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INTERNATIONAL STUDIES OF ENHANCED WASTE LOADING AND IMPROVED MELT RATE FOR HIGH ALUMINA CONCENTRATION NUCLEAR WASTE GLASSES

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ABSTRACT

The goal of this study was to determine the impacts of glass compositions with high aluminum concentrations on melter performance, crystallization and chemical durability for Savannah River Site (SRS) and Hanford waste streams. Glass compositions for Hanford targeted both high aluminum concentrations in waste sludge and a high waste loading in the glass. Compositions for SRS targeted Sludge Batch 5, the next sludge batch to be processed in the Defense Waste Processing Facility (DWPF), which also has a relatively high aluminum concentration. Three frits were selected for combination with the SRS waste to evaluate their impact on melt rate. The glasses were melted in two small-scale test melters at the V. G. Khlopin Radium Institute. The results showed varying degrees of spinel formation in each of the glasses. Some improvements in melt rate were made by tailoring the frit composition for the SRS feeds. All of the Hanford and SRS compositions had acceptable chemical durability.

INTRODUCTION

The United States Department of Energy (DOE) is currently processing (or planning to process) high-level waste (HLW) through Joule-heated melters at the Savannah River Site (SRS) and the Hanford Site. The process combines the HLW sludge with prefritted or mined mineral glass forming additives that are subsequently melted. The molten glass is poured into stainless steel canisters to create the final waste form. In preparation for the qualification and receipt of each sludge batch, development and definition of various tank blending and/or washing strategies have been or will be initiated. The various strategies are contemplated in an effort to meet critical site objectives or constraints that include tank volume space, transfer options, and settling issues. Although these objectives or constraints are critical, the ability to meet both process and product performance criteria associated with the final waste form must also be maintained. The product performance issues relate to the durability of the glass waste form. Process related issues (e.g., liquidus temperature, viscosity, electrical conductivity, and melting rate) ultimately dictate the efficiency and effectiveness of the melter operation.

Tank retrieval and blending strategies at both SRS and Hanford have identified high Al_2O_3 concentration waste streams that are scheduled to be processed through their respective

HLW vitrification facilities. For example, the Liquid Waste Organization (LWO) at SRS provided compositional projections for the next two sludge batches (Sludge Batch 4 and Sludge Batch 5) to be processed in the Defense Waste Processing Facility (DWPF). These streams have Al_2O_3 concentrations of approximately 25-40 wt% (on a calcined oxide basis). In addition, physical limitations in the Tank Farms and/or settling issues associated with the sludge coupled with the need to maintain feed for DWPF have prevented advanced washing which has resulted in relatively high Na_2O (approximately 22-26 wt%) concentrations. Current Hanford projections suggest that Al_2O_3 concentrations in sludge could be much greater than those currently projected for DWPF, with Al_2O_3 concentrations as high as 80 wt%.

While it is well known that the addition of small amounts of Al_2O_3 to borosilicate glass generally enhances the durability of the waste form (through creation of network-forming tetrahedral Na^+-$[AlO_{4/2}]^-$ pairs), nepheline ($NaAlSiO_4$) formation, which depends in part on the Al_2O_3 concentration, can result in a severe deterioration of the chemical durability of the glass through residual glass compositional changes. The primary driver for this reduction in durability is that nepheline removes three moles of glass forming oxides (Al_2O_3 and $2SiO_2$) from the continuous glass phase per each mole of Na_2O consumed. Nepheline formation produces an Al_2O_3 and SiO_2 deficient continuous glass matrix (relative to the same composition which is void of crystals), which reduces the durability of the final product. The magnitude of the reduction ultimately depends on the extent of crystallization and on the initial glass composition.

The formation of nepheline and/or other aluminum/silicon-containing crystals is a potential for both DWPF and Hanford due to the projected compositional views recently evaluated coupled with the frit development strategy (e.g., higher alkali frits have lead to enhanced melt rates at DWPF for those sludge batches). Although durability is obviously a critical constraint that the HLW glass must meet, other process-related issues must also be considered. Additionally, the inclusion of higher concentrations of Al_2O_3 will generally increase the liquidus temperature of the melt and decrease the processing rate.

