

Methodology of Qualification of CCIM Vitrification Process Applied to the Decontamination Effluent of the La Hague UP2-400 Facility – 9142

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ABSTRACT

This paper describes the methodology used to qualify a Cold Crucible Induction Melter (CCIM) process for the decontamination effluents from the UP2-400 facility (effluents coming from rinsing and decommissioning operation). This methodology consisted of five types of pilot-scale tests. The nominal and the sensitivity tests determined the operating parameters which guarantee that the industrial-scale glass has the same properties as those developed in the laboratory. The transient and degraded mode tests defined the operating parameters and the procedure for ensuring plant safety to preserve the process equipment and material. Finally, long-term testing demonstrated the viability of the operating parameters and the management of the transient and degraded modes.

On completion of this qualification program for the decontamination effluents a request for approval of production of a new glass package with the CCIM vitrification process was submitted to the regulatory authority. The main results of the qualification tests are described in this paper here.

INTRODUCTION

In 2010, the cold crucible induction melter (CCIM) [1] will replace the existing induction-heated metal melter in an AREVA vitrification cell at La Hague (France). The CCIM will allow the synthesis of several glass compositions suitable for different types of waste. The new vitrification process and the new glass must be accepted by ANDRA the agency responsible for the waste repository and by the nuclear regulatory authorities before production can begin. An application must therefore be filed for each glass variant. This file includes three sections:

- specification of the glass composition range,
- long-term glass behavior,
- process qualification.

This paper addresses only the third topic for which a process qualification methodology was developed and tested for the first time on a glass for conditioning the decontamination effluents of the UP2-400 facility.

Methodology

The process qualification program included five types of tests to specify the conditions necessary to obtain a homogeneous material.

- Tests to determine the nominal operating parameters guaranteeing the quality of the material fabricated at industrial pilot scale by final characterization of its physical and chemical properties compared with the same material synthesized in the laboratory.
- Two types of sensitivity tests:

- A chemical composition sensitivity test similar to laboratory studies intended to synthesize the potentially most difficult glass composition to fabricate at full scale, considering the technological performance possible from the selected vitrification process.
- Tests of sensitivity to the operating conditions to specify possible parameter variation ranges acceptable for the material and for the process. For CCIM glass, the parameters are the temperature, the stirring speed and the injector air flow rate.
- Transient mode tests to determine melter control parameters: the feed shutdown or calciner standby periods and the melter startup conditions were studied for decommissioning glass.
- Degraded mode tests to identify procedures for offsetting or mitigating the impact of incidents on safety, on the process equipment, and on the material. The following degraded modes were investigated:
 - the impact on the material of an interruption in the glass frit feed;
 - the consequences of restarting a full melter, to demonstrate that under the rated operating conditions a crucible can be restarted while full following an incident, and to determine the impact on the material and on the process;
 - the impact on the process of liquid feed onto the molten glass surface.
- Extended long-term testing with the main objective of demonstrating that process operation is not subject to variation, that the operating conditions specified for nominal operation as well as during transient phases are applicable, and that the material properties remain constant over time.

In this program, the tests chosen for these CCIM glass were based on these rheological, electrical and thermal properties, on the risk of crystallization or phase separation, and on the presence of volatile compounds. These tests were carried out on a full-scale pilot described below.

Vitrification Pilot

The vitrification full-scale pilot is shown in Figure 1. The feed solution and additives are supplied to the calciner. The resulting calcine is then mixed with glass frit in the melter. The off-gas treatment unit recycles particle matter and purifies the gas streams, before stack release.

The melter crucible [2] (inner diameter of 650 mm) is continuously supplied with calcine and intermittently with glass frit. The glass in the crucible is heated directly by eddy currents generated by the inductor surrounding the shell. The currents dissipate power by Joule effect that heats the calcine and glass frit to form the glass melt. Melting is initiated by a magnetic suscepter placed at the center of the glass. The monitored parameters on the melter are the temperature, the stirrer speed, and the air flow rate via the three injectors. A video camera is provided to view the molten glass.

