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Executive Summary

This report describes work on Rapid Conditioning for the Next Generation Melting System under US Department of Energy Contract DE-FC36-06GO16010. The project lead was the Gas Technology Institute (GTI). Partners included Owens Corning and Johns Manville. Cost share for this project was provided by NYSERDA (the New York State Energy Research and Development Authority), Owens Corning, Johns Manville, Owens Illinois, and the US natural gas industry through GTI's SMP and UTD programs. The overreaching focus of this project was to study and develop rapid refining approaches for segmented glass manufacturing processes using high-intensity melters such as the submerged combustion melter. The objectives of this project were to 1) test and evaluate the most promising approaches to rapidly condition the homogeneous glass produced from the submerged combustion melter, and 2) to design a pilot-scale NGMS system for fiberglass recycle.

During the course of this lengthy project, the project team examined a number of potential rapid refining approaches. Several of the planned approaches were abandoned upon investigation. Other methods were studied mathematically, through modeling, and through laboratory testing. Still other methods were conceived during the course of the project and were subsequently investigated. Dismissed rapid refining approaches included steam (insufficient theoretical support) and sonic (ultrasonic horns that can survive in glass contact are not available). Rapid refining approaches that were investigated included:

- Short residence time
- Centrifugal
- Vacuum
- Filtration
- Obstructed flow
- Thermal convective

Results are presented in this report for each of these refining methods. A US patent (7,874,179) was obtained by GTI for filtration refining, and a patent application (2012/0210751) was submitted for thermal convective rapid refining (TCRR). Each of the studied refining approaches was found to provide means to refining viscous molten glass. At the conclusion of the project, GTI engineers believed the TCRR approach was the most promising of the approaches studied. One perspective identified by the team is a two-step refining process. The needed quality of glass varies by orders of magnitude between products. For some products a single 'coarse' refining step may be sufficient, while other glass products may require a second, 'fine' refining step to meet target quality.

Work in this project and related projects that served as cost share studied a number of other aspects of the Next Generation Melting System. These areas of study included:

- A new, submerged combustion burner with a different design
- An optical sensor for real-time monitoring of the active surface of molten glass in the SCM
- A graphite tap to replace the current costly platinum tap for discharging molten glass from a submerged combustion melter
- A preheated air test to collect data over a wider SCM operating window

- A techno-economic design of a commercial-scale SCM plant melting fiberglass from scrap or raw materials

Some of the work in these areas of investigation was performed under coordinated projects. Results are presented in this report to provide a complete description of all related Next Generation Melting System project activities in the single document.

1. Project Goals/Objective

The objectives of this project were to 1) test and evaluate the most promising approaches for rapidly conditioning the homogeneous glass produced from the submerged combustion melter to serve as a second step in a Next Generation Glass Melting System (NGMS), and 2) to design a pilot-scale NGMS system for fiberglass recycle. Techniques to produce higher quality glass from the melter were to be investigated, and external fast conditioning approaches were to be analyzed. The add-on rapid conditioning techniques evaluated through modeling and limited testing were expected initially to rely on sonic energy, steam, and time discharge to speed conditioning

During the course of the project, an important change was made in the project objectives. The project team is still considering multiple rapid refining approaches. However, work in Budget Period One led to re-consideration of the actual approaches. The team concluded that:

- Short residence time refining is still the primary focus for rapid refining
- Sonic refining has been replaced with a filtration refining approach
- Steam refining has been replaced with a low-momentum centrifugal rapid refining method.

The reason for replacement of sonic refining was that no technology or materials are currently available to produce the sonic effect in long-term industrial-scale melters. Filtration was substituted because testing has shown that bubbles can be physically removed in this manner. A patent was submitted for filtration refining as a result of project work. Steam refining was discontinued because there was no modeling evidence to show that additional steam in addition to the steam produced by SCM combustion will have any large effect on refining. A third, speculative refining approach, based on centrifugal action was substituted. High speed centrifuges operating at over 1000 rpm have been shown to remove bubbles from molten glasses, but these units are difficult to operate. Analyses of early publications have shown that a much slower centrifuge speed driven solely by motion of the molten glass can produce a rapid refining action by increasing the g forces on the molten glass.

A final objective that was added for Budget Period Two is the development of a more robust, next generation burner for the SCM. The burner is the most critical SCM component. Burners must operate continuously and reliably for a minimum of one year. A new design was developed by GTI, and this more robust burner will be fabricated and tested during pilot-scale rapid refining tests.

Funds for this project were made available at several different times. This caused project delays while waiting for further funding. Overall, the total DOE funds in the original contract for made available. The find contract action was taken at the end of 2012. At that time the final $195,000 was made available. The final contract action specified completion of the rapid refining tests. This funding supported work described in this report in the sections of Batch TCRR Testing and Continuous TCRR Testing.

2. Background

Glass formation places heavy demands on the glass melting process. Quality requirements vary radically between industry segments, but all forming processes need a steady flow of molten, homogeneous glass of the chosen color (if not clear), with few to no unmelted sand grains (stones). This molten glass must be at a specific viscosity to form into products, with

means temperature must usually be controlled to within several degrees. Along with these requirements, each industry segment specifies upper limits on the number and size of bubbles (seeds) in the molten glass used to form their products. In molten glass to form most products, seeds must be below 0.1 mm in diameter, although optical glass demands are even stricter.

The U.S. Department of Energy is supporting collaborative efforts to develop a Next Generation Melting System (NGMS) that will produce glass of needed quality to the forming step of each glass industry segment. The submerged combustion melting technology, licensed and controlled by the Gas Technology Institute (GTI), is currently being developed by a GTI-glass industry consortium to serve as the melting and homogenization step of the NGMS process. NGMS envisions a segmented melting approach to achieve high energy efficiency, low emissions, and low capital cost. These goals are met by optimizing components of the melting process. SCM is particularly attractive because homogeneous, stone-free glass is produced while feed handling is less costly and simpler than for conventional furnaces, energy use is decreased, and emissions are lower, all with a far less expensive melter.

In SCM (shown below), fuel and oxidant are fired directly into the molten bath from burners attached to the bottom of the melt chamber. High-temperature bubbling combustion inside the melt creates complex gas-liquid interaction and a large heat transfer surface. This significantly intensifies heat exchange between combustion products and processed material while lowering the average combustion temperature. Intense mixing increases the speed of melting, promotes reactant contact and chemical reaction rates, and improves the homogeneity of the glass melt product. The melter can handle a relatively non-homogeneous batch material. The size, physical structure, and especially homogeneity of the batch do not require strict control. Batch components can be charged premixed or separately, continuously or in portions.

The many features and requirements for SCM have been discussed elsewhere and will not be covered in detail here. The technology requires careful and appropriate design of combustion systems, and GTI has obtained a cornerstone patent in this area (US 7,273,583). Oxy-gas firing is imperative so that heat input can be maximized while nitrogen bubbles are eliminated and total gas volume is minimized. Refractory walls would be worn away by moving glass in an SCM, so walls are built from externally cooled panels on which a frozen glass layer forms to protect the wall. GTI and a glass company consortium (Corning, Johns Manville, Owens Corning, PPG, and Schott) have been developing SCM for use as the NGMS melting and homogenization step. Soda-lime, E glass, and many other compositions have been melted to date in a lab-scale and a 1 ton/h pilot-scale SCM unit (shown below) at GTI. Construction of the pilot-scale SCM was supported by an earlier DOE project

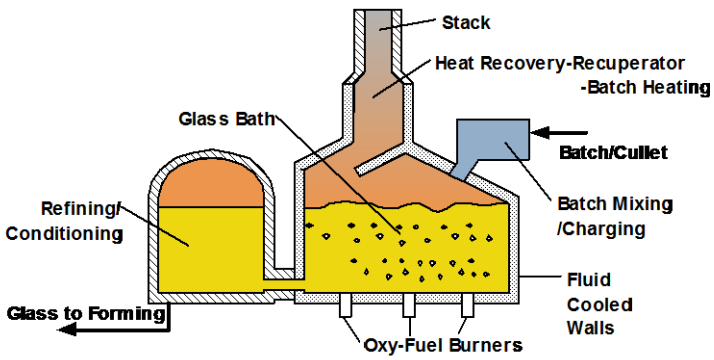
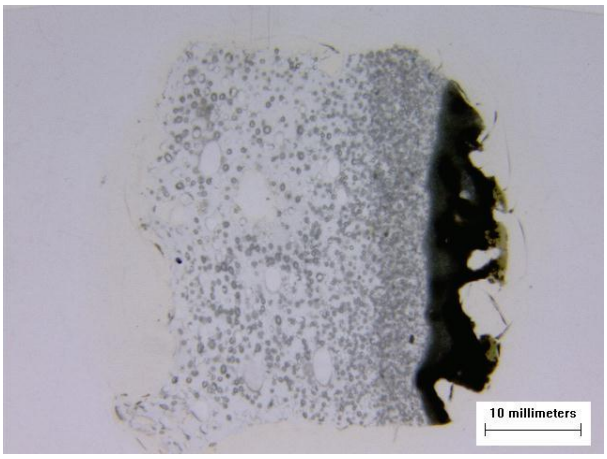


Figure 1. Submerged Combustion Melter Concept and Pilot-Scale SCM

Operation of SCM with a range of industrial glass compositions from soda-lime glass to LCD glass has verified the melter's capacity to generate homogeneous, stone-free glass. Photomicrographs of soda-lime glass produced in the pilot SCM unit are shown below.



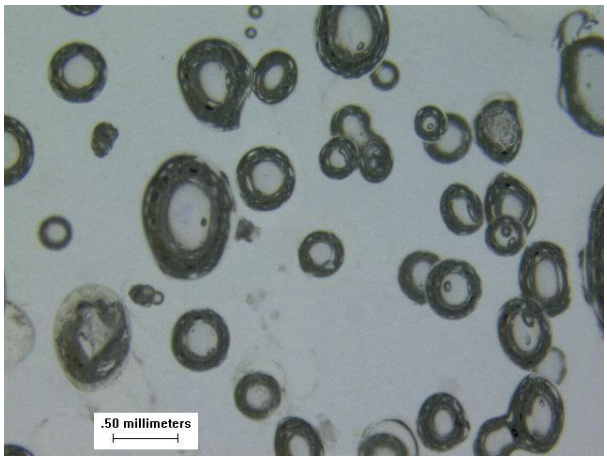


Figure 2. Bubbles Present in SCM Product Glass

Results with SCM are highly encouraging. The product glass, however, must be of higher quality, with far fewer bubbles, to meet many industrial needs. There are multiple means to improve the glass quality, and this project focused on improving the quality of SCM product glass. Many of the techniques evaluated, particularly the external conditioning methods, are also applicable to conventional glass melters, as indicated above.

SCM Operations Improvements to Improve Glass Quality

SCM glass quality can be improved through a number of melter improvements, some in components and some in operating techniques. Component improvements include

- feeding systems that control batch introduction and minimize particle and volatile discharge
- modified burners and combustion systems to give desired combustion and heat transfer patterns to generate large easily refined bubbles
- Optimum discharge taps located to discharge high quality glass and to control melt flow rate

Improved operating procedures include burner locations and firing patterns, temperature control, residence time control, and control of melter mixing patterns. All of these techniques have potential to increase the size of bubbles, decrease bubble concentration, and eliminate small bubbles (below 0.5 mm).

External Conditioning Approaches

Along with improved SCM operation, external, rapid conditioning methods can improve glass quality. Glass refining is made difficult by the small size of bubbles (0.1-2.5 mm) that must be removed, the high viscosity of molten glass (often 100-150 poise), and limitations on materials chemically and physically compatible with molten glass. The upward velocity of small bubbles in a liquid is characterized by Stokes Law.

$$V \ \alpha \ d^2 g \rho / \mu$$

which states a bubble's upward velocity (V) is proportional to the square of the bubble diameter (d), the gravitational constant (g), and to glass density (ρ) and inversely proportional to the viscosity of the glass (μ). All refining approaches modify one or more variables to increase V. Refining time is controlled by the time it takes the smallest bubbles to reach the glass surface. Gas diffusion rates through glass vary, with nitrogen and carbon dioxide being the most difficult to remove. Current practice relies on time and chemical fining agents. Fining agents release gases such as oxygen that combine with bubbles of other gases. This produces larger, faster diffusing bubbles that shorten the refining time but also can contribute to air pollution. Fining agents are expensive and can alter glass chemistry, so their application is limited. Time can be turned into a refining ally by using a thin film or 'fining shelf' that shortens bubble distance to rise. Fining shelves are employed in some furnace designs, but care must be taken to maintain temperature uniformity and to minimize heat loss from the large surface of molten glass.

The NGMS process requires rapid conditioning to produce high quality glass from the high-intensity SCM unit. Each rapid refining approach (with the exception of filtration) takes advantage of one or more variables in Stokes Law to accelerate the conditioning process. A list of the rapid conditioning approaches considered, modeled, or analytically tested in this project is shown in the table below.

Table 1. Rapid Refining Approaches Evaluated

Rapid Conditioning	**Basis for Accelerated Conditioning**	**Project Action**
Temperature/time	Lower viscosity and shorter distance for bubbles to travel, combined with no recycle of clarified glass can decrease refining time	Tested in pilot-scale SCM with batch samples and in a refining channel
Helium (inert gas)	High solubility and high diffusion leads to scavenging of small bubbles into larger	Not studied because no approaches are known for

	bubbles and fast bubble rise to surface (d_b is larger)	recovery and recycle of helium
Water (steam)	High solubility and high diffusion leads to scavenging of small bubbles into larger bubbles and fast bubble rise to surface (d_b is larger)	Not studied because no evidence was found indicating benefits of additional steam on refining
Sonic	Vibrations lead to bubble consolidation and faster rise to the surface	Not studied because no horn material compatible with molten glass is known
Vacuum	Decrease pressure leads to bubble expansion and fast rise to the surface	Studied with silicone oil but abandoned because of low glass company interest
Centrifugal	Gravitational constant changed from g to $r\omega^2$ which can greatly increase velocity	Studied at 600 and 900 rpm with simulated fluids but abandoned because of low glass company interest
Filtration	Bubbles attach to the surfaces of fine particles in a filtration bed. Coalescence of small bubbles into large, easily refined bubbles leads to clarified glass after slow passage through filter media	Studied and patented. Determined to be of limited value because flow rates must be extremely low, making equipment too large and costly
Obstructed path	Variation of time/temperature refining in which weirs force molten glass in a channel to travel in vertical as well as horizontal flow along a channel	Studied with silicone oils. Abandoned when testing found no benefits of weirs
Thermal convective	Improvement to time/temperature refining in which precise temperature fields are created in the refiner enhance convective flow of bubbles toward the surface	CFD modeling, batch and continuous TCRR testing with molten glass

3. Accomplishments

This project was carried out by a project team led by the Gas Technology Institute (GTI). Team members included Owens Corning, Johns Manville, Owens Illinois, and the Missouri University of Science and Technology. Several projects supported specific Tasks in this project and also carried out work in other areas of interest to the Next Generation Melting System (NGMS). The NGMS was assumed to consist of a segmented glass melting process with the submerged combustion melter (SCM) as the melting and homogenizing step. Earlier projects, some supported by the US Department of Energy, focused on development of a pilot-scale SCM. That earlier work will not be reviewed in this report. This report, however, does include the results of all projects providing cost share to the DOE rapid refining project. Some of this material was outside the immediate scope of the DOE Statement of Project Objectives, but all

work supported the NGMS development. These other results are presented here to provide the broadest, most complete possible information on developments toward an industrial NGMS process for glass production.

The multiple topics described in this report are organized into related groupings and are not presented chronologically. Work on refining processes began with investigation of time/temperature refining and progressing through the other approaches described below. The final refining work explored batch and finally continuous thermal convective rapid refining. Work on other topics was carried out in parallel to the refining efforts throughout the period of the DOE project. Descriptions of those other efforts and the results of the studies are presented after the discussions of the refining approaches.

Rapid Refining Approaches

A total of nine rapid refining approaches were studied. All investigations assumed the inlet molten glass was from a continuous SCM unit. The glass was assumed to be at the desired temperature and flow rate for forming, homogeneous, and containing bubbles accounting for approximately 25 percent of the glass volume. In some cases this large number of bubbles presented difficulties for a particular refining method, and in some cases the large bubble content proved to be an advantage for a refining method.

The refining approaches evaluated changed over the course of the project. Three approaches were determined to be impractical and were not studied further. Helium refining was deemed to have operational potential. But the amount of needed helium and the inability to identify a means to capture 100 percent of the helium from the exhaust gas made this approach appear impractical on a cost basis. Water (or steam) refining was dismissed because the team found no evidence that additional water in the melter would have any impact on the refining of molten glass. Sonic refining has a sound theoretical basis and has been proposed by past researchers. The project team was forced to dismiss consideration of sonic refining because no material could be identified that will survive as a horn in contact with molten glass for an extended period of time. After dismissal of these three approaches, six other rapid refining approaches were studied in this project.

Temperature/Time Refining

Clarification of a viscous liquid such as molten glass is easily accomplished if the liquid is held in place for a long enough period of time. Temperature-time refining of molten glass can take from several hours to several days depending on the glass viscosity and the level of larity demanded. Rapid refining with a goal of meeting quality targets such as fiberglass or container glass in under 30 minutes will require more aggressive engineered solutions than allowing time to solve the refining problem. To obtain an important baseline understanding of SCM product glass behavior, the initial refining study in this project was of temperature-time refining.

The first effort was to conduct a pilot-scale SCM test and to then refine samples under controlled conditions in a high-temperature electric oven. This work was needed to provide baseline information for SCM refining requirements and to determine the quality of SCM product glass after initial refining.

A pilot-scale SCM test was conducted with borosilicate glass batch. This E glass is a commercial product used for reinforcement fiber. In this test approximately 1000 lb of glass was melted and held at temperature in the pilot-scale SCM unit. Over the next several hours samples

were collected in preheated ceramic crucibles and placed in an electric oven for various periods of time up to 120 min. with the electric oven varied between 1250º and 1450ºC. The photos below show the set-up used and the careful collection and movement of samples. Note that a second electric oven at 800ºC was used for annealing. All samples were first held at refining conditions and then placed in the annealing oven for at least 60 minutes or placed directly into the annealing oven without any refining.

Figure 3. Electric Ovens and Sample Placement for Refining and Annealing Batch Samples

All samples were collected after annealing or refining/annealing and sent to Johns Manville for analysis. Each crucible was cut vertically in half on the centerline and some refined samples were sectioned for closer analysis. Results confirmed that bubbles do rise with time and that the rate of rise correlates with refining temperature. This data has provided the project team with baseline data on the rate of bubble rise as a function of temperature for this glass. Results were encouraging, with glass after refining proving both homogeneous and seed-free.

During the borosilicate melt, a series of gas samples were collected and submitted for gas chromatography analysis. Results showed that the majority of the gas on a dry basis was carbon dioxide with some oxygen (since excess oxygen was used at an oxygen to natural gas ratio of 2.15) and with some nitrogen. The nitrogen is most likely from the 1 to 3 percent nitrogen in the natural gas supplied to the GTI lab. No significant levels of CO or unburned hydrocarbons were detected. Gas samples contained 100 to 150 vppm of NO_x. This corresponds to a NO_x level of 0.11 to 0.16 lb NO_x per ton of glass. This level is significantly lower than the best oxy-gas melters which produce approximately 0.5 lb NO_x per ton. The low level of observed NO_x is expected because the flame is rapidly quenched to a lower temperature in the melt. This is an advantage of SCM over conventional furnaces.

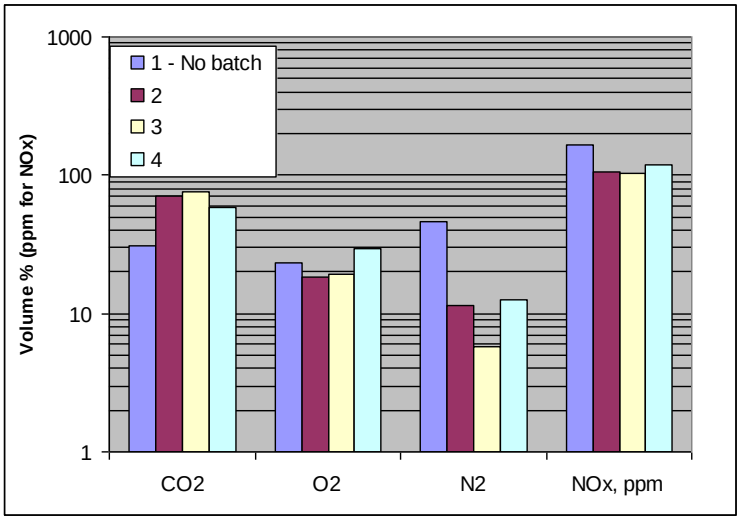


Figure 4. SCM Gas Analyses During Steady-State Melting of Borosilicate E Glass

Testing in the new pilot-scale melter for the previous project was completed in the (April through June, 2007 quarter. During that time, the project team began preparations for the rapid refining work in this project. Since two methods of refining may have been studied separately and together, a short refining section made from refractory brick was designed to allow for residence times of product glass up to 30 minutes. This section was to be attached to the melter outlet.

A photograph of the assembled melter before adding a channel at the front end is shown below along with a photograph of melt discharge from the pilot-scale SCM. The melter was erected and positioned in a test cell in the GTI industrial combustion laboratory.

Figure 5. Pilot-Scale SCM Before Adding Refining Channel

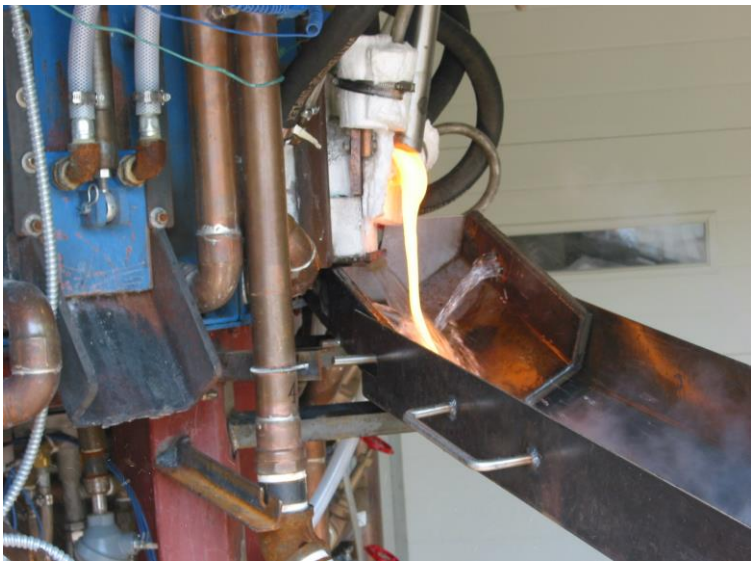
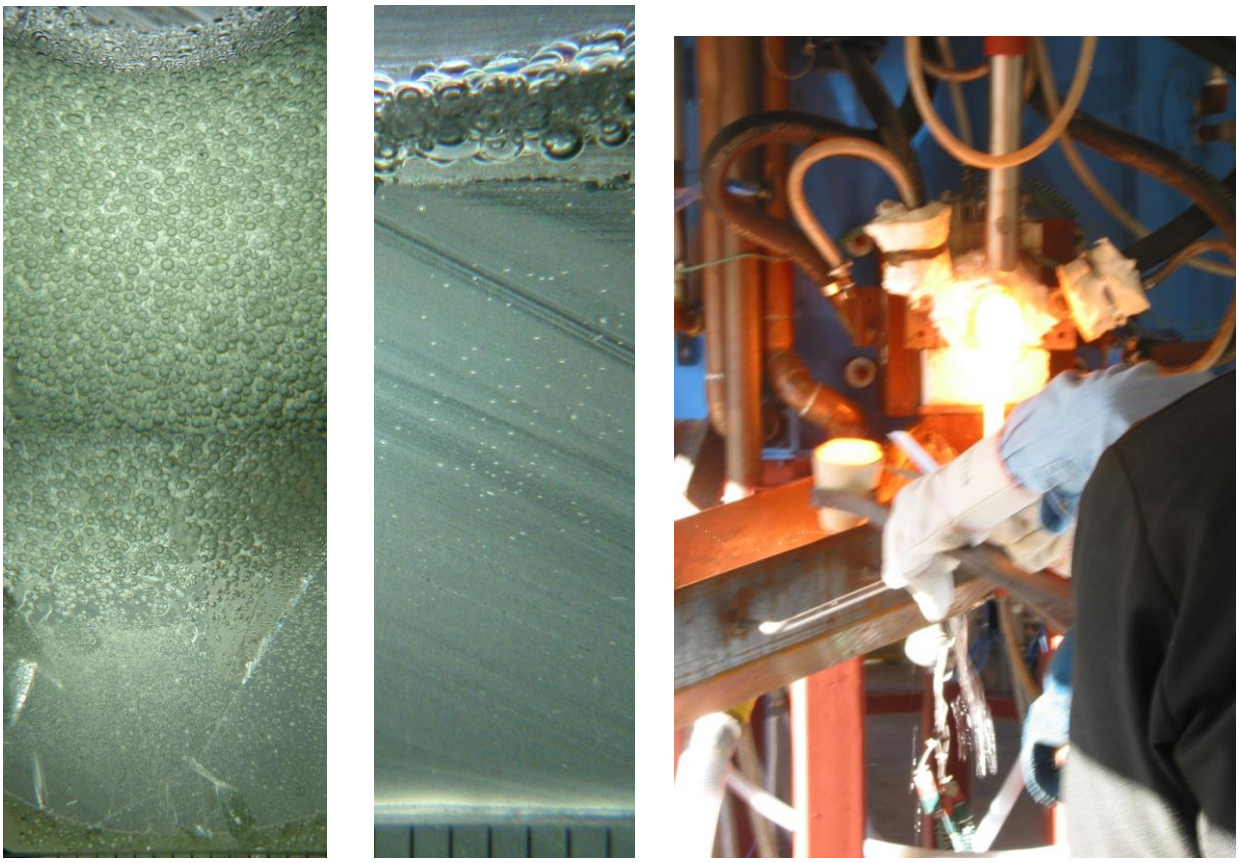


Figure 6. Typical Melt Discharge From Pilot-Scale SCM

A design concept for a temperature-time refining channel was developed and discussed among the project participants. Fabrication approaches were evaluated, and a final detailed design was approved. All needed refractories, heating elements, and steel frames and supports were ordered. The channel was to be fabricated and prepared for testing with molten E glass

starting in July, 2008. The channel was built from standard high-temperature bricks and used high temperature electric elements to control channel temperatures. Residence times was to be controlled by the SCM pull rates. One challenge is to devise a reliable means to continuously discharge molten glass from the SCM and to introduce the molten glass at high temperature into the refining channel. This challenge was to be met by using a diverter plate. The diverter plate can discharge glass into a discharge channel until a stable stream of molten glass is established. Then the diverter plate can be moved so that the glass falls as a stable molten stream into the front end channel.

The final glass product was removed from the bottom half of the channel by using a skimmer block. The level in the channel was controlled automatically by glass discharge by overflow after the skimmer block. The team originally planned to use a heated bushing to control glass flow out of the channel, but this was deemed unnecessary when the overflow of glass into an oversized exit port was devised. The major criteria for the exit flow is to have sufficient heat to avoid freezing. This was achieved by using additional electric heating elements placed on the walls and roof after the skimmer block at the end of the channel.



(a) (b) (c)

Figure 7. Images of E Glass Held at 1350 C for (a) 20 min and (b) 60 min. Image of (c) sample collection from SCM discharge tap

In summary, the forehearth channel sits on a steel frame and can be moved in and out of position relative to the melter. The forehearth is built of three layers of refractory selected for

their high temperature thermal properties. An outside layer of high-temperature refractory board is also included. The foreharth section internal dimensions are approximately 12 inches wide by 72 inches long. Glass depth can vary and is initially set for 6 inches. The lid contains the inlet channel from the melter. A total of 24 electric elements are mounted horizontally in the lid and are operated in three groups (of six, twelve, and six elements). Nine SCRs supply three-phase 480V power to the elements, and a transformer is connected to the SCRs.

A photograph of this completed device is presented below. Note that the forehearth section is mounted on rails to facilitate easy movement of the device up to and away from the melter.

The first continuous SCM-refining test was conducted with a non-boron E glass. In this test, molten E glass was continuously fed to the forehearth at different flow rates. These varying flow rates were supposed to produce refiner residence times for the glass of 20 to 60 minutes. Glass samples were collected from both before and after the refiner during each steady state period. These glass samples were vertically sectioned and analyzed.

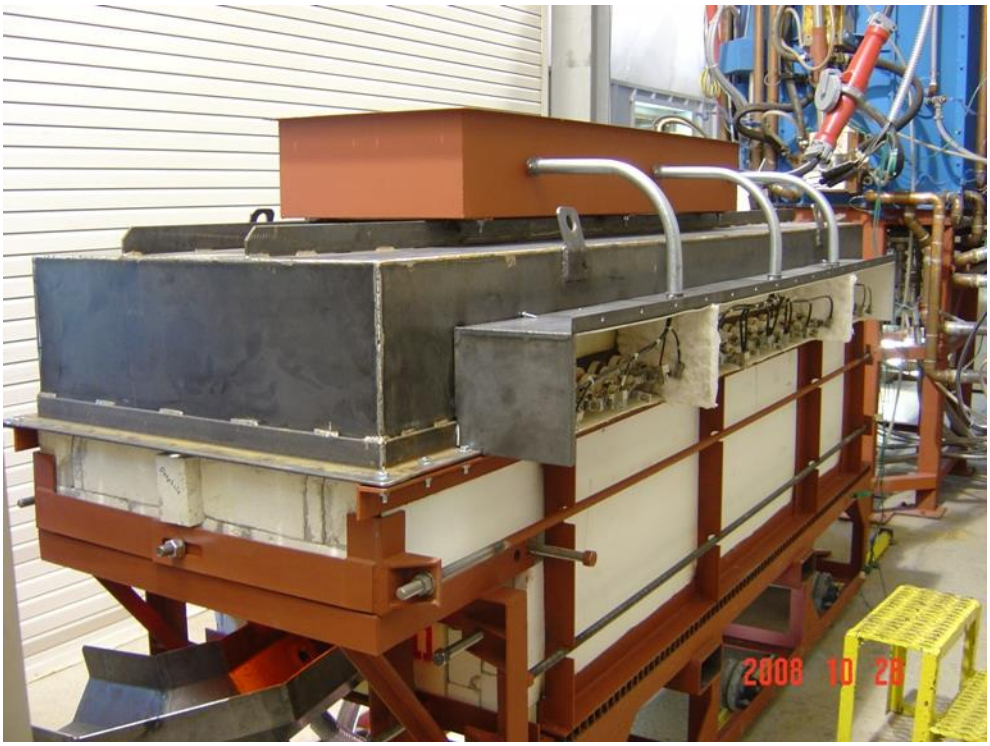
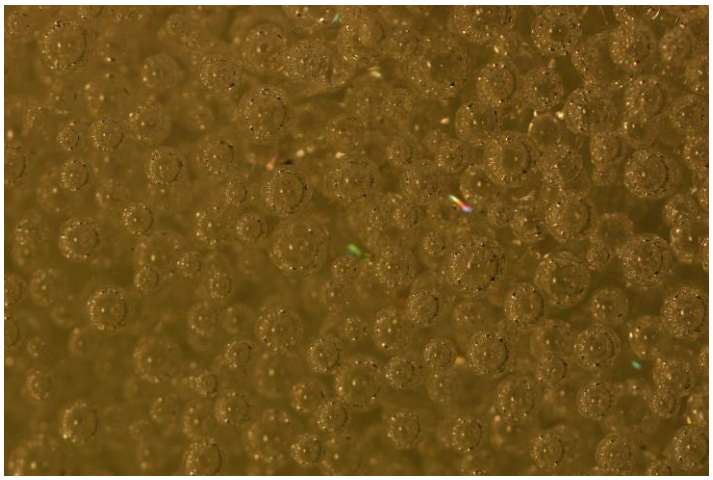


Figure 8. Refining Channel With Electric Elements Above the Glass

The test did not proceed as planned. The glass passed through the refining section much faster than planned, and the glass exiting the refining section did not show a significant decrease in bubbles. Review of the test revealed that the glass entering the refining section was too cold to flow uniformly. This led to the refining section filling with glass to the discharge level. Then the next glass charged 'floated' on top of the highly viscous glass already in the refining section. This process short circuited the refining step. The pictures below are extreme close-ups of the glass removed from the refining section after the test. The bottom sample indicates that refining

began, but that the glass was too cold and viscous to fully refine. The glass surface sample shows that bubbles do rise to the surface and break there.

