer. Microscopic characterization shows that the grain size of U–Mo fuel meat increases with the annealing temperature, as expected. The grain sizes were ~9, ~13, and ~20 μm for annealing temperature of 650, 750, and 850°C, respectively. No abnormal grain growth was observed. The U–Mo/Zr interaction-layer thickness increased with the annealing temperature with an Arrhenius constant for growth of 184kJ/mole, consistent with a previous diffusion-couple study. The interaction layer thickness was $3.2 \pm 0.5 \mu\text{m}$, $11.1 \pm 2.1 \mu\text{m}$, $27.1 \pm 0.9 \mu\text{m}$ for annealing temperature of 650, 750, to 850°C, respectively. The homogeneity of Mo improves with post rolling annealing temperature and with U–Mo coupon homogenization. The phases in the Zr/U–Mo interaction layer produced by co-rolling, however, differ from those reported in the previous diffusion couple studies.

Keywords

monolithic, annealing, nuclear fuel, U–Mo, diffusion barrier

1 Introduction

U–Mo-based fuel types are considered very-high-density fuels for the conversion of high-performance research reactors from highly enriched uranium (HEU) to low-enriched uranium (LEU).¹ Literature on U–Mo nuclear fuels is on dispersion fuel type. References [2–8] provide some examples of the performance of this fuel during irradiation testing. The need for higher uranium density than may be achieved in dispersion fuels, coupled with observation of unstable behavior at high fission densities, led to consideration of a so-called monolithic fuel design. This fuel design replaces the dispersion of U–Mo and aluminum in the fuel meat with a solid U–Mo foil. Testing of advanced monolithic fuel type has been carried out under the United States High Performance Research Reactor (USHPRR) program.^{9–15} To date, almost 100 monolithic fuel plates, ranging from 4–40 inches in length, have been irradiated. Fuel-performance data obtained indicates that the current U–Mo monolithic fuel design exhibits good irradiation performance during high-power operation to local fission densities that exceed those required for operation of U.S. high-performance reactors.¹⁵ **Current fuel design utilizes a thin zirconium diffusion barrier to eliminate/minimize the direct interaction between U–Mo and aluminum-based cladding.**¹⁰ **Zirconium was selected over other candidate materials, such as Nb and Mo, after successful application of Zr to U–Mo fuel meat using a hot co-rolling process. Currently, the co-rolling process to bond U–Mo and Zr is the standard method for U–Mo fuel foil fabrication.** Heat treatment of U–Mo foils was considered as a further improvement to the fuel design. The potential beneficial effects of heat treatment include homogenization of the U–Mo fuel foil to eliminate molybdenum segregation (chemical banding), grain growth, uniform bonding between the U–Mo fuel meat and the zirconium barrier layer, and flattening of the foils prior to application of the aluminum cladding layer. The focus of this study is on the effect of annealing treatments on the homogeneity of Mo in U–Mo fuel meat and its impacts on the U–Mo/Zr interaction layer. Results are also compared with the existing data in the literature.^{16–21}

2 Experimental Procedure

2.1 Foil fabrication

Depleted uranium (>99%) and molybdenum (Alfa-Aesar, 99.95%) were arc-melted and drop-cast into a graphite mold to yield a **U–10 weight% Mo** coupon approximately $38 \times 25 \times 3$ mm. Selected coupons were homogenized at 1000°C for 2 hours **in an argon atmosphere followed by a water quench** prior to co-rolling. The coupons were placed between 0.25 mm (0.010-in.)-thick zirconium foil in evacuated and seal-welded low-carbon-steel cans. The rolling assembly was heated to 650°C in a box furnace and hot-rolled to 74% reduction in thickness. After hot rolling, foils were annealed at 650°C for 45 minutes before removal from the can. Foils were cold-rolled to the final thickness, another 50% reduction from the hot-rolled condition. The final total thickness of the fuel foils is approximately 300 μm (including a 25- μm -thick Zr layer on each side of the U–Mo fuel foil). Post-cold-rolling annealing treatments were performed in an argon-gas, purged tube furnace for one hour at 650, 750, or 850°C. **Carbon and**

oxygen contaminations in the fuel foils are possible, but were not determined in this study.

2.2 Microstructure Characterization

Microscopy samples were cut from the fuel foil using a Buehler high-speed diamond saw. Samples were mounted in an epoxy and ground using increasingly fine silicon-carbide paper to 1200-grit. After grinding, the samples were polished with 3 and 1- μm diamond suspensions. Final polishing was conducted using a vibratory polisher with 0.05 μm colloidal silica. Polarized light was used to reveal the grain structure of the Zr diffusion barrier layer and U–Mo fuel meat. The average grain size was determined by individually measuring 25 grains for which boundaries were obvious. Specimens were sputter-coated with a thin layer of gold. Scanning electron microscopy (SEM) was performed using a JEOL 7000F field emission scanning electron microscope equipped with Oxford energy-dispersive X-ray spectroscopy (EDS) and wavelength-dispersive X-ray spectroscopy (WDS) systems. Both secondary-electron (SE) and backscattered-electron (BSE) images were taken using a 20 kV accelerating voltage and a 10 mm working distance. The precision of U–Mo/Zr interaction layer thickness measurement was 0.1 μm . Chemical-composition information was obtained using Oxford INCA EDS semi-quantitative point analysis and EDS/WDS mapping functions.