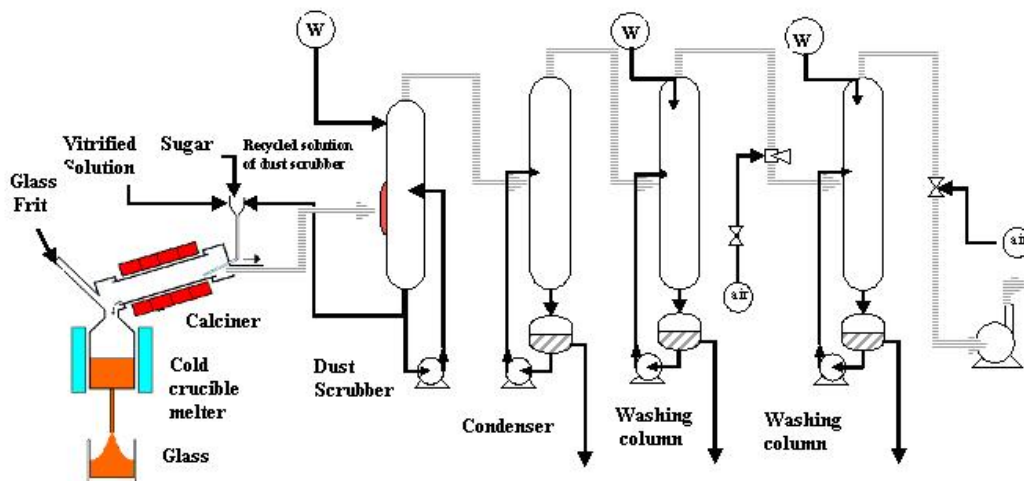


Figure 1. Vitrification full scale pilot.

Glass for effluent coming from rinsing and decommissioning operation

The glass formulation is closely related to the two-step vitrification process: calcining the solution followed by vitrification in a CCIM. The glass composition range is specified based on the possible range of effluent chemical compositions, on the calcining constraints necessary to avoid calciner fouling, and on the chemical composition of the frit.

Distance-Based Design methodology [3] was applied to quantify all the physical and chemical properties from the molten liquid state to the solid state over the full composition range. Twenty-five calculated glass compositions were specified, then synthesized at laboratory scale (20 basic glass and 5 validation glass compositions). Their physical and chemical properties were quantified: viscosity, electrical conductivity, density, and initial alteration rate r_0 . Models were constructed and validated to interpolate between the properties and chemical compositions. These models quantitatively describe (by interpolation) the properties of all the glasses within the composition range.

First step of the methodology: nominal tests

The objective of the nominal tests was to define the nominal parameters values to synthesize a homogenous reference glass with the same properties as the laboratory reference glass. The calcining parameters were defined by prior tests without vitrification and could not be modified during the vitrification process. Only the temperature in the molten glass, the stirrer rotation speed and the flow rate of the three injectors was subject to variation. The final choice of the nominal parameters depended on the results of the glass characterization, the volatility and the stability of process operation.

The nominal CCIM parameters during the synthesis of the selected reference glass were:

- 1230°C for the glass temperature in the crucible, obtained by regulating the generator power,
- 50 rpm for the stirrer rotation speed,
- 400 NL/h (Normal conditions : 1 atm and 0°C) for the flow rate from each of the three air injectors.

Under these operating conditions, foaming was observed in the laboratory reference glass, and the reference glass was modified to avoid foaming. The properties of the industrial reference glass was then compared with those given by the experimental model obtained at the laboratory scale.

Three glass samples were taken while the melt was poured: at the beginning, middle and end of each pour. All melt samples for all tests were submitted to chemical analysis. The analyses indicated in Table I are for the reference glass produced under nominal operating conditions. This table shows the analyzed glass chemical composition, the theoretical one's (named reference glass) and the expected glass composition calculated from mass measurement of the calcine and the glass frit.

