

Widening the envelope of UK HLW vitrification – Experimental studies with high waste loadings and new product formulations on a full scale non-active vitrification plant – 8348

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ABSTRACT

The Vitrification Test Rig is a full scale waste vitrification plant that processes non-radioactive liquid HLW simulants based on the active waste streams produced by the reprocessing plants in the UK. Previous work on the rig has primarily concerned increasing the operational envelopes for the active waste vitrification plants at Sellafield to accommodate higher throughputs of Blended waste streams, higher waste oxide incorporation rates in the vitrified products, and the incorporation of legacy waste streams from early reactor commissioning and reprocessing operations at Sellafield. Recent operations have focussed on four main areas; dilute liquid feeds, very high Magnox waste stream incorporation levels, alternative base glass formulations and providing an operational envelope for 28%^{w/w} Magnox waste vitrification. This paper details the work performed and the major findings of that work.

INTRODUCTION

Reprocessing of spent Magnox nuclear fuel has been practised at Sellafield in the UK for over 4 decades, and Oxide fuel has been reprocessed in the Thermal Oxide Reprocessing Plant (THORP) for 13 years. The resulting high level nuclear waste (HLW) streams from these processes are stored as a nitric acid solution prior to being vitrified in an alkali borosilicate glass matrix on one of the three vitrification lines in the Waste Vitrification Plant (WVP) [e.g.1]. The UK government has set a target to reduce stored liquid HLW to buffer stocks by 2015, and thus consistent production of vitrified products is essential to meet this target. The Vitrification Test Rig (VTR), a full scale replica of the WVP vitrification process was built in 2004 to perform research and development work for WVP with non-radioactive HLW simulants to help WVP meet this target [2]. In this paper some of the major achievements of the VTR to date are discussed along with future areas of research that will be undertaken.

VTR OVERVIEW

A schematic of the various processes carried out on the VTR is shown in figure 1 below. Simulated liquid HLW (metal nitrates dissolved in nitric acid) is mixed with sucrose solution (reducing agent) and fed into the calciner, a rotating tube furnace angled at shallow downward slope. The temperature of the calciner is adjusted so that the liquid phase is evaporated in the upper end of the tube furnace and the resulting solid metal nitrates are largely denitrated and

converted to metal oxides in the lower end¹ (typical oxide:nitrate ratio of 3:1). This “calcine” then exits the tube at the lower end and drops by gravity into an elliptical induction heated crucible. An alkali borosilicate base glass frit is simultaneously fed into the crucible, and the two ingredients are agitated via a compressed air sparge to ensure intimate mixing. Once a feed cycle² has been completed the glass is kept in the crucible until the average temperature of the melt, as measured by several internal thermocouples, reaches a predefined value (typically 1050°C) and is then poured into a storage canister. Once cool, the VTR canister can be opened allowing examination and analysis of the glass product.

