

VITRIFICATION OF INEEL SODIUM BEARING WASTE SURROGATE IN THE SIA RADON COLD CRUCIBLE INDUCTION MELTER

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ABSTRACT

Results of the INEEL sodium-based waste surrogate vitrification test are described. The test was performed at the Radon bench-scale plant equipped with the cold crucible 216 mm in inside diameter. The feasibility of the process has been demonstrated. Some disadvantages were identified such as relatively low glass productivity (~1 kg/h) and high heat expenses (up to ~50 kW×h/kg of glass) but they were believed to be attributed to the introduction of boron flux in the form of boric acid which has high volatility. This reduces the boron content in the glass and increased the glass melting temperature. The cold crucible demonstrated high resistance to molten glass corrosion even at elevated process temperatures (1350-1370 °C) caused by the boron deficiency. The glasses produced were found to be quite homogeneous with minor inclusions of unreacted raw minerals.

INTRODUCTION

The inductive cold crucible melting technology (ICCM) is considered as an alternative to vitrification of radioactive waste in Joule heated ceramic melters, which are currently applied in Russia [1] and USA [2,3]. Major advantages of the cold crucible over the ceramic melter are smaller overall dimensions and weight, no refractories and electrodes, higher specific productivity, and wide compositional and temperature variations. The ICCM technology is being successfully applied in Russia at SIA Radon to vitrification of sodium nitrate based liquid intermediate-level radioactive waste (ILW) [4-6]. Accordingly, the need to evaluate the possibility of vitrification of radioactive waste from the US Department of Energy (DOE) sites at

the Radon bench-scale facility equipped with accessory cold crucibles with variable productivity has been identified.

The goal of this test was to perform a test with INEEL sodium bearing waste (SBW) surrogate to confirm the possibility of application of the ICCM technology for DOE waste vitrification and to test the new ~200 mm diameter cold crucible.

Experimental

Equipment

The INEEL SBW surrogate vitrification test was performed in an experimental unit equipped with a cold crucible melter, off-gas system, inductor and container placed within a process box, which also serves as a protective shield for personnel from high frequency radiation. The process flowsheet is shown on Figure 1.

Energy is supplied from a high frequency power supply – VCI-11-60/1.76 generator with technical parameters as follows:

- Input voltage 380 V,
- Operation frequency 50 Hz,
- Number of phases 3
- Input power ≤ 90 kW
- Vibrating power 60 ± 6 kW,
- Operating frequency 1.760 ± 0.044 MHz,

Other electric devices were capacitor battery and copper inductor with inner diameter of 295 mm. The high frequency generator operation is monitored by indicating pointer devices installed in its frame and it is controlled by vibrating power regulation by means of voltage or current variation. Control drives are hand-operated and also installed in the frame of the generator.

The cold crucible (Figure 2) is a vessel of cylindrical shape with water-cooled walls and bottom. It is manufactured from stainless steel (Russian brand 12X18H10T) pipes. Sidewalls are fabricated from pipes 12 mm in diameter and 2 mm in thickness. Total crucible height is 521 mm, including crucible itself – 500 mm, bottom – 16 mm, cover – 5 mm. Inside diameter is 216 mm, melt surface area is 36643 mm². The outside of the crucible walls is coated with protective zirconia-based putty. The cold crucible is equipped with a pouring unit consisting of water-cooled tube-in-tube cylinder (Figure 2) with a blocking rod (gate) to stop glass pouring.

The slurry preparation system consists of a stirred vessel for slurry production placed within an electric furnace to increase solubility of some waste surrogate constituents, interim vessel to accumulate slurry before feeding the melter, and feeder – peristaltic pump to charge slurry to the melter.

The off-gas system consists of an off-gas pipe, a heat-exchanger, a condensate collector, and an electric heater that supplies an air blast. The latter is used to prevent vapor condensation in the off-gas pipe.

The slurry feed was prepared from INEEL SBW surrogate and glass forming additives (Table I). To avoid segregation of solid phase from the slurry and ensure its stability for a time necessary to feed all the slurry to the melter, about 30 wt% of the silica was replaced by a reagent-grade “Rubbersil RS-200” containing >98 wt.% SiO₂ with surface area of 180 m²/g.

