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U-10Mo Mechanical Property Study Results

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ABSTRACT

A uranium-10 wt% molybdenum billet was cast using a vacuum induction melting furnace. The uranium was retrieved from Zero Power Physics Reactor stockpiles and had previously been covered with a polymeric coating to prevent oxidation. The coating was removed by mechanically eliminating the exterior surfaces. After casting total carbon analysis, tensile testing and microstructural testing were performed. Based on the characterization results, the Zero Power Physics Reactor stockpile's depleted uranium can be used as a source of depleted uranium if all coating materials are fully removed.

CONTENTS

FIGURES

TABLES

U-10Mo Mechanical Property Study Results

1. INTRODUCTION AND PURPOSE

The Fast Burst Reactor design calls for a low-enriched uranium-10 wt% molybdenum (U-10Mo) fuel. Because this type of fuel has not recently been fabricated in monolithic form at an engineering scale, several issues need to be studied further; these issues include feedstock availability, preparation, and how they affect chemical and mechanical properties. Currently, limited amounts of depleted uranium feedstock of known pedigree with low impurity levels are available in the U.S. Department of Energy complex. A possible source of feedstock may be depleted uranium plates and blocks previously used in the now decommissioned Zero Power Physics Reactor (ZPPR). This feedstock has been exposed to an air environment during storage, but nearly all of this uranium was coated with a polymeric coating with the trade name of Kel-F in order to reduce oxidation during storage and use. This coating has proven tenacious and difficult to remove in the past and may act as a carbon source for the alloy unless fully removed. However, if the coating can be efficiently removed, this may open up several tons of possible depleted uranium feedstock.

In order to quantify if this feedstock could be used with the Kel-F coating removed, a U-10Mo billet was cast using the ZPPR uranium feedstock and characterized. Characterization included carbon analysis of the cast alloy, microstructure of the as-cast product, and, finally, ultimate tensile and yield strength. These data can be compared to literature data and previously used U-10Mo material to determine if the ZPPR blocks can be used as feedstock.

2. EXPERIMENT

2.1 Initial Billet Casting

The initial billet was cast using a vacuum induction melting system. The charge is loaded into a Y_2O_3-coated graphite crucible, which is positioned above a Y_2O_3-coated graphite mold. The crucible is designed with a pour spout in the center, which is plugged with a stopper rod until the rod is withdrawn at the time of casting. A schematic model of the billet casting system and a photograph of the actual system are shown in Figure 1. The crucible was loaded with depleted uranium and molybdenum foil. The uranium feedstock started out as a Kel-F-coated 2 x 2 x 5-in. block. The block had a 1.375-in. core cut from the center portion for another project, but the remaining scrap was available for this project's use. The Kel-F coating was fully removed from all external surfaces by electro-discharge machining away the outer surface. After all outer surfaces were removed, the block was cut up into a number of smaller pieces for ease of handling; this was also done via electro-discharge machining. The molybdenum foil (0.25-mm thick, 99.5%) was commercially procured from Alfa Aesar. Appendix A shows the certificate of analysis for the molybdenum feedstock. The molybdenum foil was also cut into smaller random shapes for ease of handling. Most of the uranium was loaded first, followed by the molybdenum, and, finally, followed by the remaining uranium. The loaded crucible is shown in Figure 2.

After loading the crucible, the furnace chamber was closed and evacuated until a vacuum level of at least 9 x 10^{-5} torr was reached. Once the desired vacuum level was achieved, the furnace was heated to 500°C at a ramp rate of 50°C/minute and allowed to soak for 5 minutes to ensure all coatings were fully dried. Next, the temperature was increased to 1550°C at a rate of 50°C/minute and immediately allowed to cool to 1500°C. After approximately 43 minutes at 1500°C, the stopper rod was lifted and the material flowed into the Y_2O_3-coated graphite mold. Figure 3 shows the temperature profile of the crucible and mold. Thermocouples are located on the outside of the crucible, approximately at the mold axial middle and approximately 1 in. above the middle.