The objective of this task was to develop glass formulations for DOE waste streams with high aluminum concentrations to avoid nepheline formation while maintaining or meeting waste loading and/or waste throughput expectations as well as satisfying critical process and product performance related constraints. At SRS, frit compositions must be developed for high aluminum concentration wastes that will avoid detrimental crystallization while enhancing melt rate. At Hanford, high aluminum concentrations have necessitated melter operation with a small volume of spinel crystallization in the melt pool. Both of these systems are evaluated in this study in partnership with the V.G. Khlopin Radium Institute (KRI) in St. Petersburg. Small-scale test melters at KRI were used to evaluate test glass compositions for both SRS and Hanford.

EXPERIMENTAL PROCEDURE

The Steklo Metallicheskie Konstruktsii (SMK) Melter System

The SMK melter system at KRI is a batch melter intended for experiments on the rate of melting (by monitoring cold cap consumption) and crystal accumulation (through sectioning of the melt crucible) of simulated HLW glass. The components of the SMK melter system make it possible to provide for a dry or liquid feeding, off-gas treatment, air bubbling, glass product pouring, and vitrification temperature monitoring, with a continuous stirring of the feed. Fig. 1 shows a general layout of the SMK melter system.

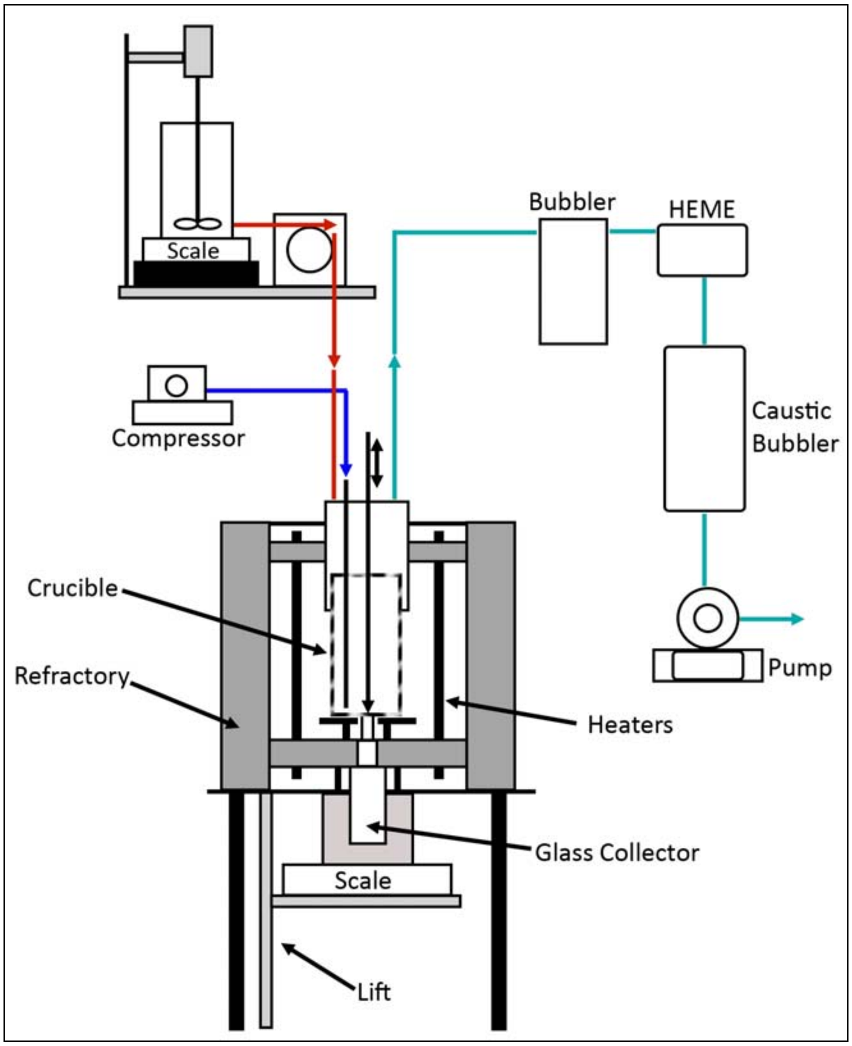


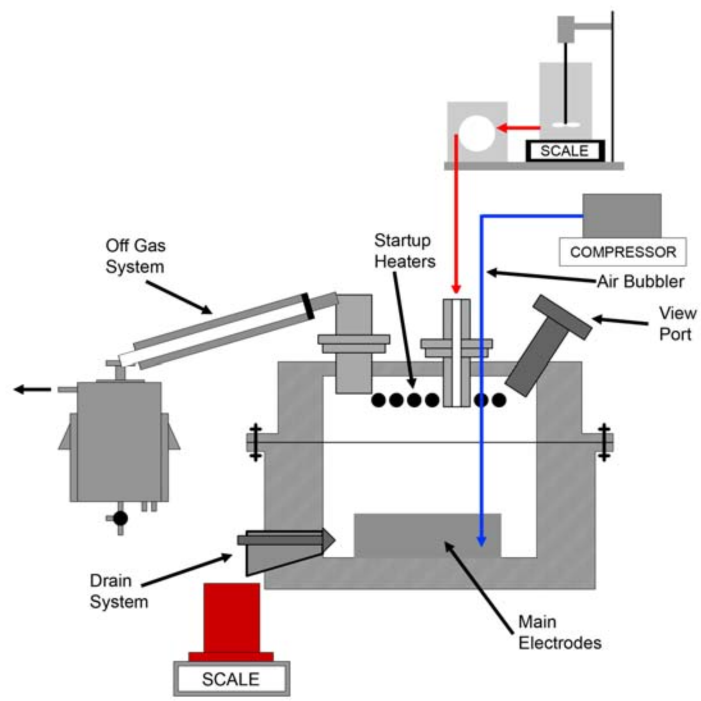
Figure 1. General Layout of the SMK Melter System.

Prior to an experiment, a quantity of melt startup product estimated to generate 500 g of glass was placed into the crucible. The composition of the melt startup product was the same as the glass composition to be evaluated. After the melter was heated and the melt startup product reached the target temperature (1150 ºC for these experiments), the bubbler was turned on 0.6 