

Casting Development Report for EFF-W Casting Furnace

Brady Mackowiak

September 2017



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September 2017

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Casting Development Report for EFF-W Casting Furnace

1. INTRODUCTION

The Experimental Fuels Facility (EFF) Casting System, which is configured for gravity casting into a permanent mold, was installed to cast billets made up of uranium, uranium alloys, copper, and copper alloys, as well as other geometries, in support of the TerraPower project. The billet caster design, installation, and testing were documented in the engineering job file #EJ-1519. This file contains Functional and Operational Requirement documents, specifications, the testing procedure, references to sketches and drawings, and other relevant material. During the billet casting furnace assembly and initial testing, several minor modifications were made, such as the addition of longer thread reliefs and radiuses on corners on graphite components, removal of the crucible skirt from around the mold, and the addition of insulation between the mold and crucible. These modifications were documented in the as-built fabrication as either sketches or drawings, as the changes were to the furnace assembly and will be provided in the equipment data package.

For feedthrough of the copper conductor, standard high temperature lava seal Conax Technologies single gland power feedthroughs were modified to be used on the furnace chamber (see Figure 1). The modification included a shouldered central conductor with an extended length sealed with two additional soft copper gaskets instead of the lava sealant system. The feedthroughs were modified to ensure a vacuum seal could be maintained at elevated temperatures. The standard lava seal is rated for increased temperature but is not vacuum-seal rated, while a Viton seal gland can maintain vacuum tightness but cannot withstand temperatures in excess of 250°C.

A solid model and photo of the EFF Casting Furnace is shown in Figure 2. The EFF Casting Furnace is contained in a stainless steel shell that uses standard ISO KF- and LF-style vacuum flanges for all sealing surfaces. The crucible is inductively heated, while the mold is heated in large part through conduction from the crucible with some small amount of inductive heating also present. A number of thermocouples can be monitored throughout the heating cycles. Modeled after other gravity casting systems designed at INL, the EFF Casting Furnace utilizes both K-type and S-type thermocouples. Two S-type thermocouples are used on the crucible with one additional thermocouple as a spare. An S-type thermocouple is also used to monitor the crucible induction coil. Two K-type thermocouples monitor the temperature of the mold; there are two spares at that location. One K-type thermocouple is used to monitor the temperature at the top outer surface of the chamber to give warning of damage to the seals. This particular thermocouple functions as an over-temperature shutdown of the heating system if the chamber becomes too hot. Three additional K-type thermocouples are available as spares and can be used where needed. The furnace uses a single-piece graphite crucible and a three-piece graphite mold.

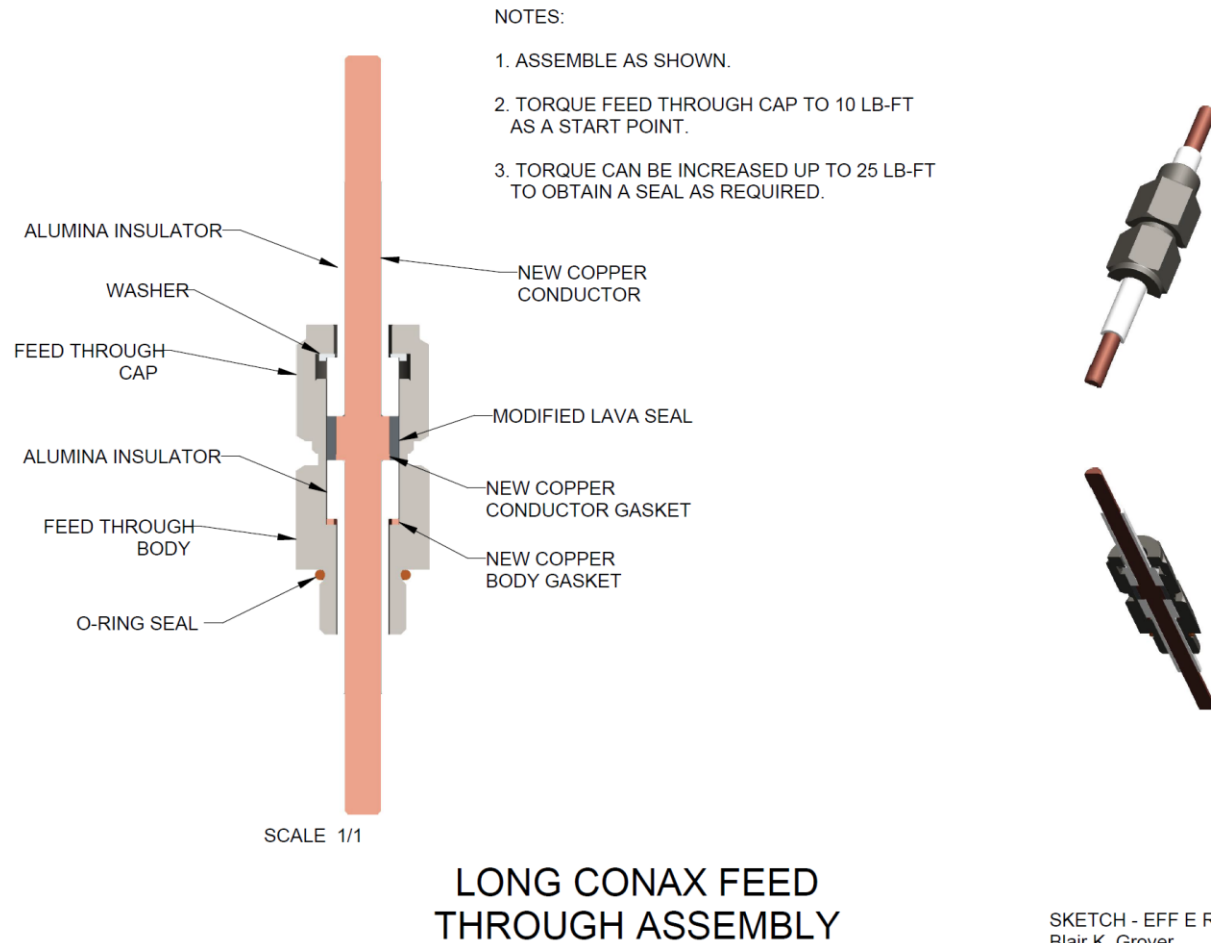


Figure 1. Modified Conax Technologies power feedthrough.

The TerraPower project calls for casting of U and U alloy billets up to 2.5 kg each for processing in the extrusion press, also located in EFF. Approximately 85 castings or casting tests have been performed in the EFF Furnace in support of the project since it was installed. These include castings of pure copper and copper alloys for use as surrogate material and billet components.

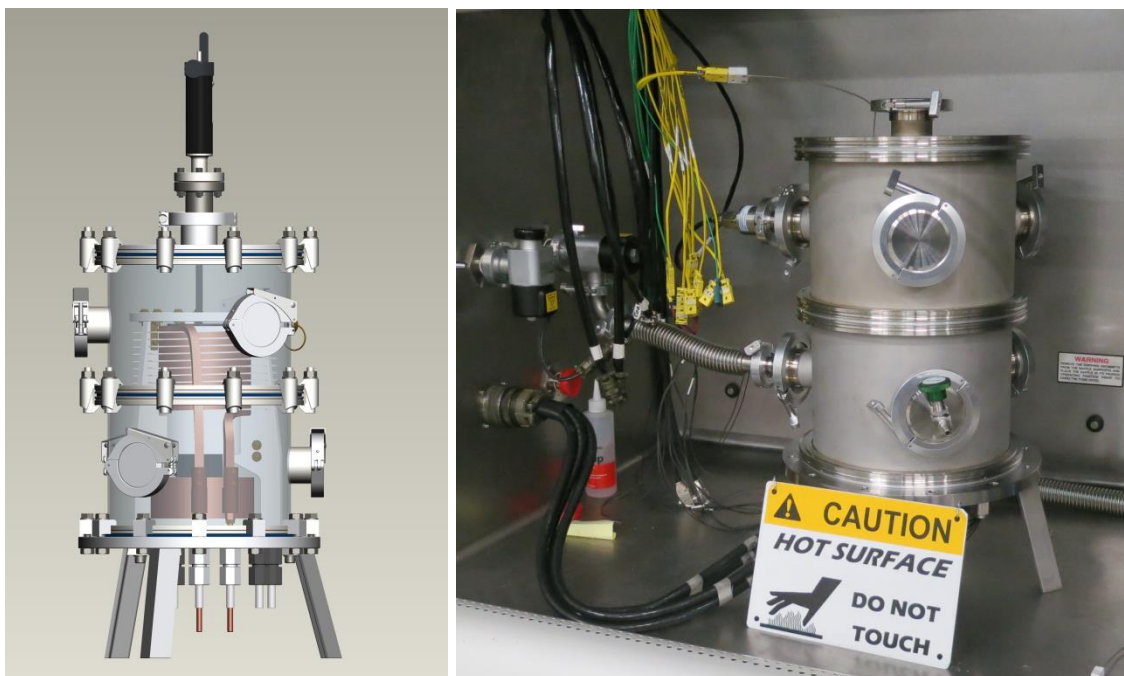


Figure 2. Model (left) and photo (right) of the EFF Furnace.

2. EFF CASTING FURNACE

2.1 Vacuum System

The EFF Casting Furnace is operated under vacuum. The vacuum is generated by an Agilent Turbo Bench packaged system comprised of a turbo molecular pump, turbo pump controller with full range gage kit, and a roughing pump. The turbo molecular pump is a 304FS TwisTorr pump with an ISO 100 inlet flange and is backed by an SH110 dry scroll roughing pump. The vacuum system is self-contained with its own control system with a push button interface and digital readout system. The Agilent Turbo Bench vacuum system is initiated manually and has no control interface to the furnace control system other than opening and closing of solenoid valves to the system chamber. Typical vacuum values for non-test castings are usually less than 5×10^{-5} Torr, as measured at the inlet to the turbo molecular pump. While this is not high vacuum, the resulting castings have not shown a negative behavior associated with these vacuum levels. Argon gas is also supplied to the furnace chamber from the facility argon supply system.

2.2 Electrical Control System

The computer control system and graphical user interface (GUI) is based on earlier designs of similar furnaces at INL. The control system software is designed to allow the user to operate the system in two modes—manual and automatic control. The induction power supply can be controlled by temperature control, power level control, or ramp rate control. Using the control system in manual mode, all solenoid valves can be operated from the GUI to either evacuate or backfill the chamber, and the induction power supply can be operated by specifying a desired power level. The automatic mode is programmed in segments that can be gated by different set points of temperature, pressure, and time, or by a combination thereof.

When using the automatic feature in temperature control mode, both chamber pressure and crucible temperature can be controlled. Temperature is controlled based on a single set point, in which the power supply will drive the system to reach the set point as quickly as possible and maintain the temperature for the specified time. In power level control, a specific power level is maintained regardless of temperature. Ramp rate can control the power level to achieve the set temperature rise in a specified time to a set point temperature. In the ramp rate function, a cooling and heating rate can be specified along with a hold time at the set point temperature. The full heating program is made by linking various segments in the program. The system can store five different programs. Each program can utilize up to 20 segments, and each segment can use up to five ramp rates and hold times (for ramp control) or a set of parameters for the segment. The operator can designate which parameter is used to gate to the next segment. If desired, the programs can also be linked to produce a more complex heating profile.