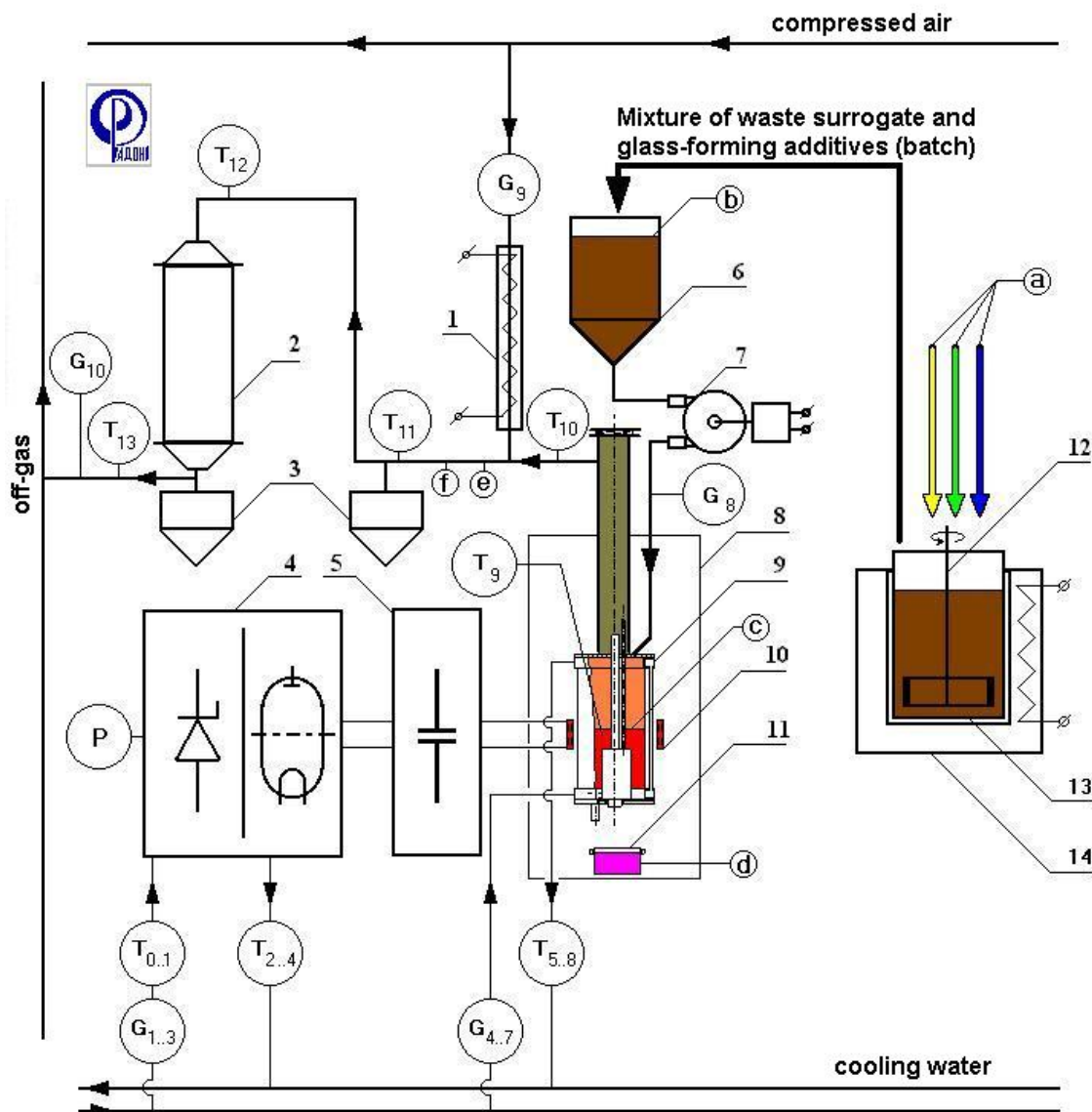


Fig. 1. Flow sheet of the cold crucible based experimental plant.

Equipment: 1- electric heater of compressed air, 2- heat-exchanger, 3- condensate collectors, 4- high-frequency energizer, 5- battery of capacitors, 6- batch vessel, 7- feeder, 8- process box, 9- cold crucible, 10- inductor, 11- container for glass product, 12- stirrer, 13- slurry batch

preparation vessel, 14- electric furnace. Sampling points: a- raw materials (source reagents), b- slurry, c- “yellow phase”, d- glass, e- aerosols, f- off-gas.