L/min air) followed by the start of liquid feeding. Note that bubbling was not used for the experiments with SRS feeds since the DWPF melter is not currently configured for bubbling. The feed tube was water cooled to minimize the possibility of plugging. At the completion of feeding and when the cold cap had been consumed, the bubbler was turned off. A portion of the melt was then poured into an insulated can and later characterized. After the final pour of an experiment, the crucible was removed from the melter for quenching the remaining glass to support an evaluation of crystallization within the melt pool.

The following parameters are monitored during the experiments: feeding rate, melt temperature, bubbling rate, voltage, and current. Visual observations are also performed. The melt temperature was measured by using a thermocouple submerged into the melt next to the crucible bottom, 3 cm away from the side wall.

The Electricheskaya Pech-5 (EP-5) Melter System

The EP-5 melter at KRI is configured for longer-term, continuous feeding and batch pouring. It is equipped with systems and mechanisms that provide for the following: dry or liquid feeding, off-gas treatment, air bubbling of the melt, pouring of the glass product, and temperature monitoring of the melting process. A schematic of the EP-5 melter system and the operational parameters of the melter are shown in Fig. 2.



Parameter	Value
Maximum Operating Temperature	1200 ºC
Melter Cavity Length	260 mm
Melter Cavity Width	130 mm
Melter Cavity Depth	210 mm
Total Glass Pool Surface Area	340 cm^2
Feed Area	229 cm^2
Melt Production Area	106.5 cm^2
Melter Cavity Height	285 mm
Average Glass Depth	110 mm
Average Glass Volume	3.7 L
Average Glass Mass	9.3 kg
Refractory Material	Bacor-33
Electrode Dimensions	200 mm x 75 mm
Maximum Current Density	2 A/cm^2
Maximum Current	300 A
Maximum Power	22 kW

Figure 2. Schematic and Operational Parameters of the EP-5 Melter System.

