ELECTROMELT FURNACE EVALUATION

MASTER

G. A. Reimann
J. M. Welch

U.S. Department of Energy

Idaho Operations Office • Idaho National Engineering Laboratory

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G. A. Reimann
J. M. Welch

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EG&G Idaho, Inc.
Idaho Falls, Idaho 83415

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ABSTRACT

An electromelt furnace was designed, built, and operated at the Idaho National Engineering Laboratory to demonstrate the suitability of this equipment for large-scale processing of radioactive wastes in iron-enriched basalt. Several typical waste compositions were melted and cast. The furnace was disassembled and the components evaluated. Calcines and fluorides attacked the furnace lining, unoxidized metals accumulated under the slag, and electrode attrition was high.

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ELECTROMELT FURNACE EVALUATION

1. INTRODUCTION

Disposal of radioactive wastes has been a problem for several decades. Efforts to find a solution have intensified recently as the capacity of various "temporary" storage facilities is approached and as political pressures have multiplied. Present disposal methods are not satisfactory for some wastes, and many tons of radioactive materials and contaminated waste are in storage awaiting development of a technology that will enable controlled, secure, and permanent disposal.

One approach to radioactive waste disposal that shows considerable promise is dissolution of the wastes in iron-enriched basalt, a man-made rock that is similar in composition to natural basalt (lava). Studies are underway to verify that the properties of the man-made rock are suitable for containing a variety of radioactive elements securely for extended time periods. It appears that iron-enriched basalt is an excellent material for this application, as it has good mechanical properties and low water solubility and maintains these attributes over the wide range of compositions that may be encountered when processing a variety of nuclear wastes.

This report describes the use of an electromelt furnace to prepare iron-enriched basalt by melting noncombustible, simulated nuclear wastes with mineral additives. Waste compositions approximated what is expected from various waste generators. The resultant melts were cast into mild-steel molds. After the melting campaign, the electromelt furnace was examined to identify problems and to determine its potential for scale-up and for remote operation.

2. ELECTROMELT FURNACE CONSTRUCTION AND OPERATION

In order to produce and evaluate man-made basalt on a pilot plant scale, a joule-heated furnace was designed and built at the Idaho National Engineering Laboratory (INEL). This furnace is an experimental unit that incorporates several of the features developed by Penberthy¹ and others for commercial glass manufacturing. The INEL electromelt furnace is designed to produce intermediate-sized (up to 100 kg) castings of iron-enriched basalt. Heating of the charge is accomplished by the resistance of the melt to the passage of electric current.

The INEL electromelt furnace is intended as a prototype design. In addition to demonstrating the feasibility of processing nuclear waste in man-made basalt, the furnace will generate data on the durability of refractories and electrodes, establish a basis for the design of large, efficient and reliable units, and enable development of skills and technology necessary for remote operations required to process radioactive wastes.

2.1 Furnace Design and Construction

An isometric drawing of the electromelt furnace is shown in Figure 1. The unit made extensive use of castable refractories; however, the tank was lined with Harbison-Walker "Ruby" (90% Al_2O_3 -10% Cr_2O_3) brick for resistance to slag attack. The bricks were fitted closely together and arranged to form a 30 x 30 x 90-cm tank. A horizontal row of four holes was bored through the bricks along each side of the tank with a 5.7-cm diamond core drill to permit insertion of 5-cm diameter molybdenum electrodes.

The body of the furnace was formed upside down, with the Ruby brick tank assembled over a plywood form. A 7.6-cm layer of Kast-o-lite 30 (A. P. Green Co.) was cast around the Ruby brick. The form was removed and the steel furnace shell was assembled and centered around the furnace interior. The remaining volume was filled with Kast-o-lite 25 and Kast-o-lite 22. Cores were inserted into the bored brick and extended to

Figure 1. Electromelt furnace

INEL-A-17 276

the furnace interior to maintain tubes for insertion of the electrodes. The steel bottom was bolted into place and the furnace was rotated to the upright position and placed on its mount after the castable refractories had set.

The furnace top was made in three lengths using "Greencast 97" (alumina) by pouring the castable into reusable molds fabricated from plywood and sheet metal. The molds were made sufficiently durable for producing several additional sections should it become necessary. The Greencast 97 was cast into 7.6-cm thick arches that spanned the width of the melting chamber. This castable was selected for its strength at elevated temperature and for its resistance to deterioration from direct flame impingement. It was intended that these arches become incandescent and radiate heat back to the molten bath or onto a fresh, cold charge.

