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Advanced Waste Form and Melter Development for Treatment of Troublesome High-level Wastes

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Abstract

A number of waste components in US defense high level radioactive wastes (HLW) have proven challenging for current Joule heated ceramic melter (JHCM) operations and have limited the ability to increase waste loadings beyond already realized levels. Many of these "troublesome" waste species cause crystallization in the glass melt that can negatively impact product quality or have a deleterious effect on melter processing. Recent efforts at US Department of Energy laboratories have focused on understanding crystallization behavior within HLW glass melts and investigating approaches to mitigate the impacts of crystallization so that increases in waste loading can be realized. Advanced glass formulations have been developed to highlight the unique benefits of next-generation melter technologies such as the Cold Crucible Induction Melter (CCIM). Crystal-tolerant HLW glasses have been investigated to allow sparingly soluble components such as chromium to crystallize in the melter but pass out of the melter before accumulating. The Hanford site AZ-101 tank waste composition represents a waste group that is waste loading limited primarily due to high concentrations of Fe_2O_3 (with higher Al_2O_3). Systematic glass formulation development utilizing slightly higher process temperatures and higher tolerance to spinel crystals demonstrated that an increase in waste loading of more than 20% could be achieved for this waste composition, and by extension higher loadings for wastes in the same group.

Introduction

The US is currently vitrifying defense HLW at the Savannah River Site (SRS) in South Carolina and plans to vitrify defense wastes currently stored in underground tanks at the Hanford Site. To date, over 15 million liters of HLW has been treated and vitrified in the Defense Waste Processing Facility (DWPF) at SRS. Although, significant advances have been made to increase waste loading and throughput in the DWPF over the 17 years of operation, practical limits have been reached regarding waste loading. At the Hanford site, projections using models based on JHCM processing show that waste loading will be limited for several waste groups due to crystallization in the glass melt and the resulting negative impacts on glass quality or melter processing. Several chemical constituents have been identified in both SRS and Hanford wastes that challenge waste loading due to crystallization concerns. High Al_2O_3 and Na_2O concentrations in the wastes have the potential to increase nepheline ($NaAlSiO_4$) crystal formation in the glass as shown by Peeler et al. (2006). The formation of nepheline can have a detrimental impact on glass durability because nepheline crystals deplete the residual glass matrix of glass forming oxides Al_2O_3 and SiO_2 (Kim et al. 1995). Mika et al. (1997) showed that high iron oxide, nickel oxide and chromium oxide (as well as other metal oxides) lead to the formation of spinel phases in waste glasses. Jain et al. (1991) described how spinel phase formation can adversely affect JHCM processing by causing pouring problems. In severe instances, Jain et al. described how significant spinel and noble metal accumulation in the melter can cause electrical conductivity issues in a JHCM.

Efforts are underway at US DOE laboratories to better understand crystallization behavior in waste glasses and to identify means to preclude crystallization in the glasses or mitigate the deleterious effects of

crystallization. At Pacific Northwest National Laboratory, researchers have studied the behavior of crystals in the melt in an attempt to develop models that predict crystal formation and settling. Studies by Hrma (2010) and Matyas et al. (2010) resulted in increased understanding of the effects of nucleation, crystal size and crystal agglomeration on settling and accumulation of crystals in the melt. The terminal objective of this work is to develop "crystal tolerant" glass formulations that can be processed using the JHCM technology at increased waste loadings allowing crystals to form in the melter and harmlessly pass from the melter without impacting melter operations. A second approach being evaluated is to leverage advances in melter technologies that allow for higher processing temperatures and improved crystal tolerance. The CCIM affords the opportunity to significantly increase processing temperatures. The CCIM melts glass inductively by creating an eddy current inside the glass pool using a water-cooled, high-frequency electrical coil that surrounds the outer walls of the CCIM. Due to a steep temperature gradient that exists near the walls, a frozen glass layer forms along the inside walls of the CCIM that separate the walls and the heating coil from the molten glass, thus, minimizing corrosion. Alternative to the CCIM, advances in JHCM technology using improved melter electrode materials will allow for modest increases in melter operating temperatures. Additionally, the introduction of bubblers into JHCMs can enhance mixing and temperature uniformity within the melter. Smith and Iverson (2011) describe the installation and operation of bubblers in the DWPF melter.

