HANFORD HIGH LEVEL WASTE PROCESSING IN A COLD CRUCIBLE MELTER: TEST RESULTS OBTAINED IN THE FRAMEWORK OF THE TWRS-P CONTRACT

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ABSTRACT

In the framework of the Hanford Tank Waste Remediation System (TWRS), the DOE has chosen a multi-step privatization approach with a phase IA proof-of-principle effort prior to the design, construction and operation of an active vitrification plant for Low Active Waste (LAW) and possibly High Level Waste (HLW).

In September 1996 the DOE selected a team led by Lockheed for the Phase IA proof-of-principle effort. Within this, team SGN was the technical leader for the High Level Waste process and proposed to implement the cold crucible technology, which offers significant advantages for processing the Hanford High Level Waste.

SGN carried out an extensive testing program to establish the process feasibility and meet contractual requirements. These tests were performed successfully by Pacific Northwest National Laboratory (PNNL) and the French Atomic Energy Commission (CEA) in Marcoule.

The test program included the following parts:

- Preliminary glass formulation study for the HLW feed, based on theoretical analyses and confirmed by experimental laboratory work.
- Variability study of the glass properties for major constituents and some minor constituents.
- Vitrification and analysis of a radioactive sludge sample provided by the DOE (Hanford tank 241-C-106).
- HLW process feasibility demonstration at laboratory scale for the HLW feed pretreatment steps and at pilot scale (40% of industrial scale) for the calcination and vitrification steps.

The work was completed in December 1997. The radioactive and inactive glass samples prepared at PNNL and in France not only met the requirements but clearly indicated that higher performance can be achieved.

INTRODUCTION

In the framework of the Hanford Tank Waste Remediation System (TWRS), the DOE has chosen a multi-step privatization approach with a phase IA proof-of-principle effort prior to the design, construction and operation of an active vitrification plant for Low Active Waste (LAW) and possibly High Level Waste (HLW). In September 1996 DOE selected a team led by Lockheed for the Phase IA proof of principle effort. Within this team SGN was the technical leader for the High Level Waste process.

Drawing on the extensive experience gained by the COGEMA/CEA group in the field of industrial High Level Waste processing and vitrification, SGN proposed to implement the cold crucible melter (CCM) technology as the best adapted to process the Hanford High Level Waste. This technology, which supports high temperature glass melting, should also provide new and efficient answers to some of the very demanding phase II challenges.

In order to establish the feasibility of treating Hanford waste using this process and to meet contractual requirements, SGN launched an extensive testing program in collaboration with Pacific Northwest National Laboratory (PNNL) in the United States and the French Atomic Energy Commission (CEA) in France. After careful examination of the data pertaining to the waste, the first step was to select glass formulations that allowed meeting the contract requirements while being processable. The two resulting baseline formulations were then completely characterized at the lab scale and used to perform larger scale pilot demonstrations. As part of the contract, a radioactive sludge sample from one of the Hanford tanks was also successfully vitrified using one of the formulations developed.

THE REQUIREMENTS

The task for the HLW facility involved immobilizing a high-level, sludge-based waste stream (envelope D) directly received from Hanford Site, together with the high level fraction separated from the Low Activity Waste (LAW) streams (envelopes A, B and C) in the LAW processing facility. The baseline envelope D stream was defined by a set of maximum component limits in the feed. Expansions of the baseline limits, including increased sodium, sulfur, aluminum, chromium, iron and nickel contents, were also to be considered.

The major contractual requirements for the process itself were to demonstrate the capability of vitrifying 60 MT of waste oxides (sodium oxide and silica excluded) over a 12 month period and to process 245 MT of waste oxides (excluding sodium oxide and silica) over 5 years (or 465 MT over 9 years).

The contractual requirements for the product were essentially:

- To meet the product and disposal requirements expressed in the WASRD (2), WAPS (3) and QARD (4).
- To achieve a waste loading of at least 25 wt% on an equivalent oxide basis for non-volatile components of waste envelope D, excluding sodium oxide, silica and other materials that result from processing Low Activity Waste. Hereafter, the waste loading calculated using this convention will be called "reduced" waste loading, as opposed to the "true" waste loading, which accounts for all the oxides from the waste.
- To vitrify a radioactive sludge "Envelope D" sample provided by the DOE and to demonstrate compliance of the vitrified sample.

SELECTION OF THE PROCESS

The core of the selected process is based on the extensive experience gained by the COGEMA/CEA group in the field of high-level waste vitrification, at Marcoule and La Hague in France. A similar process has been implemented in the BNFL-operated reprocessing plant of Sellafield to vitrify the High Level Waste solutions. The vitrification process developed by the CEA is a continuous, two-stage process in which the waste is calcined in a rotary kiln calciner prior to being melted with the glass-forming additives in a small size melter.

