Consolidation of Simulated Nuclear Metallic Waste by Vacuum Coreless Induction Melting

D. R. Montgomery

October 1984

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SUMMARY

The Pacific Northwest Laboratory (PNL) has been conducting melting studies using a vacuum coreless induction furnace for the consolidation of simulated nuclear metallic waste. This study follows the Montgomery and Nesbitt (1983) study, wherein it was recommended that a large-scale cold mock-up facility be established to refine and test the vacuum coreless induction melting system which was selected as the most viable for nuclear metallic waste consolidation. Because a large-scale facility was not available, scaled-down tests were performed by retrofitting PNL's present Inductoslag furnace.

Vacuum coreless induction melting includes the use of a graphite crucible that is bottom-poured through the use of a water-cooled copper freeze plug. The molten metal is poured from the crucible into the canister or mold, which is located directly under the crucible. The metal melted for this study was limited to the eutectic alloy of zirconium and iron. The iron in this case was in the form of Type 316 stainless steel and the zirconium was in the form of oxidized Zircaloy 4 hulls. The components of this particular alloy constitute ~80% of the metallic waste expected from the operation of a spent fuel reprocessing facility such as the Barnwell Nuclear Fuels Processing (BNFP) facility if operated. Eleven heats were made during this investigation, and the following results and conclusions were obtained:

- 1/4-in.-thick-walled stainless steel canisters or molds performed most satisfactorily.
- 1/8-in.-thick-walled stainless steel canisters or molds proved to be too thin to hold the molten metal in at least one experiment.
- There was no metal-to-mold reaction when pouring into the 1/4-in.-thick stainless steel canisters.
- Preplaced scrap in the canisters was completely encapsulated by the molten metal that was poured on top of and around it. The density of the resulting ingot appeared to be ~99%.

- After 8 consecutive heats, the only visible erosion of the crucible occurred at the top of the melt line, where an $\sim 1/32$ -in.-deep x $1/8$ -in.-wide erosion line had formed. It had previously been hypothesized that a graphite crucible would last at least 4 heats; the estimate is now 16 heats.
- No failures of the water-cooled copper freeze plug were observed in any of the 11 heats.
- All ingots formed open pipes upon solidification that could be filled by subsequent pours. Four successive pours were made into a stainless steel canister. The resulting ingot after sectioning appeared to be $\sim 99\%$ dense except for part of the last pour, which contained the open pipe.
- The best melting rate was 41 kg/h at 40 kW with a power consumption of 1.03 kWh/kg.
- Large pieces of Zircaloy 4 scrap placed in the crucible with the appropriate amount of large stainless steel scrap can be melted as long as all of the submerged metal is melted before pouring.
- The design of the pouring nozzle needs additional work to prevent a solidified drop from forming at the nozzle exit after a pour. This drop prevents the subsequent seating of the copper plug and therefore has to be removed by hand before the next heat.
- Heavily oxidized Zircaloy 4 hulls mixed with 316 stainless steel hulls melted rapidly at $\sim 1260^\circ\text{C}$ with power set at 40 kW. The best pouring temperature was 1300 to 1325°C . After the water-cooled plug was pulled, an average of 15 seconds was required for the molten metal in the crucible to melt through the skull formed in the sprue, allowing the metal to pour from the crucible.
- The melt is quieter and gives off less vapor in a static $1/3$ atmosphere of argon than in a dynamic vacuum.

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INTRODUCTION

The Pacific Northwest Laboratory (PNL)^(a) is conducting a study to investigate the feasibility of vacuum coreless induction melting, as recommended by Montgomery and Nesbitt (1983), for the remote consolidation of metallic transuranic (TRU) nuclear waste of the type that would be expected from the operation of the Barnwell Nuclear Fuels Processing (BNFP) facility. The metallic waste form used in this investigation is the eutectic alloy of zirconium and iron. The iron in this case is in the form of Type 316 stainless steel and the zirconium is in the form of oxidized Zircaloy 4 hulls. This concept involves graphite crucibles with bottom pouring, which is accomplished using a water-cooled copper freeze plug controlled by a manipulator. The metal is poured into a stainless steel canister which serves both as a mold and as a simulated burial container.

