CONSOLIDATION OF CLADDING HULLS FROM THE ELECTROMETALLURGICAL TREATMENT OF SPENT FUEL

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ABSTRACT

To consolidate metallic waste that is residual from Argonne National Laboratory's electrometallurgical treatment of spent nuclear fuel, waste ingots are currently being cast using an induction furnace located in a hot cell. These ingots, which have been developed to serve as final waste forms destined for repository disposal, are stainless steel (SS)-Zr alloys (the Zr is very near 15 wt.%). The charge for the alloys consists of stainless steel cladding hulls, Zr from the fuel being treated, noble metal fission products, and minor amounts of actinides that are present with the cladding hulls. The actual irradiated cladding hulls have been characterized before they were melted into ingots, and the final as-cast ingots have been characterized to determine the degree of consolidation of the charge material. It has been found that ingots can be effectively cast from irradiated cladding hulls residual from the electrometallurgical treatment process by employing an induction furnace located in a hot cell.

I. INTRODUCTION

Argonne National Laboratory is developing an electrometallurgical treatment process for spent nuclear fuel. The process uses an electrorefiner to dissolve spent nuclear fuel in a molten-salt electrolyte and to collect uranium as a pure metal on a stainless steel mandrel [1]. Metallic components are left behind in the process (mainly in the anode dissolution baskets) and these are consolidated into a metal waste form (MWF) [2], after adhering LiCl-KCl eutectic salt has been distilled away from the material left in the anode dissolution baskets. The metallic components consolidated into a MWF include: stainless steel (from cladding and reactor hardware), ≈15.0 wt.% zirconium (from the U-Zr and U-Pu-Zr alloy fuels), fission products noble to the process (e.g., Ru, Pd, Tc, etc.), and minor amounts of actinides. The MWF is destined for disposal in a geologic repository. The initial demonstration of this process is being performed using 100 driver assemblies and 25 blanket assemblies from the Experimental Breeder Reactor-II (EBR-II) located in Idaho.

Over 80% of the MWF comprises added stainless steel, much of it in the form of cladding hulls (where a driver cladding hull is a ≈0.25 inch long x 0.23 inch diameter x 0.015 inch thick segment of tubing). These cladding hulls must completely melt in order to produce a homogenous, well-consolidated waste form ingot. To better understand the nature of the charge material being consolidated, actual irradiated cladding hulls have been analyzed straight out of the electrorefiner and after the distillation step. The analysis was performed using a scanning electron microscope (SEM) and energy-dispersive spectroscopy (EDS) to try and determine if any layers or other microstructural features may be present in the cladding hulls that could impede melting. In addition, the composition of the cladding hulls and any adhering material has been determined using chemical analysis.

Melting experiments have been performed to determine the ease in which irradiated cladding hulls can be melted and solidified into homogeneous, high-quality ingots. Earlier work with non-irradiated materials [2] showed that by holding the charge material for 2 hours at 1600°C high-quality ingots could be generated. As a result, all melting experiments have been performed at 1600°C using hold- times of up to 3 hours. The melting experiments involved casting up to 1 kg-sized ingots in a resistance furnace located in an argon hot cell and up to 6 kg-sized ingots in an induction furnace, also located in a hot cell.

This paper reports the cladding hull SEM/EDS and chemical analysis results, along with the results from the different melting experiments that were conducted. The degree of cladding hull consolidation is noted along with the main features of some of the as-cast ingot microstructures.

II. EXPERIMENTAL

Irradiated Type 316 SS cladding hull samples were selected at various stages of the MWF casting process for SEM/EDS examination. The MWF casting process consisted of the following steps: (1) placing the cladding hulls from the electrorefiner into a stainless steel container, (2) heating the container with the cladding hulls to 1100°C (at ≈1 torr) for many hours in a distillation furnace to remove adhering salt, (3) then, transferring the salt-free cladding hulls, added Zr, and maybe stainless steel into an yttria crucible, (4) heating the loaded yttria

crucible to 1600°C for up to 3 hours in a vacuum induction casting furnace, and (5) removing the disc-shaped ingots from the crucibles and transferring the ingots into appropriate containers for storage.

