

Am/Cm TTR Testing - 3/8" Glass Beads Evaluation in CIM5

by

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Date: 12/15/99

TO: W. H. MARTIN, 730-2B

FROM: L. F. LANDON, 704-1T


AM/CM TTR TESTING - 3/8" GLASS BEADS EVALUATION IN CIM5

The attached document describes the results of the evaluation of glass beads as a potential glass former alternative to cullet. This work was in response to Task 1.02 of TTR 99-MNSS/SE-006. Please refer any questions you may have regarding the contents of this document to D. C. Witt (Ext. 7-7754)

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AM/CM TTR TESTING - 3/8" GLASS BEADS EVALUATION IN CIM5 (U)

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SAVANNAH RIVER SITE

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AM/CM TTR TESTING - 3/8" GLASS BEADS EVALUATION IN CIM5 (U)

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I. INTRODUCTION

To facilitate the procurement and handling of the glass former for Am/Cm vitrification in the F-Canyon MPPF, 1/4 inch and 3/8 inch diameter glass beads were purchased from Corning for evaluation in the 5 inch Cylindrical Induction Melter (CIM5). Prior to evaluating the beads in the CIM5, tests were conducted in the Drain Tube Test Stand (DTTS) with 1/4 inch beads, 3/8 inch beads, and a 50/50 mixture to identify any process concerns^{1,2}. Results of the DTTS tests are summarized in Attachment 1. A somewhat larger volume expansion was experienced in all three DTTS runs as compared to a standard run using cullet. Further testing of the use of glass beads in the CIM5 was requested by the Design Authority as Task 1.02 of Technical Task Request 99-MNSS/SE-006. Since the Technical Task Plan was not yet approved, the completion of this task was conducted under an authorization request approved by the SRTC Laboratory Director, S. Wood. This request is included as Attachment 2.

II. SUMMARY

Small volume expansions using 1/4 inch 25SrABS glass beads in place of cullet were observed during two vitrification runs in the CIM5³. These observations are consistent with the three runs made previously in the DTTS. Based on the results of these two glass bead runs, further testing using glass beads was abandoned in favor of 25SrABS cullet.

III. DETAILS - 1/4" GLASS BEAD EVALUATION

Two CIM5 runs (Runs #38 and #39) were made to produce a 49 wt% lanthanide (Ln) loaded 25SrABS glass made with 1/4 inch glass beads in place of cullet. Previous glass bead evaluations in the Drain Tube Test Stand resulted in volume expansions under standard operating conditions (See Attachment 1). In Run #38 the 25SrABS glass beads and oxalate precipitate were charged to the CIM vessel to produce a 49 wt% Ln loading. The drying and calcination processes proceeded as expected. However, a small volume expansion was observed during vitrification. Vessel and bed temperatures, which are shown in Figure 1, increased 40 to 50 °C above normal. The residual glass remaining in the melter at the conclusion of the pour was a lightly loaded glass phase containing a number of small bubbles as seen in previous 49 wt% Ln runs. As shown in Figure 2, typical lanthanide loading variations were observed with the first samples highly loaded and decreasing as the pour progressed.

Run #39 was the second run to evaluate the process effects of using 1/4" 25SrABS glass beads in place of cullet. The drying and calcination processes proceeded as expected. However, a small volume expansion was again observed during vitrification, consistent with Run #38 results. Based on the results of these two glass bead runs, no further testing using glass beads was planned. The glass beads were subsequently used as feed stock to produce 25SrABS cullet needed for TTR testing.