3 Results

It should be noted that all results presented in this section come from fuel foils utilizing U–Mo coupons without any high-temperature homogenization treatment.

3.1 Grain Size of Fuel Foils Subjected to Annealing

Both temperature and Mo content have impact on the stability of γ -phase in the U–Mo system. Equilibrium phases for U–10Mo below 565°C are α -uranium and γ' (U₂Mo).^{22–24} Because the γ -phase in the U–10Mo is metastable for long times,²⁵ the post-cold-rolling annealing treatment in the γ -phase field, followed by air cooling, is sufficient to retain the high-temperature γ -phase uranium at room temperature.

Figure 1 shows a polarized light optical micrograph of the grain structure of a U–Mo fuel foil that was hot-rolled and annealed at 650°C for 45 minutes with no cold-rolling. Equiaxed grains can be seen near the U–10Mo/Zr interface. After cold-rolling, as shown in Figure 2, a deformed grain structure was observed with a grain size $\sim 15 \mu\text{m}$. The U–Mo returns to a fully equiaxed grain structure after post-cold-rolling annealing at temperatures evaluated in this study. Figures 3–5 provide polarized light optical images showing the grain structure of the cold-rolled U–Mo foils after annealing for 1 hour at 650, 750, and 850°C, with grain sizes of ~ 9 , ~ 13 and $\sim 20 \mu\text{m}$, respectively. As expected, the grain size increased with temperature increases, and no abnormal grain growth was observed.

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.

3.2 Mo Distribution in U–Mo Fuel Meat

Molybdenum segregation is commonly observed in as-cast U–Mo coupons, as shown in Figure 6. The Mo segregation remained in the fuel foils after the rolling process, as evidenced by the existence of chemical banding in the U–Mo fuel meat shown in Figure 7. Mo concentration as high as 12 weight % was measured in the darker-colored bands. The light-colored regions exhibit Mo concentration as low as 7 weight %. The Mo homogeneity improved with increasing post-rolling annealing temperature. Figure 8 shows EDS plots of Mo concentration in the U–Mo fuel meat subjected to a post-rolling annealing treatment for 1 hour at 650, 750, or 850°C. The range of Mo concentration in the U–Mo fuel meat narrowed from 7–12 wt% in the fuel foil annealed at 650°C to 9–11wt% in the fuel foil annealed at 850°C.

Figure 6.

Figure 7.

Figure 8.

3.3 U–Mo/Zr Interaction Layer Growth

The interaction layer between U–Mo fuel meat and Zr diffusion barrier was characterized by scanning electron microscopy. The as-cold-rolled fuel foil did not exhibit 100% coverage of U–Mo/Zr interaction layer. Figure 9 shows an SEM micrograph of a visible interaction layer (arrowed) between U–Mo and Zr in a fuel foil subjected to a post-cold-rolling annealing treatment at 650°C for 1 hour. The interaction layer is relatively uniform in thickness. This interaction layer contains several sub-layers as shown in a higher-magnification SEM micrograph (Figure 10). The fine microstructure is typical of that observed in other monolithic fuel plates.^{10–13} Based on previous studies, the dark-gray layer closest to Zr is composed of UZr_2 . Moving from the UZr_2 toward the U–Mo phase are dark precipitates; these become a sub-layer in some locations. Formation of the MoZr_2 precipitates results in the formation of molybdenum-depleted region, usually containing 5 weight % or less Mo. The total thickness of the interaction

layer is measured to be $3.2 \pm 0.5 \mu\text{m}$ (average of 10 locations). Increasing the post-cold-rolling annealing temperature increased the thickness of the U–Mo/Zr interaction layer. SEM micrographs in Figures 11 and 12 show the interfacial area of fuel foils subjected to annealing treatments at 750 and 850°C. After annealing at 850°C, it can be seen that the UZr_2 -bearing sub-layer became discontinuous toward the pure Zr region. Figure 13 is a high-magnification SEM micrograph from the Zr/U–Mo interfacial area of the fuel foil presented in Figure 12. Needle-shaped Zr-rich phases in the low Mo sub-layer can be seen. Identification of these phases was indeterminate by SEM analysis **due to their small size**. The total interaction layer was measured to be $27.1 \pm 0.9 \mu\text{m}$ (average of 10 locations) for the fuel foil annealed at 850°C.

