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CONVERSION OF PLUTONIUM SCRAP AND RESIDUE TO BOROSILICATE GLASS  
USING THE GMODS PROCESS

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# Conversion Of Plutonium Scrap And Residue To Borosilicate Glass Using The GMODS Process

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## ABSTRACT

Plutonium scrap and residue represent major national and international concerns because (1) significant environmental, safety, and health (ES&H) problems have been identified with their storage; (2) all plutonium recovered from the black market in Europe has been from this category; (3) storage costs are high; and (4) safeguards are difficult.

It is proposed to address these problems by conversion of plutonium scrap and residue to a CRACHIP (CRITICALITY, Aerosol, and CHEMICALLY Inert Plutonium) glass using the Glass Material Oxidation and Dissolution System (GMODS). CRACHIP refers to a set of requirements for plutonium storage forms that minimize ES&H concerns. The concept is several decades old. Conversion of plutonium from complex chemical mixtures and variable geometries into a certified, qualified, homogeneous CRACHIP glass creates a stable chemical form that minimizes ES&H risks, simplifies safeguards and security, provides an easy-to-store form, decreases storage costs, and allows for future disposition options.

GMODS is a new process to directly convert metals, ceramics, and amorphous solids to glass; oxidize organics with the residue converted to glass; and convert chlorides to borosilicate glass and a secondary sodium chloride stream. Laboratory work has demonstrated the conversion of cerium (a plutonium surrogate), uranium (a plutonium surrogate), Zircaloy, stainless steel, and other materials to glass. GMODS is an enabling technology that creates new options. Conventional glassmaking processes require conversion of feeds to oxide-like forms before final conversion to glass. Such chemical conversion and separation processes are often complex and expensive.

## INTRODUCTION

In the United States, significant ES&H concerns<sup>1</sup> have been identified with the storage of plutonium scrap and residue. A similar situation is thought to exist in Russia. All of the plutonium recovered from the black market in Europe has been from this category. Storage costs are high and safeguards are difficult. These difficulties are a direct result of the characteristics of these materials. Plutonium scrap and residue normally consist of only a few weight percent plutonium, but the total volume and mass exceed that of clean plutonium. The materials have highly variable chemical and nuclear characteristics. Some of the chemical forms are hazardous and corrode their containers.

It is proposed to address these problems by conversion of plutonium scrap and residue to a CRACHIP (CRITICALITY, Aerosol, and CHEMICALLY Inert Plutonium) glass using the Glass Material Oxidation and Dissolution System (GMODS). CRACHIP refers to a set of requirements<sup>2</sup> for plutonium storage forms that minimize ES&H storage and transport risks. Conversion of plutonium from complex chemical mixtures and variable geometries into a certified, qualified, homogeneous CRACHIP glass with fixed

dimensions in standard containers (1) creates a stable chemical form that minimizes ES&H risks, (2) simplifies safeguards and security (number count safeguards), (3) provides an easy-to-store form, (4) decreases storage costs, and (5) allows for future disposition options.<sup>3</sup>

GMODS is a new process<sup>4,5</sup> for directly converting scrap and residue into glass. Earlier glassmaking processes required that plutonium feed material first be a relatively pure oxide-like material before being converted to glass. This requirement implied a complex processing step to yield an oxide form acceptable for conventional glass melters. The technical and economic difficulties in conversion of plutonium scrap and residue to CRACHIP glass have been major barriers for this treatment option. The objective of GMODS development is to provide a low cost, technically feasible process to make CRACHIP glass.

### **A NEW APPROACH TO PLUTONIUM STORAGE AND DISPOSITION**

Before any option to treat plutonium scrap and residue can be undertaken, the requirements for the anticipated product must be defined. A CRACHIP glass must (1) be mechanically stable and must not form aerosols under storage or accident conditions, (2) be chemically inert, and (3) contain sufficient neutron poisons to prevent nuclear criticality with any quantity of material and/or any geometry. This addresses the near-term ES&H issues. This glass must also allow multiple disposition options: long-term storage, recovery of plutonium (with some difficulty), and disposal of plutonium as a waste. In the intermediate term, a CRACHIP form minimizes storage costs and ES&H storage risks, and simplifies safeguards.