Larger arches, 15 cm thick, were made from Kast-o-lite 22 to cover and insulate the alumina interior arches. The assembled furnace top was covered by an alumina wool blanket 8-cm thick which in turn was protected by a sheet metal shield. Two gas exhaust ports in the furnace top were lined with castable alumina flue pipes and an exhaust hood was constructed over these ports to carry away the fumes and dust.

A lifting ring was fastened to the rear of the furnace to permit attachment of an overhead crane hook. The furnace was tilted for pouring by lifting the rear with the crane.

2.2 Power Supply

The electrical supply uses 480-V, 3-phase power through tapped transformers with a combined rating of 300 kVA. Manual switches enable selection of three ranges of voltage from the secondary. A silicon-controlled rectifier circuit controls the primary voltage, enabling variation of the power within each voltage range selected and also permitting independent adjustment of each phase in the event it is desired to vary the temperature in different zones of the bath.

The power leads are connected to the molybdenum electrodes with heavy copper clamps that are water-cooled to carry away the heat conducted by the electrodes from the molten bath.

2.3 Furnace Operation

The cold furnace charge is nonconducting and must be raised to a temperature sufficient to initiate melting before it will carry enough current to maintain bath temperature. Preheating the charge was accomplished with a propane gas burner by inserting the burner nozzle through the pouring spout opening so that the flame was directed at the charge (Figure 2). The initial charge was soda-silicate (25% Na_2O , 75% SiO_2), which was heaped along the sides of the tank to cover the electrodes. The furnace was heated slowly to avoid spalling and cracking of the fresh-cast refractories.

Thermocouples were placed on the outer surface of the alumina arches to monitor the temperature. After about six hours of careful heating, the temperature of the outer surfaces of the alumina arches exceeded 1280 K and the surface of the charge had become molten. The electrodes, which had been positioned so that they were flush with the sides of the chamber, were pushed inward manually until they protruded about 10 cm into the melt. The power supply was energized and 160 A could be induced to flow through the bath at 60 V. While it was not evident visually that any heating resulted from the passage of 160 A, the charge began settling and it was necessary to add more material to prevent the electrodes from becoming exposed. The propane burner was also kept on the charge.

After two additional hours of heating (8 h total) the propane burner was removed and the current increased to 320 A. Heating along each row of electrodes became evident. The 100 A circuit breakers protecting the transformer primary windings began tripping because heat was passing upward through conduits to the breaker boxes. Transformer temperatures began to exceed 360 K, which was the limit for the insulation on the conductors. Sealing the conduits to airflow and providing fans for the transformers allowed the primary current to be increased to 95 A.

a. Burner preheating furnace charge.

b. Removing burner from furnace after preheating pouring spout.

Figure 2. Propane gas burner.

The charge began to heat more rapidly and the last of the cold mix was added. The first melt was poured 20 h after the initial burner firing and

14 h after the electrical power was turned on. The electrical power required to bring the furnace and charge to temperature for the first pour was 375 kWh. The power equivalent of the gas used for preheating was not determined.

A charge of 68 kg (150 lb) of kyanite tailings was added to the soda silicate remaining in the chamber. This mix was poured at 1780 K within 4 h of charging. Six additional compositions were charged and poured. The compositions were formulated to resemble what is expected when various radioactive waste calcines, sludges, zeolites, and soils are processed, except that radioactive components were absent. Since the chamber contained 71 litres when filled, but only 28 litres were poured, the mixes had to compensate for the composition that remained in the chamber after each pour. The compositions are detailed in Table 1. A heat being poured from the furnace is shown in Figure 3, and the castings are shown in Figure 4. No difficulties were encountered with any of the heats except that one pour melted through the side of the receiving container because the 1870 K bath temperature was excessive. The power needed for the last 7 melts was 1275 kWh, or 182 kWh average per melt.

Figure 3. Heat of slag being poured from electromelter into drum.