The investigation included the modification of the PNL Inductoslag furnace to a vacuum coreless induction furnace as well as the installation of accessory equipment such as manipulator, sight glasses, thermocouples, and water lines to the water-cooled copper plug. The life of the graphite crucible, melting parameters, reliability of the water-cooled freeze plug, factors involved in the density and quality of ingots, and canister integrity were determined.

SELECTION OF METAL MELTING METHOD

In an earlier PNL study, Montgomery and Nesbitt (1983) identified, evaluated, and rated consolidation methods that could reduce the volume of metallic TRU nuclear waste generated in a fuel reprocessing facility. Compaction and melting techniques were rated, and each technique was evaluated based on its ability to handle and process the metallic wastes that would be produced if the BNFP facility were operated. From the numerous potentially applicable melting concepts and consolidation systems, the following processes were selected for evaluation:

(a) Operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute.

- Inductoslag melting
- rotating nonconsumable electrode melting
- plasma arc melting
- Electroslag melting with two nonconsumable electrodes
- vacuum coreless induction melting
- cold compaction.

Each process was evaluated and rated according to complexity, state and type of development required, safety, process requirements, and facility requirements. The numerical ratings for each process by criterion, a total rating for each, and the overall ranking of these processes are presented in Table 1. The ratings and numerical values shown for each criterion are the results of independent evaluations made by individuals with broad experience in radioactive waste processes, remote handling, remote process facility operation, and/or metal melting and casting.

As the table indicates, the vacuum coreless induction melting process was found to be the most viable process for consolidating nuclear metallic wastes.

TABLE 1. Composite Rating Matrix for Consolidation Processes

	<u>Inductoslag Melting (Ingot)</u>	<u>Inductoslag Melting (casting)</u>	<u>Nonconsumable Electrode Melting</u>	<u>Plasma Arc Melting</u>	<u>Electroslag Melting</u>	<u>Vacuum Coreless Induction Melting</u>	<u>Cold Compaction</u>
Complexity of process	302	362	359	320	254	435	320
State and type of development	240	254	264	244	210	284	229
Safety	227	228	212	222	210	262	282
Process requirements	157	156	217	189	140	226	242
Facility requirements	210	190	195	192	202	240	168
Total rating	1136	1190	1247	1167	1016	1447	1241
Ranking	6	4	2	5	7	1	3

CONCLUSIONS AND RECOMMENDATIONS

Vacuum coreless induction melting with bottom pouring has exceeded expectations for simplicity, reliability, and versatility when melting the zirconium and iron eutectic alloy. The melting tests have established that:

- the eutectic mixture of oxidized Zircaloy 4 hulls mixed with Type 316 stainless steel hulls can be melted at 41 kg/h at 40 kW with a power consumption of 1.03 kWh/kg and a melting temperature of 1260°C
- the life of a graphite crucible can be expected to be longer by a factor of 4 than was previously projected (i.e., 16 heats versus 4)
- the bottom-pour water-cooled copper freeze plug was 100% reliable
- an ~24-in.-tall stainless steel canister with 1/4-in.-thick walls (~6-in. inside diameter) was satisfactory in every respect
- an ingot formed from 4 consecutive heats poured into a stainless steel canister appeared to be ~99% dense after sectioning
- preplaced scrap in the canister can be encapsulated with molten metal to an ~99% density
- large pieces of Zircaloy 4 and stainless steel scrap can be melted, but have differing melting parameters
- the pouring nozzle requires further development to prevent solidified drops from forming at the hole exit after a pour.

It is recommended that a large-scale cold mock-up facility be established to refine and test a full-scale vacuum coreless induction melting system. Other options might include further scaled-down experiments to test other alloys and crucible materials under different atmospheric conditions (i.e., air melting).