Samples for SEM/EDS analysis were taken directly after the electrorefining, the distillation, and the ingot casting steps. For the samples taken directly after electrorefining, a water-washing step was implemented to remove the salt. The distillation step required exposing the salt-coated cladding hulls to a temperature of 1100°C for 1 hour at less than 10 Torr operating pressure. For the sample taken after the casting step, an unmelted cladding hull, which was adhering to an ingot after casting, was disattached from the ingot and submitted for analysis. SEM/EDS analysis was performed on all the selected samples using an ETEC Autoscan SEM equipped with a Kevex 8000 (Fisons Instruments) energy dispersive X-ray (EDX) analysis system. Before the analysis, samples were first cross-sectioned transversely, mounted, polished through 1 μm diamond, and then gold-coated.

After each electrorefining run was completed, chemical analysis was performed on ten cladding hulls samples from each fuel dissolution basket. The cladding hulls were selected from the top, middle, and bottom regions of the baskets. After the hulls were selected, they were washed in water, to remove adhering salt, and then dissolved in an acid solution. Measurement of the various components in the solution was completed using a variety of techniques: mass spectrometry, ICP-atomic emission spectrometry, and gamma spectrometry.

For the melting experiments that were conducted, a total of ten experiments were performed in a resistance furnace located in a hot cell called the Hot Fuel Examination Facility (HFEF). Six larger-scale experiments were performed in a vacuum induction furnace called the Casting Furnace (CF), which is located in a hot cell called the Fuel Conditioning Facility (FCF). All castings were generated using irradiated Type 316SS, except for two of the FCF ingots, where D9SS (a Ti-stabilized 316SS) was consolidated.

Nine of the ten HFEF experiments involved melting 5 to 10 cladding hulls in yttria crucibles (2.0 inches long x 0.5 inches inner diameter) at temperatures up to 1700°C. One ingot was generated using an yttria crucible approximately 3.0 inches diameter x 5.5 inches high, and it had a weight of ≈1.0 kg.

The FCF melting tests were conducted using the standard MWF casting procedure, as described earlier. The effects of changing some of the furnace parameters were investigated. Some of the changed furnace parameters included: hold time, temperature, and power. The shortest hold time possible and the lowest furnace temperature possible was investigated to try and mitigate wear and tear on the furnace. Changes in power levels, in terms of increasing and decreasing the power rapidly over a period of time, were evaluated to see if stirring of the melt could be initiated to try and maximize the homogeneity of the as-cast ingot.

Some of the ingots cast in HFEF and FCF were sampled. The 1 kg ingot, cast in HFEF, was sampled using a high-speed core drill. The sample that was generated was ≈1.5 inches long x 0.5 inches diameter, and it was taken from the center of the ingot. For ingots FCFMWF05 and FCFMWF06, cast in FCF, samples were taken via a vacuum injection-casting technique. Samples, several inches long x 0.170 inches diameter, were vacuum injected into quartz molds. From these core-drilled and injection-cast pins, samples exposing transverse and longitudinal cross-sections were cut, mounted, and polished. Pictures were taken of the microstructures of the samples using an AMRAY scanning electron microscope housed in a shielded glovebox in HFEF.