Bottom of Refining Section



Glass at Surface of Glass in Refining Section

Figure 9. Glass at Bottom and Top of Refining Channel

While the first effort to conduct continuous refining was not successful, the project team sought ways to address the problems encountered. The first thing the team did was to heat the glass left in the refining section and let that glass refining for two days. The photograph below shows that this operation demonstrated that glass will definitely refine in this unit. After this was shown, the glass was emptied from the refining section in preparation for modifications and further testing.

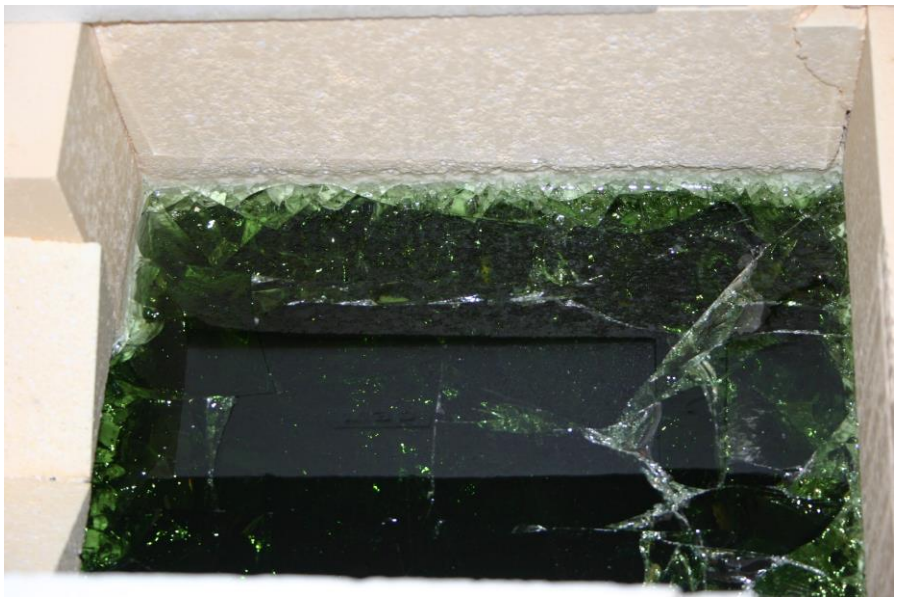


Figure 10. SCM-Melted E Glass in Refining Section After Two Days at Temperature

The project team felt the first test went well operationally, but the results were poor since the glass was not hot enough either entering or exiting the refining section. The project team, with assistance of Owens Corning engineers, has designed a ceramic feed chute to link the SCM and the refining section more closely. This component will help keep the glass hot and at the desired low viscosity entering the refining section.

The project team also is planning to brick in the discharge to the refining section. This will significantly reduce the amount of cold air induced into the refining section, and will help to decrease cooling of the product glass before that glass is removed from the refining section.

A third improvement employed was to install a flame at the discharge end of the refining section to maintain heat at the point of glass discharge. The flame is produced by a long welding torch type heater that is inserted through the bricks at the discharge end of the refining section.

All of these changes regarding the refining step of the glass-making process were installed before conducting the next overall continuous SCM-refining test. The goal was to improve the SCM to refining section glass transfer and to operate the refining section at a high enough temperature so that the desired 20 minute refining residence time under continuous operation could be achieved.

Another SCM test was then conducted (June 2009). For this teat, the glass was changed to a standard boron-containing E glass supplied as batch from Johns Manville. The test went smoothly. SCM temperatures were controlled at desired temperatures and varied during the test. The connection between SCM and refining section appeared adequate to keep the glass hot enough to prevent layering and bypass in the refining section. The refining section worked well for a number of hours. A photograph of the test in progress with molten glass being steadily discharged from the refining section is shown below.

Figure 11. Time-Temperature Refining Showing Melt Discharge From Channel

Two steady-state periods were established and maintained. The temperature was higher in period two than in period one. In both periods the residence time in the refining section was approximately 20 minutes. Visual inspection of glass samples from before and after the refining section showed that a large fraction of the bubbles were removed. Void fraction analyses confirmed the visual observations as indicated below.

Table 2. Time-Temperature Refining Results

	Void fraction in glass after SCM, %	Void fraction in glass after refining section, %
Stead-state 1 – lower temperature	32	23 to 28
Steady-state 2 – higher temperature	21	6 to 24

Gas samples were collected during this test. Analyses found CO and unburned hydrocarbons to be below the detectable limit. The amount of oxygen present was higher than expected, and the mass flow meters will be calibrated to confirm flows of oxygen and natural gas are at the desired levels.

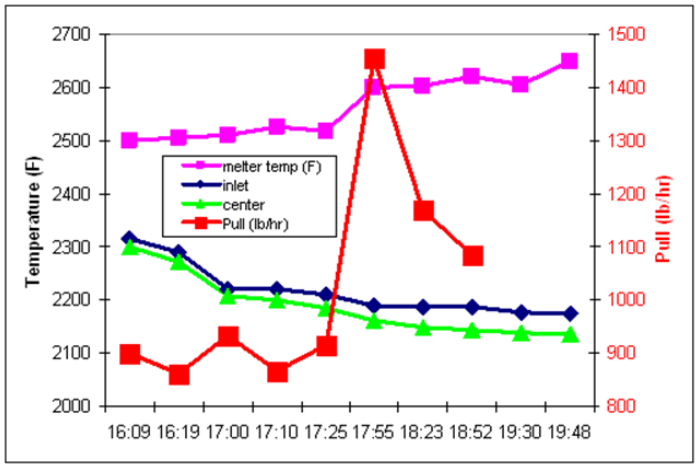


Figure 12. Measured Temperatures and Glass Pull Rates During Periods One and Two of the SCM Refining Test Conducted on 6/30/09

Visual inspection of glass samples from before and after the refining section showed that a large fraction of the bubbles were removed. Void fraction analyses confirmed the visual observations as indicated below. The first Figure provides a guide to the testing apparatus configuration and sample collection points. The Table and second Figure below provide details on the levels of refining achieved. Note that the second tap sample could be inaccurate because the sample was collected after the end of steady-state operations. Also note that the level of refining achieved in period two was much greater than in period one. This could be due to higher glass temperatures in the melter and channel in period two and to an increase of 10 to 20 percent in channel residence time during period two. Further SCM-refining testing will involve testing with more detailed analysis of the variables influencing the level of refining.

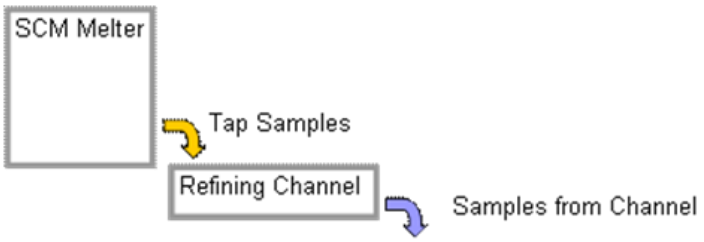


Figure 13. Layout of SCM-Conditioning Channel Refining Tests Showing Sampling Locations

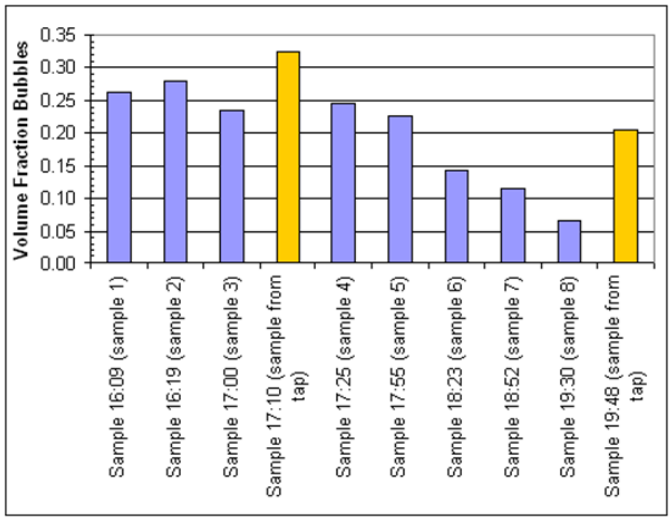
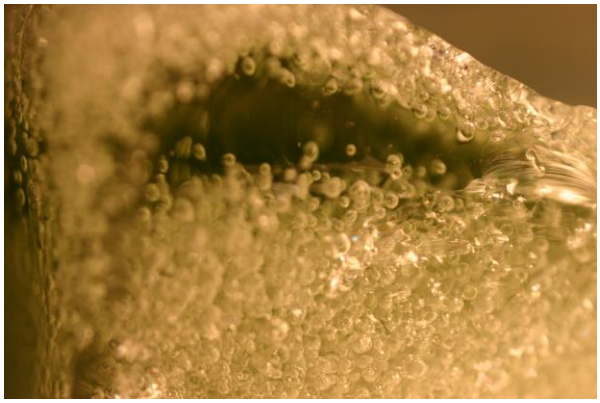


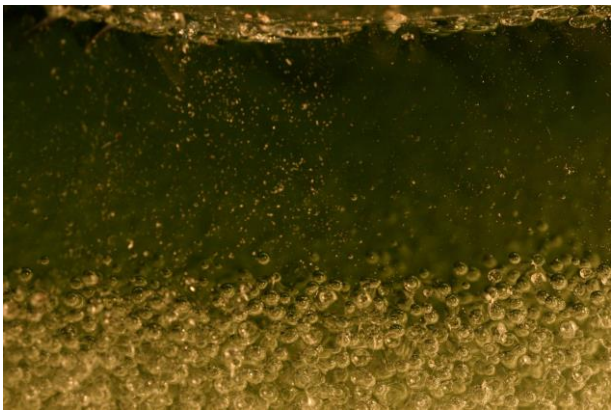
Figure 14. Void Fraction of Glass Samples From SCM Tap and From Conditioning Channel Collected During Periods One and Two of the SCM-Refining Test of 6/30/2009

Close-up photographs were taken of tap and channel glass samples collected during the 6/20/2009 test. Some of those close-up photos are presented below. The pictures were all prepared using the same level of magnification show that the SCM product glass from the melter is homogeneous. As the glass begins to refine, the number of bubbles declines, the bubbles coalesce into larger babbles, and portions of the glass become much clearer than other portions. Future refining tests with longer residence times and controlled temperature variations will study the behavior of bubbles more closely.

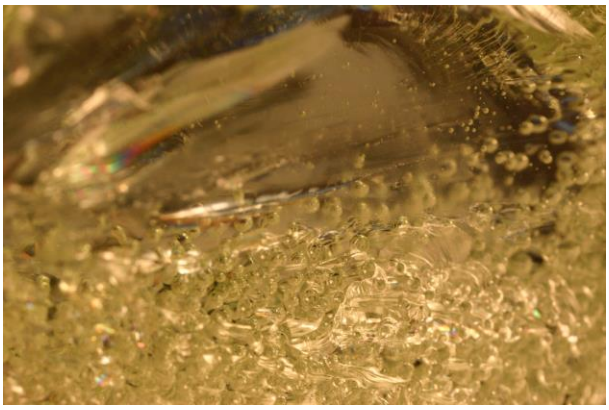
SCM Tap Sample 2



Refining Channel Sample 4



Refining Channel Sample 5



Refining Channel Sample 7

Figure 15. Tap and Refining Channel Close-Up Glass Sample Photographs

Another continuous SCM-refining test was conducted on September 30. Detailed results of this test will be assembled early next quarter and presented in the next quarterly Technical Progress Report. Only the test conditions and observations from this latest test are presented in this report.

The 9/30/09 test was conducted using the same E glass batch material as the 6/30/09 test. The 9/30 test was conducted with a single SCM and channel temperature. The SCM temperature was held at 2600F to duplicated the conditions of Period Two of the 6/30 test. The desire in the latest test was to have tighter operating control and to achieve a wider range of residence times in the conditioning channel. To achieve more accurate melt flow control from the SCM into the conditioning channel a motor controlled gate was fabricated. This gate, shown in the photograph below, is made of mullite and kept hot so glass does not freeze on the inside surface of the gate and prevent vertical adjustment to mediate glass flow rate.

Figure 16. Glass Entering Refining Channel From the Pilot-Scale SCM

The gate worked well during the test. External heating with a torch allowed the gate to slide freely as needed. Melt flow rates from the SCM were controlled over the range of 350 to 1200 pounds per hour. This allowed the residence time in the conditioning channel to be varied by a factor of approximately 3.5.

Temperatures were controlled in the SCM to within 25ºF throughout the test, and similar levels of temperature controlled were achieved in the refining channel. Channel discharge temperature measured by optical pyrometer was approximately 2470ºF throughout the testing period, over the full range of residence times achieved.

Samples of glass were collected from the tap and the discharge of the conditioning channel. Observations found that the significant level of degassing was reached. The level of degassing clearly increased as the residence time in the conditioning channel was increased.

Assessment of the short residence time refining of SCM glass has been made with samples collected from similar operating conditions of tests SCM-R-1 and SCM-R-2. The SCM was maintained under continuous steady state operation at 1425°C using the same borosilicate E glass pre-mixed batch. Samples weighing approximately 0.5 kg were collected from the SCM tap and from the conditioning channel discharge in graphite molds. Samples were allowed to cool, with some samples annealed at 800°C. Chemical analyses found the glass samples from the conditioning channel were fully melted and homogeneous, with uniform compositions across both tests. The collected glass samples were split in half, and chemists at GTI and Johns Manville independently measured void fractions of the samples.

Void fraction analyses from test SCM-R-2 tap and refiner samples are presented in Figure 17. Analyses show the void fraction of the tap samples was similar throughout the day of operation. Voids were measured between 25 and 33%. The test was conducted with melt flow rate decreasing (and refiner residence time increasing) as the day continued. Results show that as residence time increases, a large decrease in void fraction is obtained. Average void fraction of samples after the conditioning channel decreased from 16 to as low as 0.3%, equivalent to a decrease in void fraction from 45 to 99% relative to the voids in the SCM product glass. Refining with short residence time was able to convert the foamy SCM glass to a solid glass with remaining bubbles by removing 99% of the gaseous inclusions.

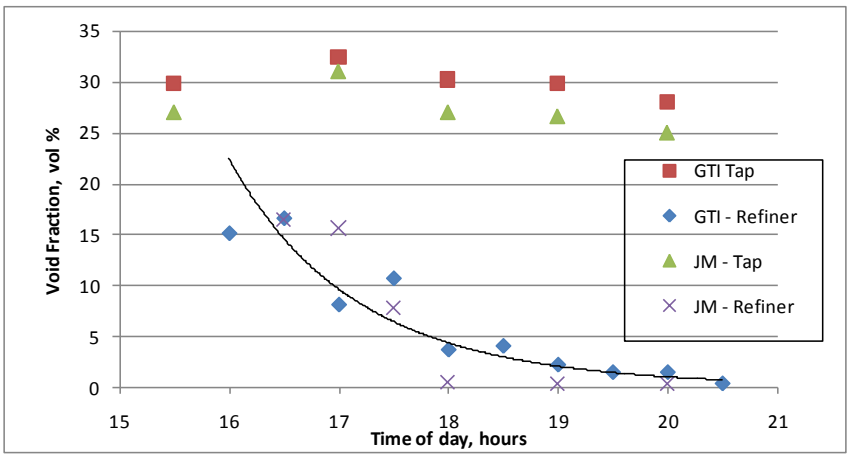


Figure 17. Test SCM-R-2 Tap Glass and Refiner Glass Void Fractions, SCM at 1425°C, Refiner at 1355°C, Pull Rate 175-450 kg/h (Samples Measured Independently by GTI and JM)

The relationship between void fraction in refined glass and residence time in the conditioning channel is shown in Figure 18 for samples collected during both tests. Residence times were shorter in the first test and were longer, covering a wider residence time range, in the second test. All samples were measured for void fraction independently by GTI and Johns Manville chemists. Glass samples from the two tests present the same pattern of reduced void fraction with increased residence time. A short relative residence time period produced a reduction in void fraction to 16%, a removal of inclusions of around 45%. By increasing the residence time, the void fraction decreased. A relative residence time of 3.5 times the initial short residence time produced glass with gaseous inclusions reduced to as low as 0.3%, a removal equivalent to 99% of the SCM glass voids.

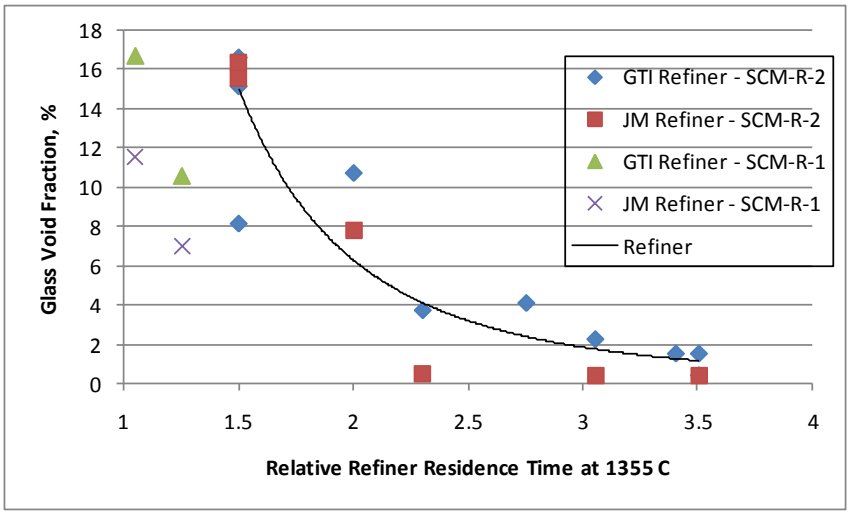
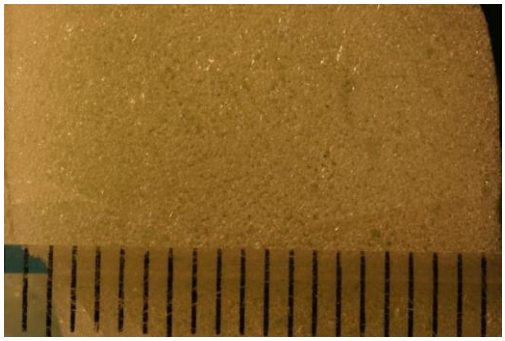
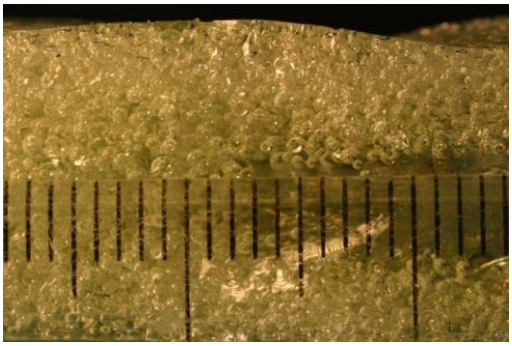


Figure18. Refiner Glass Void Fractions From Two Tests, Samples With SCM at 1425°C, Refiner at 1355°C, Pull Rate 175-600 kg/h (Samples Measured Independently by GTI and JM)

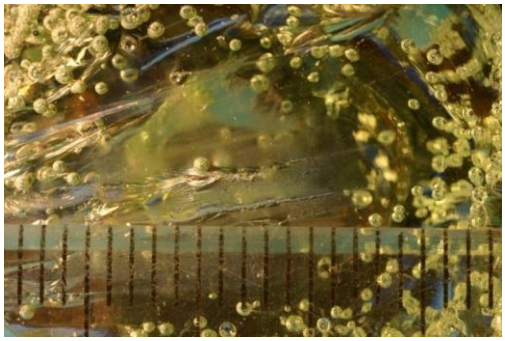
Inclusions in SCM glass have been found to range from microns to as large as 2 cm. Centimeter sized bubbles are not common, but many bubbles are in the range of 10 mm. to 1 cm. Figure 19(a) provides a close-up view of SCM glass with no refining. The bubbles are uniformly dispersed throughout the glass, and relatively large bubbles can be easily identified.



(a)



(b)



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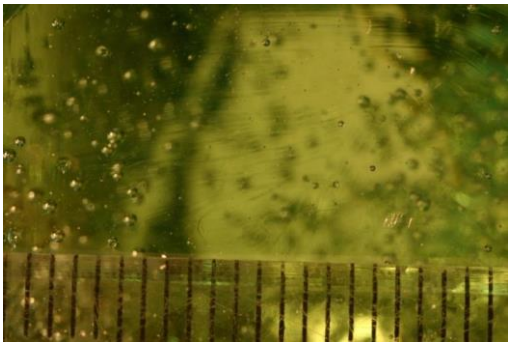


(d)

Figure 19. SCM-R-2 Glass (a) SCM Tap (29% voids) and With Relative Refining Residence Times of (b) 1.5 (16% voids), (c) 2 (11% voids), and (d) 3 (<1% voids) – 1 mm Line Spacing

The other photographs in Figure 19 show examples of the level of refining achieved with various refining time periods. With extended refining times the number of inclusions clearly decreased. The longest relative refining time of 3.5 produced isolated bubbles in the glass matrix. Viewing the photographs as a set shows that the average bubble size increases with additional refining time. This is speculated to be the result of bubble coalescence as larger bubbles rise the fastest and merge with smaller bubbles. An advantage of this process is that bubbles will rise more quickly as they merge and become larger, therefore reducing overall refining time.

As higher levels of refining are reached in future tests, different means will be needed to collect samples. Hand sampling with graphite molds will add bubbles to the molten glass. At high refining levels, this will impact the quality of the product glass.

The short residence time refining step has proven capable of removing 99% of the bubbles in SCM glass. This level of refining may be sufficient for certain market segments such as wool insulation and large diameter continuous fibers. However, most glass applications require much higher quality. Glass ready for forming into fine continuous fibers, containers, tableware, flat glass, and other items requires gaseous inclusion levels from a thousand to a million times better than has been achieved to date. Short residence time refining is anticipated to able to achieve at least a further ten-fold improvement in quality with careful control, but higher quality levels will require a different or supplemental refining approach.

The project team understands that the 99% reduction in bubbles is significantly but that another 2-4 orders of magnitude of bubble reduction is needed to produce commercial glass for fine continuous fibers and containers. This level of refining can be achieved by doubling or tripling the refining channel residence time. A residence time increase of this length lead to a long refining time that would not be considered practical or rapid.

The improved channel refining process depends on the use of a small amount of advanced materials that are highly abrasion resistant at molten glass temperatures. The project team began work this quarter to explore two approaches toward determining appropriate materials for this demanding application. The approaches being explored include:

1. Refractory metals – This approach considers the use of platinum, molybdenum, combinations or alloys of these metals, layers of these metals, or these metals with coatings. A consultant

to the project is exploring the feasibility of this approach with an eye on developing a practical solution at a reasonable cost. Platinum, for example, would meet the material requirements, but using platinum alone would be too costly. Tests are scheduled for next quarter to determine ways to use refractory metals to address the refining channel materials concerns.

2. Refractory materials – Conventional glass furnaces and forehearths are lined with high temperature refractories. The refractory materials are selected based on chemical resistance to the particular glass, thermal shock resistance, thermal insulating capability, cost, etc. Commonly used materials include silica, fused alumina, and chromium refractories. For the particular requirements of the refining process, none of the common commercial refractories is likely to have the corrosion and abrasion resistance required. However, the amount of material is much smaller than is needed for the full refining channel. Therefore, other, more costly refractory materials can be considered. A consultant has been retained who has a concept for preparing a hybrid refractory material that can meet the material requirements. This material will be tested in crucible tests with refractory 'fingers' next quarter.

Based on the results of the two material testing efforts, a material will be selected for testing with the refining channel. The refining channel was to be re-built, incorporating the selected material in needed strategic locations, and testing will be conducted as planned and outlined above. The refining channel re-building and testing were postponed because the thermal convective rapid refining approach was developed. All further refining efforts were focused on the TCRR approach

Vacuum Refining

Reducing pressure causes gas inclusions to expand. Velocity upward is increased by the square of the bubble diameter. Earlier work by multiple researchers has proven this approach to be viable and scalable. Both PPG and Asahi have operated reduced pressure refining at commercial scales. The team felt this approach to be well-enough proven that adaptation to SCM in a NGMS system is an engineering issue beyond the need for extensive investigative research. The SCM produces molten glass with more bubbles than other melting processes. A question regarding SCM is whether the expansion of so many bubbles under reduced pressure would present a foaming problem during refining.

The foaming question was investigated in this project by creating a viscous liquid of silicone oil with a high volume content of bubbles similar to SCM molten glass. The silicone oil was held in a glass flask under controlled vacuum conditions so the behavior of bubbles could be observed. Experiments were conducted by filling silicone oil in a flask with air bubbles, sealing the flask, drawing pressure down to a desired level, and then monitoring and timing the rise of bubbles while holding the pressure level constant.

The photographs below show the silicone oil during clarification at ambient pressure and at a high vacuum of 25 in. Hg. Easy visual observation of the process provided important insights into the refining process. Bubbles are significantly larger at high vacuum, with ambient pressure having a wide range of bubble diameters and high vacuum having only large bubbles. The ambient test demonstrates that large bubbles rise faster. The vacuum test shows that bubbles, particularly large bubbles, rise as a group. What the photographs do not show is that bubbles must break at the surface for lower bubbles to rise in the flask. This confirms the

importance in a rapid refining process of breaking bubbles on the surface to avoid trapping lower bubbles trying to rise by buoyancy.

(a) (b)

Figure 20. Buoyancy Removal of Air Bubbles From Silicone Oil at (a) Ambient Pressure, and (b) 25 in. Hg Vacuum

A further question on the minds of the project team was how deep a vacuum needs to be to significantly increase the bubble velocity and the rate of refining. The graph below shows that the vacuum effect is not linear. As the level of vacuum deepens, the impact on refining increases non-linearly. A vacuum of 10 in. Hg has only a modest impact on refining time. Deep vacuum of 25 in. Hg, however, can reduce the refining time by 80-90%.

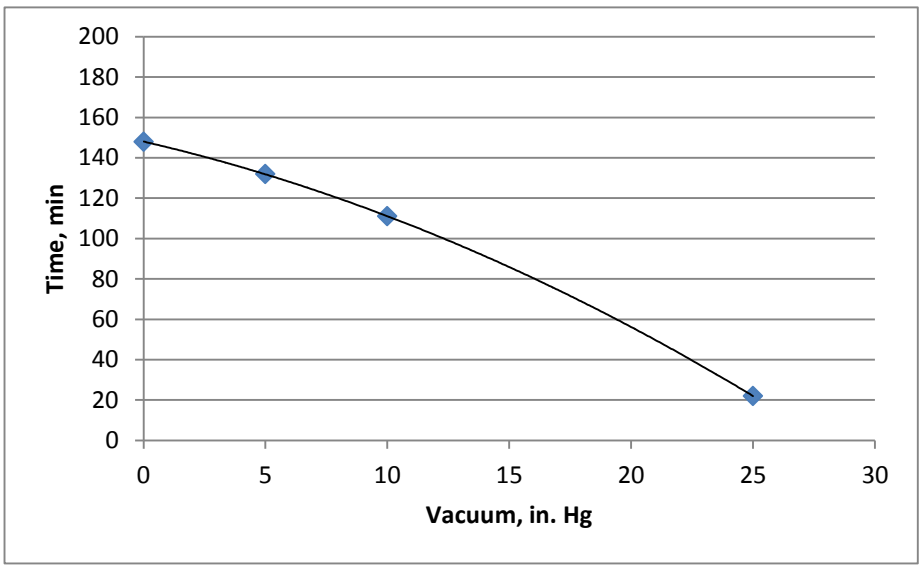


Figure 21. Relationship Between Vacuum and Refining Time of Air Bubbles in Silicone Oil

The vacuum testing confirmed several features of refining. Bubble rise rate is faster for larger bubbles. Bubbles must be broken at the surface to prevent stable surface bubbles from halting the rise of lower bubbles to the surface. Vacuum is an effective way to speed refining, but a deep vacuum of near 25 in. Hg is needed to achieve refining time decreases of 80 to 90%. Industrial plant operators are generally not comfortable with the combination of molten glass and

high vacuum processes. While a process can be built and operated, this is not a preferred path for most glass makers. Future developments may find ways to work with lower vacuum, work with pulsed vacuum, or improving the mechanical aspects of vacuum refining. All of these improvements would make vacuum refining a more attractive solution for quickly refining glass in the NGMS process.

Centrifugal Refining

N.A. Penkova[1] has studied all of the proposed approaches to rapid fining of glass. Through calculations and experimental results, she showed that centrifugal refining, particularly cyclonic centrifugal refining, can decrease refining time more than any other approach, by a factor of more than 10. St. John of Owens Illinois found non-cyclonic centrifugal refining to be effective experimentally.[2] These authors and others have pointed out the technical challenge of operating a 200- to 500-rpm centrifugal glass refiner.

The first centrifuge type studied combines thin-film and cyclonic refining. The principle of the centrifugal degassing process consists of gassed liquid being applied as a thin layer onto the inner lining of a truncated-cone rotor. This provides advantages of 1) decreasing refining time, since bubbles need only travel through the thin film to the inner surface, and 2) enabling very small bubbles (of 0.0004 in.) to be removed. (St. John's patent cites bubbles of 0.001 in. and above.). Since all bubbles may not burst on the surface, the device is specially designed to provide bulk flow of the essentially bubble-free portion of the molten glass. The thin film cyclonic refiner allows faster rotation speeds, more rapid refining, and better separation of smaller bubbles than other methods. Cone angle and gravity assist in bulk flow and separation of product glass from surface bubbles or froth. This approach also easily manages movement of the flowing glass from the rotating cone to a stationary surface.

The second centrifuge configuration analyzed is a separator in which the portion of the glass with few or no bubbles is continuously drawn from the tapered end of a rotating cone. Unrefined glass is tangentially injected into a tapered rotor filled with glass. Centrifugal action creates a bubble concentration gradient, which forces bubbles toward the cone's axis. As glass is drawn downward, the narrowing cone causes the lowest-bubble-content glass along the walls to be pulled downward and the 'frothy' glass in the center to be pushed upward. In operation, glass enters at the top of the cone and is separated into two streams. The high-bubble-count stream along the axis is pushed upward, back into the melter, and the low-bubble-count stream is pulled from the bottom of the cone. When connected with a high-intensity melter, this technique will serve as a 'bubble trap' to prevent bubbles from leaving the melter. The bubbles will be coalesced, returned to the melter, and will rise to the surface in the melter.

Both configurations were evaluated in cold-flow testing. Work included the following:

- Designing laboratory devices to evaluate both configurations
- Selecting a fluid that has visco-elastic properties similar to glass at room, temperature
- Ordering all needed components to build laboratory test stands
- Assembling the test apparatus

[1] Penkova, N. A., Glass and Ceramics, 50(9,10), 1993.
[2] St. John, Douglas F., "Method for Removing Gaseous Inclusions from Molten Glass," U.S. Patent 3,938,981, February 17, 1976.

Test series were conducted with both cold-flow apparatuses to determine the effects of a wide range of independent variables on bubble removal from a fluid that has visco-elastic properties similar to molten glass. A full list of independent variables was developed. The independent variables for both configurations include at least:

- Cyclone dimensions (length, diameter, angle of narrowing)
- Cyclone rate of rotation (rpm)
- Fluid flow rate
- Bubble size
- Bubble concentration in the fluid
- Fluid viscosity

The centrifuge designs considered here involved calculations for two approaches to cold-flow modeling of glass refining, thin-film and conical. A balance between gravitational and angular acceleration shows that the magnitude of the acceleration determines the direction of the melt flow at various centrifuge speeds, wall angles, and angular velocities. This concept is portrayed in Figure 22.

