World Premiere Industrial Vitrification of High Level Liquid Waste Produced by Uranium/Molybdenum Fuel Reprocessing in La Hague's Cold Crucible Melter – 14035

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ASTRACT

250 cubic meters of high-level liquid waste from reprocessed U-Mo-Sn-Al spent fuel, used in Gas Cooled Reactors (GCR), have been stored in stainless steel tanks in the AREVA La Hague facility since the mid-1960s. The Cold Crucible Induction Melter (CCIM) was selected for the vitrification of this particularly hard-to-process waste stream because it could not be reasonably processed in the standard hot induction melters currently used in the La Hague vitrification facilities: the waste has a high molybdenum content which makes it very corrosive and also requires a special high-temperature glass formulation to obtain sufficiently high waste loading factors (12% in molybdenum oxide).

In April 2010, a CCIM started hot operation for the first time ever in an existing very high active facility (R7) at La Hague. This was the culmination of more than two decades of R&D involving progressive process and technological development. The design and implementation phases for the industrial deployment were described in a previous paper at WM [1]. The CCIM has now been operated for more than three years, essentially processing highly active effluents from D&D operations.

In January 2013, the first industrial operation of legacy UMo waste vitrification in a CCIM was carried out in the R7 facility. During this campaign, much data was collected to confirm the process parameters that were defined during the qualification of this innovative process. The experience of the operating teams along with the support of engineering and R&D allowed for successfully management of this world premiere operation.

This paper presents the start-up methodology and history, and the lessons learned during the first UMo waste processing in the La Hague CCIM, emphasizing the benefits of close cooperation between R&D, engineering, and operation teams during this learning phase.

INTRODUCTION

Vitrification of high-level liquid waste is the internationally recognized standard to minimize the environmental impact resulting from waste disposal and the volume of conditioned waste. In France, high-level liquid waste arising from nuclear fuel reprocessing has been successfully vitrified for more than 30 years with three major objectives: durable containment of the long-lived fission products, minimization of the final waste volume, and suitability for an industrial context.

The CEA and AREVA have acquired a unique experience in the field of high-level waste vitrification through continuous efforts to improve the technology (from hot to cold crucible melter) and the associated matrix formulations, with constant emphasis on quality and volume reduction, leading to the design and qualification of the cold crucible induction melter (CCIM) technology.

250 cubic meters of legacy solutions resulting from reprocessing U-Mo-Sn-Al spent fuel in the former UP2-400 plant during the mid-1960s are still stored at La Hague facility in stainless steel tanks. These solutions are less radioactive than the current fission product concentrates coming from ongoing reprocessing activities, but are very rich in molybdenum. The high molybdenum content makes the waste very corrosive and also requires a special high-temperature glass formulation to obtain sufficiently high waste loading factors (12% in molybdenum oxide).

As standard vitrification technologies are incompatible with the specific features of UMo waste, AREVA has committed to conditioning it using the CCIM technology. In April 2010, a CCIM has started hot operation for the first time ever in a harsh environment at the La Hague R7 vitrification facility processing high active material. The CCIM has now been in commercial operation for more than three years, essentially processing active effluents from D&D operations.

In January 2013, the first industrial processing of legacy UMo waste in a CCIM was carried out in the R7 facility. This was the culmination of several years of R&D led by the Joint Vitrification Laboratory (L.C.V), a common research laboratory between CEA and AREVA in charge of qualifying new processes and matrices for waste containment.

This paper describes the process operation during this “witness” campaign, the start-up methodology and the lessons learned.

CCIM VITRIFICATION PROCESS OPERATED IN THE R7 FACILITY

Industrial French Vitrification Design

In France, highly active liquid wastes are vitrified into a two-step vitrification process, shown schematically in Figure 1. In the two-step process, the feed solution coming from reprocessing operation is fed to a rotary calciner which performs the evaporating, drying and calcining functions. Aluminum nitrate is added prior to calcination to avoid sticking issue in the calciner (melting of $NaNO_3$). Sugar is also added to the feed prior to calcination to reduce some of the nitrates and to limit ruthenium volatility. At the outlet of the calciner, the calcine falls directly into the melter along with the glass frit which is fed separately. The melter is fed continuously but is poured batchwise. The off-gas treatment unit recycles particulate material and purifies the gas streams, before stack release.

