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Melt-Dilute Spent Nuclear Fuel Form – Fabrication and Metallurgical Characteristics

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TITLE: **Melt-Dilute Spent Nuclear Fuel Form – Fabrication and Metallurgical Characteristics**

TASK: **Plan for Qualification of the Melt-Dilute SNF Form for Repository Disposal, SRT-MTS-99-2031, Rev. 2**

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1.0 EXECUTIVE SUMMARY

The melt-dilute treatment technology developed at SRS consolidates fuel assemblies by a melting/casting process in which depleted uranium is added to reduce enrichment below 20% ^{235}U. Preliminary criticality analyses have shown that minor amounts of neutron-absorbing materials are needed to demonstrate criticality control (i.e., maintain $k_{eff} < 0.95$) for melt-dilute spent nuclear fuel (MD-SNF) form with 20% ^{235}U enrichment. Therefore, it is necessary to demonstrate process and performance compatibility of neutron absorbing materials in the MD-SNF form. This report addresses the process compatibility of neutron absorbing materials for the uranium-aluminum alloy fuels.

Studies on the process compatibility of neutron absorbing materials for the silicide and oxide fuels and the performance compatibility of the resulting SNF form is in progress under the melt-dilute qualification program. The SNF form compatibility studies will provide data on the effect of neutron absorbing material additions on MD-SNF corrosion and corrosion products, microstructure, and homogeneity to support performance assessment and criticality analyses under repository relevant environments. Results of this ongoing work will be included in the final report of the melt-dilute SNF form characteristics.

2.0 INTRODUCTION

Approximately 20 metric tons heavy metal of aluminum-based spent nuclear fuel (Al-SNF), or approximately 15,000 assemblies, are being consolidated at the Savannah River Site. A significant portion of the Al-SNF contain highly enriched uranium (HEU) (> 20% ^{235}U). The melt-dilute treatment technology is being developed at SRS for ultimate disposal of these fuels in the monitored geologic repository (MGR).[1] This alternative for disposition has been selected as the preferred alternative and a Record of Decision has been issued through the EIS process.[2] The proposed repository waste package will co-dispose a canister containing the melt-dilute SNF form with several borosilicate glass logs of defense high-level waste (DHLW).

One challenge in the disposal of SNF is to assure criticality control for disposition. Analyses of degraded configurations of the MD-SNF form at 20% ^{235}U enrichment[3-4] indicate that the reactivity of some configurations exceeds the limit of $k_{eff} < 0.95$.[5] Among the possible methods available to reduce the reactivity of the SNF forms is to control the amount of fissile material disposed of in a single waste package and to integrate neutron absorbers with the diluted SNF form obtained through the melt-dilute treatment technology. The degradation characteristics of the MD-SNF form, including corrosion rate and corrosion product formation are important to the qualification of the MD-SNF form for repository disposal. A compatibility-testing program is being implemented at SRS to fully characterize the degradation behavior of the MD-SNF form. The degradation response is predicated by the fabrication (Melt-Dilute treatment) history and associated as-fabricated metallurgical characteristics. This report presents the as-fabricated metallurgical characteristics of the MD-SNF form with integral neutron absorbing materials.

3.0 BACKGROUND

Major challenges associated with the direct disposal of research reactor fuel in a repository include nonproliferation and criticality control, both of which are a concern for HEU Al-SNF. Criticality control is more readily achievable and the issue of proliferation is eliminated by reducing the uranium enrichment to below 20%. The melt-dilute technology development program is focused on the development and implementation of a treatment technology for diluting HEU Al-SNF to LEU levels (< 20%) and qualifying this LEU Al-SNF form, melt-dilute DOE SNF, for geologic repository disposal.

3.1 Criticality Control of the Melt-Dilute SNF Form

Criticality control in waste packages for disposal at Yucca Mountain is governed by 10CFR60. It must be demonstrated through analysis that the probability of criticality for 10,000 years following disposal is less than 10^{-6}. Failure of waste package containment is generally assumed to occur beginning several thousand years after emplacement. Therefore, both intact and degraded states of the waste package contents must be evaluated.

Neutron absorbing materials are needed to maintain $k_{eff} < 0.95$ in desired packaging configurations of MD-SNF with 20% ^{235}U enrichment loaded in DOE-SNF canisters. Criticality of the MD-SNF loaded in a fully flooded, intact DOE-SNF canister was evaluated using the SCALE family of computer codes.[3] Results for the MD-SNF form at various stages of degradation, at several enrichments, and with various fill levels, indicate that criticality control for the 20%-enriched MD-SNF form can be maintained through implementing a minimum fill level for the DOE-SNF canister.[3]

Preliminary analyses of several scenarios for degraded DHLW and degraded MD-SNF form within the waste package have been conducted.[4] These preliminary criticality calculations indicate that the resulting configurations require the addition of neutron absorbing material to maintain criticality control. The results of these calculations indicate that a cylindrical configuration of the degraded MD-SNF form within the intact waste package provide a greater criticality potential than the configurations originally suggested by previous studies.[6] Based upon these preliminary criticality calculations, it is estimated that the addition of less than 1% by weight of gadolinium may be sufficient to satisfy the criticality requirements for the MGR. However, the final neutron absorber specifications for the MD-SNF form requires the completion of the final criticality analysis.

The final criticality analysis of the melt-dilute Al-SNF form will provide a defensible parametric evaluation of the melt-dilute Al-SNF form in various configurations and at various stages of degradation of the melt-dilute Al-SNF form, the DOE SNF canister, and the DHLW glass within the waste package. This analysis includes consideration of geochemical and degradation behavior and the neutron absorption characteristics of the waste package contents. This analysis is underway and will be completed in FY01. The completed analysis will demonstrate that neutron absorbers incorporated in a melt-dilute SNF form provides sufficient negative reactivity to preclude the possibility of achieving nuclear criticality within an emplaced waste package. The results of the criticality analysis will provide the necessary information required to define the final composition of the melt-dilute Al-SNF form.

3.2 Neutron Absorber Options and Considerations

Candidate melt-dilute/neutron absorber systems include melt-dilute plus gadolinium, melt-dilute plus hafnium, and melt-dilute plus gadolinium and hafnium. These systems have been selected based upon thermal neutron absorption cross-section and upon geochemical considerations. The specific compositions have not been established; however it is anticipated that less than 1% by weight of neutron-absorbing species in the melt-dilute form will be required to maintain reactivity limits.

