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Crystal Accumulation in the Hanford Waste Treatment Plant High Level Waste Melter: Summary of FY2016 Experiments

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EXECUTIVE SUMMARY

Five experiments were completed with the full-scale, room temperature Hanford Waste Treatment and Immobilization Plant (WTP) high-level waste (HLW) melter riser test system to observe particle flow and settling in support of a crystal tolerant approach to melter operation. A prototypic pour rate was maintained based on the volumetric flow rate. Accumulation of particles was observed at the bottom of the riser and along the bottom of the throat after each experiment. Measurements of the accumulated layer thicknesses showed that the settled particles at the bottom of the riser did not vary in thickness during pouring cycles or idle periods. Some of the settled particles at the bottom of the throat were re-suspended during subsequent pouring cycles, and settled back to approximately the same thickness after each idle period. The cause of the consistency of the accumulated layer thicknesses is not year clear, but was hypothesized to be related to particle flow back to the feed tank. Additional experiments reinforced the observation of particle flow along a considerable portion of the throat during idle periods. Limitations of the system are noted in this report and may be addressed via future modifications.

Follow-on experiments will be designed to evaluate the impact of pouring rate on particle re-suspension, the influence of feed tank agitation on particle accumulation, and the effect of changes in air lance positioning on the accumulation and re-suspension of particles at the bottom of the riser. A method for sampling the accumulated particles will be developed to support particle size distribution analyses. Thicker accumulated layers will be intentionally formed via direct addition of particles to select areas of the system to better understand the ability to continue pouring and re-suspend particles. Results from the room temperature system will be correlated with observations and data from the Research Scale Melter (RSM) at Pacific Northwest National Laboratory, and coordinated with modeling efforts underway at Idaho National Laboratory.

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LIST OF ABBREVIATIONS

DOE	U.S. Department of Energy
HLW	High-Level Waste
LAW	Low-Activity Waste
ORP	Office of River Protection
RSM	Research Scale Melter
$T_{1\%}$	Temperature where one volume percent of spinel crystals are present in a glass melt
vol %	Volume percent
WTP	Hanford Tank Waste Treatment and Immobilization Plant

1.0 Introduction

The U.S. Department of Energy (DOE) Office of River Protection (ORP) is building the Tank Waste Treatment and Immobilization Plant (WTP) at the Hanford Site in Washington to remediate 56 million gallons of radioactive waste that is being temporarily stored in 177 underground tanks. Radioactive waste will be separated into high-level waste (HLW) and low-activity waste (LAW) fractions that will be vitrified in stable borosilicate glass with Joule-heated, ceramic refractory lined melters. Efforts are being made to increase the loading of Hanford tank wastes in glass while maintaining an adequate ability to meet process, regulatory, and product quality requirements.

Glass formulation and melter testing data have suggested that significant increases in waste loading in HLW and LAW glasses are possible over current system planning estimates.[1] Belsher and Meinert identified five constraints that were most influential on the estimated Hanford HLW glass volumes,[2] and by extension, most restricting to waste loading. One of those constraints was the limit of no more than one volume percent spinel crystals in the melt ($T_{1\%}$) at a temperature of 950 °C.

Historically, crystallization constraints are placed in process control systems to prevent premature or catastrophic failure of the melter through bulk devitrification (also described as volume crystallization) or crystal accumulation and, thus, to mitigate negative impacts of crystals as glass is produced.[a] The baseline method of controlling crystallization in the WTP HLW melter uses a model that predicts the temperature, $T_{1\%}$, at which the equilibrium fraction of spinel crystals in the melt is 1 volume percent (vol %).[4] An alternative crystal-tolerant glass approach[5] may allow higher waste loading for WTP processing while maintaining a chemically durable glass product. Some crystalline phases, such as spinel, do not impact the durability of the waste form[6] but may accumulate in the melter or riser and restrict or prevent its operation. However, prediction of spinel precipitation and accumulation could potentially allow for formulating higher waste loading, durable glasses if an alternative strategy for operating and idling a melter with some amount of tolerable crystals can be developed and implemented.