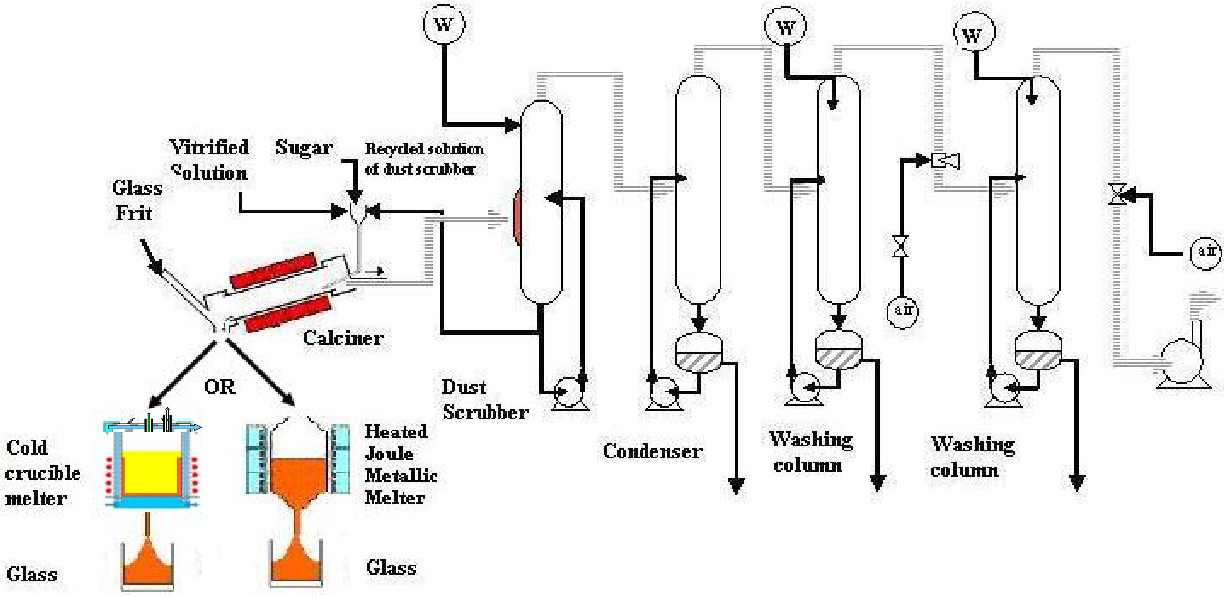


Fig. 1. Two-step vitrification process.

The calciner includes:

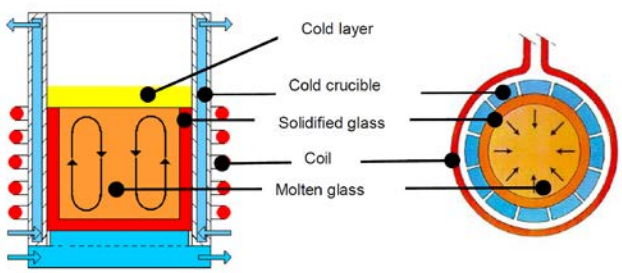
- a resistance furnace with four independent heating zones separated by interzone segments,
- a rotating tube,
- an upper end-fitting ensuring leak-tightness at the rotating upper end, with connections for exhausting the off-gas and for supplying the liquid feeds (vitrification feed solution, sugar and recycled solution),
- a lower end-fitting ensuring leak-tightness at the rotating lower end and guiding the calcine into the melter.

The calciner is controlled by assigning heating temperature setpoints to the electrical resistors. The calcining performance is observed by monitoring the heating power variations in each zone.

The off-gas treatment system is composed of a hot wet scrubber with weir plates, a water and nitric acid vapor condenser, an absorption column, a washing column, a ruthenium filter, and three HEPA filters. The most active gas washing solutions are recycled from the wet scrubber to the calciner. The other solutions are concentrated in an evaporator before recycling into the vitrification plant. Off-gas treatment must be capable of ensuring a satisfactory decontamination factor in the gas exhausted from the calcining and glass production operations. Liquid samples can be taken from each of the four process devices to estimate the quantity of volatilized or entrained species. Each device is also supplied for level, temperature, and pressure measurements.