Table I: Analysis Data for Reference Glass Fabricated under Nominal Operating Conditions


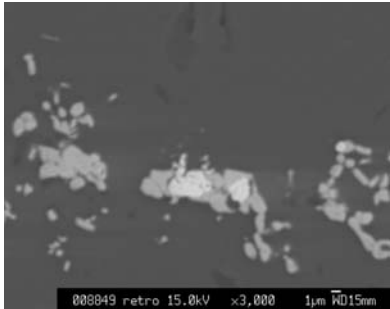
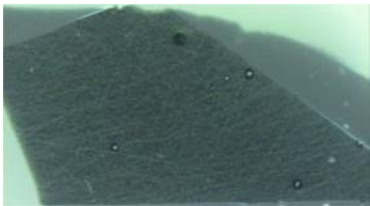
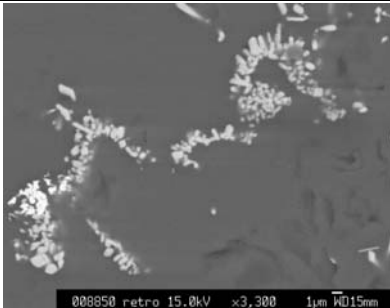
Oxide (%)	Reference glass (%)	Expected glass (%)	Analyzed glass (%)		
			Beginning	Middle	End
SiO ₂	48.66	48.68	47.52	47.52	47.45
B ₂ O ₃	13.99	13.99	13.73	13.85	13.53
Na ₂ O	12.58	12.57	11.92	11.94	11.93
Al ₂ O ₃	10.39	10.39	10.64	10.62	10.64
Fe ₂ O ₃	3.47	3.47	3.61	3.62	3.62
RuO ₂	0.12	0.12	0.16	0.16	0.16
SO ₃	0.19	0.19	0.13	0.12	0.13

The analyses of the reference glass produced under nominal operating conditions show that the glass remained homogeneous between the beginning, middle and the end of the pour. Very good agreement was observed between the theoretical and expected glass compositions and the chemical analysis results. The following remarks concerning the observed deviations are applicable to all the tests. Sulfur was lacking in the glass because of its high volatility; ruthenium was in excess because as a result of possible analytical error; silica was lacking because its actual concentration in the frit was lower than expected, although remaining within the specification limits. The deviations observed for boron and sodium are also probably due to the analytical uncertainties.

Several microstructural characterizations were carried out on glass samples. In each case the glass was homogeneous except dispersed micro-aggregates of ruthenium oxide particles and micro-grains of ruthenium.

The matrix composition analyzed by EDS (Electron Dispersive Energy) corresponded in each case to the expected composition. The SEM (Scanning Electron Microscopy) observations are summarized in Table II.

Table II: Glass Characterization

	Optical view (centimeter scale)	SEM images (high magnification of micro-domains containing insoluble noble metal particles)
<p>Middle of pour 2</p> <p>Homogeneous glass with dispersed Ru oxide (in gray) and Ru micro-aggregates (white)</p>	 <p>(optical microscopy)</p>	
<p>Middle of pour 8</p> <p>Homogeneous glass with dispersed strings of Ru and Ru oxide nanograins</p>	 <p>(optical microscopy)</p>	

Although the reference technological glass fabricated with the modified frit was not synthesized in the laboratory, the distance-based statistical method allows the physical and chemical properties of each composition in the validated range to be calculated using the interpolation models. We therefore decided to compare the measured physical properties of the industrial-scale reference glass sample with the results obtained from the models. We compared the viscosity, the electrical resistivity, the thermal conductivity and the density.