The Hanford site has developed system models called the Hanford Tank Waste Operations Simulator (HTWOS) to assist in planning for future plant operations (Bergmann 2010). These models include modules that sequence the various wastes and predict glass quantities using existing glass composition property models and relationships to specify waste loading in the glass (Vienna et al. 2009 and McCloy et al. 2010). There are also the baseline Waste Treatment and Immobilization Plant (WTP) glass property models and constraints that were developed for initial operation of WTP (Vienna and Kim 2008). The WTP constraints are more conservative than those used in HTWOS and hence typically project larger quantities of glass. The high Fe_2O_3 waste group represents about 1300 MT out of total waste of 11,000 MT at Hanford on a calcined oxides basis. The WTP baseline model predicts that approximately 3100 MT of glass will result from processing the waste group while the HTWOS model predicts about 3000 MT of glass will be produced. The AZ-101 tank waste is representative of the high Fe_2O_3 waste group. The AZ-101 tank waste has relatively high concentrations of Fe_2O_3 (38 wt %) and Al_2O_3 (25 wt %) and, thus, can be waste loading limited due to spinel or nepheline formation. This waste also includes high ZrO_2 concentrations (11%) that can lead to further crystallization concerns.

In the first phase of this Coordinated Research Project (CRP) glass formulation development activities focused on the AZ-101 composition to demonstrate increased waste loading (over the baseline maximum of 37 wt %) by taking advantage of higher processing temperatures and crystal tolerance afforded by the CCIM technology. The results of a case study with the AZ-101 composition are reported in this report. Future CRP efforts will focus on CCIM processing with the AZ-101 waste and will develop glass formulations to increase waste loading in similarly challenging HLW streams.

Experimental

Scoping glass composition development tests were initially conducted using the AZ-101 waste composition (Table 1) with concentrations of up to 20 wt % Fe_2O_3 with melting temperatures ranging from 1150 to 1300° C. Glass former additives included B_2O_3, Li_2O, Na_2O and SiO_2. Based on these initial results, a series of compositions with Fe_2O_3 concentrations ranging from 15-17 wt % were formulated. A subset of the compositions evaluated is provided in Table 2. The glasses were fabricated at 1250° C in platinum crucibles using reagent grade chemicals. It should be noted that a melting temperature of

Table 1. Composition of AZ-101 Waste Surrogate (wt %)

Component	Concentration
Al_2O_3	24.58
CaO	1.40
CdO	2.16
Ce_2O_3	0.80
Cr_2O_3	0.46
Cs_2O	0.50
Fe_2O_3	37.67
La_2O_3	0.89
MnO	0.91
Na_2O	10.58
Nd_2O_3	0.65
NiO	1.66
P_2O_5	1.34
RuO_2	0.15
SiO_2	3.77
SnO_2	0.66
SO_3	0.38
ZrO_2	11.44

Table 2. Targeted Glass Compositions for Select AZ-101 Glasses (wt %)

	AZ-10	AZ-16	AZ-17	AZ-18	AZ-29	AZ-30	AZ-31	AZ-32	AZ-33
Al2O3	11.09	10.44	11.09	11.09	9.79	9.79	9.79	9.79	9.79
B2O3	11.00	11.00	14.00	14.00	11.00	11.00	7.00	15.00	11.00
CaO	0.63	0.60	0.63	0.63	0.56	0.56	0.56	0.56	4.00
CdO	0.98	0.92	0.98	0.98	0.86	0.86	0.86	0.86	0.86
Ce2O3	0.36	0.34	0.36	0.36	0.32	0.32	0.32	0.32	0.32
Cr2O3	0.21	0.20	0.21	0.21	0.18	0.18	0.18	0.18	0.18
Cs2O	0.23	0.21	0.23	0.23	0.20	0.20	0.20	0.20	0.20
Fe2O3	17.00	16.00	17.00	17.00	15.00	15.00	15.00	15.00	15.00
K2O	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
La2O3	0.40	0.37	0.40	0.40	0.35	0.35	0.35	0.35	0.35
Li2O	3.00	3.00	3.00	3.00	3.00	5.00	4.50	3.00	3.00
MnO	0.41	0.38	0.41	0.41	0.36	0.36	0.36	0.36	0.36
Na2O	10.78	11.38	8.64	10.78	11.99	7.50	11.99	9.25	9.12
Nd2O3	0.29	0.28	0.29	0.29	0.26	0.26	0.26	0.26	0.26
NiO	0.75	0.70	0.75	0.75	0.66	0.66	0.66	0.66	0.66
P2O5	0.60	0.57	0.60	0.60	0.53	0.53	0.53	0.53	0.53
RuO2	0.07	0.06	0.07	0.07	0.06	0.06	0.06	0.06	0.06
SiO2	36.59	38.26	35.73	33.59	39.93	42.42	42.43	38.67	39.35
SO3	0.17	0.16	0.17	0.17	0.15	0.15	0.15	0.15	0.15
SnO2	0.30	0.28	0.30	0.30	0.26	0.26	0.26	0.26	0.26
ZrO2	5.16	4.86	5.16	5.16	4.55	4.55	4.55	4.55	4.55
WL, %	45.1	42.5	45.1	45.1	39.8	39.8	39.8	39.8	39.8