The requirements and criteria for CRACHIP glass are similar to those required for high-level-waste (HLW) glasses. Radioactive wastes become less hazardous with time; hence, the fundamental concept in waste management is to isolate (store) these wastes until they are nonhazardous. Glass has been chosen worldwide as the preferred HLW storage and transport form because of several of its properties: (1) acceptance of impure feeds, (2) low solubility in water, (3) chemical inertness, (4) acceptable mechanical integrity, (5) ability to handle high heat loads from decay heat, and (6) avoidance of nuclear criticality by use of neutron poisons. The similar requirements of waste management and plutonium scrap and residue management provide the basis for defining performance requirements for CRACHIP glass: storage with performance equivalent to that of HLW glass.

Several groups are developing optimum compositions<sup>6,7</sup> for high-plutonium-loaded glass. For plutonium scrap and residue, traditional HLW glass compositions may also be modified for the plutonium and other components in the feed. In this case, plutonium is a minor component in the glass. Glass compositions must be optimized to accept both the plutonium and the other components in the feed.

Regardless of the long-term disposition of plutonium scrap and residue, storage is the only viable near-term option. This implies that the near-term incentive for conversion of plutonium scrap and residue to any storage form is to minimize storage costs. CRACHIP glass reduces the *storage requirements* and, in turn storage costs for plutonium scrap and residue by the following mechanisms:

- *Nuclear Criticality.* Plutonium is currently stored in vaults in small containers (traditionally <5 kg of plutonium per container) that are widely spaced to avoid nuclear criticality. CRACHIP glass with

neutron poisons eliminates criticality control as a vault requirement and thus reduces the vault size. In large vaults, most of the space is empty for geometric criticality control and can be eliminated if the material is stored as CRACHIP glass.

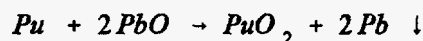
- *Volume Reduction.* Conversion of heterogeneous materials to a high-density, monolithic glass further minimizes storage costs.
- *Safeguards.* Conversion of highly heterogeneous materials to homogeneous glass in a standardized package allows for (1) more precise and reliable safeguards, (2) automated systems, and (3) number-count safeguards. This minimizes the costs of safeguards.

## THE PROCESS: GMODS

### Conversion Of Metals, Ceramics, Amorphous Solids, And Organics To Glass

GMODS converts plutonium and the other elements within the scrap and residue directly to borosilicate glass. GMODS is a batch process (Fig. 1) during which sequential process steps convert feeds to glass. The initial condition for the process is a melter filled with a molten oxidation-dissolution (lead borate) glass, which has a composition of 2 or more moles of lead oxide (PbO) per mole of boron oxide (B<sub>2</sub>O<sub>3</sub>). The PbO is a component of the glass and a sacrificial oxide. The process consists of the following steps:

- *Addition of feed material to the molten dissolution glass (Fig. 1.b).* The ceramic (plutonium oxide (PuO<sub>2</sub>), etc.) and amorphous components in the feed dissolve into the glass. While metals and organics do not dissolve into conventional molten glasses, the GMODS dissolution glass has special properties to process these materials *in situ*. The inclusion of the sacrificial oxide—PbO—in the molten glass provides a method to oxidize *in situ* (a) metals to metal oxides and (b) organics to carbon dioxide (CO<sub>2</sub>) gas and steam. When plutonium or another metal is fed to the melter, it is converted to a metal oxide. These metal oxides dissolve into the glass; carbon oxides (in gaseous form) and steam exit the melter. The reaction product, molten lead, separates from the glass and sinks to the bottom of the melter to form a separate layer,



- *Addition of glass additives [silicon oxide (SiO<sub>2</sub>) etc.] to improve the product quality (Fig. 1.c).* The optimum compositions of glasses for rapid oxidation-dissolution of materials in molten glass are different in composition from those for long-term durability; thus, additives that create a more durable glass are introduced after feed oxidation-dissolution takes place.