Direct Induction Vitrification Principles and Advantages

The direct induction process is characterized by currents directly induced inside the molten glass by a coil (Figure 2). These electromagnetic currents heat the glass inside the melter by the Joule effect. The segmented structure of the crucible enables penetration of electromagnetic field into its volume. Absorption of electromagnetic radiation allows the glass to be heated directly without heating the crucible. In addition, the walls of the crucible are cooled and sectorized to make them transparent to the electromagnetic field.



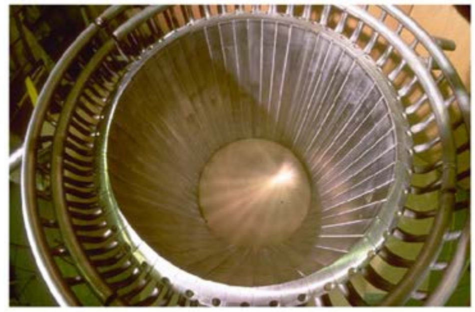


Fig. 2. Direct induction melting principle - View of the inside of the CCIM.

The CCIM technology presents a number of major advantages.

First, cooling of the crucible forms a solidified layer of glass which coats the surface of the crucible in contact with the glass. This skull layer protects the crucible from the corrosive melt. The cooling of the crucible protect from corrosive vapours. Second, the direct induction heating method allows the temperature to be increased (beyond 1300°C for some new matrix formulations still being tested) making it possible to obtain new waste containment matrices which would have been impossible to produce with the hot metallic melter.

This technology can be used to vitrify many varied types of chemical waste. By allowing higher waste loading it also minimizes the volume of packaged waste. Furthermore, the presence of the cold layer minimizes the impact of the composition of the waste on the lifetime of the crucible.

Finally, when integrated into the two-step vitrification process (calcination and vitrification), as is the case in the R7 facility at La Hague, the CCIM technology allows the industrial vitrification throughput to be significantly increased. The higher temperature allows a faster calcine digestion by the glass, and consequently allows continuous feeding.

CCIM Design Principles

The CCIM is composed of the following elements (Figure 3):

- The metallic crucible shell, which is a segmented structure, transparent to the electromagnetic field. The cooled sectors are separated by electrical insulators.
- The crucible bottom (slab), which includes the pouring valve. A cooled duct links the crucible to the container.
- The crucible is topped by a dome which supports a mechanical stirrer.
- Glass level and temperature are continuously measured by specific sensors.
- Bubblers are positioned on the crucible slab.

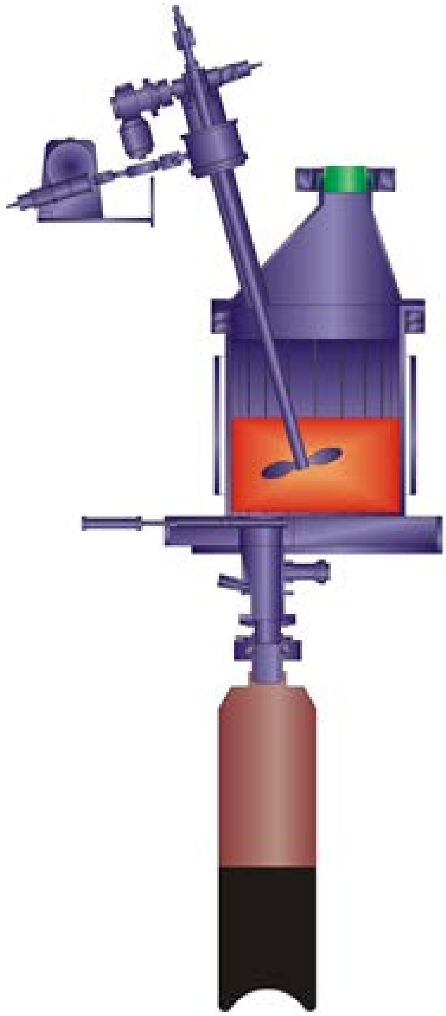


Fig. 3. Schematic drawing of a CCIM.

The crucible power supply comprises:

- A high-frequency generator with an output of around 400-600 kW.
- A high-frequency power line.
- A copper coil surrounding the crucible.