1250° C would be easily achievable in a CCIM. After fabrication, a portion of the glass was subjected to a canister centerline cooling (CCC) heat treatment intended to mimic the most extreme slow cooling profile that would be expected in glass fabricated in the plant. The glasses were analyzed using x-ray diffraction (XRD) and scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) to assess crystallinity within the glasses in the as-fabricated state and after the CCC heat treatment. Isothermal heat treatments were used to provide a measure of the equilibrium crystal fraction vs. temperature. The liquidus temperature associated with spinel crystallization (T_L) was estimated by extrapolating the lower temperature data to 0 vol % crystal content. The Product Consistency Test – Method A (PCT-A) was used to assess the relative durability of the glasses in both the as-fabricated condition and after being subjected to the CCC heat treatment. The PCT-A is an ASTM standard test method (2002) used as a benchmark test to determine glass repository acceptability. To confirm that the as-fabricated glasses corresponded to the defined targeted composition, a representative sample was chemically analyzed. Chemical content was analyzed by means of two dissolution techniques, sodium peroxide fusion (PF) and lithium-metaborate (LM) fusion both followed by acid dissolution. The resulting dissolved samples were analyzed by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES).

Results and Discussion

Visual observations of the glasses identified in Table 2 showed that the glasses were essentially crystal free after fabrication. After the glasses were subjected to the CCC treatment, XRD scans indicated the presence of a magnetite-type spinel crystalline phase in all of the samples. Baddeleyite phase was observed in the higher waste loading glasses (AZ-10, AZ-16, AZ-17 and AZ-18). The XRD scans showed that no nepheline was present in any of the glasses after the CCC treatment. The lack of nepheline reduces the risk that the chemical durability of the CCC glasses would be significantly worse than that of the quenched glasses.

The isothermal heat treatments showed that in general the volume percent of crystals decreased as the heat treatment temperature increased (Figure 1). An artifact of the testing was identified for some glasses in that at the higher heat treatment temperatures used (1200 and 1250° C), very fine spinel crystals formed during quenching the samples, i.e., they were not present at the heat treatment temperature but formed during cooling. Figure 1 also shows that crystal content also generally increased as waste loading increased.

Table 3 provides the normalized elemental releases for B, Li and Na by PCT-A. These results show that all glasses exhibited lower normalized elemental releases than the Environmental Assessment (EA) glass by at least one order of magnitude. The EA glass is a standard glass used for qualifying US HLW glasses for repository disposal as reported by Jantzen et al. (1993). The results also indicated that CCC heat treatment had no significant effect on the PCT-A responses of the glasses, as expected. Interestingly, the normalized releases were also essentially equivalent irrespective of waste loading.

Based on total crystal vol % versus temperature data in Figure 1, the three glasses with 17 wt%, 16 wt%, and 15 wt% Fe_2O_3, CCIM-AZ-10, 16, and 29, respectively, were identified as candidates for initial CCIM demonstration testing. Figure 2 displays the total crystal vol % as a function of temperature for the selected three glasses. It was evident that the estimated crystal content increased as waste loading increased in these three glasses. At a processing temperature of 1250° C, these 3 glasses provide a range of crystal concentrations that could adequately assess the ability of the CCIM to process crystal laden melts. Furthermore, the viscosity and electrical conductivity of these formulations were all measured to be satisfactory at a target T_M=1250°C to support CCIM processing.