Gadolinium has been selected due to its high neutron absorption cross-section. The solubility of gadolinium metal may become a concern over geologic times as it has the potential for being selectively removed from a waste package while leaving behind fissile uranium. However, gadolinium metal has the potential for forming a less soluble oxide, gadolinia (Gd_2O_3), or even an insoluble phosphate, $GdPO_4$, as the MD-SNF form degrades in expected repository environments. Gadolinia has been used in transport/shipment casks as a criticality control material. In addition, gadolinia provides improved solubility characteristics over gadolinium metal. The phosphate form of gadolinium, gadolinium-phosphate ($GdPO_4$), would provide significant neutron absorbing capacity while exhibiting desirable geochemical characteristics. Unfortunately, $GdPO_4$ is not commercially available at this time. Gadolinia and gadolinium-phosphate are not currently being pursued for melt-dilute criticality control since gadolinium readily alloys with aluminum and uranium and can be an integral absorber in the MD-SNF form.

Hafnium is attractive metallurgically. In its pure form, hafnium is extremely corrosion resistant and is relatively insoluble over a wide pH range. However, the thermal neutron absorption cross-section of hafnium is significantly lower than that of gadolinium. Hafnium has been selected due to its modest neutron absorption cross-section in combination with its relative insolubility. The hafnium loading required to preclude criticality over geologic times in the proposed repository is many times that of gadolinium.

The ideal melt-dilute/neutron absorber system is one that will utilize a combination of gadolinium, for its very large neutron cross-section and alloying characteristics, and hafnium, for its insolubility. This combination will provide suitable neutron absorption and solubility characteristics to allow for the demonstration of criticality control of the MD-SNF form over geologic times. The final neutron absorber mix requires degradation product species evaluation that is an integral part of the neutron absorber compatibility program at SRS.

3.3 Melt Dilute/Neutron Absorber System

Because the primary constituents of the aluminum-based SNF assemblies are uranium and aluminum, the MD-SNF form will be based on neutron absorber additions to the binary uranium-aluminum system (see Figure 1). The majority of Al-SNF assemblies are comprised of an enriched uranium-aluminum alloy in an aluminum matrix with aluminum cladding. This preliminary report represents the demonstration of a melt-dilute SNF form for these uranium-aluminum alloy based fuels. MD-SNF forms of the silicide and oxide based fuels are currently being fabricated and are also expected to be based on the binary uranium-aluminum system. Figure 1 illustrates the region of interest involved for the melt-dilute process with the use of the uranium-aluminum phase diagram. The MD-SNF form will be comprised, primarily of UAl_4 and Al, shown by the shaded region in Figure 1. In the binary system the solidification is shown to occur as a eutectic transformation (a liquid transforming on cooling to UAl_4 and Al phases. This transformation occurs at approximately 13.2 wt. % U and 642°C. The aluminum phase is a disordered face centered cubic (FCC) structure, while the UAl_4 phase is an ordered intermetallic phase, based on an orthorhombic structure. Deviation from 13.2 wt. % U will cause either primary Al or UAl_4 to form in conjunction with this eutectic microstructure. If the composition contains more than 15.5 to 16 wt. % U, primary UAl_3 (cubic $L1^2$ structure) will form followed by a transformation to UAl_4 by a peritectic reaction. Both the UAl_3 and UAl_4 phases are commonly observed to form in a faceted manner, which leads to irregularly shaped phase boundaries in the alloy system.

Ternary additions to the alloy can also cause the composition to deviate from the simple two-phase field. To accommodate additional constituents, the composition of each phase in the melt may change or new phases may form. Figure 2 and Figure 3 are the phase diagrams for the aluminum-gadolinium and

aluminum-hafnium systems, respectively. When comparing these two diagrams to the U-Al phase diagram, similar phases may be observed. Specifically, Figure 2 shows the existence of a $GdAl_4$ and a $GdAl_3$ phase and Figure 3 shows a $HfAl_3$ phase. Although the $GdAl_3$ and $HfAl_3$ phases exhibit different structures than their uranium counterpart, the $GdAl_4$ phase is isomorphous with the UAl_4 phase. Thus, from a preliminary examination of the phase diagrams and crystal structures for the aluminum-rich intermetallic phases in the three binary alloys systems, the neutron absorber additions can be expected to exhibit the following behavior: 1) partitioning to the UAl_x intemetallic phases or 2) formation of a unique ternary phase with uranium. For either case uranium will be co-located with a neutron absorbing species which will provide effective criticality control.

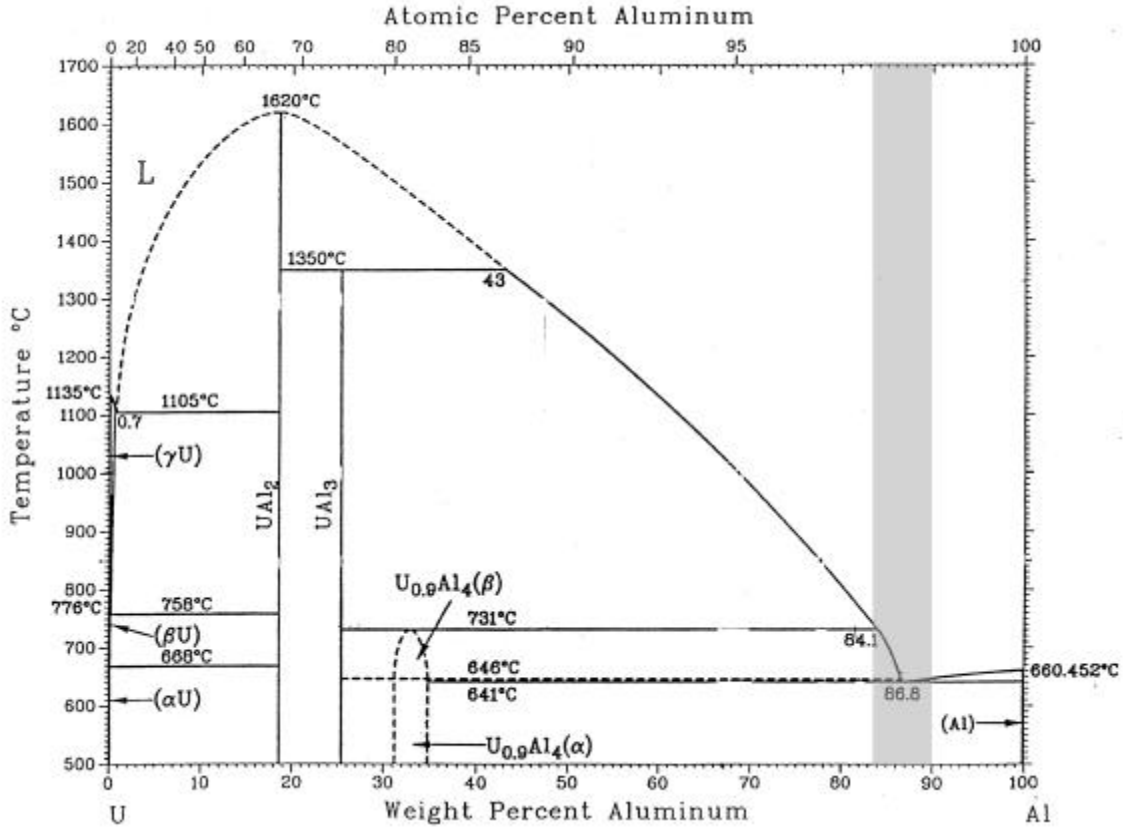


Figure 1 Uranium-Aluminum Phase Diagram.