CCIM Deployment at La Hague

In April 2010, a CCIM has started hot operation for the first time ever in an existing very high active facility (R7) at La Hague. This was the culmination of more than two decades of R&D involving progressive process and technological development. The design and implementation phases for the industrial deployment were described in a previous paper at WM [1]. The main stages are detailed below.

1981	The first CCIM prototype was put into service (350 mm in diameter).
1983	The feasibility of vitrification by electromagnetism was demonstrated. The reliability and endurance of the process were demonstrated by 800 hours of remelting inactive glass.
1985	Industrialization phase 1. A larger CCIM was built (550 mm in diameter).
1987	The continuous two-step vitrification process of R7T7 glass was demonstrated with a reduced capacity mock-up in 175 hours of inactive melting.
1992	AREVA NC decided to study the implementation of the cold crucible.
1997	A 650 mm diameter CCIM was built and tested (calcination-vitrification operation) with an industrial capacity for almost 3,000 hours (inactive tests).
2000	Industrialization phase 2. A specific CCIM prototype was designed and built for the vitrification of highly corrosive UMo fission product solutions resulting from the recycling of legacy GCR fuels.
2004	AREVA NC decided to implement a CCIM in R7.
2005	Engineering (E&P) started the summary design phase of the R7 CCIM.
2006	Industrialization phase 3. A “nuclearized” CCIM prototype was built, adapted to the La Hague vitrification process and environment. This CCIM was designed to vitrify a large variety of waste. It was used in Marcoule to qualify the process and glass quality. More than 6,000 hours of testing have been conducted on this platform. Engineering (E&P) started the Detailed design phase of the R7 CCIM.
2007	Construction in AREVA’s Beaumont testing and development laboratory (HRB) of a full-scale test platform identical in every way to the radioactive environment of R7 facility. This platform was used to carry out tests outside the nuclear zone and train personnel in 2008 and 2009.
2008	Industrialization phase 4. A nuclearized industrialized CCIM prototype was built to be implemented in the R7 facility. This one was used to qualify the equipment in the HRB. Start of the phase covering manufacturing and installation of the industrial CCIM in R7 facility.
2009	Start of the phase covering commissioning and inactive tests of the CCIM implemented in the R7 vitrification facility (5 inactive canisters).
2010	Start of the first active operation of the CCIM implemented in the R7 vitrification facility

UMO GLASS CONTAINMENT FORMULATION AND TECHNOLOGICAL PROCESS QUALIFICATION

Containment Matrix and Long-Term Behavior

The general qualification method for the UMo containment glass formulation was described in a previous paper at WM [2].

The main features of UMo legacy solutions are indicated in Table I.

Table I. Main characteristics of UMo solution

Composition (g/L)	MoO_3	137
	P_2O_5	42
	Na_2O	11
	Other	26
Volume		250 m^3
Activity		< 222×10^{10} Bq/L

Considering the elements present in the UMo high-level waste feed solutions, the molybdenum and phosphorus loading capacity of the glass has been critical for the waste loading capability of the containment matrix.

The reference UMo glass from the containment glass formulation qualification, SUMo2-10d, is a vitreous material fabricated at 1250°C. It is an opaque glass-ceramic. In the molten state the melt is homogeneous, but phase separation and crystallization phenomena occur after cooling in the canister. The glass-ceramic is characterized by secondary phases dispersed in an encapsulating borosilicate glass matrix. The reference UMo glass composition is indicated in Table 2.

Table II. UMo reference glass composition (wt%)

SiO2	38.7
Na2O	9.4
B2O3	13.9
Al2O3	7.1
P2O5	3.1
MoO3	10.0
ZnO	6.0
ZrO2	3.3
CaO	6.1
Other	2.4

The molten glass properties are compatible with fabrication in a cold crucible melter: viscosity of around 40 dPa·s at 1250°C and electrical resistivity of about 7 Ω·cm at 1250°C. The maximum range of molybdenum and phosphorus content in the final glass determine the melting temperature range, which must be higher than the phase separation temperature in order to maintain a homogeneous melt in the crucible. The phase separation temperature depends on the molybdenum and phosphorous concentrations in the glass.

The physical and microstructural properties of the UMo glass in the solid and liquid states were determined over the full specified range of compositions and process operating parameters.