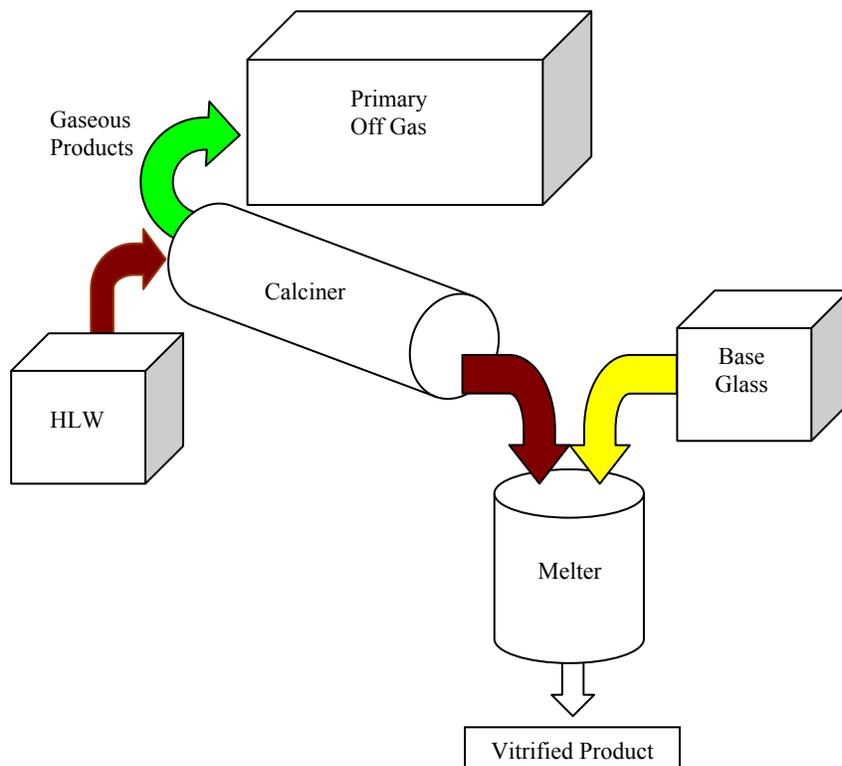


Figure 1 – Schematic of the VTR

As well as operation in vitrification mode, the VTR can also be operated in calcination only mode. In this configuration, the melter is removed from the plant and the calcine is collected after exiting the calciner, allowing it to be sampled and analysed.

The primary off gas system (POG) abates any volatile elements or fine calcine particles that are suspended in the off-gas flow and the gaseous products of the calcination process (primarily water and nitric acid vapours, and NO_x gases). The POG consists of a dust scrubber to remove solid particles, a condenser to remove water and nitric acid vapours and a NO_x absorber to remove any remaining NO_x gases. Solids in the off gas stream are dissolved in the dust scrubber and the solution is recycled back to the top end of the calciner. Condensed nitric acid and water from the condenser, along with the weak nitric acid solution generated by removal of NO_x in the

¹ Thus the actual temperature setpoints can vary considerably depending upon feed type and overall evaporative load

² A “feed cycle” is the addition of the appropriate quantity of base glass frit and calcine to the melter to produce 190kg of vitrified product

NO_x absorber are sent to effluent tanks. All of the vessels that make up the POG can be sampled for analysis. The POG, calciner and melter are kept at a constant depression in order to contain all of the process off gas within the plant and prevent the release of NO_x (and, on WVP, radionuclides) to the cell.

A more detailed description of the process and the history of the VTR, along with the operating philosophy, can be found in [3].

LIQUID HLW STREAMS

The composition of the liquid HLW that is processed by WVP is not consistent. Variations within the operational flow sheet envelope arise from a number of factors including the origin of the waste (i.e. whether it was generated by the Magnox or THORP reprocessing plants), the burn up of the parent fuel and the time spent in the evaporators. The first two of these issues are self explanatory and have obvious influences on the waste stream constitution. The evaporation time allocated to the waste streams directly influences the concentration of waste elements in the solution. Due to these variables, the exact compositions of the waste streams will not be discussed in detail in this paper, but a summary of the important differentiating factors and their major influence in the vitrification process is given below. In simplification of the HLWs for production of simulants for use on the VTR, all fission products for which there are no stable isotopes are omitted, along with a large number of elements that are present in very small concentrations.

Magnox Waste

Waste streams arising from Magnox fuel reprocessing have very high concentrations of Mg and Al that arise from the cladding of the fuel elements and also often contain high levels of Fe, Ni and Cr. Aluminium increases the viscosity of the borosilicate waste form and this can lead to higher temperatures being required to process the waste. The levels of Fe, Ni and Cr can result in increased melter heel³ enrichment, which can eventually result in blockages of the melter pour nozzle. Furthermore, the combination of Fe, Ni, Cr, Mg and Al can result in the formation of refractory spinel phases that dissolve very slowly in the melter, although the presence of these phases need not be deleterious to the process. A summary of the Magnox simulant composition used on the VTR is given in table 1 below.

Oxide Waste

Waste from reprocessed oxide fuels tend to have high levels of Zr (from the zircalloy fuel cladding), high levels of Mo and Cs as fission products and high levels of Gd which is added as a neutron poison. Traditionally, oxide only waste streams are not vitrified as laboratory studies have shown that the glass product made from this material is less resistant to chemical attack when the glass contains the desirable level of HLW loading. Instead, oxide waste is blended with Magnox waste, typically in a 3:1 ratio (known as “Blended waste”), as this ratio has been shown

³ The melter heel is a layer of ~70kg of glass that is retained in the base of the crucible at the end of a pour to act as an insulating layer

to give a product with a chemical durability similar to that of a pure Magnox product⁴. At high waste loadings, the high Mo levels in Blended waste streams can lead to the formation of yellow phase [4] which is a highly corrosive substance in the molten state and partially water soluble in the solid state. Blended wastes tend to hold greater challenges for the POG than the melter as more fines (in comparison to Magnox wastes) get carried over to the POG, and durable zirconium molybdate phases precipitate from these fines and can coagulate to block tubes and vessels. Table 1 shows the composition of the Blended waste simulant used on VTR.