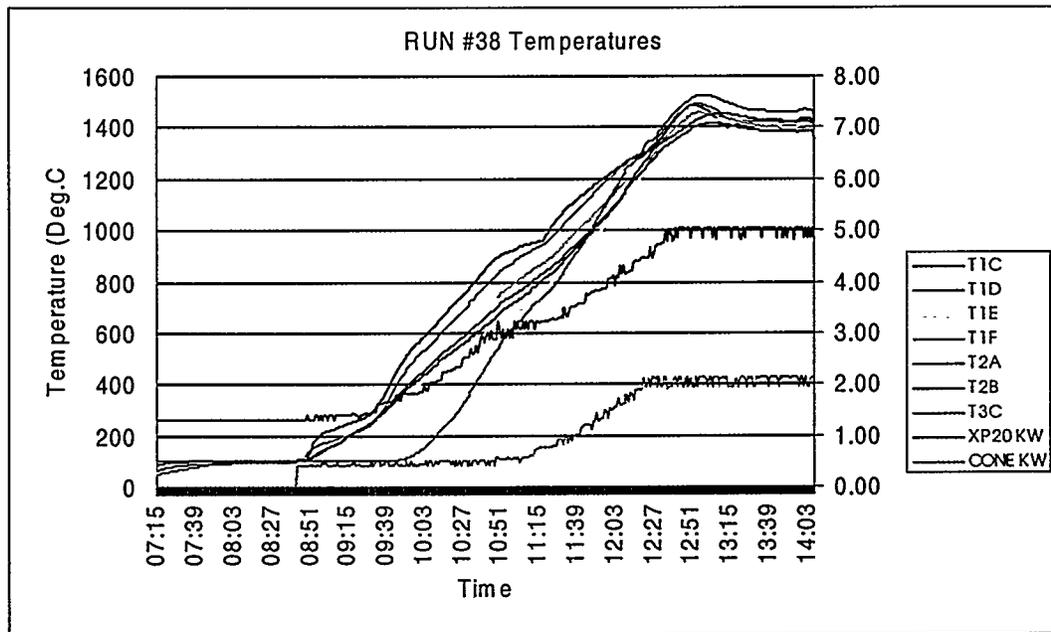


Figure 1

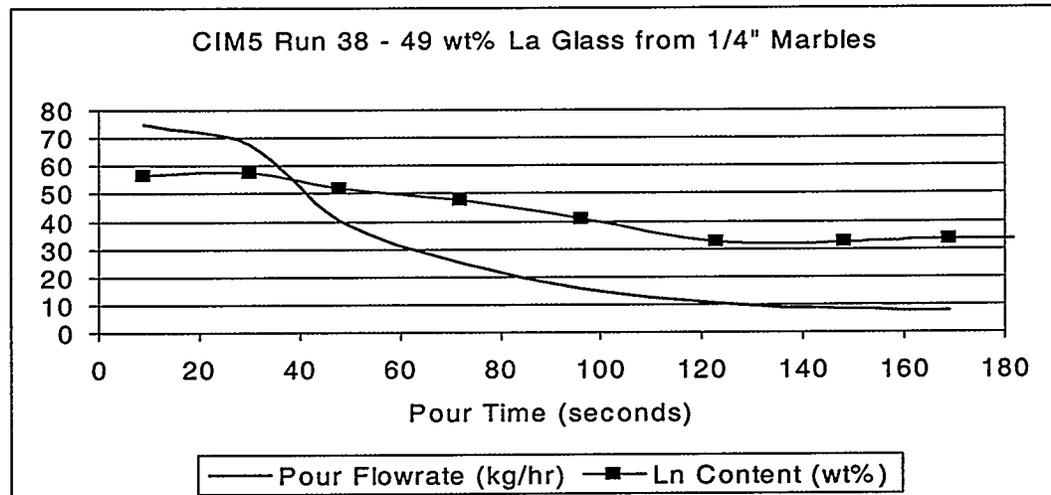


Figure 2

IV. REFERENCES

1. **WSRC-NB-97-239, Drain Tube Test Stand Laboratory Notebook**
2. **WSRC-NB-97-242, Drain Tube Test Stand Laboratory Notebook**
3. **WSRC-NB-99-, CIM5 Laboratory Notebook**
4. **T. M. Jones and D. C. Witt, CIM5 Phase III –Run #38 – 49 wt% Ln 25SrABS From CP-748 with ¼” Marbles, SRT-AMC-99-0125, June 9,1999.**
5. **T. M. Jones and D. C. Witt, CIM5 Phase III –Run #39 – 49 wt% Ln 25SrABS From CP-749 with ¼” Marbles, SRT-AMC-99-0126, June 10,1999**

ATTACHMENT 1 – DTTS EVALUATION OF GLASS BEADS

Results of Run #6 (Phase II) – 1/4" Glass Beads in the DTTS

A run batched with the 1/4" 25SrABS glass beads and wet oxalate slurry was completed on 10/8/98. The objective of this run was to determine the impact of a size specific 25SrABS glass on high temperature volume expansion due to cerium reduction. The initial height of the entire batch (marbles and oxalate slurry) in the melter was measured to be ~8", approximately 1" less than when batched with cullet. The DTTS batch was dried and heated to a bed temperature of approximately 1146°C before any notable differences were observed. At this temperature, a few larger bubbles were observed at the bed surface, evolving into many smaller bubbles (Ce reduction). However, through the remaining 30 minutes of ramping to the target bed temperature of 1390°C, the volume expansion continued to increase until the entire free board volume of the melter vessel was filled with small frothy bubbles. The frothy layer was reincorporated into the melt after approximately 1/2 hour. The batch was drained with a continuous stream at a flowrate of ~16 kg/hr. Further testing with glass beads is planned to better understand the observations from the DTTS run.

