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**PNL Vitrification Technology
Development Project High-Waste
Loaded High-Level Waste Glasses
for High-Temperature Melter: Letter
Report**

**D. Kim
P. R. Hrma**

February 1996

**Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest National Laboratory
Richland, Washington 99352**

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1.0 INTRODUCTION

For vitrification of high-level wastes (HLW) at the Hanford Site, a Joule-heated overflow type melter with bottom draining capability and capable of operating at temperatures up to 1500°C is being developed. The original proposed Hanford Waste Vitrification Plant (HWVP) melter used a 1150°C processing temperature and was tested using glasses with up to 28 wt% waste oxide loading for NCAW (Neutralized Current Acid Waste). The goal of the high-temperature melter (HTM) is the volume reduction of the final product and increase of the waste processing rate by processing high-waste loaded glasses at higher temperatures. This would dramatically decrease waste disposal and processing costs.

The aim of glass development for the HTM is to determine compositions and melting temperatures for processible and acceptable glasses with a high waste loading. Glass property/composition models for viscosity and liquidus temperature developed in the Glass Envelope Definition (GED) study (Hrma et al. 1994) were used. The results of glass formulation and experimental testing are presented for NCAW and DST/SST (Double-Shell Tank/Single-Shell Tank) Blend waste. Although the purpose of this report was to summarize the glass development study with Blend waste only, the results with NCAW were needed because glass development with Blend waste was based on the results from the glass development study with NCAW.

This work was performed in support of HTM development program (1.2.2.04.19).

2.0 OBJECTIVES

The objectives of this study are

- to develop the glass formulation approach for the HTM, and
- to determine the composition and melting temperature of a glass with 50 wt% NCAW for the first HTM runs and the maximum waste loading in glasses with DST/SST Blend waste for the future HTM runs.

3.0 GLASS FORMULATION APPROACH

An important property required for glass processibility in a continuous melter is melt crystallinity. Additionally, the durability of the HLW glass determines whether it is acceptable for disposal. Because Hanford high-level wastes are mainly composed of refractory oxides, such as ZrO_2 , Al_2O_3 , and Fe_2O_3 , their waste loading is limited by crystallinity before the durability limit is reached (Fini and Hrma 1994). HLW glasses, especially those with high waste loading and high melting temperature have reduced alkali and boron oxides content, which also improves durability. For this reason, the formulation of HLW glasses for the HTM focuses on the melt crystallinity.

Models are available for various glass properties, such as viscosity, durability, liquidus temperature, and electrical conductivity (Hrma et al. 1994). The models have been developed within the composition range defined by single- and multi-component constraints, shown in Table 1, and thus, an extrapolation to compositions beyond these constraints may generate uncertain predictions. Typically, the compositions of high-temperature glasses fall outside of at least one of these constraints. In addition, models for viscosity and electrical conductivity are validated only within temperatures between 950 and 1250°C because these models were developed for the 1150°C melter, while the intended operating temperature of the HTM is generally higher than 1250°C. Because of complexities involved in the crystallization of glasses (Hrma et al. 1994, Kim and Hrma 1994), liquidus temperature models are even further limited in terms of glass composition and temperature ranges.

Glass liquidus temperature constraint for Hanford melters has traditionally been $T_L < T_M - 100^\circ\text{C}$,* where T_L is the liquidus temperature and T_M is the melter processing temperature (melting temperature). The predicted T_M was defined as the temperature at which the predicted viscosity is 4 Pa-s (except for glasses in the preliminary tests). An effort was made to achieve the highest waste loading compatible with the requirement that $T_L < T_M - 100^\circ\text{C}$. Because of their limited

* Hanford Waste Vitrification Plant Project Technical Data Package, WHC-SD-HWV-DP-001, Rev. 6, Westinghouse Hanford Company, Richland, WA, 1992.