Figure 1. Billet casting system internal cut-away schematic showing basic internal components (left). Billet casting system in the radiological fume hood (right).

Figure 2. Loaded, Y_2O_3-coated graphite crucible loaded into the furnace (left) after molybdenum loading and (right) after final depleted uranium loading.

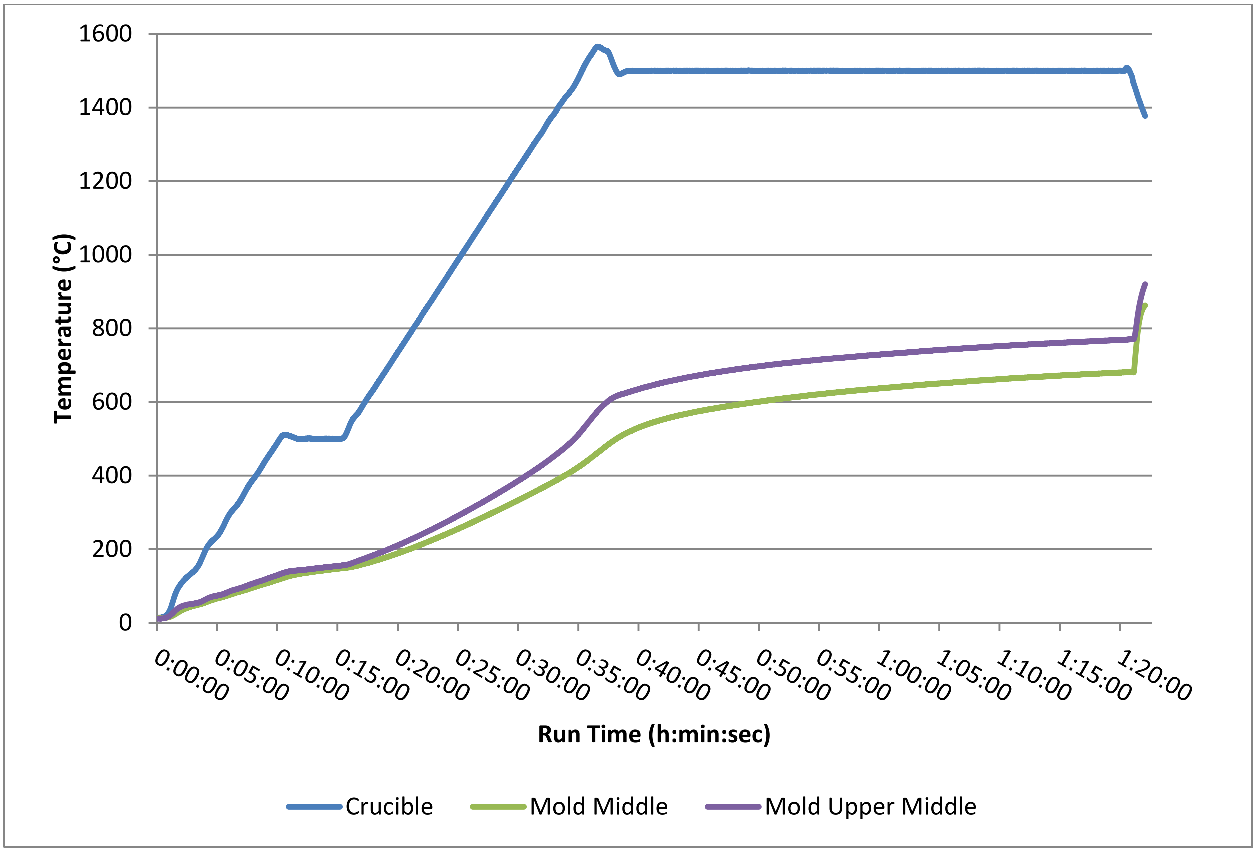

Figure 3. Thermal profile of the casting run.

