Cold Crucible Induction Melter (CCIM) Demonstration Using a Representative Savannah River Site Sludge Simulant On the Large-Size Pilot Platform at the CEA-Marcoule - 8503

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ABSTRACT

The cold-crucible induction melter technology (CCIM) is considered worldwide for industrial implementation to overcome the current limits of high level waste vitrification technologies and to answer future challenges such as: new or difficult sludge compositions, need for improving waste loading, need for high temperatures, and corrosive effluents. More particularly, this technology is being considered for implementation at the US DOE Savannah River site to increase the rate of waste processing while reducing the number of HLW canisters to be produced through increased waste loading and improved waste throughput.

A collaborative program involving AREVA, CEA (French Atomic Energy Commission), SRNL (Savannah River National Laboratory) and WSRC (Washington Savannah River Company) has thus been initiated in 2007 to demonstrate vitrification with waste loadings on the order of 50% (versus the current DWPF waste loading of about 35%) with a PUREX-type waste composition (high Fe_2O_3 composition), and to perform two pilot-scale runs on the large size platform equipped with a 650 mm diameter CCIM at the CEA Marcoule. The objectives of the demonstrations were 1) to show the feasibility of processing a representative SRS sludge surrogate using continuous slurry feeding, 2) to produce a glass that would meet the acceptance specifications with an increased waste loading when compared to what is presently achieved at the DWPF, and 3) achieve improved waste throughputs.
This presentation describes the platform and the very encouraging results obtained from the demonstration performed at temperatures, specific throughputs and waste loadings that overcome current DWPF limits. Results from the initial exploratory run and second demonstration run include 1) production of a glass product that achieved the targeted glass composition that was more durable than the standard Environmental Assessment (EA) glass, 2) successful slurry feeding of the CCIM, and 3) promising waste processing rates (at 1250°C and 1300°C melt pool temperature) that could result in processing of the Savannah River HLW faster than could be currently achieved with the existing Joule Heated melter in DWPF.

INTRODUCTION

In order to demonstrate the advantages of the cold crucible induction melter (CCIM) vitrification technology developed jointly by AREVA and CEA over the current DWPF ceramic melter, a feasibility demonstration has been proposed on the CFA 2001 large size platform of Marcoule [1], equipped with a 650 mm diameter CCIM. The demonstration was performed with a liquid feed surrogate, representative of a waste stream previously treated at the DWPF.

The program was divided into two runs:

- Run #1, designed as an exploratory run, in order for CEA to get acquainted with the waste, the glass, the type of feeding, while enabling the derivation of optimum operating parameters necessary for the performance of run #2. The operating temperature for this run was 1250°C.
- Run #2, during which stable and controlled operation at maximum throughput was to be demonstrated, for 1250°C and higher temperatures.

This paper describes the activities performed to prepare this demonstration, as well as the performance of the two demonstration runs. Preliminary conclusions that can be drawn from this first demonstration are presented.

DESCRIPTION OF THE PLATFORM

The demonstrations have been performed at the Marcoule site by CEA/DTCD/SCDV using the existing Large Scale Integrated CCIM Pilot Platform (see Figure 1), which was adapted to the specific requirements of the project.

The demonstration platform is self-contained and comprises all the systems and components necessary to perform large-scale 72-hour continuous demonstration runs: a melter feed system, a 650-mm diameter CCIM, a glass pouring station, a canister filling station, a complete off-gas treatment system, and related auxiliary equipment, including the control system. The platform is installed on four floors covering 160 m² at ground level.

Fig. 1. General view of the platform.

Melter Feed System

For this demonstration, the existing slurry feed system of the demonstration platform was used. This melter feed system includes a preparation and feed tank located inside the building and equipped primarily with heating coils (heated with steam provided by the site) and a mechanical stirrer capable of mobilizing and homogenizing highly concentrated slurries. The stirrer is equipped with three levels of blades: 1 lower, smaller blade below two levels of full size blades. The useful volume of the feeding tank is 8 m^3. This tank can be dry-fed from the top and this capability has been used to add glass frit to the sludge surrogate. The tank is also equipped with dip tube bubbler-based level and density measurements, and a thermocouple for temperature measurement. The built-in stirring capability of the feeding tank is complemented by the implementation of a high flowrate (> 5 m^3/h) recirculation loop extracting the slurry from the bottom of the tank and re-injecting it at the top of the tank. Sampling can also be performed on this loop, directly below the bottom of the tank.