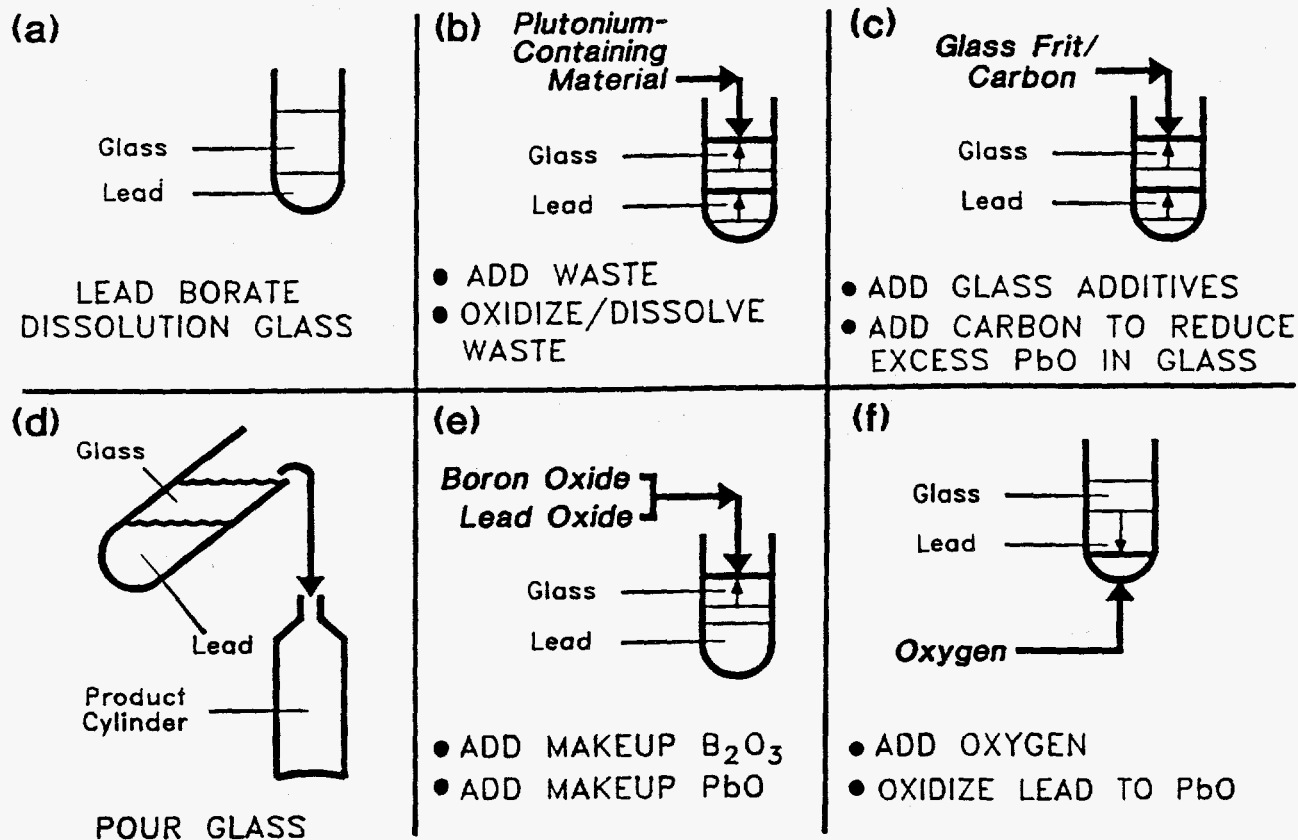
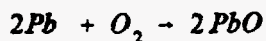
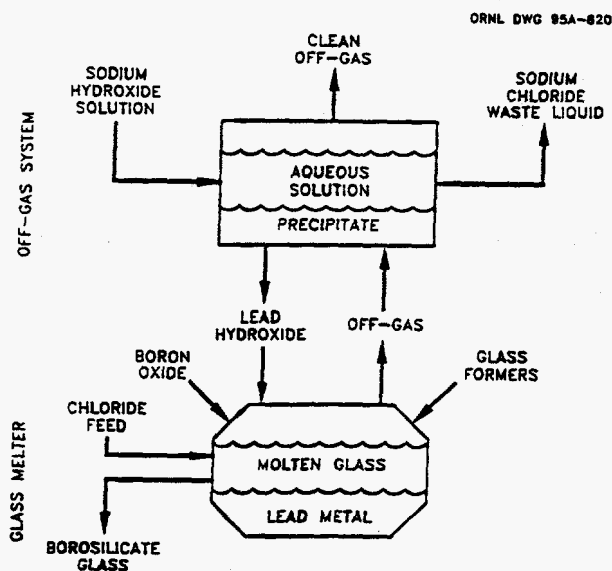