The long-term behavior of the matrix was also investigated, and particularly the matrix behavior under irradiation (UMo glass doped with americium and curium) and its leaching resistance.

The data from the leaching tests, carried out on glass samples synthesized in a cold crucible melter under conditions representative of the industrial process, confirm that the chemical durability of the glass-ceramic matrix is comparable to homogeneous borosilicate glasses for the alteration rate regimes investigated (initial rate and rate drop with time).

The result of the irradiation behavior study (doped glass) confirms its stability under irradiation.

Inactive Technological Process Qualification

Qualification of the CCIM process, for UMo glass production, has consisted in different types of full-scale pilot tests with inactive surrogate solutions. These tests, carried out by the LCV's R&D team at Marcoule in a full-scale pilot of the industrial process including the CCIM, are described below.

- The nominal tests have defined the nominal parameter values which guarantee that the industrial-scale glass has the same characteristics as the laboratory reference glass.
- The sensitivity tests have validated an operating range for operating parameters over the entire composition domain, to maintain the nominal throughput.
- The transient mode tests have defined the operating parameters adjustments necessary to guarantee the chemical composition and microstructure of the final glass and to avoid strong volatility during transient phases.
- The degraded mode tests have defined the operating parameters to preserve the process equipment and the material properties. Means of detection have been determined and management procedures have been defined.
- Finally, a 21-day endurance test has demonstrated that the process is reliable, and that the material properties of the product remain constant over time.

The calcining parameters (heating power of each zone and rotation speed) have also been defined by specific tests without vitrification, and optimized during the qualification process for different throughputs. The feed solution composition adjustment has been also defined during the inactive qualification. UMo solutions have a strong tendency to stick in the calciner due to the high molybdenum content. Compliance with calcining and adjustment parameters ensure a proper calcine is obtained and prevent sticking issues.

Qualification of the cold crucible melter for vitrification of UMo solutions also required changes in the process: specific devices, at the outlet of the calciner, were defined and qualified to limit the sticking of molybdenum from calcining exhaust stream in off-gas treatment equipment.

The cold crucible melter control modes were defined by the CEA process licensor. They are specified in the process data book, which includes three levels of information, in accordance with the scope of CEA responsibilities:

- The requirements of the CEA process licensor.
- The operating recommendations based on CEA experience and on the limits of the test program.
- The lessons learned from operating experience.

Another full-scale pilot of the CCIM is installed in AREVA's Beaumont testing and development laboratory. This pilot has no calciner and is specifically devoted to technological development for the CCIM nuclearization and defining some additional operating parameters and procedures for application to the industrial facility. This pilot is operated by AREVA E&P (former SGN) teams.

DESCRIPTION OF THE FIRST UMO WASTE VITRIFICATION CAMPAIGN

Context and Procedure

UMo solutions are very hard to process because of the high molybdenum content. The main characteristics of the waste behavior in the process are detailed below.

- The waste is very corrosive.
- The solutions have a strong tendency to stick in the calciner.
- Vitrification and calcining exhaust stream may cause strong clogging issues in off-gas treatment equipment.

On January 3, 2013, a "witness" campaign of UMo waste vitrification was carried out in a cold crucible melter in the R7 facility. The vitrification campaign lasted 5 days with the following objectives:

- Validate the calcining and adjustment parameters defined during inactive tests.
- Validate the effectiveness of the devices implemented at the calciner outlet with respect to molybdenum sticking behavior.
- Check that the process parameter values are consistent with those obtained during inactive tests.
- Check the stability of the process parameters in nominal operation.
- Check that the operation of the off-gas treatment system is consistent with the results obtained during inactive qualification.
- Check that the energy balance is consistent with the results obtained in the inactive pilot facility under similar operating conditions.
- Check for satisfactory equipment operation and endurance.

This first "witness" campaign produced four CSD-U canisters weighing 380 kg each, and ended with a programed melter draining pour.

The volume of effluent vitrified was about 2.5 m^3. The glass production rate was 25 kg/h for batch 1 and 30 kg/h for batches 2 to 8.

During this "witness" campaign, data was collected to confirm the process parameters defined during the qualification of this innovative process.