Given the identification of the $T_{1\%}$ constraint as one of the most influential constraints for estimated Hanford HLW glass volumes, ORP has initiated a program to evaluate whether this constraint can be relaxed or whether new constraints could be developed to replace the current $T_{1\%}$ approach.[7,8] A road map was developed to guide research and development efforts for a crystal tolerant glass processing strategy for WTP.[9] The basis of this potential, alternative approach will be an empirical model predicting the crystal accumulation in the WTP glass discharge riser and melter bottom as a function of glass composition, time, and temperature.[5,10] When coupled with an associated operating limit, this model could then be integrated into the process control algorithms to formulate crystal tolerant HLW glasses targeting higher waste loadings while still meeting other process related limits and melter lifetime expectancies.

Actual melter operation is likely to involve situations where accumulation of spinel crystals can occur. Unexpected events may hamper the use of a crystal accumulation process control model. Methods of recovering from such an event will make the crystal tolerant approach more robust, and allow for continued use of a melter in the event of excessive crystal accumulation.

To better understand crystal settling, accumulation, and resuspension in critical areas of the WTP HLW melter, a full-scale, room temperature test system has been designed and constructed.[11] The road map for development of crystal-tolerant HLW glasses noted that an accumulation of crystals in the melter riser could prevent discharge of the molten glass into canisters, especially when considering frequent and periodic idling.[9] Therefore, the test system focuses on the throat and riser of the WTP HLW melter. The

[a] Jantzen and Brown provide a brief review of the potential, negative effects of crystallization within a melter.[3]

system uses transparent materials to allow for the observation of particle behavior under a variety of process conditions. Data collected will support the development and implementation of a crystal accumulation process control model. The system will also be used to develop and demonstrate potential methods for recovery in the event of an unacceptable amount of crystal accumulation.

This report describes the results of experiments completed in Fiscal Year 2016 (FY16) with the full-scale, room temperature WTP HLW melter riser system. Observations from the experiments are provided and discussed, and recommendations are made for future testing. This work was performed following a Task Technical and Quality Assurance Plan.[12] Experimental plans were developed and issued for system design and material selection,[13] as well as preliminary particle settling experiments.[14]

2.0 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Savannah River Site Manual E7, Procedure 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Laboratory data for this study were recorded in the SRNL Electronic Laboratory Notebook system, experiment L0008-00162-01.

3.0 Experimental Runs

3.1 Test Conditions

The design, materials selection and operation of the full-scale, room temperature WTP HLW melter riser test system were described in a previous report.[11] Five experimental runs were completed using the system in FY16:

- The first test was used to load the system with particles and monitor their settling behavior.
- The second and third tests were used to demonstrate restarting of pouring with particles in the system, to demonstrate the ability to maintain a consistent pour rate, and to verify visual observation as a viable method for identifying particle resuspension and settling. A secondary intent was to increase the thickness of the settled layers of particles within the throat and riser.
- The fourth test monitored changes in the thickness of the settled layers during a pouring cycle to identify repeatability.
- The fifth test was used to observe potential differences in particle behavior when pouring at a reduced volumetric flow rate.

The system was allowed to settle for several days between each of the experimental runs. Particle loading in the fluid was 0.1 vol % for the first series of experiments to maintain visibility through the fluid. A nominal pouring rate of 3.18 lpm (0.84 gpm) was targeted for the first four tests. This pouring rate is the volume of glass planned to be poured per unit time in the actual melter. The rate was calculated using a nominal WTP HLW melter pour rate of 520 lbs of glass in a period of 29 minutes (8.13 kg/min),[15,a] and an arbitrary glass density of 2.56 g/cm^3. The pouring rate for the fifth test was reduced to approximately 2/3 of that of the previous tests (2.3 lpm or 0.60 gpm) to identify potential differences in particle re-suspension.

3.2 Observations

General observations noted during the first three tests are provided in the preliminary testing report.[11] In summary, the results of these tests demonstrated that the system was able to maintain the targeted pour

[a] Note that the production rate of 4 MT/day given in Reference 15 is higher than the design capacity production rate of 3 MT/day given in the IHLW Waste Form Qualification Report.[16] The higher rate production rate, and therefore higher pouring rate, was used in this study and considered to be an upper bound.

rate; that suspension, flow, settling, and accumulation of particles could be readily observed visually using real-time and time-lapse recording; that the settled layer of particles at the bottom of the riser appeared to be unaffected by pouring and settling cycles; and that the accumulated layer of particles along the bottom of the throat was somewhat reduced after a pouring cycle and built back up during idle periods.[11]

The temperature of the slurry during the five experiments and the calculated[11] viscosity of the silicone oil at those temperatures are shown in Table 3-1. The ambient temperature in the laboratory varied from day to day, resulting in changes to the viscosity of the silicone oil among the five experiments. Qualitatively, this did not appear to impact particle settling behavior among the experiments.