Vitrification System

The melter selected for the demonstration was an existing 650-mm diameter cold-crucible induction melter that had been used previously on another platform at Marcoule. This melter was an early version used for development, and not representative of the robust design which has been qualified for radioactive operations in one of the La Hague HLW vitrification plants.

The melter was powered by a ~270 kHz, 600 kW high frequency generator delivering power into a copper inductor wrapped around the melter sectored vertical wall. The power supply line to the inductor integrates a high frequency / high voltage line and an impedance adaptation device (capacity assembly) to adapt the generator to the load in the crucible. The tuning of this impedance adaptor is based on modeling and calculations run prior to the demonstration, which take into account the physical properties of the glass versus temperature (thermal conductivity, electrical conductivity, and viscosity).

The platform includes a cooling loop with separate branches for the melter, the dome, and for ancillary equipment, as well as an emergency cooling system for the melter. Each branch is individually equipped with temperature and flow rate measuring devices to gather data inputs necessary for the development of a thermal balance.

The melter was equipped with a water-cooled retractable mechanical stirrer and its motorization, 3 cooled sparge tubes to assist melt homogenization, and a pouring device consisting of two cooled sliding valves and their actuators. The melter is also equipped with thermocouples for measuring the melt temperature, either from the top or from the bottom; in this case they are inserted into molybdenum inserts. Only one of the thermocouples is required to be operational for successful demonstration performance.

A viewing system which included a video camera and a viewing port was mounted onto the dome of the CCIM and provided for the remote monitoring of the cold cap.

Glass Pouring Enclosure and Canister Filling Station

During glass pouring, the receiving canister was located inside an insulated enclosure equipped with weighing scales. The canisters were made of standard carbon steel and can hold approximately 400 kg of glass.

Glass sampling could be performed below the outlet of the CCIM pouring system by grabbing small quantities of glass into steel or cast iron pans.

Off-gas Treatment System (OGTS)

At the melter off-gas outlet, the vertical part of the off-gas pipe is equipped with a mechanical device (ram) allowing recycling any deposit into the melter. The horizontal pipe to the dust scrubber is equipped with water injection systems for the removal of potential deposits and with fittings for pressure and temperature measurements. The off-gas then flows through a dust scrubber, a condenser, and a washing column. A centrifuge extractor extracts the off-gas and provides for a slightly negative pressure in the system. The configuration of this off-gas treatment system is similar to the La Hague HLW vitrification facilities and is different from the DWPF OGTS. See Figure 2.

(L = Level; P = Pressure; T = Temperature, D = Density; pH = pH; F = Flowrate; S = Sampling)

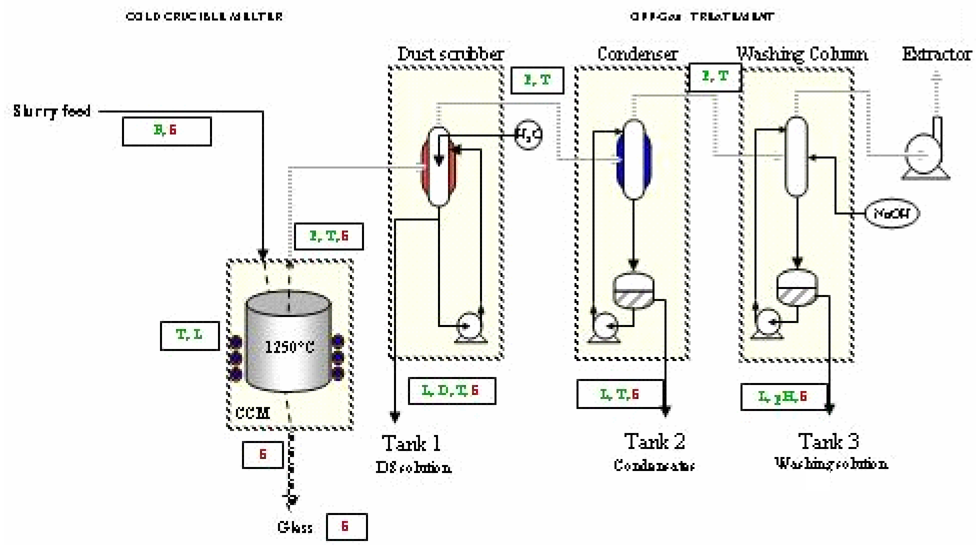

Fig. 2. Diagram of the off-gas treatment system – CCIM platform – CEA Marcoule.