III. RESULTS

A. Characterization of Cladding Hulls

Figure 1 shows a typical microstructure for a

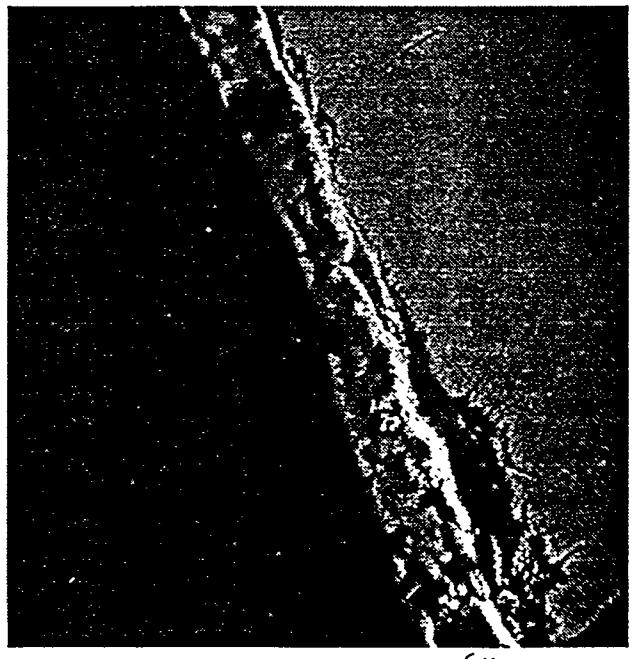
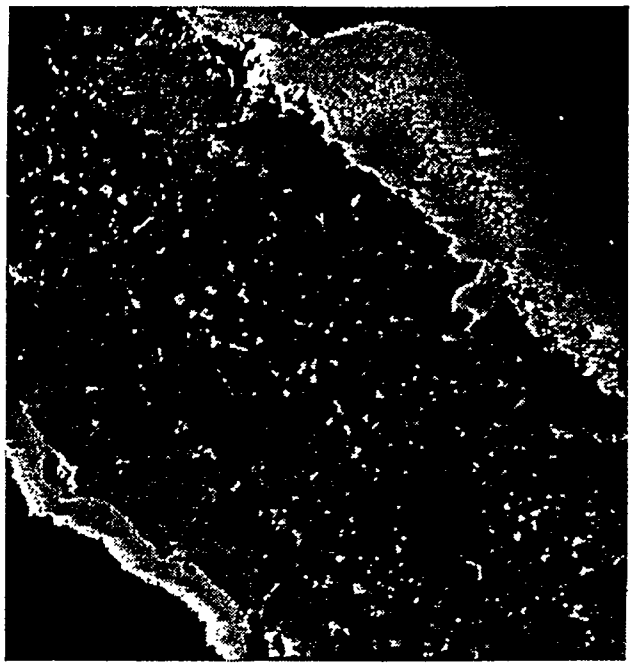


Figure 1. Scanning electron micrograph showing the inner surface of a transversely cross-sectioned irradiated cladding hull taken from the electrorefiner and water-washed. The dark-contrast region to the left is mount, followed by a medium-contrast layer of adhering LiCl-KCl salt, a bright-contrast Zr-rich layer, and then the cladding hull itself.

cladding hull that has been water-washed after coming out of the electrorefiner. These water-washed cladding hulls still had some adhering LiCl-KCl salt (as seen in Figure 1), but nearest the cladding inner and outer surface there was also another layer. This layer was confirmed by SEM/EDS to be a metallic Zr-rich layer with some U. This layer seems to have been deposited on the cladding hulls during the electrometallurgical treatment process [3].

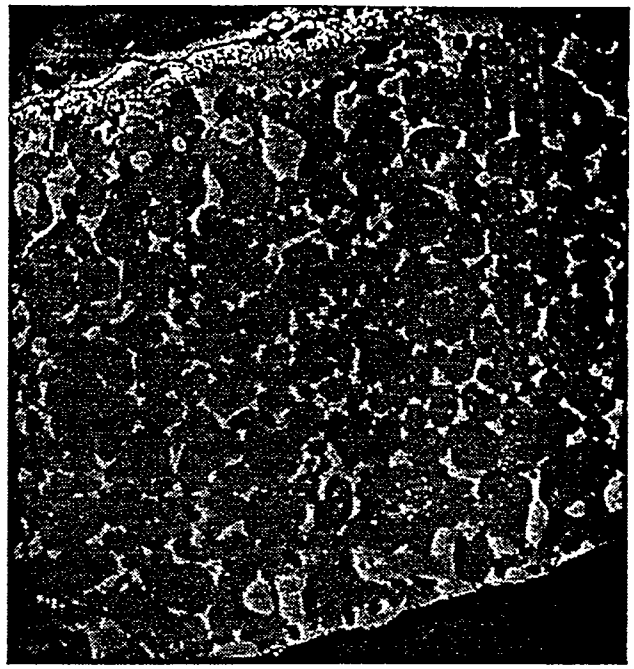
Figure 2 shows a cladding hull after the distillation step. The initial Zr+U layer has interdiffused with the cladding hull components during the many hour treatment