2.2 Characterization

The as-cast billet was characterized by a variety of methods. First, the density of the billet was determined using the immersion density method. Three measurements were made using a copper standard of a similar volume. Following standard verification, three dry and three wet masses were taken of the as-cast billet and the density was calculated using a water density of 0.998722 g/cm^3. After density was determined, a total of six tensile samples were machined from the center portion of the billet. Tensile samples were sized per American Society of Testing and Materials (ASTM) E8. Samples were also taken from the top and bottom portion of the billet for carbon and microstructure analysis. All samples were removed by electro-discharge machining. The microstructural samples were prepared using standard metallographic techniques and examined using a Phenom XL tabletop scanning electron microscope. Carbon analysis was performed via IR measurements using an ELTRA CS-800. Tensile testing was performed per ASTM E8.

3. RESULTS

The solid U-10Mo billet was cast with charge and final masses as described in Table 1. A significant amount of dross was also present, but it stayed in the crucible (Figure 4). Upon visual examination of the color and form, the dross phase appeared to be made up of oxides from both the uranium and molybdenum feedstock. It should be noted that much of the dross layer was composed of pieces in similar shape, but in thinner pieces, to the molybdenum feedstock. Table 2 shows the dimensions of the billet. Length was measured from the bottom of the taper (ignores the sample tip) to the top of the billet assuming a flat top. Diameter was measured along the length of the billet near the top, middle, and bottom. Two measurements were taken at each location approximately 90 degrees apart. The immersion density measurements are described in Table 3. The average density value was 17.21 g/cm^3, which corresponds to a 98% theoretical density assuming a density based on the rule of mixtures.

Table 1. Summary of the casting masses.

Item	Mass (g)
Uranium Charge	2,404.63
Molybdenum Charge	267.79
As-Cast Billet	2,581.50
Casting Dross	90.924

Table 2. Summary of the as-cast billet dimensions.

Location	Measurement (in.)	
Length	5.742	
Top	0 to 1.454	90 to 1.456
Middle	0 to 1.465	90 to 1.461
Bottom	0 to 1.460	90 to 1.461

Table 3. Immersion density results.

	Dry (g)	Wet (g)	Density (g/cm^3)
Measurement-1	2,581.65	2,431.79	17.205
Meaurement-2	2,581.55	2,431.84	17.222
Measurement-3	2,581.59	2,431.82	17.218
Average	2,,581.60	2,431.83	17.215

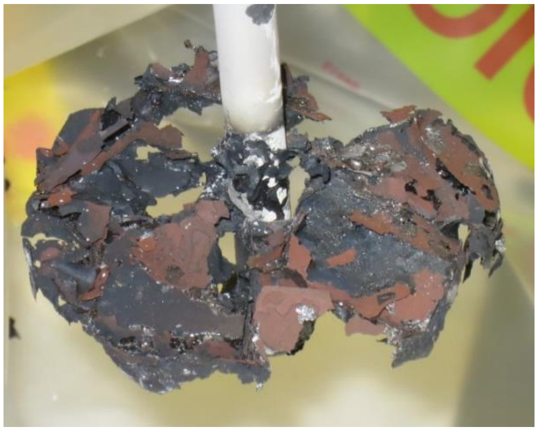

Figure 4. Stopper rod with residual dross adhering to it.

A total of six tensile samples were made and found to be acceptable per ASTM E8. Figure 5 shows a basic sketch detailing where dimensional measurements were taken. Samples thickness was measured at T1 and T2, sample widths were measured at W1, W2, and Wc, and gage length measured at GL. The samples were tested using a standard Instron load frame at room temperature. Samples were not dimensionally characterized after testing; therefore, uniform elongation, elongation, and reduction of area cannot be reported per ASTM E8. An extensometer was used for testing so strain data are reported. A typical stress strain curve is shown in Figure 6 to provide a general idea of sample behavior. Table 4 shows the pre-tested dimensions, 0.2% offset yield, ultimate tensile strength, and slope. The reported slope is the initial slope in the elastic region, which is similar to the modulus. It should be noted that this test did not meet all standard requirements for modulus testing; slope information is provided as general information for material behavior but should be used only as an approximation. Note that Sample 1279-B-DB-1 (i.e., Sample ID 1279-bottom portion-dog bone-1) slipped in the grips during testing and no strength or slope are reported. Figure 7 shows the samples after testing.