This process allows designing a very compact vitrification cell, where all the equipment can be designed in small modules, easy to access and replace and generating little secondary waste. A failed melter can be size-reduced in the maintenance cell adjoining the vitrification cell and fit into a volume representing less than a glass canister. Melter replacement can be achieved in less than a week, as demonstrated in the R7 and T7 facilities at La Hague.

The calcination operation is a proven technology, with demonstrated safety. The calciners in R7 and T7 vitrification plants at La Hague have provided uninterrupted service since the plants started in 1989 and 1992, respectively. The selected process proposed to use a calciner of the same type with increased throughput (120 to 150 l/h evaporating capacity).

For glass fabrication, the cold crucible melter (CCM) technology has been proposed, since its unique characteristics were felt to be particularly well suited to the various challenges involved in this project. In the CCM concept, the glass "self-heats" by the Joule effect created by currents directly induced into the melt from external high frequency inductors. Because heat is not transmitted by conduction, the melter crucible remains cool (hence the name "cold crucible"). This causes a thin layer of vitrified material to coat the walls, protecting them from the corrosive glass melt. In addition, since the source of energy is outside the melter, the recurring problem of electrode corrosion is eliminated.

Those two features, in addition to providing a considerable lifetime for the melter, allow overcoming the temperature limit imposed in more conventional systems by electrode and/or refractory corrosion considerations (1050 to 1150 °C). Corrosive glass melts, which would be excluded from processing elsewhere, can be treated in such melters. No extraneous material, resulting from refractory or electrode corrosion, is added to the glass. The possibility to operate at high temperature has been recognized by many workers as a prerequisite for efficient waste minimization when the waste contains high amounts of refractory compounds or when high liquidus temperatures (T_L) are expected (5, 6). The formulation range is extended. The CCM thus seems particularly well suited to Hanford waste, which is rich in alumina and iron, with significant amounts of chromium and, in some cases, substantial quantities of zirconium.

Another favorable feature of the CCM is the pouring system: the glass is poured periodically through a bottom drain that is well adapted for the removal of bottom deposits. The drain is fit with a specifically designed progressive valve that allows adjusting the glass-pouring rate. At each pouring operation, a large part of the melt is drained. Since Hanford waste is rich in spinel promoters, such features could considerably help operation, especially when coupled with efficient melt agitation as proposed.

The CEA has considerable experience with this technology. Two pilot facilities were built at Marcoule. One of them has been operating for more than fifteen years, corresponding to several thousands hours of R&D. The technology has been able to process materials as diverse as enamels, lead glass, glass-ceramics, basalt (for heavy metals immobilization) and asbestos, at temperatures sometimes higher than 1600 °C. Two larger production units directly derived from the pilot facilities have been provided to an industrial glass producer for specialty productions. The bigger of these two melters has a throughput ranging from 200 to 400 kg/h according to the temperature (1200 to 1400 °C). A pilot melter is being run in collaboration with South Korea to process simulated power plant solid waste, mainly resins (7). The first radioactive melter of this type (liquid fed) is to be implemented at Sallugia, Italy, to process waste from MTR fuel reprocessing (8-10).

For Hanford phase I, it has been proposed to implement a CCM of 50-kg/h capacity, dry-fed through a rotary calciner. The small size of this equipment allows implementing the melter and calciner in a cell array derived from that existing in the present R7 and T7 vitrification cells of La Hague, with specific emphasis on operability and maintainability. The proposed process is described in another publication (11).

TESTING PROGRAM FOR THE DEMONSTRATION

From the beginning, it was decided to give preference to an integrated demonstration illustrating all the aspects of vitrification process design, starting from the evaluation of waste characteristics and culminating in the continuous production of fully acceptable waste forms on a 40% scale industrial pilot facility, with emphasis on the identification of potential difficulties or improvements along the line. The vitrification of the radioactive sample, required by contract, was to be incorporated into this scheme. The capacities of three complementary partners (SGN, CEA, and PNNL) were combined to reach this objective. The testing program is described in the following paragraphs. It consisted of four parts: glass formulation and characterization; pilot demonstration at 40% of the proposed industrial capacity; vitrification of a radioactive sludge sample into an acceptable glass; and further study of the influence of feed variability and minor constituents.