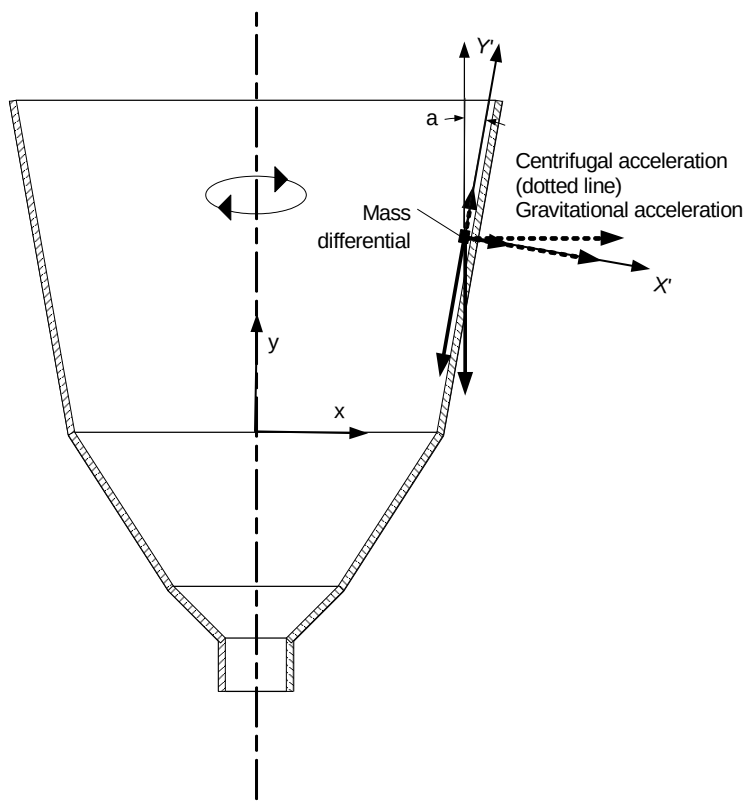


Figure 22. Angular and Gravitational Acceleration Diagram

Force-balance equations can be used to estimate the movement of a bubble within a centrifuge. Figure shows the acceleration balance as a function of wall angle for a centrifuge with a diameter of 0.5 m and various rotational speeds.

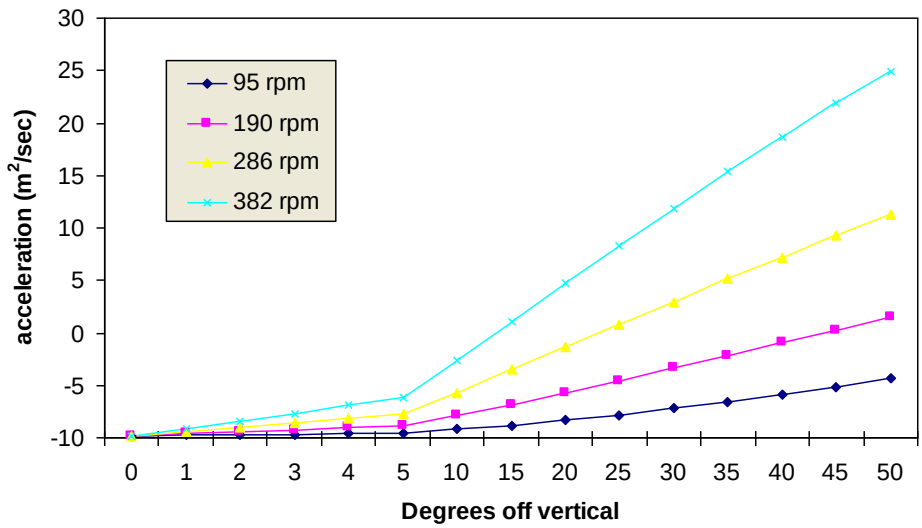


Figure 23. Mean Relative Acceleration for a Centrifuge Diameter of 0.5m at Various Wall Angles and Angular Velocities

The acceleration factor, which is the ratio of angular acceleration to gravitational acceleration, is a very important parameter to consider for reducing fining time. As the acceleration factor increases, the bubble velocity increases due to the density difference between the bubble and the melt. As the angular velocity or radius is increased, the ratio becomes larger causing the relative effect of gravity to diminish. The equation used for calculating the acceleration factor is shown below:

$$z = \frac{\omega^2 R}{g}$$

where z is the acceleration factor, ω is the angular velocity, R is the centrifuge radius, and g is the acceleration due to gravity. As shown in Figure 24, this factor can reach values over 600 times that of gravity with a centrifuge diameter and velocity of 5 m rotating at 477 rpm. It is desirable to have a large acceleration factor to create an increase in pressure towards the wall of the centrifuge. This generates a larger density gradient between the fluid in the melt and the gaseous bubble, accelerating the bubble velocity.

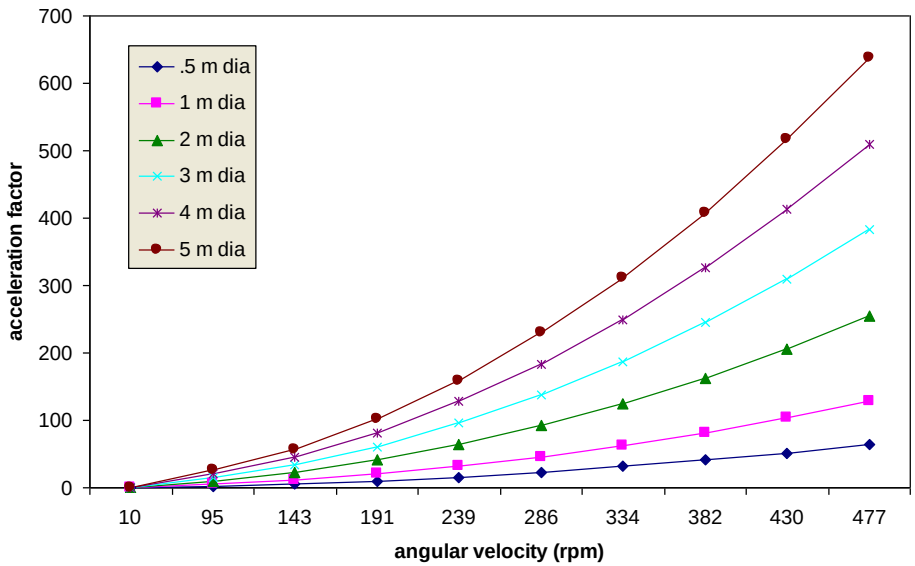


Figure 24. Acceleration for Various Centrifuge Diameters, Wall Angles, and Angular Velocities

When the acceleration factor is known, the bubble velocity through the melt can be determined. The velocity of the bubble can be calculated using the following equation:

$$W_c = \frac{1}{3}\frac{r^2 \Delta\rho \omega^2 R}{\upsilon}$$

where W_c is the bubble velocity, r is the bubble radius, $\Delta\rho$ is the density gradient between the gas and the fluid, ω is the angular velocity, υ is the kinematic viscosity, and R is the centrifuge radius. This relationship is portrayed in Figure 25. Using this principle and assuming a film thickness of the melt, the length of the centrifuge needed to remove gaseous bubbles can be calculated.

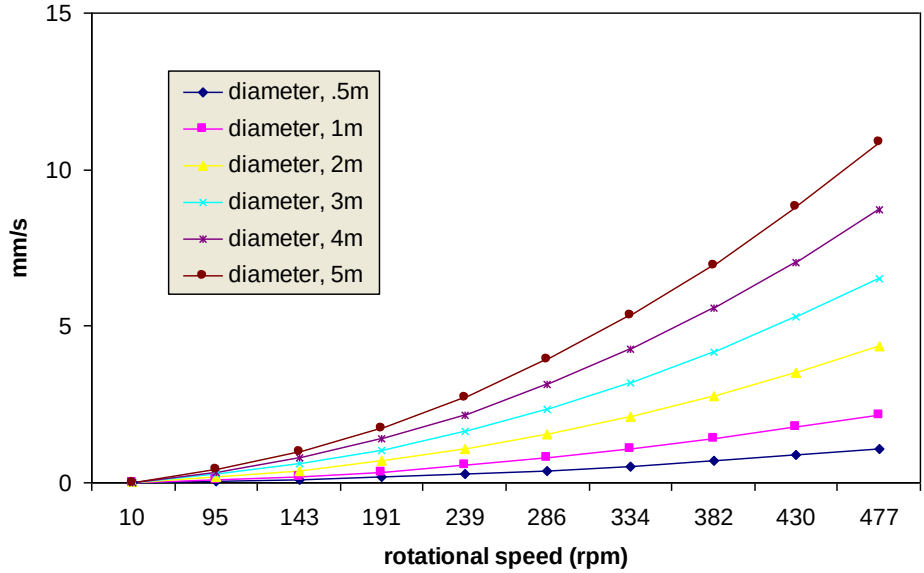


Figure 25. Bubble Velocity (mm/s) for Various Rotational Speeds and Centrifuge Diameters

The volumetric flow rate of the fluid through a thin-layer centrifuge refining system is determined by the following equation:

$$\dot{V} = 2\pi Rgzb^3 \frac{\sin\alpha}{3\upsilon}$$

where $\dot{V}$ is the fluid volumetric flow rate, R is the centrifuge radius, g is the acceleration due to gravity, z is the acceleration factor, b is the fluid thin-film thickness on the centrifuge wall, α is the angle of the centrifuge wall off of vertical, and ν is the fluid kinematic viscosity. With the volumetric flow rate and velocity of the bubble known, the length of the centrifuge can be determined by calculating the average velocity of the melt through the centrifuge for various angular accelerations, diameters, and flow rates.

A two-dimensional bench-scale experimental centrifuge was designed, based on the equations above. This bench-scale centrifuge was designed to have a diameter of 4 inches and a thin-film cylinder section of approximately 8 inches. This centrifuge was designed to be capable of removing bubbles of approximately 100 μm diameter.

Calculations were made to compare the glass production rates of thin-film centrifugal refining those of current refining methods. The calculations showed that, to remove minimum bubble sizes of 330 μm from a glass melt (based on a design from Sokolov[3]), using standard thin-film gravity methods, 75 ton/day is the production capability whereas with a thin-film centrifugal refining system (centrifuge diameter of 2.6 ft, height of 5.6 ft, and a rotational speed of 300 rpm) has the capability to produce over 300 ton/day. A cyclonic centrifuge with comparable dimensions to the thin-film centrifuge is calculated to yield 75 ton/day. These calculations point to the thin-film centrifuge as being the better approach for centrifugal refining.

A glass fining experimental setup was used to experimentally verify the centrifugal bubble calculations. The arrangement, shown in Figure 26, consists of a data acquisition system

[3] V.I. Sokolov, Modern Industrial Centrifuges, Mashgiz, Moscow (1968).

and a 1-HP variable frequency electric motor with speed controller, rotating table top. The table top can rotate at over 1000 rpm to study the effect on bubble velocity. The data acquisition system consists of a SLR camera fitted with a macro lens to capture the rate of bubble migration. Polybutene H-100 was used to simulate molten glass . At the temperatures tested in the laboratory, this fluid matches closely the viscosity of molten glass.

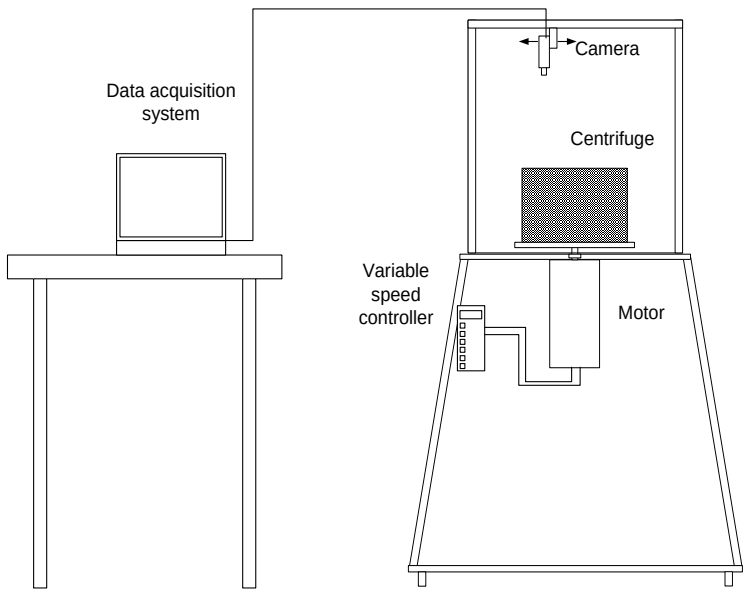


Figure 26. Experimental Arrangement for Tracking Movement of Bubbles within a Fluid
The testing procedure involved creating a bubble with a needle-syringe, rotating the centrifuge for a set time at a set rpm, and tracking the distance that the bubble traveled using the camera system. The results were then plotted and compared to theoretical calculations.

Bench-Scale Experimental Arrangement – 2D

An experimental centrifuge prototype was designed and built to incorporate both the thin-film and conical approaches to centrifugal glass fining. 27 shows schematics of this centrifuge. The centrifuge was designed with a spinning 4-inch cylinder section that transitions to a static conical section. The same motor used in the prior experiments was used to drive the centrifuge through a belt connection.

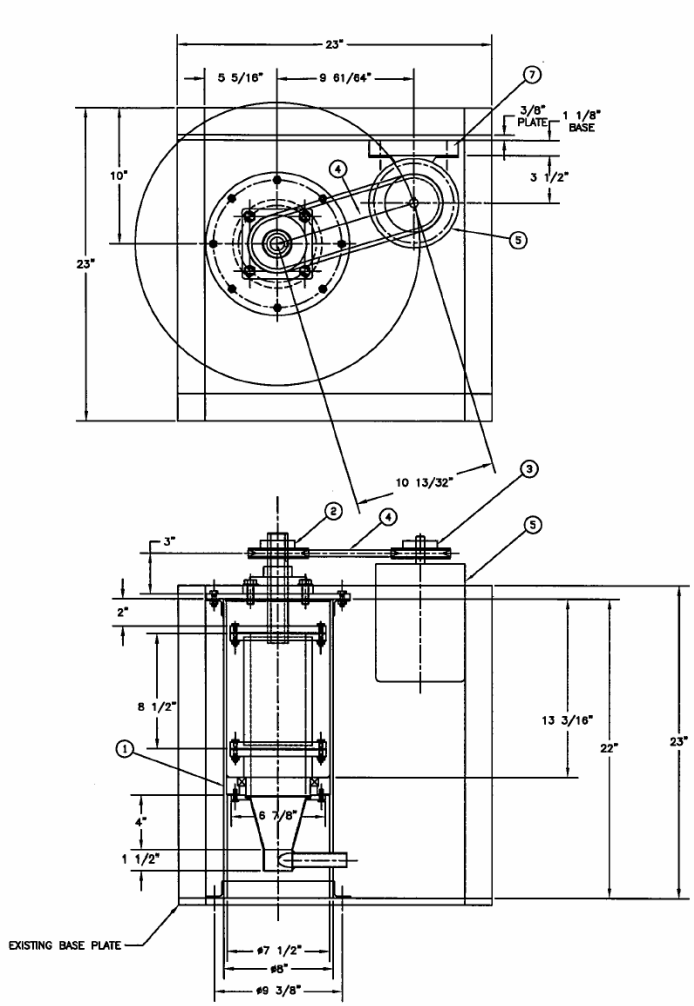


Figure 27. Schematics of the Bench-Scale Centrifuge Prototype

The fluid chosen for this experimental arrangement was 100% corn syrup. Polybutene proved to be difficult to handle and was too viscous for these tests. A sample of pure corn syrup was tested at the GTI analytical laboratory, using a rotating viscometer. The viscosity curve obtained from this test is shown in 28. For all tests performed, the corn syrup was at 70 to 73°F, correlating to a kinematic viscosity (viscosity divided by density) of approximately 1400 cSt (centistokes). Molten soda-lime glass has a kinematic viscosity of approximately 4500 cSt. Since the experimental setup here is a scaled-down version of what would be used industrially, the lower viscosity fluid was needed to proportionally test the effects of centrifugal forces on bubble removal.

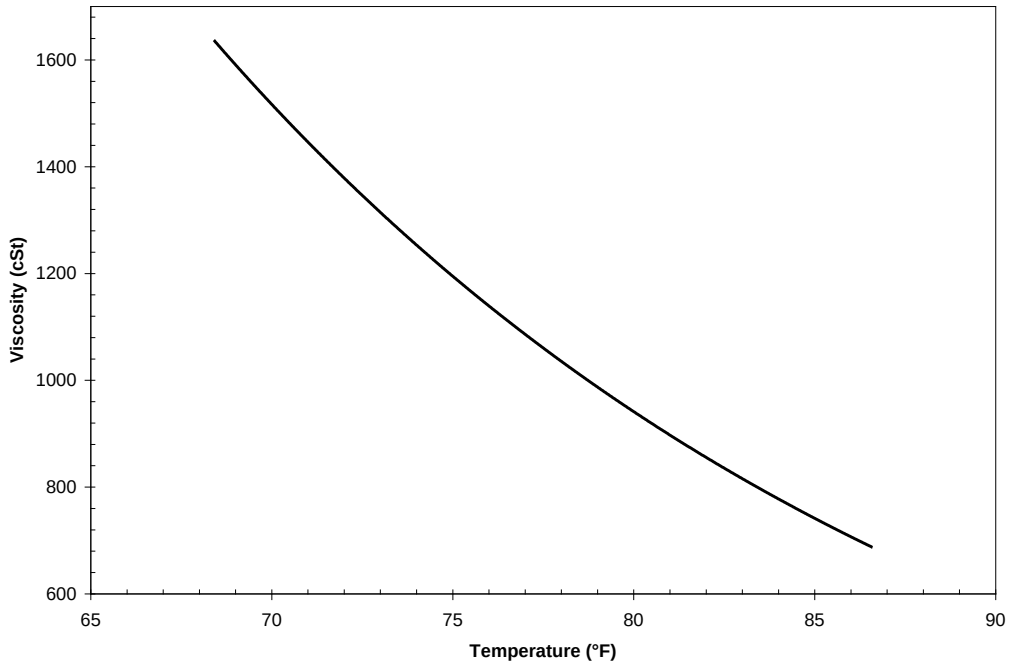


Figure 28. Corn Syrup Viscosity as a Function of Temperature

The flow diagram for the prototype refining system test configuration is shown in Figure 29. The fluid flowed from a supply tank by means of gravity and pressurized nitrogen. Bubbles were generated in the tank by supplying nitrogen through a tube within the fluid. Bubble size and distribution was observed and recorded through a Plexiglas window. From the tank, the fluid flowed into the centrifuge section. After being refined, the fluid was once again viewed through another observation window and the temperature was measured again. Finally, the fluid flowed into a drain tank. A vacuum line connected to the drain tank was used to facilitate the flow of the fluid from the centrifuge to the tank.

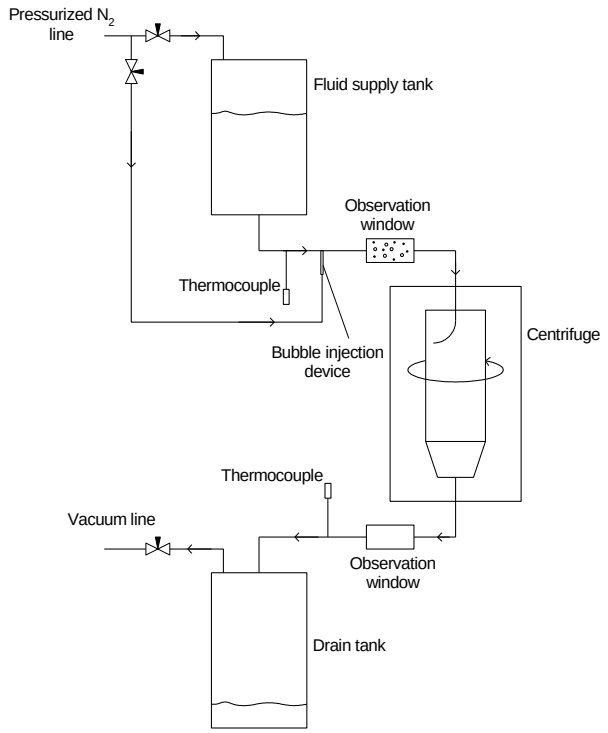


Figure 29. Centrifuge Bench-Scale Experimental Flow Diagram

Figure 30 shows the completed experimental arrangement. Prior to each test, the system was allowed to equilibrate at the chosen flow-rate and rotation rate for 10 minutes. Tests were performed at 200 and 500 rpm for two different fluid volume levels at a flow rate of 4.3 ml/min. Tests were also performed at flow rates up to 20 ml/min with no visible change in refining performance.

Bubble sizes were observed and acquired using a Canon digital SLR camera coupled with a Canon 65 mm lens. The fluid flow was halted long enough to take photographs during the experiments. Bubble sizes were manual measured from the photographs.

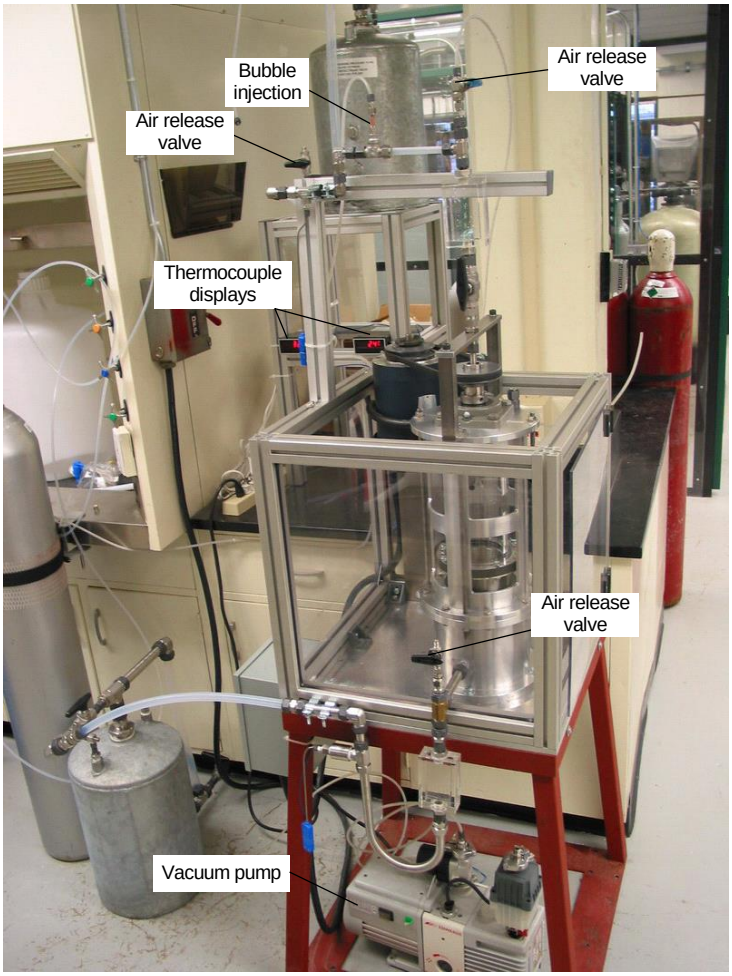


Figure 30. Glass Refining Experimental Arrangement

Lab-Scale Centrifuge – 1D

The first series of static tests measured bubble velocity under gravitational force. This work enabled the project team to acquire baseline data on travel of bubbles in the viscous liquid as a function of their size. Figure 31 shows that the velocity-bubble size relationship parallels the theoretical velocity-bubble size curve. The figure also shows that this experimental relationship is proportional to the square of the bubble diameter, as predicted by theory.

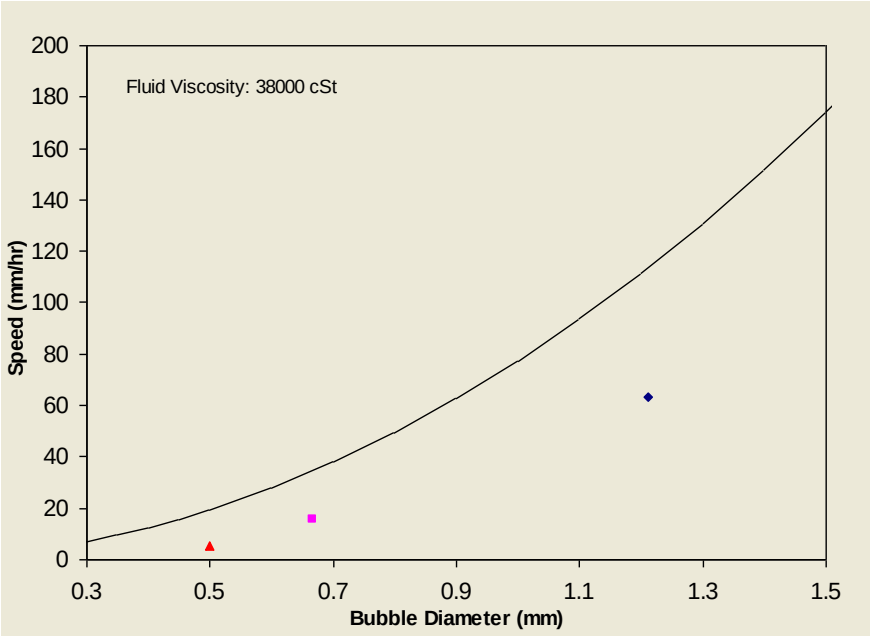


Figure 31. Vertical Bubble Velocity Within Polybutene During Static Tests Compared with Theoretical Prediction

A series of lab-scale rotational tests was performed in which individual bubble migrations were tracked and analyzed at rotational speeds of 600 and 300 rpm. The data was processed, compared to the theoretical calculations, and graphed to show the bubble velocity in mm/hr as a function of the radial position. Experimental data from the tests is shown in Figure 32 and Figure 33. Here, individual bubbles were tracked; their radial position is denoted on the x-axis and their velocity on the y-axis. These results show that the radial velocities of the bubbles are lower than the theoretical expectations. The important finding here is the matching of the rate of change of the bubble velocities with the centrifuge radius. Discrepancies between experimental and theoretical findings are probably due, in part, to uncertainty in the fluid viscosity.

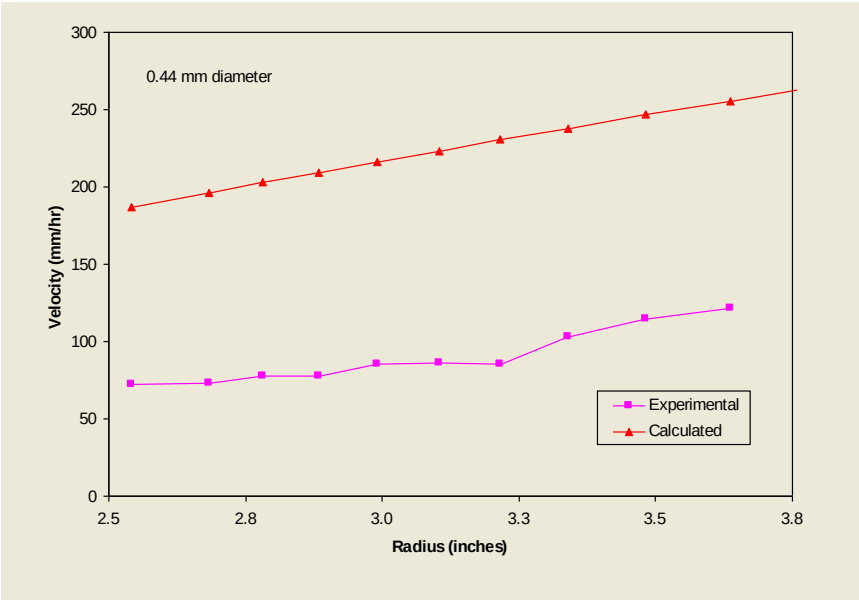


Figure 32. Lab-Scale Centrifuge Test Data Showing Radial Speed as a Function of Radial Position for a 0.44 mm Bubble

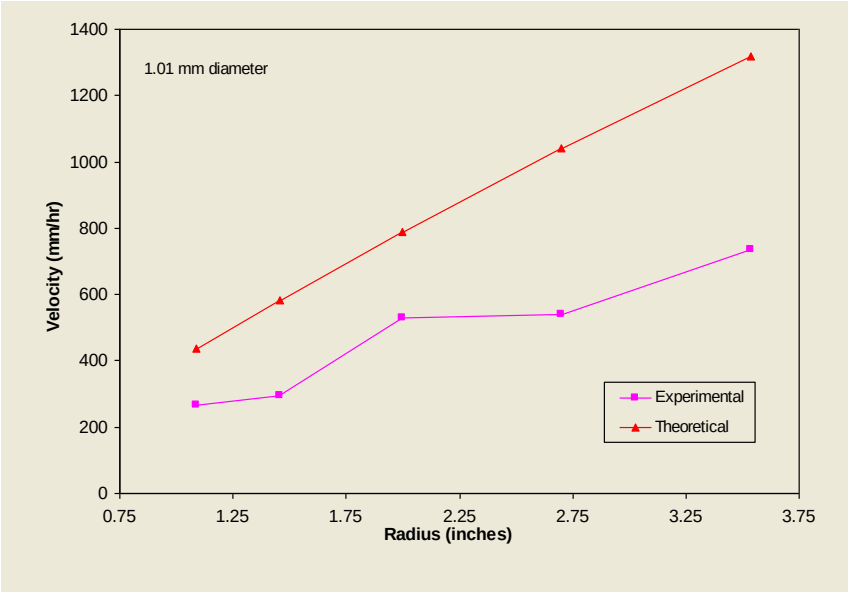
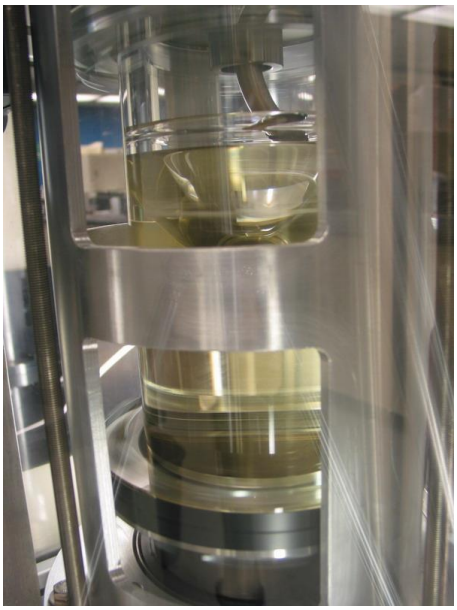


Figure 33. Lab-Scale Centrifuge Test Data Showing Radial Speed as a Function of Radial Position for a 1.01 mm Bubble

Results from this centrifuge are encouraging and clearly show the benefits of centrifugal force on more rapid bubble motion. As predicted by theory, the bubble velocity increases with bubble size and increases with larger distance from the center of the centrifuge (higher centrifugal force). The quantitative results provided the detail needed to design the bench-scale cyclonic centrifuge testing apparatus. The bubble travel speeds observed in the lab-scale unit were not large enough to justify the use of centrifugal glass refining, but the positive results warranted extension of the study to the two-dimensional bench-scale batch centrifuge.

Bench-Scale Centrifuge – 2D

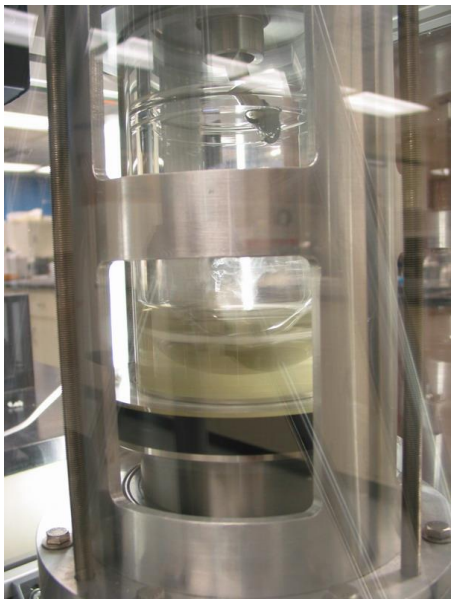
The bench-scale testing yielded positive experimental results. Tests were conducted with a 4.3 ml/min flow rate and were each given 10 minutes to equilibrate at their respective rotational speeds. Tests were performed with two different volumes of fluid in order to realize the effects fluid volume would have on the refining process. One series of tests was performed with the fluid level at the bottom of the centrifuge cylinder flange, while higher volume tests were performed with the fluid level 4 inches above the flange. The lower volume tests showed the benefits of implementing the thin-wall method, and the larger volume tests did not. Figure 34 and Figure 35 show photographs of the centrifuge during tests for the two fluid volumes. Figure portrays visibly the conical shape formed within the centrifuge. The thin-wall method was achieved most closely in the experiment shown in Figure for the 500-rpm condition, where the cone is very steep, with a thin wall of fluid extending the length of the cylinder.