The dust scrubber is a heated vessel topped with a column in which the off-gas is contacted with a counter-current flow of liquid circulated from the vessel in order to trap the entrained particles. The dust scrubber is heated so that no condensation occurs in it. In normal operation, the dust scrubber is continuously fed with a small flow of fresh water, and the excess liquid, loaded with the collected material, is recycled to the CCIM with the main feed. This represents around 10 % of the feed. For the 2 demonstration runs described in this paper, the excess liquid was not recycled to the CCIM with the main feed but was diverted from the scrubber into a dedicated receipt tank, in order to measure the maximum achievable throughput in a configuration compatible with the actual DWPF system. The cleaned off-gas then flows through a condenser where the moisture is condensed. The condensates are collected into a specific cooled tank. Downstream of the condenser, the off-gas flows through a washing column where it is contacted with a caustic solution to remove the acidic gases (NO_x and others).

The three components can be sampled to support the evaluation of the distribution of entrained and volatilized species. Each of them is equipped with level and temperature measurement devices. Pressure is measured before and after each piece of equipment.

Control and Monitoring

The Large Scale Integrated CCIM Pilot Platform is fully instrumented and operated remotely using a Process Logic Controller and a Digital Control System with a multi screen display. All the process parameters can be monitored, time-stamped, and recorded to provide historical trend information. The control system includes warning thresholds on each critical measurement and automatic shut-down sequences to assure safe operation of the system.

FEED SELECTION

A Sludge Batch 3 PUREX-type waste composition was selected for the demonstration (see Table I), since it was representative of a moderately washed, iron-rich sludge that was being processed at the DWPF. DWPF melt rate and waste throughput capability with real SB3 radioactive waste was well characterized, thus allowing a meaningful comparison with the results obtained using the CCIM with SB3 simulant. However, as a result of various practical constraints, the simulant did not undergo the acid addition steps used in the DWPF Chemical Processing Cell (CPC) so an alkaline feed was used, uranium has been removed from the composition and concentration has been limited (no possibility to evaporate more water using the slurry feed tank at the demonstration window).

Table I. Characteristics of the Simulant[a]

	Target	Batch 1	Batch 2	Batch 3
Oxides (wt %)				
Al_2O_3	16.9	17.39	17.13	17.13
CaO	3.31	2.79	3.49	3.47
Cr_2O_3	0.224	0.20	0.19	0.20
CuO	0.064	nm	0.18	0.18
Fe_2O_3	37.3	35.04	33.89	34.89
K_2O	0.339	0.74	0.84	0.84
MgO	3.81	4.08	3.88	4.03
MnO	7.09	9.05	8.39	8.63
Na_2O	24.4	22.55	23.36	23.29
NiO	1.93	1.29	1.75	1.81
SiO_2	3.97	3.95	3.96	3.98
ZrO_2	0.19	0.17	0.16	0.17
Total	100.00	97.22	97.22	98.61
Anions (mg/kg)				
NO_2	36242	36300	39150	37200
NO_3	35348	16000	30000	29200
SO_4	1902	1895	1785	1710
C_2O_4	1111	1340	1885	1765
Physical properties				
Total solids (wt %)	33	30.17	32.5	30.82
Calcined solids (wt %)		21.31	22.40	21.13
Density (g/ml)		1.25	1.29	1.26
pH	12.7	nm	nm	nm

[a]Batch 1, batch 2 and batch 3 are 3 batches prepared separately.