It was recognized early that well-defined waste feed compositions were necessary for glass formulation, process design and testing. It was therefore decided to focus on the compositions of the first wastes that would be processed in the plant during phase IB, since it would give additional positive meaning to the demonstration. Sludge from the NCAW (Neutralized Current Acid Waste) double shell tanks 241-AZ-101 and 241-AZ-102 and from the high-heat, singe-shell, tank 241-C-106 were identified. These wastes are particularly rich in aluminum and iron, with significant amounts of zirconium in some instances.

GLASS FORMULATION AND LABORATORY WORK.

The formulation work was achieved through an efficient collaboration between PNNL and the CEA.

The preliminary definition of glass formulations was performed using property/composition models developed by PNNL over the specific Hanford waste composition ranges and based on the fabrication and characterization of numerous glass samples (12-17). The following models were available: viscosity, electrical conductivity, liquidus temperature T_L, glass transformation temperature T_g, nepheline formation and phase separation, PCT releases. The target glasses had to meet the contractual requirements for waste loading and the WAPS requirement for PCT leach resistance. They also had to be processable in the proposed equipment.

The target melting temperature (e.g. temperature at which the viscosity is in the desired range) was set conservatively at 1200 °C.

The outcome of these calculations was a set of glass compositions that met the criteria, with varying viscosities at 1200 °C. These compositions were further studied by the CEA drawing on its experience of formulating durable glasses that can be fabricated with the proposed process, to yield the two baseline compositions given in table I.

TABLE I: Baseline glass compositions

Oxides	AZ-Blend baseline (wt%)	C-106 Baseline (wt%)
SiO_2	50.23	50.55
B_2O_3	10.34	8.43
Na_2O	13.14	14.96
Li_2O	1.37	1.13
CaO	0.23	0.92
MgO	0.08	0.14
Fe_2O_3	9.89	13.84
Al_2O_3	7.40	6.81
ZrO_2	1.98	0.10
Others	5.33	3.32

Following the selection of these two compositions, dry-batched glasses were characterized both at the CEA and at PNNL. Although the models were extrapolated outside of their qualified range of validity, most of the calculated values were consistent with the measured values. The major difference to be noted concerned viscosities, which were higher than calculated, even slightly outside the selected range for AZ-Blend (15.1 to 15.5 Pa.s at 1200 °C). Despite this discrepancy, the glass has been used unchanged for the pilot test described later. Both glasses had densities around 2.63 and glass transition temperatures around 500 °C. The liquidus temperatures were 1043 °C for AZ-Blend glass and less than 925 °C for C-106 glass. Testing confirmed the absence of detrimental crystallization or phase separation upon heat treatment. To be noted was the very good leach quality of both glasses, with 7-day PCT releases far better than the specification (around 0.2 g/m² for boron and 0.25 g/m² for sodium), for heat-treated glasses as well as for quenched glasses.

The knowledge acquired during this operation suggested that no glass formulation limit was reached, and that flexibility towards waste composition could be expected. The two baseline glass compositions were thus the starting point for all the demonstration studies: pilot demonstrations, vitrification of the active glass sample. In some cases, higher waste loadings were looked for and achieved.

EXPERIMENTAL DEMONSTRATION: PILOT TESTING

The prototype in Marcoule, France, was set up for demonstration testing on the two compositions. It consisted of a rotary calciner with 40 l/h evaporation capacity feeding the 550 mm diameter CCM. The CCM was representative of the proposed industrial equipment. The off-gas treatment consisted of a dust scrubber at the outlet of the calciner, a condenser, and NO_x absorption columns. The resulting glass production rate was between 19.5 and 22.5 kg/h, that is 40% of the proposed industrial capacity. The feed for both tests was slurry simulating the effluents pretreated as proposed for the industrial facility. The glass formers were added as a frit. The target "reduced" waste loading was 25 wt%.

AZ-Blend pilot test. The test took place in June 1997. The first step of the test was to determine the operating parameters for the calciner. The full pilot was then operated continuously for 61 hours. Calcination ran smoothly. At first, the melting temperature was set at 1200 °C. But, as already noticed in the laboratory, the glass was viscous at that temperature (15.5 Pa.s). It was then decided to increase the processing temperature to 1300 °C for the last three pourings and the melter flush. The viscosity of the glass was then satisfactory (about 7.4 Pa.s from the laboratory results) and pouring became easy. This experiment is a good illustration of the flexibility provided by the process. About 1400 kg of glass were fabricated, to yield six pourings at 1200 °C and three pourings at 1300 °C followed by the melter flush.