VACUUM CORELESS INDUCTION MELTING AND CASTING INVESTIGATION

DESCRIPTION OF MELT STOCK WEIGHTS AND ALLOY

Eleven melting heats were fabricated, of which seven were made under a dynamic vacuum within the range of 0.05 to 10^{-3} torr and four were made under 1/3 atmosphere of argon. The melt stock included 316 stainless steel (SS) cladding from the Hanford Fast Flux Test Facility (FFTF). A mixture of two sizes of cladding were used: 1) outside diameter (OD) = 0.092 in.; inside diameter (ID) = 0.076 in.; wall thickness = 0.008 in.; and 2) OD = 0.131 in.; ID = 0.095 in.; wall thickness = 0.018 in.

The lighter cladding (1) weighed 0.565 g/cm^3 , which resulted in an approximate 14:1 volume reduction by melting. The heavier cladding (2) weighed 1.04 g/cm^3 with an ~7.7:1 volume reduction. Clean oxidized Zircaloy 4 hulls with oxide layers ranging from 0.021 g/dm^2 to 0.074 g/dm^2 and weighing ~960 g/liter constituted the remaining melt stock. When melted, the volume reduction was ~7.1:1. The charge for melting from heats 4 through 11 was ~2.42 kg of mixed stainless steel and ~13.40 kg of oxidized Zircaloy 4 hulls. This charge consisted of 18 wt% of 316 SS, including ~12 wt% iron. The remaining 6 wt% consisted of the alloy elements nickel, chromium, molybdenum, manganese, silicon, and carbon.

INDUCTOSLAG FURNACE MODIFICATION

The existing PNL Inductoslag furnace (Figure 1) was modified as follows:

- Inductoslag crucible and water manifold were removed.
- A water-cooled copper freeze plug was fabricated and installed.
- Two W 5% Re W 26% Re thermocouples were installed.
- A thermocouple feedthrough was installed.
- An ingot withdrawal stool was removed and replaced with a flat water-cooled copper plate.

FIGURE 1. Inductoslag Furnace Before Modification

- A manual manipulator was installed to hold and move the copper freeze plug.
- Sight glass was installed in the furnace door.
- The power supply and leads were changed from 150 kW and 10 kHz to 200 kW and 3 kHz.
- Part of the heat shield that fit around the Inductoslag crucible was removed.
- An induction coil, ~9 in. high and ~8 in. ID, was fabricated and installed.

After modification (Figure 2), the furnace was tested for proper operation.

SUMMARY OF HEATS 1 THROUGH 11

Heat 1

The objective of Heat 1 was to establish melting parameters. Approximately 20 kg of clean Zircaloy hulls and 3.6 kg of clean 316 SS hulls were mixed and dumped into the furnace feed hopper. A graphite crucible with outside measurements of 9-7/8 in. tall by 6-3/8 in. OD and inside measurements of 9-1/2 in. tall by 5-1/2 in. ID was used. A 3/4-in.-diameter hole was drilled in the bottom center of the crucible and the outer surface of the hole was radiused with a 1-in.-diameter drill bit. A 1/8-in.-thick-walled SS canister or mold was placed under the crucible, the crucible was placed in position, and the furnace door was closed. The water-cooled copper freeze plug (Figure 3) was positioned in the crucible pouring hole. The furnace was evacuated to 10^{-3} torr and the crucible was heated to 1550°C at the top (the thermocouple was located ~4 in. down from the crucible lip) and to 1208°C at the bottom (the thermocouple was located about 1/4 in. into the crucible bottom sidewall, 1/2 in. from the bottom). The charge was vibratorily fed into the crucible. This eutectic mixture was expected to start melting at ~950°C and to be completely melted at 1100°C. The hulls started melting immediately after contacting the bottom of the crucible and, from all indications, verified the expected melting parameters. The hulls were fed slowly in order to observe the

FIGURE 2. Inductoslag Furnace After Modification

FIGURE 3. Water-Cooled Copper Freeze Plug in Place in Crucible Pouring Hole

action around the freeze plug. The plug could be seen clearly and appeared to be holding the metal in the crucible without leaking. After 1.0 kg of charge was fed to and melted in the crucible, a sight glass cracked and caused a vacuum leak. The heat was terminated and the crucible and molten metal were allowed to cool. The vacuum leak did not cause any problems. Upon opening the furnace it was seen that the copper freeze plug had functioned as expected and had prevented any leakage. A slight metal-to-crucible reaction made it necessary to break the crucible to free the ingot. The ingot was $\sim 7/16$ in. thick and the sprue area that had been in contact with the plug appeared to contain unmelted material, as expected.