As shown on Figure 2, the measured viscosity was in very good agreement with the value calculated using the model developed by the distance-based design method and of course with the process constraints. For example, the viscosity was 50 poises at 1250°C (model 59 poises) and 78 poises at 1200°C (model 80 poises), consistent with pouring requirements. The measured electrical resistivity was consistent not only with process constraints but also with value predicted using the model, despite a slight difference. At 1250°C the measured value was 3.4 ohm-cm (equivalent to a conductivity of 29 S·m⁻¹), whereas the model predicts a value of 3 ohm-cm (i.e. 33 S·m⁻¹). The average measured thermal conductivity (Figure 5) was 5.5 W/mK, consistent with process requirements.

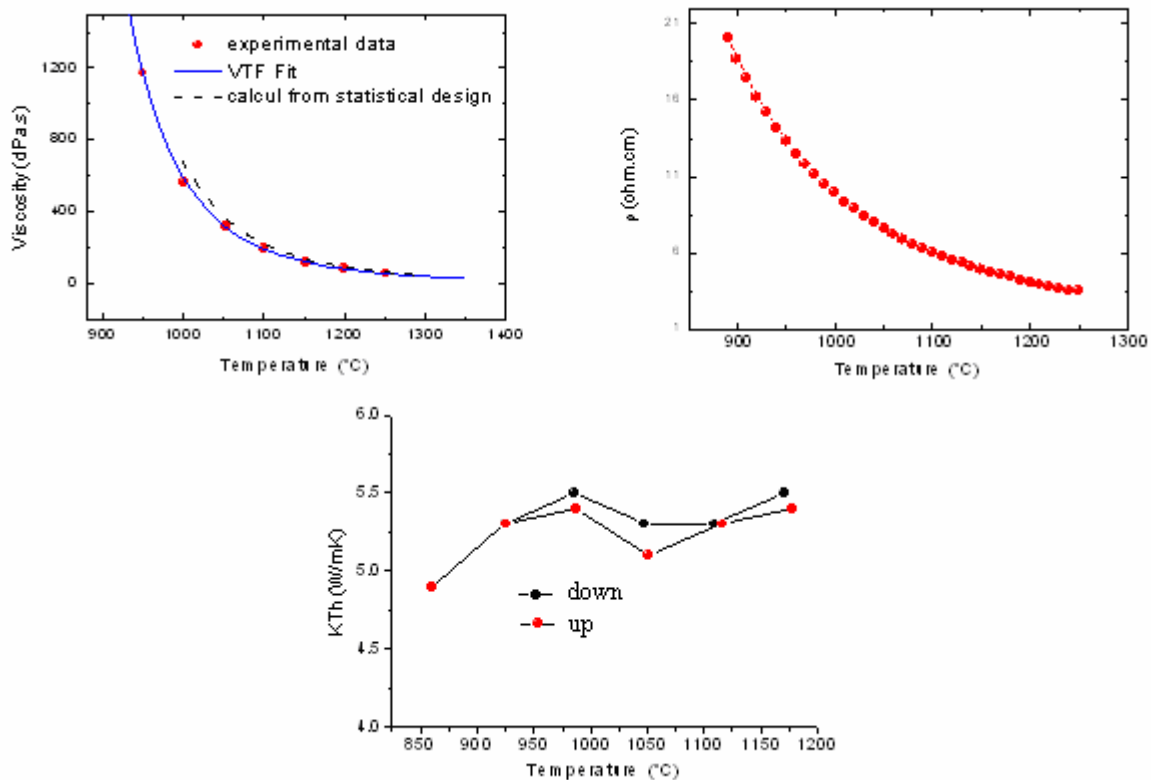


Figure 2: Viscosity, electrical resistivity and thermal conductivity of reference glass

The measured relative density was 2.549, corresponding to the density of 2.556 calculated by the model. All the measured properties of the reference glass are thus in good agreement with results given by the model

The equipment performance in a melting facility is estimated by calculating the decontamination factor under active conditions equivalent to the decontamination factor under inactive conditions. For each compound, the decontamination factor was calculated as the ratio of the output content to the input content. Ten elements were analyzed in the off-gas treatment system. Only the decontamination factors for sodium, ruthenium, boron and sulfur are indicated, as the volatility for the other elements is very low. For the reference glass fabricated the results are also indicated under nominal operating conditions (Table III). For example, the DFCv equal to 200 for the boron means that there is 200 times less boron in exit of the calcination and vitrification equipment that it takes in.