Extended glass characterization was performed for run no. 6 (last run at 1200 °C). The glass was homogeneous throughout the whole pouring operation. The "reduced" waste loading was found to be about 26 wt%. The PCT releases were slightly lower than those obtained from crucible melts (< 0.20 g/m² for both B and Na). No distinction could be made between quenched or canister-cooled glass. Some inclusions were detected: polyhedral micro-crystals consisting of chromite (O, Fe, Ni and O, Cr, Fe, Ni) and silver micro-spheres, bigger in the glass sampled from the container. No extensive crystallization was found.

In order to collect information on the behavior of volatiles at 1200 °C and 1300 °C, the solutions from the dust scrubber and the condenser were sampled during runs no. 6 (last run at 1200 °C) and no. 9 (last run at 1300 °C). No significant increase in volatility was observed for Na, B, Te, Li, F except, possibly, for sulfur.

C-106 pilot test: a similar pilot test was performed on C-106 composition in October 1997. 1300 kg of glass were fabricated during a 65-hour continuous run. In this case, no viscosity problem was experienced. The target processing temperature was 1200 °C. Sampling and testing were performed according to the same pattern as for the AZ-Blend test. The results were similar: good homogeneity, satisfactory quality, with a reduced waste loading between 24.5 and 25 wt%.

PREPARATION AND VITRIFICATION OF THE RADIOACTIVE SAMPLE

The DOE provided a radioactive envelope D sample for performance demonstration. The sludge originated from Hanford Tank C-106 and had been pretreated with two caustic leachings and three dilute hydroxide washings, following a possible industrial pretreatment scheme.

Since it was required to meet the 25 wt% reduced waste-loading criterion, the target waste loading had to be set higher, to provide a margin for experimental or analytical uncertainties. PNNL models were used to support the determination of the achievable waste loading. All waste loadings up to 31 wt% (reduced, corresponding to a true waste loading of 58 wt%) seemed acceptable. The major constraint in this case was nepheline formation.

Prior to the actual vitrification of this sample (at PNNL), both the CEA and PNNL fabricated several glass samples with target "reduced" waste loadings of 28 wt%, either from slurry simulant to test the behavior of the melt with the proposed hot-cell procedure, or dry-batched to investigate the durability of the proposed formulation. No operating difficulty was experienced. Again, high quality, durable glasses were produced. The TCLP results performed on a specific dry-batched sample spiked with 0.8 wt% CdO were also fully satisfactory. These results gave good confidence in the success of the sludge sample vitrification operation and allowed confirming the proposed vitrification procedure. A target reduced waste loading of 27 wt% was selected for the vitrification of the radioactive sample.

The vitrification of the radioactive sample itself was performed at PNNL. Glass preparation followed the pre-tested procedure, with calcination at 600 °C for 2 hours and melting at 1200 °C for 2 hours. The radioactive glass melted quietly with very little foaming and no observed fuming. The glass appeared to have a higher viscosity than the simulant glass. Part of the glass was poured onto a quenched surface. The cooled glass was close in appearance to the dry-batched simulant glass. For analysis, Envelope D glass was batched with three other samples, including a sample of ARG-1 analytical reference glass, to provide confirmation of analytical accuracy. The normalized composition of the radioactive glass sample, as determined by averaging four replicates and normalizing the total oxides to 100 wt%, is given in table II.

TABLE II: Normalized composition of the glass fabricated from the radioactive envelope D sample

Oxides	Wt %	Oxides	Wt %	Oxides	Wt %
Ag_2O	0.19	Fe_2O_3	16.69	SeO_3	0.03
Al_2O_3	8.02	La_2O_3	0.01	SiO_2	49.04
As_2O_3	0.02	Li_2O	0.84	SnO_2	0.10
B_2O_3	6.51	MgO	0.20	SrO	0.01
BaO	0.04	MnO	0.32	ThO_2	0.06
Bi_2O_3	0.06	MoO_3	0.01	TiO_2	0.15
CaO	0.74	Na_2O	14.17	Tl_2O_3	0.03
CdO	0.01	Nd_2O_3	0.04	UO_2	0.18
CeO_2	0.04	NiO	0.20	Y_2O_3	0.01
Co_2O_3	0.01	P_2O_5	0.77	ZnO	0.05
Cr_2O_3	0.23	PbO	0.32	ZrO_2	0.50
CuO	0.16	Sb_2O_3	0.10		

The "reduced" waste loading computed in various ways from this chemical analysis ranges from 27.2 wt% to 29.4 wt%. The 25 wt% requirement was thus notably exceeded.

7-day PCT testing of the radioactive Envelope D glass was successful and yielded results similar to simulant and model calculations. The 7-day normalized releases were 0.19 g/m^2 for B, 0.26 g/m^2 for Na, 0.13 g/m^2 for Si and 0.25 g/m^2 for Li. The radioactive glass easily complied with the PCT requirement established in the WAPS.