TABLE 1. Single- and Multi-Component Constraints for Glass Property/Composition Models

(a) Single-component constraints

Component	Lower limit (wt%)	Upper limit (wt%)
SiO ₂	42	57
B ₂ O ₃	5	20
Na ₂ O	5	20
Li ₂ O	1	7
CaO	0	10
MgO	0	8
Fe ₂ O ₃	0.5	15
Al ₂ O ₃	0	17
ZrO ₂	0	13
"Others"	1	10

(b) Multi-component constraints

Oxide sum (wt%) or ratio	Upper Limit
CaO + MgO	10
Al ₂ O ₃ + ZrO ₂	18
CaO + MgO + ZrO ₂	18
Fe ₂ O ₃ + Al ₂ O ₃ + ZrO ₂ + Others	24
Al ₂ O ₃ /SiO ₂	0.33

applicability, the models were only used as initial predictive tools, especially those for liquidus temperature, and it was necessary to test the glasses by measuring their properties in the laboratory.

4.0 EXPERIMENTAL METHODS

Table 2 shows the simulated composition of NCAW and Blend waste used in this study. NCAW has been well characterized and extensively used in past melter runs. The Blend waste composition represents the estimated average composition of all the Hanford double-shell tank (DST) and single-shell tank (SST) wastes.* The composition is expressed in weight percents of nine major oxide components and "Others," the "Others" component being comprised of the remaining waste components.

For each glass, the nine major components were batched as oxides or carbonates, while "Others" were batched together in constant proportions. Each glass was melted at the temperature for a predicted viscosity of 4 Pa·s (except for glasses in the preliminary tests) for 1.5 h in a platinum crucible, under a lid, using an electrically heated resistance furnace. For better homogeneity, the glass was crushed and remelted for 1.5 h under the same conditions. The glass for the crystallinity test was poured into a $2 \times 2 \times 7$ cm bar, air quenched, and annealed for 2 h, followed by furnace cooling. The remaining glass was poured onto a steel plate and air quenched. This glass was used for viscosity measurement.

Crystallinity was evaluated in as-melted samples and 24-h heat treated samples at T_M and $T_M - 100^\circ\text{C}$. Cubical specimens were cut from the glass bar and placed in a Pt-foil box in a preheated furnace for the heat treatment. Thin sections, prepared from the glass samples and mounted in polymer resin, were examined using an optical microscope with both reflected and transmitted light to determine the type of crystalline phases present. The volume fraction of crystals were estimated by an Image Analysis System attached to the optical microscope. The chemical composition of the crystalline phases was qualitatively determined by SEM/EDS (Scanning Electron Microscopy/Energy Dispersive Spectroscopy).

Viscosity was measured using a rotating spindle viscosimeter at temperatures in the range of $T_M \pm 150^\circ\text{C}$. Electrical conductivity and 7-day PCT (Product Consistency Test) (Janzen and Bibler 1990) elemental releases were determined for

* Memo, from Bob Watrous, WHC, to Pavel Hrma, PNL, "Composition Limits for Blended DST & SST Waste Glasses," dated June 11, 1993.

TABLE 2. Compositions (in mass fraction) of Simulated NCAW (Neutralized Current Acid Waste) and Blend Waste

Component	NCAW	Blend Waste
SiO ₂	4.02	12.37
B ₂ O ₃	0.01	0.09
Na ₂ O	21.35	24.67
Li ₂ O		
CaO	0.79	2.52
MgO	0.20	0.09
Fe ₂ O ₃	28.13	13.60
Al ₂ O ₃	9.01	9.74
ZrO ₂	15.07	8.75
"Others"	21.43	28.16
Total	100.00	100.00

"Others" components

BaO	0.38	
Bi ₂ O ₃		2.75
CdO	2.88	0.11
CeO ₂	0.58	3.38
Cr ₂ O ₃	0.48	1.06
Cs ₂ O	0.58	
CuO	0.58	
F	1.15	0.68
La ₂ O ₃	2.40	0.53
MnO ₂	0.58	2.25
MoO ₃	1.15	0.09
Nd ₂ O ₃	4.73	12.04
NiO	2.21	2.81
P ₂ O ₅	0.38	1.59
PdO	0.19	
Pr ₆ O ₁₁	0.38	
Rb ₂ O	0.19	
Rh ₂ O ₃	0.19	
RuO ₂	0.58	
SO ₃	1.06	0.41
Sm ₂ O ₃	0.19	
SrO	0.38	
WO ₃		0.21
Y ₂ O ₃	0.19	
Subtotal	21.43	28.16

the selected "best" glass composition. The viscosity (η) and electrical conductivity (ϵ) data as a function of temperature were fitted to the Arrhenius equation, $\ln(\eta, \epsilon) = A + B/T$, where A and B are constants, and T is the absolute temperature. The viscosity data at temperatures in this report represent the values calculated from the Arrhenius fits.