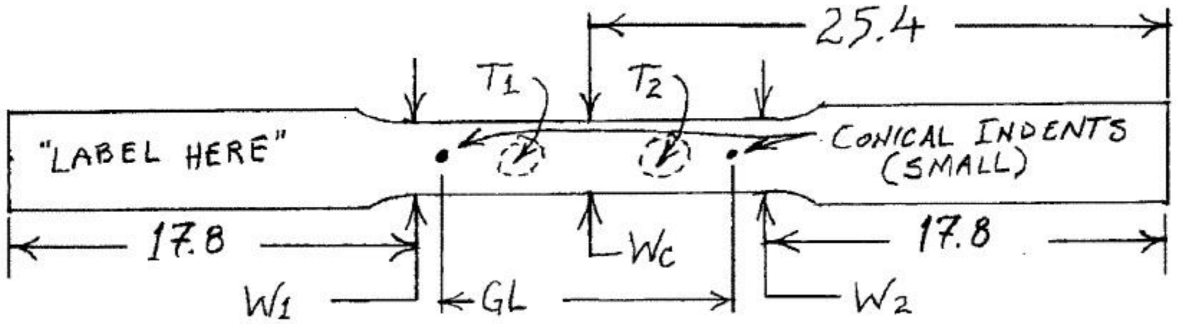

Figure 5. Sketch of the tensile samples showing locations of dimensional measurements.