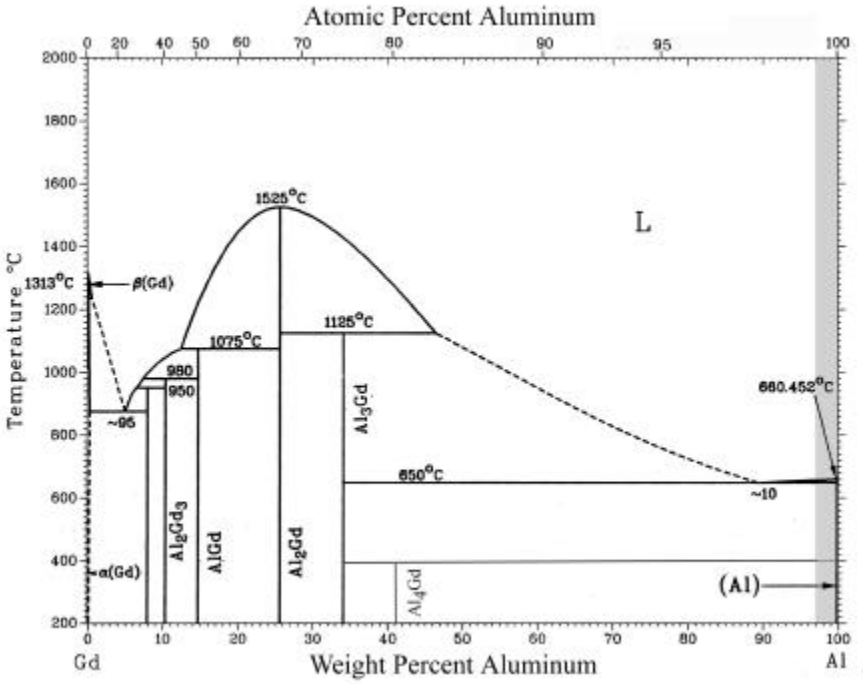


Figure 2 Gadolinium-Aluminum Phase Diagram.

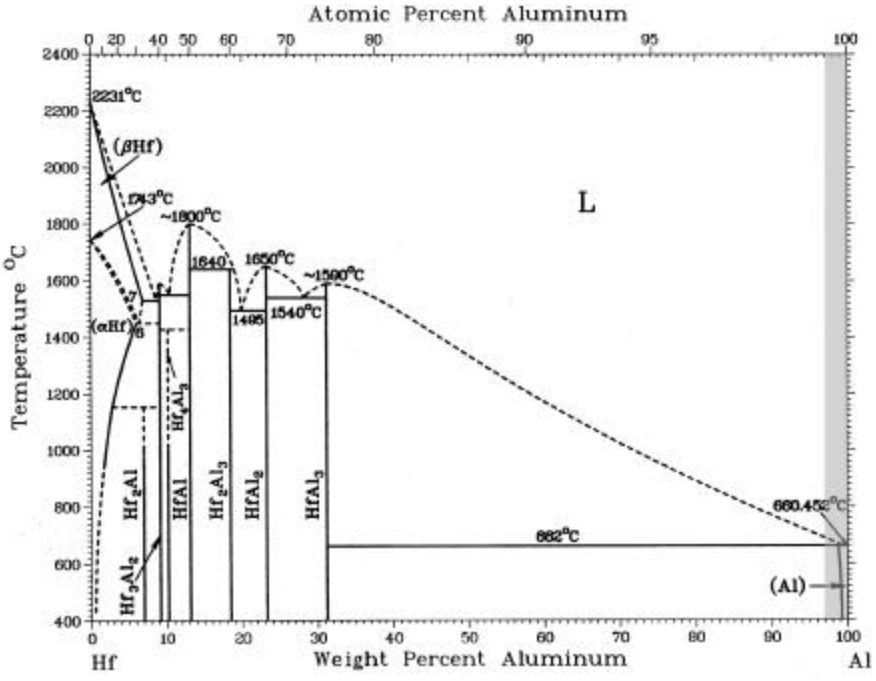


Figure 3 Hafnium-Aluminum Phase Diagram.

3.4 The Melt-Dilute/Neutron Absorber System Fabrication

The Al-SNF assemblies are typically fabricated using enriched uranium and aluminum alloy, with an aluminum alloy cladding. During reactor service, fission products are produced within the assemblies. The relatively small quantities of fission products by mass, however, are not expected to significantly alter the microstructure of the MD-SNF form from a simply binary mixture of uranium and aluminum. Therefore, the compatibility program at SRS utilizes a surrogate fuel assembly fabricated using depleted-uranium and aluminum to simulate the behavior and characteristics of actual MD-SNF. The radiation effects are simulated through gamma radiolysis studies. These surrogate fuel assemblies are melted and alloyed with additional aluminum to obtain a near eutectic composition (~13.2 percent by weight U in Al). Neutron absorber materials are added (in levels of 1.5 to 3 wt. %) during the melt-dilute treatment to produce samples used in the compatibility program, as necessary. These levels are higher than expected for the MD-SNF but allow for easy detection in phase segregation and degradation studies.

Neutron absorber doped aluminum-uranium alloys were prepared using a commercial grade 1100 Al alloy, reactor grade depleted uranium and 99.9% purity gadolinium and hafnium. These alloys were prepared using an induction casting furnace (see Figure 4) operated at approximately 25 kW. The alloys were melted in HLM grade graphite crucibles at melt temperatures of 850°C. The melting procedure for these alloys involved melting approximately 8380 gms of 1100 Al alloys followed by induction stirring during which additions of depleted uranium (≈ 1320gms) and neutron absorber (Gd/Hf ≈ 300gms) were added. The melt was induction stirred for 3 minutes. The alloys were re-heated to 850°C at which time furnace power was shut-off and the melts were allowed to furnace cool. Samples were cut from the solidified ingot (see Figure 5) using a wire EDM and characterization was performed using light optical microscopy, x-ray diffraction, and scanning electron microscopy with EDS.