Prior to an experiment, a melt startup product consisting of crushed glass remaining from the SMK experiments was placed into the melter. The chemical composition of the startup product matched the targeted glass composition. After the melter was heated and the startup product was melted at 1150 ºC, the bubbler was turned on (for the Hanford glass only) followed by the start of liquid feeding. After a sufficient quantity of glass was generated, the melt was poured without interrupting the feeding.

Feed Selection and Preparation

KRI prepared the sludge simulants in accordance with recommendations provided by Savannah River National Laboratory (SRNL) and Pacific Northwest National Laboratory (PNNL). Details of the sludge stimulant preparation methodology are available elsewhere.[1, 2] Table I provides the Hanford simulated (i.e., no radioactive components) sludge, frit and glass compositions selected for this study. The glass composition is referred to as HAL-17 and represents an oxide waste loading of 48.6 wt % sludge in glass. The HAL-17 composition was selected based on its high waste loading, high alumina concentration and acceptable physical and chemical characteristics of the final product.

Table I. Composition of the HAL-17 Glass and Its Components (wt % calcined oxides).

Oxide	Sludge	Frit	Glass composition (48.6 wt % WL)
Al_2O_3	53.27		25.87
B_2O_3	0.42	31.0	16.14
BaO	0.12		0.06
Bi_2O_3	2.54		1.24
CaO	2.39	12.0	7.33
CdO	0.054		0.03
Cr_2O_3	1.16		0.56
F	1.48		0.72
Fe_2O_3	13.11		6.37
K_2O	0.31	5.0	2.72
Li_2O	0.38	7.42	4.0
MgO	0.26		0.13
Na_2O	7.96	4.28	6.07
NiO	0.89		0.43
P_2O_5	2.34		1.14
PbO	0.91		0.44
SO_3	0.44		0.22
SiO_2	10.88	40.30	26.0
TiO_2	0.022		0.01
ZnO	0.18		0.09
ZrO_2	0.88		0.43

KRI used two types of simulated sludge for the SRS feeds: Sludge 1 which represents a projected Sludge Batch 5 (SB5) without the implementation of the Al-dissolution process and Sludge 2 which represents a projected SB5 composition after the completion of Al-dissolution. Their compositions are given in Table II.

Table II. SRS Target Sludge Compositions (wt % calcined oxides).

Oxide	Sludge 1 (without Al-dissolution)	Sludge 2 (with Al-dissolution)
Al_2O_3	33.22	16.60
BaO	0.11	0.15
CaO	2.09	2.90
Cr_2O_3	0.20	0.28
CuO	0.07	0.11
Fe_2O_3	26.40	36.77
K_2O	0.16	0.22
MgO	1.41	1.96
MnO_2	6.33	8.87
Na_2O	24.40	24.36
NiO	2.31	3.23
P_2O_5	0.12	0.11
PbO	0.10	0.14
SO_3	0.96	1.35
SiO_2	1.82	2.53
ZnO	0.07	0.10
ZrO_2	0.23	0.32

Three frits were evaluated for combination with the simulated SRS sludges to evaluate the impact of frit composition on melt rate. The frit compositions are given in Table III. The process used to select these frit compositions is documented elsewhere.[3] The frits batched and then melted at 1250 ºC, with the melt being held at this temperature for 30 minutes. The frits were ground and sieved to a particle size of -80/+200 mesh (-177/+74 µm, respectively). Melter feed targeted a waste loading of 35 wt %.

Table III. Target Frit Compositions (as wt% oxides) for the SRS Feeds.

Frit	B_2O_3	CaO	Li_2O	Na_2O	SiO_2
503	14	0	8	4	74
517	17	0	10	3	70
520	8	1	10	4	77

Durability Testing

The Product Consistency Test (PCT), Method A[4] was performed in triplicate on samples of the glasses resulting from the melter runs to assess their chemical durability, which is a critical HLW glass performance criterion for acceptance into the federal repository. Included in the experimental test matrix was the Environmental Assessment (EA) glass, which is used for comparison to assure acceptable durability.[5] Glass samples were ground, washed, and prepared according to the ASTM procedure. At the completion of the 7-day test, the solutions were sampled (filtered and acidified) and analyzed by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES). Samples of a multi-element standard solution were included in the analyses as a check on the calibration of the ICP-AES instrument. Normalized release rates were calculated based on the target or measured (when available) glass compositions.