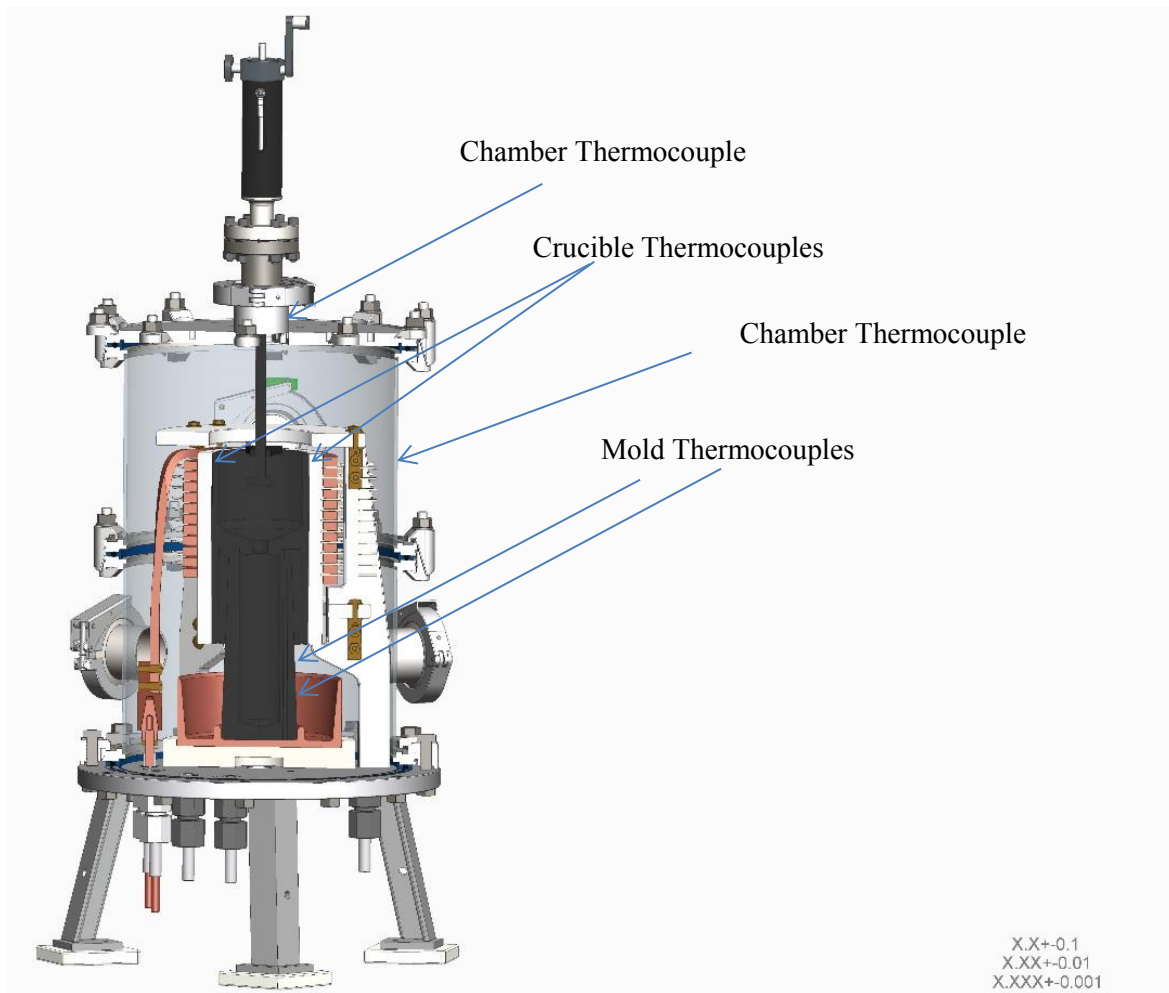


Figure 3. Schematic representation of the billet caster showing approximate thermocouple locations.
NOTE: Schematic shows an earlier version of the billet caster, which included an extended crucible skirt.

The temperatures of the mold, crucible, and furnace chamber are monitored with several thermocouples. The mold and vacuum chamber use K-type thermocouples, whereas the crucible and induction coil use S-type. **Error! Reference source not found.** shows typical locations of the thermocouples. Chamber temperature is monitored to ensure the sealing O-rings are not damaged. The maximum allowable temperature of the chamber is 250°C, at which point an independent over-temperature controller will shut down the furnace induction power supply. The mold temperature is monitored in a midpoint and bottom location to determine the thermal gradient of the mold to the chill basin and the temperature differential between the mold and crucible. The induction coil temperature is monitored to prevent overheating of the non-cooled solid copper coil. Overheating of the induction coil could result in excessive coil deformation, leading to possible electrical shorting of the coil segments, or, in extreme cases, melting of the coil. An administrative limit of 650°C has been placed on the induction coil. Three high-temperature S-type thermocouples are used to monitor the crucible and induction coil. Two thermocouples are located between the refractory and crucible. One of the two is selected in the control software as the “control thermocouple,” which the programmable logic controller will use as the process variable.

It is possible to run the system with only one crucible thermocouple; however, this carries with it an increased risk. The high-temperature crucible thermocouples are fine wire S-type thermocouples, and the

induction coil thermocouple is a sheathed platinum probe type. The induction coil thermocouple is placed at the midpoint of the induction coil between spiral wraps and is electrically isolated from the induction coil by an alumina tube. If a single thermocouple is used on the crucible and it fails during a run, the run will be shut down. Also, there is a risk of the thermocouple providing a faulty temperature reading. An example of this occurred when a single thermocouple was being used; it is assumed that the thermocouple wires touched inside of the ceramic coupler. This provided a reading, but the reading was several hundred degrees lower than the actual crucible temperature.

2.3 Induction Power Supply

Several casting systems at INL have used the Ajax TOCCOtron 5/10 KW power supply. This power supply is an air-cooled, 10-50 kHz, 5-10 KW power supply. Based on the extremely high possible heating rates and the low power required for these other furnace systems, it was decided that the same power supply would be adequate for the billet caster. However, upon early testing it was seen that the required power was more than expected, especially during ramp rate heating to set-point holding temperatures. In an attempt to try to decrease the power levels required, the mold was insulated from the crucible with a 0.25-in.-thick alumina refractory insulation (Zircar Refractory RS-99R). Although this did reduce the power required and decrease the overall mold temperature a little, the power supply was still under-powered. The power supply was further investigated, and it was discovered that the 5/10 KW power supply had a rather low duty cycle above 5KW, which caused the power supply to shut down and not provide the necessary heating. Ajax Tocco was consulted, and based on the amount of graphite, melt mass, desired temperatures/ramp rates, and melting points of the constituents, a larger power supply was recommended. The next larger size that was still air-cooled was the TOCCOtron AC 25 KW power supply with 100% duty cycle at 25 KW power output, with the same frequency range. After changing the power supplies, the system had adequate power to maintain the needed temperature and heating rates in excess of 75°C/min (7 to 9 KW continuous output up to melting/alloying temperature).

2.4 Mold and Crucible

2.4.1 Configuration

The design of the graphite crucible, stopper rod, and mold were based on best practices and configurations for similar type furnaces. The graphite crucible was designed to allow material to flow out of the orifice at the bottom with the removal of a nearly flat-bottomed stopper rod (see Figure 3). The removal was accomplished by a linear motion actuator that grabs the top of the stopper rod and lifts it inside the chamber while maintaining the vacuum. When the crucible is loaded, the plunger also provides a slight positive force to keep the stopper rod in place over the crucible orifice.

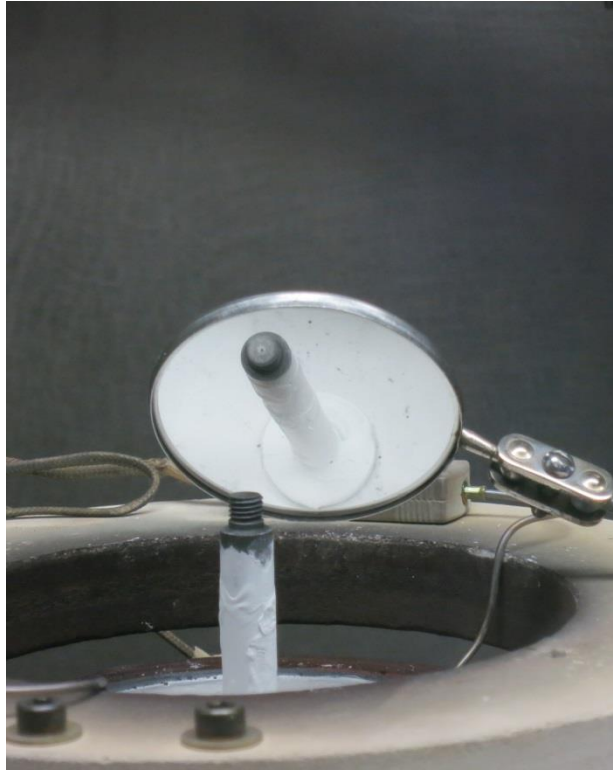


Figure 3. Crucible placed in the casting furnace with the flat-bottomed stopper rod in place.

The graphite mold is a three-piece mold with a collar to keep the two halves together (see Figure 4). The mold has thermocouples located at approximately 1 in. and 3.25 in. from the bottom mold surface, inserted to monitor the temperature during a casting cycle. These two thermocouples provide temperature indications throughout the casting process and indicate when the alloy melt material has left the crucible and has filled the mold. The mold is shaped to reduce the amount of machining needed to prepare the billet for extrusion. The bottom of the mold is formed to closely coincide with the shape of the trailing end of an extrusion billet. The bottom also has a built-in protrusion that is machined off and used for chemical analysis; this is the ICP tip, so named for the Inductively Coupled Plasma Mass Spectrometry analysis used to measure basic alloying elements in the material.

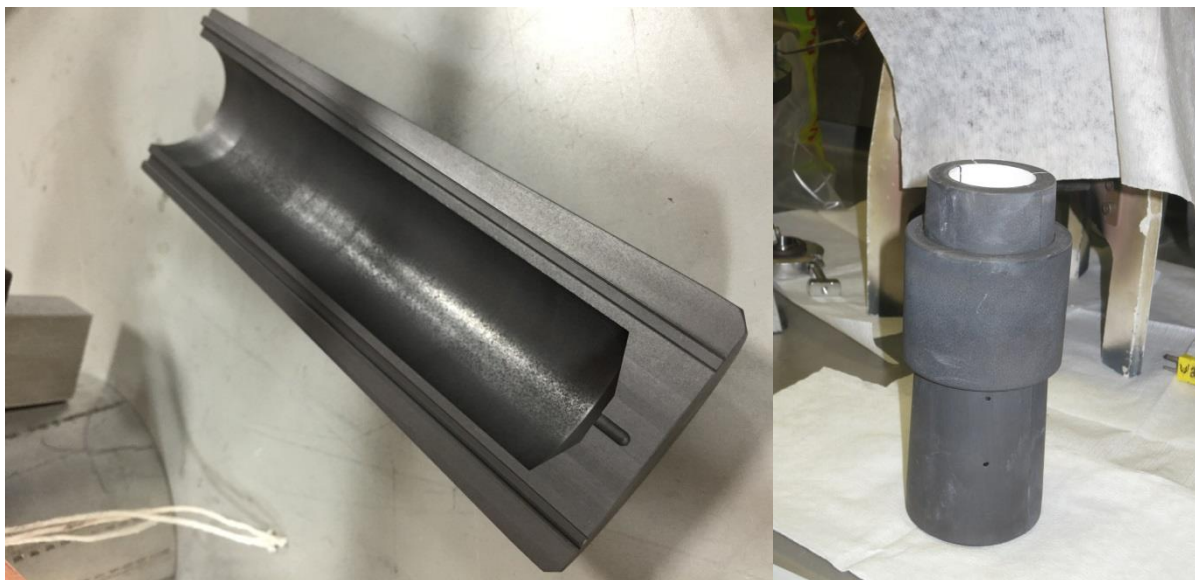


Figure 4. Half of the graphite billet mold with ICP tip at bottom (left). Mold stack assembled (right).