Figure 4 Induction Furnace Used to Produce Surrogate MD-SNF Ingots.

Figure 5 Surrogate MD-SNF Ingot Produced in the Induction Furnace.

4.0 MELT-DILUTE/NEUTRON ABSORBER SYSTEM MICROSTRUCTURE

4.1 Microstructure of Binary Uranium Aluminum Alloys

The general microstructure of the MD-SNF form is that of a simple binary eutectic. The eutectic composition is usually reported as 13.2 wt. % U but deviation from this value is common because of the nature of solidification in a faceted/non-faceted eutectic microstructure. The eutectic composition is observed to be sensitive to cooling rate or impurity level, and hence, will not be the same under varying processing conditions.[7] If the melt composition deviates slightly from the eutectic composition, the microstructure will form primary Al or UAl_4 followed by the formation of a eutectic between regions of the primary phase. An example of this microstructure is presented in Figure 6. In SEM micrographs, UAl_x phases appear as light phases contrasted to the dark appearance of the Al matrix. In this figure, blocky primary UAl_4 is observed surrounded by a layer of the aluminum phase and then a coupled eutectic of Al + UAl_4. From this microstructure, we can discern that the actual composition of the alloy is higher in uranium content than the eutectic composition (i.e., hypereutectic). The presence of an intermediate aluminum layer between the primary phase and the eutectic region is common in faceted systems and results from the sluggish growth kinetics of the faceted UAl_4 phase in the coupled eutectic.[7]

In some cases the composition may vary enough (> 15.5 wt. % U) or impurities can change the microstructure such that other UAl_X phases form. In Figure 7a, the UAl_x phases are surrounded by an Al matrix in a binary U-Al alloy that has iron (Fe) as its primary impurity. Figure 7b presents the x-ray map that indicates the presence of uranium. Higher uranium density leads to lighter appearance in the x-ray map. These figures suggest the presence of two separate uranium-containing phases. The phases are UAl_3 and UAl_4. The UAl_3 phase appears lighter, of the two phases due to its enriched uranium content. In this particular case, the UAl_4 phase also contains Fe (see Figure 6) in solid solution while no iron was observed in the UAl_3 or the Al. Iron is a common impurity in commercial grade aluminum and is expected to be present in the MD-SNF form.

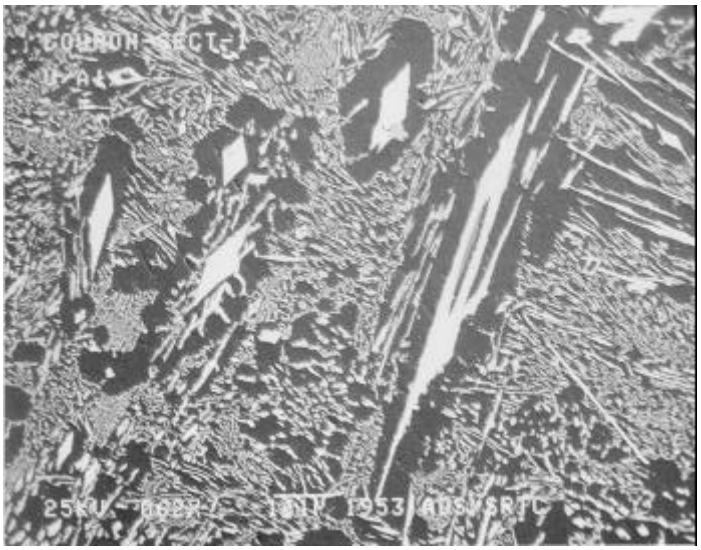
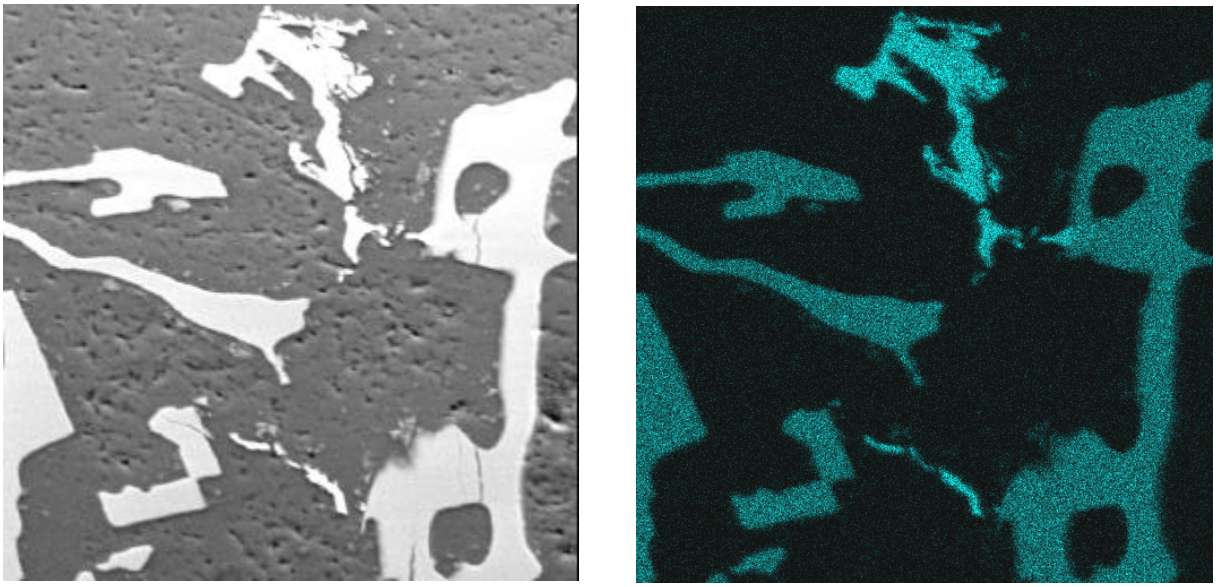


Figure 6 General Microstructure of the Binary U-Al Melt-Dilute Form.



a) b)

Figure 7 Back-Scattered Electron Micrograph (a) and a X-ray Map of Uranium in the Same Region (b) of the Surrogate MD-SNF Form.