GLASS FORMULATION AND CHARACTERIZATION

One of the major advantages identified for the CCIM is its ability to operate at a higher temperature than the current DWPF melter, a fact which allows processing melts with liquidus temperatures higher than those that can be processed in a traditional Liquid Fed, Joule-Heated Ceramic Melter. For those glass systems which are liquidus (TL) limited, use of the CCIM technology offers the potential to increase waste loading. The demonstration initially targeted a waste loading of 50% (on a calcined oxide basis), corresponding to an increase of 30 % when compared to the best performance of the DWPF with this type of waste (38% waste loading). As will be discussed an even higher waste loading (52%) was targeted for run #2.

SRNL developed a glass formulation and a frit compatible with processing in the CCIM (see Table II), using the existing DWPF process control models (which did not cover the exact composition and temperature domains of the demonstration) as a guide followed by subsequent experimental scoping studies to characterize specific process and product performance properties. Due to schedule constraints, the glass composition was not optimized for melt rate and/or waste loading or other glass properties such as liquidus for instance. The resulting nominal composition had a waste loading of 50%, and a liquidus between 1250 and 1300°C, that is close or above the melting temperature. The formulation studies with this waste type and the associated characterization also indicated a tendency to form nepheline upon heat treatment for waste loadings between 45 and 50%. When waste loading increased, this tendency to form nepheline seemed to disappear, thus actually improving glass quality. With this composition, it seems that the usual predictions regarding nepheline formation did not fully correspond to the observed glass behavior. This would require further evaluation to develop a better model prediction for future applications at high waste loadings and high temperature. The complete formulation and characterization report has been published by SRNL [2,3].

Table II. Nominal Frit and Glass Composition (50 % Waste Loading)

Oxides	202-A-11 Frit	HTLG-21 target glass
Al_2O_3		8.45
B_2O_3	9.00	4.50
CaO		1.65
Cr_2O_3		0.10
CuO		0.04
Fe_2O_3		18.65
K_2O		0.17
Li_2O	6.00	3.00
MgO		1.91
MnO		3.54
Na_2O	3.00	13.59
NiO		0.96
SO_4		0.36
SiO_2	82.00	42.97
TiO_2		0.03
ZrO_2		0.09
Total	100.00	100.00

In addition, it was necessary to determine other key physical properties of the molten glass in order to, on one hand, verify its processability in the CCIM and, on another hand, obtain the data required as input into the electrical model for the melter for the pre-configuration of the power supply system. The measured properties were viscosity, electrical resistivity and heat conductivity. They are given in Figure 3. All the properties are suitable for a CCIM processing of the HTLG-21 glass. However, a tendency to foam was detected from about 1250°C upwards.