Table 1 - Comparison of elemental compositions of the simulated HLW waste streams used on VTR

	w/w % in Magnox feed	w/w % in Blended feed
Mg	17.2	4.9
Al	14.2	4.9
Cr	2.5	1.7
Fe	12.5	7.3
Ni	1.8	1.2
Zr	6.4	9.7
Mo	5.9	7.7
Cs	6.1	7.6
Gd	0.0	18.3
Others	33.4	36.7
Total	100.0	100.0

PLANT OPERATION

Early development work on waste vitrification in the UK was based around proving flow sheet operations for WVP. At this time, waste oxide (WO) concentrations⁵ in the WVP feedstock were expected to be fairly consistent at ~150g/l, and the overall waste incorporation rate in the product at 25%^{w/w}. The overall throughput⁶ of the plant was 19 - 21kg/h, and base glass was (and still is, under standard operations) fed in direct proportion to WO to keep the WO incorporation level in the melter constant.

Upon installation of VTR, two main objectives that were immediately apparent as having potential to increase WVP efficiency were increasing plant throughput and increasing WO incorporation in the product. Increasing the throughput requires a proportional increase in the HLW liquid feed to the calciner, thus increasing the challenge in terms of the total amount of liquid to be evaporated and the quantity of off gas to be processed. Increasing the WO incorporation can have implications for melter operation and the quality of the product, established using various criteria including chemical durability.

⁴ Lower Blend ratios are also sometimes used (e.g. 1:1) as any lower ratios will give a product with properties similar to Magnox glasses

⁵ A convenient way of describing HLW concentrations is as the WO content in the liquid feed, with units of g/l. To calculate this quantity, the elemental composition of the liquid feed is considered as fully oxidised compounds of each element, the state that they would be in if charge balanced in a large oxide molecule, i.e. a glass

⁶ Plant throughput, expressed in kg/h, is the total amount of base glass frit fed plus the total amount of WO fed.

Further to these aims, the VTR has also been used to examine the processing of dilute feeds, novel waste compositions with very high loadings of troublesome elements, new plant components, alternative base glass frit compositions and ways of reducing plant component corrosion. Various trials are also carried out with a water only feed to the plant, allowing various internal inspections to be carried out with the plant hot and functioning, but without exposing the operational staff to unacceptable risks. These trials have provided important data for use in modelling calculations that could not have been obtained using a scaled down plant replica.

To date, the VTR has already proved plant operability and product quality at throughputs of 25kg/h and WO incorporation rates of 25%^{w/w} such that these operating regimes have been adopted as standard for WVP. Operation at 28%^{w/w} waste oxide incorporation for Blended waste has also been demonstrated, and is currently being implemented on WVP.

RECENT EXPERIMENTAL RESULTS

Dilute feeds

In the coming years, WVP expects to receive feed stocks that are more dilute than currently provided by the reprocessing plant. Therefore, if the liquid feed rate to the calciner is kept constant, the WO throughput (and therefore the overall production rate) will be reduced. To maintain the current production rate and waste loading, the volume of liquid HLW fed to the calciner will need to be increased. Experiments were performed on the VTR with a feed at a WO concentration of 110g/l. At this concentration, the VTR feed system had to be operated at close to full speed in order to produce a container of 25%^{w/w} WO at a production rate of 25kg/h. This increased feed rate required an increase of calciner temperatures in the upper half of the calciner to >880°C (with 940°C being the operational limit). The VTR proved that operation at this level could be achieved and maintained whilst simultaneously producing an acceptable calcined product.

However, other factors also needed to be considered before WVP operation under these conditions could be recommended. The calciner is heated by 8 banks of electrical elements, 4 banks below the tube and 4 above. Each bank of heaters is known as a “half shell”. If a half shell fails, it is possible to replace the unit, but this requires isolation of the remaining half shells from the power supply and therefore suspension of plant operations. Under standard feed conditions, VTR has previously demonstrated the ability to safely continue operations and maintain calcine quality with one half shell powered down (simulating a half shell failure) thus allowing the failed half shell to be replaced during a planned maintenance period and therefore not interrupting production. This has been achieved by adjusting the temperatures in the remaining operational half shells. In the case of 110g/l WO feed stocks, operation at 25kg/h was demonstrated under the half shell failure scenario when that failure occurred in the lower half of the calciner. However, it was found that when the failure occurred in the upper half of the tube (where liquid phase evaporation takes place), it was not possible to get enough heat into the tube to complete the evaporative process, thus necessitating a reduction in throughput to maintain calcine quality.