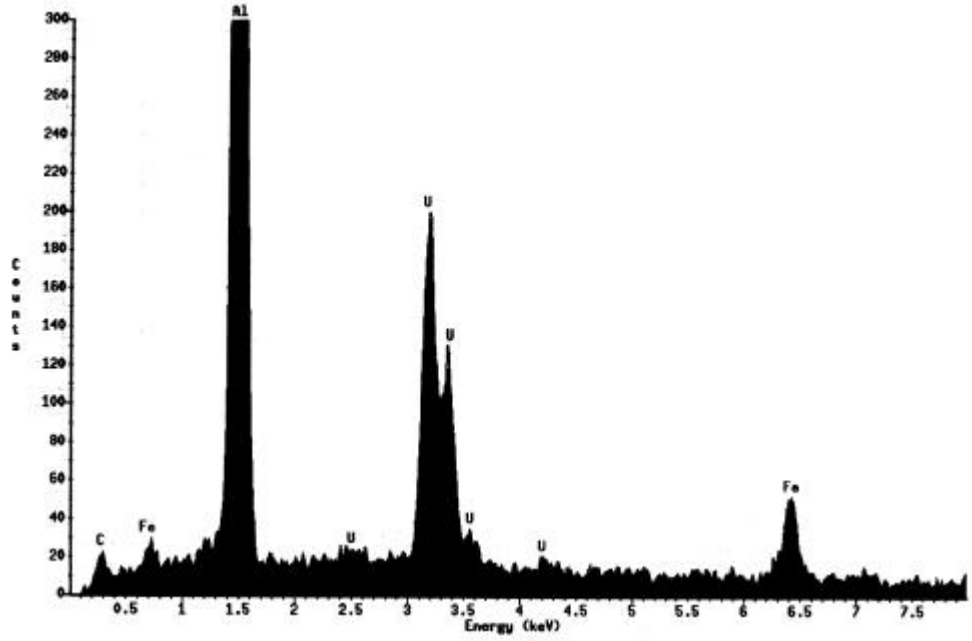


Figure 8 An Energy Dispersive Spectroscopy (EDS) Scan of the UAl_4 Phase Observed in Figure 7 (right side) Showing Iron in Solid Solution.

4.2 Microstructure of Uranium, Aluminum and Gadolinium Alloys

The addition of gadolinium in amounts up to a total of three percent by weight, to the MD-SNF form does not substantially alter the microstructure of the form. An example of this microstructure is observed in Figure 9. A comparison of Figure 9 with Figure 6 shows the microstructures with and without gadolinium additions are similar. Once again, large blocky UAl_4 phases are surrounded by a thin aluminum layer followed by a eutectic of Al + UAl_4. No UAl_3 is observed in this sample (either by SEM analysis or by x-ray diffraction). The detailed illustration of microstructure and elemental partitioning of this alloy are presented in Figure 10 and Figure 11, respectively. Figure 10 shows a high magnification SEM micrograph of the UAl_4 + Al eutectic. Figure 11a presents the x-ray map that indicates the presence of uranium within the MD-SNF form, while Figure 11b presents the x-ray map indicating gadolinium location. A comparison of these figures shows that the gadolinium added to the melt-dilute form collocates with the uranium present in the UAl_4 phase.

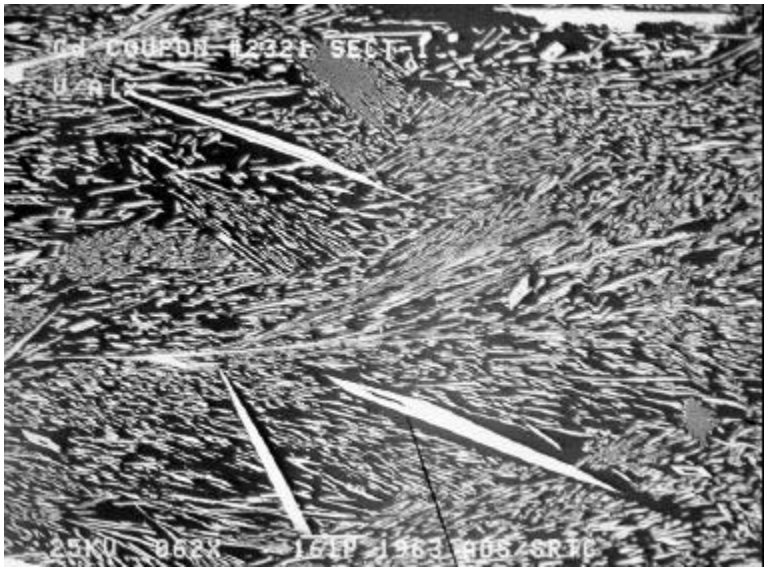


Figure 9 General Microstructure of the Binary U-Al Melt-Dilute Form with 3% Gd by Weight.

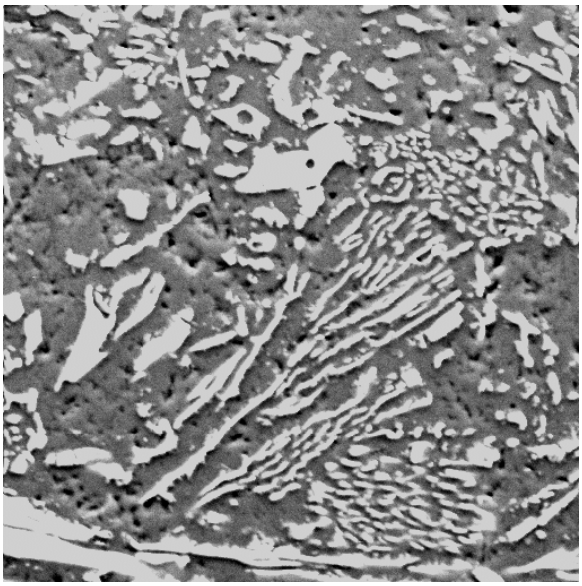
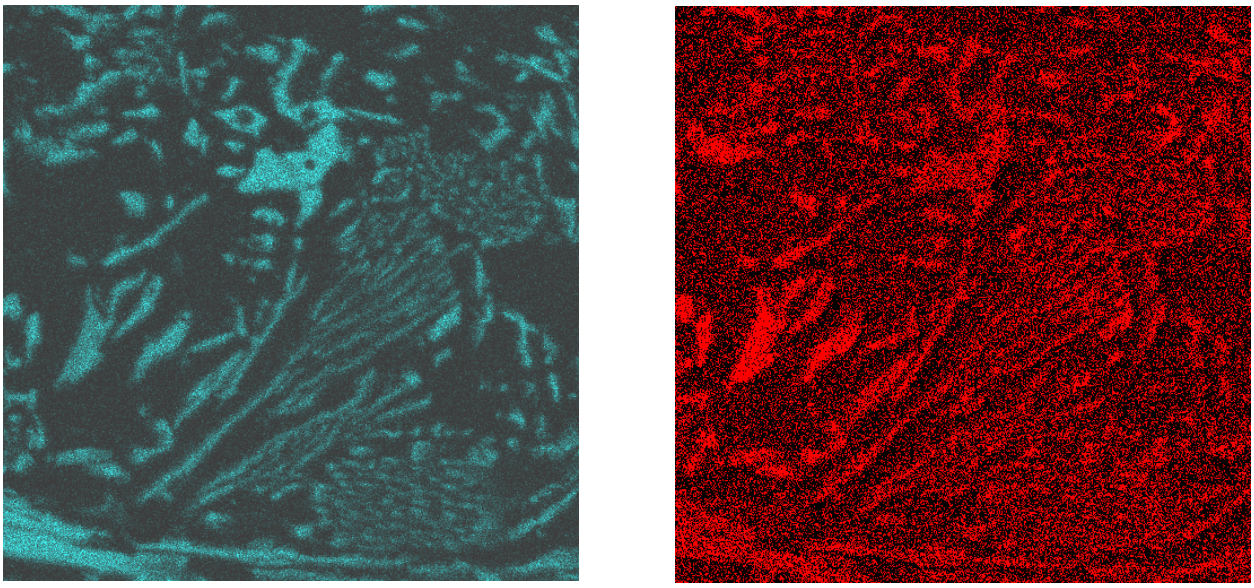