Heat 2

The objective of Heat 2 was to establish melting parameters. The same weight and material were charged to the furnace feed hopper as for Heat 1.

Melting took place at 925°C. Pouring was done at 1148°C. The copper freeze plug did not leak and the crucible emptied cleanly. The freeze plug did not retract far enough away from the bottom of the crucible, however, and the molten stream was diverted by the plug, hit the side of the 1/8-in.-thick-walled SS canister, and melted out an ~2-in.-wide strip the entire length of the canister (~24 in. high). The diverted stream also hit the SS flexible tubing used to cool the freeze plug, causing a large water leak. The vacuum pumps were left on to help remove any hydrogen or explosive gases from the furnace chamber. The cooling water was left on to the vacuum chamber for ~3 minutes. The vacuum pumps were turned off and the furnace chamber was backfilled. Several gallons of water leaked out when the chamber door was opened. The spill was cleaned up and preparations were made for Heat 3.

Heat 3

The objective of Heat 3 was to determine the melting parameters of large pieces of melting stock preplaced into the crucible followed by vibratory feeding of Zircaloy 4 hulls into the molten pool. A length of SS pipe weighing 2.43 kg and measuring ~4 in. tall by 4 in. OD by 3 in. ID was placed into the crucible. Sections of a Zircaloy 4 ingot were set on top of the SS pipe. These sections weighed a total of 8.06 kg. Next, 5.35 kg of clean Zircaloy 4 hulls were added to the furnace feed hopper.

It was necessary to heat the crucible to a top thermocouple temperature of 1640°C and a bottom temperature of 1400°C in order to initially melt the larger sections of metal. After melting had progressed for a time, the melting temperature leveled out at ~1200°C. The heat was poured at 1200°C. Ten seconds elapsed after the copper freeze plug was removed before the metal poured. Because they were submerged under the melt and were not visible, 1.779 kg of the large sections of Zircaloy 4 ingots did not melt; consequently, the molten pool was not held long enough for the pieces to go into solution. This fact also accounts for the higher pouring temperature than that of Heat 2, because the molten pool had not yet attained the complete eutectic composition before pouring took place.

A solidified drop formed on the edge of the pouring hole in the crucible and could not be removed without damaging the crucible. The copper freeze plug

worked perfectly. The heat took ~10 seconds to pour after removal of the plug. The ingot (Figure 4) had a secondary pipe with a volume of $\sim 11 \text{ cm}^3$.

Heat 4

The objective of Heat 4 was to determine the melting parameters when melting slightly oxidized ($\sim 0.021 \text{ g/dm}^2$) Zircaloy 4 hulls. Before this heat, a newly designed crucible (Figure 5) and a replaceable pouring nozzle (Figure 6) were fabricated. Heat 4 was the first to utilize the new crucible and nozzle. For this heat, 13.42 kg of the oxidized Zircaloy 4 hulls mixed with 2.43 kg of 316 SS hulls were charged to the furnace feed hopper. Some hulls were fed into the crucible with the bottom temperature at 1148°C . Partial melting occurred

FIGURE 4. Ingot Showing Secondary Pipe

FIGURE 5. Newly Designed Crucible

FIGURE 6. Pouring Nozzle

at 1260°C. With the bottom temperature at 1349°C, melting increased rapidly. The heat was cooled to 1226°C in preparation for pouring. The copper freeze plug was pulled, but nothing happened. After five minutes had elapsed and the crucible still had not poured, the bottom temperature was raised to ~1450°C, whereupon the skull melted in the sprue and the metal poured. The freeze plug worked well. Early in the heat some molten metal oozed between the crucible nozzle and the freeze plug, but it immediately froze out and did not cause any problems. This shows that the freeze plug is very forgiving of a poor fit at the seal surface. The ingot (Figure 7), which weighed 15.55 kg, had an open pipe and a good surface. Approximately 0.30 kg remained in the crucible. The ingot had not reacted with the graphite canister or mold and was removed simply by turning the mold upside down and allowing the ingot to fall out.