5.0 GLASSES WITH NCAW

Preliminary tests were conducted for glasses with 45 to 50 wt% NCAW before the liquidus temperature models were available. Table 3 shows the summary of the preliminary test results on glasses with 50 wt% NCAW. Glass N501 consisted of waste and SiO_2 . Glasses N502 and N503 also contained B_2O_3 . These glasses were melted at predicted viscosities different from 4 Pa·s. Their crystallinity was determined only in as-melted samples.

At least 3 vol% spinel was found in all samples. Glass N503 had 0.4 vol% ZrO_2 in addition to 3.6 vol% spinel. If glasses N502 and N503 were melted at lower

TABLE 3. Crystallinity of Glasses Containing 50 wt% NCAW with Addition of SiO_2 Only or SiO_2 and B_2O_3 (Preliminary Study)

Glass	N501	N502	N503
NCAW (wt%)	50	50	50
SiO_2 (wt%)	50	46	35
B_2O_3 (wt%)	0	4	15
T_M (°C)	1450	1450	1400
Predicted η at T_M (Pa·s)	5.1	3.0	1.0
Crystallinity (vol%) in as melted glass	spinel ~5	spinel ~3	spinel ~3.6 ZrO_2 ~0.4

temperatures corresponding to a predicted viscosity of 4 Pa·s, their crystallinity would be slightly higher than the values shown in Table 3.

Liquidus temperature models were used to evaluate the effect of additions of the alkali oxides Na_2O and Li_2O and B_2O_3 on the crystallinity, and to further improve glass composition. Figure 1 shows plots of predicted T_L [(a) spinel and (b) Zr-containing phases (ZrSiO_4 , ZrO_2 , and Na-Zr silicate)] versus T_M (for predicted viscosity of 4 Pa·s) when the additions of B_2O_3 , Na_2O , and Li_2O were varied. The calculated data points correspond to 2 wt% steps from 0 up to 14 wt% (right to left) of each oxide in the additive mix.

Because the composition and melting temperature of the glasses shown in Figure 1 are outside of the model validity range, uncertain predictions, especially for the T_L , are expected. This can be illustrated by a relatively high concentration of spinel found in glass N503 melted at 1400°C: the predicted T_L of spinel in this glass was 1050°C (Figure 1). Therefore, the information regarding the "relative" effect of each component on the liquidus temperature was used to aid glass formulation.

Figure 1 shows that the T_L decreases as melting temperature decreases with the addition of these components except for the effect of B_2O_3 on the T_L of Zr-containing phases. Addition of Na_2O was most effective for spinel and Li_2O for Zr-containing phases, while B_2O_3 was least effective for both crystalline forms. This result suggests that the crystallinity observed in glasses N502 and N503 may decrease if Na_2O or Li_2O is used instead of B_2O_3 .

Based on the information from preliminary tests and model predictions on the "relative" component effect, four glasses with Na_2O and Li_2O addition were formulated and tested. Table 4 summarizes the results.

In the as-melted samples, the glasses with alkali addition (Table 4) exhibited lower crystallinity than the two glasses with B_2O_3 addition (Table 3). Table 4 shows that the ability for predicting viscosity was reasonably good considering that the glass composition and temperature were outside the model validity range.

Crystallinity was lower in as-melted glasses with $T_M = 1350^\circ\text{C}$ (N508 and N509) as compared to those with $T_M = 1400^\circ\text{C}$ (N506 and N507). This indicates that the 1350°C glasses with a higher alkali content dissolve the spinel crystals formed

FIGURE 1. Predicted Liquidus Temperature (T_L) of (a) Spinel and (b) Zr-Containing Phases Versus Predicted Melting Temperature (T_M) for Glasses with 50 wt% NCAW + 50 wt% [(100-x)SiO₂ + xY], Where Y Represents B₂O₃, Na₂O, or Li₂O and x Is the wt% of Y in the Additive Mix. The Value of x Varied by 2 wt% for Each Data Point from 0 to 14 wt%.