Figure 10 Back-Scattered Electron Micrograph of the Surrogate MD-SNF Form with 3% Gd by Weight.



a) **b)**

Figure 11 X-Ray map of Uranium (a) and of Gadolinium (b) in the Surrogate MD-SNF Form with 3% Gd by Weight.

4.3 Microstructure of Uranium, Aluminum and Hafnium Alloys

The microstructure of the melt-dilute form with an addition of 3% hafnium by weight is presented in Figure 12 and Figure 13. In Figure 12, bright, blocky particles are surrounded by dark gray regions of aluminum dendrites, followed by light gray areas of simple eutectic. Figure 13 illustrates the partitioning of elements in this alloy with Energy Dispersive Spectroscopy (EDS) scans from each region of the

microstructure presented. These EDS scans clearly show that the bright blocky particles contain the majority of the hafnium, as well as significant levels of uranium and aluminum. This phase was identified as a hafnium containing (U, Hf)Al_3 solid solution by x-ray diffraction. Adjacent to these blocky phases is the binary form of UAl_3 (i.e., almost no Hf) along with pure aluminum dendrites. Both of these phases are observed to contain almost no hafnium. Finally, the light gray regions of the UAl_4 + Al coupled eutectic surround these regions. An EDS scan of the UAL_4 phase in the eutectic region show no hafnium present.

From these figures, it is evident that the hafnium added to the melt-dilute form is located primarily in a (U, Hf)Al_3 phase, as opposed to the orthorhombic UAl_4. Once the hafnium partitions to this phase, a mixture of binary UAl_3 and aluminum dendrites nucleate at the solid/liquid interface prior to the coupled eutectic of UAl_4 + Al. The presence of this intermediate layer between the primary phase and the eutectic region is a result of sluggish growth kinetics of the faceted UAl_4 phase in the coupled eutectic.

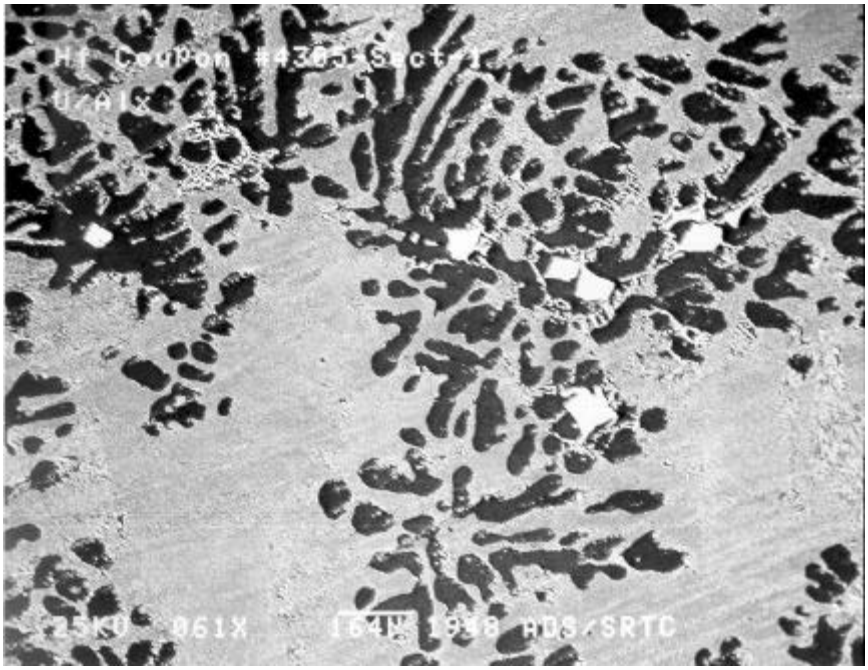


Figure 12 SEM Photomicrograph of the Surrogate MD-SNF Form with 3% Hf by Weight.