The parameters to be controlled: $T_{0...8}$ – cooling water temperature, T_9 – melt temperature, T_{10} – off-gas temperature at crucible outlet, $T_{11...13}$ – off-gas temperature at heat-exchanger inlet and outlet, $G_{1...7}$ – cooling water flow rate in the induction system, G_8 – slurry feeding rate, G_9 – compressed air flow rate, G_{10} – off-gas flow rate.

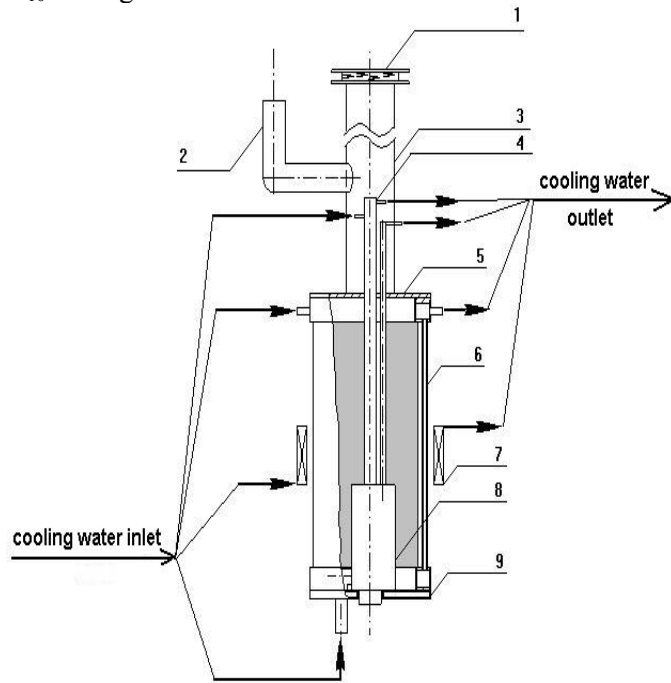


Fig. 2. The scheme (upper left), general view (upper right), installation of the cold crucible in the process box (lower left), and the pouring unit (lower right).

1 – operation control window, 2 – off-gas pipe, 3 – operation control port, 4 – pour bolt, 5 – crucible cover, 6 – pipe-fabricated water-cooled crucible wall, 7 – inductor, 8 – water-cooled pour unit, 9 – water-cooled crucible bottom.

Table I. INEEL SBW Surrogate and Glass Former Compositions.

Source chemicals	Salt content, g/L	Oxides	Oxide content in SBW surrogate, wt. %	Oxide content in glass, wt. %	Glass formers	Content, g/L	Weight content, %	Oxides	Oxide content, wt. %	Oxide content in glass, wt. %
Al(NO ₃) ₃ *9H ₂ O	0.0030	Al ₂ O ₃	27.57	5.52	H ₃ BO ₃	52.6	9.92	B ₂ O ₃	6.37	5.10
Ba(NO ₃) ₂	0.01457	BaO	0.007	0.00	Ca(OH) ₂	32.5	6.13	CaO	5.29	4.23
H ₃ BO ₃	0.7605	B ₂ O ₃	0.349	0.067	Fe ₂ O ₃	7.5	1.42	Fe ₂ O ₃	1.61	1.29
Cd(NO ₃) ₂ *4H ₂ O	0.2326	CdO	0.079	0.016	LiOH *H ₂ O	42.1	7.94	Li ₂ O	3.22	2.58
Ca(NO ₃) ₂ *4H ₂ O	11.1400	CaO	2.16	0.432	Mg(OH) ₂	12.4	2.34	MgO	1.83	1.46
Ce(NO ₃) ₃ *6H ₂ O	0.0205	Ce ₂ O ₃	0.006	0.000	NaOH	13.6	2.57	Na ₂ O	2.27	1.81
Cr(NO ₃) ₃ *9H ₂ O	1.3410	Cr ₂ O ₃	0.21	0.042	SiO ₂	333.4	62.91	SiO ₂	71.69	57.35
Co(NO ₃) ₂ *6H ₂ O	0.0056	CoO	0.0013	0.000	V ₂ O ₅	23.9	4.51	V ₂ O ₅	5.14	4.11
Cu(NO ₃) ₂ *3H ₂ O	0.1684	CuO	0.0452	0.000	ZrO ₂	12	2.26	ZrO ₂	2.58	2.06
Gd(NO ₃) ₃ *5H ₂ O	0.0768	Gd ₂ O ₃	0.0262	0.000						
Fe(NO ₃) ₃ *9H ₂ O	8.7790	Fe ₂ O ₃	1.42	0.283						
Pb(NO ₃) ₂	0.4329	PbO	0.238	0.048						
LiNO ₃	0.0234	Li ₂ O	0.0041	0.000						
Mg(NO ₃) ₂ *6H ₂ O	3.0850	MgO	0.396	0.079						
Mn(NO ₃) ₂	5.0460	MnO	0.816	0.163						
H ₂ MoO ₄	0.0326	MoO ₃	0.023	0.000						
Ni(NO ₃) ₂ *6H ₂ O	0.4278	NiO	0.090	0.018						
KNO ₃	19.8300	K ₂ O	7.55	1.509						
NaNO ₃	174.900	Na ₂ O	52.0	10.41						
Sr(NO ₃) ₂	0.0252	SrO	0.010	0.000						
TiO ₂	0.0046	TiO ₂	0.0038	0.000						
Zn(NO ₃) ₂ *6H ₂ O	0.3121	ZnO	0.0696	0.000						
ZrF ₄	0.0113	ZrO ₂	0.0067	0.000						
HCl	2.4490	Cl	0.925	0.185						
HF	1.17037	F	0.5331	0.107						
KI	1.1810	K ₂ O	0.000	0.000						
HNO ₃	51.6900									
H ₃ PO ₄	0.9384	P ₂ O ₅	0.7929	0.159						
H ₂ SO ₄	3.8770	SO ₃	4.5562	0.911						
Total			100	20		530	100		100	80