RESULTS AND DISCUSSION

SMK Experiments with Hanford Feeds

KRI performed three runs using the Hanford HAL-17 composition in the SMK melter. The feeding took 3 hours in the first run, 7 hours in the second run, and 11.2 hours in the third run, all at a temperature of 1150 ºC. The poured glass distributed evenly in the can, with a sink hole forming on the surface upon cooling. KRI sampled the glass from both the can and the crucible upon completion of the runs. The samples were taken from upper, middle, and bottom part of the can. The glass samples were analyzed using scanning electron microscopy (SEM) for identification and volume fraction determination (using image analysis) of the crystalline phases.

A summary of the feeding rates and the amount of glass produced during each melter run is provided in Table IV. The feeding rate was reduced in order to achieve longer feeding times since the first run showed that the melter would not be able to handle evaporation if operated at higher feeding rates for an extended time. The glasses appeared heterogeneous when poured.

Table IV. Summary of SMK Melter Runs for Hanford Feeds

Run Number	Feeding Rate (L/hour)	Feeding Time (hours)	Number of Pours	Mass of Glass Poured (g)
1 (1150 °C)	1.23	3	1	1415
2 (1150 °C)	0.53	7	2	1420
3 (1150 °C)	0.48	11	4	2035

SEM analyses of the glasses from each of the three runs showed that spinels were present in all areas of the poured glass as well as in the glass remaining in the crucible after the final pour at concentrations of about 1-4 vol %. An example of the measured spinel distribution for Run 3 is given in Table V. Increasing the residence time in the melter appeared to have some influence on reducing the spinel content in the poured glass. There may also have been an effect of the startup material on crystallization of the glass.

Table V. Spinel Distribution in HAL-17 Glass after 11 hours of Feeding

Sampling Location	Spinel Content (vol %)				
	Pour 1	Pour 2	Pour 3	Pour 4	Quenched Glass in the Crucible
Top	1.6	1.1	1.1	1.3	1.2
Middle	1.8	1.1	1.1	1.2	1.5
Bottom	1.6	1.2	1.2	1.2	1.4

Fig. 3 shows a representative micrograph of the spinels identified in the HAL-17 glass sampled from the can (poured glass) after melting in the SMK for 11 hours. Similar crystallization was seen in all regions of the poured glass regardless of feeding rate or feeding time. Energy dispersive spectroscopy (EDS) analysis indicated that the spinels consist mainly of Fe, Cr and Mn.

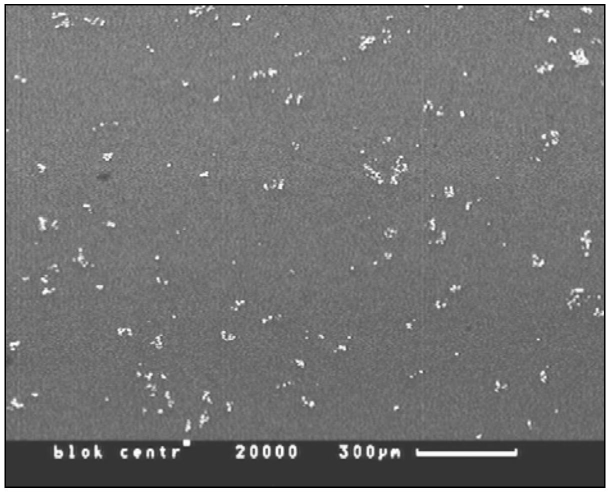


Figure 3. Representative Micrograph of Spinel Crystallization in HAL-17 Glass.

EP-5 Experiments with Hanford Feeds

KRI processed 41 L of the HAL-17 composition in the EP-5 melter. The feeding lasted 39 hours, and the overall duration of the EP-5 melter experiment was 50 hours. Approximately 20 kg of glass were produced during this experiment, including about 16 kg of glass generated from the feed. The remaining quantity of glass was generated from the startup material. Table VI summarizes the EP-5 operation with the HAL-17 feed.