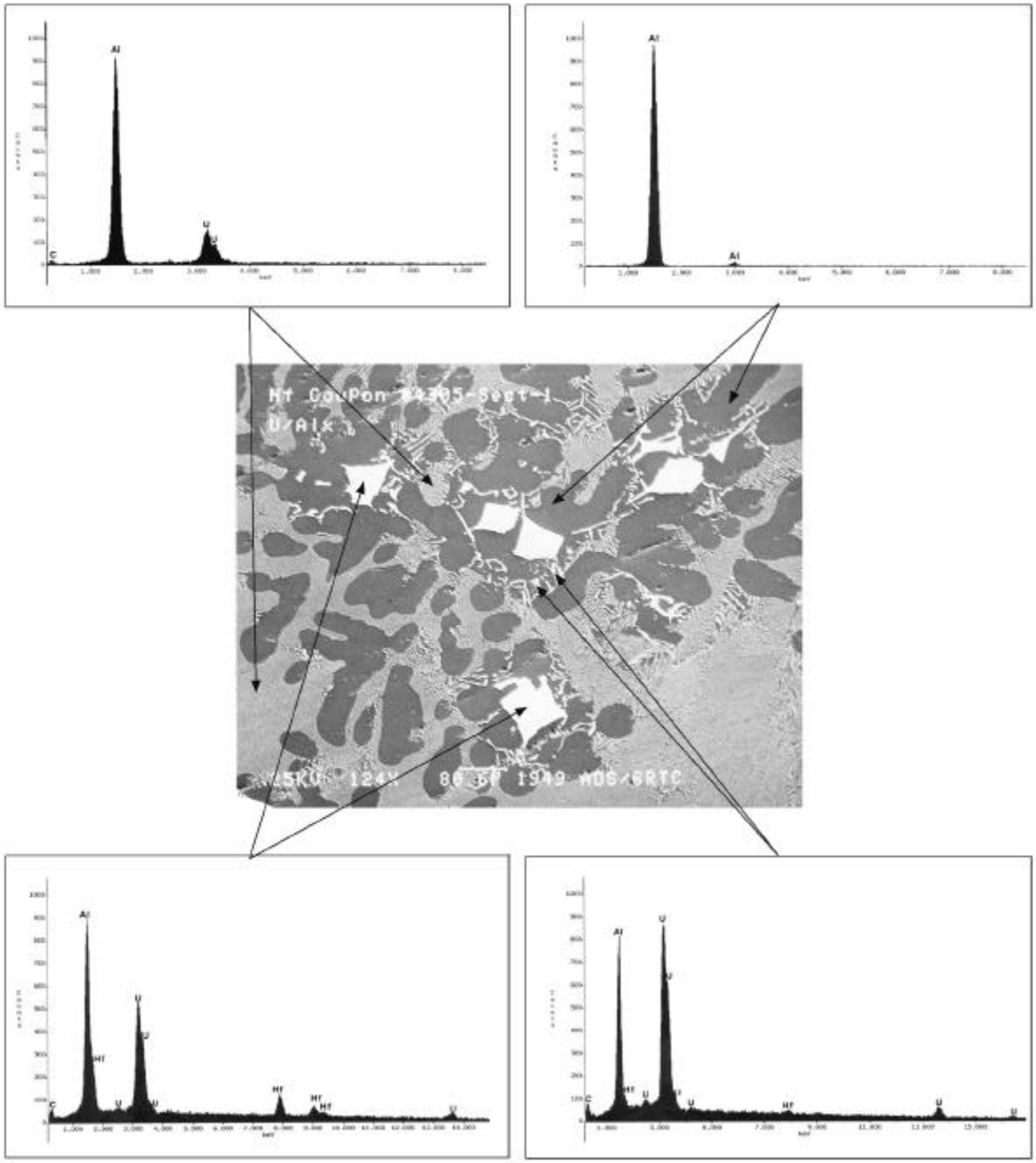


Figure 13 Detailed Microstructure of the U-Al System with 3% Hf by Weight along with EDS Scans of Individual Phases.

4.4 Microstructure of Uranium, Aluminum, Gadolinium and Hafnium Alloys

The microstructure of the melt-dilute form with an addition of 1.5 % gadolinium and 1.5 % hafnium (by weight) is presented in Figure 14. The microstructure in this alloy closely resembles the microstructure in the U-Al + Hf alloy with the exception of a new phase present. In addition to the primary blocky (U, Hf)Al_3 phase (exemplified in Figure 12), a binary $HfAl_3$ phase is observed. Figure 15 illustrates the partitioning of the elements in this alloy with EDS scans from specific phases of the microstructure presented. In this figure, the presence of a binary $HfAl_3$ phase, a (U, Hf)Al_3 phase, a (U,Gd)Al_4 phase and an Al phase are all observed by their EDS spectra. From these figures, it is apparent that a binary $HfAl_3$ phase nucleates heterogeneously from the liquid followed by blocky (U, Hf)Al_3 phases at the solid-liquid interface. An intermediate layer of aluminum dendrites then form before the UAl_4 + Al coupled eutectic. From the EDS scan of the (U, Hf)Al_3 phase, small levels of Gd can be observed to be present in this phase. However, Gd preferentially partitions to the UAl_4 phase and Hf preferentially partitions to both (U, Hf)Al_3 type phases. No binary UAl_3 was observed in this alloy which suggests a different liquidus path than that of the U-Al + Hf alloy.

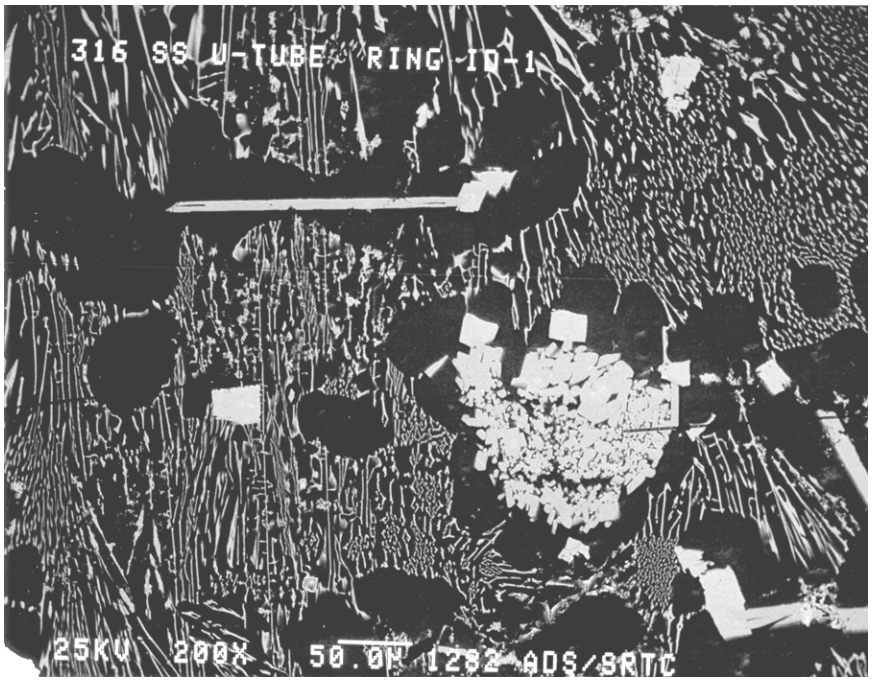


Figure 14 SEM Photomicrograph of the Surrogate MD-SNF Form with 1.5% Gd and 1.5% Hf by Weight.