Table 3-1. Slurry Temperature and Calculated Silicone Oil Viscosity

Particle Settling Experiment	Slurry Temperature (°C)	Calculated Silicone Oil Viscosity (Poise)
1	23	58
2	17	66
3	25	56
4	26	54
5	22	59

Further observations during the fourth and fifth tests were made using both real-time and time-lapse video recordings. During the pouring cycles, a reduction in the thickness of the accumulated layer within the throat was observed. This indicates that the flow of material through the throat was sufficient to re-suspend some of the settled particles. Note however that the smaller particles typically settled last, and therefore are easier to re-suspend in the subsequent pouring cycle as compared to larger particles. Changes in the thickness of the settled layer at the bottom of the riser were not evident during pouring cycles.

Experiments 3, 4, and 5 were run with a continuous purge (0.2 scfh) of the air lance in the riser to better simulate planned operation of the WTP HLW melter. The idle purge flow was observed to produce a more complex flow pattern in the riser during idle periods. It did not appear to impact the accumulation of particles in the riser or throat relative to the experiments run without the idle purge.

3.3 Accumulated Layer Measurements

A review of the time-lapse videos recorded during the idle periods showed sliding of the settled particles along the inner wall of the throat, which may not be representative of the actual melter should the spinel crystals adhere to the refractory. As was the case during pouring cycles, changes in the thickness of the settled layer at the bottom of the riser were not evident during idle periods.

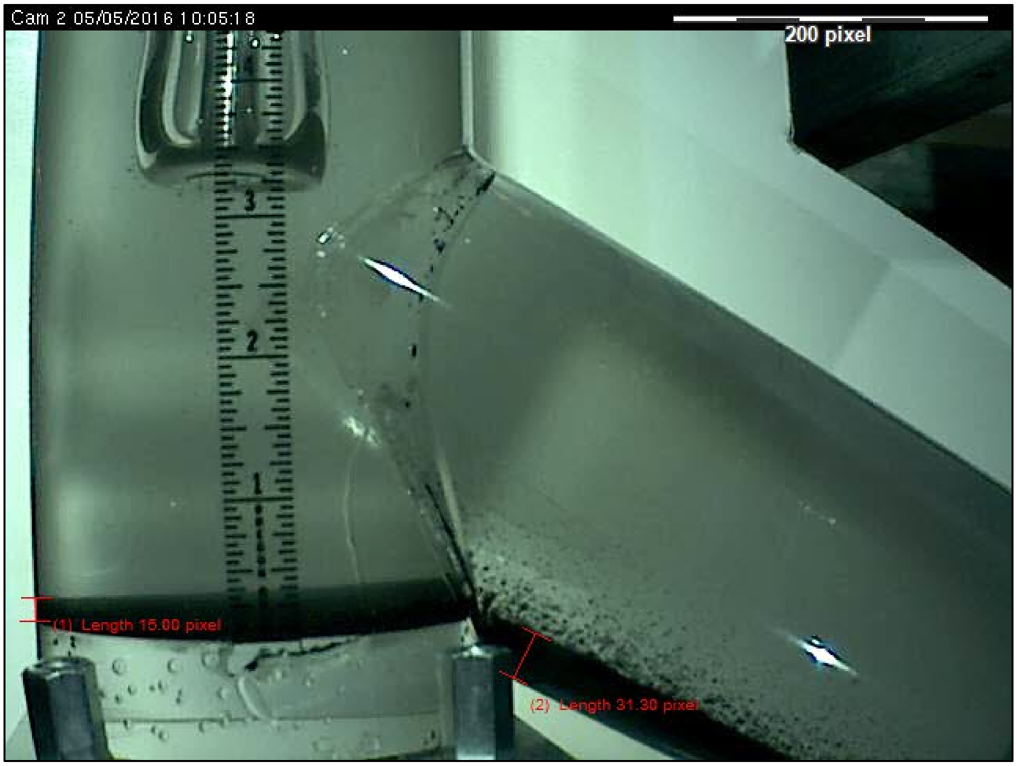
A more quantitative approach to determine changes in the thickness of the accumulated layers during pouring and idle periods was attempted using still images from the video recordings. Images of the junction between the throat and the riser were used for reference since the position of the camera was not changed during the series of experiments. Image analysis software was used to measure the thickness of the accumulated layers at the bottom of the riser and throat before and after the pouring cycles, and at the beginning and end of the idle periods. Example measurements are shown in Figure 3-1 for the beginning and end of the pouring cycle from Particle Settling Experiment 5. The thickness of the accumulated layer at the bottom of the riser was measured from a reference point on the bottom left of the riser, as shown in the figure. In this example, the difference in the measurements at the beginning (15 pixels) and end of the pouring cycle (16 pixels) is considered insignificant, as the resolution of the measurements is no less than one pixel. The thickness of the accumulated layer at the bottom of the throat was measured from a reference point where the bottom of the throat visually intersects one of the stand-offs supporting the riser, as shown at the bottom center of the images in Figure 3-1. The change in accumulated thickness at this location was considered significant (31.3 pixels versus 25.5 pixels).