Table VI. Quantities of Processed Sludge and Produced Glass for Hanford Waste Test

Pour No.	Feeding rate (L/hour)	Specific productivity ($L/dm^2 \cdot hr$)	Feeding Time (hours)	Mass of Glass Poured (g)
1	0.8	0.35	5	1580
2	0.78	0.34	5.5	1640
3	1.0	0.43	4	1584
4	0.8	0.35	4.25	1390
5	1.0	0.43	3.4	1300
6	1.3	0.57	5	2600
7	1.2	0.52	5	2875
8	1.2	0.52	3.8	1470
9	1.2	0.52	3	1490
Total			39	15929

The glass from all of the pours appeared heterogeneous. The spinel content in the poured glass as measured using SEM varied from 2-5 vol %. There was no clear relationship between the volume fraction of spinel crystallization and melter feeding time. Spinel crystals were distributed throughout the glass, with some agglomerates of smaller crystals appearing as striations.

SMK Experiments with SRS Feeds

The results of the SMK runs with the SRS feeds are summarized in Table VII.

Table VII. Results of the SMK Testing with the SRS Feeds

Frit Type	503		517		520	
Sludge Type	Sludge 1	Sludge 2	Sludge 1	Sludge 2	Sludge 1	Sludge 2
Feeding Time (minutes)	195	320	215	260	200	130
Average Feeding Rate (L/hour)	1.03	0.55	0.90	0.67	1.03	0.95
Mass of Glass Poured (g)	1490	1400	1460	1382	1470	1120
Pouring Time (minutes)	35.00	20.00	15.75	11.58	29.00	20.00

Visual observations of the melter experiments provide some insight into the behavior of the frit-sludge systems. When Sludges 1 and 2 mixed with Frits 503 and 517 were fed onto the melt surface, the cold cap distributed unevenly, mostly accumulating in the area where the feed entered the system. When the cold cap coverage reached the target 80-95%, mounding of the feed was observed on the cold cap. Melting of Sludge 1 mixed with Frit 503 resulted in a calcinate accumulation in the pour closure rod due to spills of the sludge. Melting of Sludge 1 mixed with Frit 517 resulted in a calcinate accumulation on the crucible wall. The calcinate accumulation did not make it possible to accurately measure the time required for consumption of the cold cap. No calcinate formations were associated with Frit 520. The feed material and the cold cap distributed evenly on the melt surface and the melting process continued as intended. Partial loss of the feed during feeding of Sludge 2 mixed with Frit 520 was experienced due to a damage of a hose in the peristaltic pump.

SEM analysis showed that the Sludge 1 glass formulated with Frit 503 glass had heterogeneous areas in the upper part of the poured glass. These areas were identified as spinel crystals via EDS. No spinel was found in the glass samples taken from the middle and lower part of the poured glass. The middle part of the Sludge 1 glass formulated with Frit 517 contained 14 vol % spinel crystals. No spinel was found in the samples taken from the upper and lower parts of the poured glass. The Sludge 1 glass formulated with Frit 520 contained spinel in the upper (0.1 vol %) and middle parts (0.5 vol %) of the poured glass, but not in the lower part.

No crystallization was visible via SEM in the Sludge 2 glasses formulated with Frits 503 and 520. The glass formulated with Sludge 2 and Frit 517 contained approximately 0.1 vol. % spinel crystals

The melt rate provided by the three frits was evaluated by comparing the average feeding rates for each experiment. In general, the melt rate for the Sludge 1 glasses was higher than that for the Sludge 2 glasses. For the Frit 503 and Frit 517 glasses, the melt rates differ significantly between the two sludges, while for Frit 520 glass the melt rates for the two sludges differ insignificantly. Frit 520 provided a fairly high melt rate for processing both sludges. Frit 520 also produced a reasonably homogenous glass when combined with either sludge. Frit 520 was therefore selected for processing Sludges 1 and 2 in the EP-5 Melter.