TABLE 4. Viscosity and Crystallinity of Glasses Containing 50 wt% NCAW with Addition of SiO₂ and Na₂O or Li₂O

Glass	N506	N507	N508	N509
NCAW (wt%)	50	50	50	50
SiO ₂ (wt%)	46.3	48.3	44.0	47.3
Na ₂ O (wt%)	3.7	0	6.0	0
Li ₂ O (wt%)	0	1.7	0	2.7
T _M (°C)	1400	1400	1350	1350
Viscosity Coefficients*				
A	-14.15	-12.85	-14.93	-13.97
B (K)	26716.79	24122.39	27376.12	25080.41
Measured η at T _M (Pa·s)**	6.2	4.8	6.9	4.4
Crystallinity (volume %)***				
as-melted	~1.5	~2.8	<0.1	~0.2
24 h at T _M	0	<0.1	<0.1	0
24 h at T _M - 100°C	<0.1	~0.2	0	~0.5

*Obtained from Arrhenius fit, $\ln(\eta) = A + B/T$, of experimental data.

**Predicted viscosity was 4 Pa·s at melting temperature for all glasses.

***Spinel was the only crystalline phase observed.

during the initial melting faster than the 1400°C glasses. As shown in Table 4, the crystals almost completely disappeared after 24-h heat treatment at T_M. The crystallinity after 24 h at T_M - 100°C was <0.1 in glasses with Na₂O (N506 and N508) and a little higher in glasses with Li₂O (N507 and N509), the latter possibly having a slightly higher liquidus temperature. The glass N508, therefore, was selected for the first SSHTM (Small-Scale High-Temperature Melter) test and subjected to further testing (Smith et al. 1994).

Table 5 shows the viscosity and electrical conductivity of glass N508 as a function of temperature, obtained from the Arrhenius fits to experimental data. The chemical durability of N508 was determined by a 7-day PCT (Product Consistency Test) (Janzen, et al. 1992). The normalized elemental releases were obtained using the formula $r_i = m_i / (F_i A)$, where m_i is the mass of the i-th element dissolved in solution (g), F_i is the i-th element mass fraction in glass, and A is the glass surface area (m²). The results for Na and Si were

$$r_{\text{Na}} = 0.46 \text{ g/m}^2/7 \text{ days} \quad r_{\text{Si}} = 0.17 \text{ g/m}^2/7 \text{ days}$$

These results exceed the HLW glass PCT standard, $r_{\text{Na}} = 6.67$ and $r_{\text{Si}} = 1.96$ (g/m²/7 days), for EA (Environmental Assessment) glass (Jantzen, et al. 1994).

TABLE 5. Viscosity and Electrical Conductivity of Glass N508 As a Function of Temperature*

Temperature (°C)	Viscosity (Pa·s)	Electrical Conductivity (S/m)
1250	20.99	25.00
1300	11.86	29.47
1350	6.94	34.40
1400	4.19	39.78
1450	2.61	45.62
1500	1.66	51.91

*For viscosity (Pa·s) $A = -14.93$ and $B = 27376.12$ K, and for electrical conductivity (S/m) $A = 8.40$ and $B = -7892.54$ K from Arrhenius fit, $\ln(\eta, \epsilon) = A + B/T$, of experimental data.

6.0 GLASSES WITH BLEND WASTE

Compared with NCAW, the Blend waste had lower concentrations of Fe_2O_3 and ZrO_2 , and a slightly higher concentration of all other components except MgO (Table 2). This suggests that a higher waste loading can be achieved with the Blend waste. Table 6 shows formulations and test results of four glasses with Blend waste and SiO_2 addition within a waste loading range of 57 to 71 wt%. As in NCAW glasses, the predicted melting temperature was determined as that at which the predicted viscosity was 4 Pa·s; it decreased as the waste loading increased.