Organization and Methodology

A specific organization was set up for the first UMo solution vitrification campaign, with the following participants:

- The industrial operator of the vitrification units: AREVA NC.
- The Joint Vitrification Laboratory (LCV) as CEA process licensor.
- AREVA E&P (Engineering & Projects).

AREVA E&P was in charge of manufacturing the specific equipment implemented at the outlet of the calciner to limit molybdenum sticking. AREVA E&P was also in charge of manufacturing some CCIM equipment optimizations.

The feedback obtained with the two inactive prototypes (at Marcoule and Beaumont) was used to verify that the operation of the equipment, during the "witness" run, was consistent with the inactive tests.

During the campaign, the operating parameters were monitored by support teams from the LCV (R&D) and AREVA E&P (engineering). The operation of the main equipment on the vitrification line was analyzed in real-time and advices and recommendations were provided during the daily debriefing meetings. When necessary, control adjustments were implemented to improve process performance.

Process parameters were monitored and analyzed in the following areas:

- Energy balance; Thermal parameters
- Electrotechnical parameters
- Technological operation of the melter (stirring, bubbling, glass pouring, etc.)
- Calciner operation
- Off-gas treatment operation
- Material feeds; Material balance.

Dedicated software is implemented in R7 to allow real-time observation of all the vitrification process parameters evolution. This system, augmented by the experience of all the participants, provided a detailed analysis of the operation of the facility and compiled precise diagnostics of process performance.

Start-up Phase

For each new matrix processed in the CCIM, the start-up procedure has to be adapted to the electrical, chemical, and thermal characteristics of the glass frit. The start-up of the "witness" campaign for the actual UMo effluents has allowed confirming the adequacy of procedure that had been defined jointly by the R&D, engineering and operator teams on the inactive prototypes with simulated effluents.

The data acquired during the startup phase of the active campaign were consistent with the results of the inactive tests and validated the control mode.

CCIM Operation

Special attention was given to cross-checking parameters during the campaign. The power injected in the glass and the thermal power dissipated in the structures were specifically monitored. The power injected in the glass is an indication of the power input necessary for glass synthesis under given conditions. The power injected in the glass depends on the load characteristics (mass, temperature, stirring, and composition) and the feed rate. The thermal power dissipated on each structural component is used to estimate the thermal distribution in the melter.

Analyzing the injected power showed good stability of the energy balance (figure 4), reflecting the absence of glass melt temperature drift during the campaign.