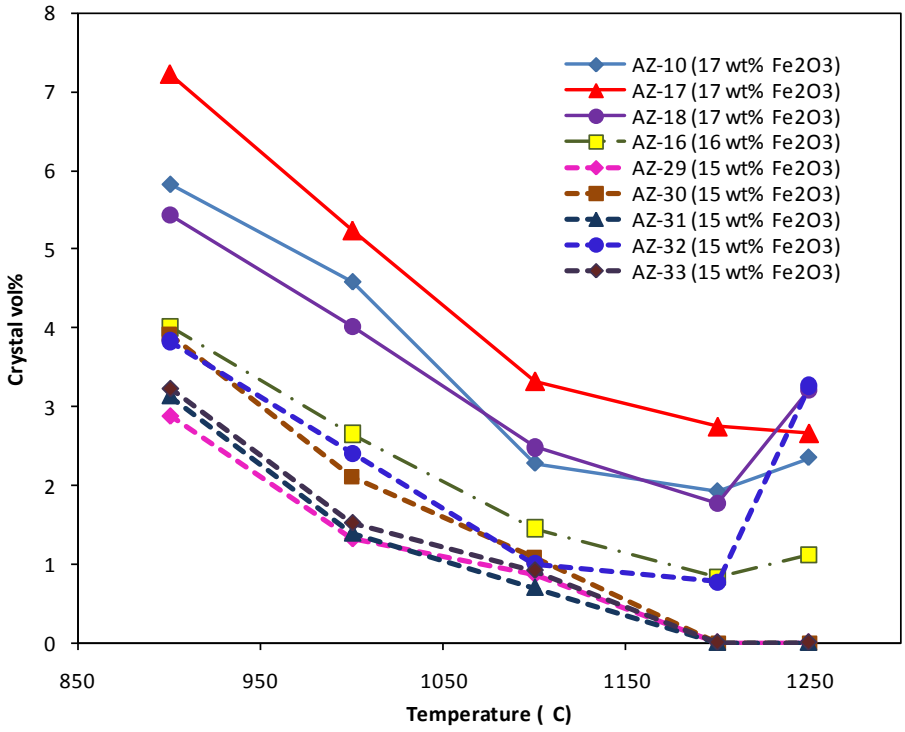

Figure 1. The volume percent of crystals in select AZ-101 HLW glasses as a function of heat treatment temperature. Note that some glasses erroneously showed an increase in crystal content at 1250° C due to spinel crystals forming during cooling from the heat treatment temperature.

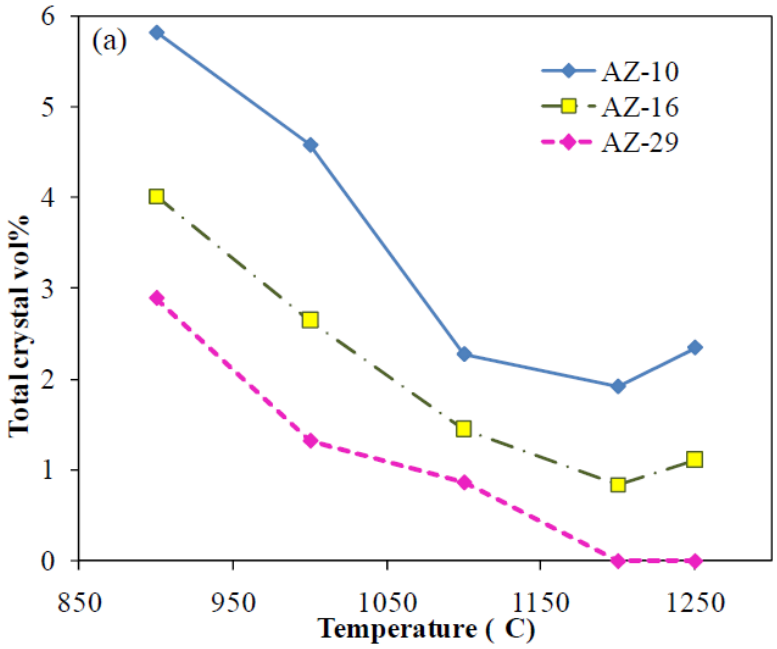

Figure 2. The volume percent of crystals for the three glasses identified as candidates for CCIM testing.