Melting run

Initial (starting) melt is a melt with weight and volume providing start-up of the ICCM process. In this test, a starting melt in the amount of 10 kg (or about 30 L) was obtained for 25 min using a patented method [7]. To achieve steady-state conditions 13 hours were required. Vibrating power of the generator ranged between 44 and 65 kW. At 44 kW of vibrating power, feed melt rate was low. The increase of vibrating power resulted in process intensification accompanied by foaming. The melt was viscous with low flowability. Three pours of glassmelt containers were performed during this step of the test in and 8.9 kg of glass was obtained.

INEEL SBW surrogate vitrification under steady-state conditions was performed for 48 hours. The third step consisted of cycles from the beginning of slurry feeding to complete glassmelt pouring. Slurry was batch fed manually using agitation. Slurry batches of 7.7 to 9.6 kg were fed before melter pouring was initiated. Feeding was started when melt surface was approximately 15% visually open. The melt pool was retained long enough to ensure melting of the residual calcine and homogenization. The melt temperature was then increased to 1350...1370 °C, the pour gate was opened, and then pouring of the melt was initiated through the pour channel (8 on Figure 2). When problems with opening the pouring gate and melt pouring occurred, the melt was retained and the pouring gate was closed and re-opened. This took from 7 to 30 min.

At a generator vibrating power of 56 kW, the melt temperature at depth of 35 mm (T_9 on Figure 1) measured during the melt homogenization was 1310 to 1375 °C and during slurry feeding was 1240 to 1250 °C. The average vibrating power of the generator for this period was 57.5 kW.

For 48 hours of steady-state conditions 172 kg of slurry was processed and 50.1 kg of glass was produced. Batch melt pours occurred 19 times.

After filling, the containers with glass were placed in a box insulated with quartz sand. Twelve containers were obtained in total. After cooling to room temperature, the glass was cracked and sampled.

The average slurry feed rate was 4.7 kg/h. The average slurry feed rate takes into account time spent for melt homogenization, pouring and replacement of containers (capacity) was 3.7 kg/h. Variations in slurry feed and melt pour rates were due to additional problems occasionally experienced with pouring of the viscous melt that impeded glass pouring to the initial level.

Average glass productivity was 1.1 kg/h. Specific heat expenses on slurry vitrification by the cold crucible and on glass production were 15.5 kW·h/kg and 52.3 kW·h/kg, respectively. The latter value corresponds to a specific glass pour rate of 730.3 kg/(m²·day).