200 rpm

500 rpm

Figure 34. Centrifuge Photo with 4” Fluid Above Bottom Flange



200 rpm

500 rpm

Figure 35. Centrifuge Photo with 0” Fluid Above Bottom Flange

Figure 36 shows an image of the exit window at static conditions. The image represents the quality of the fluid-bubble mixture with no centrifugal refining imposed. Bubble sizes range from micron-scale to 0.5 mm.

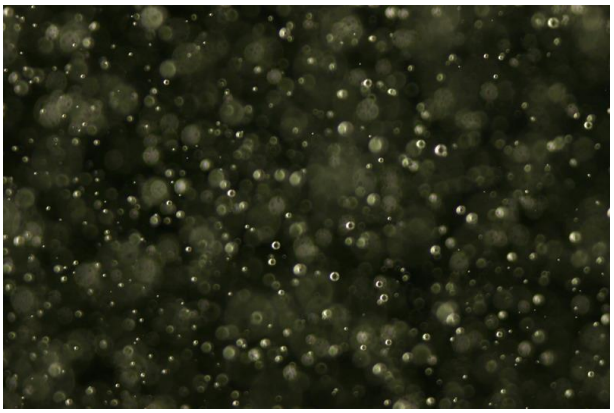
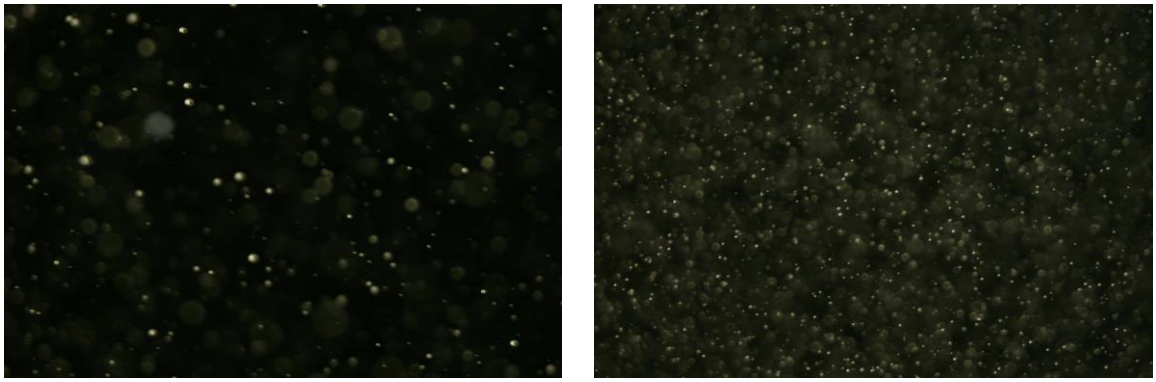


Figure 36. Centrifuge Outlet Window at 0 rpm

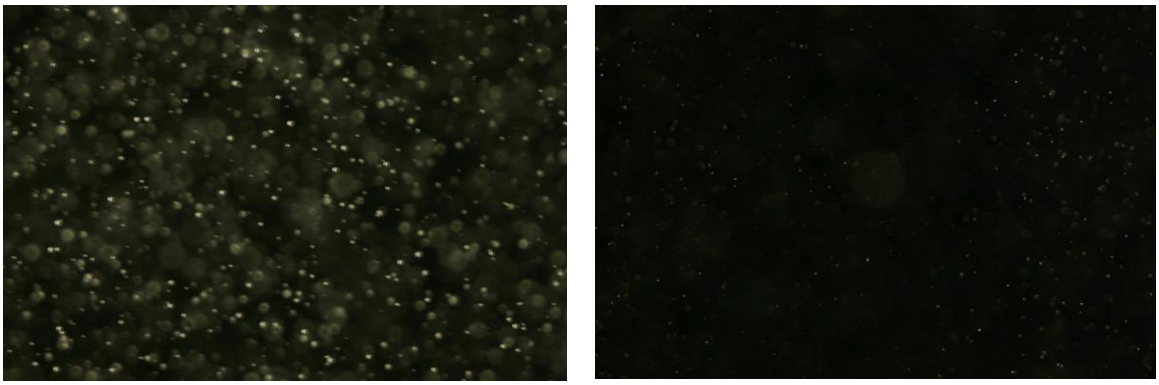
Figure 37 shows representative outlet window photographs from the large-volume tests. In the 200-rpm case, only the largest bubbles in the population left the fluid. The 500-rpm condition shows a much smaller population of bubbles than the static base case. For 500 rpm, the bubbles ranged from 0.1 mm to micron in diameter, but there was a larger total bubble population than the base case.



200 rpm 500 rpm

Figure 37. Centrifuge Fluid Output with 4” Fluid Above Bottom Flange

Figure 38 shows the results from the lower-volume experimental tests. The 200-rpm condition yielded a bubble size distribution ranging from 0.08 mm to microns. The bubble population from this test case was also larger than the base case population, indicating that some of the larger bubbles broke down to form smaller bubbles. The 500-rpm case yields the most promising results. There was a range in the population bubble diameter size of 0.05 mm to microns. The small bubble sizes made it difficult to compare the number density to the base case. It is evident, however, that the small bubbles are much more abundant here than the base case, indicating that there was some breakdown of the larger bubbles to smaller ones. These results verify the expectations of the refining ability of this centrifuge based on the calculations made. It was predicted that this centrifuge would be able to remove bubbles with diameters greater than approximately 100 microns (0.10 mm).



200 rpm 500 rpm

Figure 38. Centrifuge Fluid Output with 0” Fluid above Bottom Flange

Filtration Refining

Project engineers read as much literature as possible on possible approaches to refining. This led to brainstorming of alternate ways to remove bubbles from viscous liquids. All methods relied on manipulating the independent variables on the right hand side of the Stokes Law equation. Then engineer studied the properties of bubbles. One interest bubble behavior is the tendency of surface tension to weakly attach bubbles to a solid surface. A concept for refining

was developed in which a viscous liquid, in this case molten glass, is passed through one or more fine mesh screens or beds of particles. As the flow follows a tortuous path, bubbles will attach to the solid screens or beads. The attached bubbles were presumed to coalesce into larger bubbles that will then rise to the surface. In this way bubbles were predicted to be filtered from the viscous liquid.

This approach to fine refining was proposed by GTI. First, use a coarse refining process to removes all bubbles down to under 1 mm. Fine refining would then take this product glass and removes all the remaining bubbles. Tests were conducted under laboratory steady state conditions with high viscosity silicone oil serving as a surrogate liquid. These tests were positive in removing all fine bubbles, but the rate is too slow for an industrial process. Initial efforts to increase the production rate were positive leading to a rate that was still 2 to 3 times slower than desired for an industrial process. Parametric tests with the surrogate liquid confirmed the initial results. The project team believes there may be a way to increase the velocity and production rate to improve and industrial rate to an acceptable level. Tests were conducted with molten glass to assess the potential success of the fine refining approach.

A patent application covering the two-stage refining approach was filed and was granted. The patent describes the two stage refining approach, with stage one as the short front end 'coarse' refiner, and stage two as a filter bed 'fine' refiner. While project resources were insufficient to investigate all aspects of this approach to refining, the project team feels results validate the overall approach and the two stages of refining. Further development would be needed to develop filtration refining into a commercial process.

Shown below is a graph with silicone oil flux through different filter layers. While high fluxes can be achieved, the photographs show that high fluxes do not stop all bubbles from passing through the filter bed.

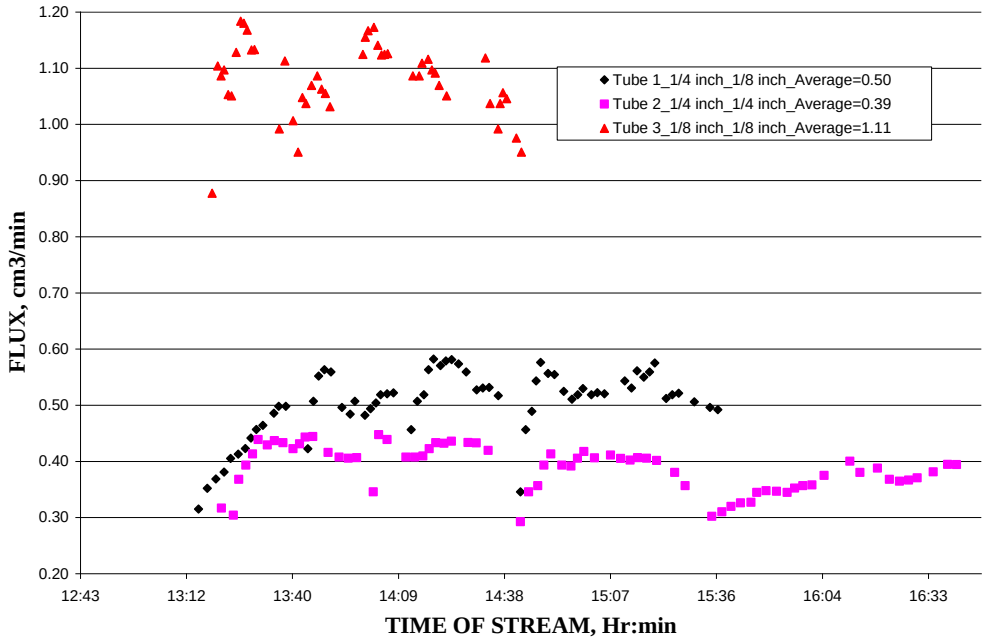
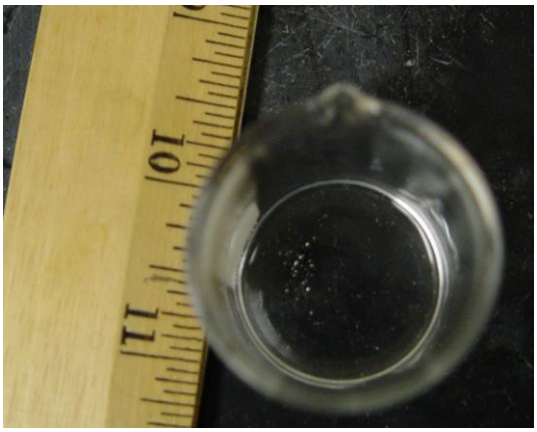
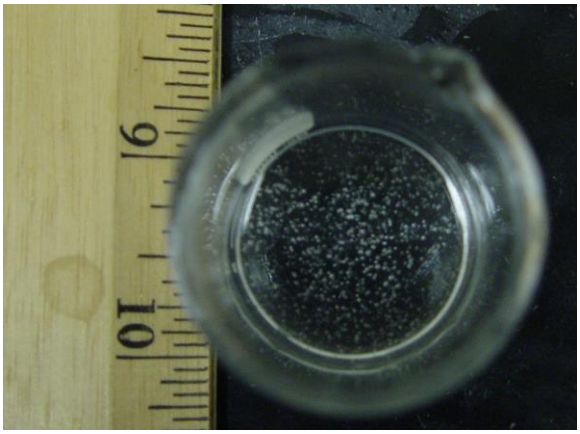


Figure 39. Flux of Silicone Oil Through Multiple Screens



Tube 2 Filtrate



Tube 3 Filtrate

Figure 40. Bubbles in Filtrate After Passing Through Multiple Screens

The practical limits of filtration refining were not explored in this project due to limited resources. A practical system would need 3 to 20 times higher fluxes. Also, there may be a lower limit to the size of bubbles that can be removed. Bubbles that are too fine may continue through the filter. Decrease the pore sizes of a filter may have practical limits because a filter with pores below a certain size will prevent flow of a molten glass through the bed. All of these and other issues could be addressed with additional testing and modeling.

Tests of the fine refining step were conducted with molten soda-lime glass melted in a laboratory electric furnace. Bubbly SCM product glass was melted and passed through high-temperature resistant refractory beads to determine the ability of the surfaces to retain bubbles. The tests were unsuccessful because the flow rates, residence times, and velocities could not be controlled well enough. Some improvement in the bubble count was achieved, but below the level of control required for an industrial process. Future work would need to repeat this testing with better controls on velocity and residence time in place.

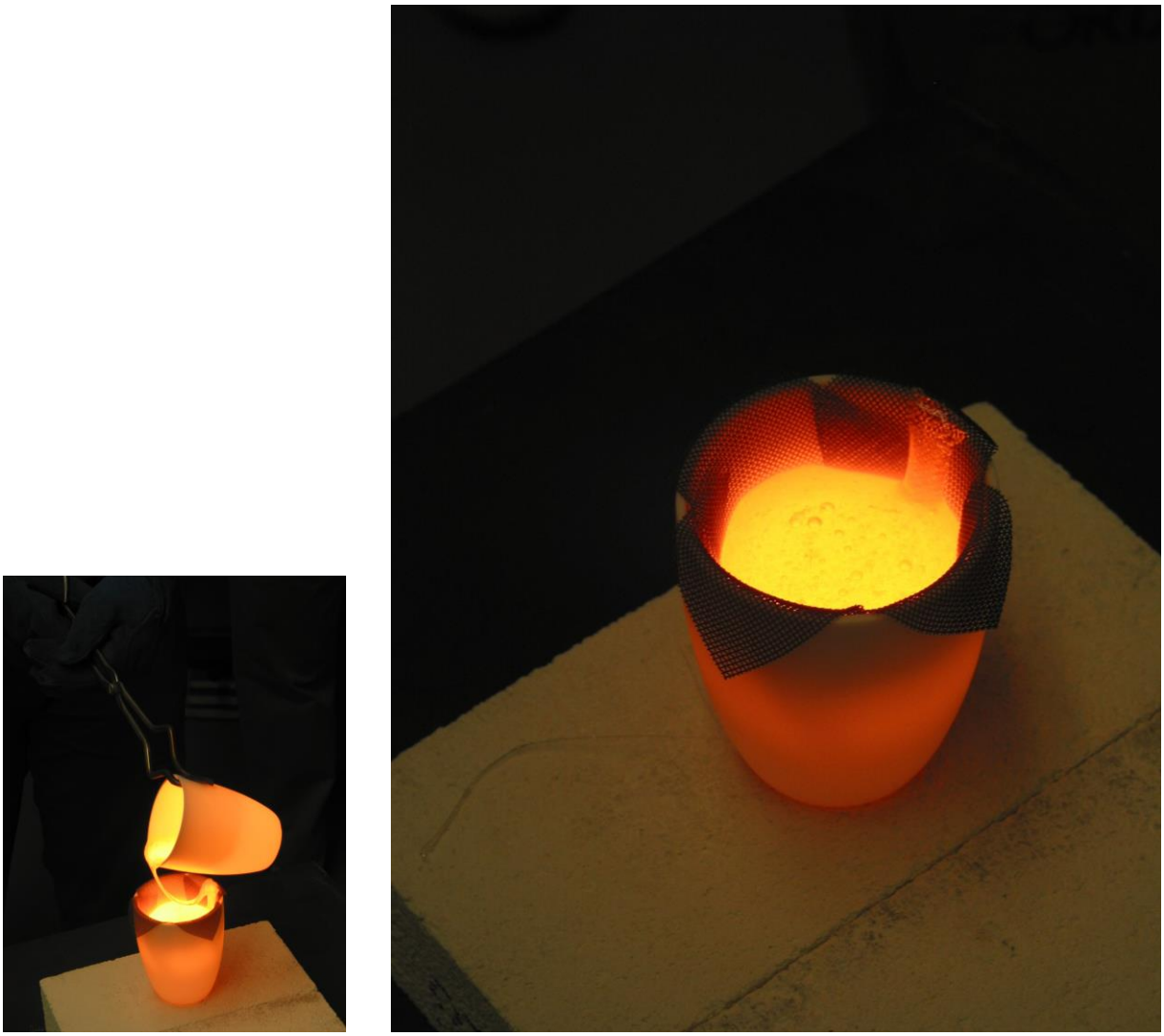


Figure 41. Molten Glass After Filtered Through Ceramic Beads

Obstructed Path Refining

In considering the movement of rising bubbles in a molten glass traveling along a channel, questions were raised regarding separation of clarified bubble-free glass from the glass still containing bubbles. As the glass refines, bubbles rise toward the surface and clear, bubble-free glass is left at the bottom of the channel. The project team carried out a series of tests with silicone oil as a simulated fluid to find if there are ways to use obstructions to isolate bubbly glass from the refined, bubble-free glass.

A rectangular Plexiglas tank was built to simulate a refining channel. For different tests, the chamber was empty or had a number of obstructive weirs (1 to 4). Weirs were placed to force the silicone oil to either move above or below the obstruction. Most tests were conducted at ambient pressure, but testing was also conducted at 10 in. Hg vacuum. The tank was filled with silicone oil, a simulated liquid with viscosity similar to molten glass during refining. Silicone oil was added at one end of this box and discharged through a tap in the lower part of the wall at the far end of the box. Air bubbles were introduced at the bottom of the chamber at

the feed end. The time needed for bubbles to travel from the feed to the discharge end of the chamber and exit through the discharge port was recorded. The depth of silicone oil in the box was kept constant at all times.

The photographs bellow show the behavior of bubbles as move along the simulated channel. The weirs were found to provide no restriction to bubble movement for small bubbles. While larger bubbles moved to the surface and broke, smaller bubbles followed the streamlines of fluid flow and were carried over and under weirs. The idea that bubbles could be trapped did not appear to take place. Following the streamlines mirrors bubble behavior observed during the filtration refining tests. Multiple forces are acting on the bubbles as they travel along from feed to discharge port. The weirs serve to block flow, but they also introduce an additional convective force because velocity must increase through the narrowed opening created by a weir. The photographs do not clearly show this behavior, but observers conducting the tests were easily able to track individual bubbles. Small bubbles could be seen 'pulled' along by the flow as it increased in velocity going over or under a weir. The obstructive benefits of weirs might be improved by designing a channel with deeper sections below weirs so velocity increases do not occur. There were insufficient resources in this project to pursue this potential improvement to obstructive refining.

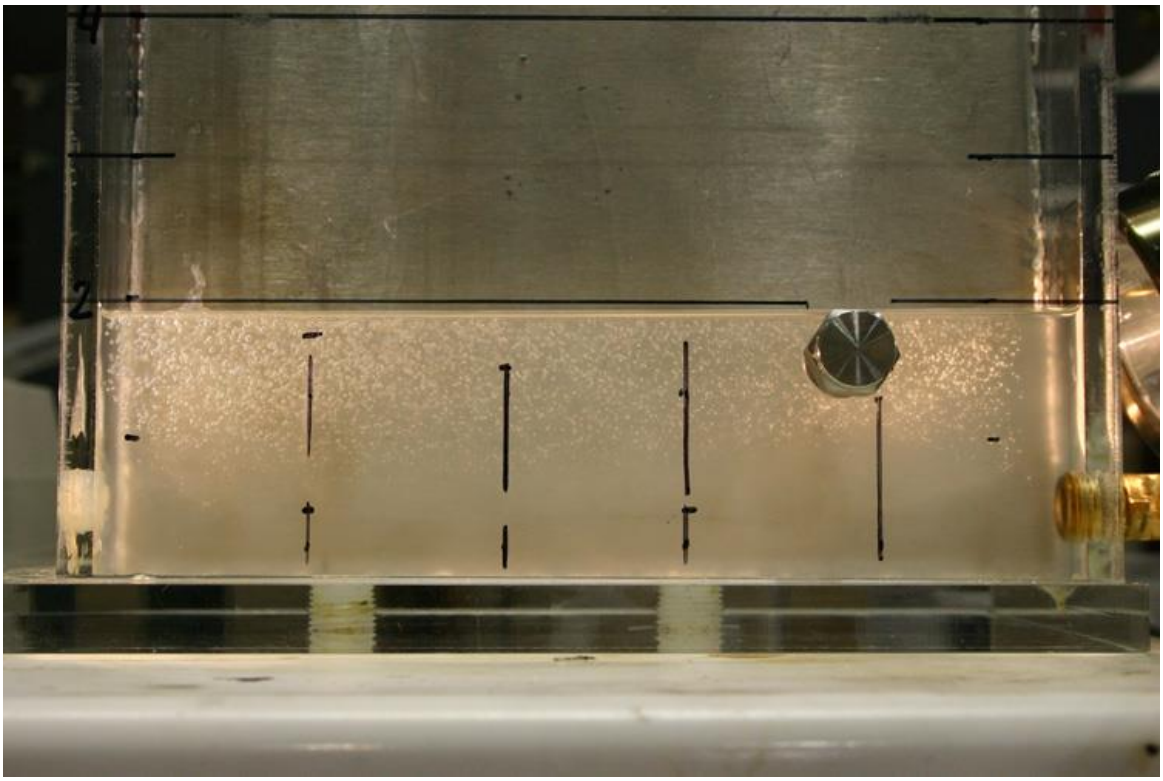


Figure 42. Ambient Pressure, No Partitions, 20 min

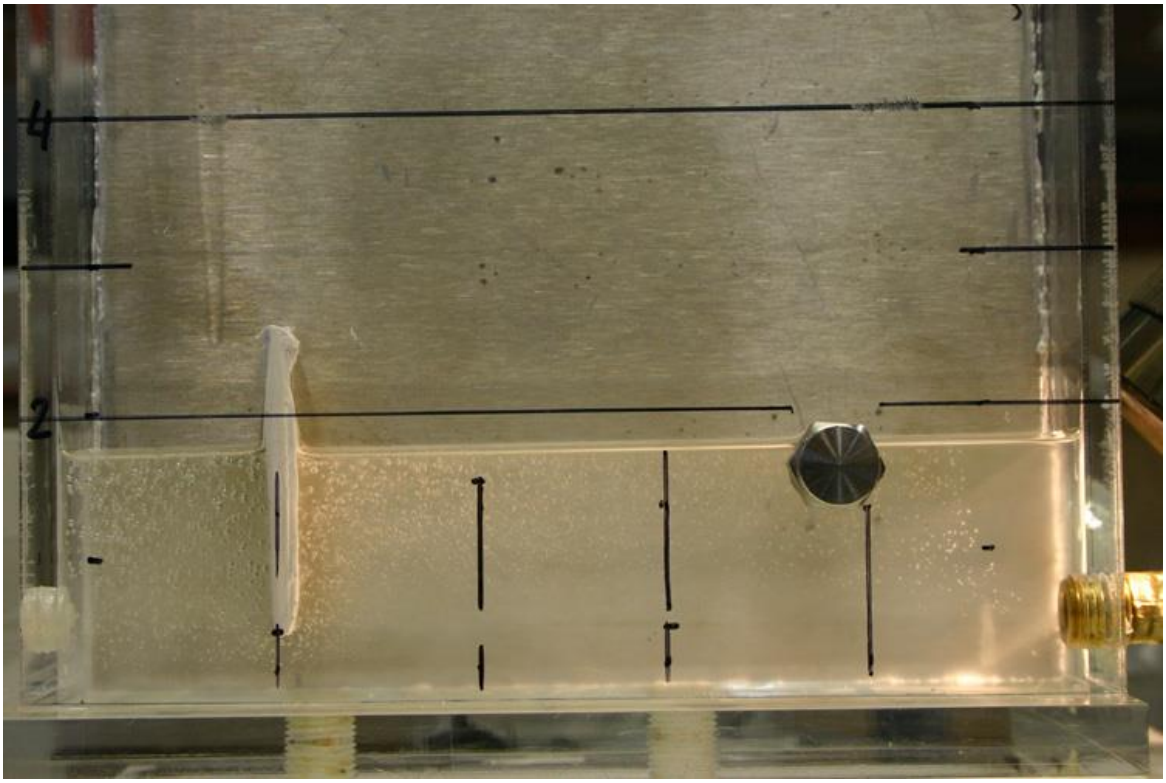


Figure 43. Ambient Pressure, One Partition, 18 min

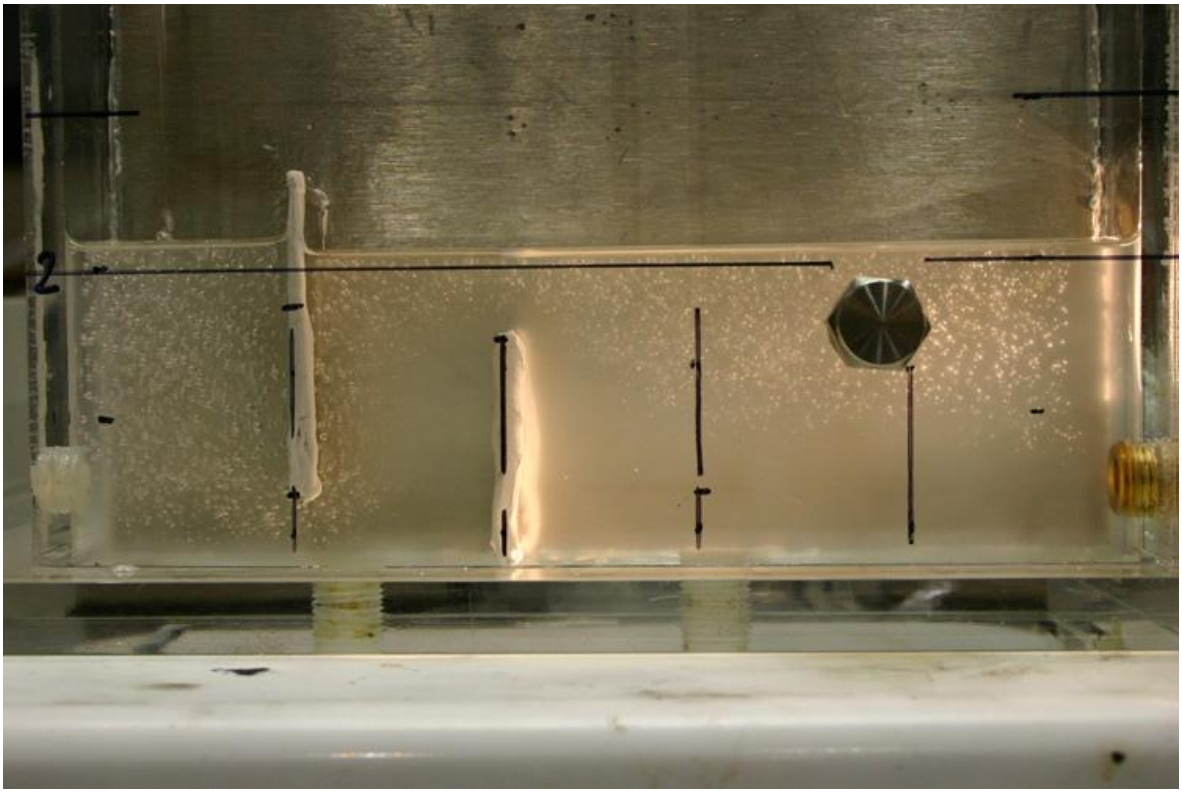


Figure 44. Ambient Pressure, Two Partitions, 19 min

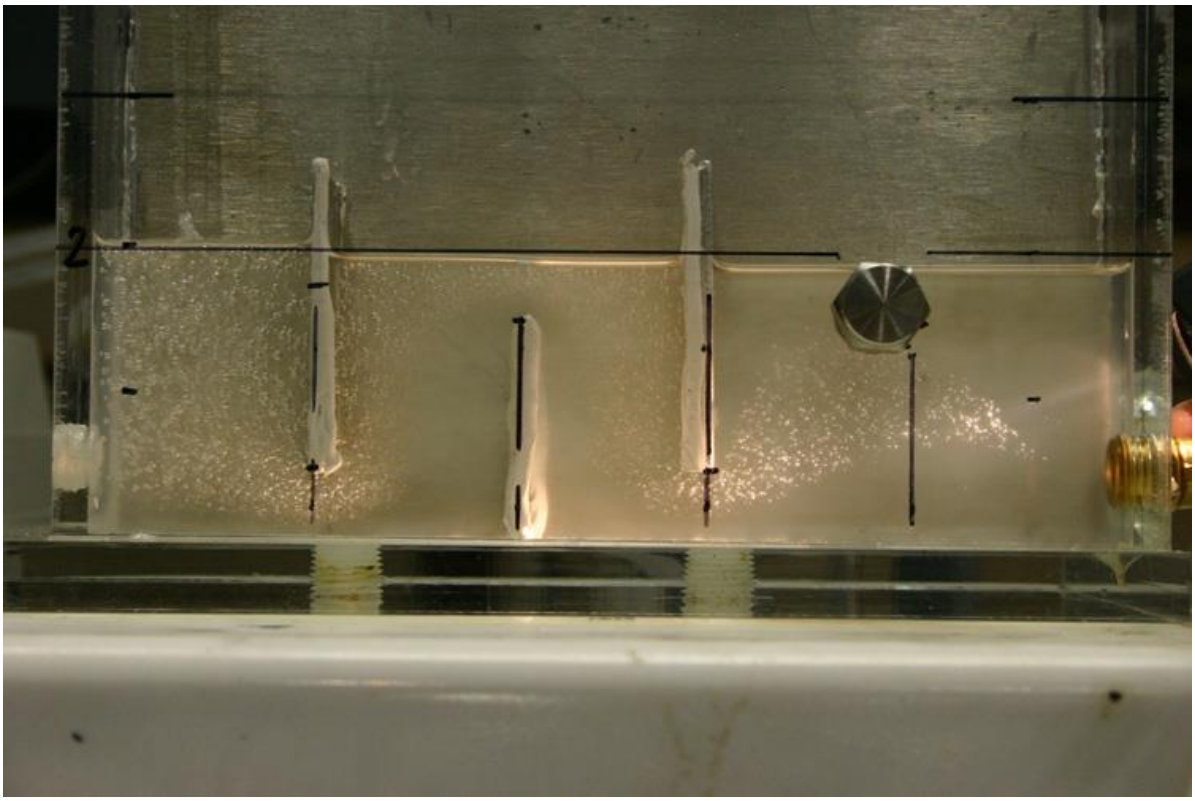


Figure 45. Ambient Pressure, Three Partitions, 15 min

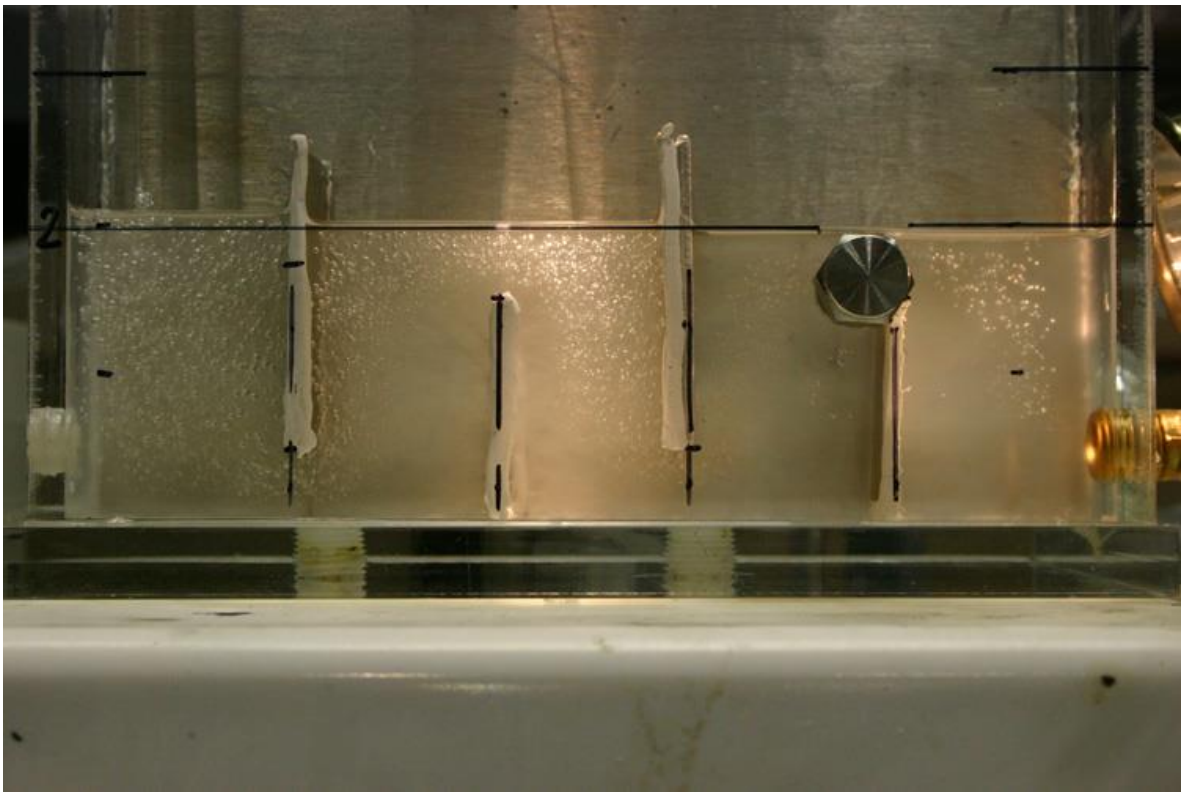


Figure 46. Ambient Pressure, Four Partitions, 15 min

Researchers considered that elimination of small bubbles that follow streamlines could make obstructive refining viable. The text box was sealed and testing was repeated with no partitions and a 10 in. Hg vacuum. Results were the same as in the same test as ambient pressure. The vacuum either did not provide any benefits or was too low to create large enough bubbles to be more easily refined from the silicone oil. The setup was not operable at higher vacuum levels to test the idea of operation at deeper vacuum.