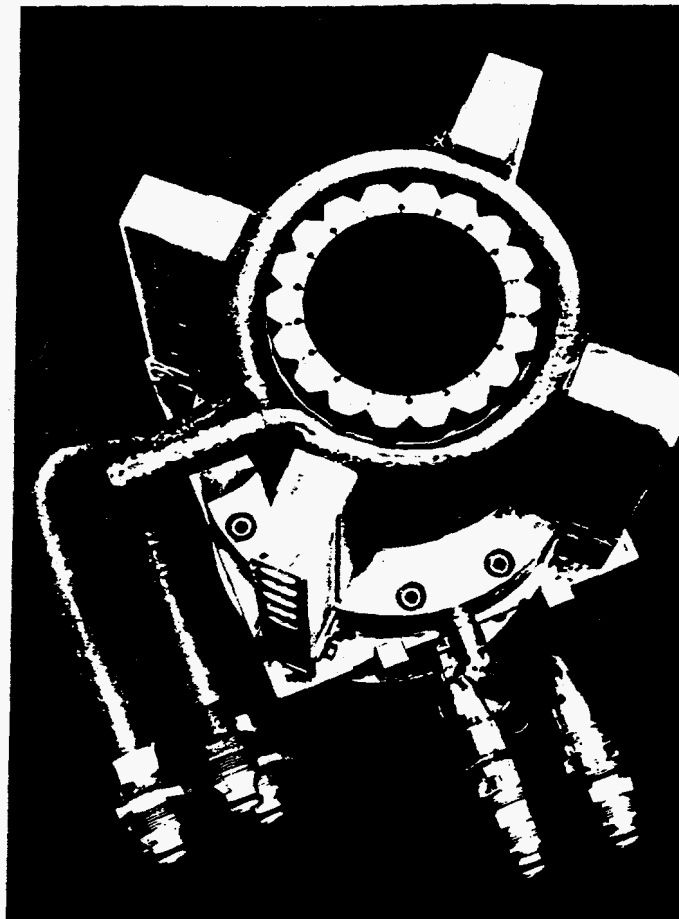
Fig. 1. GMODS batch processing of plutonium-containing material to borosilicate glass.

- *Addition of carbon to remove excess PbO (Fig. 1.c).* Carbon reduces the PbO to lead metal while producing gaseous  $CO_2$ . Excess PbO is removed from the dissolution glass for multiple reasons: (1) more durable glass, (2) reduction of the volume of glass, and (3) avoidance of the costs to provide added sacrificial PbO. The final glass may contain some or no lead, depending on the final desired glass composition.
- *Pouring glass from the furnace followed by solidification (Fig. 1.d).*
- *Addition of  $B_2O_3$  and PbO, as needed, to the melter for processing the next batch of materials (Fig. 1.e).*
- *Reoxidation of the lead at the bottom of the melter to PbO by addition of oxygen (Fig. 1.f).* This oxidation creates the new dissolution glass for the next batch of feed to be processed. Lead is an oxygen carrier that does not leave the system. The oxidation reaction is





**Fig. 2. GMODS processing of chloride-containing feed materials.**



**Fig. 3. Small commercial, cold-wall induction-heated melter (Courtesy of Consarc)**

### **Conversion Of Chlorides to Low-Chloride Borosilicate Glass And A Secondary Sodium Chloride Stream**

GMODS is designed to convert chloride-containing plutonium residues to glass and create a separate nonradioactive sodium chloride (NaCl) waste stream. Halogens, such as chloride, make poor-quality storage forms; hence, they must be separated from other components in plutonium residues. The analogy used in waste management is that good storage forms (silica, titanates, etc.) for radioactive materials can be found at any ocean beach. Materials that dissolve in seawater (chlorides, etc.) make poor storage forms.

The separation process for chlorides is shown in Fig. 2. In the dissolution glass, chlorides in the feed form lead chloride ( $PbCl_2$ ), which is volatile at glass melter temperatures and exits to the aqueous sodium hydroxide (NaOH) scrubber. In the scrubber, the  $PbCl_2$  reacts with the NaOH to yield insoluble lead hydroxide [ $Pb(OH)_2$ ] and soluble NaCl salt. The insoluble  $Pb(OH)_2$  is recycled back to the melter where it decomposes to PbO and steam, while the aqueous salt stream (NaCl) is cleaned and discharged as a chemical waste.