Table III: Process Decontamination Factors for Reference Glass under Nominal Operating Conditions

Element:	B	Na	Ru	S
DFcv (calcination vitrification)	200	150	7.5	3.5
DFpsep (particle separator)	7.5	2	3	1.25
DFcond (Condenser)	50	50	250	50

The order of magnitude of these values is reproducible from one test to another. The elements that pass through the dust scrubber in significant quantities are boron, sodium, ruthenium and especially sulfur—i.e. volatile elements during calcining (this is true in particular for ruthenium, for which sugar is added to reduce the volatility) and melting.

The reference glass was synthesized without difficulty in the PEV prototype. It was homogeneous and its physical and chemical characteristics were in close agreement with those predicted by distance-based statistical design models.

Sensitivity tests

The first type of sensitivity tests concerned the process operating conditions: the impact of the melt stirring conditions and the glass synthesis temperature on the material quality and on volatility phenomena. The test objective was to determine an operating range for these parameters.

For decommissioning glass, the process temperature, the stirrer rotation speed and the air injector flow rate had been identified as key process parameters in the course of nominal tests. The goal was to quantify the impact of variations of these parameters on the process behavior and on the glass quality. The tests were conducted in the following order: variations of the stirring conditions were tested first, then variations of the process temperature.

The stirring conditions affect the thermal homogeneity of the melt in the CCIM and on heat transfer at the surface of the melt. Insufficient thermal homogeneity diminishes the glass production capacity, and would reduce the melting rate of the calcine with the glass frit.

At a constant melt temperature, the effect of reducing the stirring rate was determined by video observation of the melt surface as well as measurement of the energy released to the dome. The final glass homogeneity was then checked by microscope observations.

The temperature also directly impacts the reactivity of the glass frit with the calcine. The glass composition range was chosen so that all glass compositions can be fabricated in a CCIM between 1200 and 1250°C (glass temperature is a key factor to increase solubility of hard to vitrify specific element coming from rinsing and decommissioning operation). The objective of the temperature sensitivity test was to demonstrate the very minor impact, if any, of the process temperature on the glass homogeneity at the microscopic scale. The goal was first to demonstrate that the stirring conditions do not impact the material, and that they have a minor impact on the throughput, then to quantify the impact of the temperature under constant nominal stirring conditions.

For all the operating conditions tested, the reference glass was visually homogeneous. The chemical analysis results for the glass produced during the tests of sensitivity to operating conditions are shown in the following table. As before, the tables show the glass chemical analysis data and the theoretical glass composition (named reference glass).

Table IV: Glass Chemical Analyses for Sensitivity Tests

Oxide %	Reference glass %	1230°C 50 rpm, 3 × 500 NL/ h	1230°C 60 rpm, 3 × 500 NL/ h	1230°C 40 rpm, 3 × 300 NL/ h	1230°C 50 rpm, 3 × 400 NL/ h	1210°C 50 rpm, 3 × 400 NL/ h	1250°C 50 rpm, 3 × 400 NL/ h
		Analyzed glass %	Analyzed glass %	Analyzed glass %	Analyzed glass %	Analyzed glass %	Analyzed glass %
SiO ₂	48.66	47.83	47.62	47.58	47.52	47.76	47.78
B ₂ O ₃	13.99	13.53	14.19	14.10	13.85	13.65	13.81
Na ₂ O	12.58	12.10	12.06	12.14	11.94	11.94	11.90
Al ₂ O ₃	10.39	10.58	10.45	10.42	10.62	10.63	10.76
Fe ₂ O ₃	3.47	3.56	3.58	3.62	3.62	3.64	3.62
RuO ₂	0.12	0.14	0.14	0.15	0.16	0.16	0.16
SO ₃	0.19	0.13	0.12	0.13	0.12	0.13	0.12

Very good agreement was observed between the theoretical and analyzed glass compositions. No effect of the operating conditions on the glass composition was observed, confirming the volatility results shown above.