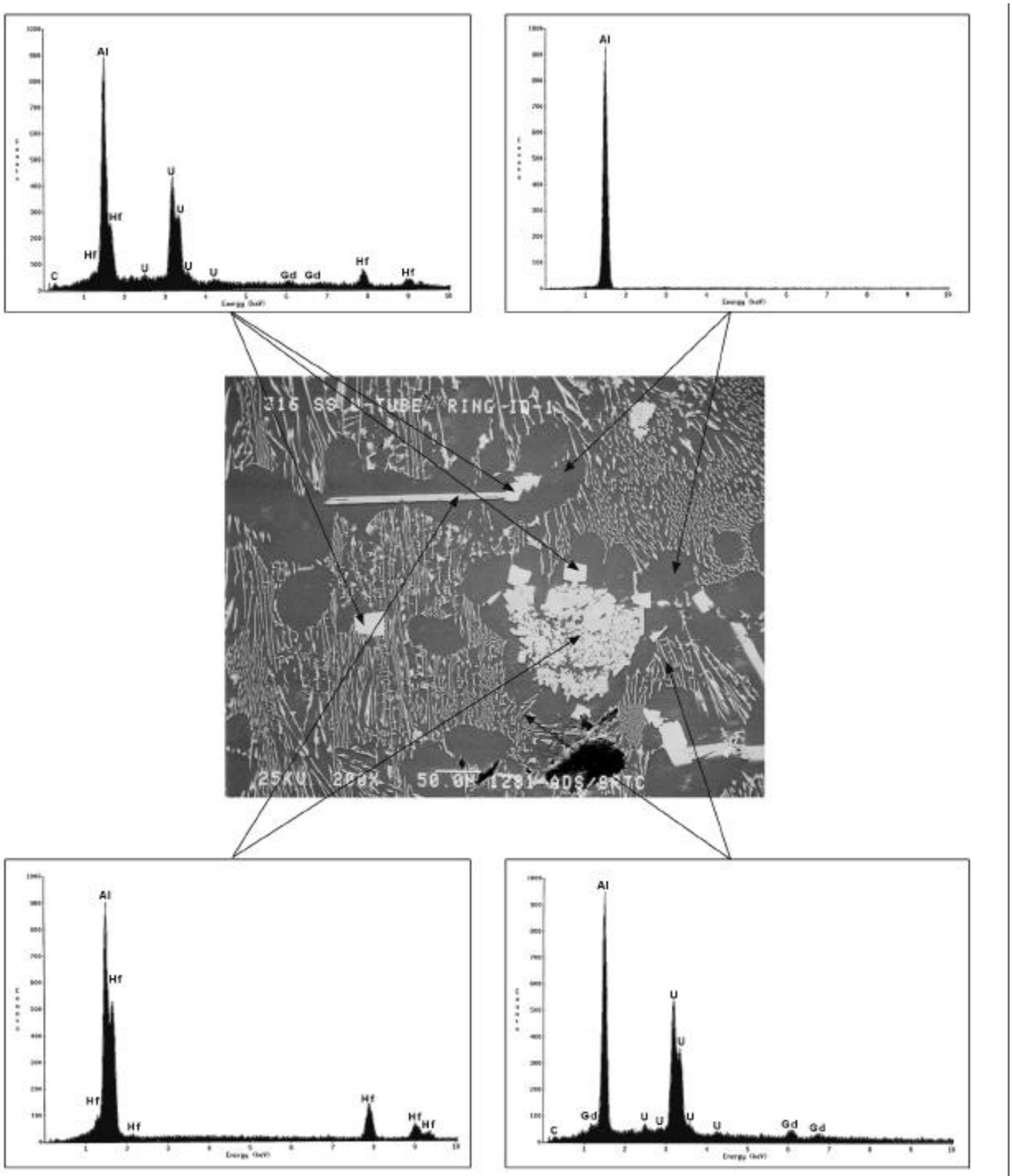


Figure 15 SEM Photomicrograph of the Surrogate MD-SNF Form with 1.5% Gd and 1.5% Hf by Weight along with EDS Scans of Individual Phases.

4.5 Summary

Al-U Melt-dilute SNF Form surrogates with integral neutron absorbers were successfully fabricated using the Melt-Dilute treatment process. Samples with Gadolinium and Hafnium neutron absorbers were metallurgically characterized. Gadolinium additions tend to concentrate in the UAl_4 phase that is more corrosion resistant than the bulk MD-SNF form. Therefore, the gadolinium is expected to stay closely associated with fissile uranium in the UAl_4 phase as the MD-SNF form degrades, providing an additional barrier against the solubility facilitated removal of gadolinium from the waste package. Hafnium additions tend to concentrate with aluminum in a $(U, Hf)Al_3$ phase with or without trace quantities of gadolinium. This makes the distribution of Hf in the waste package more heterogeneous than Gd and may make the hafnium more susceptible to release from the MD-SNF form by corrosion. However, the relative insolubility of hafnium would prevent it from being removed from the waste package. The combination of gadolinium and hafnium for criticality control provides the optimum microstructure and phase distribution for maintaining criticality control of the MD-SNF form.

5.0 PATH FORWARD

There are three major areas of continued activity for the qualification of the melt-dilute SNF form in support of the license application and subsequent repository disposal. These activities are the final criticality analyses, including degradation and geochemistry modeling, the continued fabrication and characterization of the MD-SNF forms for the silicide and oxide based Al-SNF assemblies, and the reconfiguration experimental testing program. The results of these activities will be included in the final report of the melt-dilute SNF form characteristics.

The final criticality analysis of the melt-dilute Al-SNF form will provide a defensible parametric evaluation of the melt-dilute Al-SNF form in various configurations and at various stages of degradation of the melt-dilute Al-SNF form, the DOE SNF canister, and the DHLW glass within the waste package. This analysis includes consideration of geochemical and degradation behavior and the neutron absorption characteristics of the waste package contents. This analysis is underway and will be completed in FY01. The completed analysis will demonstrate that neutron absorbers incorporated in a melt-dilute SNF form provides sufficient negative reactivity to preclude the possibility of achieving nuclear criticality within an emplaced waste package. The results of the criticality analysis will provide the necessary information required to define the final composition of the melt-dilute Al-SNF form.

Research activities to date have demonstrated that the neutron absorbers, gadolinium, hafnium, and a combination of the two, can be incorporated into a homogeneous melt-dilute SNF form for the U-Al spent fuel assemblies. The metallurgical evaluation of the surrogate melt-dilute/neutron absorber systems provides evidence that the melt-dilute treatment can integrate neutron-absorbing materials into the MD-SNF form uniformly throughout the ingot in U-Al phases. Both neutron-absorbing elements exhibited desirable alloying characteristics. Work to determine the phase boundaries of MD-SNF form for surrogate uranium-aluminum alloy fuels are ongoing. Additional studies on surrogates of silicide and oxide alloy fuels are being initiated.

The reconfiguration experimental testing program includes continued vapor and aqueous corrosion tests that will provide data on the degradation characteristics and products of the melt-dilute SNF forms in repository relevant chemical environments. This data will provide validation of the assumptions and results used in the geochemical and degradation analyses that support the criticality analysis in demonstrating criticality control in the waste package. In addition, solubility data and data on corrosion product particle size distribution will be generated in the corrosion testing program in support of the criticality analysis.

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