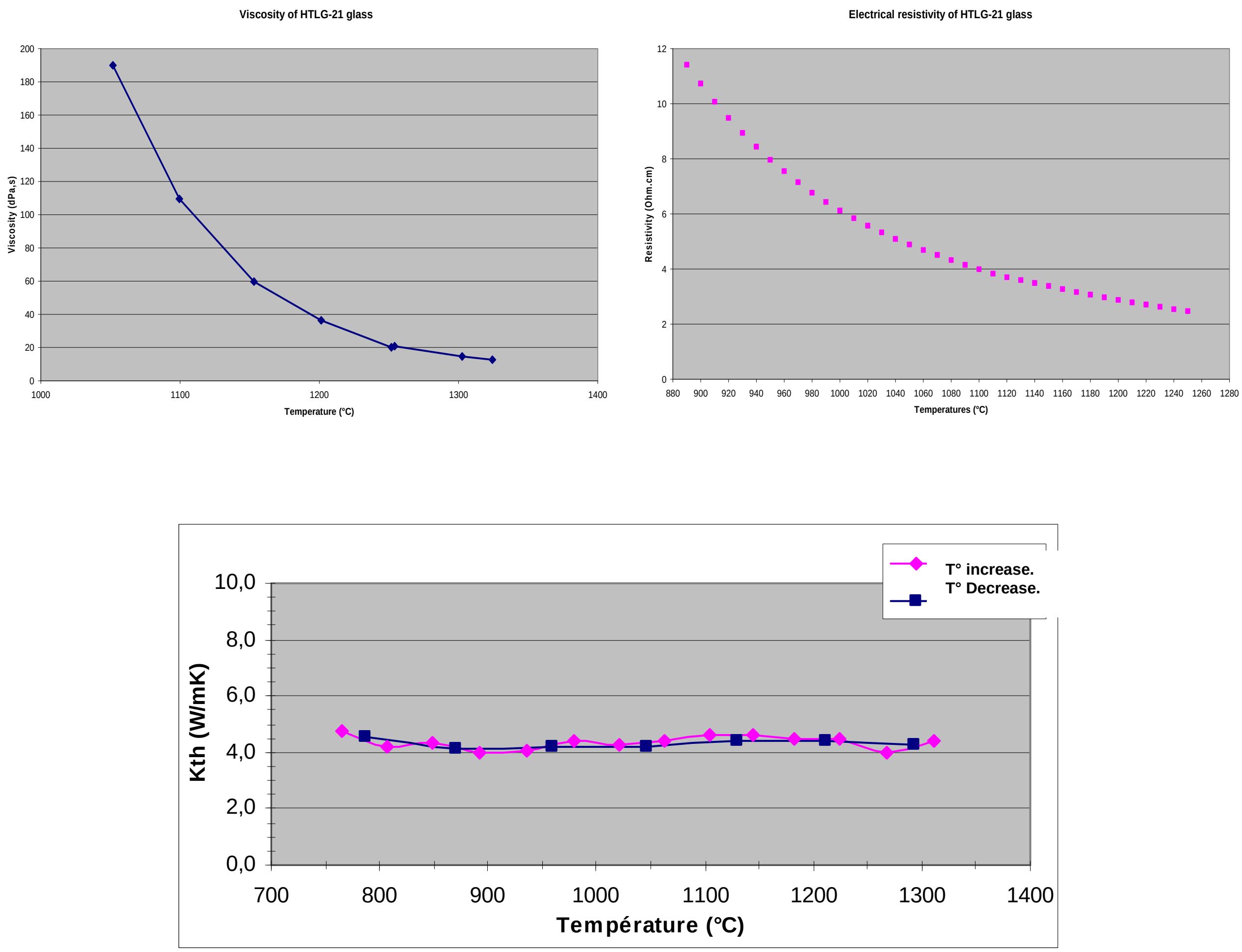

Fig. 3. Viscosity, electrical resistivity, thermal conductivity as a function of temperature – Nominal HTLG-21 glass

EXPLORATORY RUN (RUN #1)

Sampling of feed surrogate was performed downstream from the recirculation pumps and the samples characterized (see Table III).

Table III. Measured Characteristics for Feed Surrogate inside the Feed Tank for Exploratory Run (Run #1)

Density of the slurry mixed with glass frit (g/cm^3)	1.36
Solids contents (computed from solids/oxide ratio in sludge)	43 wt% (instead of 45 wt% expected for the alkaline material)
Oxide contents (Measured LOI at 1000°C)	35 wt%
Weight of glass produced per L of feed	476 g
Weight of water per L of feed	775 g

The exploratory run was dedicated for CEA to obtain an initial knowledge of the feed and glass behavior in the CCIM and on the platform, at a temperature of 1250°C, with a target waste loading of 50%. Start-up, melting and pouring were very easy. Progressive feed rate increase was started during this run at 1250°C, and the feed rate reached was 26 L/h of feed, corresponding to a glass production rate of 12.4 kg of glass per hour (27.3 lb/hr) (glass flux of 37.4 kg/h/m²)) or a waste throughput of 6.2 kg/h (or 13.7 lb/hr) (waste flux 18.7 kg/h/m²).