TABLE 1. COMPOSITIONS OF ELECTROMELT FURNACE HEATS

Heat No.		SiO ₂	Na ₂ O	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	NiO	MnO ₂	ZrO ₂	CaF ₂	Fe
1	Add: 150 kg SiO ₂ + Na ₂ O Melt Composition: Percent Pour: 50.3 kg, 99.8 kg balance	112.6 75 74.8	37.5 25 24.9										
2	Add: 68.0 kg Kyanite tailings Melt Composition: 167.8 kg total Percent Pour: 52.2 kg, 115.7 kg balance	11.3 86.1 51.9 59.3				16.9 16.9 10.1 11.7	39.9 39.9 23.8 27.5						
3	Add: 34.0 kg SiO ₂ , 39.2 kg Dolowhite, 23.2 kg Kyanite tailings Melt Composition: 193.3 kg total Percent Pour: 65.8 kg, 127.6 kg balance	34.0 3.9 97.2 50.3 64.1		12.4 12.4 8.9 8.2	8.0 8.0 4.2 5.4		5.8 17.5 9.0 11.5						
4	Add: 51.0 kg zeolite, 11.9 kg Fe ₂ O ₃ , 5.4 kg CaO Melt Composition: 195.8 kg total Percent Pour: 58.5 kg, 137.3 kg balance	34.7 98.8 50.5 69.4	2.8 14.1 7.2 9.9	1.7 15.9 7.8 10.7	0.8 6.1 3.1 4.3	8.4 19.9 10.2 14.0	2.1 11.9 21.0 28.8	0.6 0.6 0.03 0.4					
5	Add: 27.2 kg Kyanite tailings, 31.8 kg SiO ₂ , 1.9 kg Na ₂ O, 2.1 kg MnO 11.2 kg dolowhite, 0.4 kg Na ₂ SO ₄ 1.4 kg NiO, 2.3 kg zeolite Melt Composition: 210 kg total Percent Pour: 64.4 kg pour, 145.7 kg balance	4.5 31.8 1.5 107.2 51.0 74.3	1.9 0.14 0.14 12.1 5.7 8.3	3.5 2.3 0.1 14.3 6.8 9.9	2.3 0.05 0.4 6.6 3.2 4.7		6.8 16.0 0.1 21.1 21.4 31.2						
6	Add: 27.2 kg SiO ₂ , 0.5 kg Na ₂ O, 7.3 kg Al ₂ O ₃ 11.7 kg dolowhite, 0.15 kg K ₂ O 24.1 kg Kyanite tailings Melt Composition: 210.8 kg total Percent Pour: 61.7 kg, 149.1 kg balance	27.2 4.0 105.6 50.1 70.2	0.5 3.7 8.8 4.1 6.1		2.4 6.0 7.1 3.3 4.9		7.3 14.2 45.4 21.5 32.1	0.15 0.4 0.2 0.3					
7	Add: 24.0 kg SiO ₂ , 11.2 kg dolowhite, 13.6 kg Fe 16.8 kg Kaolin, 2.1 kg Na ₂ O 2.3 kg zeolite Melt Composition: 211.1 kg total Percent ^a Pour: 40.0 kg, 157 kg balance ^a	24.0 7.6 1.5 107.9 54.6 85.9		3.5 2.1 0.1 13.2 6.7 10.6	2.3 6.6 0.05 7.3 3.7 5.8								13.6
8	Add: 23.7 kg SiO ₂ , 1.1 kg Al ₂ O ₃ , 5.4 kg ZrO ₂ 19.1 kg kyanite tailings, 10.9 kg CaF ₂ 9.1 kg Kaolin Melt Composition: 225.4 kg Total ^a Percent ^a Pour: 68.9 kg, 156.5 kg balance ^a	23.7 3.2 4.1 116.9 51.9				1.1 4.8 3.6 30.8 13.6					5.4 10.9		13.6

a. w/o Fe

a. Several slag castings. Thermocouples protrude from container in foreground.

b. Sectioned casting from Heat No. 4.

Figure 4. Slag castings from electromelt furnace.

As the mass of the furnace reached an equilibrium temperature and the melt composition became more complex, the current required to maintain a 1680-1780 K temperature decreased from 320 to 240 A. Occasionally, a crust would form on the bath or a fresh charge would be difficult to melt due to crusting. Such problems could be eased with additional heat from the burner, or by breaking the crust and stirring it into the melt with a steel rod.

An exhaust hood over the furnace carried off combustion products from the gas burner as well as dust from charging the furnace and gases from decomposition of the charge. The hood operated by natural thermal convection as an exhaust fan had not been installed when the furnace was operated. Air samplers placed on the furnace showed the airborne particulate content to be (in mg/m^3): sodium 0.009 to 0.016, calcium 0.004, and fluoride 0.022.

The furnace was set at 100 A to "idle" after the last pour. This setting maintained a steady bath temperature of 1680 K. The bath could have been maintained fluid at about 1280 K with 60-70 A, however, it was feared that some crystallization and segregation would occur that would affect adversely the composition of the remaining liquid.

The furnace was inspected after about 60 hours of idling, and it was noted that the Ruby brick was deteriorating in the vicinity of the electrodes. The power was shut off, the tank was tilted to pour out as much material as possible, and a 10-cm layer of silica sand was added to protect the exposed electrodes and to retard the cooling rate.