Another important factor when considering dilute feed processing is the ability to switch between HLW feeds and water feeds. The calciner is switched to water feed at the end of a feed

cycle whilst the glass is dwelling in the melter in order to achieve the required pouring temperature, and during routine short term maintenance operations. If all feeds were simply halted to the calciner, the lack of latent heat removal could lead to the tube expanding in its frame and destroying the seal at the upper end which prevents process gases from escaping. The current procedure for the transition from HLW feed to water feed involves reducing the calciner setpoints, stopping the HLW flow and incrementally increasing the flow of water to the calciner. This approach was adopted to prevent film boiling occurring, which could result in pressurisation of the system as droplets of water skate through the calciner and rapidly vaporise in the melter. Experiments were performed to develop a methodology for controlling the HLW to water feed transition at the high setpoints required in the calciner for dilute feed processing. Previous experience has suggested that higher temperatures increase the probability of film boiling. Initial experiments were performed in calcination mode which allowed direct observation of film boiling through inspection of the calcine collection vessels, as the occurrence of film boiling would result in the presence of water in these vessels. An initial procedure was developed successfully and simplified the technique currently used on plant. This methodology will be trialled in vitrification mode in the upcoming experimental campaign to ensure its suitability on for WVP application.

High and Very High Magnox Incorporation

Previous work [3] has proved that product quality can be maintained to acceptable standards⁷ upon increasing the incorporation of Magnox waste streams in the glass, indicating that operation at 28%^{w/w} is feasible. However, to justify implementing this waste loading on WVP, it was necessary to prove long term continuous operation of the plant under these conditions, and to define the operational envelope within which an acceptable product could be manufactured. A VTR program was undertaken during which 57 pours were completed at a Magnox WO incorporation of 28wt% with a single operational sparge⁸ and the plant operating under a realistic WVP type regime. Low and high limits were established for melter wall setpoints and sparge flow rates beyond which glass quality would be unacceptable or melter operation became problematic. The POG was regularly analysed for signs of blockage formation and to monitor dust carryover, to ensure that there were no problems such as the formation of excessive fines or volatile compounds. The experiments were completed successfully without any indication of POG problems, thus it was possible to devise an implementation procedure for WVP to move to 28%^{w/w} WO oxide incorporation. With the assistance and advice of the VTR technical and operations teams, this procedure will be implemented on WVP during the next Magnox HLW vitrification period.

In order to further the understanding of Magnox vitrification along with testing the abilities of the plant, a series of 25 pours was completed with multiple operational sparge pipes and very high Magnox incorporation levels of 38%^{w/w}. At these levels, some of the waste stream elements are present in the product at levels in excess of some of the base glass formers, and are thus playing a major structural role in the formation of the glass matrix, tending to increase the

⁷ cf. in-house analysis of previously accepted lower incorporation products

⁸ The VTR crucible is equipped with 3 sparge pipes which can be used individually, in pairs or all at once. Currently, most WVP crucibles are only fitted with 1 sparge pipe in down the centre of the crucible, but the plant may move to consistent multiple sparging in the future

viscosity of the glass. Furthermore, spinel formation (figures 2a and 2b) was becoming prevalent in the process, with the major components being Al, Mg, Fe, Ni, Cr, forming particles up to ~5mm in diameter. Traditionally, spinel formation has been viewed as an undesirable phenomenon in waste vitrification at Sellafield due to the potential for the melter heel to become enriched with the spinel (which could increase the viscosity of the heel and lead to the blockage of the pour nozzle) and movement away from the idealised homogeneous product glass. However, chemical analysis of samples taken from the heel of the melter after 25 very high incorporation pours had been completed revealed that the levels of all of the elements in the heel were the same as the average level in the poured glass products. Image analysis on the macro scale indicated that the vitrified product was effectively a homogeneous glass containing an even distribution of refractory spinel particles and Soxhlet leach testing showed that the vitrified products had similar bulk leach rates to Magnox waste vitrified products containing 25%^{w/w} WO. Overall, the quality of this product was classed as acceptable when assessed using the standard Nexia Solutions quality assurance procedure for simulated waste glass compositions.