Table 3. Liquidus Temperature and PCT Normalized Elemental Releases for AZ-101 Glasses

	Glass Composition								
Normalized Elemental Release (g/L)	**AZ-10**	**AZ-16**	**AZ-17**	**AZ-18**	**AZ-29**	**AZ-30**	**AZ-31**	**AZ-33**	**EA (Jantzen 1993)**
B (quenched)	0.48	0.48	0.57	0.75	0.48	0.53	0.48	0.48	16.70
B (CCC)	0.44	0.44	0.73	1.08	0.43	0.44	0.47	0.33	--
Na (quenched)	0.40	0.43	0.35	0.55	0.39	0.26	0.48	0.41	13.35
Na (CCC)	0.39	0.39	0.42	0.70	0.37	0.21	0.46	0.30	--
Li (quenched)	0.63	0.65	0.80	0.81	0.62	0.69	0.60	0.65	9.56
Li (CCC)	0.56	0.54	0.91	0.97	0.53	0.60	0.55	0.47	--
T_L (spinel) (°C)	1398	1330	1311	1633	1146	1168	1132	1196	--

The results of the glass formulation development effort with the tank AZ-101 composition demonstrated that increased waste loading could be achieved with increasing crystal content for a given temperature. The higher operating temperature and apparent tolerance of the CCIM to crystallization within the melt could allow for processing the glass with up to 45.1 wt % waste loading (equivalent to 17 wt % Fe_2O_3) resulting in over a 20% improvement in waste loading over the current maximum projected waste loading of 37 wt % for AZ-101.

Summary for this Phase of CRP

Several "troublesome" US defense HLW chemical constituents may lead to crystallization within the melt when waste loadings are increased. The formation of these crystals can negatively impact product quality or have a deleterious effect on melter processing. Therefore, waste loading is limited due to these waste components. Efforts are underway at US national laboratories to develop "crystal tolerant" glasses that control crystallization within the melt allowing crystals to harmlessly pass from the melter. Other efforts are attempting to leverage advances in glass melter technologies to develop glass formulations that take advantage of increased melter processing temperatures, thus, leading to increased waste loading.

A case study was conducted using the Hanford tank AZ-101 waste composition (representative of a high Fe_2O_3 waste group with high Al_2O_3) to demonstrate that higher melt temperature and higher crystal content glass compositions could be developed to significantly increase waste loading yet meet product quality requirements. The results showed that waste loading increases of over 20% could be realized for this waste group by processing at temperatures that could be readily achieved using a CCIM. Extrapolating these higher waste loading results to model projections used in Hanford site operations planning, indicated that for the high Fe_2O_3 waste group approximately 600 MT less glass would need to be produced.

Future Work

CCIM Testing

The successful glass formulation development effort with the AZ-101 Hanford waste simulant support CCIM testing to demonstrate the significant waste loading increases that can be achieved using advance glass compositions in combination with the CCIM technology. The demonstration testing will be conducted on the Idaho National Laboratory CCIM test platform.

The Idaho National Laboratory (INL) CCIM platform is comprised of liquid and solid feed systems, melter system, cooling system and off-gas system (Figure 3). The slurry feed system employs a feed tank equipped with a stirrer, sparger and recirculating loop. The slurry is fed to the melter using a recirculating pump that meters feed to the melter and recirculates excess feed to the feed tank. The 267 mm inner diameter CCIM vessel is constructed from 304L stainless steel and consists of a lower manifold, upper manifold and cooling tubes. The cooling tubes are oriented vertically to form the crucible cylinder. The induction power system consists of a variable high frequency generator that can operate in frequency ranges from 200-400 kHz and 1.5-4 MHz to produce a maximum power of 75 kW (however, overpower interlocks limit the maximum power to 60 kW). A conductive metal (copper) cage encloses the melter and induction coil system to minimize worker exposure to electromagnetic radiation. Figure 4 provides a view of the INL CCIM melter in the protective metal cage. The molten glass is drained semi-continuously through a resistively heated bottom drain assembly. The off-gas system consists of a heated duct to the thermal reaction chamber, thermal reaction chamber, off-gas quench section, wet scrubber system and induced draft fan. The off-gas system is specifically designed to facilitate off-gas sampling making the INL CCIM system well suited for melter off-gas characterization tests.

The objective of the AZ-101 melter test campaign will be to demonstrate CCIM operations over an extended period (in excess of 72 hours). Initially, the AZ-16 composition will be targeted because the crystal content is in the middle of the range of the candidate glasses that were identified. Melter parameters will be monitored over the duration of the testing. The glass will also be characterized to determine chemical composition and relative glass quality as measured by the PCT.