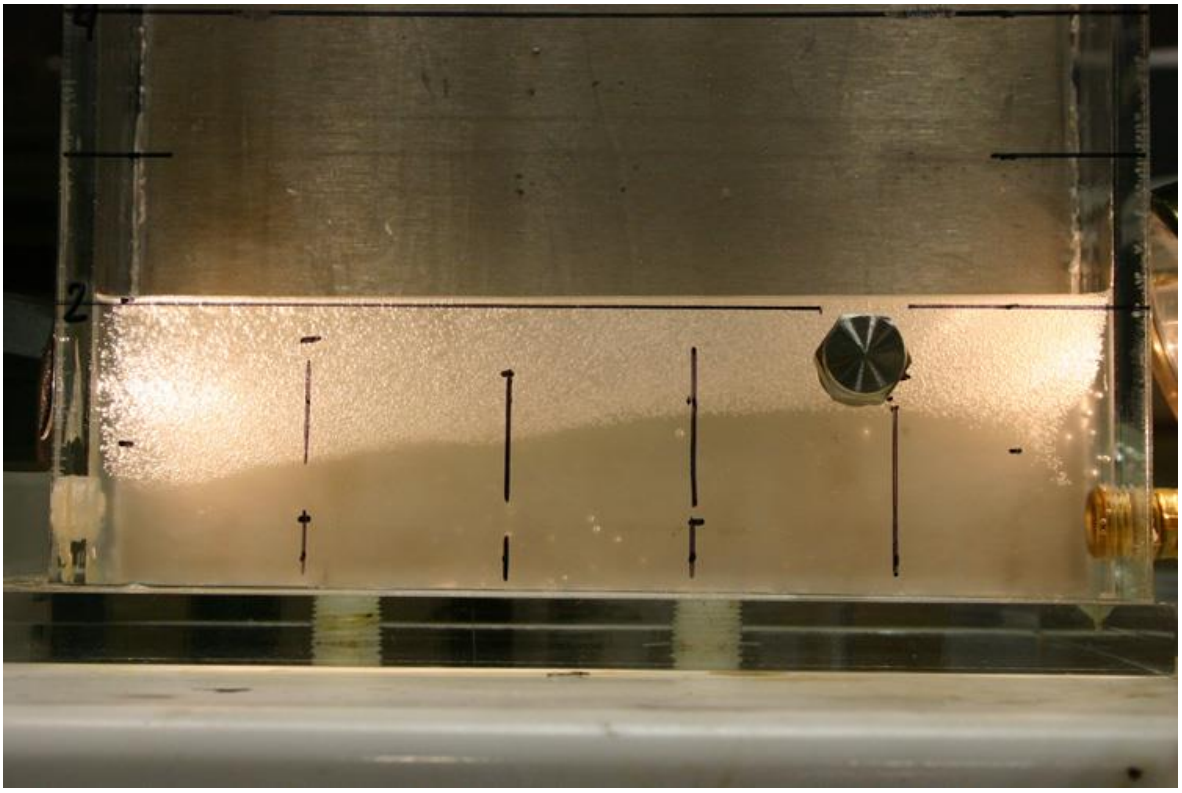


Figure 47. Vacuum 10 in. Hg, No Partitions, 19 min

Testing summarized in the Table below found that introduction of weirs decreased the time for bubbles to move the feed point to the discharge tap. This is the opposite the desired effect of increasing the time for bubbles to enter the discharge port or to be prevented from entering the discharge port. Operating the experiment under 10 in. Hg vacuum had no effect on the time needed for bubbles to move from the feeder to the discharge pot. Higher vacuum may have produced a more positive outcome because bubbles would be increased in diameter.

Table 3. Time From Feeder to Discharge Port of Bubbles in Silicone Oil

Pressure	Weirs	Time from feed to discharge, min
Ambient	0	20
Ambient	1	18
Ambient	2	19
Ambient	3	15
Ambient	4	15
10 in. Hg vacuum	0	19

Thermal Convective Rapid Refining

If a glass product requires refining (and most products do require refining), then a rapid, inexpensive melting process followed by a large, time-consuming refining step makes little sense. Therefore, the project team set a target of a rapid refining process that is industrially is practical, is scalable over the typical range of glass production, is cost effective, does not require any unproven technology, and can meet container glass quality in a residence time of under 30 minutes. The first patent obtained by the team for refining using filtration (U.S. Pat. 7,874,179) was set aside as not yet able to meet these targets on a large scale.

Earlier refining work with a channel after the pilot-scale SCM found that a residence time of an hour or more is needed to decrease bubble content from 30% to 0.3%. This is a significant improvement in clarification, but the process is too slow to meet the goals of the desired rapid refining process step, and higher levels of refining are needed to meet fiberglass and container glass requirement. Work with modeling and simulated fluids showed that refining could not be significantly escalated by using baffles or by putting the liquid under partial vacuum. Modeling did, however, show that the speed of refining can be increased by careful location of heat to the glass in the refining channel.

A new approach was needed to meet the rapid refining targets because none of the proposed approaches met all targets. This realization sent researchers into further evaluation of bubble behavior. Review of modeling and test results from evaluation of other refining approaches led to the development of the thermal convective rapid refining method. The concept of convective-thermal refining is to apply heat on the floor of the refining channel. This creates an upward convective flow of glass that slowly pushes the bubbles toward the surface. The bubbly SCM glass is ideal for this process because the large number of bubbles rise together in a mat that scavenges smaller bubbles by coalescing them with the larger bubbles. Once the bubbles are near the surface, heat focused from above is used to break the bubbles on the surface. This approach is calculated to provide rapid refining that meets and exceeds the project targets. The method was evaluated through computational fluid dynamic modeling (CFD), batch testing with molten glass from the pilot-scale SCM, and continuous testing with a specially designed channel integrated with the pilot-scale SCM.A US patent application has been submitted for the TCRR process.

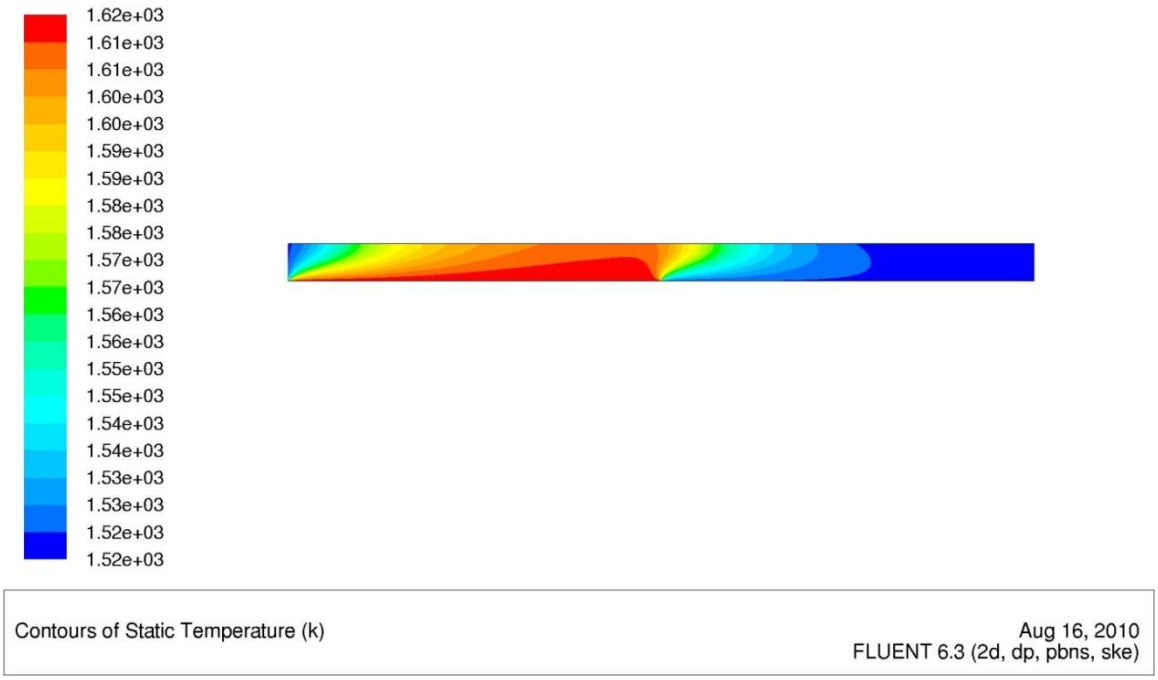
CFD Modeling

Fluent CFD software was used to model glass refining by the thermal convective rapid refining process. Selection of boundary conditions were considered reasonable, but they arbitrary to a specific baseline set of conditions. A common E glass was chosen for all calculations, and the temperature-viscosity profile that E glass was used for fluid flow calculations. Glass was assumed to travel at a uniform velocity along a channel with a depth of 15 cm. (6 inches) of glass. Boundary conditions were varied to evaluate means of applying the thermal convective technique for rapid refining.

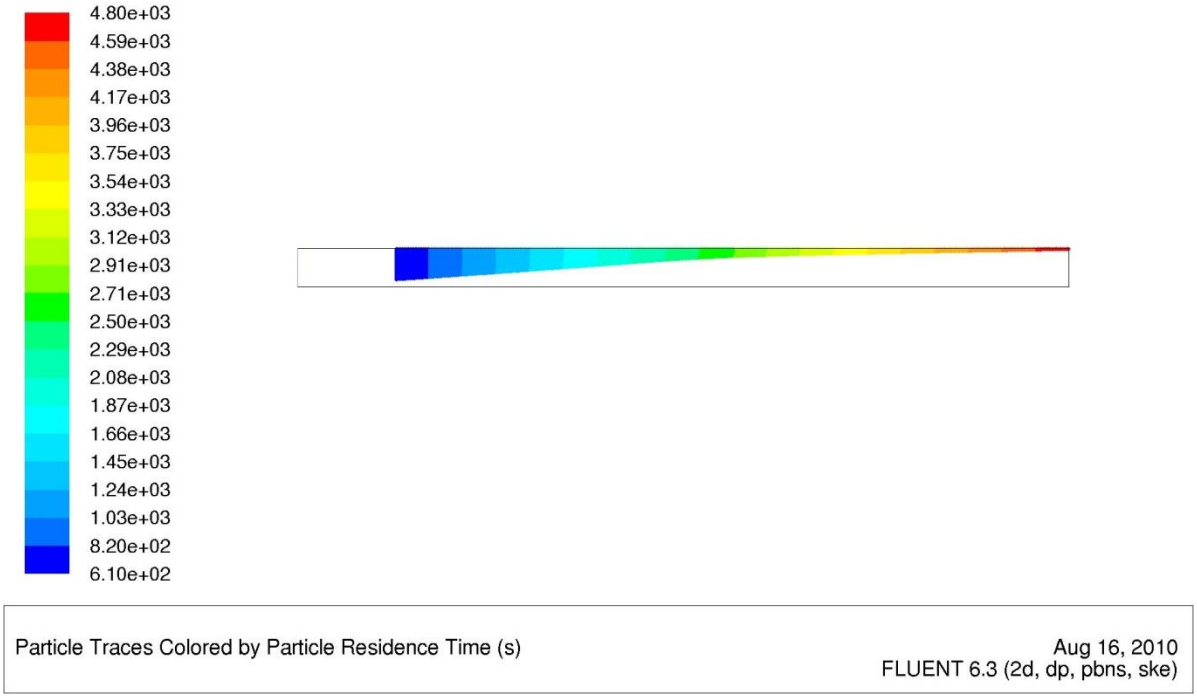
A large number of cases were calculated. Three of those cases are presented here to illustrate the impact of carefully controlling the TCRR process on glass refining. The figures below show the temperature field, bubble location map, and temperature field for a case in which

heat is applied to the floor of the first 60 inches of a 120 inch channel but there is no heat applied to the top surface of the glass.

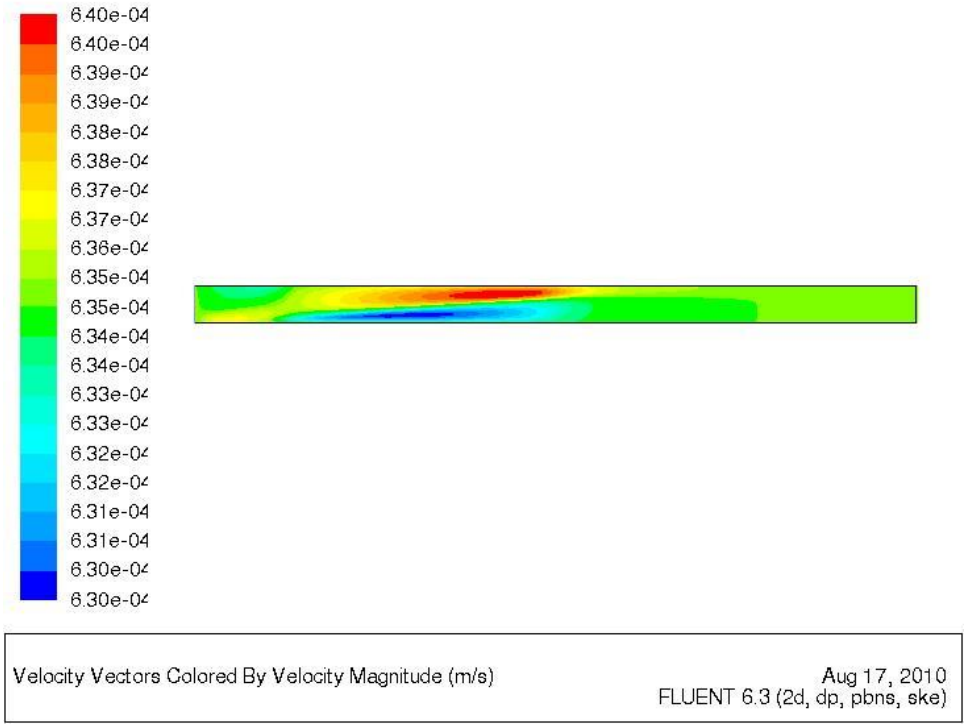
The desired type of temperature profile is created in this case. Warmer glass is on the floor and cooler glass is located toward the top of the channel. This profile produces a convective force that pushes bubbles toward the surface and makes refining faster than can be achieved by buoyancy alone. Based on the conditions of this case, the uniform sized bubbles rise at a rate of 13.78 min/in. No heat is applied to the glass surface. This prevents bubbles from quickly breaking. The figure below shows a long 'tail' of unmelted bubbles as the glass moves down the channel.



(a)



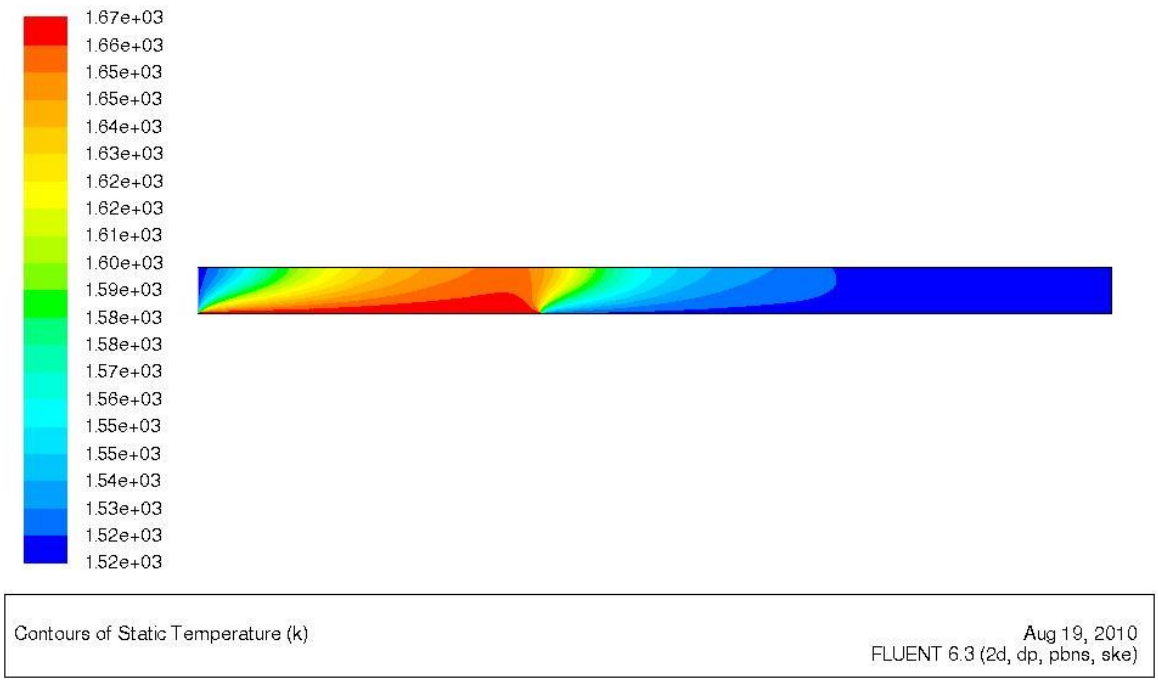
(b)

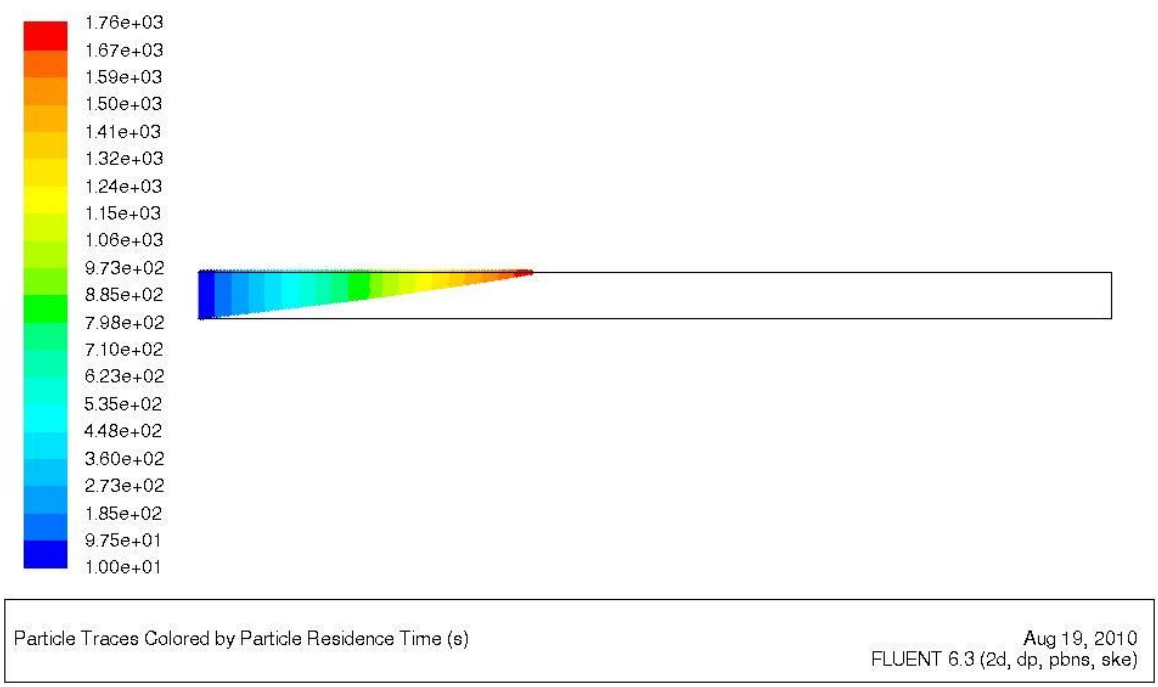


(c)

Figure 48. Temperature: Top – (Heat loss, -1 F/inch) heat flux = -1378 W/m^{2}K, Bottom – (1st 60 inch) 1620K, (2nd 60 inch) 1520K. Bubble rising speed = 13.78 min/inch. (a) temperature profile, (b) bubble map showing bubbly and clear glass, (c) velocity profile

Modeling found that the refining time can be greatly reduced by careful placement of heat in the refining channel. The case below is a good example of the effect. Heat is applied at floor level at the start of the channel. This creates the connective motion that pushes bubbles upward. Further down the channel, where bubbles are reaching the surface in large numbers, heat is applied to the surface to break the foam layer. The calculation determined that the bubbles now only require 4.49 minutes to travel one inch upward. This is a reduction in refining time of nearly 2/3 compared with the case with no heat applied to the melter surface.





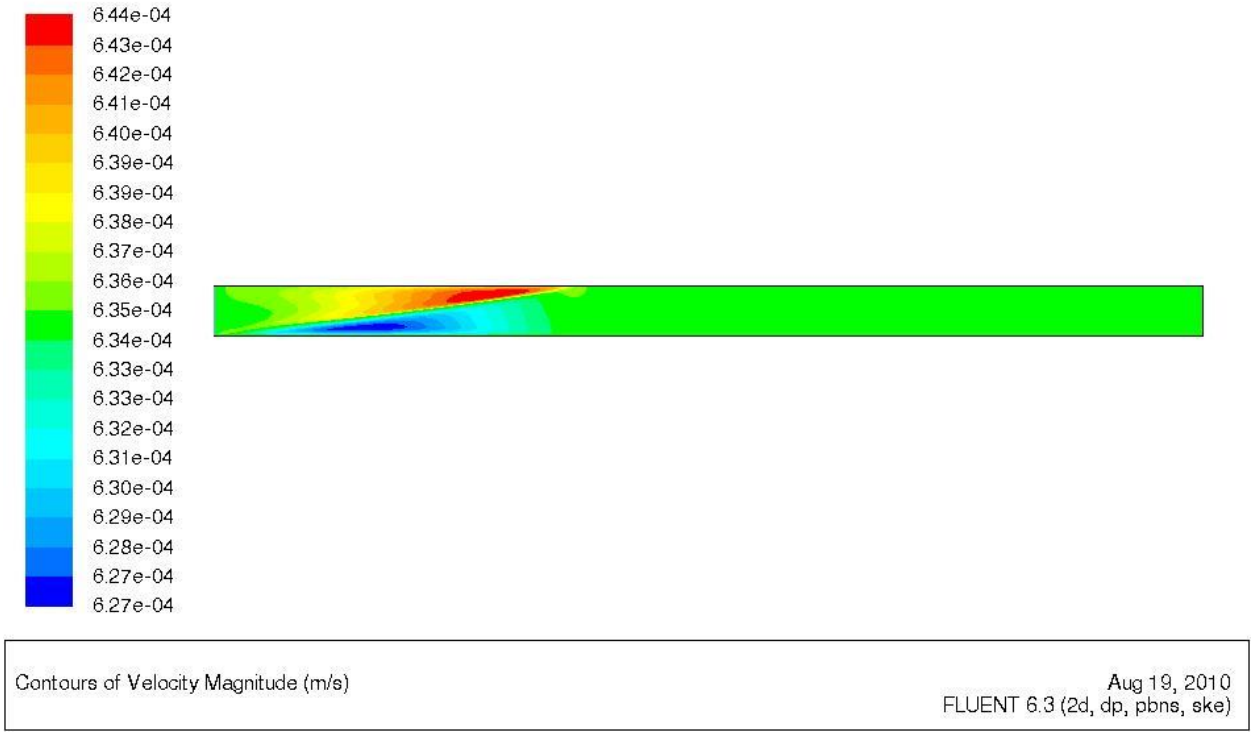


Figure 49. Temperature: Top – [Heat loss (heat flux = -1378 W/m^{2}K), -1 F/inch from 0 to 60 inch and 80 to 120 inch] & [Heat gain (heat flux = 1378 W/m^{2}K), 1 F/inch from 60 to 80 inch], Bottom – (0 – 9 inch) 1670K, (9 – 15 inch) 1670K, (15 – 45 inch) 1670K, (45 – 120 inch) 1520K. Bubble rising speed = 4.49 min/inch: (a) temperature profile, (b) bubble map showing bubbly and clear glass, (c) velocity profile

A large number of CFD cases were calculated. The two cases shown above illustrate the results well. Heat at floor level is most effectively applied in the early part of the channel to create convective motion to move bubbles toward the surface. Applying floor heat later along the channel has little effect on the refining time. The modeling also showed that a burner placed near the center of the refining channel can break the foam later and provide the energy input needed to greatly reduce refining residence time. Modeling did not attempt to optimize the TCRR process. Instead, the CFD work has confirmed that proper placement of heat can shorten reefing time. These promising results encouraged the project team to move forward with batch and continuous TCRR trials with molten glass produced in the pilot-scale SCM unit.

Batch TCRR Testing

Before conducting continuous TCRR testing, the team decided to conduct batch rapid refining tests. A refractory box was built with an opening on the side as shown in the two photographs below. Before testing, a burner firing upward into the chamber heated the refractory to the desired refining temperature. Thermocouples were installed above and below the position of a graphite sample holder that was slid into the refining box. The burner was turned off or kept at low fire during refining to create a temperature gradient in the glass sample that was collected from the pilot-scale submerged combustion melter. The second photograph below shows a graphite sample holder in place in the refining box.

Figure 50. Chamber for Batch Refining to Simulate Thermal Convective Rapid Refining

A series of batch refining tests was conducted to evaluate the thermal convective rapid refining (TCRR) approach. The same glass was used for all tests. The glass was a relatively low temperature of melting fiberglass composition. Viscosity of the glass is –

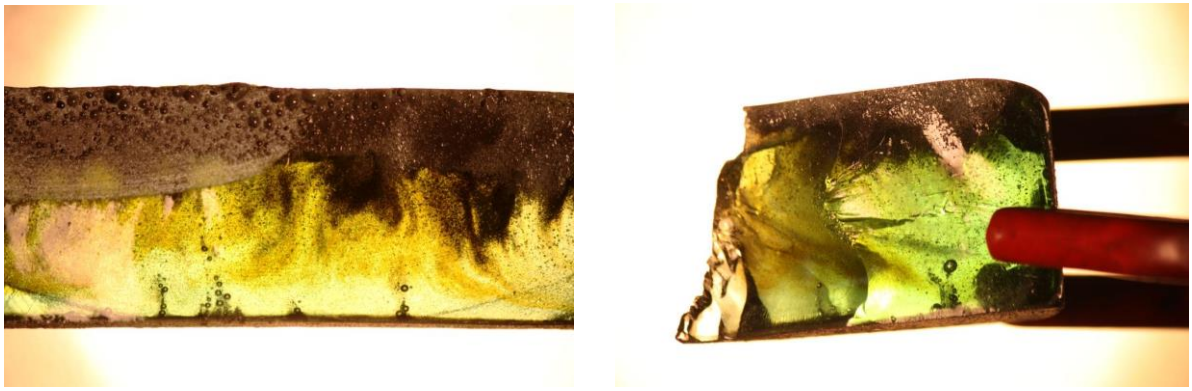
2100°F $\log_{10} \mu = 2.8$
2200ºF $\log_{10} \mu = 2.5$
2300°F $\log_{10} \mu = 2.25$

Two types of rapid refining tests were conducted. 'Cold batch' tests were conducted as follows. The refining box was heated to the desired refining temperature. Approximately 2.1 lb. of bubbly SCM product glass was placed into a graphite crucible. The crucible was then placed into the refining box and heated from below for the desired time at the desired temperature. 'Hot batch' tests were conducted as follows. Glass was melted and held in the pilot SCM furnace. Graphite crucibles were preheated to the desired refining temperature in the refining box. When all temperatures were stable, the melter tap was opened and molten glass was collected in the preheated crucible. The crucible holding the molten glass was then placed back in the refining box and heated from below for the desired time at the desired refining temperature.

Figure 51. Pilot-Scale SCM and Box to Test TCRR Process Step

The cold batch tests confirmed the experimental technique was practical and showed the potential for the TCRR approach. Time-temperature lag from bringing the crucible up to refining temperature and holding at that temperature made these tests less accurate than desired and less representative of a real world process. The hot batch tests provided good temperature control and better simulated real world conditions. The hot batch tests do not simulate the flow conditions and more precise temperature profiles that are possible with a continuous thermal convective rapid refining process.

Cold batch tests were conducted with glass samples held at temperatures of 2175, 2225, 2275, and 2325°F for 15, 20, and 25 minutes. The photographs below show the results at 2325F. Note that the bubbles have risen to the top as a mass while leaving a small number of small millimeter and sub-millimeter bubbles behind.

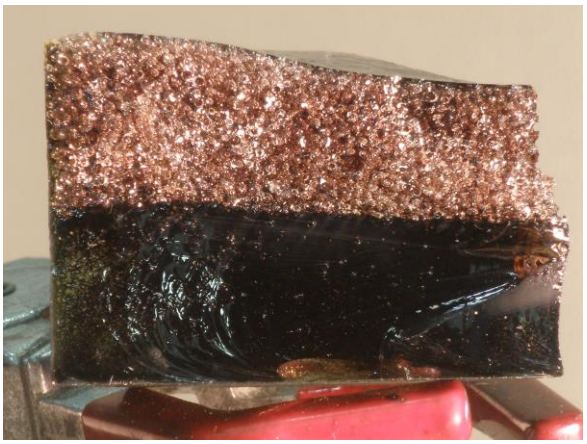


2325°F – 20 minutes 2325°F – 25 minutes

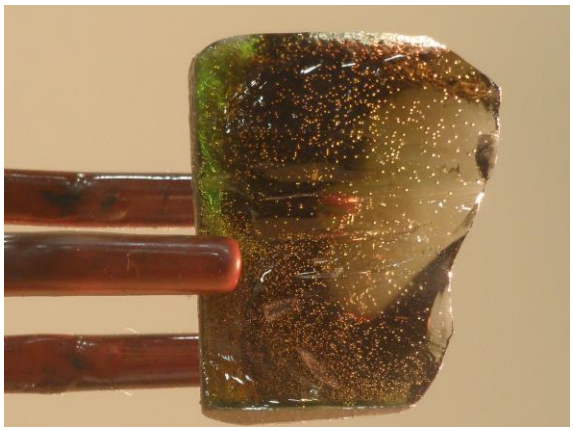
Figure 52. Glass Samples After Cold Crucible Batch TCRR Testing

Hot batch tests were conducted at 2100, 2200, and 2300°F for 15, 20, and 25 minutes. The photographs below show samples refined at 2100 and 2300°F for 20 and 25 minutes.

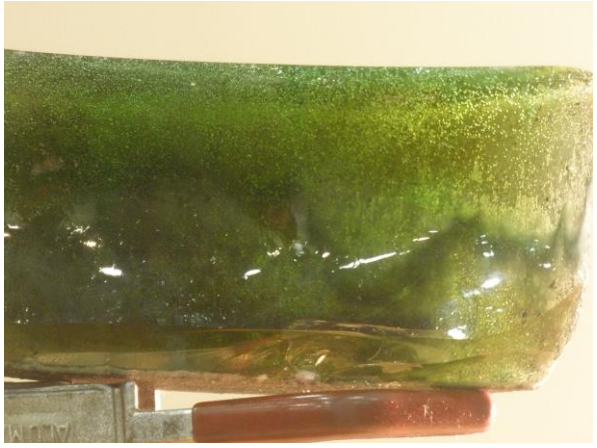
2100°F – 20 minutes



2100°F – 25 minutes



2300°F – 20 minutes



2300°F – 25 minutes

Figure 53. Glass Samples After Hot Crucible Batch TCRR Testing

The hot batch test samples refined at 2100°F show clearly that the bubbles rise as a mass. As the glass refines in the TCRR process, there is a layer of clear glass on the bottom and a layer of very bubbly glass on top. As the samples are held for longer times, the clear layer increases and the bubbly layer decreases.