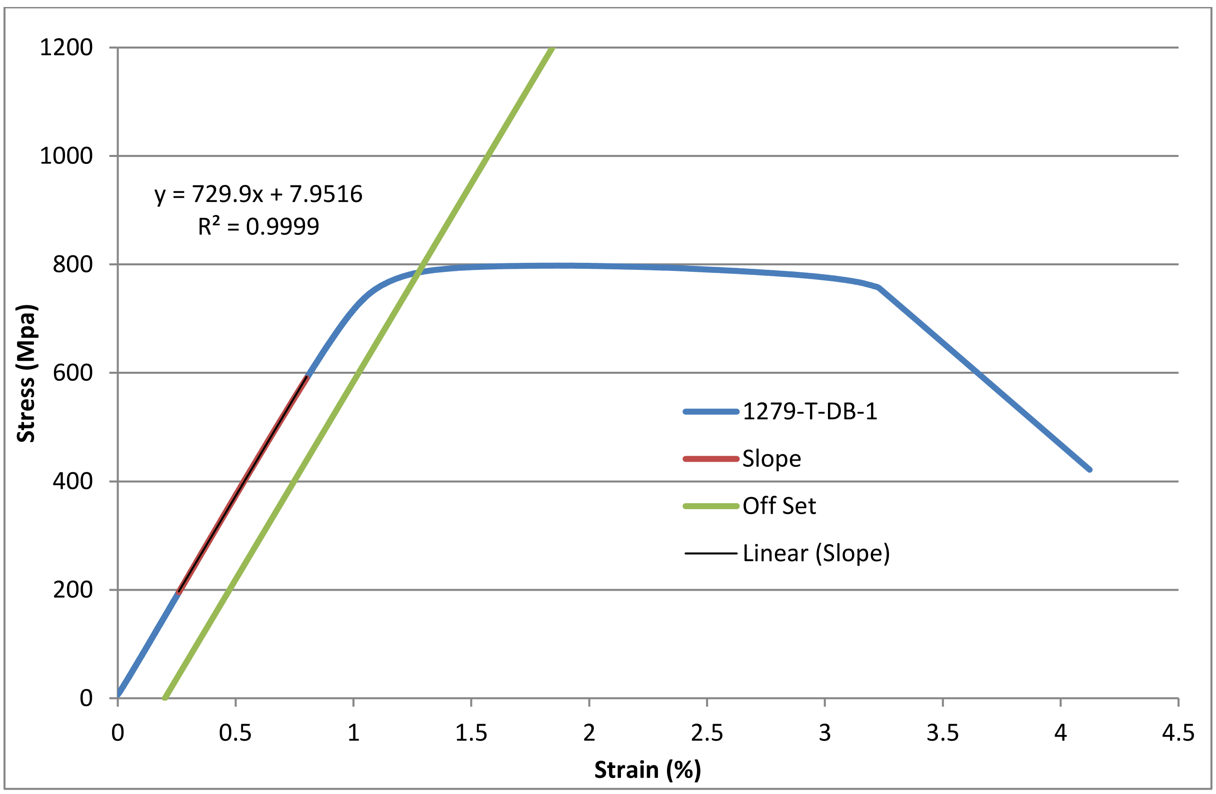

Figure 6. Typical stress strain curve for a top section sample.

Table 4. Mechanical test sample dimensions and resulting tensile test data.

	W1 (mm)		**W2 (mm)**		**Wc (mm)**		**GL (mm)**			**T1 (mm)**		**T2(mm)**		**Slope (MPa/%)**	**0.2% Yield (MPa)**	**UTS (MPa)**
Specimen ID	**#1**	**#2**	**#1**	**#2**	**#1**	**#2**	**#1**	**#2**	**#3**	**#1**	**#2**	**#1**	**#2**			
1279-B-DB-1	2.682	2.680	2.675	2.675	2.657	2.658	12.703	12.678	12.693	0.960	0.961	0.964	0.964	---	---	---
1279-B-DB-2	2.676	2.676	2.666	2.666	2.650	2.652	12.750	12.738	12.747	0.952	0.953	0.952	0.952	**764**	**828**	**837**
1279-B-DB-3	2.918	2.918	2.912	2.912	2.902	2.902	12.768	12.765	12.784	0.960	0.960	0.960	0.961	**767**	**827**	**827**
1279-T-DB-1	2.753	2.753	2.763	2.762	2.730	2.732	12.795	12.777	12.785	0.950	0.950	0.951	0.951	**730**	**785**	**797**
1279-T-DB-2	2.772	2.771	2.759	2.759	2.753	2.752	12.864	12.851	12.841	0.951	0.951	0.948	0.948	**758**	**785**	**790**
1279-T-DB-3	2.746	2.745	2.740	2.741	2.737	2.738	12.658	12.660	12.662	0.954	0.954	0.951	0.951	**745**	**793**	**798**