2.4.2 Yttria Coating

The mold, crucible, and stopper rod are coated with a soluble yttria wash prior to being used in the casting furnace. The yttria wash is a pre-mixed Type Y (SKU: 3295) coating sourced from ZYP Coatings and shaken in a turbula prior to application. Proper application of the wash has gone through several iterations to determine the optimum coating. For all applications, the graphite is heated by placing the graphite parts in a small furnace set to $\sim 160^{\circ}\text{C}$ for 20–30 minutes. This ensures adherence of the yttria to the surfaces. In early iterations, the wash was applied with a small paint brush, and just the interior of the mold and crucible were coated. However, the uncoated graphite surfaces of the crucible presented an area where heat loss was experienced during the casting runs. To overcome the heat loss, the entire crucible was coated with the yttria wash. This improved the retention of heat in the crucible. Another reason to coat the outside of the crucible was the possibility of the graphite interacting with the insulating parts of the induction coil assembly; the yttria would prevent that interaction. This was suspected based on knowledge of this and other systems, but not definitively proven.

The paint brush method did not provide a uniform coating, and it took several minutes to coat each piece as it went into and out of the furnace several times to get the coating completed. An alternative application method was a rinse application that was done by pouring some of the mold wash into the crucible and swirling it around. This again provided uneven coatings. Applying the wash via airbrush provided the best results and is the method used for the bulk of the castings (see Figure 5, Figure 6, and Figure 7). It gave the operator good control of the amount applied and of the evenness of the coating. With the graphite preheated, the coating appears dry during the application. When it starts to appear wet on the surface, the crucible has become too cool for correct application and is returned to the furnace. The process is repeated, and coatings are applied as necessary for full coverage, which may take two or three coatings. Good contrast between the graphite material and the white yttria wash provided good visibility for identifying areas with a thin coating (see Figure 8). No measurement on the thickness of the coating has been taken.



Figure 5. Spray system for yttria coating (left). Mold jig to minimize overspray on the mold pieces (right).

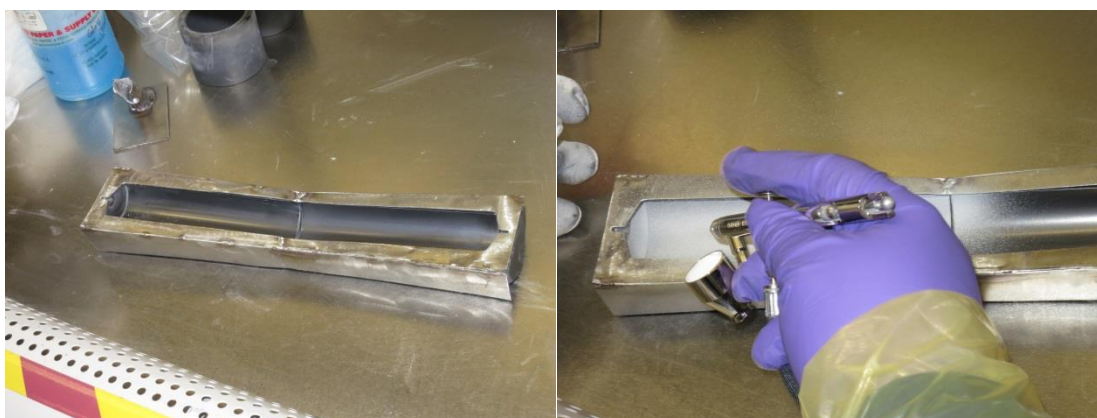


Figure 6. Heated mold in mold jig ready to be coated (left). Spraying yttria wash on heated mold piece (right).

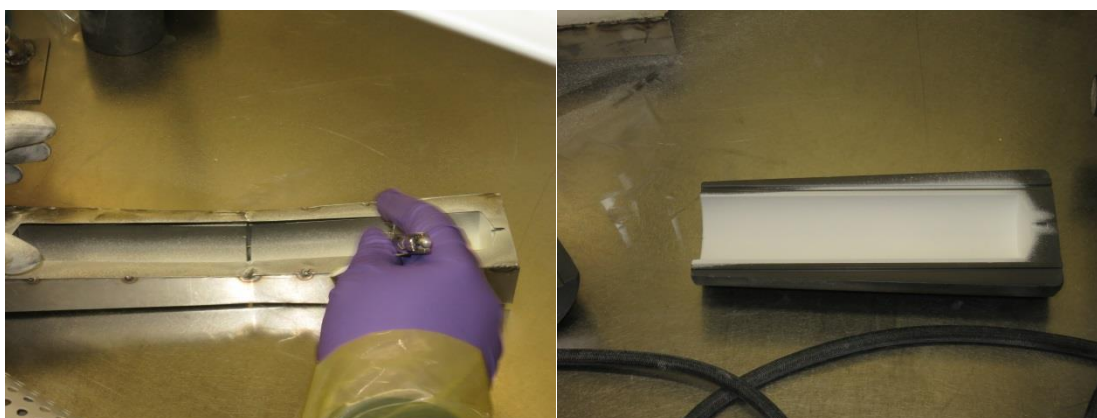


Figure 7. Partially coated heated mold in jig (left). Coated mold piece; note minimal overspray (right).



Figure 8. Crucible being coated; wet area on left side indicates crucible is too cool to coat (left). Partially coated crucible; dark area indicates insufficient coating (right).

2.4.3 Mold and Crucible Cleaning and Recoating

Molds and crucibles are used only once for a casting before requiring cleaning and recoating with yttria wash. Due to radiological concerns, the dross is left in the crucible and removed in a benchtop glove bag. The main concern is keeping the material contained when applying force to remove the dross from the crucible. The applied force may be sufficient to overcome the airflow in a hood and allow material to exit the hood. Some of the casting dross has had high activity, but a short half-life, such that the time period from casting to cleaning is several weeks. This problem has been partially controlled by appropriate DU feedstock material selection, as some sources produced higher dross activity levels than others. Material usually has to be removed by mechanical force, which includes using a screwdriver applied to the dross material through the bottom of the crucible while being lightly tapped by a small hammer (see Figure 9. Images are taken through a glove bag and are somewhat blurry).



Figure 9. Removal of dross from crucible using a screwdriver (left) and hammer (right).

The crucibles are then moved to a fume hood. Molds are held in the fume hood and are not placed in the glove bag. To clean the molds and crucibles, they are first wetted down with water to help control contamination. They are then wiped to remove any loose material or flaking yttria wash. Some alcohol is used as necessary to clean off material not soluble by water. Using wipes, the yttria coating is removed with water and light to moderate pressure. Any coating that is well bonded to the graphite is lightly

scraped off with a delicate wire brush or a small blade screwdriver. Care is taken to not gouge the graphite during this process. Usually, all the coating wipes off without the need for mechanical force. When the crucible is sufficiently clean, it is checked for contamination levels. The molds are cleaned in the same way, but since they aren't subjected to the same heat levels as the crucible they are much easier to clean.

2.4.4 Mold and Crucible Lifetime

Molds and crucibles are examined for defects and wear after each run and changed out when they are no longer viable. Failed castings present a high likelihood of damage from removal. A large amount of material in the crucible makes it more difficult to remove, and a partially cast billet can cause damage to the mold as it is removed (see Figure 10).



Figure 10. Failed casting from 11/9/15 (left) and damage to the mold from the casting (right).

The project manufactured a total of four molds and 23 crucibles to support the casting process. All four molds have been used in development, and 18 of the crucibles have been rotated through the casting process. Losses include one mold and six crucibles. The stopper rods inventory history and lifetime is covered in later sections.

2.4.5 Mold Temperature during Casting

The design of the casting furnace is such that the mold is heated during the alloying process (the heating cycle in which the component feedstock materials are allowed to combine) so that it is preheated prior to the actual casting. The design provides for a thermal gradient from the bottom of the casting to the top. Two K-type thermocouples are used to monitor the temperature of the mold (see Figure 4, right). These thermocouples also provide an indication of a successful casting. See Appendix A for typical mold temperatures during a casting run.

2.5 Stopper Rod Design

The stopper rod worked as initially designed for surrogate material and uranium castings. However, as the project moved to U-Zr castings at higher temperatures, the stopper rod would not lift or separate from the crucible when trying to cast. It was determined that the mold wash was too thick at the interface of the crucible and the nearly flat-bottomed stopper rod. The surface area between the two was very high, and it is believed that the wash was bonding to itself when U-Zr alloys were being cast (see Figure 11).

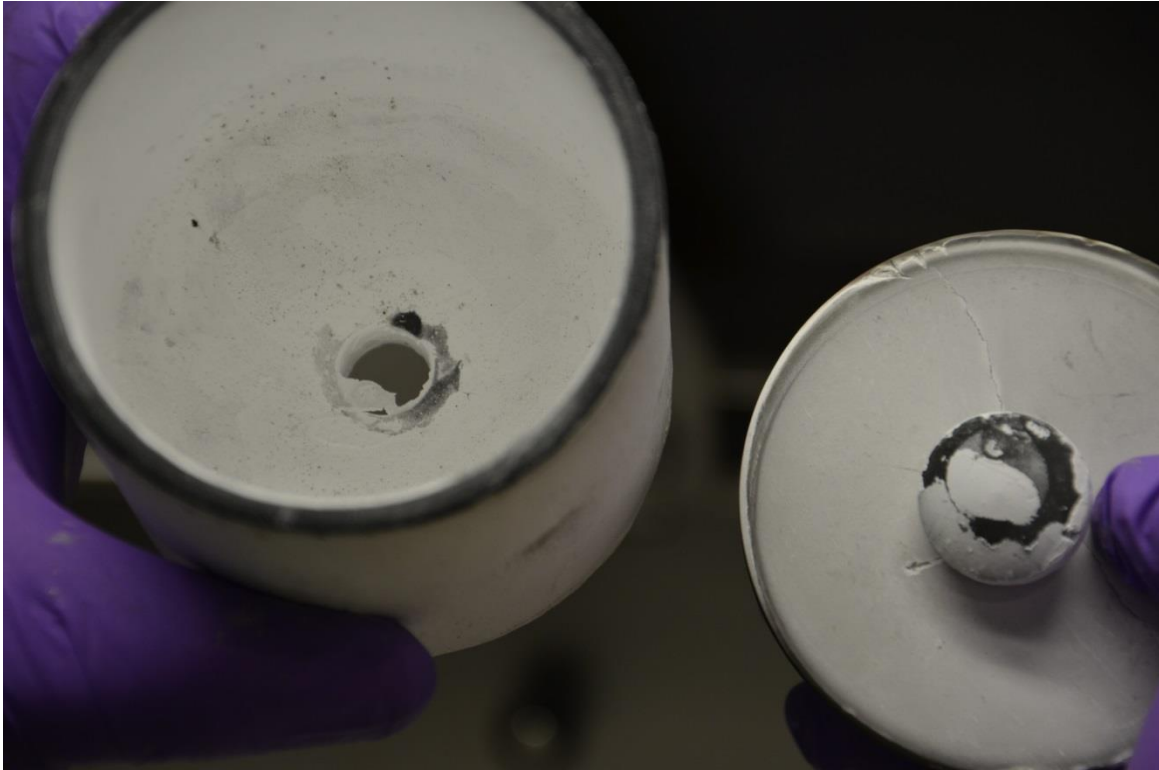


Figure 11. Yttria wash bonding between crucible and stopper rod.