The hot batch test samples refined at 2300°F show a much higher degree of refining. This is expected since the viscosity is decreased from log 2.8 to log 2.25. At this temperature, the thick bubbly layer is completely gone with only a few bubbles remaining on the surface. Bubbles remaining in the glass are all in the range of 0.5 mm and smaller. There are no larger bubbles left behind. The bubbles look larger in the photographs because the photos are enlargements. Also, the photographs are looking through glass samples that are several inches thick. This increases the number of bubbles that are normally seen in a thin piece of glass. A final note is that many of the remaining bubbles are on the glass surfaces that were in contact with the graphite crucible. Often, bubbles attach to surfaces and do not rise in a viscous liquid.

The photographs of samples from the cold batch and hot batch tests show that the hot batch tests provide a higher level of refining. This confirmation of the TCRR approach is expected to extend further to the rapid refining of glass in a hot continuous TCRR step.

Continuous TCRR Resting

With the promising completion of the batch thermal-convective refining tests with fiberglass, the project team began designing tests to evaluate the thermal-convective refining process on a continuous basis with molten glass from the pilot-scale SCM at GTI. To conduct the continuous thermal convective refining tests, the project team decided that the most straight-forward approach would be to modify the pilot SCM unit with the addition of a refining channel. The front wall (the molten glass discharge end) of the existing melter would be removed and replaced with a panel that has an opening for melt discharge. The photograph below shows the front end of the melter to be replaced. The sketch below shows a simplified depiction of the modified melter with an integrated continuous rapid refining channel.

The melt discharge would be located higher, at 20-25 inches above the melter floor. This higher location and the refining unit design allow the batch feed rate to control the rate of glass processed in the refiner. Raising the refiner higher off the floor also makes working with the product refined glass much easier and safer. The refiner is to be 5.5 feet long, 1 foot wide, 0.5 feet deep in molten glass, and to be built with 1 foot thick refractory brick. This refining channel will hold approximately 400 pounds of glass during operation. Design refining rates are 400-800 lb/h. The continuous refining channel is designed with controlled floor heating using electric elements below the refractory. This approach will enable the project team to assess thermal convective refining over a range of residence time and temperature profiles. The floor heating is arranged to be controlled in three zones to provide the ability to control molten glass temperature profiles. A roof burner is to be installed in the refining channel to help maintain the desired temperature profile. Note also that there are two sampling ports located along the length of the channel. These ports will allow engineers to take glass samples during operation so refining levels and refiner performance can be determined. These ports are located in the channel roof.

Figure 54. Front End Wall of SCM to be Replaced to Allow TCRR Channel Installation

Several tests were planned with a commercial fiberglass composition as the base glass. This glass would be refined over a residence time range of 10 to 40 minutes, at a production rate of 200 to 400 lb/h, and over a temperature range at the floor of up to 300°F above the average melt temperature. Viscosity would be kept in the range of 100 to 1000 Poise to match common glass production conditions. Glass samples were to be collected and analyzed for bubble size and count (bubbles per cubic inch).

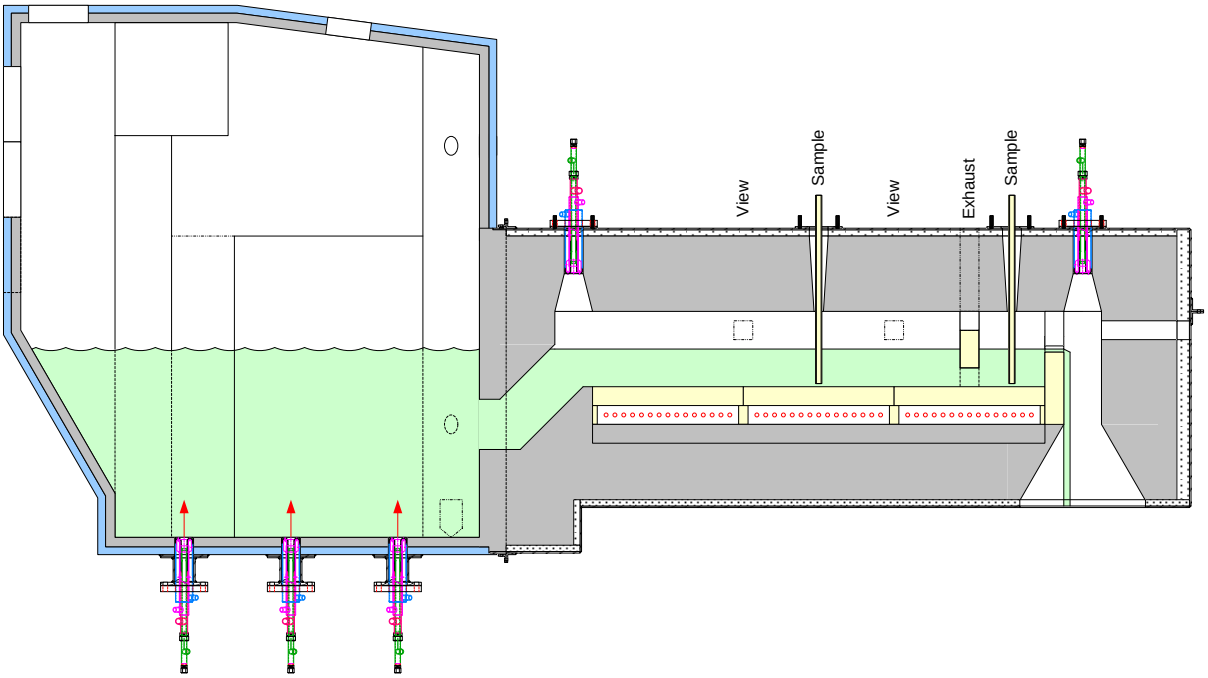


Figure 55. Side View Layout of Pilot-Scale SCM With TCRR Channel

The two layout diagrams below show the final version of the TCRR channel as built. A wide throat connects the melter with the refiner to minimize glass cooling during transfer into the channel. A burner is also positioned in the transition section to help maintain steady temperature at this key location. The channel, shown in sideview and endview had a depth of 6 inches and a width of 12 inches. Two weirs were positioned. The weirs were placed to ensure only glass from the bottom of the channel reached the discharge and to control the depth in the channel. A burner is positioned in the discharge end of the channel to keep glass molten during discharge. Cooling glass at the discharge will have higher viscosity and flow more slowly as temperature drops. This can lead to stoppage of the molten glass, a situation that can end all process operations.

Two probe openings were positioned on the top lid of the channel box. These ports, located before and after the weirs, allowed insertion of ceramic tubes designed to collect glass samples at different depths from the channel during operation. Glass was collected in the hollow-tube probes by dropping the probe vertically into the glass, capping the open upper end of the tube, and drawing the alumina tube up through the sample port. This sampling approached allowed researchers to collect glass samples during RCRR channel operation. Other than replacing the front wall of the melter to accommodate the TCRR channel, no other changes were made in the pilot-scale SCM. All burners, controls, feeder, and exhaust system were kept the same as for earlier SCM trials.

The second sketch below shows the location of the electric elements. Three groups of 15, for a total of 45, elements were located below the top layer of floor insulation. Independent control of power to

the three groups of elements was planned as a means to control floor temperature profile that creates the thermal convective effect.

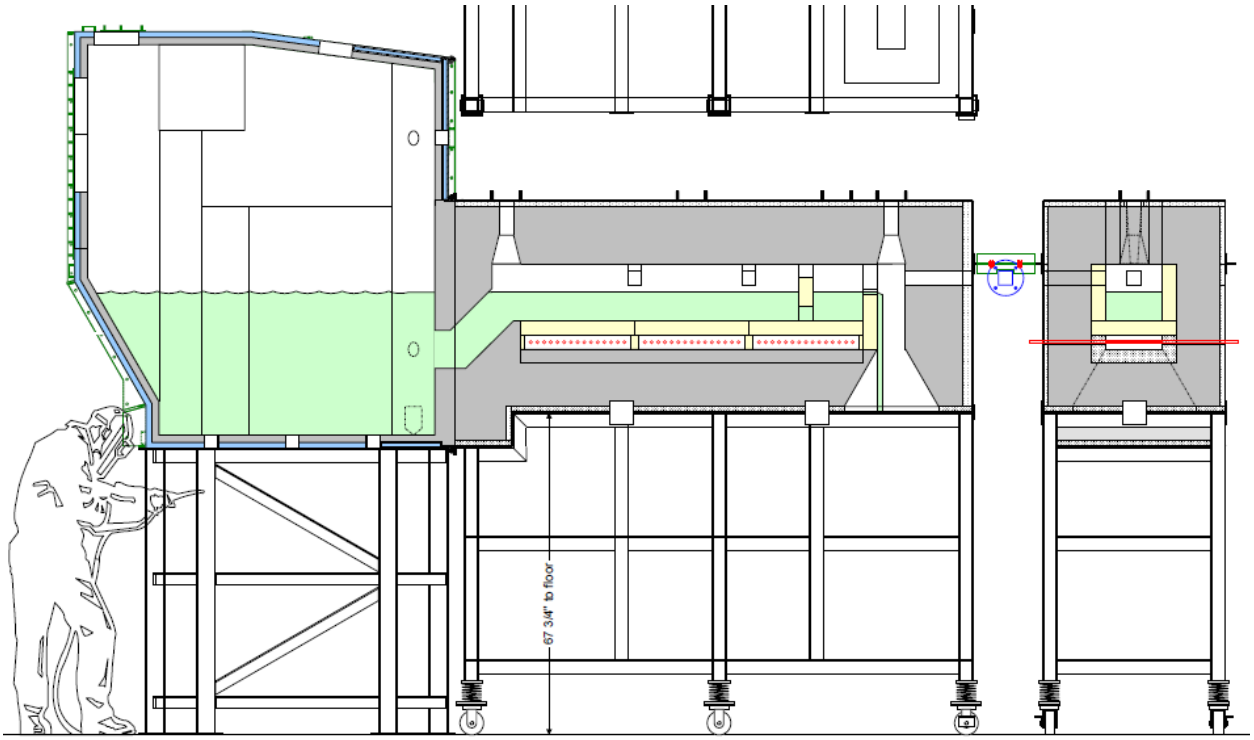


Figure 56. Side View Showing TCRR Dimensions and Framing

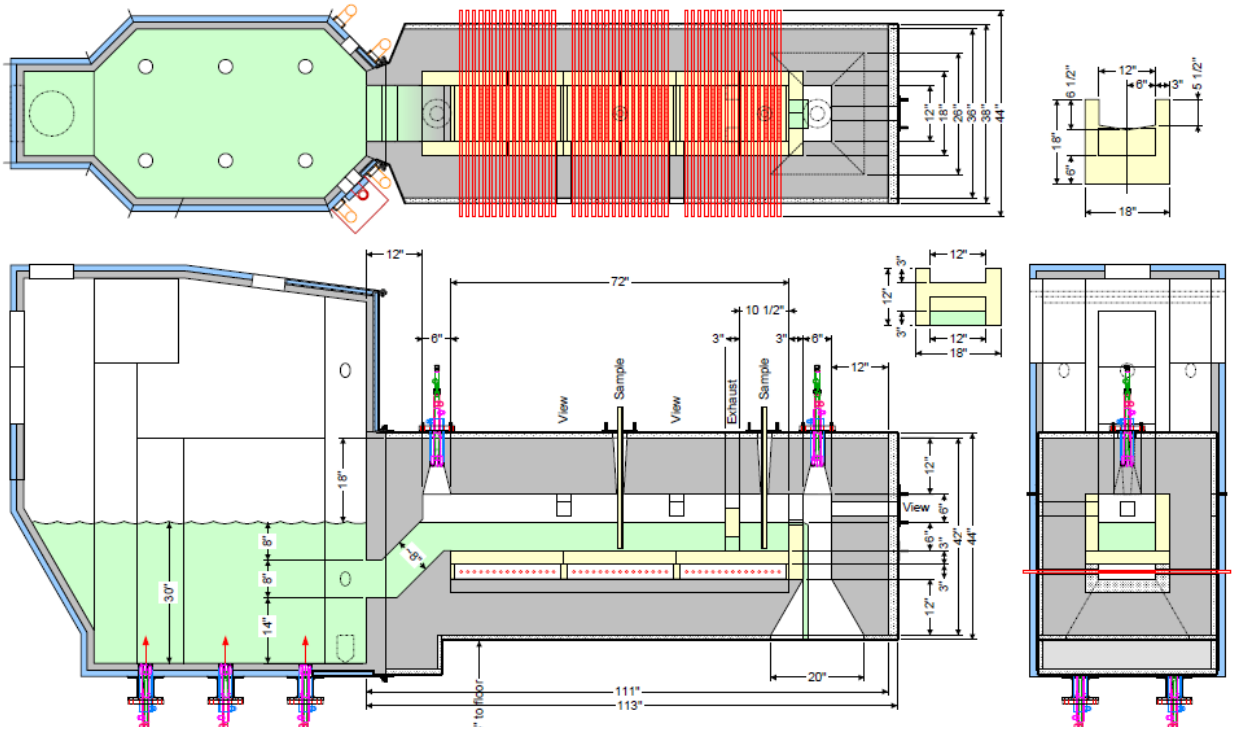


Figure 57. Side View of TCRR Channel Showing Electric Elements

Successful conclusion of this project will be demonstration of the TCRR technology to the glass industry partners and collection of sufficient continuous process data to verify the concept. With that focus clearly in mind along with the objective of meeting fiberglass quality with refining times under 30 minutes, the following independent variables were to be evaluated.

- Glass composition – can be varied but will be the same fiberglass composition for all TCRR tests
- Refining temperature – will vary over a range so that molten glass in the refiner is similar to glass melter temperature and the glass has a viscosity between 100 and 1000 poise
- Refiner residence time – will vary between 15 and 30 minutes
- Refiner temperature profile – temperature profile, with hotter glass at the floor, will have a profile with bottom glass temperature between 50 and 250°F above the bulk glass temperature.

The goal was to reach fiberglass quality. Target quality of product glass is –

- No gaseous inclusions larger than 0.1 mm in diameter
- Gaseous inclusions to be under 1.0 percent of the product glass by volume.

To accomplish the objectives, a minimum of two SCM-TCRR tests were planned. Each test was to be continuous with the molten glass from the SCM processed in the close-coupled rapid refining unit. A Summary of the conditions of the two tests is below.

Table 4. Conditions for Continuous Thermal Convective Rapid Refining Tests

Test	Glass	Temperarue F	Residence Time, min	TCRR Bottom Tecmperature Rise, F
1	JM fiber	2350 to 2550	15 to 30	150
2	JM fiber	2450	30	50 to 250

The first test was to include multiple steady state operating conditions to study variable temperature and residence time. The second test was set to examine the critical impact of changing refiner temperature profile on refined glass quality.

A photograph of the TCRR channel in place and attached to the front end of the pilot-scale SCM unit is shown below. The channel was taken down and sent off site in January, 2015 for installation of refractory. The channel was then reattached, burners and heating elements were installed, and the full system was prepared for testing.

Figure 58. TCRR Channel Attached to Pilot-Scale SCM During Assembly

The melt discharge was located higher, at 20-25 inches above the melter floor. This higher location and the refining unit design allow the batch feed rate to control the rate of glass processed in the refiner. Raising the refiner higher off the floor also makes working with the product refined glass much easier and safer. The refiner is 5.5 feet long, 1 foot wide, 0.5 feet deep in molten glass, and built with 1 foot thick refractory brick. This refining channel holds approximately 400 pounds of glass during operation. Design refining rates are 400-800 lb/h. The floor heating electric elements are commercial equipment controlled by transformers and separate SCRs, installed below the refractory, and in three groups to enable the project team to assess thermal convective refining over a range of residence time and temperature profiles. The floor heating is controlled in three zones to provide the ability to control molten glass temperature profiles. A roof burner installed in the refining channel helps maintain the desired temperature profile. A weir has been included to make sure glass from the bottom of the channel is delivered to the discharge block. The intention is to prevent 'bypassing' of glass on the channel surface to the discharge block that would prevent the clarified glass on the bottom from reaching the discharge block. Note also that there are two sampling ports located along the length of the channel. These ports allow engineers to take glass samples during operation so refining levels and refiner performance can be determined. These ports are located in the channel roof.

A photograph of the TCRR channel in place and attached to the front end of the pilot-scale SCM unit is shown on the left below. The channel was taken down and sent off site in January, 2015 for installation of refractory. The channel was then be reattached, burners and heating elements were installed, and the full system was prepared for testing. The photograph on the right shows the refractory installation in a nearly complete state.

Figure 59. TCRR Channel and TCRR Channel Refractory

Testing was planned with a commercial fiberglass composition as the base glass. The goal was to refine this glass over a residence time range of 10 to 40 minutes, at a production rate of 200 to 400 lb/h, and over a temperature range at the floor of up to 300°F above the average melt temperature. Viscosity was to be kept in the range of 100 to 1000 Poise to match common glass production conditions. Glass samples were to be collected and analyzed for bubble size and count (bubbles per cubic inch). The photographs below show the refining box in operation. The first photo shows the refining box with electric elements in operation to heat the floor. The second photo shows the flame from the burner used to heat the throat between the melter and the refiner. The third photo shows the melt discharge from the refiner box discharge port.

Figure 60. Photographs From Continuous TCRR Testing

Rapid refining testing was carried out in March, 2015. Two fiberglass compositions were tested, a lower temperature C glass and a higher temperature E glass. Both glasses were run in a single continuous TCRR trial. There were not sufficient project resources to make repairs and conduct additional continuous TCRR trials. With both glasses, the team had difficulty in operating the refiner under continuous melt flow. The refiner filled with bubbles of increased size that did not break. This meant that less glass refining or clarification was achieved than expected. The graphs below show temperatures and energy streams for the continuous TCRR test.

The 'molten glass' temperature is the temperature of glass in the SCM measured by a direct contact platinum thermocouple, and this temperature was held around 2500ºF as planned. The TCRR thermocouples (with zone 1 at the inlet and zone 3 toward the discharge) showed significantly higher temperatures at the floor and lower in the bulk glass, also as desired. But the floor temperatures were higher than desired, at nearly 500ºF above the SCM bulk glass temperature. The first two steady state periods were with E glass, and the second two periods were with a lower temperature C glass. The average pull rate of E glass in the first two periods was 11 lb/min, corresponding to a 36 minute average residence time in the TCRR channel. The average pull rate of C glass was 17.5 lb/min, corresponding to a 23 minute residence time in the TCRR channel.

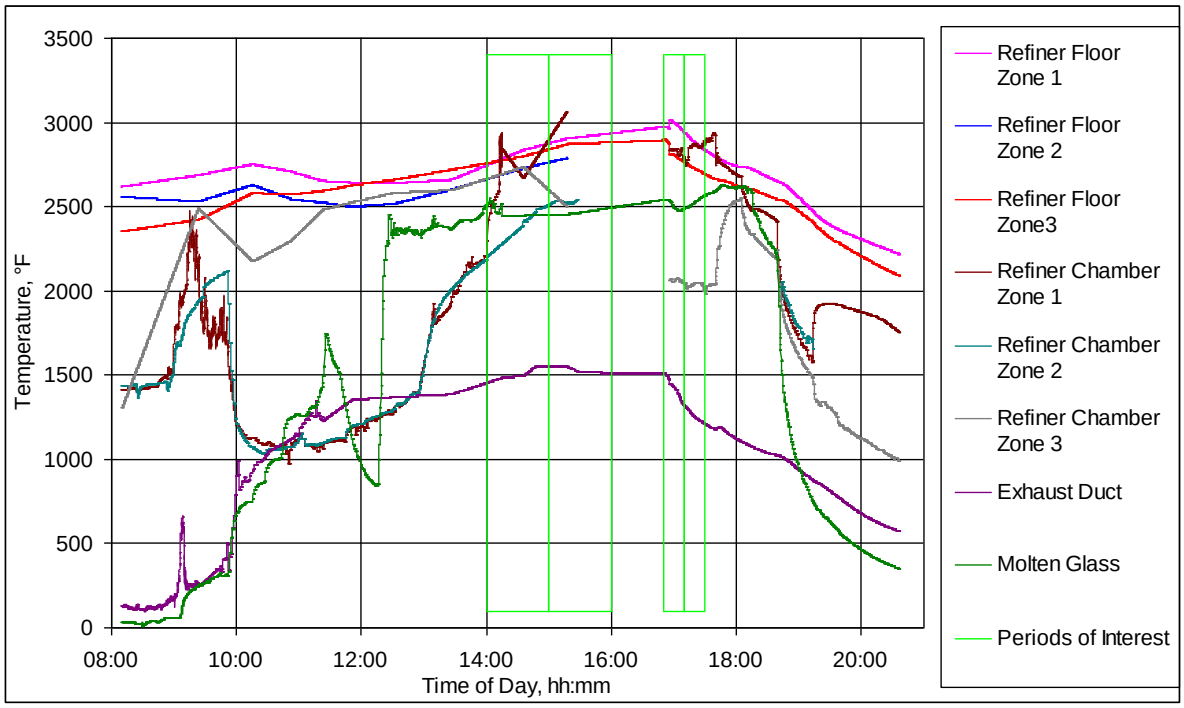


Figure 61. Continuous TCRR Test Data – Flow in Channel is From Zone 1 to Zone 3. Molten Glass is the SCM Temperature Before the TCRR Channel

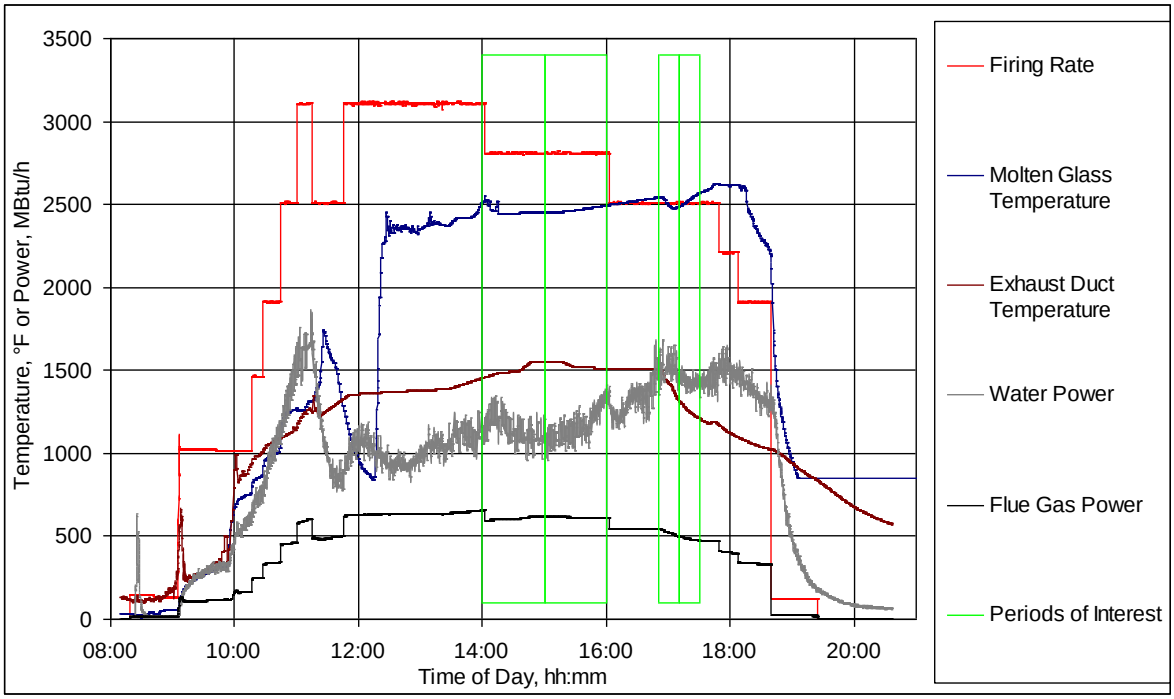


Figure 62. Continuous TCR Test Data for the SCM Feeding the TCR Channel

Operation of the continuous TCRR trial was unsteady. While steady flow was establish through the refining channel for some periods of time, long term stability was not achieved. The glass recovered from the channel discharge was nearly as bubbly as the SCM product glass, with the exception that the largest bubbles were removed. Some refining did occur in the channel. Two of the dip probe samples are shown in the photograph below. The end, showing more refined glass, is glass from the floor of the channel. The table below the photographs show bulk density of glass from the probes. Compared with the typical SCM product glass with a void fraction around 30 percent, the glass at the bottom of the product has a much lower void fraction of around 13 percent. Above the floor, the glass bulk density dropped significantly, indicating void fractions well above the SCM glass void fraction. This suggests that refining was starting to take place, but temperatures were not well enough controlled, and bubbles were not being broken when they reached the surface. Unbroken bubbles on the surface can prevent bubbles lower in the glass form rising, essentially stopping the refining process.

Figure 63. E Glass in Dip Probes From Continuous TCRR Trial. Probes Were Inserted Vertically With the Ends at the Channel Floor

Table 5. TCRR Channel E Glass Sample Bulk Densities

	Bulk density, g/cc	Void content, %
E glass, true density	2.5	--
SCM product glass	1.71	31.6
Probe 1, bottom (floor of TCRR channel)	2.19	12.4
Probe 1, 2 inch up from bottom	1.58	36.8
Probe 1, 4 inch up from bottom	1.39	44.4
Probe 1, 6 inch up from bottom	1.00	60.0
Probe 2, bottom (floor of TCRR channel)	2.16	13.6
Probe 2, 2 inch up from bottom	1.45	42.0
Probe 2, 4 inch up from bottom	1.28	48.8
Probe 2, 6 inch up from bottom	1.34	46.4

Despite the preliminary work with modeling, simulated fluids, and batch glass refining that show positive TCRR clarification, the continuous TCRR testing showed less refining. The reasons for this lack of refining are unclear, but there is certainly some physical effect that is

preventing the refining process from proceeding as expected. Possible inhibitors of rapid TCRR refining include:

- Refining was too aggressive. By overheating the floor of the channel the bubbles were pushed too quickly toward the glass surface. This can cause bubbles to recirculate back into the refined glass lower in the channel.
- The bubbles could have grown in size and created a stable foam with bubbles that could not be broken quickly enough. This would prevent bubbles lower in the glass from rising which would stop the refining process.

The team believes the TCRR process is functional but that the conditions used were inappropriate. Better control of the process by filling the channel more slowly, not overheating the floor which cause bubbles to rise too quickly, and heat in the correct position above the glass would all greatly improve the thermal convective rapid refining process. Further funding, if made available, would allow testing over a wider range of heating conditions with more careful control to enable better convective upward movement of bubble rise and breakage of bubbles at the glass surface.

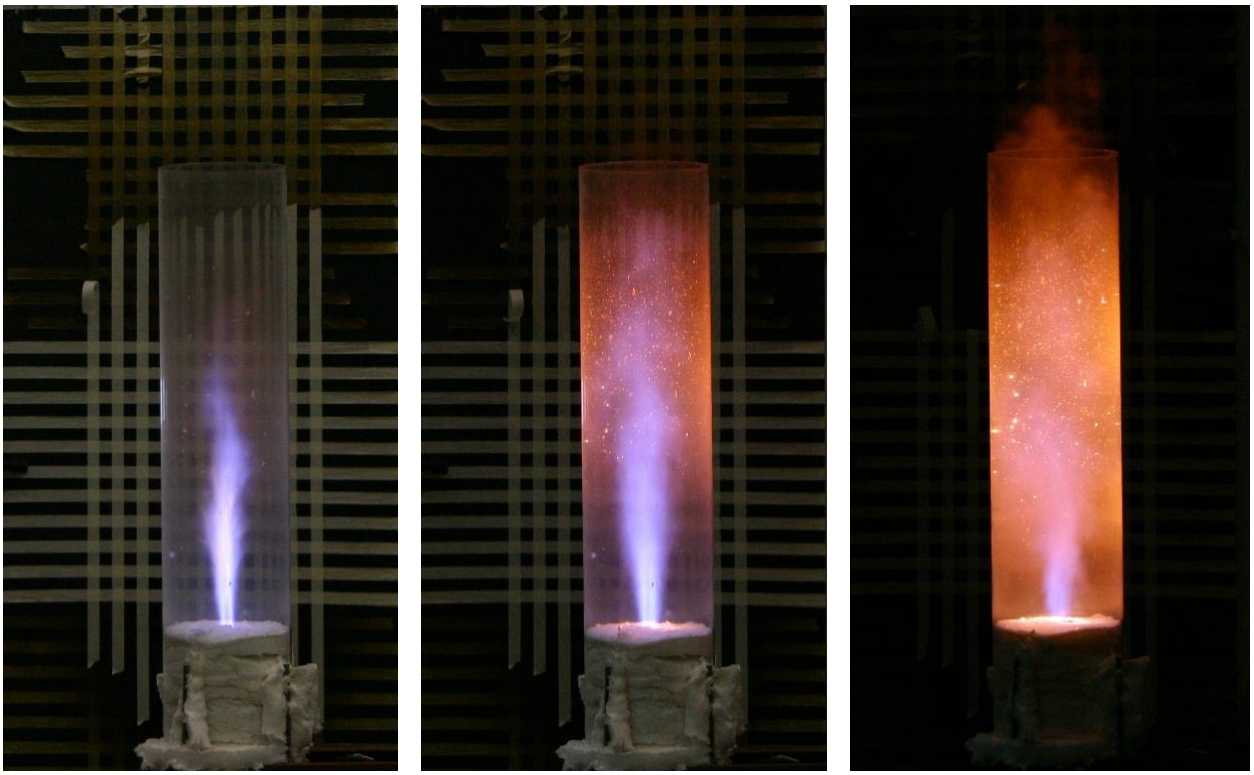
Next Generation Melting System Enabling Technologies

The ultimate goal of the project team, beyond the scope of this project, is to commercialize the NGMS approach to glass production. The NGMS approach is expected to provide significant benefits over conventional glass making methods. Benefits are higher efficiency, lower emissions, lower capital cost, and greater operational flexibility. The development of rapid refining technology in this project is essential to NGMS advancement. Other needed advancements to spur NGMS implementation were also studied in this project. All of these areas of investigation are discussed below.

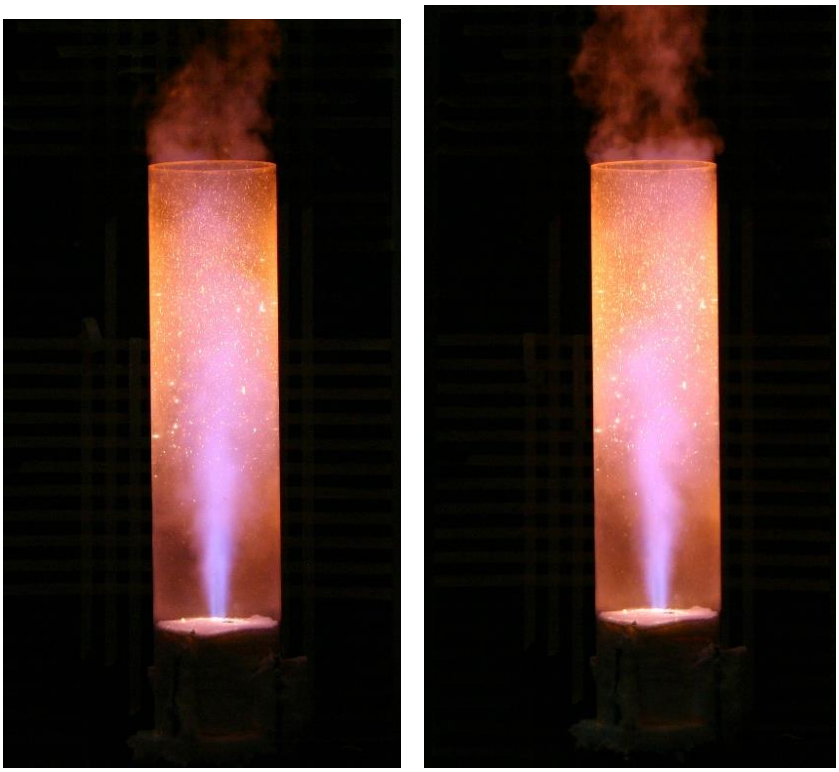
Advanced Oxygen-Gas Burner

A new, more rugged SCM burner has been designed, modeled, and tested. This new burner is expected to have much longer operational life in harsh industrial service. The burner has the added advantage of releasing heat closer to the floor of the melter so that efficiency is improved and batch stones are fully melted and homogenized into the molten glass. Early versions of this burner were tested in open air firing during earlier quarters. That testing determined optimum firing conditions, optimum burner physical dimensions, and the best operating modes to reduce noise. The prototype burner has also been tested in the pilot-scale melter during a melting trial. The burner performed well at high firing rates (>500 SCFH) but pulled flame into the burner nozzle at low firing rates. A minor design change was made after CFD modeling confirmed that dimensions could be changed to eliminate this concern.