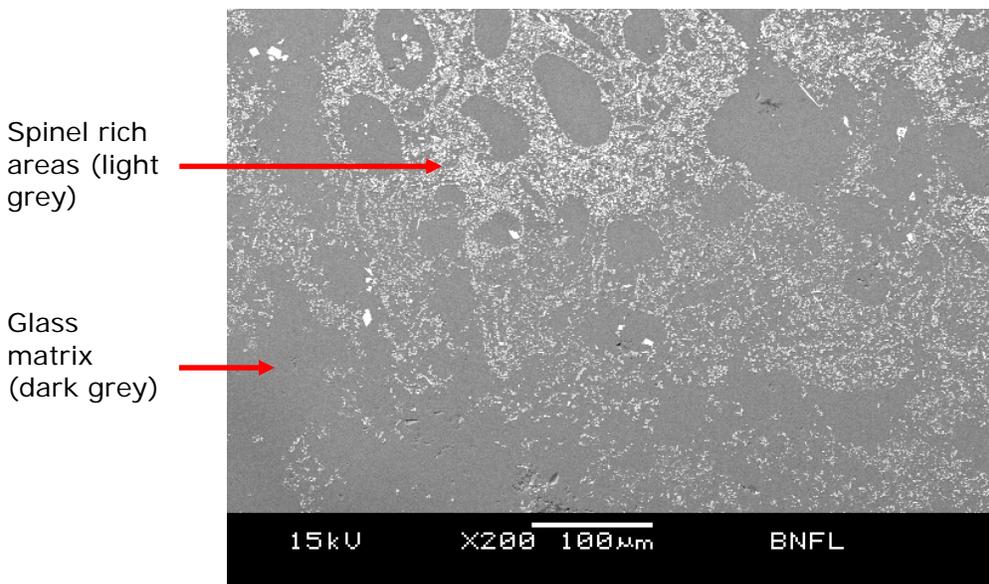


Figure 2a – Spinel particle in a simulated HLW glass, taken on a Jeol JSM-5600 Scanning Electron Microscope in backscattered mode

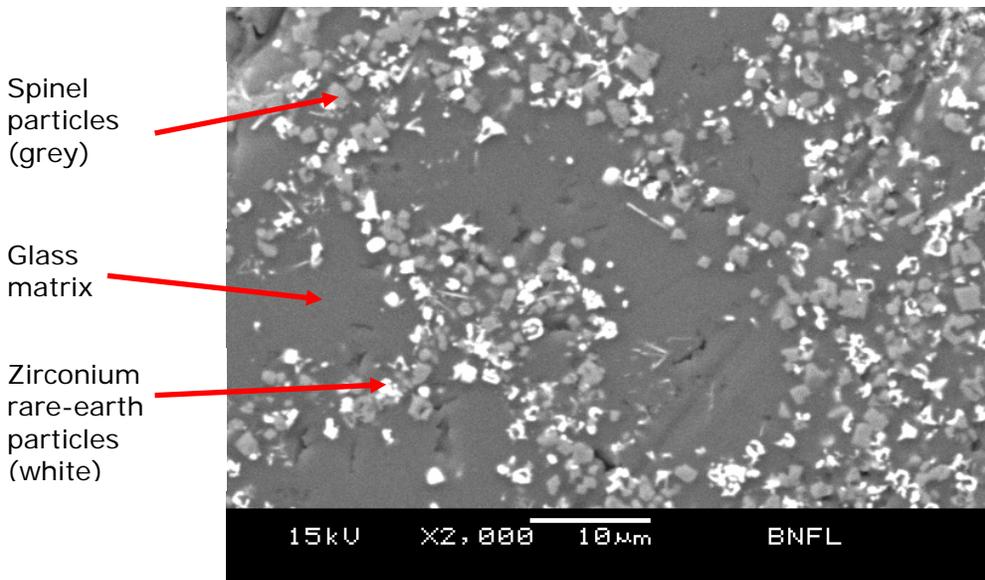


Figure 2b – Spinel particle in a simulated HLW glass at higher magnification, taken on a Jeol JSM-5600 Scanning Electron Microscope in backscattered mode