40 μm ——

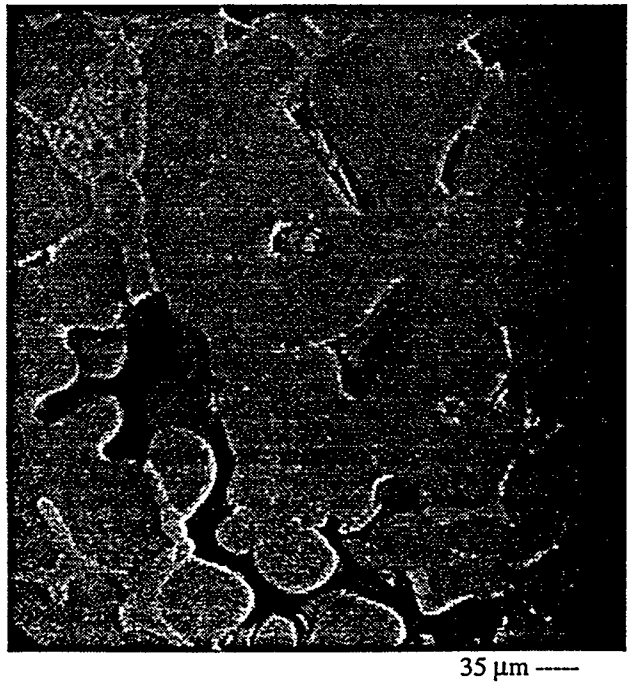
Figure 2. Scanning electron micrograph showing a transversely cross-sectioned irradiated cladding hull after the distillation step. An Fe_2Zr type of intermetallic appears as a bright-contrast, single-phase layer on the inner and outer circumference of the cladding hull. Other intermetallic phases containing cladding components, Zr, and U appear at the interior of the hull. The dark material is mount.

at 1100°C, resulting in the formation of multiphase, interdiffusion zones. The main features of these zones are a single-phase Fe_2Zr-type intermetallic phase on the outer edges of the inner and outer surfaces of the cladding hulls, along with other phases internal to the cladding hulls that contain the major cladding components (Fe, Ni, Cr, Mo, etc.) along with Zr and U. In some cases, one distillation step has been inadequate in getting off all the adhering salt, and as a result, another distillation step has been employed. In these cases, the single-phase Fe_2Zr intermetallic layer has nearly been consumed, and mostly a two-phase interdiffusion zone remains (see Figure 3).



40 μm ——

Figure 3. Scanning electron micrograph showing a transversely cross-sectioned irradiated cladding hull after being run twice through the distillation step. Intermetallic phases containing cladding components, Zr, and U appear at the interior of the hull.



35 μm ——

Figure 4. SEM micrograph showing a cross-sectioned irradiated cladding hull, heated to 1600°C in the casting furnace. The bright-contrast phase has melted during the process, and either drained away or resolidified to form a eutectic-like microstructure (upper left corner).

Table 1. Measured Concentrations of U and Zr Left With Specific Batches of Cladding Hulls After Electrorefining

Cladding Hull Batch	Distillation Furnace Batch	Wt.% U	U Standard Deviation	Wt.% Zr	Zr Standard Deviation
ERBF01A	CPMW02A	6.8	1.9	7.4	3.6
ERBF02A	CPMW03	15.6	7.8	8.1	2.6
ERBF04B	N/A	11.0	1.3	18.3	2.8
ERBF05A	CPMW04	0.8	0.1	3.9	1.2
ERBF06A	CPMW07	3.5	2.4	13.4	2.9

One irradiated cladding hull that did not melt during a casting run in the FCF induction furnace was analyzed using SEM/EDS. A cross-section of this cladding hull is shown in Figure 4. It looks as if the high-contrast phase did melt during the casting procedure at 1600°C, leaving behind some voids in the cladding hull. The solidified structure of some of the melted material can also be seen.

Results from the chemical analysis of the cladding hulls, which have been analyzed using SEM/EDS, confirm that significant Zr and U is left behind with the cladding hulls (see Table 1). The electrorefiner conditions can be tailored to get almost all the U out the cladding hulls, but then significant Zr has been found transported to the U product, deposited on the cathode. Therefore, some U is intentionally left behind with the cladding hulls, so that a majority of the Zr will also be left behind.

B. Melting Tests

1. HFEF Melting Experiments. For the small-scale HFEF melting experiments, incomplete melting of 5 to 10 cladding hulls in yttria crucibles was observed at temperatures up to 1700°C. Figure 5 shows cladding hulls that have not melted. These tests were run in the atmosphere of the hot cell (argon with ppm levels of water and oxygen) and the Zr layers on the cladding hulls may have been stabilized, due to gettering of atmosphere impurities, resulting in a Zr-rich intermetallic with a very high melting temperature(the binary Fe_2Zr phase has a melting temperature of 1673°C according to the Fe-Zr phase diagram [4]).