No sulfate-chloride salts (“yellow phase”) on the melt surface or cold crucible walls were observed. The melt had high viscosity even at temperature about 1350 °C that impeded melt pouring and container filling.

The cold crucible melter at the experimental plant operated continuously for a total of 76 hours including 48 hours under steady-state conditions. In total, 70 kg of glass was produced. Fifty-

nine kg of glass was poured through the pouring unit. The remainder of the glass in the cold crucible was 11 kg. The glass produced was visually homogeneous.

Visual observation of the cold crucible melter was performed after complete residual glass removal. It was established that:

- no metal inclusions in the glass or metal deposits on wall pipes occurred;
- no corrosion of pipes on the outside;
- visual traces of corrosion existed on the inside of the crucible walls at the pour level in the form of dark and very small caverns. The maximum corrosion of the crucible tubes was observed at the melt pour level through the drain or pour spout;
- no visual changes of the pouring unit at pouring level were seen – no traces of corrosion of the drain. Minor traces of corrosion in the form of very small caverns were noticeable on the pouring unit at a level of 40 to 50 mm under the pour level; a noticeable change in the pouring unit color can be also seen (Figure 2, lower right).

Off-Gas Characterization

Off-gas

Off-gas temperature (T_{10} on Figure 1) during slurry feeding was 40 to 110 °C and during melt homogenization reached 115 °C; the average off-gas temperature at melter outlet was 63 °C (during the test run, it ranged between 40 and 115 °C). Off-gas volume flow rate at the melter outlet ranged between 6 and 21 m³/h, with an average value of 12 normal m³/h. Calculated air leakage flow rate was 9.6 normal m³/h.

Aerosols

The average aerosol concentration in the off-gas diluted by heated compressed air at melter outlet was 142 mg/m³. Aerosol carry-over from the melter did not exceed 0.5 wt.% from the total slurry fed; average carry-over rate from the melter was 15.3 g/h. Weight concentration of aerosols in the off-gas at melter outlet varied widely, and direct connection between it and test operations was not found. Approximately 33 wt% of the total aerosols are particles $\leq 1 \mu\text{m}$ in size and smaller and about 60 wt.% were $\leq 2 \mu\text{m}$.

Acid gases

Average concentrations in the off-gas diluted by heated compressed air at melter outlet were: SO_x (recalculated to SO₂) – 22 mg/m² and HCl – 13 mg/m³. Carry-over from the melter included: NO_x (recalculated to NO₂) – 581.1 g/h; SO_x (recalculated to SO₂) – 2.4 g/h; and HCl – 1.4 g/h.

Glass Characterization

Glass sampled from the containers were quantitatively analyzed by X-ray fluorescent spectroscopy using a PW-2400 spectrometer (Philips Analytical B.V., The Netherlands) equipped with quantitative analytical Philips SuperQuantitative&IQ-2001 Software, atomic absorption spectroscopy using a Perkin-Elmer 403 spectrometer, and Emission flame photometry (EFP) to determine Na and K using a PFM-U 4.2 flame photometer (Russian design). The occurrence of crystalline phases was verified by X-ray diffraction (XRD) using a DRON-4

diffractometer (Cu K_{α} -radiation). The chemical composition of glass matrix and inclusions were determined by scanning electron microscopy with energy dispersive system (SEM/EDS) using a JSM-5300+Link ISIS analytical unit.

Glassmelt viscosity and electric resistivity were measured using a modified GOI vibrational viscosimeter designed at SIA Radon. Glass density was measured by gravimetric (Archimede) method [8].

As seen from Table II, chemical composition of the glasses obtained was slightly different from the target glass composition. The most significant difference was in B_2O_3 content and this discrepancy increases with run duration. High B_2O_3 volatilization is due to introduction of boron in the feed in the form of boric acid (H_3BO_3), which has high volatility with aqueous vapors [9].

Table II. Chemical Composition of the Glasses.