Table 3-2 summarizes the accumulated layer thickness changes during the pouring cycles of the five experiments, measured as described above. Measured differences in the thickness of the accumulated layer at the bottom of the riser were all within one pixel, indicating no significant (measureable) change between the start and end of each pouring cycle. Differences in the thickness of the accumulated layer at the bottom of the throat were measureable and on the order of a 30% reduction in thickness when the flow rate targeted 0.84 gpm. When the targeted flow rate was reduced to 0.60 gpm for Experiment 5, the thickness of the accumulated layer in the throat was reduced by about 20% after the pouring cycle. This indicates that, as expected, the lower flow rate re-suspended less of the accumulated particles in the throat. Additionally, the flowrate applies a shear stress at the wall, which is the mechanism that moves/suspends particles and as the flowrate increase, so does the applied shear stress.

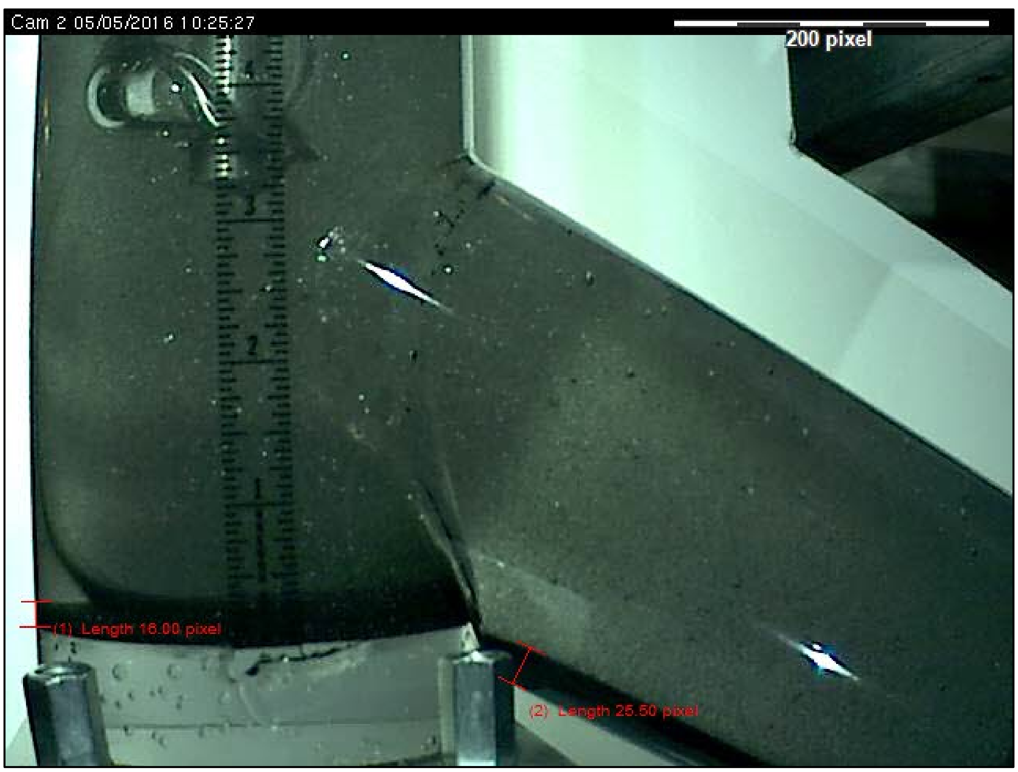
Table 3-3 summarizes the accumulated layer thickness changes during the idle periods of the five experiments.[a] Again, measured differences in the thickness of the accumulated layer at the bottom of the riser were all within one pixel, indicating no significant (measureable) change between the start and end of each idle period. The thickness of the accumulated layer at the bottom of the throat increased significantly (~60-80%) during the idle periods. Note that the accumulated layer thickness at the start of each pouring cycle was somewhat less than that at the end of the previous idle period. This indicates that the particles continued to settle in the days that passed after time-lapse recording of the idle period was stopped (roughly 20 hours).