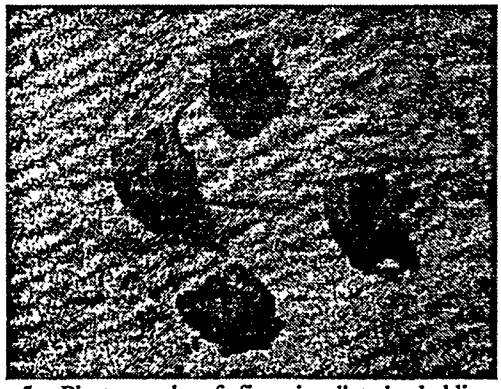


Figure 5. Photograph of five irradiated cladding hulls (two are fused together) that did not melt when held at 1700°C for 2 hours (Picture at 10x magnification).

To look at the effects of stable phase layers on the cladding hulls, some cladding hulls were etched with a nitric acid solution, to dissolve the layers (approx. 30% of an individual hull was dissolved). If cladding hulls could be melted after dissolving away surface layers, then the surface layers were probably impeding melting. Therefore, etched cladding hulls were heated to 1600°C to see if hulls without layers would melt. In fact, the hulls did melt completely. Another test, looking at the effects of the surface layers, was run and involved adding pieces of unirradiated stainless steel and Zr to the irradiated cladding hulls to see if melting would occur. The idea was that the steel and Zr additions would melt to form a low melting alloy rich in Zr, which would dissolve the Zr-layered cladding hulls. This test was run at 1600°C, and melting of the irradiated cladding hulls was observed.

Based on the results of the small-scale tests, a larger-scale melting experiment was performed using 700 grams of irradiated cladding hulls and 330 grams of unirradiated stainless steel. The stainless steel was added to assist in dissolving the cladding hulls. The charge was heated to 1600°C in a resistance furnace and held at temperature for 3 hours. Almost complete consolidation of the cladding hulls was observed, and Figure 6 is a photograph of the as-cast ingot.

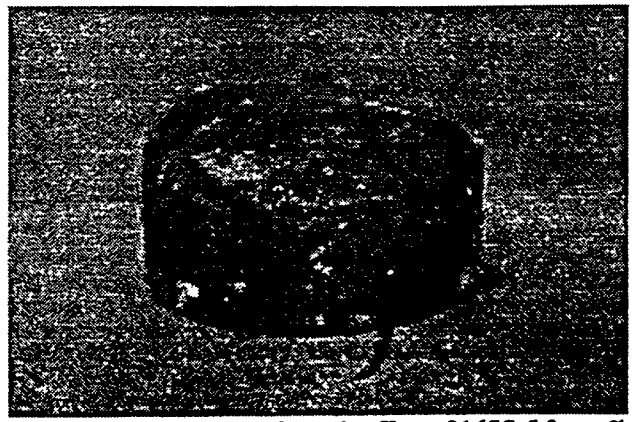


Figure 6. Photograph of a 1 kg Type 316SS-5.0 wt.% Zr alloy ingot (≈3.0 inch diameter x 1.0 inch thick) cast in a resistance furnace in HFEF.

Table 2. MWF Ingots Cast in HFEF and FCF Furnaces at 1600°C.

Ingot Designation	Cladding Hull Batch Melted	Type of SS	Ingot Weight (kg)	Hold Time (hrs)	Material Added to Hulls	Complete Melting?	Sampled?
HFEFMWF01	ERBF02A	irr. 316SS	1.03	3.0	SS	Y	Y
FCFMWF01	simulated	un-irr. 316SS	4.72	0.5	N/A	Y	Y
FCFMWF02	ERBF01A	irr. 316SS	1.93	0.5	none	N	N
FCFMWF03	ERBF01A	irr. 316SS	3.11	2.0	SS+Zr	N	N
FCFMWF04	ERBF05A	irr. D9SS	4.61	3.0	SS+Zr	N	N
FCFMWF05	ERBF02A	irr. 316SS	5.63	3.0	SS+Zr	Y	Y
FCFMWF06	ERBF06A	irr. D9SS	5.51	2.0	SS+Zr	Y	Y

To check the homogeneity of the ingot, a 0.5 inch diameter sample was core-drilled from the center of the ingot and then analyzed using an SEM. An SEM micrograph of the observed microstructure in a transverse cross-section of the core-drilled sample is presented in Figure 7. Since chemical analysis data was available for the irradiated cladding hulls used to generate this ingot, it was determined that the ingot, with the added SS, had a Zr concentration of ≈5.0 wt.%. The microstructure that developed for this ingot (as shown in Figure 7) is consistent with the microstructure reported for a SS-5 wt.% Zr alloy generated from un-irradiated SS and Zr [5].