	SBW-0 (calc.)	SBW-1	SBW-2	SBW-3	SBW-4	SBW-5	SBW-8	SBW-9	SBW-10	SBW-11	SBW-12
B_2O_3	5.10	2.30	2.40	2.06	1.73	1.80	1.82	1.37	1.70	1.96	1.17
Na_2O	12.8	15.0	13.9	12.1	12.3	12.5	13.3	13.4	13.5	12.9	12.8
MgO	1.54	1.41	1.40	1.42	1.37	1.35	1.64	1.65	1.56	1.56	1.62
Al_2O_3	5.91	6.64	6.60	7.11	8.80	7.85	8.42	10.52	10.79	10.81	10.78
SiO_2	56.1	54.7	54.9	54.4	54.4	53.1	52.9	51.9	51.3	51.8	51.6
P_2O_5	0.16	0.21	0.35	0.34	0.23	0.29	0.22	0.19	0.18	0.17	0.16
SO_3	0.95	0.7	0.69	0.73	0.69	0.77	0.71	0.85	0.81	0.77	0.64
Cl	0.180	0.038	0.031	0.040	0.020	0.030	0.028	0.000	0.023	0.000	0.034
K_2O	1.58	1.14	1.34	1.33	1.47	1.54	1.57	1.44	1.47	1.47	1.47
CaO	4.64	4.96	4.96	4.96	4.26	4.31	4.69	4.85	4.14	4.00	4.24
TiO_2	0.008	0.160	0.100	0.085	0.070	0.060	0.060	0.054	0.060	0.051	0.050
V_2O_5	4.11	4.08	4.67	4.90	5.26	5.23	5.70	6.21	6.25	6.39	6.53
MnO	0.17	0.23	0.23	0.22	0.28	0.27	0.23	0.27	0.28	0.27	0.30
FeO_x^*	1.57	2.28	2.84	2.64	2.40	2.25	2.17	1.83	1.73	1.81	1.84
NiO	0.030	0.015	0.025	0.031	0.039	0.043	0.048	0.050	0.049	0.048	0.046
ZrO_2	2.04	0.94	0.96	1.09	1.48	1.61	1.78	1.88	1.96	2.02	2.05
Ba	0.002	0.000	0.080	0.130	0.130	0.150	0.180	0.170	0.190	0.198	0.210
Pb	0.049	0.000	0.036	0.029	0.045	0.048	0.053	0.053	0.050	0.052	0.053
ZnO	0.015	0.000	0.000	0.000	0.000	0.021	0.026	0.020	0.021	0.019	0.025
CuO	0.009	0.000	0.000	0.000	0.000	0.000	0.021	0.000	0.000	0.026	0.024
Li_2O	2.04	2.10	2.34	2.46	2.75	2.97	3.08	3.16	3.24	3.25	3.34
SrO	0.0021	-	-	-	-	-	-	-	-	-	-
Cr_2O_3	0.043	-	-	-	-	-	-	-	-	-	-
Total	99.0	96.9	97.9	96.0	97.7	96.2	98.6	99.9	99.3	99.6	99.0
FeO_x^*	1.57	2.28	2.84	2.64	2.40	2.25	2.17	1.83	1.73	1.81	1.84
FeO		0.42	0.42	0.42	0.92	0.42	0.74	0.64	0.84	0.72	0.83
Fe_2O_3		1.86	2.42	2.22	1.48	1.83	1.43	1.19	0.89	1.09	1.01

* iron oxide content was recalculated to Fe_2O_3 , - lower than detection limit

Table III shows that viscosity changes are more prevalent at high temperatures and slightly increase with longer test duration (probably due to reduction in B₂O₃ content and some increase in Al₂O₃ content), while electric resistivity reduces insignificantly because of the increase in alkali oxides content.

The samples of the glasses produced were selectively examined with XRD and SEM/EDS. From XRD data, all the glasses were amorphous. SEM/EDS data demonstrated that the glass matrix was quite compositionally and texturally homogeneous (Table IV). Individual gas bubbles up to 400 μm in diameter occurred. At higher resolution, some inhomogeneities in the glass matrix became visible. There were baddeleyite grains up to 3 μm in size surrounded by cord, as well as rare grains of residual quartz and zircon (Figure 3 and Table IV).

Table III. Viscosity and Electric Resistivity of Glassmelts with SBW-5 and SBW-10 Compositions.