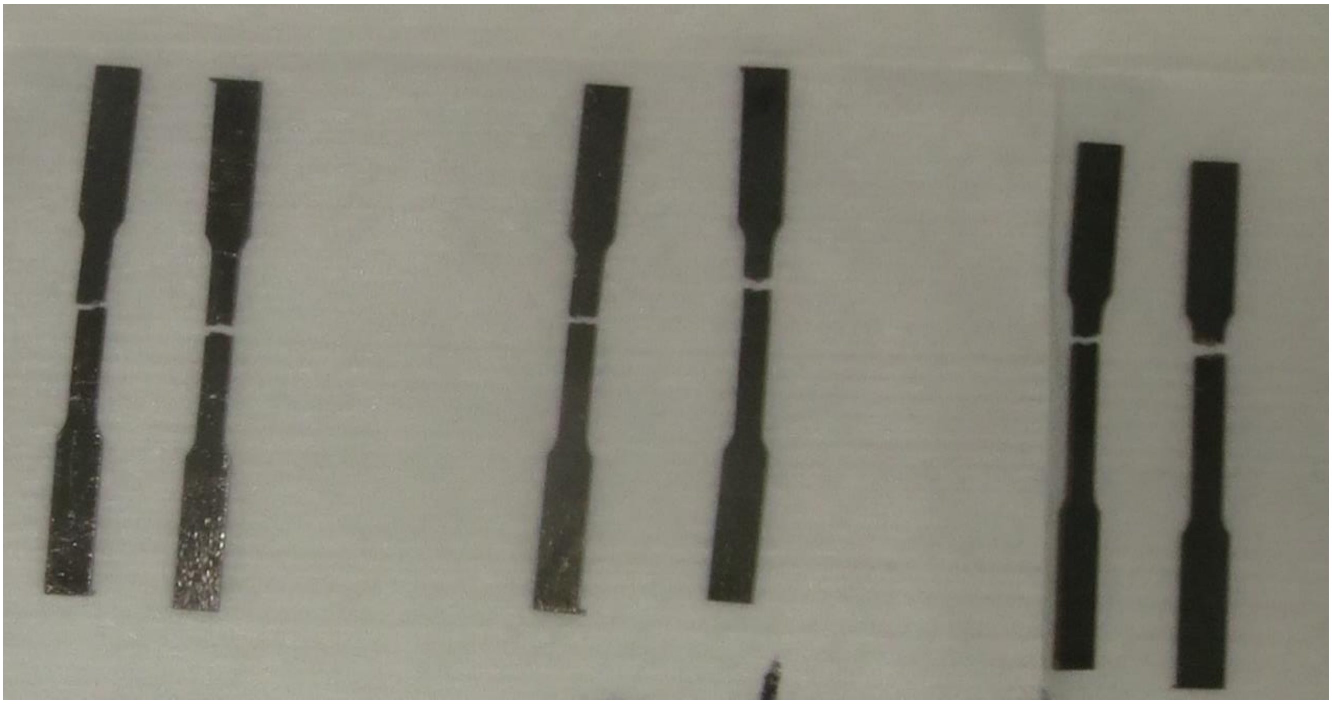

Figure 7. Tested tensile tests: (left to right) Sample 1: top, bottom; Sample 2: top, bottom: Sample 3: top, bottom.

Multiple samples were taken from the billet for carbon analysis. Table 5 shows the results. The measured carbon content was very consistent along the length, averaging 323 ±14 ppm.

Table 5. Summary of the carbon content in the depleted uranium feedstock and alloy.

Sample ID	Carbon Content (ppm)
Bottom-1	320
Bottom-2	328
Bottom-3	326
Middle-1	296
Middle-2	352
Middle-3	329
Top-1	316
Top-2	320
Top-3	322

Microstructure samples were taken from the top, middle, and bottom portions of the as cast U-10Mo billet. Samples were cut so the analyzed surface corresponded to the radial section of the billet and were polished using standard metallography techniques. The samples were first examined via backscattered electron imaging. The microstructure of each sample was consistent throughout the sample; however, the structure was progressively more refined further toward the bottom of the billet. Throughout the billet areas, slight molybdenum concentration variations were seen. This mottled appearance (left side of Figure 8) was qualitatively examined using energy dispersive x-ray spectroscopy. The darker areas were areas of slightly increased molybdenum content as opposed to surrounding lighter areas, which were slightly depleted in molybdenum. Also seen throughout the sample were precipitates on the order of 5 to 10 µm in diameter and length. Energy dispersive x-ray spectroscopy data showed the precipitates to be of higher uranium content. The right side of Figure 8 shows a higher magnification of the top portion of the billet, highlighting the precipitates and size and shape. The billet bottom portion microstructure is very similar and can be seen in Figures 10. In the left side of Figure 10, the higher degree of refinement can be seen with smaller and more numerous areas of decreased molybdenum content. Although the general microstructure appears more refined, the precipitates are present with similar size and distribution along the length of the billet. As might be expected, samples from the middle portion of the billet showed an intermediate microstructure between the top and bottom section, showing progression of the grain refinement.