Several microstructural characterizations were carried out on glass samples from the two tests at the middle of the pours. In each case the glass was homogeneous with dispersed micro-aggregates of ruthenium oxide. The final volume of insoluble RuO₂-based particles was very low because the content was low (expected equal to 0.12 wt%). The matrix composition analyzed by EDS corresponded in each case to the theoretical composition.

The volatility results show that the operating conditions have little impact on the volatility. For example, the sulfur decontamination factors ranged from 3 to 4 for calcination vitrification, from 1.2 to 1.3 for the dust scrubber, and were equal to 50 for the condenser.

The tested stirring and temperature ranges demonstrate the effectiveness of the vitrification process. Moreover, the volatility results were unaffected. Chemical analysis of the glass samples fabricated during these tests corresponded to the expected compositions. SEM observations of the glass showed no effect of the operating conditions on the microscopic homogeneity. The microstructural characteristics were consistent with those of the glass fabricated under nominal operating conditions. The glass contains a very small amount (0.12 wt%) of small insoluble RuO₂ particles, which does not impact the basic properties of the material.

Considering the slight impact of the stirring conditions on the thermal distribution in the melt, the recommended and validated normal operating ranges are therefore:

- Stirring speed (rpm): [40–60],
- Sparging air flow rate (NL/h): [2 injectors at 300– 3 injectors at 500]
- Glass fabrication temperature (°C): [1210–1270].

The chemical composition sensitivity test consisted in demonstrating the feasibility of fabricating the potentially most difficult glass in the range from the standpoint of glass homogeneity. For the studied decommissioning glass the most difficult compositions with respect to glass homogeneity are rich in P

and Mo (nucleation agents). In order to compare its properties, we chose a glass comparable to one of the samples from the statistical selection method studied at laboratory scale.

This glass was characterized in particular detail on samples taken during pouring as well as in the canister. As for the reference glass, the chemical composition of the sensitivity test glass corresponded to the expected and theoretical compositions (Table V).

Table V: Chemical Analysis of the Chemical Composition Sensitivity Test Glass

Oxide	Theoretical glass (%)	Expected glass (%)	Analyzed glass (%)	
			Pour sample	Canister sample
SiO ₂	45.00	44.99	44.91	44.93
B ₂ O ₃	13.50	13.50	13.70	13.65
Na ₂ O	14.35	14.35	14.26	14.20
Al ₂ O ₃	12.86	12.86	13.22	13.22
Fe ₂ O ₃	3.83	3.83	3.82	3.83
RuO ₂	0.23	0.23	0.34	0.34
SO ₃	0.40	0.40	0.29	0.30

Several microstructural characterizations were carried out on glass samples, at the beginning, middle and end of the pour. Homogeneous glass was observed in each case, with dispersed micro-aggregates of ruthenium oxide particles as in the reference glass

The differential thermal analysis (DTA) measurements of the sample taken at the end of pour 7 were compared with those obtained for the laboratory glass with the same composition (Figure 3). The close fit of the measurements confirms the quality of the technological-scale test of this glass composition at the limits of the range. The glass transition temperature was 494°C.