Figure 9.

Figure 10.

Figure 11.

Figure 12.

Figure 13.

4 Discussion

4.1 Zr/U–Mo Inter-diffusion Behavior in Post-rolling Annealed Fuel Foils

The Zr/U–Mo interaction-layer thickness as a function of temperature is shown in Table 1. The growth rate for a specific temperature can be calculated using the relationship $K = (\Delta L)^2 / 2t$, where ΔL is the change in thickness, t is time (in seconds) and K is the rate constant (in m^2/sec). Assuming the initial interaction-layer thickness before the post-rolling-annealing treatment is negligible, the calculated growth rate of the interaction layer, K , is given in Table 1. The activation energy for growth of the interdiffusion can be determined using equation

$$K = K_0 \exp(-Q/RT) \quad (1)$$

where K_0 is a constant, R is gas constant and T is annealing temperature (in Kelvin). Arrhenius-type behavior was confirmed with an activation energy for growth of 184kJ/mol, and K_0 is calculated to be $3.76 \times 10^{-5} \text{ m}^2/\text{sec}$. A previous work using U–Mo/Zr diffusion couples prepared in the absence of thermo-mechanical processing provided a similar activation energy for growth of the interaction layer (183.8 kJ/mol) **and slightly higher growth rate at 850°C (by interpolating $K = 1.02 \times 10^{-13} \text{ m}^2/\text{sec}$ at**

800°C and $5.76 \times 10^{-13} \text{ m}^2/\text{sec}$ at 900°C); however, the phases observed in the interaction layer differed in that a β -zirconium layer was identified, instead of the combination of a UZr_2 layer and a Mo-depleted zone.¹⁹ A more recent diffusion-couple study by Park et al. measured a slightly lower growth rate, K , at 650°C ($7.75 \times 10^{-16} \text{ m}^2/\text{sec}$), and an activation energy of 219.4KJ/mol (with an assumption of an incubation time of 159 hours at 650°C).²¹ Again, the UZr_2 phase was not observed in that study. The reason for the difference is not clear; impurities, such as oxygen and iron on the Zr/U–Mo interface, and the cooling rate after annealing could affect the formation of phases at the interface.

Table 1.

4.2 Effect of 1000°C/2-hour High-temperature Homogenization Treatment of As-cast Coupon

Some U–Mo coupons were subjected to homogenization treatment at 1000°C for 2 hour in order to eliminate or minimize the segregation of Mo in the fuel foils. Identical rolling and annealing procedures were carried out on the fuel foils utilizing U–Mo coupons subjected to a homogenization treatment at 1000°C for 2 hours. Based on the results of the EDS chemical analysis, as shown in Figure 14, this homogenization treatment resulted in variations of Mo content less than 1 weight %. Another Mo profile from the same sample in Figure 8(a), but sampled from a different area, is also included in Figure 14 to highlight the effect of coupon annealing. Table 2 is a summary of the measured Zr/U–Mo interaction-layer thickness of the fuel foils utilizing coupons subjected to a homogenization treatment at 1000°C for 2 hours prior to hot- and cold-rolling. The difference between the interaction-layer thickness in these homogenized foils and that in fuel foils utilizing coupons without the coupon-homogenization treatment (i.e., an as-cast coupon) are shown in Figure 15. The fuel foils with the homogenization treatment at 1000°C for 2 hours show a slightly thinner Zr/U–Mo interaction-layer thickness. The activation energy of interaction-layer-thickness growth for the fuel foils with the homogenization treatment at 1000°C for 2 hours, 212.4 kJ/mol, is close to that of fuel foils without such treatment.

Figure 14.

Table 2

Figure 15.

5 Conclusions

Mo segregation was observed in the as-cast coupons and the resulting U–Mo fuel foils. Chemical banding was observed in the fuel foils subjected to a post-rolling-anneal treatment between 650 and 850°C. Homogeneity of Mo in U–Mo fuel meat improves

with increasing post-rolling-annealing temperature. Grain size in the U–10Mo fuel meat increases with the temperature of post-rolling-annealing treatment. The grain sizes were ~9, ~13, and ~20 μm for the annealing temperature of 650, 750, to 850°C, respectively. No abnormal grain growth was observed.

A pre-rolling U–Mo coupon homogenization treatment at 1000°C for 2 hours was performed on some as-cast coupons to minimize the Mo segregation in the resulting U–Mo fuel foils. This heat treatment effectively reduced the Mo variation in U–Mo fuel foils to less than 1 weight %. Homogenization treatment of starting U–Mo coupons slightly decreased the U–Mo/Zr interaction-layer thickness in the fuel foils.