## **Conversion Of Heterogeneous, Poorly Characterized Feed Materials To High-Quality Homogeneous Glass**

A certified, qualified, high-quality, homogeneous glass product is required. Thus, an approach similar to that used to produce speciality metals and glass is used, where scrap and residue are fed to the melter and a homogeneous molten glass solution is produced. With a homogeneous glass solution, composition can be determined by limited sampling using mass spectrometric analysis. From the chemical analysis, the required compositions of additives can be determined to produce an appropriate product glass. This strategy, which depends on the ability to create a homogeneous glass from poorly characterized feed materials, is achieved by two mechanisms:

- GMODS can accept wide variations in the chemical composition of the feed and convert the materials to a homogeneous glass. This capability is a prerequisite because it avoids the need for detailed sampling of feed materials to ensure processability.
- The GMODS melter uses process tomography instrumentation<sup>8,9</sup> to determine in real-time when a homogeneous glass solution has been created. Homogeneous solutions imply homogeneous radiation fields that instrumentation can detect. With variable feeds, dissolution times will vary widely. Instrumentation ensures homogeneous feeds without requiring that tests be conducted on every feed to determine required dissolution times.

## **EQUIPMENT**

The primary GMODS equipment is an induction-heated, cold-wall melter (Fig. 3 shows a small commercial type), which is required because of the corrosive characteristics of the initial dissolution glass. Cold-wall melters have cooling jackets in the wall to produce a "skull" of solidified material that protects the wall from the melter contents. They are used to melt high-temperature materials (e.g., titanium and superalloys) and to produce ultrapure materials (e.g., glass for fiber optics). Russia, France, and the United States are modifying such equipment for processing various radioactive wastes. Batch size may be as large as several hundred kilograms for plutonium scrap and residue with low plutonium concentrations. In Europe, cold-wall melters are currently being developed for throughputs of up to 800 kg/h - far in excess of the size required for this mission.

## **STATUS OF DEVELOPMENT**

### **Investigations of Process Steps**

Some steps of the GMODS process are new, while others are parts of standard industrial processes. Experiments were performed to understand and prove the unique features of GMODS. Literature searches have been conducted to understand those parts of the process that are used in other industrial processes. Each step has also been accomplished in our laboratory.

Laboratory experiments were conducted in platinum and high-fired aluminum oxide crucibles within vertical tube furnaces. Platinum was used for experiments that did not involve lead (lead dissolves into platinum at high temperatures). Various ceramic crucible materials were investigated for use in oxidation process experiments. While the dissolution glass dissolves oxides, the rate of dissolution with Coors<sup>TM</sup> high-fired aluminum oxide crucible is sufficiently low for short-time experiments.

A typical experiment involved several hundred grams of material, with uranium and cerium being used as plutonium surrogates. Plutonium tests have been proposed. The plutonium content of scrap and residue is, at most, a few weight percent; hence, in terms of chemical processing, plutonium is a minor component.

*Addition of feed material to the molten dissolution glass (Fig. 1.b).* The addition of feed materials involves oxidation, dissolution, and mixing of feeds with the molten dissolution glass. Each of these steps has been investigated.

Tests demonstrated the dissolution of  $\text{UO}_2$ ,  $\text{ZrO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Ce}_2\text{O}_3$ ,  $\text{MgO}$ , and other oxides. The glasses were examined by a variety of methods to ensure complete dissolution. As expected, the high-boron oxide glass melt had good dissolution capabilities for oxides. In analytical chemistry,  $\text{B}_2\text{O}_3$  is the standard chemical reagent for fusion dissolution of unknown oxides because of its capability to dissolve such materials. Boron oxide is also the key component in many welding fluxes, which are used to dissolve iron oxides into a glassy slag during the welding process so that they are not incorporated into the weld.

Oxidation-dissolution tests demonstrated the oxidation of the following metals and alloys followed by the dissolution of their oxides into the melt: U, Ce, Zircaloy-2, Al, stainless steel, and other metals. Figure 4 shows cerium glass and lead by-product from a test of oxidation of cerium metal (plutonium surrogate).

Oxidation-dissolution tests also demonstrated the oxidation of carbon and graphite, with production of  $\text{CO}_2$ . For centuries, lead oxide has been used to oxidize organics<sup>10</sup>. It is the basis for the fire assay method for recovering noble metals (primarily gold) from silicate rock. Lead oxide, various organics, and silicate rocks are mixed together and heated. As the mixture melts, the lead oxide is reduced to metal by the organic. The noble metals in the molten mass then dissolve into the lead, which forms a separate layer that sinks to the bottom. This layer is then processed to separate the noble metal from the lead.