A final ‘beta’ version of the new burner was designed. Six thermocouples were installed in this burner to allow cooling data to be collected from an SCM burner in melter operation for the first time. The fully instrumented burner was tested this quarter in open air. Photographs of the prototype burner firing upward over a range of firing rates inside a quartz tube are shown below. Note that the base of the flame is quite ‘stiff’ as desired and that flame length is relatively short. For all of these tests the fuel-oxygen ratio was set with 10% excess oxygen.



150,000 Btu/h 300,000 Btu/h 500,000 Btu/h



650,000 Btu/h 750,000 Btu/h

Figure 64. New SCM Burner Firing in Quartz Tube

Thermocouples were positioned inside the burner body touching the surface of the outside wall. They were located on the inner and outer top of the burner (the tip) and on the inside wall adjacent to the flame. The plot below shows the rise in burner surface temperature as a function of water flow rate with the burner firing 750,000 Btu/h on the open air test stand.

Results are encouraging with no temperature rising more than 75ºF. More importantly, the data show that for water flow rates of 4 gallons per minute or more, all temperature increases are kept to less than 50F. Doubling the water flow rate to 8 gpm only decreases the water temperature increase from 50 to 40ºF. This data provides important guidance for tests to be conducted next quarter in the melt bed.

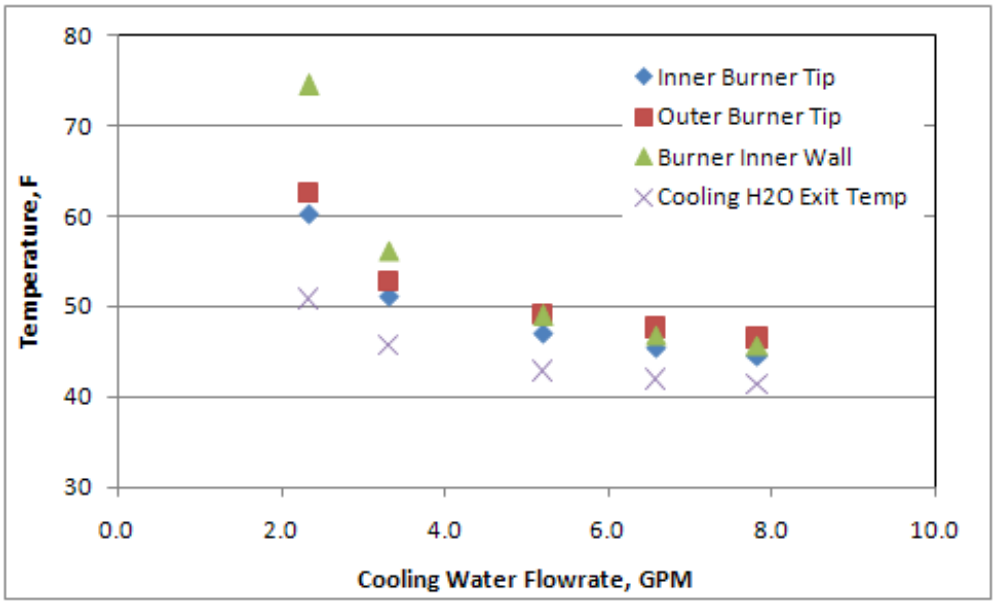


Figure 65. New SCM Burner Temperatures as a Function of Water Flow Rate

An invention disclosure has been prepared for the new burner. One note to SCM operators is that externally the new burner is a one-to-one replacement for the current SCM burner. The new burner has the same gas, oxygen, and water connections, and the new burner is designed to be mounted on the same flange. The differences in the new burner are all internal to the burner body.

A test was conducted in the pilot-scale SCM with non-boron E glass to evaluate the performance of a prototype version of the new oxy-gas burner. This was the same test used to evaluate the new optical sensor for melt observation. The burner was instrumented with six internal thermocouples. Two groups of three thermocouples were installed, with each group consisting of thermocouples attached to the inside surface of the outer wall of the burner's stainless steel housing. One TC was at the burner tip, one on the inside wall, and one on the outside of the burner tip. These TC locations were selected to monitor the effects of 2500°F melt, flame radiation, and potential flame impingement on the burner body, respectively.

Burner surface temperatures were monitored over a ranged of water flow rates through the burner for several different burner firing rates. Initial data from the tests are presented below for one of the groups of TCs. As expected, the burner body temperature increased with higher firing rate. Note that the highest temperature was observed at the burner top, in contact with the

glass, and the temperatures on the inside wall and outer top surface were similar but lower. All three temperatures were somewhat higher for higher firing rate, reflecting an increase in flame radiation heat transfer and an increase in glass melt temperature. Burner surface temperatures were affected by water flow rate through the burner. As expected, higher water flow rates led to lower surface temperatures. Results indicate that any flow rate over 3 gallons per minute will adequate cool the burner. At flow rates over 4 gallons per minute the burner body temperatures become nearly independent of water rate because heat is removed so quickly.

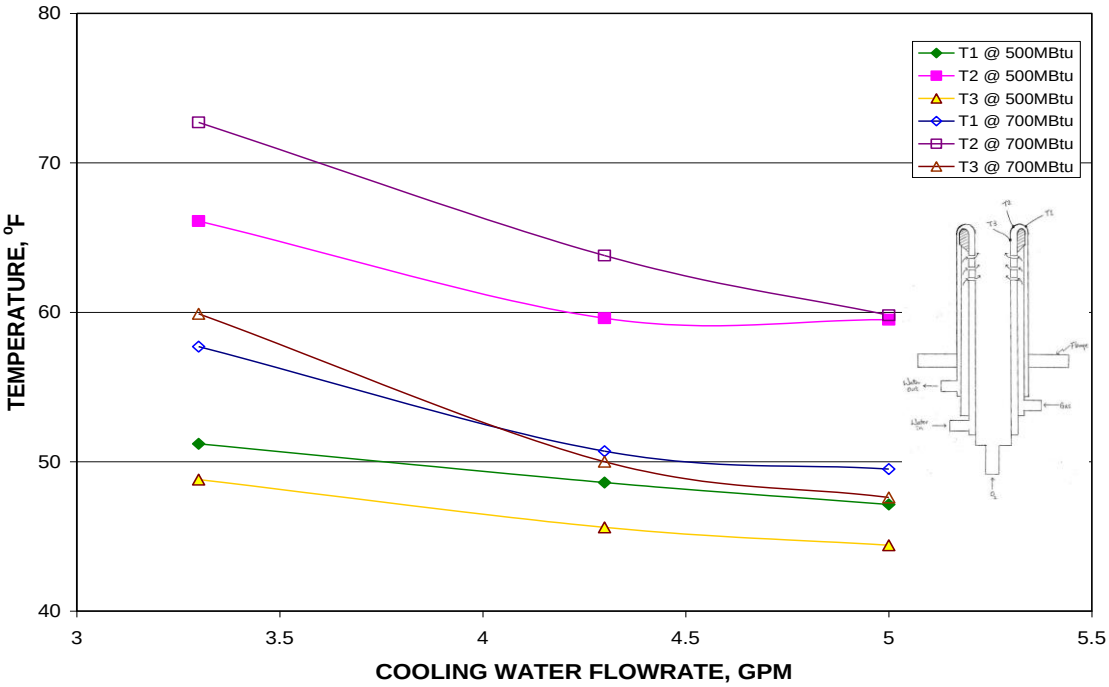


Figure 66. New Burner Temperatures During Pilot-SCM Operation

The new burner was kept in the SCM during later tests. The burner testing evaluated an even wider range of burner firing rates and cooling water rates. The objective remains to develop a burner with a service life of 10,000 to 20,000 hours. To accomplish this, all burner surfaces must be kept at low temperatures to avoid thermal damage. The surface temperatures observed in this test are all well below 100°F, temperatures low enough to provide extremely long life regarding thermal damage. The first test was approximately 12 hours in length, so durability testing was not included, but observation after the test showed no visible wear or surface discoloration of the prototype burner.

Optical Melt Monitoring Sensor

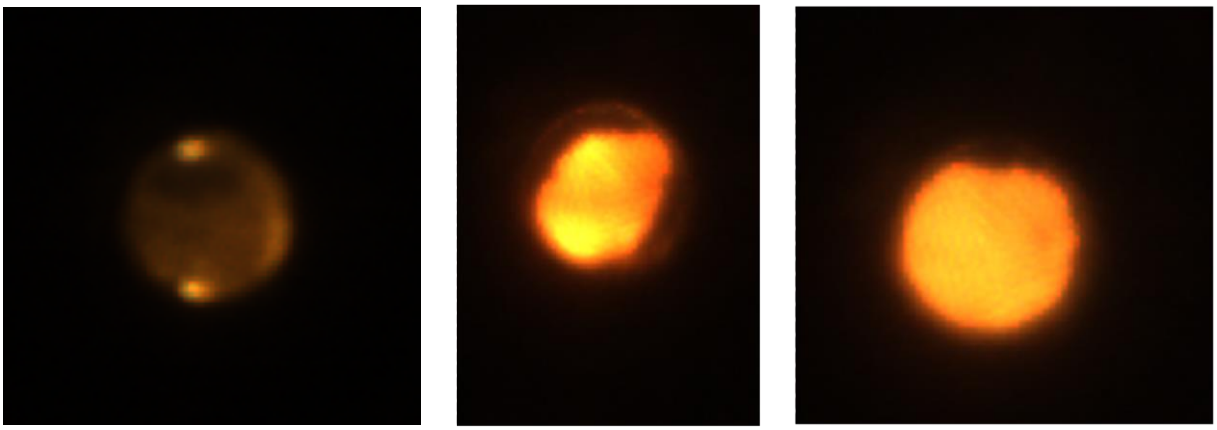
An industrial process to produce glass must be robust and easily monitored. Work on improvements in the original SCM burner and development of the new SCM burner is one way in which a more rugged technology can be readied for industrial use. Process monitoring is also important since a team of combustion engineers will not be constantly observing and controlling the unit and operations will extend over long periods of time up to months. A visual observation sensor would provide important information to the melter operator regarding bed behavior. Potentially this same sensor could be used to obtain real-time process data including temperature

and bed level. For now, the project team focused on development of a working optical sensor. This is a challenge for the high-intensity melting because the melt routinely covers all internal melt chamber surfaces and will certainly cover any observation port.

Most optical sensors require large ports with diameters of 2 in. or larger. The team has always believed that any mechanical means to keep such a port open would be impractical and would fail with operation at such high temperatures and harsh conditions. Air curtains used to keep such ports open are also impractical because extremely large volumes of gas would be need to keep all molten glass away from a large port.

The team has taken advantage of new technology to develop a sensor requiring a much smaller port with a diameter of 0.25 to 0.75 in. At 0.5 in., for example, the port area is only 1/16 the area of a 2 in. port and would therefore require a much lower purge volume to be kept clear. The project team built a sensor with an opening of 0.75 in. and installed a fiber-optic boroscope with a partial view of the melt surface in the melter roof pointing downward. This crude design can be engineered in later models to an opening of 0.5 in. or smaller with a full field of view. At this time, a crude conceptual probe was built and installed to learn if such a sensor can be operated continuously in a working SCM.

The team wanted to test the optical sensor concept during a pilot-scale melt test to evaluate the performance of the new burner in the melter. The sensor was installed and monitored throughout the test. Note that a full melt bed was established, so normal SCM operating conditions during start-up and operation were established. The boroscope images below show two of the six burners (a) during start-up, (b) when the melt bed is half full, and (c) when the burner flames are fully submerged below a full 90 cm. (35 in.) melt bed. Efforts were made using purge control to keep the observation port clear.



(a) (b) (c)

Figure 67. View Inside Pilot Melter (a) During Start-up Showing Two Burners, (b) during Bed Build-up Showing Two Burners, and (c) During Steady Operation With a 90 cm. Bed and Fully Submerged Flames

The observation port was kept open through 4 hours of start-up and the first 2 hours of steady-state operation. Eventually the port became obscured by a layer of frozen glass. The team, however, was encouraged with these results for several reasons. First, the port size can be made smaller. A reduction from 0.75 to 0.5 in. will decrease area by more than 50% which will

increase the ability to keep the port open with a purge. Second, improved purge patterns have been identified and can be implemented in future trials of the optical sensor.

The most encouraging result was observed when the SCM door was opened after the melt test. The glass blocking the sensor view had gradually built up and formed a 6 inch long protrusion from the melter crown that looked like a stalactite. Over 6 hours this protrustion must have slowly grown. The nitrogen purge was not preheated, and this cold nitrogen is projected to have cooled molten glass and allowed the protrusion to gradually grow. Use of a smaller purge, redirection of the purge, and preheating of the purge gas will all contribute to eliminating the buildup of sensor obscuring glass.

Alternative Graphite Melt Discharge Tap

During earlier SCM development, an electrically-heated platinum discharge tap was developed and deployed for the pilot-scale SCM unit. The platinum tap has proved reliable and serviceable for a wide range of glasses and is an excellent research tool. In industrial settings the use of platinum is possible but is regarded as a solution of last resort because of cost and security of the metal. Several means exist for replacing the platinum discharge tap including large diameter throats, floor taps, externally cooled steel alloy taps, ceramic taps, and graphite taps. Several of these approaches have been implemented on commercial demonstration SCM units. In this project the team explored the option of installing a graphite tap. Although graphite will eventually wear and need replacement, the material is so inexpensive that regular replacement is a viable option so long as a graphite tap is reliable and has a sufficiently long service life. To determine the feasibility of using a graphite tap, the team built and installed a new pilot-scale melter discharge tap. Graphite was used for the slide gate and all glass contact surfaces. Shown below are a sketch of the tap and a photograph of the tap in place with the slide gate open.

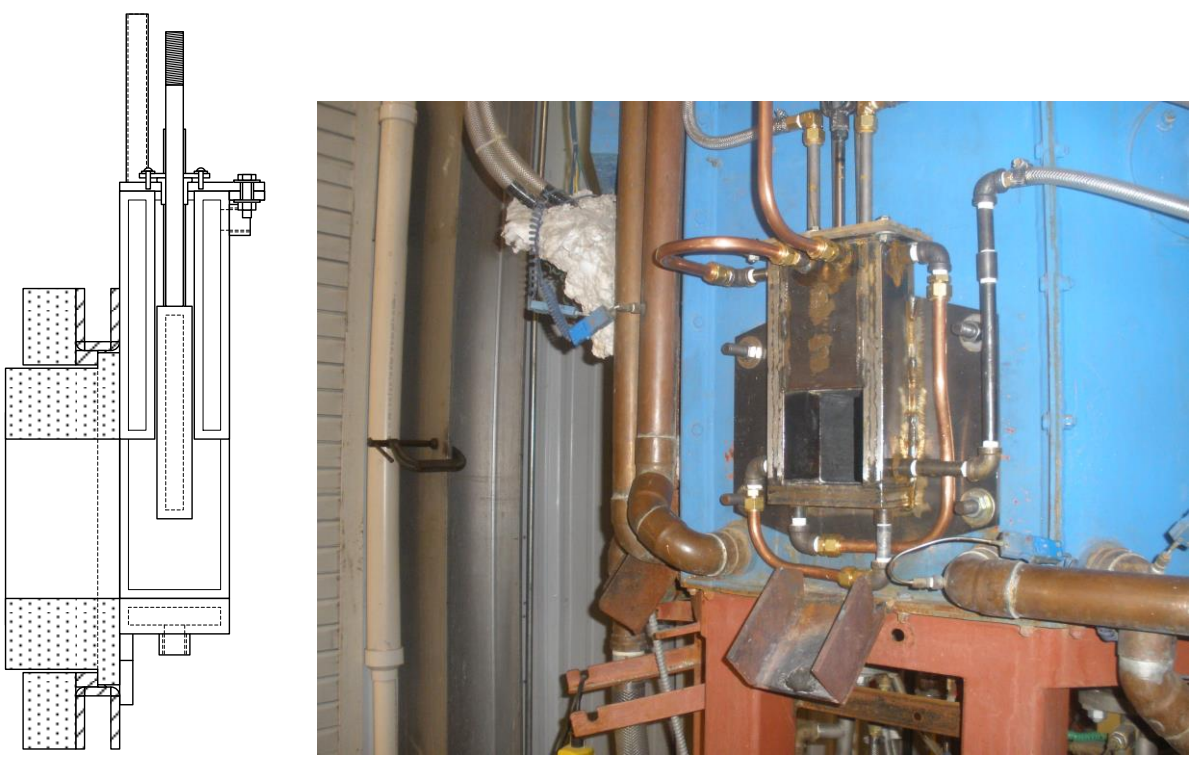


Figure 68. Graphite Discharge Gate

The tap was installed in the melter wall as shown with the left side in contact with the glass inside the melt chamber. The shaded areas are graphite. A vertical water-cooled slide gate is integral to the tap on the outside of the melt chamber. The slide gate was controller with an electric motor and used to control melt flow rate. The objective was to produce a stable melt stream at a control rate between 200 and 1000 pounds per hour. The testing goal was to determine if graphite will survive under glass melt conditions for long enough to conduct several trials before replacing the tap. The graphite can color the glass, making it not suitable for many commercial uses, but this is not a concern for the testing conducted in this project.

The new graphite discharge tap was operated in a pilot-scale SCM trial. The tap was designed to be able to mate closely with the components of a rapid refining channel. Temperature of the glass must be controlled into the channel so that glass viscosity and surface temperature do not increase. Along the length of the channel, temperature must also be controlled, and heat must be applied as desired form below and above the melt. The graphite tap could help maintain stable temperatures as the molten glass moved from the SCM to a refining channel.

The graphite tap failed after several hours of operation during a first SCM trial. While the graphite survived and did not fail catastrophically, the gate piece cracked and could not be operated stably for longer than the single day of a pilot-scale trial. The project team concluded that graphite is not a suitable material for make a discharge tap for a commercial SCM unit.

Refining Channel Improvements Including Advanced Refractory Materials

The thermal-convective refining approach depends on heating on the floor of the refining channel. This heat can be provided in a number of ways. The most obvious, direct approach is to use platinum or other refractory metals that can be electrically heated. The platinum would be in the form of plate, strip, or wire flush with the refiner floor. This approach is being explored by the project team with the help of outside experts in refractory metals and refractory metal heating. If the amount of platinum and rhodium can be minimized, the cost of the metal needed may be reasonable enough to pursue this route. Approaches being studied include the use of thin platinum layers, hybrid metals, and the combination of platinum with molybdenum as appropriate.

A second possible approach is to place heating elements on the floor, or build them into the floor. The heating elements would likely interfere with the desired convective flow path of the glass as it is heated. Some materials, such as molybdenum, may work with certain glass compositions so this approach should not be forgotten. A decision was made to not pursue this approach at this time.

A third approach is to build part of the refining channel floor out of a refractory that is oxidation, corrosion, and abrasion resistant and can also be heated to a temperature high enough to drive the convective flow. Typically, refractories for channels are chosen based on glass composition. For example, AZS can be used with soda-lime glass but not with borosilicate glass. Borosilicate glass requires chromite refractory in order to avoid high recession rates. This approach was considered promising when a consultant confirmed that new classes of refractories may be able to meet our requirements. Testing was conducted at Missouri University of Science and Technology.

Initial tests were conducted with crucibles of molten borosilicate and soda-lime glass held at temperatures of 1300-1600ºC. Coupons of the material were immersed in the molten glass for various lengths of time, up to several days. Analysis found that the material was not corroded or oxidized by the two glass compositions. Plans were made to conduct a final crucible test in which a flat coupon of the material is placed at the bottom of the crucible. The glass would be melted while the coupon is heated to 200°C above the glass melt temperature.

A number of samples were subjected to isothermal, static corrosion tests. These simple tests involved placing a small (~1/2 in^3) specimen into a crucible with glass batch and heat treating to a variety of temperatures depending upon the chemistry of the glass. In every case the actual temperature selected was at least 50°C above what would be considered the maximum temperature that would be encountered in service. Each test also included standard refractory samples that were selected based upon discussions with end-use companies. After heat treatment, samples were removed from the solidified glass and imaged. Care was taken to qualitatively determine the amount of material removed and the degree of glass interaction. In every case the boride based specimen was not wet and not penetrated. There were some limited signs of surface modification that were likely the result of minor oxidation of the boride sample. A series of high resolution images that track the 1400°C, 24 hour test were taken and show this result. It is obvious that the boride specimen performed far better than the standard oxide refractories.

Resistivity measurements were conducted using the ASTM standard technique "Designation: F 76 – 08, Standard Test Methods for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Single-Crystal Semiconductors". Measurements have been made on boride specimens having chemistries ranging from pure ZrB_2 to $ZrB_2$with up to 30 vol% addition of additives such as carbon, silicon carbide, other borides, etc. A range of resistivity values that have been achieved by inclusion of the various second phase additives.

The data provide a means to tailor the composition to provide a specific resistance to allow self-heating. Current resistances require that the specimen be quite thin ((<1mm) for self-heating to be effective. Additional attempts to further enhance the resistivity will be necessary for use with a much thicker specimen. Otherwise significant machining to provide the necessary thickness reduction will certainly lead to additional cost.

Preheated Air Firing Instead of Oxygen Firing

A pilot-scale SCM test was conducted with preheated air. The objective was to better understand energy balances around SCM. A low temperature glass batch was used so the melt bath temperature could be kept below 2200°F. New air-fired burners were fabricated that matched the two burners already available. The melter floor was replaced with a new floor panel, and four air-gas burners were installed. New castable refractory was poured on the floor and cured. Four of the natural gas flow meters were used. New air flow meters and flow control valves were installed to meter and control air flow. Natural gas and air flow were set on ratio and controlled by the SCM flow control system. An air compressor was rented and used to supply air. The air was metered through the control valves and then sent through electric heaters to simulate preheating. The air was preheated to as high as 750°F before being sent to the burners.

The test went well operationally. Despite the greatly increased gas volume and velocity, batch entrainment was not severe. The high exhaust gas volume prevented operation of the

melter at high pull rates. Pull rate was limited to approximately 350 pounds per hour. Product glass was found to have void fractions similar to those of SCM oxy-gas melted glass. However, the large volume of gas may have contributed to a lower density melt bath. This resulted in a lower mass of glass in the bath and a shorter than expected residence time.

The Figures below show how the SCM input energy is distributed between exhaust gas, walls, and gas as a function of glass pull rate.

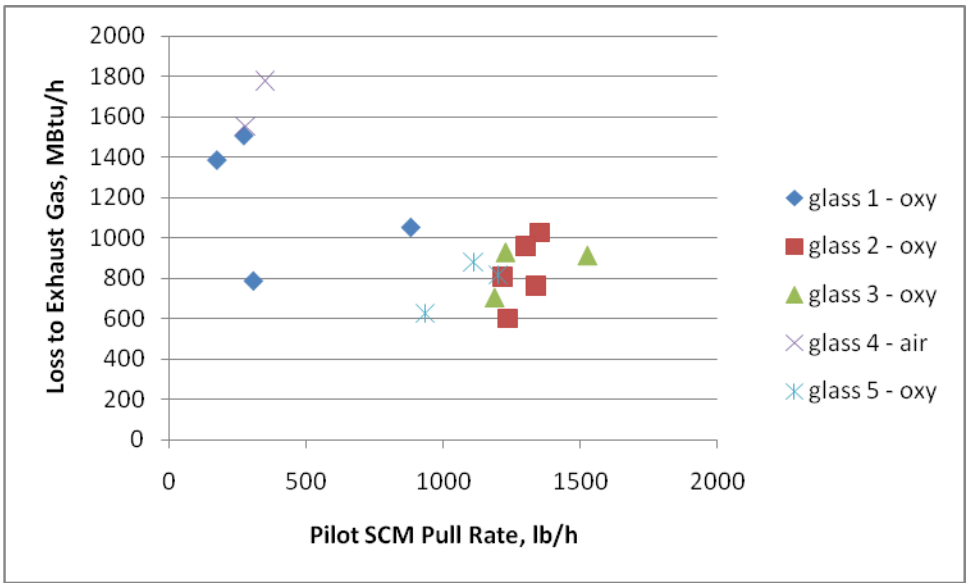


Figure 69. Energy to Exhaust Gas for a Wide Range of Glasses and a Wide Range of Pull Rates in the Pilot-Scale SCM

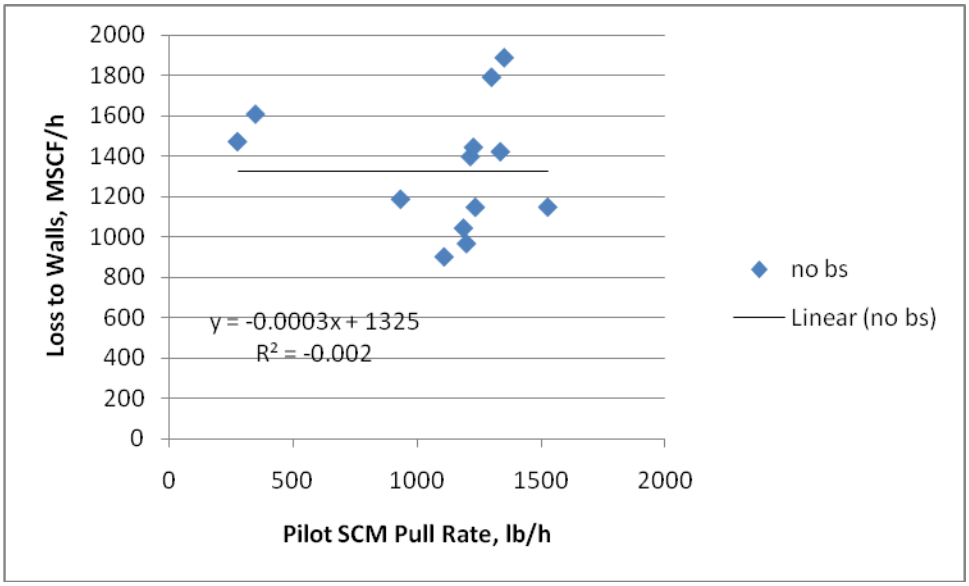


Figure 70. Energy to the Walls for a Wide Range of Glasses and a Wide Range of Pull Rates in the Pilot-Scale SCM

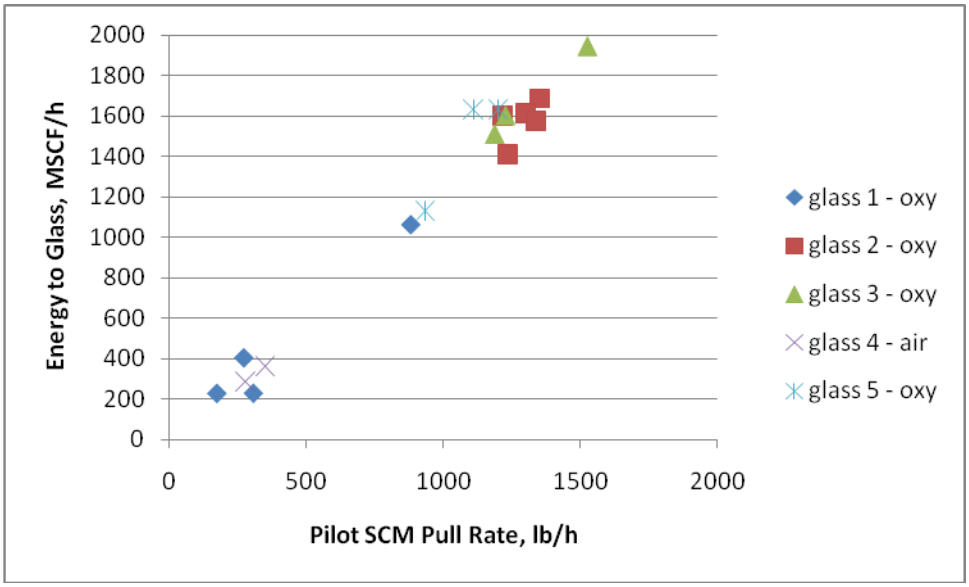


Figure 71. Energy to the Product Glass for a Wide Range of Glasses and a Wide Range of Pull Rates in the Pilot-Scale SCM

Combining data from a wide range of pilot-scale SCM tests covering melt temperatures up to 2900°F shows strong correlations on energy distributions. The energy to the walls is relatively constant in Btus per hour excluding the high temperature borosilicate glass test. The energy to the exhaust gas declines slightly with increasing pull rate. This happens because higher charging rates provide colder batch and melting glass to absorb heat. Therefore energy to the exhaust gas and exhaust gas temperature decrease as pull rate increases. Note that the test with preheated air loses significantly more heat to the exhaust gas than the oxygen-gas tests. This confirms that overall efficiency for preheated air operation will be lower than the overall efficiency for oxygen-gas firing. This is an expected result and similar to the trend seen in other oxy-gas melters relative to preheated air firing.

The energy to the product glass is a strong linear correlation with the pull rate. As pull rate increases, the energy to the glass increases linearly, leading to higher overall efficiencies. This trend holds across all glasses, across all melter temperatures, and across firing with oxygen or air.

Techno-Economic Analysis for a Commercial Scrap Fiberglass SCM

NYSERDA requested the team carry out a techno-economic analysis for the production of insulation fiberglass using submerged combustion melting. The goal was to put quantitative numbers on the benefits of SCM for glass production in New York State. Numbers will vary for different locations, melter sizes, and glass types, but this type of analysis is the first step needed by a glass company in deciding on adoption of a new type of melting technology. A good example of a fiberglass melter is the melters used by Owens Corning in their Delmar, NY plant. The following basis, which is similar to current production rate per melter, was used:

- Glass type – insulation fiberglass
- Glass chemistry – silicate glass containing boron
- Raw material – sand and other minerals in granular form

- Fiberglass recycle – ranging from 0 to 50 weight percent scrap
- Production rate – 5 ton/h
- System components – feeder, SCM (melter), burners, stand and bracing, discharge throat, refining and conditioning channel, gas and oxygen supply and safety controls, control system, and flue gas system
- Estimated burners – 20
- Melter internal dimensions – 125 x 77 in. (internal), 120 x 80 in. (external)
- Refining and conditioning channel – 25 min. maximum residence time, 17.2 ft. length

Glass from the refining and conditioning channel will be assumed to be delivered to bushings for fiber production as in the current practice.

Equipment sizes, component estimated costs, utilities requirements and costs, and equipment layouts will be presented in later reports. The table below summarizes the energy balances, overall gas and oxygen needs, and sizes of melter and refiner for a Next Generation Melter System producing insulation fiberglass from raw materials (no scrap recycle). These figures are shown as a function of glass production rate while recognizing that the base case is a 5 ton per hour system.

Table 6. Summary of Energy Balances and NGMS Dimensions

Rull Rate, ton/h		1	3	5	7
Walls, %		38.0	29.9	27.4	25.9
Glass, %		39.0	47.1	49.6	51.1
Exhaust, %		23.0	23.0	23.0	23.0
Total Energy, %		100.0	100.0	100.0	100.0
SCM Nat Gas, SCF/h		5028	12497	19767	26851
SCM Oxygen, SCF/h		10307	25618	40521	55044
SCM Energy MMBtu/ton		5.13	4.25	4.03	3.91
SCM+Ref, MMbtu/ton		5.33	4.42	4.19	4.06
SCM	Length, in	58.0	92.2	124.6	147.8
(internal)	Width, in	36.0	62.4	77.4	91.7
	Bed Height, in	36.0	36.0	36.0	36.0
	No. of Burners	6	12	20	26
Refiner	Length, ft	7.1	12.7	17.2	19.6
(internal)	Width, in	18.0	30.4	37.4	45.7
	Depth, in	6.0	6.0	6.0	6.0

Melter energy distribution changes with melter size. The larger the melter, the lower the surface to volume ratio and the lower the fraction of energy lost to the walls. The figure below illustrates this effect by showing the energy distribution to walls, glass, and exhaust gas as a function of melter size. Larger melter are more efficient than smaller melters.