Heat 5

The objective of Heat 5 was to determine the melting parameters when melting heavily oxidized ($\sim 0.074 \text{ g/dm}^2$) Zircaloy 4 hulls. The same crucible and nozzle were used as in Heat 4. For this heat 13.40 kg of heavily oxidized Zircaloy 4 hulls and 2.43 kg of 316 SS hulls were mixed and charged to the furnace feed hopper. The crucible was heated to 1204°C at the bottom, with the top temperature at 1426°C. Some hulls were fed into the crucible at this temperature and some melting occurred. When the level of molten metal approached the area of the top thermocouple, the bottom and top temperatures equalized. With a full molten pool and some unmelted material the temperatures leveled out at ~1260°C. The maximum temperature during this heat was 1648°C at the top and 1371°C at the bottom. The freeze plug was pulled at ~1430°C and the heat poured in ~5 to 10 seconds. During the heat some skull formed on the surface of the melt and some large gas bubbles were observed around the circumference. It is believed that the skull is excess zirconium oxide which dissolves over time and that the gas bubbles are caused by hydrogen as a result of the auto-claved oxidized Zircaloy 4 hulls. The ingot (Figure 8) weighed 14.97 kg with a melting loss of 0.881 kg. The ingot was poured on top of a tamped, ~5/8-in.-thick fiberfrax cloth, which had been placed in the bottom of the graphite mold in an effort to reduce the stream erosion on the bottom of the graphite mold.

FIGURE 7. Ingot 4 Showing an Open Pipe

FIGURE 8. Ingot from Heat 5 Showing an Open Pipe

The molten metal penetrated 90% of the cloth, which probably reduced the mold erosion, but the benefits were not sufficient to warrant continuation of this practice.

Heat 6

The objective of Heat 6 was to pour molten metal over some preplaced scrap in the bottom of the canister or mold and to determine how well the molten metal encapsulated the scrap. The scrap consisted of a SS pipe ~3-1/2 in. high by 4-1/2 in. OD with a 3/8-in. wall thickness. The pipe was set on top of a SS plate ~4-1/2 in. square by 3/8 in. thick. Inside the pipe were a handful of heavily oxidized Zircaloy 4 hulls and several 316 SS hulls. On top of the hulls were several pieces of angled SS scrap ~3/8 in. to 1/2 in. thick by ~1 to 2 in. dimension. The total weight in the mold was 3.632 kg. The charge weight to the furnace feed hopper was 13.415 kg of heavily oxidized Zircaloy 4 hulls mixed with 2.434 kg of 316 SS hulls. The feeding of hulls to the crucible began when the bottom temperature of the crucible was at 1204°C and the top was at 1520°C. The temperature equalized at 1260°C while there was still some unmelted material in the melt. The temperature was raised to 1371°C and the freeze plug was pulled. It took ~30 seconds before pouring started. The molten metal encapsulation of the scrap was complete. The ingot (Figure 9) had an open pipe and weighed ~18.86 kg with a melting loss of ~0.62 kg. Most of the loss remained in the crucible. The graphite mold cracked longitudinally due to the thermal expansion of the SS pipe after being filled in and around with molten metal that subsequently solidified. Figure 10 is a closeup of the encapsulated scrap and illustrates the type of hulls that were included in the encapsulated scrap metal. This heat was the third to use the same crucible and nozzle.

Heat 7

The objective of this heat was to gain further information about melting parameters and also to test a new nozzle design (Figure 11). For this heat, 13.40 kg of heavily oxidized Zircaloy 4 hulls were mixed with 2.42 kg of 316 SS hulls and charged to the furnace feed hopper. Feeding of hulls to the crucible was initiated with the top crucible temperature at 1430°C and the bottom temperature at 1226°C; some melting occurred. When the freeze plug was pulled,

FIGURE 9. Ingot 6 Showing Encapsulation of Preplaced Metal in the Canister (note: ingot is upside down)

FIGURE 9. Example of Encapsulation Showing Zircaloy and Stainless Steel Holes
 in the Canister (note: indor is upside down)