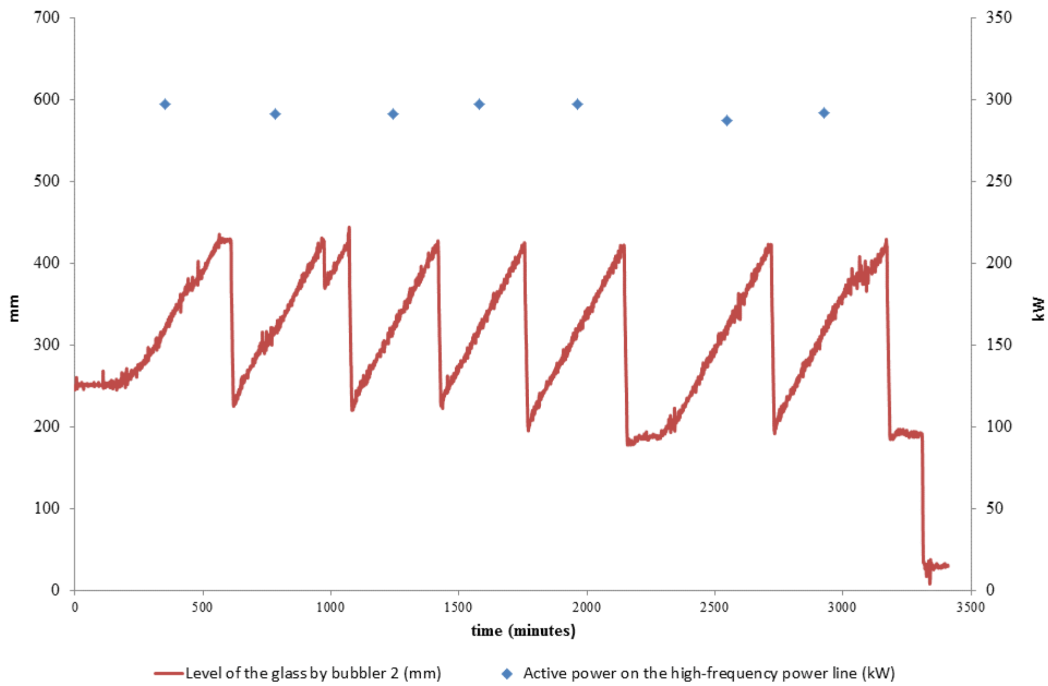


Fig. 4. Power injected in the glass for each batch

The evolution of the power dissipation in the melter equipment versus the glass melt level was consistent with the behavior generally observed in the LCV prototype. The variation of the thermal of the molten glass (and particularly in the cold layer) and of losses by Joule effect into the structures were as expected, and corroborate the data from the inactive tests.

The operation of the stirrer was satisfactory. The various descent cycles were suitable and did not create any significant heterogeneity in the molten glass. Operating feedback from the prototypes permitted satisfactory management of transient modes associated with the stirrer.

The bubblers and glass level specific sensor allowed monitoring of the mass of glass in the melter. The bubblers process parameter values were consistent with the material balance, and confirmed the absence of variations in the feed and in the composition of the poured glass. The glass level specific sensor values confirmed the feed stability. The injectors and glass level specific sensor operated as expected.

The initiation of pouring and the regulation of the pouring rate were satisfactory. The pouring parameters were adjusted with the assistance of the LCV. The pouring parameters tweaks allowed optimizing the flow rate after pouring was initiated with respect to temperature regulation of the molten glass.

The characteristics of UMo glass — and especially its high electrical resistivity — require inductor operation at high voltage and current levels. The electrotechnical parameters obtained during the campaign were stable and comparable to those obtained during inactive qualification tests. The electrotechnical operation of the process was validated.

The melter draining pour was performed satisfactorily. The process control parameters ensured that the melter was emptied under satisfactory conditions. The residual amount of glass remaining in the melter is low, it corresponds to the glass skull present during its operation and to a small fraction of the melt that remained in the crucible after it was emptied. The glass remaining was easily detachable, and did not adhere to the melter structures.

Inspection of the melter after removal of the glass remaining showed that its structure was clean and corrosion-free.

The melter operation was satisfactory and confirmed the results obtained in the R&D and technological development facilities.

Calciner Operation

The UMo solution composition was adjusted in compliance with the recommendations of the CEA process licensor.

The calciner parameters — feed rates, heating power values, and rotation speed — were applied in compliance with the recommendations of the LCV during the calciner start-up phase and in nominal operation.

The heating power values in the four zones of the calciner were stable throughout the campaign. This satisfactory result shows that there was no drift in the process liquid feed streams — and in particular no clogging.

The interior of the calciner was inspected after the campaign and the tube was found to be clean, confirming that no clogging occurred in operation (figure 5).