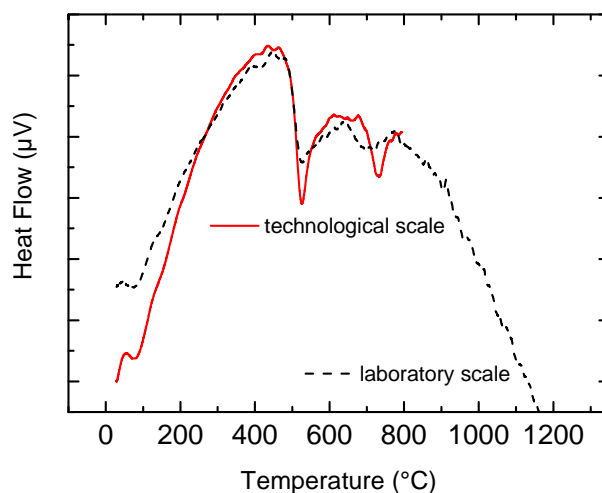


Figure 3: DTA results for chemical sensitivity technological-scale glass and the laboratory-scale glass from the statistical selection method studied at laboratory scale.

This test demonstrated that a penalizing glass at the limits of the range could be fabricated.

The three tests have demonstrated that the CCIM is properly controlled under all operating conditions: temperature deviation only occurs after each pour; glass sampling during each test, followed by optical and SEM examinations and chemical analysis, confirmed the homogeneity at microscopic scale; the volatility was unaffected.

For any glass composition in the validated glass composition range, the previous operating parameters (stirring speed....) determined previously can be applied.

Transient operating modes

During the transient operating phases, the operating parameters must be adjusted to guarantee the chemical composition of the material and avoid strong volatility. For glass to be produced, considering the presence of volatile elements, and various startup options, the following tests were chosen:

- startup tests with glass frit or with glass to determine the procedure for obtaining a homogeneous glass with acceptable composition;
- short and extended tests of calciner standby period (CSP). In operation, waiting periods occur during which the process is no longer supplied with decontamination solution and frit, i.e. the calciner standby period. For the melter, the CSP can be considered equivalent to a glass refining period. The impact of these phases must be determined on the glass quality, on the process, and on melter operation.

We have shown that the startup with frit glass or glass is possible for the CCIM. The analysis findings for the first melt show no enrichment or depletion of any of the glass constituent elements. We may therefore conclude that any possible higher volatility of some elements during the startup phase would have no consequences on the glass composition.

Short CSPs did not disturb the crucible and off-gas treatment management cycles, nor were any differences in behavior identified under CSP operating conditions. It did not affect the material, as shown by the results of chemical analysis and microstructural characterization. It will not be necessary to modify the glass fabrication conditions during short calciner standby periods. The glass synthesis temperature, the melt stirring speed, and the injector airflow rates remained stable at their nominal values.

Extended CSPs did not disturb the crucible and off-gas treatment management cycles, nor were any differences in behavior identified under CSP operating conditions. Analysis of the glass revealed a sulfur deficit, although this element is a minor glass constituent. The deficit was independent of the operating parameters during extended calciner standby periods. From these observations we can conclude that it is unnecessary to modify the crucible control parameters during extended calciner standby periods.

The nominal tests, the sensitivity tests and the transient operating mode tests covered nominal process operation. The next requirement was to investigate degraded modes and to carry out an endurance test to finish the process qualification study.

Degraded operating modes

Operating incidents can cause the process to deviate from nominal operating conditions. Degraded operating modes must be examined to minimize their impact on safety, on the process equipment, and on the material. Means of detection are determined and management procedures are defined. In our case we chose to study three degraded modes:

- the impact on the material of an interruption in the glass frit feed: the purpose of this test was to determine the consequences of failure of the glass frit feed stream on the material and its impact on process control; another objective was to determine a suitable means of detecting a frit feed failure;
- the consequences of restarting a full melter, to demonstrate that under the rated operating conditions a crucible can be restarted while full following an incident, and to determine the impact on the material and on the process;

- the impact on the process of liquid feed onto the molten glass surface.

The glass frit feed failure had little impact on the off-gas treatment and on the volatility of the various elements, all of which remained below the values observed during testing under nominal conditions. The chemical composition was in good agreement with the theoretical and expected compositions. The microstructural characterization showed that the glass was homogeneous and contained ruthenium particles like the reference glass produced under nominal conditions.