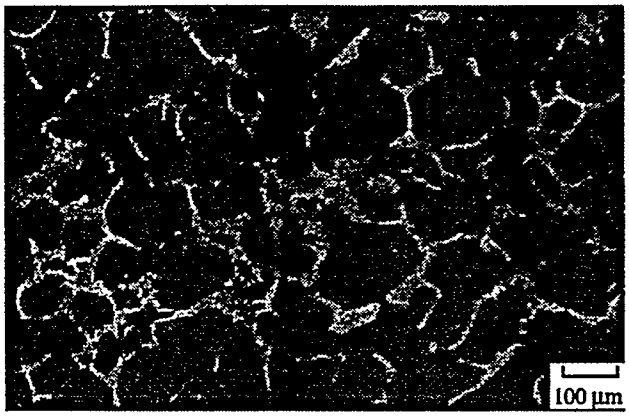


Figure 7. Scanning electron micrograph showing a transversely cross-sectioned, core-drilled sample taken from the center of the ingot cast in HFEF.

2. FCF Melting Experiments. Six metal waste form ingots have been cast in the FCF induction furnace (see Table 2). For casting the first five ingots, yttria crucibles were employed that were approximately 3.0 inches high x 0.1875 inches thick x 8.17 inches diameter at the top (the taper was 3.5°). The sixth ingot was cast using a crucible 3.0 inches taller. Since cladding hulls have a low packing density in these crucibles (≈1g/cc), a crushing procedure (using a jaw crusher) was employed with the cladding hulls used to cast FCFMWF05 to try and increase the overall loading in the yttria crucible.

The first MWF ingot (FCFMWF01) contained unirradiated Type 316 stainless steel and Zr. This ingot was the only ingot to date that has been cross-sectioned (the other ingots contained irradiated materials making them too radioactive to take out of the hot cell for cross-sectioning). Figure 8 shows the microstructure of the as-cast FCFMWF01 ingot; it consists of an Fe solid solution phase and a $Zr(Fe,Cr,Ni)_{2+x}$ intermetallic phase, which are the primary phases comprising the MWF microstructure [2]. The ingot appears to be homogeneous with only a thin <5 µm thick oxide layer present on the ingot's top surface.

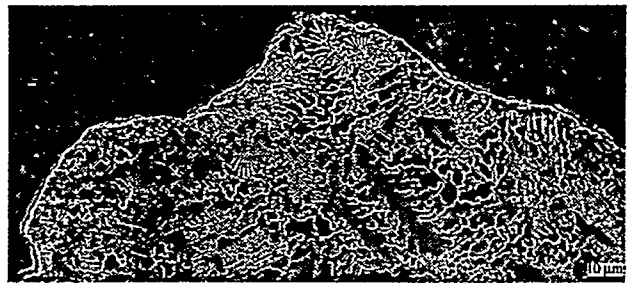


Figure 8. Montage of SEM micrographs showing the upper portion of the vertical cross-section of FCFMWF01.

Varying degrees of cladding consolidation were noted for the next three MWF ingots. Only cladding hulls were melted to cast FCFMWF02, while FCFMWF03 and FCFMWF04 consolidated cladding hulls and added SS+Zr. The Zr was added to increase the final Zr content for the alloy to near 15 wt.% (the baseline composition). The SS was added to help dissolve the cladding hulls. For all of these ingots, a large fraction of the cladding hulls were melted, resulting in large reductions in cladding volume and the formation of disc-shaped ingots. Yet, evidence of embedded, unmelted cladding hulls was observed on top of the ingots. An example of an ingot with some unmelted cladding hulls is shown in Figure 9. This partial melting of some of the cladding hulls was attributed to a high degree of heat loss from the surface of the melt. As a result, the furnace was modified so as to mitigate any heat loss. Insulation was added in specific regions of the furnace to close off any heat loss pathways, and tantalum disks and zirconia felt were fashioned into a lid to be placed on top of the yttria crucible.

With the lid and furnace modifications in place, MWF ingot FCFMWF05 was cast at 1600°C using a hold time of 3 hours. This ingot comprised 1,713 g of added Type 316SS (to help dissolve the cladding hulls), 604 g of Zr (to make the ingot a 15 wt.% Zr alloy, based on the chemical analysis of the individual cladding hulls), and 3,312 g of irradiated cladding hulls. The solidified ingot that was cast is shown in Figure 10.