Another factor was that U-Zr alloys require a higher temperature than pure U or Cu, and the higher temperature probably played a role in the bonding as well. It became too risky to end the process with a full crucible that could not be cast; all components may have to be replaced, and the melt sent to waste. To fix the problem, the stopper rod design was changed from nearly flat bottomed to a large radius (see Figure 12). This provided the ability to maintain the melt in the crucible but with a very small contact surface area. After successful initial testing with a thick-bodied stopper rod, the remaining flat-bottomed stopper rods were machined to the same larger radius (see Figure 13). This repurposed several stopper rods.

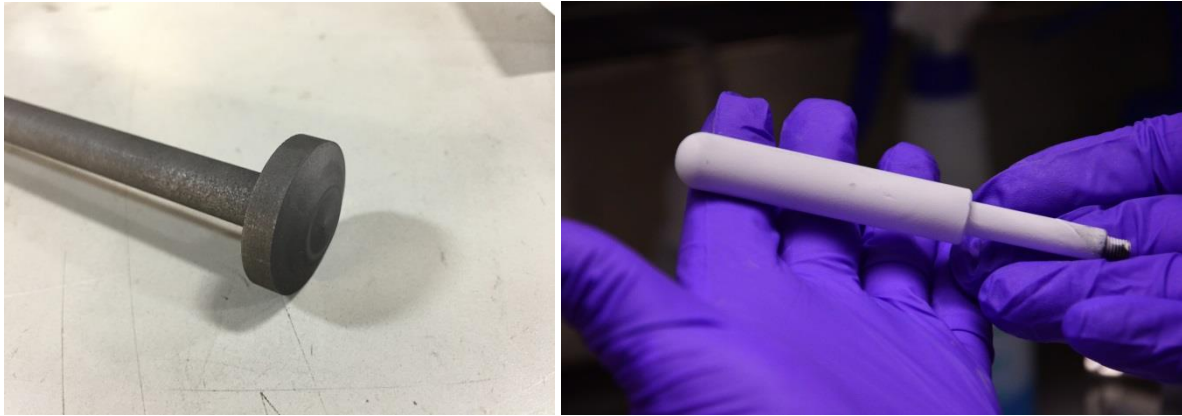


Figure 12. Initial design of nearly flat-bottomed stopper rod (left). Modified stopper rod design with larger radius (right).

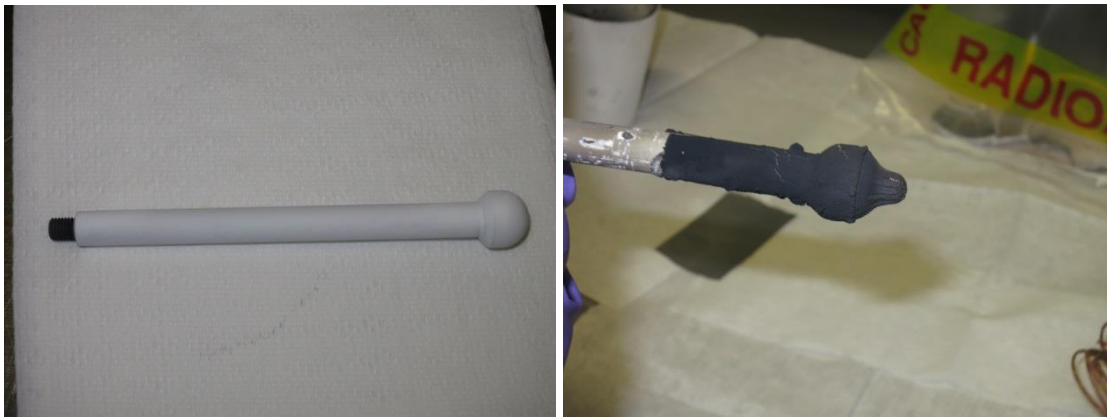


Figure 13. Final design of stopper rod (left). Used stopper rod after a casting (right).

The graphite stopper rods can easily be broken, and the threaded area is the most susceptible to breaking. In the early casting runs, some stopper rods were broken during the disassembly of the casting furnace. As operators have gained practice in removing the stopper rod from the plunger assembly, the number of broken rods has decreased to just those that are broken during the normal casting process. The process that is most likely to cause a stopper rod to break is the act of casting. During casting the plunger assembly grabs the lava hat to raise the stopper rod and allow the melt to flow out of the crucible into the mold. As described above, there can be sufficient bonding between the crucible and stopper rod to prevent the stopper rod from lifting, causing the threads of the stopper rod or the threaded area of the lava hat to break (see Figure 14). The occurrences of such a breakage are rare, especially since the design of the bottom of the stopper rod has changed.

The graphite crucibles and molds are reused after each casting. The stopper rods are made of the same graphite material, but because of the time spent in the melt, they do not lend themselves to being cleaned and reused. Cleaning the stopper rod has been attempted and yielded a success rate of 3 out of 10. The low success rate is due to the difficulty of removing the dross from the brittle graphite rod. Also, while cleaning the rods has been successful, it does appear to weaken the stopper rod. One reused stopper rod was used in a casting, and during the run, the threads between the rod and the lava hat broke, resulting in

a failure to cast. It was determined that the stopper rods should be designated consumables, as it is too risky to attempt casting with a questionable stopper rod.

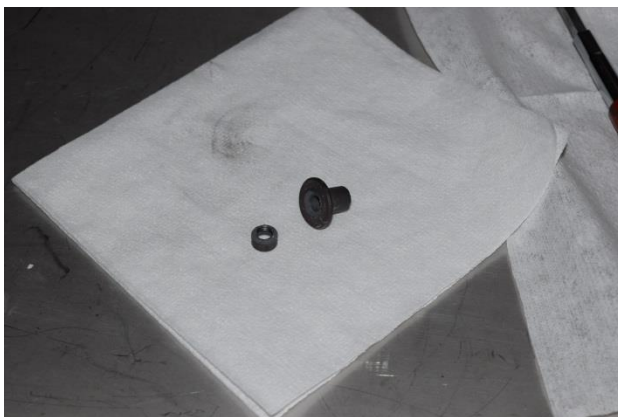


Figure 14. Broken lava hat from casting on 8/6/15.

3. CASTING PROCESS

3.1 Insulation and Heat Loss

One of the early difficulties was the amount of heat loss in and around the crucible during the melting process. In some castings, U and/or Zr material was left undissolved in the crucible. One solution, painting the entire crucible with the yttria wash, was mentioned earlier. Another included adding a refractory lid on top of the crucible. These helped contain the heat in the crucible and kept it from radiating to the chamber, where the gaskets could be damaged if the temperature was high. An increase in hold time at temperature was also a benefit. The lid is susceptible to cracking and breaking during the heat cycles of casting and is replaced when indications of cracks are present.

The top of the crucible is inside the refractory stand and induction coil. The lid protrudes about $\frac{1}{4}$ of an inch above the refractory. This area also presented high heat loss, and a refractory ring was fabricated to surround the protruding section of the crucible.

The crucible was designed to rest on top of the mold. The contact of the coated crucible to the mold presented an area of high heat loss from the crucible. An insulation ring was added to rest on the mold collar and surround the mold up to the contact point of the crucible (see Figure 15). This reduces the amount of heat transferred to the mold, but sufficient heating of the mold is still achieved. The crucible would then rest on top with the stopper rod and lid in place (see Figure 16).



Figure 15. Mold stack with insulation around top of mold (left).
RS-99R spacer added on mold stack (right).



Figure 16. Crucible added on mold stack (left). Stopper rod and lid added on mold stack (right).

3.2 Bake Out

“Baking out” components to remove moisture and/or impurities is a standard practice used in heated vacuum chambers. A bake-out process was used for new parts and components of the EFF furnace as it was assembled. Generally, the new components were heated to 1,000°C in a separate furnace for at least four and up to 24 hours prior to being assembled in the vacuum chamber.

The molds, crucibles, and stopper rods were designed to be removed from the furnace and reused. Each is cleaned of the existing yttria coating and any other residual material. The items are then heated and re-coated with yttria wash. Finally, the components are put back in place in the casting furnace and heated using a bake-out recipe programmed to drive off any residual moisture, which could cause oxidation of the U and U alloy materials. Table 1. Typical program for EFF Casting Furnace bake out (temperatures are in °C). Table 1 has a representative bake-out heating program.

Table 1. Typical program for EFF Casting Furnace bake out (temperatures are in °C).

	Set point (deg)	Ramp (deg/min)	Hold time (min)	Action
Bake-out Seg 1	500	50	5	Next segment
Bake-out Seg 2	1,200	75	30	Off

Initially, all casting setups (mold, crucible, and stopper rod) are baked out prior to using them in the furnace. Each was again baked out after coating with yttria wash by running a bake-out program with the casting furnace. However, as the castings moved to U-bearing charges, it was found that the yttria wash was friable and not bonded well to the stopper rods after exposure to the high heat (see Figure 17).



Figure 17. Stopper rod after bake out and attempt to load fuel in crucible.

Thus, the bake out allowed flaking of the wash from the stopper rod, and as U and/or alloy components were loaded into the crucible after exposure to the heat, the coating would come off the

stopper rod. This presented the possibility of exposing the U melt to a graphite surface, which could cause carbide creation at high temperatures. An unbaked, as-coated stopper was, however, found to be able to withstand the rigor of loading material without flaking, so the stopper rod was no longer subjected to a bake out.

As casting and extrusion development progressed, the demand for the casting furnace to produce usable billets for extrusion grew to higher throughputs, and the bake-out process was re-examined. Test castings were performed to determine the changes to a casting if the coated items were not subjected to a bake out each time they were coated with mold wash. While the duration of a bake out was relatively short, the amount of time needed for it to cool sufficiently to reduce the elevated temperature hazard to the operator was high. The standard practice was that an empty furnace would be put through a bake out first thing on the morning of Day 1 and would cool then be allowed to cool. At the end of Day 1, the furnace could be cool enough to open and load the charge for the casting. The chamber would then need to pump down to acceptable levels and would generally be left overnight. On Day 2, the furnace would then go through the casting process. The morning of Day 3, the chamber would be cool enough to open, remove the cast billet, change out the mold and crucible, and begin to pump down to perform the next bake out. This standard process allowed for a maximum output of two billets per week at best.