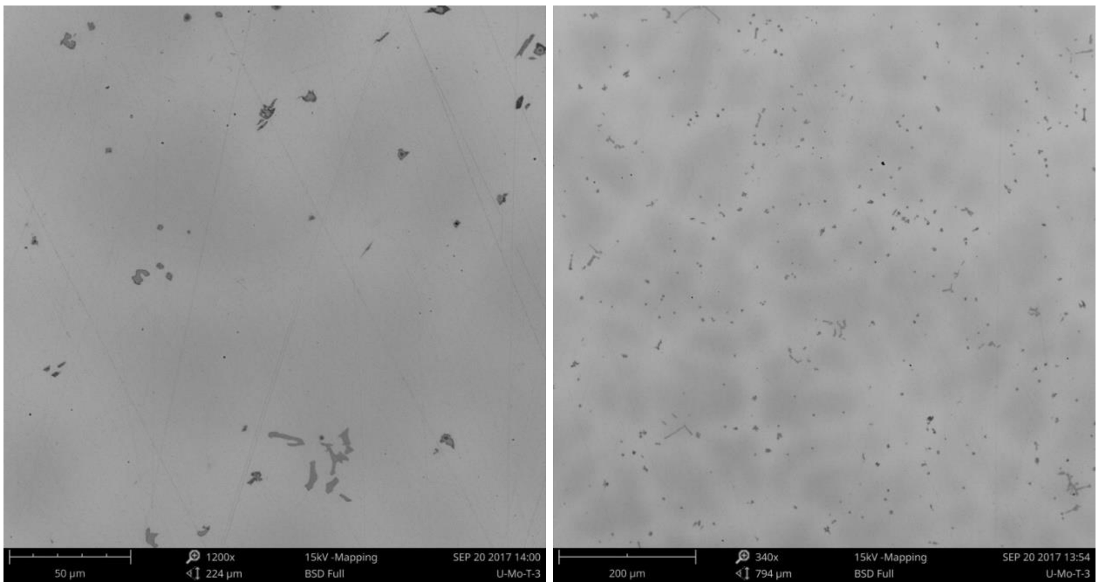

Figure 8. Micrograph of the upper portion of the as-cast billet showing general structure (left) and micrograph of upper portion of the as-cast billet highlighting the precipitates (right).