During this exploratory run, 336 kg of glass were fabricated; two pouring operations and a draining operation were performed. The product sampled during both pouring operations was analyzed at SRNL for chemical composition, crystallization, and durability. The as-poured glass was compositionally homogeneous, with some crystallization, as could be expected from the composition. The composition was close to the expected composition, with an estimated waste loading slightly below 50%. The PCT results for the as-poured glass were fully acceptable. However, as expected from the formulation studies, the modified DWPF Centerline Canister Cooling (CCC – adjusted for the higher nominal melter temperature of 1250°C) heat-treated glass developed some nepheline and aegirine and, although still strictly acceptable when compared to the EA glass, it displayed a quality significantly degraded when compared to the as-poured glass, which is typical for CCC glasses. The glass samples taken from the canisters (which undergo a cooling cycle faster than the CCC-curve) displayed only magnetite-type crystals under XRD, without any sign of nepheline. As a result, and although the modified CCC cooling curve may be conservative, it was decided, for the baseline demonstration run, to target a higher waste loading, in order to test the hypothesis of quality improvement with increased waste loading with this feed. The target waste loading was set at 52% for baseline demonstration run (run #2). The decision to target the higher waste loading was based on the results of a variability study performed by SRNL which indicated that the probability of avoiding the nepheline formation region (upon slow cooling) increased as waste loading increased above the 50% mark. It was also noted that as higher waste loading were targeted, the T_L of the glass system would increase so a balance between process and product performance issue was met for the baseline demonstration.

Other lessons learned from this run dealt with the behavior of the feed in the feed tank and its tendency to plug the feeding nozzles if not adequately rinsed immediately after feeding is stopped.

BASELINE DEMONSTRATION RUN

For the baseline demonstration run (run #2), the amount of sludge simulant transferred to the feeding tank was 2546.5 kg. To this, 479.3 kg of frit was added resulting in a batch of 3025.8 kg of feed (2140 liters). 1.3 kg of cesium nitrate (molar weight 194.91 g) (886.4 g Cs) was also added to the feed batch in order to get a cesium concentration of ≈ 414 mg/l Cs to the feed, which with the expected calcine contents of the feed, would have correspond to a Cs_2O concentration in the glass of about 0.092 wt%.

Sampling was performed prior to run #2 downstream from the recirculation pumps and confirmed the fact that the solution was more diluted than expected (density : 1.35, LOI @ 105 °C : 38.2 wt%, LOI @ 1000°C : 30.8 wt%). This is amplified by the fact that the waste loading is 52 %, which means that less frit is added per g of waste oxide in the sludge, thus decreasing the wt% oxides.

The run #2 was performed with a schedule for feed ramp-up from 20 L/h in order to determine the maximum capacity on the basis of a cold cap coverage surface of 95 %, corresponding approximately to the bubblers surface impact. A maximum feed rate of 50 L/h was determined at 1250°C and 60 L/h at 1300°C (see Table IV). Stable operation was demonstrated for a 13 hour period at 45 L/h. During the run, 3 start-up operations, 3 draining operations and 7 pouring operations were performed. 1232 liters of feed surrogate were processed and 477 kg of glass were produced.

With this specific feed composition, the maximum feed rate value of 50 L/h (feed flux of 150 L/h/m²) at 1250°C corresponds to a glass production rate of 20.9 kg/h (or a glass flux of 63 kg/h/m²) and a waste throughput of 10.9 kg/h (waste flux 32.7 kg/h/m²), that is about 44% of the waste throughput of the DWPF for the SB3-Frit 418 system. The feed value of 60 L/h (feed flux 180 L/h/m²) at 1300°C corresponds to a glass production rate of 25 kg/h (or a glass flux of 75.4 kg/h/m²) and a waste throughput of 13 kg/h (or a waste flux of 39.2 kg/h/m²).

Table IV. Calculation of Throughputs Achieved during Baseline Demonstration Run (Run #2)[a]

Temperature	Feed rate (Feed flux)	Glass throughput @ 417.1 g glass/l of feed (glass flux)	Waste throughput @ 52 % WL (waste flux)	Waste throughput @ 53 % WL (Waste flux)
1250°C	50 L/h (150 L/h/m²)	20.9 kg/h (63 kg/h/m²)	10.9 kg/h (32.8 kg/h/m²)	11.1 kg/h (33.4 kg/h/m²)
1300°C	60 L/h (180 L/h/m²)	25 kg/h (75.3 kg/h/m²)	13 kg/h (39.1 kg/h/m²)	13.25 kg/h (39.9 kg/h/m²)

[a]The waste loading was calculated from the glass composition (see table V) to lie between 52 and 54 %.