The concern regarding elimination of the bake-out step was the amount of water remaining in the yttria wash and whether that would increase the oxidation of the U material to an unacceptable level, or if it was sufficient to cause changes to the cast billet. There was not a discernable change in the amount of oxidized material remaining in the crucible when the stopper rod was no longer a part of the bake-out process, so the risk appeared to be low. A few castings were performed without a bake out, and no discernable changes in billet properties or oxidation levels were found. The throughput of the furnace can double by omitting the bake out. The furnace is opened and loaded in the morning, the cast is made in the afternoon, and it is allowed to cool overnight, making four castings a week possible. The limit then becomes having a mold, crucible, stopper rod, and charge ready and available to load. However, new molds and crucibles are still baked out prior to their first use to remove any impurities and residual moisture from the fabrication process. Molds and crucibles are stored in sealed poly bags to prevent any re-absorption of moisture.

3.3 Uranium Form

While the eventual goal is to cast uranium-bearing fuel of different enrichments, the only type of uranium that has been cast in the EFF Furnace has been depleted uranium (DU). Initially, a stock of 1×1×3” pieces was used (see Figure 18). These pieces had an outside oxidation layer that was not removed prior to casting. Occasionally, a shell of one or more of the DU pieces was observed in the crucible after the casting (see Figure 19). One of the difficulties with this form was that it was too large to place over Zr and still fit within the crucible. Some of the blocks were cut to smaller 1×1×2” and 1×1×1” pieces. While this helped with the loading of the crucible, it was still not ideal.



Figure 18. Image of DU feedstock used for initial casting on 9/10/15.



Figure 19. Image of DU shells (rectangular pieces) and Zr turnings (curved pieces) remaining in crucible after melt on 9/21/15.

A different source was identified that had sufficient volume for use in the casting furnace. The DU is in rod form and is sheared into $\frac{3}{4}$ " slugs (see Figure 20). This geometry allows for a high packing factor in the crucible and completely covers all of the Zr alloying addition at the bottom of the crucible. The material is wiped clean using alcohol when it is sheared, and no other preparation is done prior to casting.



Figure 20. Image of sheared DU feedstock loaded in crucible prior to melt on 2/7/17.

3.4 Alloy Addition Forms

Zirconium has been the most prominently used alloy addition in the casting process. The forms of Zr material used in casting have varied. Initial casting runs used Zr turnings filled in around the large $1 \times 1 \times 3$ DU pieces (see Figure 21). The turnings facilitated obtaining the desired mass easily due to their low mass and varied sizes. However, their high volume made it difficult to ensure that U material was able to melt over the charge completely. Note that uranium melts at a lower temperature ($1,132^{\circ}\text{C}$) than zirconium ($1,852^{\circ}\text{C}$) and a maximum casting temperature of $1,500$ – $1,550^{\circ}\text{C}$ is only possible by letting the uranium melt over the lower-density zirconium to dissolve it. A few turnings were observed to be nearly whole and un-melted after some castings (see Figure 19). Zirconium foil (0.010 – 0.040 " thick) was used in one casting but this option was abandoned. It was a poor option not only because of the cost of the thin foil compared with other forms, but because of the manual labor needed to prepare it for use in the crucible (see Figure 22). Sheared Zr chips (approximately $0.75 \times 0.75 \times 0.125$ ") were obtained and provided a balance between flexibility in obtaining correct mass values and the high volume the turnings presented (see Figure 23). Options that were discussed but have not yet been used include Zr rods and Zr wire. All of the forms of Zr have been used as-is without any additional cleaning process.



Figure 21. Zr turnings with $1\times 1\times 3$ DU pieces (left).
Zr turnings and DU loaded in crucible (right) from casting run on 11/9/15.

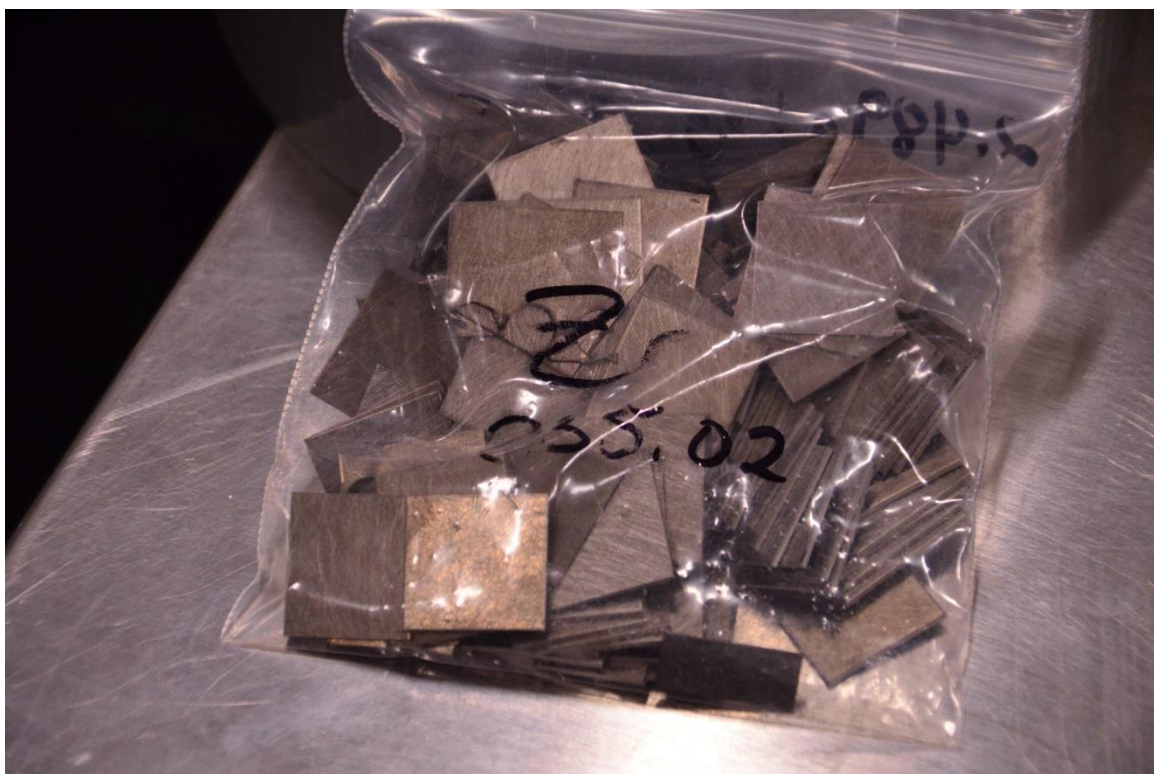


Figure 22. Image of sheared Zr foil used in casting on 12/14/15.



Figure 23. Sheared Zr chips (not from a casting).

3.5 Melting and Alloying

3.5.1 Copper and Copper Alloys

The system was used to cast Cu and Cu nickel billets to practice and discover any issues with programming or components. The first casting was done as the system was originally configured with Cu slugs as the feedstock. While some of the material did cast, it was found that the heat loss at the top of the crucible was high, and not all of the slugs were hot enough to melt. The remaining material solidified around the stopper rod. The billet was cut open to examine the solidification of the material (see Figure 24).



Figure 24. Cu feedstock from run on 4/2/15 melted around the stopper rod and the short cast billet resting in the mold (left). Cast Cu billet sectioned in half revealing low density Cu (right).

The material did not solidify well and left a collection of voids in the billet. The hold time for this run was only 15 minutes, which may not have allowed sufficient time for the entire charge to melt. From this data it was determined to run DU and DU alloy casts at a hold time of 45 minutes. A lid was fabricated of RS-99R to go on top of the crucible to retain more heat in the crucible. The next copper casting melted all of the feedstock and cast a billet with a large shrinkage void in the center of the billet (see Figure 25). When the billet was machined, there were some surface voids on the outside of the casting (see Figure 26). Other runs were completed with Cu and Cu-Ni castings, and the changes listed above improved the castings.



Figure 25. Cast copper billet from 4/9/15 with large shrinkage void down the center of the billet.



Figure 26. Machined billet with surface voids (left). The extent of the shrinkage void (right).

3.5.2 Uranium

The initial DU billet casting turned out fairly well. The casting had some dark discoloration at the top, but other than that, the appearance was good.



Figure 27. Initial DU casting from 8/6/15.

3.5.3 Uranium Alloys

Alloying accuracy and repeatability is important to the TerraPower project. While actual limits have not yet been set in a fuel specification, the assumed functional loading target for zirconium alloying has been set at $\pm 0.05\%$, half that of production for EBR-II driver fuels. For a target of a U 10wt% Zr alloy, this represents approximately a 3 g tolerance window of Zr mass for a 2.5 kg charge of U. Obtaining a measured mass of Zr material to fit within the tolerance window has not been a challenge. The challenge has been getting all of the alloying additions to sufficiently go into solution in the uranium melt. Zirconium has a melting temperature of 1,855°C, well above the temperature range that the furnace is equipped for (around 1,500°C). However, the phase diagram of U and Zr shows that for alloys up to 20wt% Zr, 1,500°C is sufficient to achieve a full liquid solution. The casting program (see Table 2) is designed to provide sufficient time at temperature for the Zr to go into solution with the uranium. Zirconium material is placed at the bottom of the crucible, and the U is placed above it. This allows the U to melt around the Zr and disperse through the solution (note that the Zr is less dense than U so it may float in the melt).

Table 2. Initial casting program for EFF Casting Furnace (temperatures are in °C).

	Set point (deg)	Ramp (deg/min)	Hold time (min)	Action
Casting Segment 1	500	50	5	Next Segment
Casting Segment 2	1,550	50	0	Next Segment
Casting Segment 3	1,500	0	43	Cast

Billets are sampled from the bottom and top of the castings. Samples can be examined via ICP methods at the Analytical Laboratory (AL) at INL. Several DU-6Zr samples were run, but the results were not as consistent as desired. An issue of mass balance showed the material to be larger than the sample was at the start of the examination. Since these issues cast doubt on the results, and since the material was DU-6Zr (a stepping stone alloy to get to DU-10Zr), the results will not be reported here.

To examine DU-10Zr material, a billet was selected and pieces from that billet were sent off-site to be examined via Inductively Coupled Plasma Mass Spectrometry (ICP MS). The results are listed below (see Figure 28). For this examination the bottom tip (location 0'), one of the top ICP samples (see Figure 29), and three samples from the extruded billet (leading end, center, and trailing end) were examined for uranium and zirconium content. The top of the casting is extruded first, so the LE piece is from the top of the casting, and the TR piece is from the bottom. The three extrusion samples were full diameter pieces that were each about 2 mm thick. The billet examined was targeted to be a DU-10wt%Zr.