Glass B711 (71 wt% waste loading) contained both spinel and ZrO_2 in all samples tested, whereas glass B571 (57 wt% waste loading) did not have any crystals at all. Glass B671 (67 wt% waste loading) formed spinel in the as-melted sample and $T_M - 100^\circ\text{C}$ 24-h heat treated samples, whereas glass B621 (62 wt% waste loading) formed spinel in the $T_M - 100^\circ\text{C}$ 24-h heat treated sample only. The size distribution of spinel in this sample (B621; 24-h heat treated at $T_M - 100^\circ\text{C}$) was different from that in other samples: the total crystallinity of 0.5 vol% consisted of about 90% very small ($< 1\mu\text{m}$) and about 5% large spinel crystals (10-40 μm). The size of spinel crystals in other samples ranged from 1 to 10 μm . The small crystals in B621 were, possibly, formed during cooling. If so, glass B621 might be acceptable for HTM. Because the measured viscosity of glass B621 was 7 Pa·s at the melting temperature 1350°C , the amount of crystals at $T_M - 100^\circ\text{C}$ for T_M at measured viscosity of 4 Pa·s will be lower. This suggests that a waste loading B621 than 62 wt% might be possible assuming a requirement of < 0.1 vol% spinel at $T_M - 100^\circ\text{C}$.

If the HTM can process glasses with up to 1 vol% spinel crystallization at $T_M - 100^\circ\text{C}$, the maximum waste loading may increase to between 67 and 71 wt%: glass B671 (67 wt% waste loading) had 0.8 vol% spinel at $T_M - 100^\circ\text{C}$. However, it is possible that the processing rate of glasses may become unacceptably slow at a high waste loading of 67 to 71 wt%. The possibility of slow melting rate may be indicated by the 0.8 vol% spinel in an as-melted sample, which dissolve after 24-h heat treatment at T_M .

TABLE 6. Viscosity and Crystallinity in Glasses Containing Blend Waste with Addition of SiO₂ Only

Glass	B571	B621	B671	B711
Blend waste (wt%)	57	62	67	71
SiO ₂ (wt%)	43	38	33	29
T _M (°C)	1400	1350	1300	1250
Viscosity Coefficients*				
A	-13.36	-13.87	-14.18	-14.17
B (K)	25835.63	25667.56	25164.83	24286.19
Measured η at T _M (Pa·s)**	8.0	7.0	6.2	5.9
Crystallinity (volume %)***				
as-melted	0	0	~0.8	~0.4 ZrO ₂ ~0.2
24 h at T _M	0	0	0	ZrO ₂ <0.1
24 h at T _M - 100°C	0	~0.5^	~0.8	~1.2 ZrO ₂ ~1.2 unidentified ~5

*Obtained from Arrhenius fit, $\ln(\eta) = A + B/T$, of experimental data.

**Predicted viscosity was 4 Pa·s at melting temperature for all glasses.

***Spinel crystals unless otherwise specified.

^Consisted of 0.05 % large spinel (10-40 μm) and 0.45% very small spinel (< 1 μm). In other glasses, the spinel crystals were between 1 and 10 μm .

The estimated composition of Blend waste has been updated since this work was initiated. The new Blend waste composition* is slightly different from the one

* Letter, R. W. Powell, WHC, to J. M. Creer, PNL, "Double-Shell Tank/Single-Shell Tank Waste Blend Composition for High-Level Waste Vitrification Process Testing," 9452712, received May 19, 1994.

used in this study. The new Blend waste is expected to allow waste loadings between 60 and 65 wt% if <0.1 vol% of spinel is acceptable for HTM.

7.0 CONCLUSIONS

Glass formulations for HTM from two Hanford waste types were formulated using property/composition models, and tested experimentally. Different approaches were used for different waste types: for NCAW, the glass development effort was focused on finding the optimum additive at a target waste loading of 50 wt%, and for Blend waste, the additive was fixed as SiO₂ only, and the waste loading was varied from 57 to 71 wt%, to find the maximum waste loading. An NCAW glass acceptable for repository and processable in the HTM was developed by adding the SiO₂ and Na₂O, and is being planned to be used in the first melter run (SSHTM-1). The maximum waste loading that can be achieved with <0.1 vol% crystallinity after 24 h at T_M - 100°C in the Blend waste glass was determined to be between 60 and 65 wt%, with only SiO₂ as an additive.

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