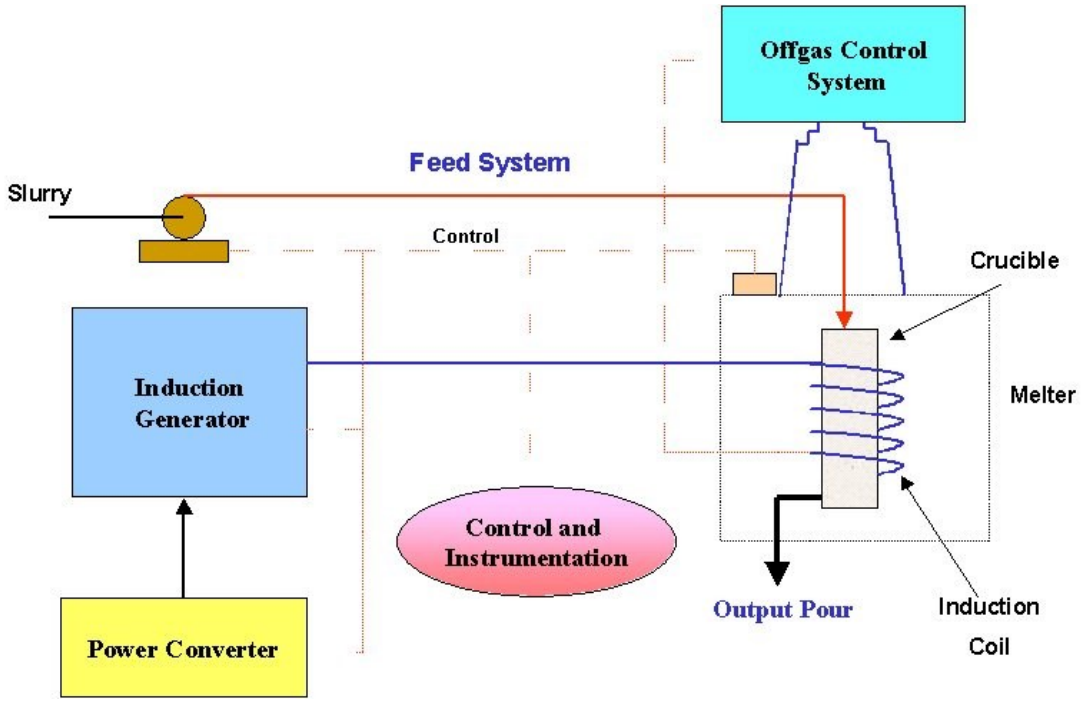

Figure 3. Simplified schematic diagram for the INL CCIM pilot-scale platform. (Soelberg 2009)