Limited chloride dissolution tests with  $\text{NaCl}$  demonstrated that lead exits the dissolution glass as  $\text{PbCl}_2$ , thus providing a separation of the chloride from other materials. This is a major mechanism for lead to escape from processes where lead and chlorides coexist at high temperatures.<sup>11</sup> The basic chemistry is well understood.

Experimental measurements were made of the viscosity of the dissolution glass with various added materials. Experience in the glass industry indicates that molten glass viscosities should be below 100 centipoise (about the viscosity of olive oil) for good mixing and creation of homogeneous glasses. Based on our experimental data, the GMODS dissolution glass temperature will need to be between 800 and 1000°C. The final processing temperature after addition of the silica will be above 1000°C because this addition increases glass viscosity.

*Addition of glass additives [silicon oxide ( $\text{SiO}_2$ ) etc.] to improve the product quality (Fig. 1.c).* This process step is essentially identical to that used for producing many specialty glasses.<sup>12</sup>



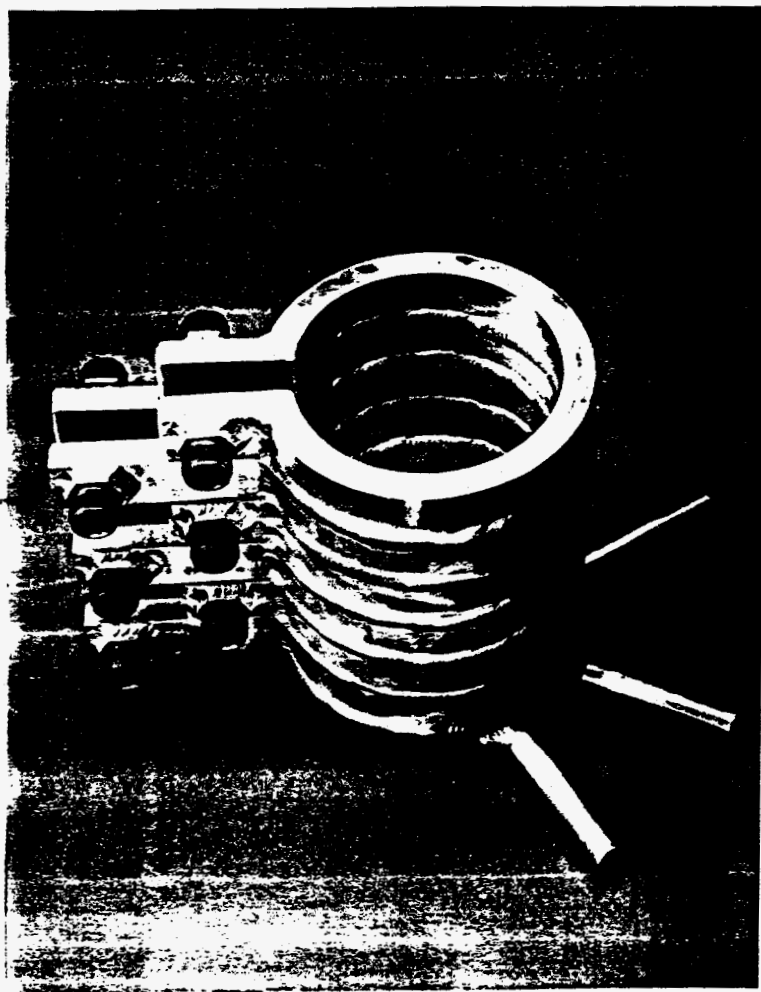
**Cerium Loaded Glass**



**Lead Reaction Product**

**Fig. 4. Cerium glass and lead metal from completed oxidation-dissolution test with cerium metal.**

**Fig. 5. ORNL single-turn, cold-wall, induction-heated glass melter.**



*Addition of carbon to remove excess PbO (Fig. 1.c).* This process step is used in several lead-smelting processes, such as the QSL process, to recover lead metal from lead oxide in molten slag.<sup>13</sup> This step has also been demonstrated with HLW glass in hot cells for recovery of fission product noble metals at Pacific Northwest Laboratory,<sup>14</sup> using a modification of the fire-assay method described above. Because some proposed plutonium glasses are variants of HLW glasses, this experience is particularly relevant.

*Pouring glass from the furnace followed by solidification (Fig. 1.d).* This is a standard operation used by the glass industry.