A glass frit feed failure was detected by the simultaneous occurrence of three phenomena:

- The slope of the molten glass contour line established from the injector air pressure significantly diminished.
- The slope of the curves for the power supplied to the instrumentation tubes was much less pronounced than usual.
- Decreases of large amplitude were observed for the current flow, voltage and power.

Failure of the glass frit feed stream had no impact on the material quality or on the process.

The feasibility of startup with a melter full of glass was demonstrated. The microstructural characterization showed that the glass was homogeneous and contained ruthenium particles like the reference glass produced under nominal conditions.

Injecting demineralized water on the surface of the molten glass to simulate liquid entering the crucible had no impact on the material. The brief duration of the injections did not modify the CCIM control settings. Only the off-gas treatment system was affected. An injection of 0.075 L of water was sufficient to create a pressure transient that shut down the feed streams and caused the system to revert to the wet calciner standby configuration. In the event of continuous injections on the crucible surface, a detailed analysis of the operating parameters of the particle separator, the calciner coupling sleeve, and the condenser should result in diagnosing an extra liquid feed in the core of the process.

Endurance test

The main objective of the endurance test was to demonstrate that the process is not subject to variation, that the operating conditions specified for nominal operation as well as during transient phases are applicable, and that the material properties remain constant over time.

For the glass in development we based this study on the three consecutive sensitivity tests, each lasting one week, during which it was possible to characterize the glass heel at the bottom of the crucible, which is the first sign of a potential deviation in material quality during extended operation.

No major operating difficulties were encountered during the three tests. Two glass compositions were fabricated at various temperatures and stirring conditions without any problem occurring in the crucible, nor did any difficulties appear during pouring of the 30 melts fabricated during this period.

The macroscopic properties of the heel were similar to those obtained throughout the qualification program. The chemical analysis results for the glass heel are indicated in Table VI.

Table II: Chemical Analysis of Glass Heel at the Bottom of the Crucible after Three Sensitivity Tests

Oxide %	Theoretical glass %	Expected glass %	Analyzed glass %		
			Melt 11	Melter dump	Crucible heel
SiO ₂	48.66	48.64	47.99	47.92	47.81
B ₂ O ₃	13.99	13.99	13.91	14.16	14.01
Na ₂ O	12.58	12.59	11.90	11.94	11.90
Al ₂ O ₃	10.39	10.41	10.67	10.73	10.58
Fe ₂ O ₃	3.47	3.47	3.65	3.66	3.64
RuO ₂	0.12	0.12	0.16	0.16	0.17
SO ₃	0.19	0.19	0.11	0.10	0.12

No difference in chemical composition was observed between melt 11 (the last melt of the third sensitivity test), the melter dump, and the glass heel at the bottom of crucible. Chemical analysis thus confirms that no enrichment of any compound occurred in the glass heel; this is very favorable for long-term operation with this glass.

CONCLUSION

This methodology gradually allowed us to qualify the process. By beginning with the tests under nominal operating conditions—that is the nominal tests, the sensitivity tests and the study of the transient modes—it was possible to quickly detect possible issues, and thus to modify the operating conditions or the glass composition range if necessary. In our example, the glass frit composition was changed to avoid foaming in the CCIM.

The study of degraded operating conditions identified means of detecting incidents leading to these conditions and for implementing procedures to protect safety, the process equipment and the material. In this application the degraded modes tested had no effect on the material, on the process equipment or on safety.

Finally, the endurance test validated the nominal operating conditions over an extended time period. In our example, we simply examined the glass heel at the bottom of the crucible because it was the main risk for these glass.

This process qualification methodology applied to the decontamination effluents from the UP2-400 facility allowed us to draft the package qualification file for new decommissioning glass canisters in 18 months. In addition to the technological data, this file also describes the study of the glass composition range and the study of its long-term behavior, which were carried out at the same time.

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