The thickness of Zr/U–Mo interaction layer increases with temperature during post-cold-rolling annealing according to the classical Arrhenius relationship. The activation energy for growth was calculated to be 184KJ/mole, consistent with a previous diffusion-couple study and is close to another one. Also in this study, the activation energy of interaction-layer thickness growth—for fuel foils with the homogenization treatment at 1000°C for 2 hours, 212.4kJ/mol—is close to that of fuel foils without such treatment.

The existence of low-Mo and UZr₂-bearing sub-layers on the Zr/U–Mo interface differs from the phase array reported in previous studies in which U–Mo/Zr diffusion couples were used in the absence of thermo-mechanical processing. The reason for the difference is not clear; impurities, such as oxygen and iron on the Zr/U–Mo interface, and the cooling rate after annealing could affect the formation of phases at the interface.

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Figure Captions

Figure 1. Polarized-light optical micrograph of a fuel foil subjected to a 74% hot-rolled reduction at 650°C, followed by a 650°C annealing treatment for 45 minutes.

Figure 2. Polarized-light optical micrograph of a U-10Mo foil after hot-rolling to 74% reduction at 650°C, annealing for 45 minutes at 650°C, and a further 50% cold-rolling reduction.

Figure 3. Polarized-light optical micrograph of a fuel foil subjected to a post-cold-rolling annealing treatment of 650°C for 1 hour.

Figure 4. Polarized-light optical micrograph of a fuel foil subjected to a post-cold-rolling annealing treatment of 750°C for 1 hour.

Figure 5. Polarized-light optical micrograph from a fuel foil subjected to a post-cold-rolling annealing treatment of 850°C for 1 hour.

Figure 6. SEM micrograph and EDS elemental mapping of molybdenum, showing the segregation of Mo in an as-cast coupon. Lighter regions in the EDS Mo element mapping have higher Mo concentration (up to 12 weight %). The black features are carbides/oxides.¹²

Figure 7. SEM micrograph showing the chemical banding in the U–Mo fuel foil subjected to a post-cold-rolling annealing treatment at 650°C/1hr. Wavy dark features are chemical bands with higher Mo concentration. Black precipitates are carbide/oxides.

Figure 8. Mo concentration profiles in U–Mo fuel foils subjected to post-cold-rolling annealing treatments for 1 hour at (a) 650°C, (b) 750°C and (c) 850°C. **The Mo profiles were taken starting from the edge of the U-Mo fuel meat to the center of the U-Mo fuel meat.**

Figure 9. An SEM micrograph of the U–Mo/Zr interface in a fuel foil subjected to a post-cold-rolling annealing treatment at 650°C for 1 hour.

Figure 10. A higher-magnification SEM micrograph of the region in Figure 9 shows the U–Mo/Zr interfacial area from a fuel foil subjected to a post-cold-rolling annealing treatment at 650°C for 1 hour.

Figure 11. An SEM micrograph from the U–Mo/Zr interface of a fuel foil subjected to a post-cold-rolling annealing treatment of 750°C for 1 hour.

Figure 12. An SEM micrograph from a fuel foil subjected to a post cold-rolling annealing treatment of 850°C for 1 hour.

Figure 13. A higher-magnification micrograph of the region shown in Figure 12 shows the U–Mo/Zr interfacial area from a fuel foil subjected to a post-rolling annealing treatment at 850°C for 1 hour. **The left hand side of this image is toward the pure Zr and the left hand side is U-Mo.**

Figure 14. Mo and U concentration profile in U–Mo fuel foils after post-cold-rolling annealing treatment for 1 hour at 650°C utilizing U–Mo coupons which are (a) as-cast, (b) homogenized at 1000°C/2hrs. Note that no significant Mo segregation (less than 1 weight %) was observed in the fuel foil fabricated using homogenized coupon.

Figure 15. A plot shows Zr/U–Mo Interaction layer thickness verses post-rolling annealing treatment temperature, with (red) and without (blue) homogenization treatment on coupon.

Figures

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.

Mo La1
Figure 6.

Figure 7.

Figure 8.

Figure 9.

Figure 10.

Figure 11.

Figure 12.

Figure 13.

Figure 14.

Figure 15.

Tables

Table 1. Zr/U–Mo interaction layer thickness and growth rate of fuel foils subjected to a post rolling annealing treatment at different temperature.

	650°C	750°C	850°C
IL thickness (μm)	3.2 ± 0.5	11.1 ± 2.1	27.1 ± 0.9
K (m ² /sec)	1.42 × 10 ⁻¹⁵	1.71 × 10 ⁻¹⁴	1.02 × 10 ⁻¹³

Table 2. Zr/U–Mo interaction layer thickness of fuel foils using 1000°C/2hr homogenized coupon subjected to post-rolling annealed at different temperature.

	650°C	750°C	850°C
IL thickness (μm)	2.0 ± 0.5	6.2 ± 0.4	22.7 ± 1