As well as confirming the quality of the product with the high Magnox incorporation, the ability of the plant to deal with such compositions was challenged by using very high WO throughputs in the calciner to achieve a reasonable feed cycle time, and relatively low melter wall setpoints to assess the capability of the melter. For comparison, the waste oxide throughput of the plant was 1.6 times that used for standard 28%^{w/w} operation, while the baseline melter wall temperatures were on average ~15°C lower than the baseline temperatures used during the 28%^{w/w} experiments. As expected, the higher WO throughput led to a higher overall solids abatement rate in the dust scrubber than would normally be expected, although there was no indication of blockage formation in the POG. In the melter, dwell periods to reach pouring criteria had to be extended (due to the lower wall temperatures used), although under these conditions the extra time required to produce each container would be countered by the increase in the overall processing rate of the HLW stocks as a function of the high waste loading per container. Furthermore, this effect could also be countered by turning up the melter temperatures, and with the wall setpoints at ~15°C on average higher than used for standard 28%^{w/w} operation, the melt was at pour criteria upon completion of feeds, thus reducing the feed cycle times.

Alternative Base Glass Formulations

The standard base glass used in HLW vitrification (termed “MW”) in the UK is composed, on a molar basis, of 60.5% SiO₂, 18.5% B₂O₃, and 10.5% each of Na₂O and Li₂O⁹. This composition was found to be suitable for vitrification of both Magnox and Blended waste streams during early development work for WVP and to give a product that had similar physical properties regardless of the waste type. However, moving to higher waste loadings, the desire to increase the plant throughput and increase component longevity has led to the examination of alternative

⁹ Some of the total amount of Li₂O in the final glass product is added as a LiNO₃ solution to the liquid HLW prior to calcination. As the LiNO₃ addition precedes the vitrification step, the final composition of the base glass matrix depends on the overall HLW loading of the product

glass compositions. To this end, a series of laboratory trials were performed with a calcined simulated Blended waste stream and a range of base glass formulations containing Si, B, Na, Li, Ca, Zn and Al in various ratios. The performance of these compositions was rated based on the reactivity of the base glass with the calcine, the viscosity of the product glass, the chemical durability of the product glass, the stability of the product upon heat treatment and the corrosion rate of Inconel (from which the melting crucibles on WVP and VTR are fashioned) in the glass. After the trials were completed, the composition chosen for full scale trials on the VTR consisted, on a molar basis, of 54% SiO₂, 18.9% B₂O₃, 1.8% CaO, 4% ZnO and 10.6% each of Na₂O and Li₂O¹⁰ (termed “CZ”)

As laboratory trials had been performed with a Blended waste simulant at ≤ 31%^{w/w} incorporation, it was decided to perform the VTR trials with a Magnox simulant at very high waste loadings (38%^{w/w}) as these conditions were most likely to give a highly viscous and poorly reacted product. This also allowed direct comparisons with the results of the very high waste loading experiments performed with the MW base glass described above. Evaluation of the results (table 2) showed significant benefits over MW glasses in terms of the viscosity of the glass during pouring, with pour rates for the CZ containing products of nearly 200kg/h more than for the MW base glasses when comparing glasses with the same average internal temperatures upon pouring. It was also found that with an average glass temperature in the melter of 36°C less than for the MW glass, the pouring rate of the CZ glass was still higher than for MW glass.

Table 2 - Comparison of pouring rates of MW and CZ glasses at a range of temperatures

	MW base glass 38% ^{w/w} Magnox loading	Ca & Zn base glass 38% ^{w/w} Magnox loading
Pour rate with 1053°C internal average (kg/h)	281	473
Pour rate with 1017°C internal average (kg/h)	-	344
Pour rate with 985°C internal average (kg/h)	-	186

Being able to achieve higher pour rates (i.e. lower viscosity) at lower temperatures is beneficial to the process as it reduces the corrosion rate of the crucible and helps to reduce the stress of high temperature gradients. Analysis of the CZ products is still in progress, but indicates that they performed slightly better than the equivalent MW products in terms of leach testing and contained similar or lower levels of spinel phase on the macro scale. Chemical analysis of the glass products, and of the melter heel after the experimental run of 19 CZ pours had been completed indicated that no heel enrichment had taken place. Thus, initial experimentation on the full scale plant indicated that CZ glass could be a better host matrix than standard MW glass for high waste to base glass ratios.