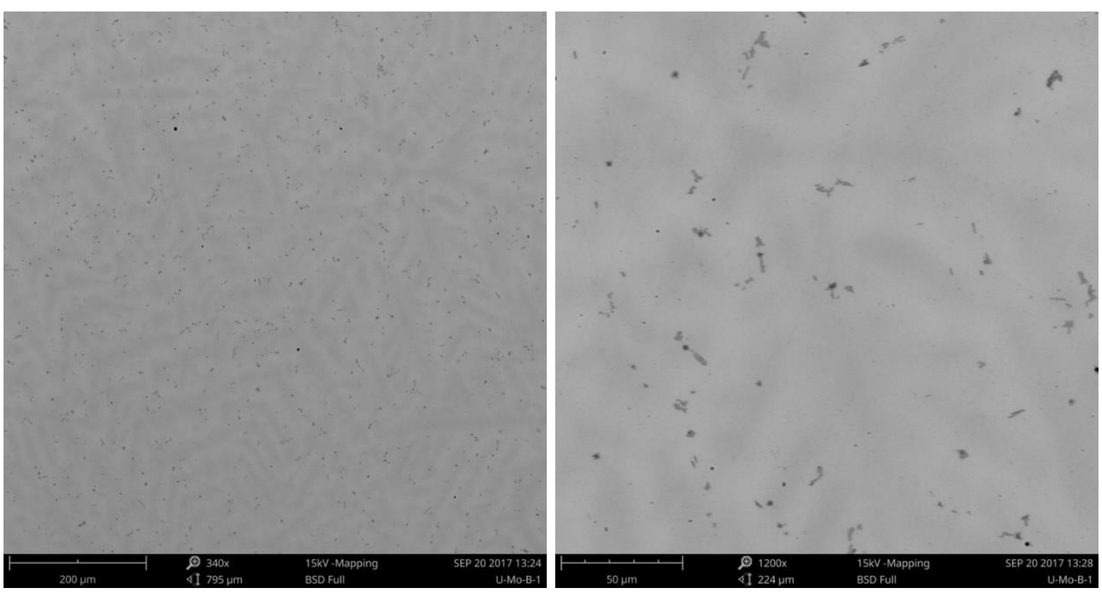

Figure 9. Micrograph of the lower portion of the as-cast billet showing the refined structure (left) and micrograph of the lower portion of the as-cast billet (right).

4. CONCLUSIONS/DISCUSSIONS

A U-10Mo billet was cast using depleted uranium feedstock from the ZPPR stockpile. The Kel-F coating was removed through mechanical means to ensure it did not add further carbon contamination to the final billet. Characterization of the resulting billet found that the carbon content of the billet was very consistent along the length, averaging 323 ±14 ppm.

Data from the microstructure analysis showed that the matrix material consisted of discrete areas of high and low molybdenum concentrations throughout the billet. The upper portion shows discreet areas of lower molybdenum concentration approximately 50 to 100 µm in width surrounded by a continuous higher molybdenum phase. While the bottom portion shows the discrete lower concentration areas to be approximately 10 µm in width and a more lenticular morphology surrounded by the continuous higher molybdenum phase. Uranium-rich precipitates are distributed throughout the sample. The size and distribution of the precipitates did not vary as much as the matrix microstructure based on axial location; however, the lower sample precipitates are slightly smaller and more numerous. It is also of note that the precipitates appear to more closely associated with the areas of higher molybdenum concentration.

Tensile samples were also taken from the as-cast billet. The samples were electro-discharge machined from the center, upper, and lower portions and tested per ASTM E8. The results were also quite consistent, although the upper portion had a consistently lower 0.2% offset yield strength and ultimate tensile when compared to the lower portion. The difference in yield and ultimate tensile strengths can be explained by the variation in microstructure. It is reasonable to assume that the increased strength of the lower billet samples was caused by the increased grain refinement of these samples. Increased grain refinement is driven by the furnace design used to cast the billets. As seen in Figure 1, the mold sits on a copper chill basin and the lower portion of the mold is not actively heated. This causes a thermal gradient in the mold, with the bottom portion being cooler (Figure 3). The bottom portion of the billet then cools faster, causing the greater amount of grain refinement, which in turn can affect the physical properties. However, the difference between the samples was not large.

Based on these results, using the ZPPR stockpile of depleted uranium is feasible and a mechanical cleaning method removes any residual Kel-F coating. Also, for this experiment, the outer surface of the feedstock was removed through electro-discharge machining; however, other methods of mechanical cleaning are likely as efficient for removal and may be more time and cost effective as well, depending on the available equipment. By effectively removing any coating material, several tons of pure depleted uranium feedstock can be made available; however, a pedigree has not been established for much of this material.

Appendix A

Alfa Aesar

Certificate of analysis

Product No.:	10044
Product:	Molybdenum foil, 0.25mm (0.01in) thick, annealed, 99.95% (metals basis)
Lot No.:	J17Y022

Mo: > 99.95

C	< 0.003	Ti	< 0.001	Cr	< 0.001
Pb	< 0.001	O	< 0.007	Fe	< 0.0014
Ca	< 0.001	Co	N/R	N	< 0.002
Mn	< 0.001	Na	N/R	Mg	< 0.001
H	N/R	Si	< 0.001	Al	< 0.001
K	N/R	Ta	N/R	Sn	< 0.001
Mo	> 99.95	W	N/R	Nb	N/R
Ni	< 0.001	Cu	< 0.001	O_2	N/R
N_2	N/R	H_2	N/R		

N/R = Not reported
Values given in percent unless otherwise stated

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