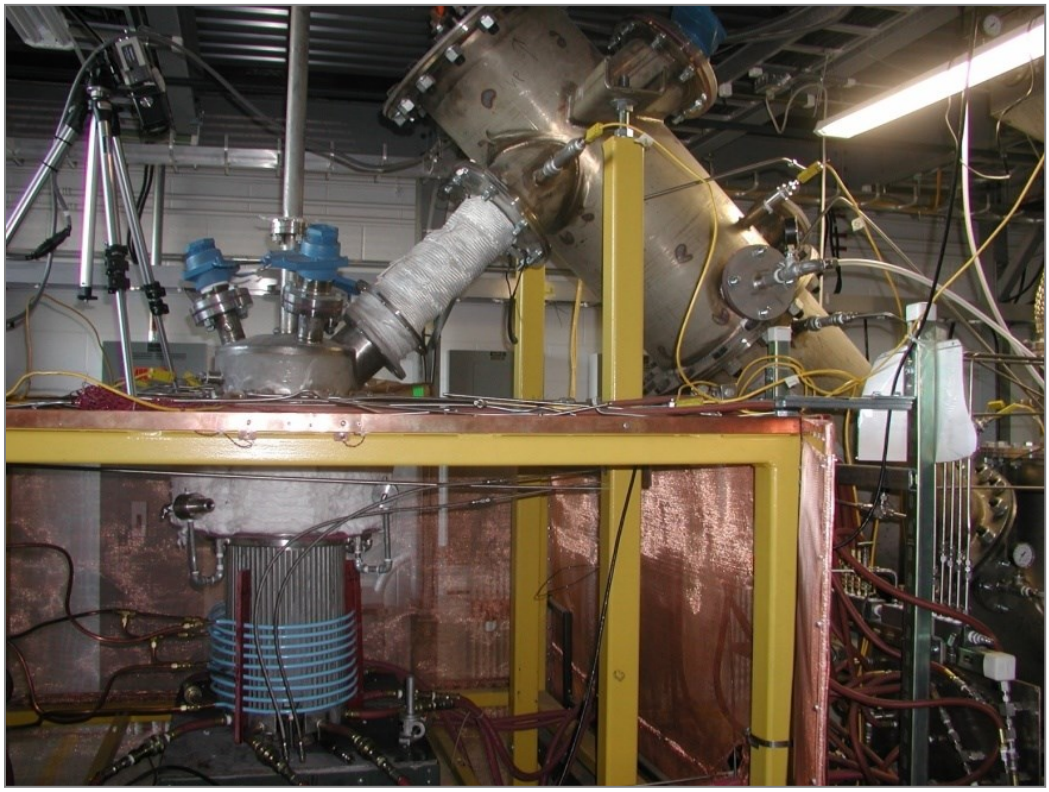

Figure 4. INL CCIM melter in the copper protective metal cage (INL 2011).

Glass Formulation Development

The selection of wastes for the glass formulation and testing efforts to demonstrate increased waste loading are focused on Hanford waste streams because they are likely to show the highest cost benefit to implementation, considering the size and cost of the Hanford tank waste cleanup program and the timing of startup. The AZ-101 glass composition development effort was the first Hanford HLW stream evaluated. Several tank wastes have been recently identified that provide further challenges to waste loading due to unique characteristics of the wastes.

Settling of plutonium-bearing particles > 10 µm in WTP process vessels is a concern for the WTP pretreatment facility (PTF) (Sams 2012). Recently it was estimated that approximately 30 kg of plutonium present in the tank farms was delivered as >10 µm particulate Pu oxide and Pu metal. This inventory is located in 16 tanks; eight with minimal quantities, and eight with appreciable quantities that could challenge the Criticality Safety Evaluation Report (CSER). The eight tanks with appreciable quantity (> 750 g) are TX-105, TX-109 and TX-118, 244-TX, SY-102, C-102, AN-101, and S-108.

Alternative treatment approaches for these tanks with safety concerns for processing within the WTP PTF are being considered. Direct vitrification using a near-tank CCIM unit is a strong candidate.

The tank wastes 244-TX and C-102 were identified for glass formulation development for CCIM processing for the following reasons. The 244-TX composition has an exceedingly high iron oxide concentration (>59 wt%). This high iron concentration would significantly limit waste loading using the current JHCM technology with a process temperature limit of 1150° C and relatively low crystal tolerance. The C-102 composition has a high Al_2O_3 concentration (>60 wt%). The high alumina

concentration in the waste limits waste loading due to the propensity to form nepheline and spinel in glasses with high alumina contents. These wastes also represent Hanford HLW composition families in general. Therefore, the information gained from this study may also be helpful for future glass formulation development efforts for Hanford HLWs in general. Table 4 shows the composition of these two wastes after adjusting the full composition by removing radioactive components (UO_3), by replacing PuO_2 and ThO_2 with ZrO_2 and lanthanide and actinide oxides with La_2O_3 (molar equivalent), and by removing minor components with <0.01 wt%. Each waste is also spiked with Cs_2O to ensure adequate analysis of this key volatile component.

Table 4. Composition of 244-TX and C-102 Waste Surrogates (wt %)

Comp.	244-TX	C-102
Ag_2O	0.01	--
Al_2O_3	0.81	62.5
As_2O_5	0.07	--
Bi_2O_3	--	0.32
CaO	0.37	1.10
Cl	0.38	0.21
Cr_2O_3	0.23	0.11
Cs_2O	0.20	0.20
F	0.06	0.49
Fe_2O_3	60.8	3.01
K_2O	0.94	0.17
La_2O_3	0.02	0.02
MgO	3.36	--
MnO	--	0.23
Na_2O	31.5	17.1
NiO	--	0.97
P_2O_5	0.38	1.46
PbO	0.04	0.21
SiO_2	--	8.96
SO_3	0.83	0.71
SrO	--	0.02
ZrO_2	--	2.21
SUM	100	100

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