3. FURNACE EVALUATION

The furnace was allowed to cool for two days before the dismantling and inspection of components began. All of the castable arches were suitable for reuse (Figure 5). The sand was removed, the solidified slag was chipped out, and the bricks and electrodes were removed for examination. A layer of iron was found on the bottom of the melting chamber which originated from the 13.6 kg of iron added for the seventh melt. Less than 20% of the iron was oxidized in nearly 100 h of operation. The compositions in Table 1 were adjusted so that the unoxidized iron present does not appear in the slag as Fe_2O_3 .

The deterioration of the Ruby brick between the electrodes and corrosion of a molybdenum electrode are shown in Figure 6, where the original electrode diameter is visible after the sidewall bricks were removed. Other views of the sidewall brick corrosion are shown in Figure 7, where a new brick was placed alongside for comparison. Solution of the sidewall brick between the electrodes is believed to have developed as a result of the presence of CaF_2 , which is an excellent flux for alumina, and as the result of excessive temperature that developed in the bath between the electrodes while idling for two days at 100 A. Bath

Figure 5. Deterioration of castable insulation around furnace vent due to overheating.

a. Electrodes and slag.

b. Condition of Ruby brick between electrodes after slag was poured out.

c. Condition of molybdenum electrode; removal of brick reveals original diameter.

Figure 6. Electrodes, brick, and slag after cooling and disassembling electromelt furnace.

- a. Comparison of dissolved brick with new brick. Sloping line was caused by last pour.

- b. Reassembled Ruby bricks showing corrosion along sidewall and layer of unoxidized iron.

Figure 7. Sidewall bricks after disassembly.

temperature measurements using an optical pyrometer would not detect localized overheating between the electrodes. No brick deterioration was detected visually during the course of the first seven melts. It developed only after idling the furnace with CaF_2 present.

Test bricks of Serv, Serv-N, Monofrax K-3, and Monofrax E were incorporated into the lining at the rear of the furnace to assess their durability. There was little slag attack on these bricks (about 8 or 10 mm), however, the rear of the furnace was cooler and less suitable for obtaining durability information as a location between electrodes.

Samples were selected for more detailed study, and polished sections were examined using reflected-light microscopy and SEM/EDS analysis. These samples included a specimen of a corroded Ruby brick, the undrained slag that contained a metallic iron globule, slag resting on the hearth bricks, and a section from a molybdenum electrode.

3.1 Slag-Refractory Interaction

A Ruby refractory brick was sectioned perpendicular to a visible corroded surface and polished. The microstructure at the brick surface, shown in Figure 8, was observed to include a layer of FeAl_2O_4 - FeCr_2O_4 spinel that separated the slag from the brick, indicating that iron oxide in the slag reacted with the sesquioxides (Al_2O_3 - Cr_2O_3) of the brick to form the spinel which in turn dissolved into the slag. The sesquioxides of the brick do not seem to be dissolved into the slag directly, but first form an intermediate product (spinel) by reaction with the slag. This reaction layer could have provided protection for the refractory brick during the processing of the first melts. This phenomenon was observed in preliminary tests by Seymour.² However, this protection may have been destroyed by the 5% CaF_2 contained in the final melt. CaF_2 is a fluxing medium that will lower the temperature at which a crystalline material will be dissolved by a slag. Thus, a spinel barrier would be less resistant to a CaF_2 -bearing slag.

Figure 8. Micrograph of slag/refractory interface.

3.2 Undrained Slag Containing Metallic Iron

A sample was selected from the solidified slag in contact with metallic iron that remained in the electromelter. The polished section, shown macroscopically in Figure 9a, has reaction rings that formed around the metal globule. At the metal/slag interface, the metal and slag are interfingered (Fig. 9b). The amount of metal present decreases abruptly away from the metal globule such that there is $\sim 5\%$ Fe^0 present in the inner reaction ring, $\sim 1\%$ Fe^0 present in the outer reaction ring, and no detectable iron in the area of the slag outside the reaction rings.

During the cool-down of the electromelter, the residual slag partially devitrified. The oxide phases present are listed in Table 2. Both the fayalite and the glass near the metal/slag interface have very high iron

a. Macrograph of polished section.

0.5 cm

50 μm

b. Micrograph of metal/slag interface. White = metal, light gray = fayalite, and dark gray = glass.

Figure 9. Electromelter sample containing metallic iron globule.

oxide contents (from EDS analysis) and the iron-oxide content of the bulk slag decreases away from the metal-rich areas. The high iron oxide content of the slag in metal-rich areas indicates that the metal was being oxidized and dissolved by the slag, then dispersed in response to a concentration gradient.