EP-5 Experiments with SRS Feeds

The goal of the EP-5 experiments with the SRS feeds was to identify the optimal feeding rate and determine whether a difference in melt rate could be observed between the sludges combined with Frit 520. The evaluation was made by monitoring the cold cap growth associated with a gradual increase of the feeding rate. The initial feeding was performed at a given rate for 30 minutes. If, during this time, the melt surface was not 80-90% covered with the cold cap, the feeding was stopped until the cold cap was consumed. The feeding rate was then increased and the process repeated. The feeding rate was subsequently reduced if the melt surface was 100% covered with the cold cap within 30 minutes. At the completion of feeding and cold cap consumption the melted glass was held in the melter for one hour to be followed by complete pouring into a collector can.

The Sludge 1 glass was poured once (6840 g). The feeding rate ranged from 1.6-2.0 L/hr, with the optimal feeding rate being 1.7 L/hr. The Sludge 2 glass was poured twice (805 g and 4156 g). The feeding rate ranged from 1.4-1.8 L/hr. The optimal feeding rate was 1.6 L/hr. There was no obvious difference in melt rate between the two sludge types.

Table VIII shows data on the spinel content in the glass from the two EP-5 experiments. The poured glass from the Sludge 1 experiment had a zoned distribution of spinel. The middle part of the glass contained a larger amount of spinel than the upper and bottom parts, which may relate to cooling rate. No spinel was observed in the glass from the first pour with Sludge 2. The spinel content in the glass from the second pour of Sludge 2 ranged from 0.05 to 0.8 vol %.

Table VIII. Spinel Content in the SRS Glasses Melted in the EP-5 (averages of 3 measurements)

Sludge 1		Sludge 2	
Sample Location	Spinel Content (vol %)	Sample Location	Spinel Content (vol %)
Top	0.4	Top	0.5
Middle	1.6	Middle	0.1
Bottom	0.2	Bottom	0.3

Chemical Durability Analysis

Representative samples of each of the glass systems were evaluated for chemical durability using the PCT. A select set of the results of the PCT for some of the study glasses in terms of the normalized leachate releases for boron (NL [B]) is given in Table IX. All of the glasses had NL [B] values that are well below that of the EA reference glass (16.695 g/L).[5] This indicates that these glasses would be acceptable for disposal in the federal repository. The PCT results also indicate that the crystallization (spinels) identified in the glasses does not have a significant impact on their chemical durability.

Table IX. Product Consistency Test Results for the Study Glasses

Melter	SMK	SMK	SMK	EP-5	EP-5
Sludge	2	2	2	1	HAL-17
Frit	503	517	520	520	
NL [B] (g/L)	0.84	1.15	0.95	1.08	0.22

CONCLUSIONS

The goal of this study was to determine the impacts of simulated high level waste glass compositions with high aluminum concentrations on melter performance, crystallization and chemical durability for SRS and Hanford waste streams. Glass compositions for Hanford targeted both high aluminum concentrations in waste and a high waste loading in the glass. Compositions for SRS targeted the next sludge batch to be processed in the DWPF, which also has a relatively high aluminum concentration. Three frits were selected for combination with the SRS waste to evaluate their impact on melt rate. The simulated sludges and frits were mixed and then melted in two small-scale test melters at KRI. The SMK melter allows for a determination of optimum feeding rate using a liquid feed over relatively short time periods. The EP-5 melter allows for longer feeding times between pours to evaluate longer term melter operation and glass crystallization.

The results showed varying degrees of spinel formation in all of the glass compositions using both of the melters. Spinels are known to have little influence on chemical durability and therefore were not of significant concern. Spinel accumulation within the melter did not appear to be an issue for the Hanford feeds, even after melter feeding times of more than 30 hours. Melt rates were estimated based on the feeding rate that provided optimum cold cap coverage on the melt pool. Some improvements in melt rate were made by tailoring the frit composition for the SRS feeds. Differences in cold cap behavior (flowing of the feed material) were also noted. All of the Hanford and SRS glass compositions had measured chemical durability performance that was acceptable for disposal in the federal repository. The highest measured NL [B] value was 1.15 g/L, which is significantly lower than that of the EA benchmark glass (16.695 g/L). Future studies will continue to assess the ability to tailor frits for improved melt rate through the control of boron and alkali additives, as well as other additives that may influence reactions in the cold cap.

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