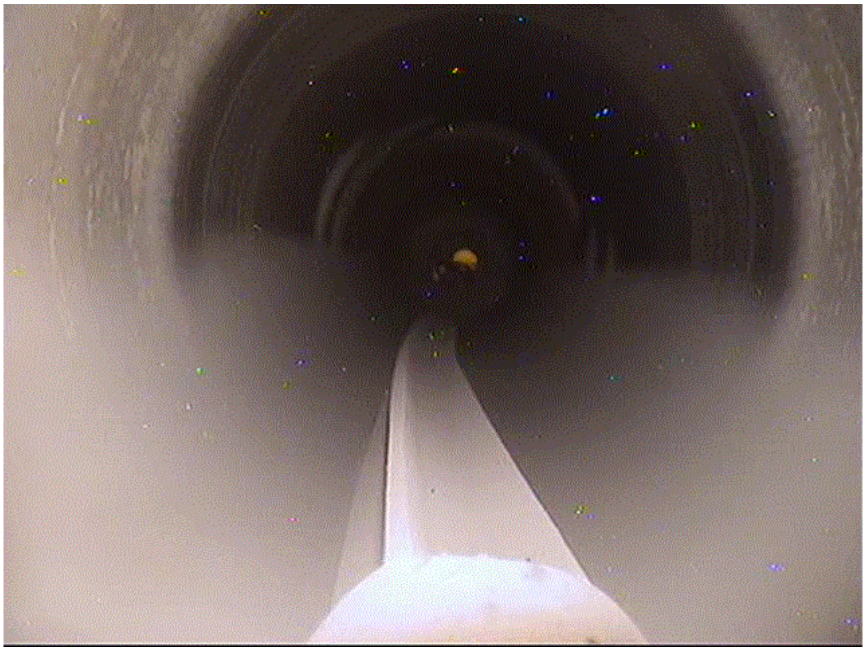


Fig. 5. Interior of the calciner after the campaign

Compliance with the parameters of UMo solution adjustment and calcining produced a satisfactory calcine and prevented clogging of the tube. Analysis of the calciner process parameters revealed satisfactory calciner operation and validated the qualification obtained under inactive conditions.

Off-Gas Treatment Operation

The process parameters of the off-gas duct from the calciner to the scrubber were specifically monitored because this item is critical with respect to clogging by molybdenum compounds in particular. The pressure drop variation in this link was monitored as a good indicator of the degree of clogging. During operation the increase in the pressure drop over time was consistent with what is generally observed in the inactive prototype in a similar configuration. The process control recommendations defined by the LCV and applied by the operators ensured satisfactory operation of this functional unit.

The installation of specific devices at the outlet of the calciner therefore limited the sticking of molybdenum from the calcining exhaust stream in the off-gas treatment system. These devices were optimized by teams from the LCV. In tests performed with the optimized devices in the inactive pilot facility, clogging was significantly reduced in the line connecting the calciner to the scrubber. The implementation of these design optimizations in R7 could significantly reduce the downtime arising from clogging of the calciner-scrubber line.

The operation of the off-gas treatment was satisfactory and remained stable throughout the “witness” campaign. Specific process control parameters for the dust scrubber were applied (with respect to molybdenum clogging behavior) in accordance with the one defined during the process qualification (inactive tests). The liquid density at the bottom of the scrubber was consistent with what is generally observed in the inactive prototype at the same feed rate.

Some optimizations, tested on the inactive pilot by R&D teams, enable drastically reduce clogging occurrences of scrubber pressure and level measurements. The implementation of those optimizations in R7 could significantly reduce the downtime arising from clogging of the scrubber equipment.

The off-gas treatment operation was satisfactory and corresponded to the expected performance.

CONCLUSION

The UMo effluent vitrification campaign lasted from January 3 to January 8, 2013. The first “witness” campaign produced four CSD-U canisters and ended with a melter draining pour. This world premiere was the outcome of more than 20 years of R&D and close collaboration between R&D and engineering teams and the industrial operator.

The volume of UMo effluent vitrified was about 2.5 m^3. The glass production rate was 25 kg/h for batch 1 and 30 kg/h for batches 2 to 8.

Analysis of the process parameters showed satisfactory overall operation of the CCIM. Analysis of cross-checking parameters (energy balance) revealed no process drift. The electrotechnical parameters obtained during the campaign were stable and comparable to those obtained during inactive qualification tests. The electrotechnical operation of the process was validated.

The bubblers, glass level specific sensor and stirrer operated as expected.

The glass pouring parameters were adjusted during the campaign, in particular to optimize the flow rate after pouring was initiated.

The glass remaining in the melter after draining pour was easily detachable, and did not adhere to the melter structures. Inspection of the melter after removal of the glass remaining showed that its structure was clean and corrosion-free.

Analysis of the calciner process parameters revealed satisfactory operation and validated the qualification obtained under inactive conditions. Compliance with the adjustment and calcining parameters produced a satisfactory calcine and prevented clogging of the tube.

During the production phase the rate of clogging of the calciner-scrubber line was consistent with the operation of the inactive prototype. The off-gas treatment operation was satisfactory and corresponded to the expected performance.

The organization set up between the industrial operator, the process licensor, and the engineering teams ensured a detailed analysis of process operation. The process control adjustment applied allows improving the performance of the process.

The feasibility of vitrifying UMo fission product solutions in a cold crucible melter at industrial scale has been demonstrated. The operation of the vitrification line was satisfactory and confirmed the results obtained in the R&D and technological development facilities.

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