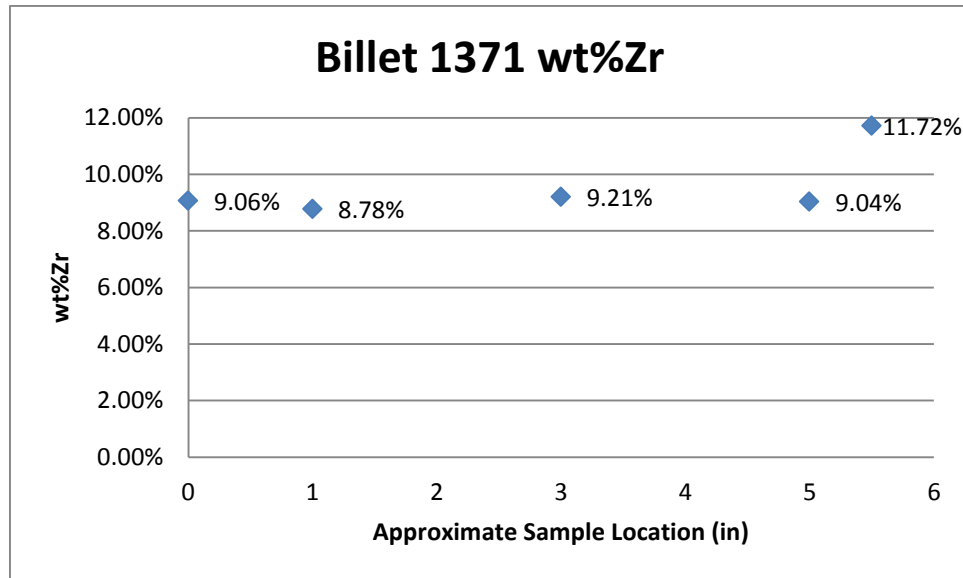


Figure 28. ICP results from billet 1371.

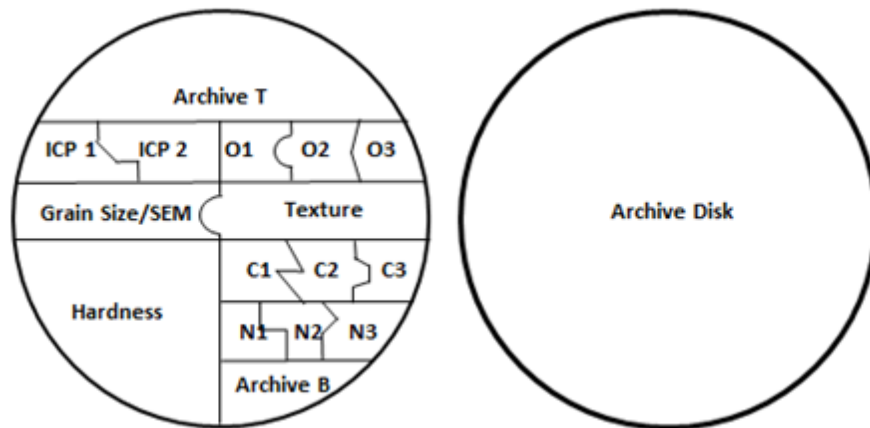


Figure 29. Billet sampling diagram; each disk is about 0.050" thick.

While the change in Zr form from turnings to chips has helped with the intended result of having U melt over Zr to allow it to go into solution, there are times when there are still some Zr remnants left in the crucible after the casting, as seen in Figure 30. It is unknown if this material is submerged under the melt during the casting but doesn't go into solution, or if it floats on top of or near the top of the melt where it is much less likely to alloy. Not all melts show the leftover Zr, and the amount of remaining material varies from casting to casting. An ICP examination on a 1.5 g piece of dross material found that it contained up to 17wt% Zr as opposed to the target 10wt%. The higher concentration of Zr material would explain the lower than desired results for the billet.

With a 6% average charge loss (see Section 3.8 for further discussion) to casting dross that is 17% Zr, we can see that the Zr content in the billet should be about 9.4%, which is not as low as the actual results displayed above. However, the top of the billet has a reported elevated Zr content as well, which might further explain the Zr content issues. Further examination of ICP results on billets would be well advised.



Figure 30. Possible remaining Zr (metallic item at 5 o'clock position) and Zr husk (rectangular, rusty item directly above metallic item) after casting on 4/24/17.

An attempt was made to develop a method to stir the melt and therefore increase the likelihood of all the alloy additions going into solution. The method included using variation in the power applied to the coil while the melt was in the crucible. A window was installed on the lid of the furnace and a bismuth melt was run. Different power levels were applied to the coil, starting with low powers (1 kW), and stepping up to higher levels (10 kW). With the application of lower powers (3 kW), ripples in the melt were visible. At higher powers (>5 kW), the melt would still ripple, but it would also “lift” or arch in the crucible. A new program was added to the casting furnace software and attempted with DU (see Table 3). No discernable improvements were seen with the pulse program, but several internal logic issues were found in trying to run the new program, and it never ran in fully automatic. The initial casting program in Table 2 was reinstated.

Table 3. Pulse casting program for EFF Casting Furnace (temperatures are in °C).

	Set point (deg)	Ramp (deg/min)	Hold time (min)	Power level (kW)	Action
Segment 1	500	50	5	Auto	Next segment
Segment 2	1,550	50	0	Auto	Next segment
Segment 3	1,500	-	10	Auto	Next segment
Segment 4	1,500	-	-	Auto	Next segment
Segment 5	1,475	-	-	0	Next segment
Segment 6	1,550	-	-	6	Next segment
Segment 7	1,475	-	-	0	Next segment
Segment 8	1,500	-	-	6	Next segment

Segment 9	1,500	-	10	Auto	Next segment
Segment 10	1,500	-	-	6	Next segment
Segment 11	1,475	-	-	0	Next segment
Segment 12	1,550	-	-	6	Next segment
Segment 13	1,475	-	-	0	Next segment
Segment 14	1,550	-	-	6	Next segment
Segment 15	1,500	-	10	Auto	Next segment
Segment 16	1,550	-	-	6	Next segment
Segment 17	1,475	-	-	0	Next segment
Segment 18	1,550	-	-	6	Next segment
Segment 19	1,475	-	-	0	Next segment
Segment 20	1,500	-	10	Auto	Next segment

3.6 Casting Results

Overall casting results have been as desired. Cast billets have had minor issues or variations that will be discussed in depth, but the majority have run without issue (see Figure 31). Most notably, there have been no major casting voids or inclusions as were seen in the copper billets. Casting voids would cause problems for density measurements and would cause issues for the extrusion process.



Figure 31. Cast billet from 2/4/16.



Figure 32. Cast billet from 12/14/15 with good casting results.

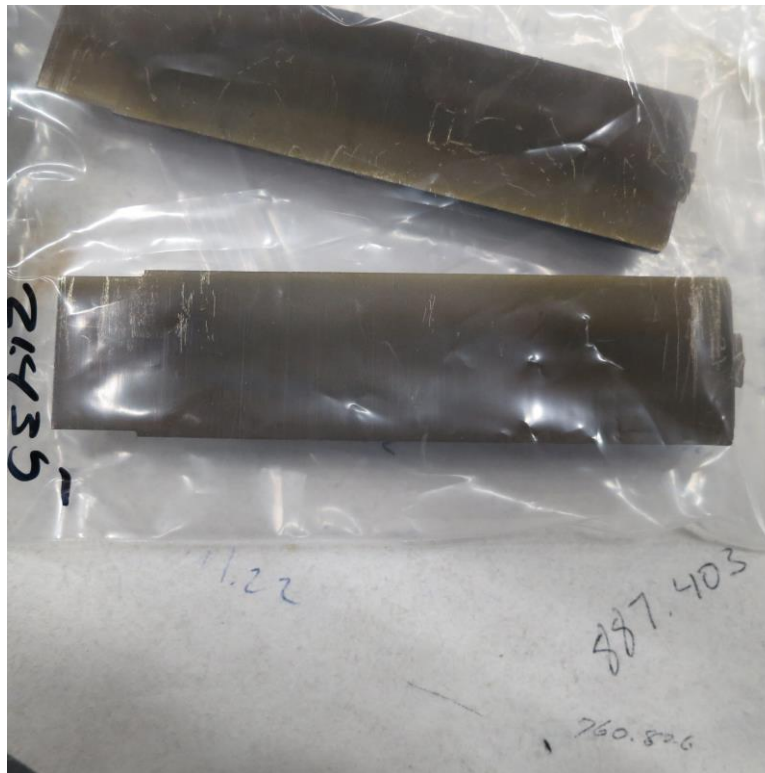


Figure 33. DU-10Zr billet cut cross section showing no voids nor inclusions.

The dimensions of the billets have been consistent overall. There has been some variation, but it is very small. Measured diameter dimensions were analyzed on a sample of twenty DU-10Zr billets that were cast into a mold with a diameter of 1.50". Measurements were taken at the top, middle, and bottom of the castings, then taken again as each was rotated 90 degrees. The overall average was 1.454" with a standard deviation of 0.007". The top of the casting averaged 1.451" with a deviation of 0.009". The minimum diameter measurement was 1.428", and the maximum was 1.469". Examining the data over time shows no discernable trend in growth that would indicate the size of the mold has changed over time.

In most billets there is a diameter variation from heat shrinkage that results in a necking effect on the top end of the billets, which correlates to the diameter variation above (see Figure 34 and Figure 35). These heat shrinkage features are fairly consistent in shape, size, and location. They are generally about 0.025" deep, or three times the standard deviation for the DU-10Zr billets. The shrinkage isn't severe enough to remain after machining. The depth is still small enough that when the billet is machined down to 1.350" for extrusion, there is no un-machined surface from the shrinkage. However, this shrinkage could affect the useable length of a billet that is intended to be used in the as-cast condition.



Figure 34. Billet cast on 5/22/17 showing a shrinkage void almost all the way around the top diameter.



Figure 35. Billet cast on 4/24/17 showing a shrinkage void on one side of the top of the billet.

The surface finish of the bottom of the casting has generally not been a concern as the process of machining the billet cleans any porosity or defects from the casting. But with the movement toward extruding an as-cast billet or just reducing the amount of machining waste from a billet, a change was proposed for the bottom of the mold. During the process of coating the mold with yttria wash, the bottom of the mold is cleaned of any yttria. This was thought to increase the rate of cooling from the copper cooling plate through the mold, to help the billet solidify from the bottom. The billets cast in this manner displayed small voids in the bottom of the casting (see Figure 36). It is believed that the exposed graphite was reacting or interacting with the cast material and causing the voids.