Results of Run #7 (Phase II) – 3/8" Glass Beads in the DTTS

A run using 3/8" glass beads in place of cullet was completed on 10/16/98. The initial height of the batch (marbles and oxalate slurry) in the melter was measured to be about 8", approximately 1" less than when batched with cullet, as was observed in the previous glass bead run. The change in total batch volume was due to deeper penetration of the oxalate slurry into the glass bead bed. During the initial boiling of the free water, the surface activity appeared to be more violent and erratic than with cullet, at times splattering small amounts of material onto the upper vessel flange. Power was manually backed down to reduce the surface activity and splattering. This violent boiling is uncharacteristic of similar batch runs using cullet, and because the power had to be backed down, the removal of the free water (excess oxalic) took approximately twice as long. Once the free liquid was gone, the batch continued through the drying phase without incident. At a temperature of ~1170°C, a sudden increase in the rate of glass temperature rise was observed, indicating bubbling or frothing of the melt. Observation of the bed revealed that indeed a frothy, foamy layer of small bubbles had risen about 1 to 2" from the previously observed level in the melter (Ce reduction). The volume continued to increase to within approximately 3" of the melter top (approximately 4X increase), when a small opening in the bed surface emerged. The small opening revealed a large void beneath the frothy surface. The surface layer melted back into the glass pool in about 20 minutes. The batch was drained in a continuous stream at flowrates of ~16 kg/hr.

ATTACHMENT 1 (continued) - DTTS EVALUATION OF GLASS BEADS

Results of Run #9 (Phase II) – Mixed (1/4" & 3/8") Glass Beads in the DTTS

A run was completed in the DTTS on 10/30/98 which incorporated an equal mixture of 3/8" and 1/4" 25SrABS glass beads and wet oxalate slurry. This run was designed to examine the effects of tighter packing of the glass beads on the high temperature volume expansion that has been notably more severe when using glass beads. As the bed temperature approached 1160°C, the bed surface began to show evidence of small frothy bubbles at the outer edge. The surface then began to rise and continued to gradually increase in height over the next 30 minutes until the volume expansion was at the top of the melter vessel. The volume expansion surface layer was probed and found to be thin and soft, with the volume underneath the surface layer void of bubbles. The surface layer of the volume expansion was pushed back into the melt pool and after 50 minutes showed no signs of residual bubbles in the melt pool. The batch was then poured at 12-14 kg/hr.

The volume expansion observed with the beads was initiated at lower temperatures than previously experienced with either cullet or frit. The thin, very gradual volume expansions noted when using glass beads may be the end effect of the enhanced melting characteristics of glass beads, coupled with enhanced fluxing by the lanthanides in the surrogate. Glass beads (marbles) are used in the glass industry to decrease the time required to melt and this effect may be contributing to the early volume expansions seen with the beads.

ATTACHMENT 2 – AUTHORIZATION REQUEST APPROVAL FOR TASK 1.02

**WESTINGHOUSE SAVANNAH RIVER COMPANY
SAVANNAH RIVER TECHNOLOGY CENTER**

SRT-AMC-99-0124

June 8, 1999

To: S. Wood, 773-A

From: L. F. Landow, 704-1T



**REQUEST FOR AUTHORIZATION TO INITIATE A TASK
PRIOR TO HAVING AN APPROVED TASK TECHNICAL PLAN**

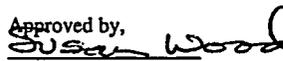
SRTC's Immobilization Technology Section has received a Technical Task Request (TTR) from NMSS to execute 34 additional Americium-Curium development tasks at the Am/Cm Pilot Facility located in Building 672-T. One of these tasks requests evaluating the effects on the nominal Cylindrical Induction heated Melter (CIM) processing characteristics if we were to use glass former beads rather than cullet.

It will be several weeks before a Technical Task and QA Plan is developed and the many, required approvals obtained. Authorization to perform this specific task prior to having an approved Task/QA Plan is requested for the following reasons:

- We will need a great deal more cullet to perform all the TTR tasks requested. Currently, we have on hand sufficient cullet (~40 lbs) to perform only 14 additional melter runs in the 5-inch CIM.
- We currently have about 200 lbs of the glass former beads. If these beads were to prove to be a viable alternative to cullet, we would have a reasonable supply of glass formers to initiate the other tasks while allowing us ample time to procure the additional supply of beads needed to complete the TTR tasks.
- If the use of glass former beads in lieu of cullet proves unsatisfactory, we can then immediately begin to melt the beads in the 5-inch CIM and Drain Tube Test Stand (DTTS) to produce the additional cullet we need to complete the tasks.

This task will be performed in compliance with all existing operational procedures, quality assurance requirements and Safe Practice/Personal Protective requirements.

Approved by,



S. Wood, SRTC Laboratory Director

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