In figure 4, TC1 and TC2 are the two thermocouples inserted from the top of the CCIM measuring the melt pool temperature. It can be seen from these measurements, that the bath temperature was quite stable during the whole run and at its target value, except of course when the generator was stopped for cleaning operations; once during the run, a rectifier bridge defect of the power generator caused a short power shutdown promptly corrected. It can be noted that during this test, the temperature was manually set by action on the power by the operator; however this regulation can be made automatically once the temperature response is well known for a specific glass.

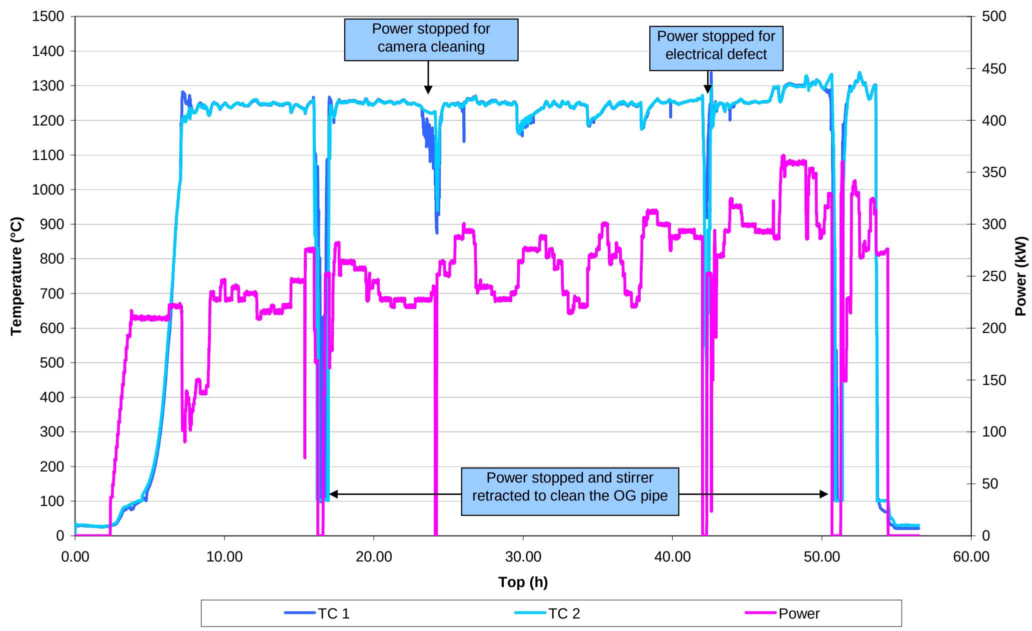

Fig. 4. Evolution of power and temperature in the melt during demonstration run.

During the run, some OGTS (Off Gas Treatment System) pipe cleaning system improvements performed quite well (cooled vertical part equipped with a mechanical ram that could be actuated manually), resulting in smaller head losses and less deposits in the pipe than during exploratory run. However, when the feed rates became really high the OGTS was handicapped by the existence of high non condensable gas flow which disturbed condenser operation. The feeding system was operating correctly until the 2/3 of the run, when, owing to the high feed rates involved, the level in the feed tank became too low to ensure proper stirring. It was then decided to add the remaining aged feed from exploratory run which had become significantly more viscous, since the frit had remained mixed with this very alkaline sludge for long enough (10 weeks) to start dissolving. From that point, the operation was significantly disturbed by repeated plugging events, in the feeding nozzles as well as in the whole feed lines. Since the maximum throughput had been reached at 1300°C, it was decided to terminate the run. These observations led to consider a complete re-configuration of the whole feeding line, including the tank, if, necessary, for future runs, and to decrease air inleakage as much as possible.

Glass samples from the pours taken during the part of the run when the system was operated at or close to its maximum throughput were analyzed by SRNL for chemical composition, crystallization and durability. The as-poured product was homogeneous, and a little more crystallized than the product from demonstration run (Run #2), a fact that had to be expected since the waste loading was higher (estimated between 52 and 53 wt%). The chemical composition (see Table V) was stable in time and close to what was expected. The XRD results on the as-poured and CCC-treated samples confirmed the findings of the formulation studies: no trace of nepheline or aegirine was detected in both the as poured and CCC-treated samples. SEM/EDS performed on samples taken from the canisters confirmed these observations. The only detectable crystalline phases were iron-rich spinels with varying levels of Cr and other elements.