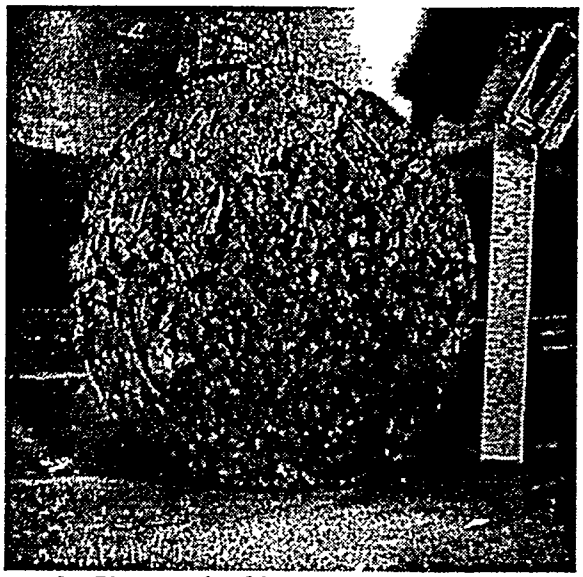


Figure 9. Photograph of ingot FCFMWF03. Unmelted cladding hulls were observed embedded in the ingot.

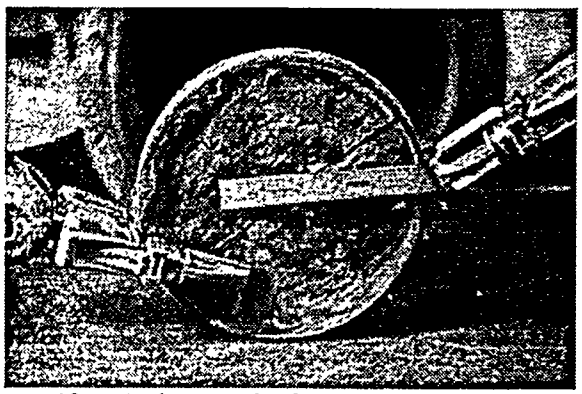


Figure 10. A photograph of a metallic waste form ingot prepared from irradiated stainless steel cladding and Zr. The ingot was generated in an induction furnace located in an argon hot cell.

Two vacuum injection cast pins were generated from the ingot melt of FCFMWF05, and each one was ≈18.0 inch long x 0.170 inches diameter. Chemical and SEM analyses were performed on samples taken from both pins. The chemical analysis results showed that the ingot contained approximately 14.2 wt.% Zr and 9.3 wt.% U. Since this ingot was generated using cladding hulls from batch ERBF02A, there should be reasonable agreement between the chemical analysis results for the injection cast pins and the starting cladding hulls. Looking at Table 1, the original ERBF02A cladding hulls had 15.6 wt.% U and 8.1 wt.% Zr. The additions of SS and Zr to these hulls reduced the total U levels to the 9.3 wt.% found in the injection-cast pin and increased the Zr levels to 14.2 wt.%.

The microstructure of the injection-cast pins is presented in Figure 11. The nominal Fe solid solution phase and $Zr(Fe,Cr,Ni)_{2+x}$ phases constitute the alloy microstructure. Some localized areas of the $Zr(Fe,Cr,Ni)_{2+x}$ phase are bright in contrast. Similar areas in other MWF alloys doped with actinides have been observed to be enriched in actinides [6]. Due to the fast cooling rate seen by the injection-cast sample, no formation of dendrites was observed in the pin samples. Whereas, dendrites are typically observed in slower-cooled, as-cast MWF microstructures [7]. This elimination of dendrite formation at faster cooling rates is not uncommon and has been reported for other alloy systems [8].

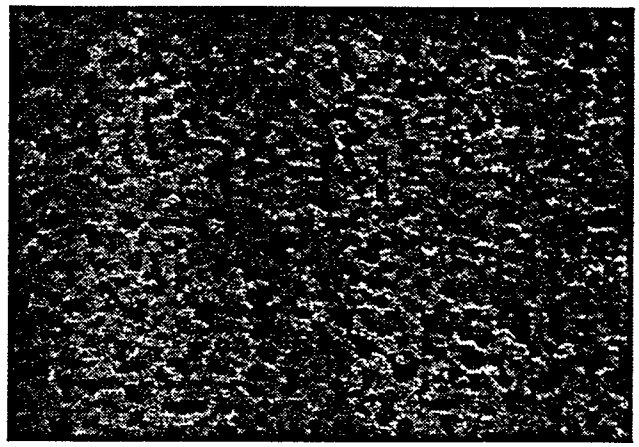


Figure 11. SEM micrograph showing the microstructure of a transverse cross-section of an injection-cast pin produced from ingot FCFMWF05.