These data indicate that the settled layer at the bottom of the riser quickly (after the first settling experiment) reached a stable thickness. The data also indicate that the settled layer at the bottom of the throat is partially re-suspended during pouring, and re-forms at approximately the same thickness during the idle periods. Neither accumulated layer appears to increase in thickness in a step-wise manner with each pouring and settling cycle, as was first anticipated. The reason for this observed behavior is not yet clear.

[a] Note that there were slight differences in timing when transitioning the video recording system from real-time to time-lapse modes, which resulted in small (1-2 pixel) differences in the thickness measurements of the accumulated layer thickness in the throat. Thus, the throat layer thickness measurements at the finish of each pouring cycle do not necessarily match those at the start of the subsequent idle period. These differences were considered insignificant relative to the magnitude of the changes measured over the course of each idle period.



(a)



(b)

Figure 3-1. Example accumulated layer thickness measurements at the beginning (a) and end (b) of a pouring cycle

Table 3-2. Accumulated Layer Thickness Measurements for the Pouring Cycles

Particle Settling Experiment	Targeted flow rate	Start time	Finish time	Riser layer thickness, start (pixels)	Riser layer thickness, finish (pixels)	Change	Throat layer thickness, start (pixels)	Throat layer thickness, finish (pixels)	Change
1	0.84 gpm	1/28/2016 10:43:16	1/28/2016 10:53:00	0	0	--	0	0	--
2	0.84 gpm	2/10/2016 10:19:05	2/10/2016 10:36:06	11	10	-9%	29.79	20.12	-32%
3	0.84 gpm	4/25/2016 09:38:27	4/25/2016 09:52:02	15	14	-7%	32.65	23.71	-27%
4	0.84 gpm	4/27/2016 12:58:41	4/27/2016 13:14:15	15	14	-7%	33.54	22.36	-33%
5	0.60 gpm	5/5/2016 10:05:18	5/5/2016 10:25:27	15	16	7%	31.3	25.5	-19%

Table 3-3. Accumulated Layer Thickness Measurements for the Idle Periods

Particle Settling Experiment	Start time	Finish time	Riser layer thickness, start (pixels)	Riser layer thickness, finish (pixels)	Change	Throat layer thickness, start (pixels)	Throat layer thickness, finish (pixels)	Change
1	1/28/2016 10:52	1/29/2016 07:35	0	10	--	0	35.78	--
2	2/10/2016 10:35	2/11/2016 02:50	11	12	9%	20.12	37.12	84%
3	4/25/2016 09:52	4/26/2016 05:00	15	15	0%	22.36	37.12	66%
4	4/27/2016 13:13	4/28/2016 11:50	15	15	0%	21.02	33.54	60%
5	5/5/2016 10:25	5/6/2016 04:30	16	16	0%	24.6	39.36	60%

3.4 Particle Flow During Idle Periods

It was hypothesized that the unexpected behavior of the accumulated layers may be due to flow of the particles through both the riser and throat back to the feed tank, as outlined in Figure 3-2. Review of the time-lapse recordings identified flow of the suspended particles through both the riser and throat toward the feed tank, as well as recirculation of the suspended particles in the opposite direction. This may result both in a thicker than expected accumulated layer in the throat, and an overall reduction in the expected thickness of the accumulated layers if particles return to the feed tank.

To test this hypothesis, a small quantity (~100 ml) of silicone oil with suspended magnetite particles was collected from the make-up tank and poured directly onto the surface of the fluid in the riser. This allowed for observation of the flow of a small “group” of particles. Time-lapse images of the particle flow are shown in Figure 3-3, where the group of particles is highlighted with arrows. The group of particles is seen settling through the riser in Figure 3-3a, and flowing into the throat in Figure 3-3b. The group of particles continues to flow down one third to one half the length of the throat, as shown in Figure 3-3c and Figure 3-3d. It was not clear (using the current camera locations) whether any of the solids flowed all the way back to the feed tank. This experiment was repeated with similar results observed the second time. The next series of experiments will include tests designed to better understand the impacts of this particle flow during idle periods.