The PCT results (see Table VI) were in agreement with this observation: the leach rates were about an order of magnitude better than the standard EA glass for both the as-poured and CCC-treated samples.

A preliminary evaluation of cesium volatility in the technology at these high temperatures conditions can be estimated. The glass analyses indicate that between 88 and 93 % of the cesium remained in the glass in these high temperatures, high melt turbulence, but short residence time conditions. Although the duration at 1300°C was short, there was no sign of increased volatility when the temperature was raised from 1250 to 1300°C.

Table V. Chemical Analysis of Glass Sampled on Pouring – Baseline Demonstration Run (Run #2)[a]

Oxide wt%	Expected based on normalized sludge composition	Target based on as-analyzed sludge composition	Pour #7 and 8 average	Pour #9	Pour # 10 average
Al2O3	9.03	8.91	8.93	8.87	8.74
B2O3	4.25	4.25	4.07	3.95	3.96
CaO	1.83	1.80	1.74	1.64	1.60
Cr2O3	0.10	0.10	0.11	0.11	0.11
CuO	0.09	0.09	0.04	0.04	0.04
Fe2O3	18.40	18.14	18.1	18.2	17.9
K2O	0.44	0.44	0.52	0.41	0.40
Li2O	2.76	2.76	2.53	2.56	2.56
MgO	2.12	2.10	2.13	2.12	2.12
MnO2	4.55	4.49	4.69	4.71	4.65
Na2O	13.61	13.44	15.4	15.6	15.0
NiO	0.96	0.94	0.83	0.87	0.88
SO4			0.35	0.37	0.29
SiO2	41.77	41.74	40.5	40.8	40.8
TiO2			0.114	0.07	0.06
ZrO2	0.09	0.09	0.13	0.13	0.14
Cs2O		0.092	0.081	0.086	0.082

[a](pour #7 & 8 : 1250 °C, 45 L/h, pour #9 : 1250 °C, 50 L/h, pour # 10 : 1300 °C, 60 L/h)

Table VI. PCT Testing of Pour Samples from Baseline Demonstration Run (g/glass/L Leachant)

	B	Li	Na	Si
EA glass	18.54	9.71	13.82	3.90
Pour #7 as-poured	1.01	0.92	0.97	0.51
Pour #7 – CCC	0.72	0.77	0.84	0.46
Pour #8 – as poured	0.94	0.92	1.00	0.53
Pour #8 – CCC	0.72	0.77	0.88	0.47
Pour #9 – as poured	0.96	0.89	0.94	0.52
Pour #9 – CCC	0.67	0.72	0.83	0.45
Pour #10 – as poured	0.91	0.89	0.97	0.51
Pour #10 – CCC	0.63	0.70	0.85	0.44

CONCLUSIONS

In conclusion, this joint effort conducted by CEA, AREVA, SRNL and WSRC led to very encouraging results, demonstrating waste throughputs 44 % that of the DWPF ceramic melter throughput in a 650 mm CCIM melter for the same waste type with a Sludge Batch 3 PUREX-type waste feed flux of 150 L/h/m² demonstrated @ 1250 °C.

The very high waste loading (above 52%) allows reducing the amount of glass to be produced by about 27% to treat the same amount of waste when compared to previous DWPF operation for this specific type of feed, since 27 % less glass is needed to immobilize the same amount of waste. It was also demonstrated, for this type of feed, an unusual behavior with regard to nepheline formation, which would require further evaluation for future applications. The product from the baseline demonstration run, with a waste loading of at least 52%, displayed a very good quality. Stabilized operation close to the maximum throughput was demonstrated. Cesium volatility was apparently between 7 and 12 % (based on glass analysis); however this value is only preliminary.

This demonstration also allowed the CEA to better understand the SRS slurry feed behavior and to propose adaptations to the platform for any future demonstrations using this type of feed. Finally, use of a large diameter CCIM (~1 meter) may allow faster processing of the SRS HLW than can be achieved with the current DWPF melter.

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