Temperature °C	Viscosity Pa*s	Resistivity Ω*m	Temperature °C	Viscosity Pa*s	Resistivity Ω*m
SBW-10			SBW-5		
1300	8.8	0.025	1300	7.8	0.023
1250	11.2	0.028	1250	10.6	0.026
1200	15.4	0.032	1200	13.9	0.029
1150	19.9	0.037	1150	19.4	0.035
1100	26.4	0.044	1100	27.0	0.041
1050	36.2	0.054	1050	36.6	0.051

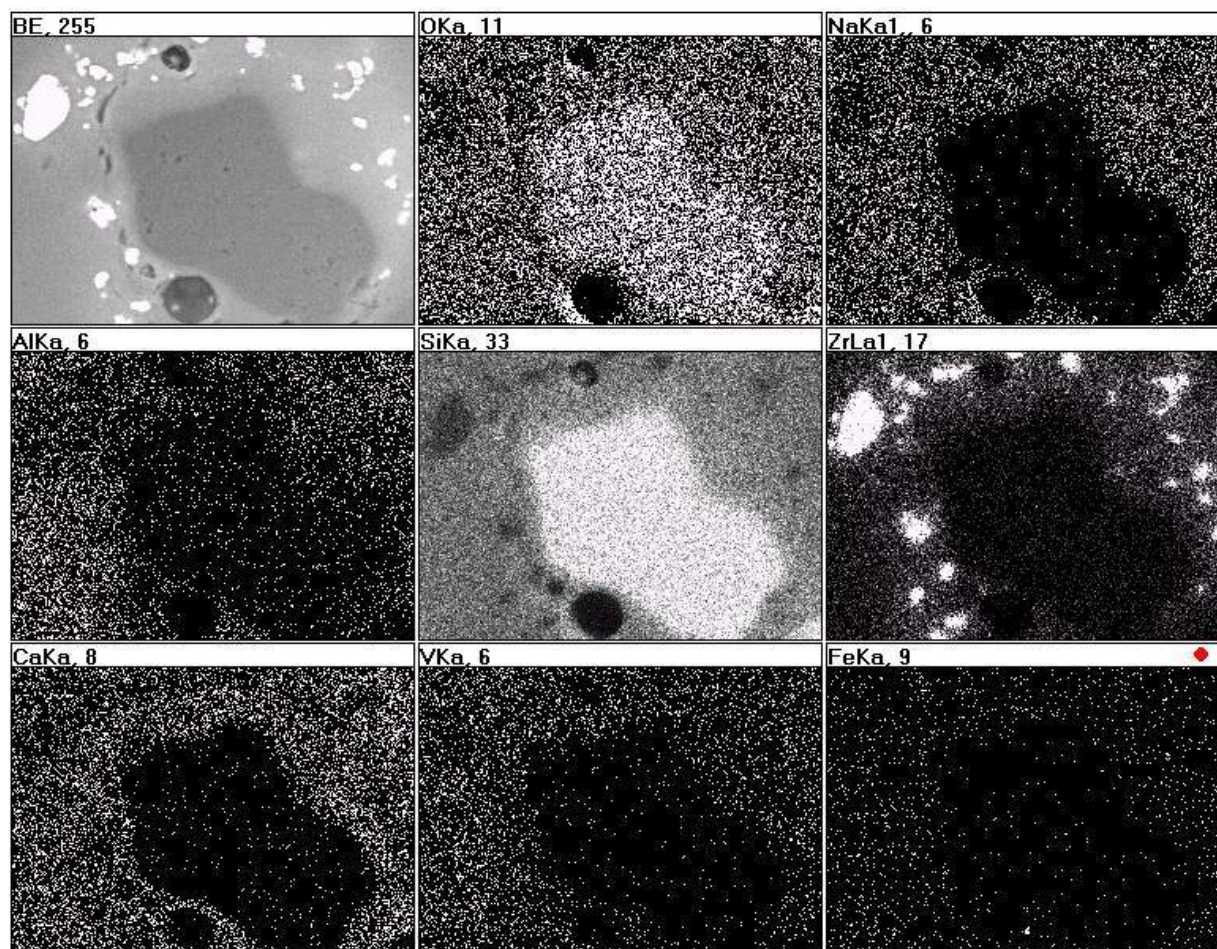


Fig. 3. SEM image and EDX mapping of unreacted grains of quartz, baddeleyite and zircon in the matrix of the SBW-4 glass.

The density of two selected glasses, SBW-5 and SBW-10, was 2.6 and 2.7 g/cm³, respectively. The slightly higher density of the SBW-10 glass as compared to the SBW-5 glass is due to elevated sodium, vanadium, and zirconium oxides content.