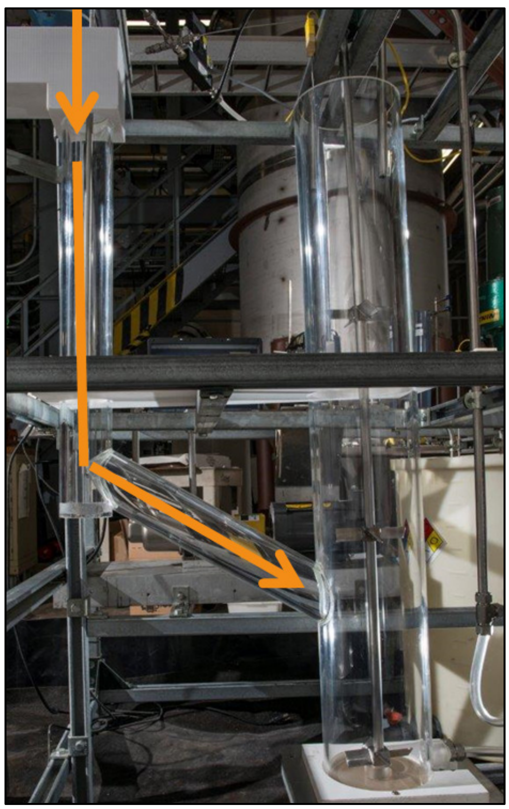


Figure 3-2. Hypothesized Path of Particle Flow During Idle Periods

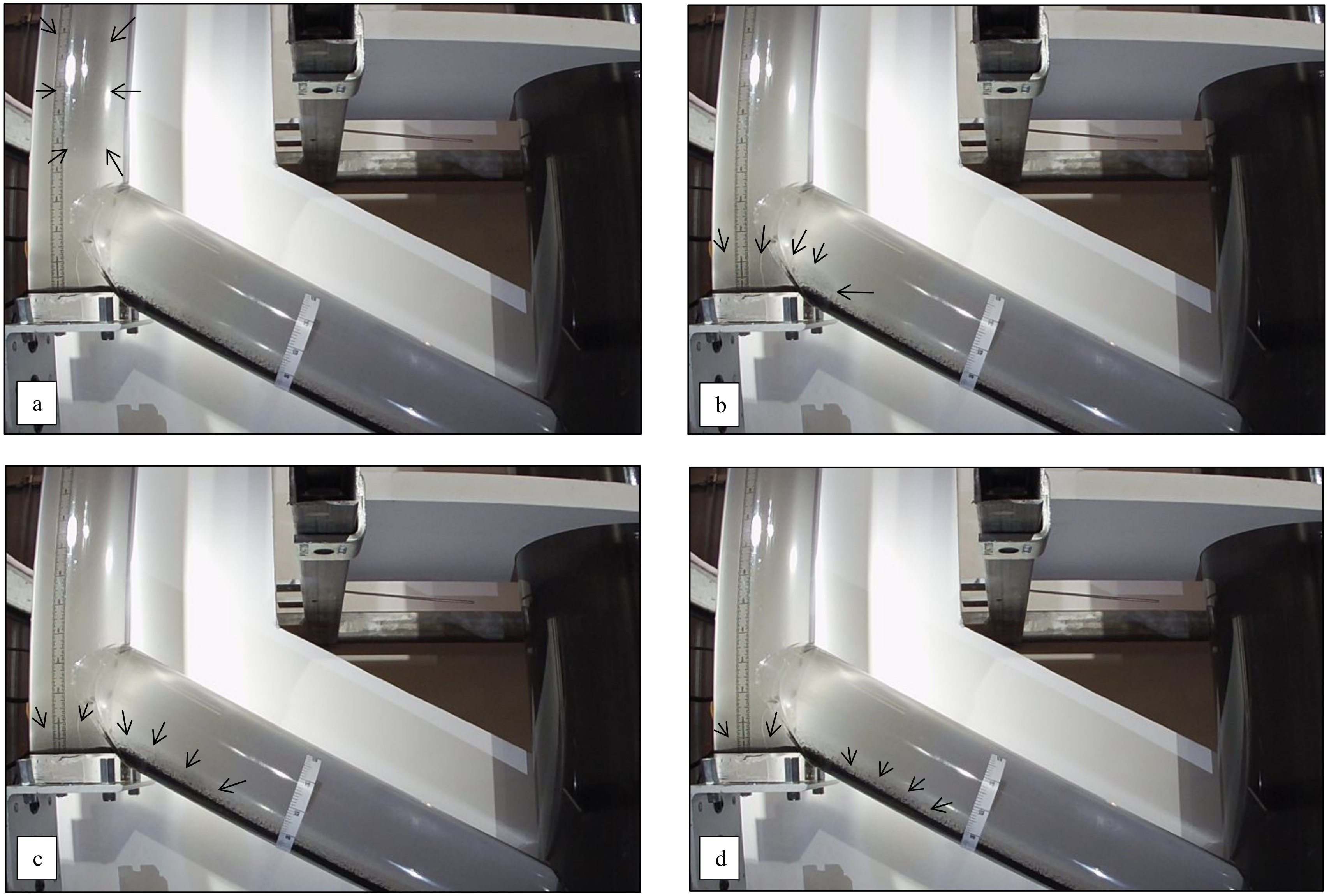


Figure 3-3. Time lapse photos of particle settling and flow into throat (elapsed time 62 minutes)

4.0 Summary

Five experiments were completed with the full-scale, room temperature WTP HLW melter riser test system to observe particle flow and settling in support of a crystal tolerant approach to melter operation. A designed pour rate was maintained based on the volumetric flow rate. Accumulation of particles was observed at the bottom of the riser and the bottom of the throat after each experiment. Measurements of the accumulated layer thicknesses showed that the settled particles at the bottom of the riser did not vary in thickness during pouring cycles or idle periods. Some of the settled particles at the bottom of the throat were re-suspended during subsequent pouring cycles, and settled back to approximately the same thickness after each idle period. The cause of the consistency of the accumulated layer thicknesses is not yet clear, but was hypothesized to be related to particle flow back to the feed tank. Experiments reinforced the observation of particle flow along a considerable portion of the throat during idle periods.

As noted in the preliminary testing report,[11] these experiments may be conservative in that the system is constructed of smooth wall material rather than refractory. Therefore, resuspension of the particles may require less effort than in the actual melter. Smaller particles are expected to have settled last, and may therefore be easier to re-suspend in a subsequent pouring cycle as compared to larger particles. Also, the particles are unlikely to agglomerate or adhere to the walls during testing, which may not be true of crystals in a molten glass in a refractory lined melter. The particle size distribution in the experiments was intentionally kept narrow, with removal of fine particles to support visual observations. A broader particle size distribution may form a better packed settled layer that would be more difficult to re-suspend. Also of note is the non-prototypic difference in density between the silicone oil and magnetite particles (0.98 vs. 5.2 g/cm^3) as compared to molten glass and spinel crystals (2.6 vs. 5.3 g/cm^3).[11] This impacts particle flow and settling behavior.