All future MWF ingots to be cast in FCF will contain 2 EBR-II subassemblies worth of cladding hulls (≈3500 g), and to date, one ingot (FCFMWF06) has been cast using this prescribed amount of cladding hulls. In order to accomodate the increased amount of hulls, 3.0 inches has been added to the height of the yttria crucibles. This added space will also allow for the cladding hulls to be added in an uncrushed state. A picture of the FCFMWF06 ingot is shown in Figure 12. No evidence of any unmelted cladding hulls was observed. The Zr concentration in the alloy was approximately 11 wt.%.

With regards to studying the effects of specific changes in the furnace operating parameters, it has been determined that stirring cycles are probably not necessary to melt cladding hulls. In terms of the operating temperature, 1600°C seems an appropriate temperature, but

one future test will be run at 1550°C to provide more data.

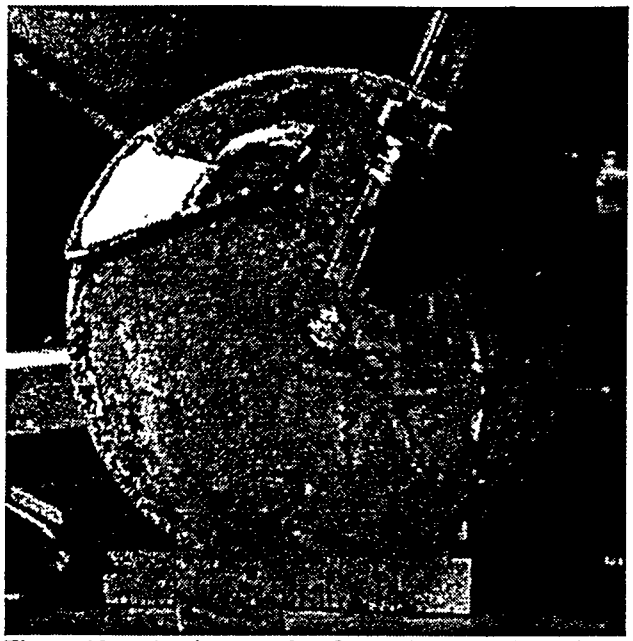


Figure 12. A photograph of metal waste form ingot FCFMWF06. A piece of the yttria crucible is still adhering to the upper left portion of the ingot.

Currently, the holding time of 2 hours has been adopted, but one test still remains where a hold time of 1 hour will be employed.

IV. DISCUSSION

The SEM/EDS and chemical analysis of the irradiated cladding hulls that are residual from the electrometallurgical treatment of spent EBR-II fuel confirmthat Zr-rich layers, with some U, are present circumferentially on their inner and outer surfaces. Mechanisms for this deposition have been identified and are reported in another paper [3]. The Zr and U in these layers interdiffuse with the cladding components, when adhering salt is distilled from the hulls at 1100°C in an induction furnace. The result is the formation of multiphase diffusion structures throughout the thickness of the cladding. Deposited (Zr+U) layers on cladding hulls are not problematic as long as all the material can be completely melted. Extensive work has been completed investigating SS alloys containing Zr and U [6], and the results show that the microstructures of these as-cast alloys are homogeneous and should exhibit good waste form properties.

Complete consolidation of irradiated cladding hulls is possible using either a vacuum induction or resistance furnace operated at 1600°C in a hot cell, based on the melting tests performed on the Zr-layered cladding hulls. Yttria crucibles can be employed to contain the charge materials, and either vacuum injection casting or core-drilling can be employed to sample the alloys.

V. CONCLUSIONS

Melting experiments have been conducted on actual irradiated cladding hulls, leftover from the electrometallurgical treatment of spent nuclear fuel; the results from these small and large scale melting experiments have shown that homogeneous, MWF ingots can be produced from small segments of irradiated cladding tubing by employing a vacuum induction furnace located in a hot cell.

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