TABLE 2. PHASES PRESENT IN ELECTROMELTER SAMPLE WITH METALLIC IRON

Location	Phases Present (in order of abundance)
Slag/metal interface	fayalite ¹ > glass
Inner reaction ring	glass > fayalite
Outer reaction ring	glass > fayalite > spinel ²
Outside reaction rings	glass > spinel > fayalite > augite ³

1. Fe_2SiO_4

2. Fe_3O_4 - FeCr_2O_4 solid solution

3. $\text{Ca}(\text{Fe},\text{Mg})(\text{Si},\text{Al})\text{Si}_2\text{O}_6$

During EDS analysis it was noted that the metallic iron phase present in the reaction rings contained substantial amounts of molybdenum. The molybdenum electrodes are the most likely source for this component, and it is possible that molten iron present beneath the slag will accelerate electrode corrosion without iron being in direct contact with the electrodes.

3.3 Molybdenum Electrodes

The molybdenum electrodes were found to be tapered over the 9-cm length that they were immersed in the slag bath (Figures 3 and 4). The tip of the electrode measured 4 cm in diameter, compared to the original diameter of 5 cm. Near the tip of the electrode was a deposit of yellowish-green fibrous crystals which was identified as molybdate (MoO_3). These crystals developed when the hot electrodes were exposed to air during the final draining of the electromelter. At this time it was observed that the electrodes "smoked" (due to vaporization of molybdenum oxides); they were then quickly covered with silica sand to isolate them from the air. Although some corrosion of the electrodes undoubtedly took place at this time, most of the 1 cm decrease in diameter is due to corrosion during the operation of the electromelter and to momentary exposure to air during pours.

3.4 Slag Resting on Hearth Bricks

One sample was taken from the slag where it was in contact with the bottom bricks of the hearth. The portion of the sample in contact with the hearth bricks contained coarse spinel and plagioclase crystals in a cumulate textured layer approximately 2 mm thick. Some spinel crystals exceeded 100 μm across the grain. Above this layer a second cumulate layer, approximately 2 mm thick, was composed of spinel, plagioclase, and augite. The main components of the spinel are iron, chromium, and aluminum that would form the spinel components FeCr_2O_4 , and FeAl_2O_4 . The plagioclase crystals are generally elongate and have the general chemistry, $(\text{Ca},\text{Na})(\text{Al},\text{Si})_4\text{O}_8$, where $\text{Ca} > \text{Na}$.

In the upper cumulate layer, the grain size of the crystalline phases was approximately one-quarter of the lower cumulate layer. The spinel in the upper layer contained less aluminum and more iron than the spinel in the lower layer, but the plagioclase compositions were similar in the two layers. The augite of the upper cumulate layer contained the components of the general formula $\text{Ca}(\text{Fe},\text{Mg})(\text{Si},\text{Al})_2\text{O}_6$, where $\text{Fe} > \text{Mg}$.

Cumulate layers generally result from crystals forming in a cooling melt and sinking through the molten slag to form the layers. Before the slag was drained, the electromelter was idled at 1480 K for two days. It was probably during this time that the crystals formed and settled. When the slag was reheated to 1780 K for the final pour, the bottom layers were not heated for a sufficient time to allow the phases to be redissolved into the slag. Above the cumulate layers the partially devitrified slag containing spinel and dendrites of augite seems to have cooled in situ.

4. CONCLUSIONS AND RECOMMENDATIONS

Generally, the application of the electromelter principle to melting and pouring of iron-enriched basalt was quite successful, and further work should be directed toward refinement of the furnace and adapting it for remote operation.

It is evident that some method must be devised to accelerate oxidation of metals entering the bath so that they readily dissolve into the slag and cannot accumulate to the extent that they short-circuit, or alloy with, the electrodes. Reactive metals such as zirconium will oxidize readily, but the stainless steels and high-nickel alloys will be difficult. A bubbler for injecting air will be devised for the next series of tests.

A more desirable electrode arrangement could enhance convection in the slag. Locating the electrodes so that they protruded vertically through the furnace bottom would provide better convection and prevent overheating of the sidewalls. However, a means to prevent accumulation of metalics around the electrodes must be devised to avoid short circuiting and/or destruction of electrodes by alloying. The application of a bubbler to oxidize metalics may generate enough stirring in the bath to assure more uniform temperatures with the present electrode arrangement.

Several compositions of refractory brick should be incorporated into the sidewall to compare performance with Ruby brick, particularly when using corrosive fluoride slag.

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