5.0 Future Work

Topics for future experiments are listed in this section. A more detailed experimental plan for FY2017 will be developed and issued by the end of calendar year 2016.

- Experiments at lower pouring rates will be performed to verify that less re-suspension of particles occurs.
- Experiments will be run with feed tank agitation left on during idle periods to identify whether there is influence on the formation of accumulated layers.
- The position of the air lance will be adjusted to determine whether it can effectively re-suspend particles accumulated at the bottom of the riser. Experiments will then be performed to determine whether accumulated layers with “equilibrium” thicknesses form again.
- A method for sampling the accumulated particles will be developed to support particle size distribution analyses.
- Thicker accumulated layers will be intentionally formed via direct additional of particles to select areas of the system to better understand the ability to continue pouring and re-suspend particles.
- Methods for increasing the density of the silicone oil (or an alternative fluid) will be explored.
- Thus far, the experiments have targeted a relatively low volume fraction of particles in the fluid to ensure that flow and settling could be visually observed. In actual operations, the amount of spinel crystals in the melter may be 1 vol % or more. Future stages of testing will utilize higher volume fractions (e.g., 0.5 to 1 vol %) of particles in the fluid.
- Results from the room temperature system will be correlated with observations and data from the Research Scale Melter (RSM) at Pacific Northwest National Laboratory.
- It may be possible to collect more quantitative information from the system in order to support crystal accumulation modeling efforts. This will be coordinated with efforts underway at Idaho National Laboratory.

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