Table IV. Chemical Composition of Matrix Glasses and Inhomogeneities by SEM/EDS.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	TiO ₂	V ₂ O ₅	Cr ₂ O ₃	MnO	FeO	CoO	NiO	CuO	ZnO	ZrO ₂	Σ*
SBW-1																		
Matrix	11.73	0.65	4.83	53.73	-	1.18	5.61	0.39	2.57	-	0.30	2.12	-	-	-	0.38	1.54	85.02
Matrix	11.89	1.01	5.80	52.67	0.57	1.07	5.47	-	2.56	-	0.28	1.73	-	0.16	-	-	1.64	84.85
Point	10.04	0.98	5.48	53.87	0.60	1.15	5.35	0.19	2.75	-	0.17	1.91	-	-	-	-	1.58	84.07
SBW-4																		
Matrix	10.19	1.18	7.52	53.97	0.60	1.41	5.12	0.19	3.08	-	0.12	1.42	-	-	0.22	0.27	2.21	87.49
Matrix	9.78	1.13	7.47	52.89	0.63	1.33	5.01	-	3.31	-	-	1.63	-	-	0.22	-	2.34	85.76
Cord	6.71	1.18	6.43	49.59	0.36	1.36	4.79	-	2.85	-	0.21	1.34	-	0.21	-	0.24	6.03	81.31
Zircon	5.10	0.43	2.84	25.75	-	0.79	2.40	-	1.42	-	-	0.59	-	-	-	-	58.28	97.60
Quartz	-	0.13	0.30	98.66	-	-	0.12	-	0.15	-	-	-	-	-	-	-	0.98	100.33
SBW-9																		
Matrix	9.59	1.45	8.65	50.20	0.66	1.54	5.52	-	3.79	0.25	0.28	1.55	-	-	-	-	2.33	85.81

Cord	9.26	1.36	15.29	55.21	0.71	2.68	9.00	0.18	2.85	-	-	1.00	-	-	-	-	2.52	100.06
Point	9.16	1.41	8.70	49.38	0.49	1.63	5.57	0.20	4.00	-	0.12	1.25	0.14	-	-	-	2.98	85.04
Point	8.07	1.28	8.89	51.00	0.59	1.59	5.84	-	3.70	0.22	0.15	1.33	-	-	-	-	2.69	85.35
SBW-10																		
Matrix	9.86	1.54	9.28	49.79	0.68	1.61	5.51	0.14	3.83	-	0.34	1.49	-	-	-	-	2.79	86.87
Matrix	10.10	1.54	9.10	49.88	0.44	1.53	5.47	0.11	3.87	-	-	1.51	-	-	-	-	2.56	86.11
Point	8.86	1.31	9.24	49.61	0.69	1.51	5.28	0.14	3.91	-	0.20	1.36	-	0.21	-	-	2.84	85.16

Note: matrix – scan by area 1 mm x 1 mm; point – electron beam 5 µm in diameter; * the rest – Li₂O, B₂O₃.

CONCLUSION

The possibility to vitrify INEEL SBW using the ICCM technology at engineering scale has been demonstrated in principle. Vitrification processing with slurry feeding and a semi-continuous (batch) mode of operation was performed in the 215 mm inner diameter x 521 mm high cold crucible designed by SIA Radon. Processing occurred for 48 hours at steady-state conditions and for 76 total hours. A total of 70 kg of glass was produced, with 59 kg of glass poured into containers through the pouring unit. The process temperature ranged between 1350-1370 °C (melting) and 1240-1250 °C (slurry feeding). The elevated process temperature, as compared to the lower target, was due to boron volatilization caused by volatility of boric acid with aqueous vapors. Corrosion of the cold crucible components, including the pouring unit, was negligible.

Off-gas temperature at the melter outlet ranged between 40 and 115 °C depending on the process step (average value was 63 °C). The average gas volume flow rate was 12 normal m³/h. The average aerosol concentration at the melter outlet was 142 mg/m³ and 33% of the total aerosols weight was ≤1 µm particles. The average concentrations of acid gases at the melter outlet were 22 mg/m³ for SO_x and 13 mg/m³ for HCl.

Chemical analysis of the glass product showed that the glass composition was depleted with boron during the run. The glass was structurally and compositionally homogeneous with minor inclusions of gas bubbles and rare grains of residual quartz, baddeleyite and zircon.

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