INFLUENCE OF FEED VARIABILITY: MAJOR AND MINOR CONSTITUENTS

From the above studies, the achievability of 25 wt% or higher reduced waste loadings, with the proposed CEA frit in the proposed process, was demonstrated experimentally at the crucible and pilot scales, for the two baseline compositions, including the radioactive envelope D sample.

However, the waste from Hanford Tanks is not always fully characterized and local variations are expected within each tank. In addition, before vitrification, the HLW sludges will undergo various pretreatments aimed at reducing the amount of HLW product and the products resulting from the decontamination of the low active waste stream will be added to the HLW stream. In order to be efficient, the process should thus be capable of accepting waste varying around the baseline compositions, at the best possible waste loading.

For the purpose of demonstration, a paper study was performed around the proposed baseline compositions. Plausible variation ranges for glasses were defined on the basis of tank and pretreatment data available in the literature, and considering reduced waste loadings ranging from 25 to 30 wt%. Over the defined glass composition regions, glass properties were calculated using PNNL models (Since the problem under consideration did not fall exactly in the previous field of application of these models, the calculations must only be considered as giving estimations).

According to this study, for both baseline waste types, the minimum waste loading was achievable with margins to allow for compositional and process uncertainties. Higher waste loadings were achievable with some additional compositional constraints to adjust, for instance, viscosity (AZ-Blend) or chemical durability (C-106). It was also shown that, by increasing the operating temperature in the 1200 to 1300 °C range (which has actually been done on the pilot), the acceptability range for viscosity could be widely expanded. Potentials for further waste loading improvements were identified, among which optimization of additives (for both waste types) and increased melting temperature (especially for AZ-Blend waste type) were the most promising. Some expected potential limits to the improvement of waste loading were confirmed: alkalis in the C-106-type waste, since they could induce lower durability glasses (remedial actions could be optimization of the frit and/or additional sludge washing), or aluminum, if its amount brought the glass into the nepheline formation region.

In parallel with these calculations, the CEA performed exploratory testing to investigate the influence of some minor, potentially limiting constituents on the C-106-type glass. It was confirmed that the various precipitations to be expected (such as silver micro-particles, spinels, and calcium-rare-earths phosphates) had no detrimental effects on the durability of the glass, at the higher levels expected for these elements for this waste type. It was also found that sulfate solubility in the glasses tested was in the range of 0.50 wt%. Chlorides were digested at levels reaching about 1 wt%.

CONCLUSIONS

In a very short period of time, careful design and efficient collaboration between three dedicated partners: SGN, the CEA and PNNL, have enabled a very positive response to all the contractual requirements and obtaining useful information for process design and identification of further needs. Owing to the strict schedule, and to the level of details existing for the data, a direct, realistic, focused approach was preferred to a more comprehensive and general, but time-consuming one. By combining the complementary experience and knowledge of the CEA and PNNL, the definition of two good quality, fully compliant and processable baseline glass compositions was very rapid. The radioactive sample was vitrified, using the same frit composition, with a waste loading of at least 27 wt% and its durability was similar to that of all the other glasses made until then.

Two pilot tests at 40% scale demonstrated that the baseline feeds could be calcined and vitrified with the proposed process, yielding glasses with highly satisfactory quality, very close in appearance and durability to the corresponding laboratory glasses. This pilot testing also demonstrated the flexibility of the CCM process, since, when the AZ blend glass displayed a high viscosity, pouring was improved by increasing the melting temperature to 1300 °C with no detrimental consequences. No excessive increase in the volatility of elements from the glass was observed during this temperature increase.

Potentials for waste loading improvements were identified, among which optimization of additives (for both waste types) and increased melting temperature (especially for highly refractory waste) were the most promising. On the other hand some waste components liable to limit the waste loading, and thus requiring more detailed specific studies, were identified.

In summary, the above R&D activities covered most of the important glass formulation or process aspects. Technical issues were clearly identified. The simplified procedure applied allowed us to show that efficient responses to the contractual requirements existed, with a potential for waste loading improvements, both at the level of processing (by operating the melter at higher temperature) and of glass formulation (use of more definitive data, adaptation of specific frits for each waste type). The glasses prepared at PNNL and in France not only met the DOE requirements but also clearly indicated that higher performance can be achieved. In addition, the process demonstrated its unique flexibility and its good adaptation to this type of waste. No major processing problem was encountered that could prevent process scale-up to the expected industrial throughput. These tests demonstrated the potential for improvement brought on by the proposed technology.

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