*Addition of B<sub>2</sub>O<sub>3</sub> and PbO, as needed, to the melter for processing the next batch of materials (Fig. 1.e).* This is a standard operation used by the glass industry for producing lead borosilicate glass (fine crystal).

*Reoxidation of the lead at the bottom of the melter to PbO by addition of oxygen (Fig. 1.f).* This is one of several processes used for producing lead oxide for batteries and other uses.<sup>15</sup>

### **Flowsheet Analysis**

An analysis of GMODS was performed using the process simulator FLOW.<sup>16</sup> The simulator includes a set of rules to choose glass compositions that meet process (viscosity, etc.) and performance requirements, using Savannah River HLW glass as a basis. The analysis identified critical process parameters when processing plutonium scrap and residue to a borosilicate glass, which is designed to be equivalent in performance to HLW glass. The two key observations were as follows:

- Incentives exist to maximize the GMODS operating temperature. For example, processing 1 kg of plutonium-containing chloride salt residues at 1103°C yields 6.5 kg of glass under standard conditions. Allowing the processing temperature to increase to 1167°C reduces the final glass quantities to about 3 kg. In this case, the waste loading in the glass is limited by the need to minimize molten glass viscosity during process operations to ensure good glass mixing. Increasing the processing temperature decreases the glass viscosity, minimizes the need to add sodium oxide to lower glass viscosity by changing the chemical composition, and allows a higher waste loading in the product glass. With the use of cold-wall, induction-heated melters that are not temperature-limited, the limitation on the maximum process temperature is volatilization of selected glass components.
- Incentives also exist to blend different feeds to minimize glass volumes. For example, blending plutonium salt and ash residue streams and converting them to glass reduces the final volume of glass by about 50% as compared with separate conversion of the two materials to glass. Final glass volumes are minimized because the ash stream provides necessary silica and aluminum to the final product glass, while the chloride stream provides necessary sodium to the final product glass.

The flowsheet simulator also afforded a bounding estimate of glass quantities if scrap and residue at Rocky Flats were converted to glass by GMODS. For "lean" scrap, 232 m<sup>3</sup> of glass would be produced with an average plutonium content of 0.06%. For "rich" scrap, 34 m<sup>3</sup> of glass would be produced with

an average plutonium content of 2.4%. The actual quantities of glass may be significantly less when higher process temperatures are used.

The quantities of glass produced from processing plutonium scrap and residue are not determined by the plutonium (due to its low concentration) but, rather, by other components in the scrap or residue. The set of constraints includes: glass processing temperatures, solubility limits of specific elements in the product glass, and glass durability under repository conditions. If the goal is to minimize glass volumes in order to minimize storage or disposal volumes, selected pre-processing of some residues can be undertaken to minimize specific elements that most impact glass volumes. This choice involves a series of trade-offs between multiple processes. (Note that organics and chlorides in feeds have little impact on final glass volumes because GMODS is a separations, as well as a glassmaking process.)

### **Equipment**

In making a survey of cold-wall, induction-heated melters, the range of operating conditions was found to substantially exceed the potential range of GMODS operations. Industrial melters are designed to operate at up to 2500°C; some of them operate with molten metal and slag. Recently, a small experimental melter has been built in our laboratory (Fig. 5) to provide a better understanding of this technology. Tests of this melter are under way.

### **Development Perspective**

The analytical testing and laboratory development work have demonstrated each step required for GMODS and identified equipment, instrumentation, and other components required for GMODS. A significant effort, however, will be required to convert GMODS into an industrial technology. This effort will include a better understanding of the process, integration of process steps into a system, and development of equipment.

### **SUMMARY**

GMODS is a new process for the direct conversion of plutonium scrap and residue to CRACHIP glass. It is designed to (1) convert metals, ceramics, and amorphous solids to glass; (2) oxidize organics with conversion of residues to glass; and (3) convert chlorides into a chloride-free borosilicate glass and a secondary clean NaCl stream. GMODS is an enabling technology, since it creates new plutonium scrap and residue management options. Because these options address common national security, non proliferation, and ES&H concerns, they may be acceptable to both the United States and Russia. As a new technology, however, GMODS has significant technical uncertainties that must be resolved in additional studies.

### **DISCLAIMER**

The views expressed in this paper are those of the authors and do not necessarily reflect any biases, proposed actions, or decisions of the United States Government or any agency thereof.

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