Water feed trials

WVP has recently fitted new equipment to plant to enable the unblocking of pressure tappings using a high pressure water jet. This offers significant advantages over mechanically clearing the blockage (the current methodology for removing blockages) as the water jet can be used whilst

¹⁰ See ⁹ above

the plant is hot and in feed, thus causing minimum disruption to plant operation. Most of the pressure tappings on the plant are located within the POG and are capable of absorbing the pulse of water that the pressure washer emits without any effect on their operability. However, some of the pressure tappings are located in positions that can lead to the pulse of water from the pressure washer entering the calciner. Whilst the total volume of water emitted by the water jet is small (<2500ml), the flow rate is high and thus there is potential for calefraction to occur in the calciner and for the wash water to enter the melter and rapidly evaporate, causing a pressurisation.

Therefore, prior to use on WVP, the pressure washing equipment was trialled on VTR. In water feed, the VTR was taken to standard operating temperatures for 28%^{w/w} and the melter charged with a full feed cycle equivalent of base glass and allowed to equilibrate in temperature. The pressure washing equipment was then used to fire various volumes of water into the pressure tappings that could allow forward flow to the calciner. The pressure in the calciner and the POG was monitored as the pressure washer was fired and the maximum pressure logged. The experiment determined that the maximum volume of water that could be fired into the least restrictive tapping was 30ml, to give a 99% confidence limit of not causing a pressurisation of greater than -3mbar in the calciner¹¹, and also established the level of pressurisation caused by various injection volumes >30ml. This allowed WVP to set appropriate restrictions on the use of the high pressure jet for unblocking pressure tappings.

The VTR team has also developed a temperature probe that has been deployed inside the calciner tube during water feed operations at representative calcination temperatures. Under normal operation, the VTR measures the temperature of the outside of the tube via four pyrometers spaced equidistantly along the length of the tube. However, due to the lack of compensation for radiant heat from the furnaces these pyrometers do not give a true temperature reading and furthermore there is a temperature gradient between the inner and outer surfaces of the tube. It was possible to surmise that the surface of the tube in contact with the liquid phase in the upper half of the calciner had to be $\leq 120^{\circ}\text{C}$ (the approximate boiling point of the HLW liquid feed) although the temperature of the inner surface of the tube at alternative points on its circumference, or indeed anywhere below the evaporative front, was unknown. Using the temperature probe allowed the measurement of temperatures at any point along the entire inner surface of the calciner tube. The experimental programme mapped the temperature profile at a series of equidistant points on the inner surface at a range of temperature setpoints. This work provided detailed information on the location of the evaporative front in the calciner, an important parameter in the production of a calcine of consistent quality. It is now also being used by WVP to generate an accurate computer model of the thermodynamics inside the calciner.

FUTURE WORK

In 2008, the VTR will embark on its 9th operational campaign, during which an operational envelope for the vitrification of Magnox waste streams at 32%^{w/w} incorporation levels will be

¹¹ As the pressure in the calciner and POG varies constantly, it is not possible to give an absolute value at which pressurisation will not occur. Also, whilst the calciner itself is capable of containing positive pressures of up to 75mbar prior to experiencing a release of pressure via its safety seals, a safety trip cuts HLW feed to the plant if the pressure in the calciner rises above -3mbar

determined. In addition to this work, experiments will be performed with dilute feeds in vitrification mode to confirm the product quality, and some trials will be carried out using a Blended HLW simulant with very high levels of Ru and Pd to simulate the worst case scenario for these elements and to determine their effect on heel enrichment and glass viscosity.

Campaigns beyond 2008 are yet to be defined, but are likely to address such issues at waste arisings from post operation clean out of the HLW storage tanks and more extensive investigations with alternative base glass compositions and HLW feeds.

SUMMARY

The VTR has been successfully used to determine operational envelopes and product quality for several HLW feed variations that will allow WVP to increase overall plant throughput via increased waste loading in canisters, increased HLW feed rates or a combination of both. The VTR has also demonstrated the ability to go to waste incorporations, feed rates and glass compositions that are currently beyond WVP specified limits, but that are feasible for future vitrification regimes. In addition, the VTR has trialled dilute feeds similar to those that are likely to be received by WVP in the future and the data obtained from these experiments will allow WVP to prepare adequately for the high throughput challenge of such feeds. Furthermore, new equipment has been trialled on the VTR in water feed mode to determine its suitability and operational limitations for WVP. Future operations will, in the short term, be concerned with increasing the throughput of WVP and are likely to focus on HLW decommissioning operations waste streams in the longer term.

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ACKNOWLEDGEMENTS

The authors would like to thank Sellafield Ltd for sponsoring this work on behalf of the Nuclear Decommissioning Authority.