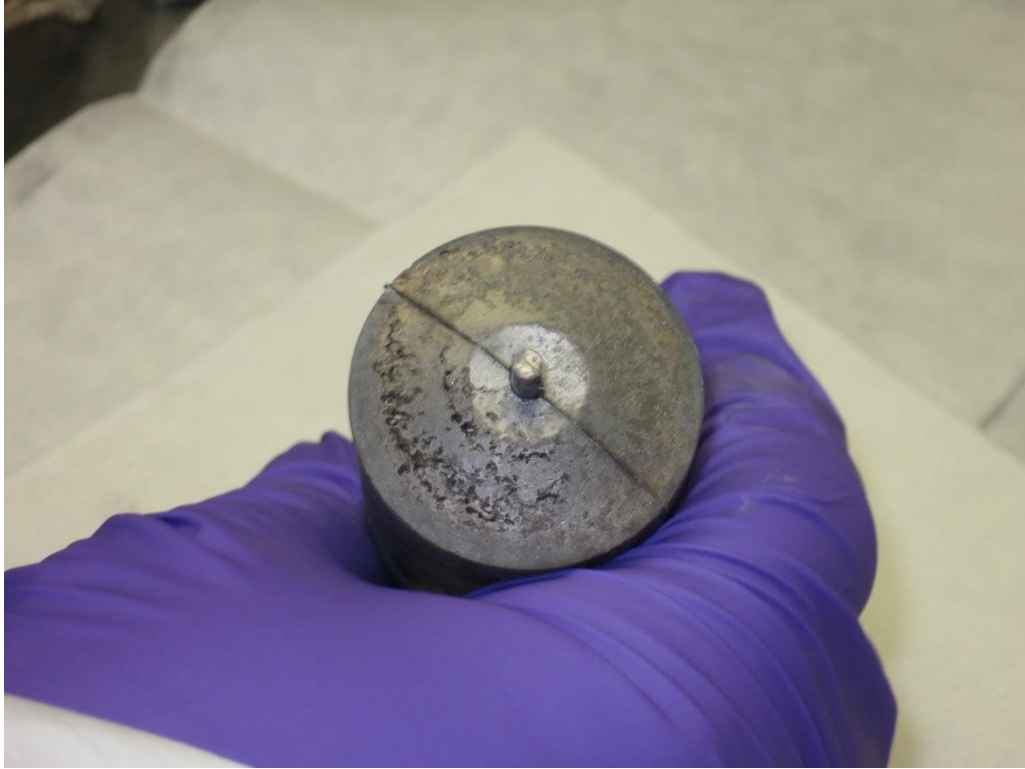


Figure 36. Bottom of billet cast on 4/24/17 showing surface voids from the bottom of the casting.

After a discussion with the subject matter experts, a casting was performed with DU-10Zr with the bottom coated like the rest of the mold. The result was a much cleaner surface finish on the bottom of the casting with no noticeable effect on its geometry (see Figure 37). The accepted practice is now to coat the bottom of the mold.



Figure 37. Bottom of billet cast on 07/19/17 showing no surface voids on the bottom of the casting.

Cast billets also present color variation on the casting. The most common variation is dark discoloration at the bottom of the casting; this is present on virtually all castings. It is most likely caused by the heated melt hitting the cooler bottom of the mold. The mold does see some inductive heating from the coil and from being in close contact (1/4" insulation separation) with the crucible. This thermal gradient is desired for solidification of the casting from the bottom to the top. It may be that the bottom of the mold is too cool and the thermal shock from the molten material is too great. This may cause the yttria coating to break down or impart some of the graphite from the mold to the billet.

Another color variation is a tarnish or discoloration at the top of the casting. It is unknown what the cause of the discoloration is (see Figure 38).



Figure 38. DU-10Zr billet cast on 5/23/17 showing little to no color variation (left). DU-10Zr billet cast on 5/4/17 showing a high amount of color variation (right).

Another feature that varies from casting to casting is material that is not part of the melt, but falls on top of the cast billet. This material is present on most of the billets to some degree. The material is not associated with the discoloration, as billets with the globs of material on top are discolored while others aren't, and billets without the material show discoloration and normal billet color (see Figure 34, Figure 35, and Figure 38). The material is most likely from the slag floating on top of the melt, which does not see the same heat as the bulk of the melt. It is possible that when the stopper rod is lifted and the bulk of the material is cast, the remaining material moves down in the crucible and is subjected to the more direct heating zone. The material then melts and slowly deposits on the top of the casting. With the stopper rod removed, the material would be free to drip as it melts.

There seems to be a direct link between the magnitude of the shrinkage void and the presence of material that drips on top of the casting. This is presumably from material continuing to melt after the bulk of the casting material has already dropped, but at a lower temperature, therefore drawing heat out of the top of the casting. Castings that have a high amount of drippings have a more pronounced shrinkage void. The casting from 8/1/17 contained fresh and previously cast material. The run yielded a non-typical casting slag that did not fall during the casting, and therefore would not have seen the increase in heat. The top of this billet has no dripping material on it at all and has a minimal shrinkage void (see Figure 39).



Figure 39. Non-typical dross material in crucible from billet cast 8/1/17 that did not fall in the crucible (left). Cast billet from 8/1/17 with little to no shrinkage void (right).

One possible way to fix this is to turn off the power immediately after casting. The current program gives two minutes for the cast to occur. It is set to hold for 45 minutes, and the operator casts at 43 minutes. This time difference is designed to give the operator time to attempt to recover from an issue, like a stuck stopper rod, and still be able to cast. When the casting happens on time, it leaves the dross in the heat zone for up to two minutes. Turning off the power immediately after casting and not letting the program continue should reduce the amount of material on top of the cast billet.

3.7 Density

The theoretical density of DU-10wt%Zr is about 16.16 to 16.25 g/cc. The results of the measured density of several cast billets are shown in Figure 40, along with the average. Most of the castings track very close to each other, and the average is within the theoretical range. One outlier is present in the graph for ID-1388. It is not known if this result is from an actual density deviation or if it is an error.

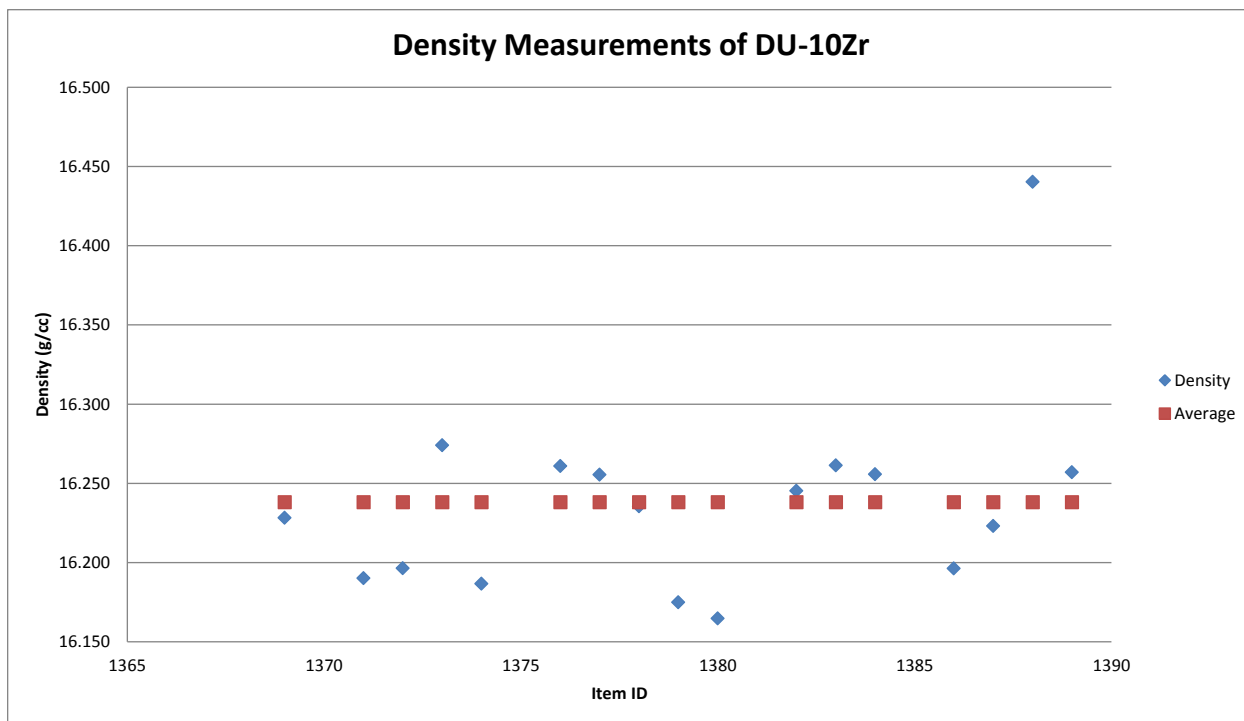


Figure 40. Density data of cast billets.

3.8 Mass Balances

The castings do exhibit some process loss from melting and alloying. Impurities and slag from the materials are left in the crucible and separated from the billet (see Figure 41). The average charge and process loss for each type of casting is listed in Table 4, along with the standard deviations. While there isn't a lot of information for pure uranium and low Zr percentages, the percentages of loss generally increase with an increase in alloying components.



Figure 41. Bottom of casting dross (left), top of casting dross (right) from casting on 11/24/15.

Table 4. List of average charges, process loss, and variation of each type of casting.

Casting Type	Average Charge	Std. Deviation	Average Loss	Std. Deviation	# of Castings
DU	2626.960	N/A	3.76%	N/A	1
DU-05Fe-05Si	2694.094	40.739	3.67%	0.10%	2
DU-2Zr	2633.79	N/A	3.12%	N/A	1
DU-4Zr	2686.93	N/A	3.23%	N/A	1
DU-6Zr	2315.776	282.347	4.80%	1.47%	27
DU-10Zr	2423.849	44.520	5.67%	1.38%	29

The charges of the DU-6Zr varied quite a bit more than the other fuel types. Some of this variation is from changing DU sources. The Aerojet material was in large blocks that didn't allow for discrete mass selection. Single pieces could range from 250–400 g each. The EBR II feedstock material came from already fabricated DU rods that were sectioned. These had little to no variation in mass. As such, the DU-10Zr batches were more consistent than the DU-6Zr charges.

4. POSSIBLE CASTING IMPROVEMENTS

Positive pressure stopper rod – The stopper rod has been known to stick to the crucible, even with the improvements to the design and a reduction of the contacting interface between the two. The current stopper rod is designed to lift up from the lid of the chamber. Designing a system that prevents the crucible from lifting during casting would increase the number of successful casts.

Casting furnace window – The placement of the window in the casting furnace as designed accomplishes two goals: (1) it allows the operator to see the teeth of the stopper rod lifting mechanism and verify that it has a hold on the stopper rod, and (2) it provides visibility into the system for observation of the process. The visibility obtained for both is poor. A location change or addition of another window would help to reduce the strain the operator encounters when manipulating the system when it is closed. Moving the window higher would give better visibility into the system to observe any issues, such as a broken stopper rod, or whether or not the stopper rod is lifting with the plunger at the top.

Stopper rod and plunger design – In addition to having positive control of the separation of the stopper rod and crucible, it would also be beneficial to change the material of the plunger and re-design the stopper rod into one piece. The stopper rod and lava hat help to isolate the stopper rod plunger from the frequency present in the chamber, but the threads present a focal point for stress and make it easier to break. Changing the stopper rod to a solid graphite piece that includes the shape of the current stopper rod and lava hat together and making the teeth of the plunger out of the same material as the lava hat would accomplish the same objective of isolation, but would eliminate the weak fracture location. The stopper rod and plunger system could also be designed to rotate to provide a stirring motion during the alloying process. This would give the operator control to attempt to improve homogeneity in the cast billet.

Inlets/outlets location in chamber – The location of the several inlets/outlets in the system are not ideal, specifically the location of the Ar inlet on the back side of the furnace. Not only is it cumbersome to reach behind the chamber to hook up and release each time it is opened, it doesn't provide any flow over the heated portion of the system if it were used. Relocating it opposite the vacuum outlet would be preferred.