FIGURE 10. Example of Encapsulation Showing Zircaloy and Stainless Steel Holes

FIGURE 11. Latest Nozzle Design

the bottom crucible temperature was 1226°C and the top was 1280°C. After ~2 min the crucible had not dumped so the bottom crucible temperature was raised to 1280°C, whereupon the crucible dumped. Immediately after pouring, the molten metal began spitting out of the mold because of an obvious metal-to-mold reaction which produced large amounts of gas. The reaction is thought to have been caused by a thin film of oil on the interior surface of the mold which came from the compressed air that had been used to blow out any loose residue before placement in the furnace. The performance of the newly designed nozzle could not be evaluated because the splatter from the mold had coated the nozzle with solidified metal. It was reuseable after replacing parts 2 and 3 as shown in Figure 11. The portion of the ingot that had remained in the mold (Figure 12) could not be removed without breaking the graphite mold. This was the fourth consecutive heat using the same crucible.

Heats 8, 9, 10, and 11

The objective of these heats was to obtain further information on melting parameters and to determine whether four consecutive heats, one poured

FIGURE 12. Ingot from Heat 8, Showing Result of Gas in the Mold

on top of the other, would create an ingot with an acceptable density. Other factors such as the integrity of the SS canister, metal-to-canister reaction, and whether or not each consecutive ingot formed an open pipe were investigated.

All charges consisted of 13.40 kg of heavily oxidized Zircaloy 4 hulls mixed with 2.42 kg of 316 SS hulls. The charges for Heats 9 and 10 contained some zirconium fluoride on the surface of the Zircaloy 4 hulls. All heats were done at 1/3 atmosphere of argon. The best melting parameters were obtained in Heat 11, which shall be described in detail below.

At 10:54 a.m. power was turned on at 40 kW and the crucible temperature was raised to $\sim 1430^{\circ}\text{C}$ top and $\sim 900^{\circ}\text{C}$ bottom. At 11:00 the crucible was filled with hulls. At 11:04 the crucible contents had melted down to $\sim 1/2$ the volume. The crucible was refilled with hulls, with the top thermocouple at $\sim 1580^{\circ}\text{C}$ and the bottom at $\sim 1150^{\circ}\text{C}$. Refilling was repeated two more times under approximately the same conditions, and was thereafter followed by incremental feedings. Feeding of hulls was completed at 11:23 with the bottom thermocouple at 1230°C and the top thermocouple at 1371°C . The pool was held molten until the bottom thermocouple was at the pouring temperature, 1300°C . The freeze plug was pulled at 11:28. About 20 seconds elapsed before the crucible dumped. The pouring nozzle worked well, excepting a solidified drop that formed on the nozzle exit after each pour, which required hands-on repair before a subsequent heat.

After each heat the ingot was visually inspected to determine whether it had developed open pipes, so that the subsequent molten metal poured on top would fill the void caused by the open pipe and create a satisfactorily dense ingot. The canister containing the four heats (Figure 13) was sectioned as shown in Figure 14, which plainly shows that each pipe had been filled with metal. The top views of the ingot sections (Figures 15 and 16) also show very few voids with the exception of some small gas pockets. Figure 17 shows that there was very little reaction between the molten metal and the SS canister and that the canister maintained its integrity. The crucible was also sectioned (Figure 18) and revealed that the only erosion of any significance occurred at the top of the melt line, which was $\sim 1/32$ in. deep by $\sim 1/4$ in. wide. A graphite crucible used for melting this type of alloy should easily last for 16 heats.

FIGURE 13. Stainless Steel Canister with Four Ingots
Poured Consecutively

FIGURE 14. Cross Section of Ingot Shown in Figure 13

FIGURE 15. Tops of Ingots 3 (left) and 4 (right)

FIGURE 16. Tops of Ingots 1 and 2 (bottom of Ingot 2 showing an attached section pulled out from the top of 1)

FIGURE 17. Ingot Showing No Metal-to-Mold Reaction

FIGURE 18. Cross Section of Crucible Showing Minimal Reaction After Eight Successive Heats

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