Insulation in the top of the chamber – The top of the chamber gets heated during the casting run and has a thermocouple that monitors how hot it gets. This is a control thermocouple; if the temperature reaches 250°C, it stops the program to protect the seals. A few runs have been cut short because of this

limit. While it is not linked to any specific defect, it is preferable for the program to run through fully prior to casting. Insulation on the lid or increasing the distance would help to reduce the amount of heat it receives from the casting. Insulation is preferred over increasing the size of the casting furnace, as it keeps contaminants lower and does not increase the mass of the vessel. Increasing the mass of the vessel may necessitate installation of a lifting assist mechanism.

Appendix A: Thermal Profile of Example U-Zr Casting Runs

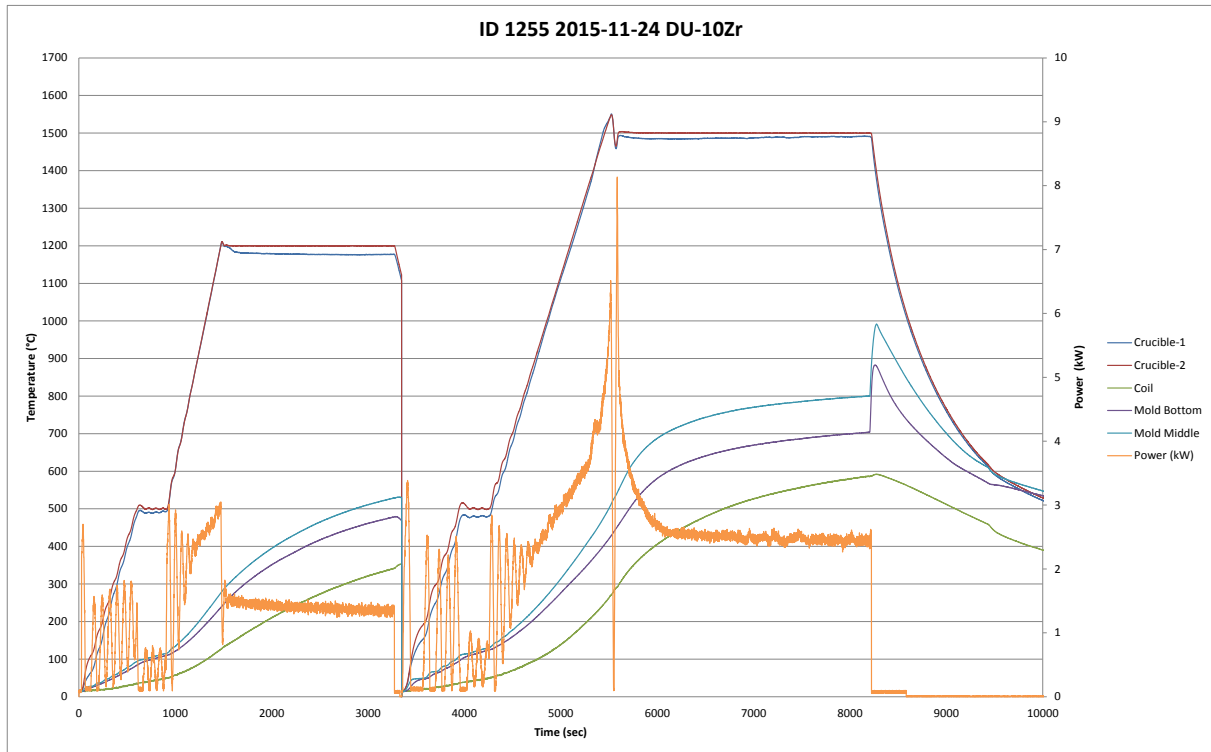


Figure A-1. A typical non-pulsing DU-10Zr run.

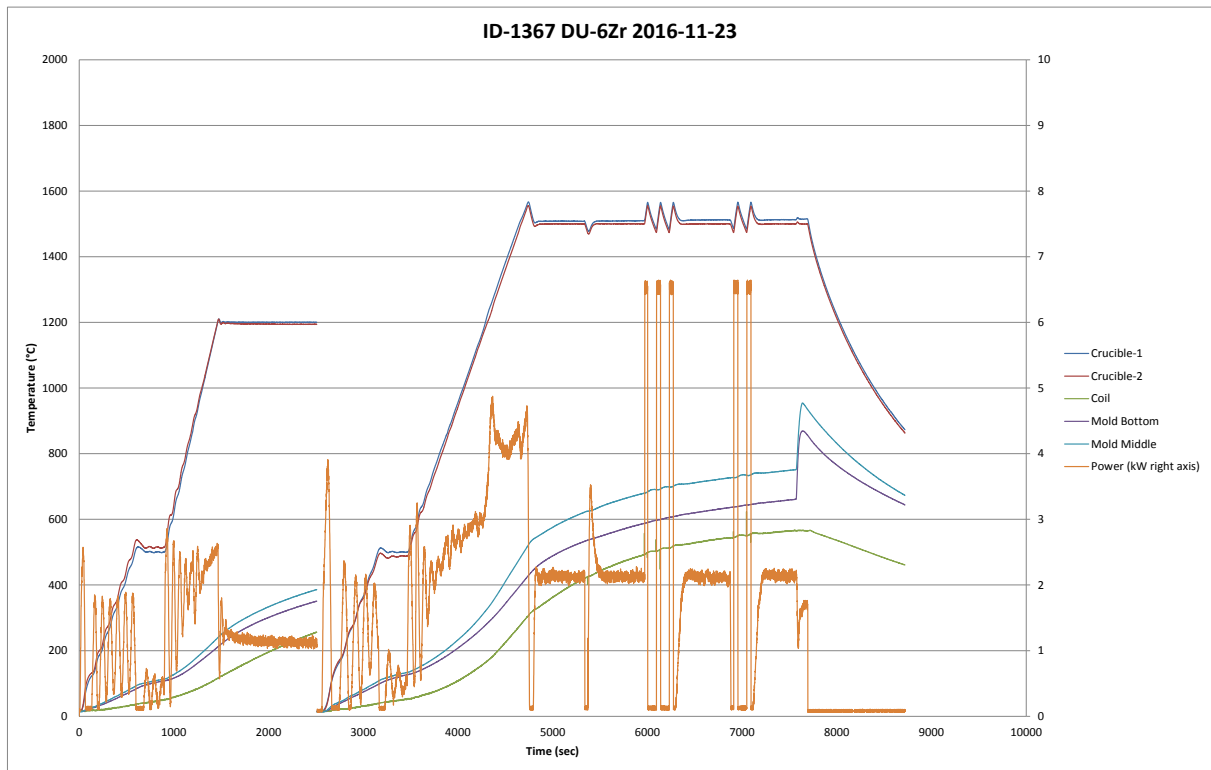


Figure A-2. Pulse program example with bake-out data included.

Appendix B: IDs of Castings (Including Failed Castings)

Date	ID	Material	Notes
4/2/2015	N/A	Cu	
4/9/2015	N/A	Cu	
4/30/2015	N/A	Cu-Si Bronze	
6/4/2015	N/A	Cu-3Ni	
6/16/2015	N/A	Cu-3Ni	
6/18/2015	N/A	Cu-3Ni	
7/1/2015	N/A	Cu-3Ni	High Temp Run
7/8/2015	N/A	Cu-3Ni	
7/13/2015	N/A	Cu-3Ni	
8/6/2015	1209	DU	
8/18/2015	1216	DU-2Zr	
8/31/2015	1226	DU-4Zr	
9/10/2015	1236	DU-10Zr	Failed
9/21/2015	1237	DU-10Zr	Failed
10/12/2015	1238	DU-MA	Failed
10/26/2015	1239	DU-MA	
11/2/2015	1240	DU-MA	
11/9/2015	1241	DU-10Zr	Failed
11/24/2015	1255	DU-10Zr	Filled mold, charge too high
12/14/2015	1242	DU-10Zr	Sheared Zr foil
2/4/2016	1275	DU-10Zr	
3/31/2016	1258	DU-6Zr	
4/21/2016	N/A	Cu-3Ni	For Extrusion process
4/27/2016	1259	DU-6Zr	
5/19/2016	1261	DU-6Zr	
5/25/2016	1262	DU-6Zr	
6/7/2016	1311	DU-6Zr	
6/22/2016	1260	DU-6Zr	
7/6/2016	1312	DU-6Zr	
7/18/2016	N/A	Cu-2Ni	For Extrusion process
8/1/2016	1349	DU-6Zr	
8/9/2016	1351	DU-6Zr	
8/15/2016	1350	DU-6Zr	
8/18/2016	1352	DU-6Zr	Failed
8/23/2016	1353	DU-6Zr	
8/25/2016	1354	DU-6Zr	
9/6/2016	1356	DU-6Zr	
9/13/2016	1348	DU-6Zr	Broken Crucible, failed to alloy
9/19/2016	1358	DU-6Zr	
9/22/2016	1359	DU-6Zr	
9/28/2016	1361	DU-6Zr	
10/12/2016	1362	DU-6Zr	
10/25/2016	1363	DU-6Zr	

Date	ID	Material	Notes
11/3/2016	1364	DU-6Zr	Started using EBR II Feedstock
11/14/2016	1365	DU-6Zr	Broken Lava Hat
11/17/2016	1366	DU-6Zr	
11/23/2016	1367	DU-6Zr	Large Casting
12/12/2016	1368	DU-10Zr	Large Casting
12/19/2016	1370	DU-10Zr	Half Casting
1/23/2017	1377	DU-10Zr	
1/30/2017	1371	DU-10Zr	
2/1/2017	1372	DU-10Zr	Crucible TC lost
2/7/2017	1373	DU-10Zr	
2/9/2017	1374	DU-10Zr	
2/13/2017	1375	DU-10Zr	No Bake Out Test, no further bake outs
2/15/2017	1376	DU-10Zr	
2/21/2017	1348-1	DU	Melt test with window
2/22/2017	N/A	Bismuth	Pulse Test
2/23/2017	1377	DU-10Zr	Zr Turnings and Zr Chips used from here on
2/28/2017	1378	DU-10Zr	
3/2/2017	1379	DU-10Zr	
3/7/2017	1380	DU-10Zr	
3/20/2017	1381	DU-10Zr	
3/22/2017	1382	DU-10Zr	
3/28/2017	1383	DU-10Zr	
3/29/2017	1384	DU-10Zr	
3/30/2017	1386	DU-10Zr	
4/10/2017	1387	DU-10Zr	
4/11/2017	1388	DU-10Zr	
4/13/2017	1389	DU-10Zr	
4/18/2017	1390	DU-10Zr	
4/19/2017	1391	DU-10Zr	
4/24/2017	1392	DU-10Zr	
5/4/2017	1394	DU-10Zr	
5/17/2017	1395	DU-10Zr	
5/22/2017	1396	DU-10Zr	
5/23/2017	1397	DU-10Zr	
6/5/2017	1398	DU-10Zr	
7/17/2017	1502	DU-10Zr	
7/19/2017	1503	DU-10Zr	
7/24/2017	1504	DU-8Zr	Fresh and Previously Cast
7/27/2017	1393	DU-10Zr	Small Casting
8/1/2017	1505	DU-10Zr	Fresh and Previously Cast
8/22/2017	1506	DU-10Zr	
8/28/2017	1507	DU-10Zr	Failed
9/5/2017	1516	DU-10Zr	As-Cast

Appendix C: INL Document List

1356-07-FASB, “EFF FASB General Laboratory Work”

LI-702, “Billet Casting System”