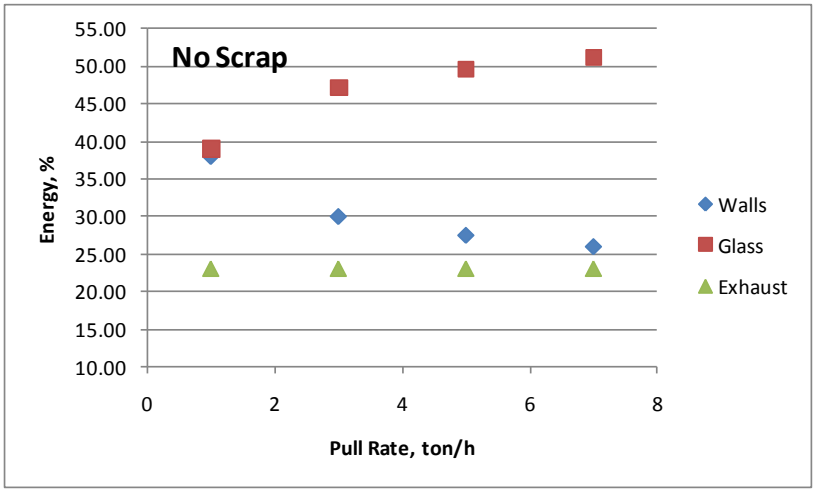


Figure 72. Energy Balances as a Function of SCM Pull Rate, No Scrap

The figure below shows the SCM and SCM plus refiner energy demand as a function of furnace size when no scrap is included in the feed. The Refiner energy requirement is low and is equal to the heat needed to balance wall heat losses. That added heated is provided from below to establish the thermal-convective refining environment.

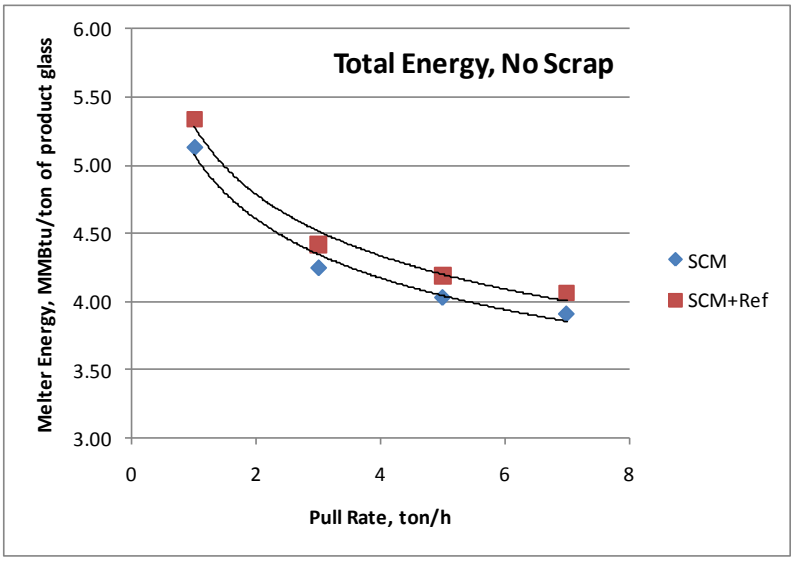


Figure 73. Total NGMS Energy as a Function of Pull Rate, No Scrap

Scrap fiberglass is coated with a resin that serves as a fuel during the melting process. This source of fuel decreases the melter natural gas requirement based on the fraction of scrap in the feed and the loading of resin on the scrap. The figure below shows that at high scrap feed fractions and high resin loading on the scrap the natural gas demand is significantly reduced. The data is plotted for the baseline melter produced 5 tons per hour of molten glass.

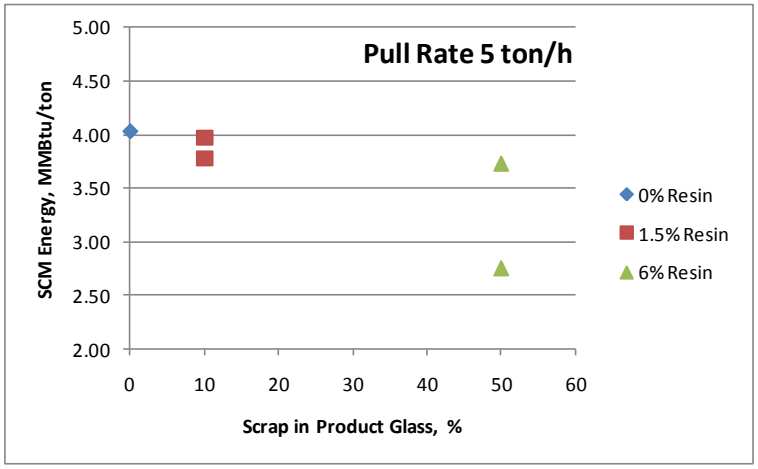


Figure 74. Effect of Scrap Resin Content on SCM Energy Requirement

Glass melting furnaces are built designed to produce certain types of glass. For example, a container glass furnace may be used to produce glasses with different colors and with some changes in composition, but the furnace is never used to produce other types of glass such as flat glass or fiberglass. This practice reflects corporate business practices and plant equipment. Furnaces share many features, but the forming process for different products is distinctly different. To switch from container glass to flat glass would require changing the entire front end of the plant. Equipment would need to be replaced. The buildings themselves might need to be replaced.

Although glass furnaces are not utilized for different products, they are often used to make a range of glasses within a type of product. A fiberglass furnace can produce a wide range of different fibers of different compositions. Changing the bushings allows production of fibers of different diameters. Composition changes are realized by changing the mineral raw materials. Another common variation is to change the amount of cullet used in the raw batch to a furnace. The use of cullet is common in container glass production. Flat glass makers use less cullet because of product quality constraints. Fiberglass is produced with some cullet. Material cost savings and melting energy savings are driving the push for increased cullet in fiberglass production.

The Next Generation Melting System (NGMS) based on the submerged combustion melter is capable of melting glass with any known commercial formulation and with any ratio of batch and cullet. The decision to deploy the NGMS is based on a number of technical and business factors, and each case must be evaluated separately. Information is needed for specific plants and production requirements to determine overall NGMS dimensions, costs, furl and oxygen use, water and electricity requirements, and other critical decision factors.

Glass from the refining and conditioning channel is assumed to be delivered to bushings for fiber production as in the current practice. The figures below show top and side views of the melter and refining section. Dimensions are approximate but give a reasonable idea of the scale of the 5 ton/h system. The melter is built of water cooled panels with a thin layer of castable refractory. The refiner is built of refractory brick. Refiner heating is not shown but includes heating of the refiner floor near the melter and a roof heating zone roughly in the center of the refining section. Overall refiner heat duty is less than 10 percent of the melter heat duty. The sideview sketch shows the depth of the molten bed of glass in the melter and shows the height of the disengaging section above the glass and below the crown. The disengaging section is sized to prevent carryover of molten glass droplets into the exhaust duct.

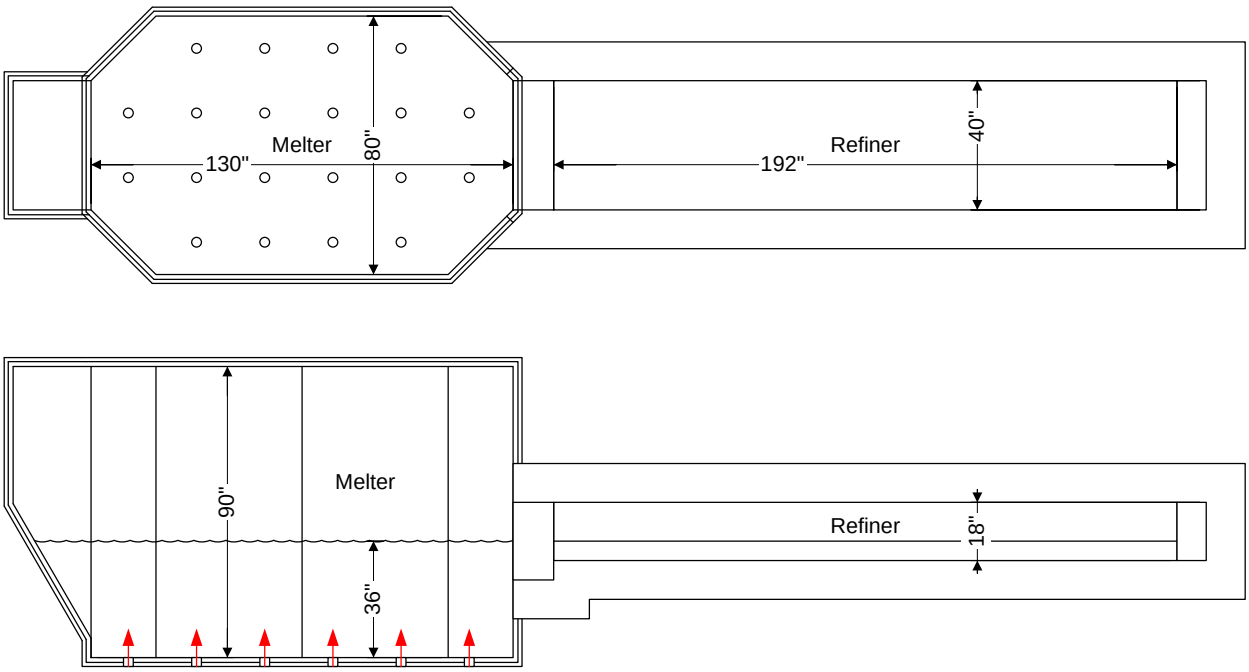


Figure 75. Top and Side Views of 5 ton/h Fiberglass SCM Unit With Refiner

An expanded sideview of the NGMS melting system is shown in the figure below. The melter and refiner are at the heart of the process. Other system components include the feed preparation area, the feeding system with hoppers, the gas and oxygen flow trains, The water system and cooling tower, the exhaust duct, fans and compressors, and the baghouse. This organization of system components is presented as illustrative. Actual plant space availability and process needs can lead to variations in this NGMS layout. System components are shown to scale as much as possible. The high intensity melter is actually smaller than a number of the components.

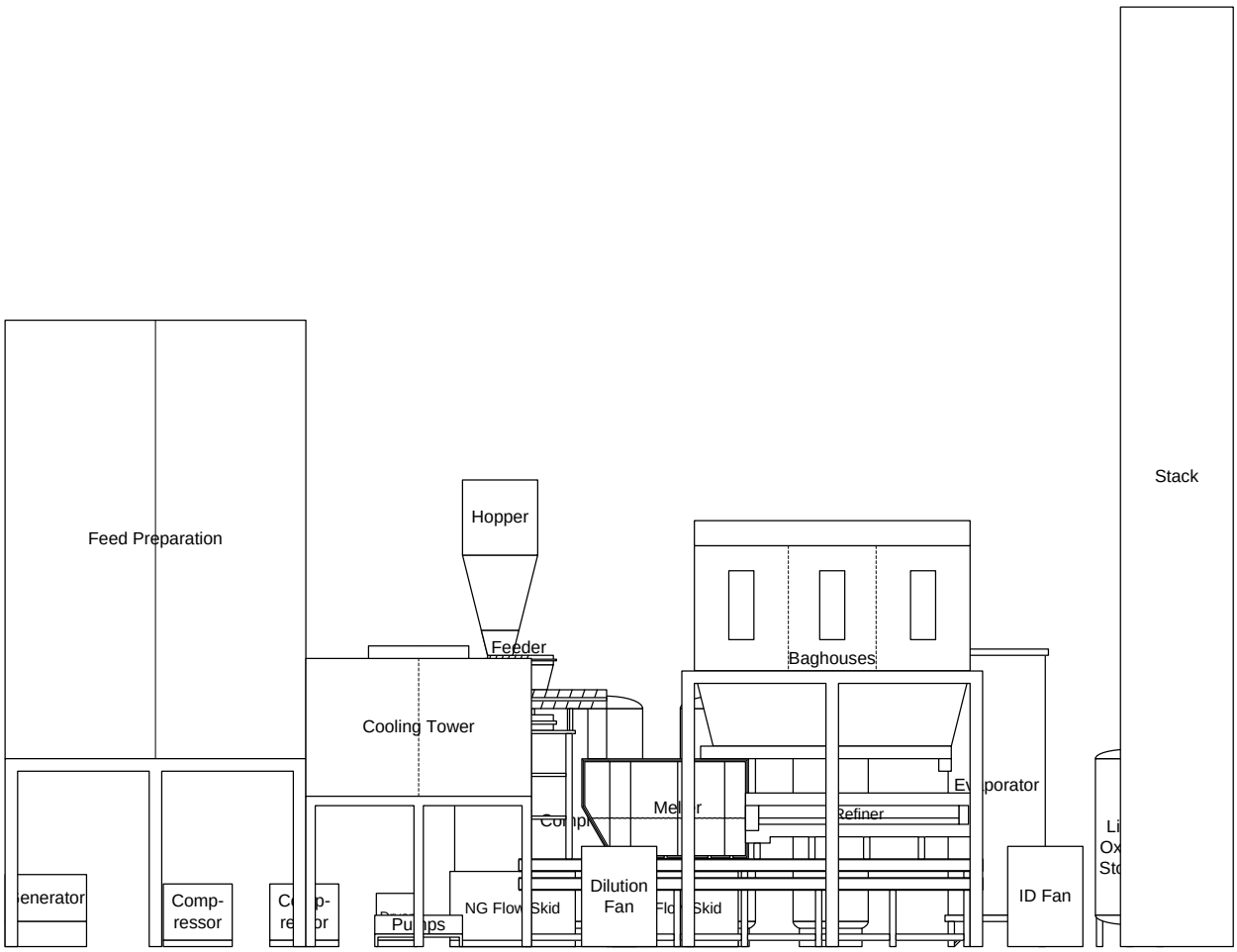


Figure 76. Side View of Full 5 ton/h NGMS Fiberglass Facility

A number of factors influence the location of melters and supporting systems inside a industrial glass plant. The sketch below is provided to show one possible arrangement of NGMS components. This plant layout is one possible arrangement and is not meant to represent a finalized plant design. To keep the sketch easy to understand, A number of smaller components are not show.

The table below provides a summary of equipment sizes, estimated component costs (in 2013 dollars), utilities requirements and costs (in 2013 dollars), and equipment layouts. The first table summarizes the energy balances, overall gas and oxygen needs, and sizes of the melter and refiner for a Next Generation Melter System producing insulation fiberglass from raw materials (no scrap recycle). These figures are shown as a function of the glass production rate while recognizing that the base case is a 5 ton per hour system. The melter is designed to be flexible enough to melt batch containing as much as 50 percent scrap fiberglass.

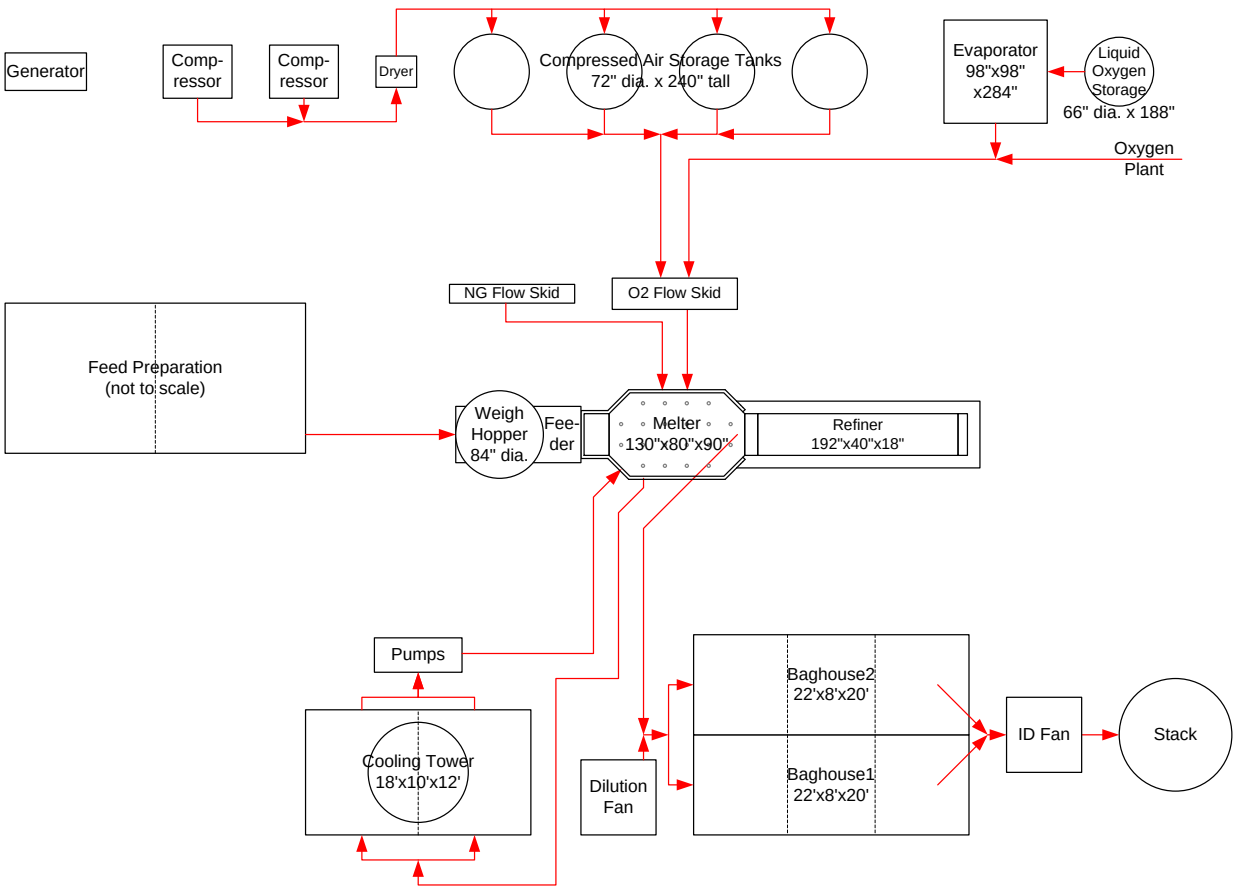


Figure 77. Top View of Full 5 ton/h NGMS Fiberglass Facility

Table 7. Energy Balances and Dimensions of a 5 ton/h NGMS System for Fiberglass

Rull Rate, ton/h		1	3	5	7
Walls, %		38.0	29.9	27.4	25.9
Glass, %		39.0	47.1	49.6	51.1
Exhaust, %		23.0	23.0	23.0	23.0
Total Energy, %		100.0	100.0	100.0	100.0
SCM Nat Gas, SCF/h		5028	12497	19767	26851
SCM Oxygen, SCF/h		10307	25618	40521	55044
SCM Energy MMBtu/ton		5.13	4.25	4.03	3.91
SCM+Ref, MMbtu/ton		5.33	4.42	4.19	4.06
SCM	Length, in	58.0	92.2	124.6	147.8
(internal)	Width, in	36.0	62.4	77.4	91.7
	Bed Height, in	36.0	36.0	36.0	36.0
	No. of Burners	6	12	20	26
Refiner	Length, ft	7.1	12.7	17.2	19.6
(internal)	Width, in	18.0	30.4	37.4	45.7
	Depth, in	6.0	6.0	6.0	6.0

Melter energy distribution changes with melter size. The larger the melter, the lower the surface to volume ratio and the lower the fraction of energy lost to the walls. The figure below illustrates this effect by showing the energy distribution to walls, glass, and exhaust gas as a function of melter size when melting raw batch materials (no scrap fiberglass). Larger melters are more efficient than smaller melters. The same effect of size on energy consumption holds when melting scrap fiberglass. Scrap glass requires less energy because the resin on the fiber serves as a fuel, displacing part of the natural gas, and because recycled glass requires less inergy than raw batch to melt.

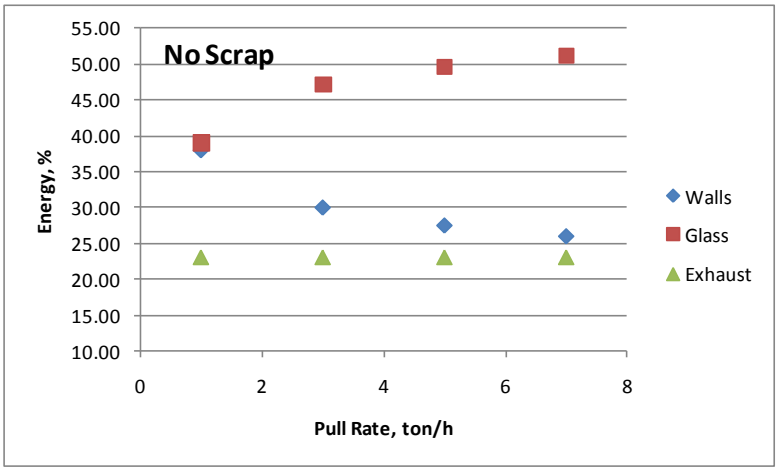


Figure 78. SCM Energy Distribution as a Function of Pull Rate

Estimated electrical requirements for the subsystems of the 5 ton/h NGMS facility for insulation fiberglass are shown below. Vendor quotes are included where this data is available. Feed preparation subsystem electricity requirements are not shown because this power demand is equipment, feed material, and scrap fraction dependent. Several of the power requirements are combined under the Control System power demand.

Table 8. Electricity Requirements for a 5 ton/h NGMS System for Fiberglass

Subsystem	Current	Unit	Power	Unit	Power	Unit	
Feed Preparation		A		kW		HP	
Feeder	143.7	A	119.5	kW	125	HP	5x GTI unit
Feeder Controls	62.7	A	52.1	kW	--	HP	(5^0.8)x GTI unit
Cooling Tower	17.2	A	14.3	kW	15	HP	www.evapco.com - AT 110-218
Pump(s)	69.0	A	57.4	kW	60	HP	www.mcmaster.com - 3x
Flow Skid	--	A	--	kW	--	HP	included w/control system
Dilution Fan	34.5	A	28.7	kW	30	HP	combustiondepot.com - 3x Pyronics 4121 LC-8-OZ-12-10-
Particulate Removal	64.0	A	53.2	kW	--	HP	4x GTI unit (discharge
ID Fan	86.2	A	71.7	kW	75	HP	IAP Fan Pro - Size 23 -
Compressor	138.0	A	114.7	kW	120	HP	emaxcompressor.com - 2x rotary screw 60 hp
Dryer	5.7	A	4.8	kW	5	HP	www.buyquincy.com - QPNC-
Sensors	--	A	--	kW	--	HP	included w/control system
Control System	28.2	A	23.4	kW	--	HP	(5^0.8)x GTI unit
UPS/Generator	0	A	0	kW	--	HP	
Melter	--	A	--	kW	--	HP	included w/control system
Refiner	425.0	A	353	kW		HP	
Total	1074.3	A	893.2	kW	--	HP	

Utility requirements are shown below. The values for natural gas and oxygen reflect a feed with no scrap fiberglass. Demand for natural gas and some oxygen demand decrease as the fraction of scrap fiberglass is increased in the feed to the melter. Oxygen plant rental costs are not included because this would be determined through competitive bids by oxygen suppliers. The chemicals needed for the water cooling tower cannot be priced at this time because plant site water quality will determine the type and quantity of water treatment chemicals. The baghouse bags are specific to the selected equipment brand and cannot be priced at this time without knowing the specific equipment to be use for particulate capture.

Table 9. Utility Requirements For a 5 ton/h NGMS System for Fiberglass

Utilities	Rate	Unit	Usage	Unit	Cost/yr	Unit	Cost
Natural Gas	20.3	MMBtu/h	178,120	MMBtu/y	$841,000	4.72	$/MMBtu
Oxygen	42,700	SCFH	1,870	ccf/y	$1,120,000	30	¢/ccf
Oxygen Plant Rental	43.0	ton/d	50	ton/d	competititve Bid		
Electricity - not counting feed prep	1074.3	A	7,824,157	kWh/y	$524,000	6.7	¢/kWh
Water (make-up)	14.5	gpm	10,191	ccf/y	$10,300	1.01	$/ccf
Chemicals for CT					Site Dependent		
Bags for Baghouse					Equipment Dependent		

This scrap melter estimate assumes the NGMS will be sited in an existing building with available utilities. Because the actual location is not known, some of the associated costs cannot be estimated at this time. The table below provides capital costs for major NGMS system components and systems. Smaller items such as utilities access, valves, tubing, insulation, etc. are not included in the costs. These items would be covered under materials and services in a construction project. Costs are shown in 2013 dollars and reflect vendor quotes when possible and GTI estimates for one-of-a-kind components such as the melter and the refiner.

Costs do not reflect site preparation, installation, or shake down. Although siting is assumed to be in an existing facility, the NGMS will need new gas and oxygen trains along with water lines and a cooling tower. The total capital cost of approximately $1.5 million is less than 10 percent of the cost of a traditional melter of similar capacity.

Table 10: NCMS Capital Costs – 5 ton/d Insulation Fiberglass With Up to 50% Scrap in Feed

Subsystem	Stream	Nominal	Capacity	Unit	Size	Unit	Style	Est. Cost	Basis			Cost Basis	
Feed Preparation	Batch	2.78	6.00	ton/hr				beyond boundary					
	Scrap FG	2.78	3.00	ton/hr					50%				
Feeder	Batch+Scrap	5.56	6.00	ton/hr			Loss-in-Weight	$195,000	10%	LOI	45000	GTI unit	
	Comp. Air	185	200	SCFM	100	psi	Support Structure	Contractor Bid					
Cooling Tower	Water	1130	1,250	gpm	417	ton	ID-Counterflow	$121,000	27.82%		35000	GTI unit option	[[Evapco AT-110-218]]
	All	--	--	--	--	--	Framework & Piping	Contractor Bid					
Pump(s)	Water	1130	1,250	gpm	35	psi	Centrifugal	$15,000	10	°F rise	3	McMaster	8256K48 (460 gpm at 80'wc)
Flow Skid	Natural Gas	20,335	24,000	SCFH	30	psi	Mass Flow Controller	$48,000	1200	per burner		Instrumart	Alicat MCR-1000SLPM-D/1M,CIN,Gas: Methane
							3" Shut Off Valve (2)	$5,700				Lesman	Maxon 5000 300CMA11-BA11-BB21A0
							1/2" N.C. Solenoid	$2,100				McMaster	4738K602
	Oxygen	42,700	50,400	SCFH	30	psi	Mass Flow Controller	$58,000	2.1	ratio		Instrumart	Alicat MCR-1500SLPM-D/1M,CIN,Gas: Oxygen
							4" Shut Off Valve	$5,200				Lesman	[Maxon 5000 400CMA11-BC54-BB21A0]
					100	psi	3/4" N.C. Solenoid	$3,200				McMaster	4738K603
	Comp. Air	1,240	1,240	SCFM	100	psi	3/4" N.O. Solenoid	$2,400	3720	SCFH		McMaster	4738K722
	All	--	--	--	--	--	Framework & Piping	Contractor Bid					
Exhaust Duct - before dillution air	Exhaust Gas	1,051	1,240	SCFM	2.36	ft.dia.	Refractory-Lined	Plant Specific	19.69	ft/s	3		
Dilution Fan	Air	5,845	6,894	SCFM	10	in.wc.	Centrifugal	$12,000	23.00%	1740	°F	Combustion Depot	[Pyronics 4121 LC-8-OZ-12-10-T] (2465 cfm at 10"wc)
Flue Duct	Flue Gas	6,892	8,134	SCFM	3.66	ft.dia.	Refractory-Lined	Plant Specific	5.56	ratio	JFlame		
Particulate Removal	Flue Gas	6,892	8,134	SCFM	350	°F	Baghouse	$626,000			200000	GTI unit	
	Comp. Air	86	100	SCFM	576	bags	Support Structure	Contractor Bid					mpeas.com - Flex-Kleen - 2x 84-WAB-288
ID Fan	Flue Gas	6,892	8,134	SCFM	20	in.wc.	Radial Blade	$29,000			5781	GTI unit second	
Stack	Flue Gas	6,892	8,134	SCFM	6.34	ft.dia.	Brick	Plant & Regulatory Specific	6.56	ft/s			
Compressor	Comp. Air	1,515	500	SCFM	100	psi	2 Rotary Screw 60 hp	$50,600				21825	www.compressedairstore.com - Emax 50 hp
Dryer	Comp. Air	--	500	SCFM			Refrigerated	$6,400					www.buyquincy.com - QPNC-500
Comp. Air Storage	Comp. Air	--	15,000	gal	150	psi	4 Vertical Tanks	$84,000	11362.85	SCF	10.3581	min	www.buyquincy.com - T3750-150
Sensors	Temperature	2,500	3,000	°F	molten	glass	R-Type, Pt Thermowell	$4,900				Omega	XPA-P13R-U-125-30-A-Q-12 [E-Quote# 303908140]
	Temperature	120	400	°F	cooling	water	20 T-Type	$600	18	panels	30	Omega	TQSS-18U-6
	Emissions	8.86	25	%O2			In-Situ Monitor	$10,100	105%	stoich	JFlame		
	Pressure	10	25	psig	4-20	mA	Transducer	$4,900				Lesman	Wika Tronic A-10 Transducer 50426401
	Weight	123,000	160,000	lb	8	cells	Button Type	$5,900				Omega	LC401-25K (25,000 lb)
Control System	All	--	--	--	--	--	PLC	$7,100				Automation Direct	P3000:550;11B;01AC;6x04ADS;4x06DAS-1;08THM;10xRTB
Transformer	Electricity	23.4	30	kW	480VAC to	208/120	delta to wye	$1,400				McMaster	7131K43
UPS	Electricity	1,952	2,100	Wh	24.3	kW	9 units, 5 minutes	$11,000	2700	w	9	APCGuard.com	SMT3000
Generator	Electricity	23.4	35	kW	480	VAC3ph	NG/Propane,liq.cooled	$13,700				Grainger	2ZNG5 - Generac QT03624KNAX
Transfer Switch	Electricity	23.4	60	kW	480	VAC3ph	Automatic	$1,200				Grainger	3HFD8 - Generac RSTN100K3
Melter	Fuel	20.3	24.0	MMBtu/h	20	burners	SCM	$39,400	49.18%	3+4x4+3	pattern	Ertl	\\C-More:T8C;PGMSW;AC
	Glass	5.00	5.00	ton/hr	130"x	80"	Low-Iron Castable	$141,000		1017		GTI unit	
	Water	1130	1,250	gpm	35	psi	Carbon Steel	$143,000	2.00	MMBtu/ton		GTI unit	
Refiner	Glass	5.00	5.00	ton/hr	40"x	192"	Low-Fe Refractory Bric	Contractor Gid					
					12"	high	Carbon Steel	$32,400					
	Heat Input	0.77	1.00	MMBtu/h	293	kW	72 1.25in SiC Elements	$64,000			163.8	GTI Unit	24.6x1
		100	125	A	480	V3Ph	4 Phase Angle SCRs	$9,000			2239	Omega	SCR 39P-48-125-S9
Structure	Steel						I-Beam,Channel,Angle	Plant Specific					
Piping	Natural Gas						Carbon Steel	Plant Specific					
	Oxygen						Stainless Steel	Plant Specific					
	Water						Cooper/Carbon Steel	Plant Specific					
	Air						Carbon Steel	Plant Specific					
Electric	Electricity	1074.3	1200	A			Wire/Cable/Conduit	Plant Specific					
									1206.802	ft3/d			
									9027.509	gal/d			

Utilities	Rate	Unit	Usage	Unit	Cost/yr	Unit	Cost					
Natural Gas	20.3	MMBtu/h	178,120	MMBtu/y	$841,000	4.72	$/MMBtu				industrial:	www.eia.gov/dnav/ng/ng_pri_sum_dcu_nus_m.htm
Oxygen	42,700	SCFH	1,870	ccf/y	$1,120,000	30	¢/ccf				r-o-t	[www.chemicool/elements/oxygen: 0.1¢/lb]
Oxygen Plant Rental	43.0	ton/d	50	ton/d	competitive Bid							
Electricity - not counting feed prep	1074.3	A	9,824,157	kWh/y	$524,000	6.7	¢/kWh				industrial:	www.eia.gov/electricty/monthly/xls/table 5 03.xlsx
Water (make-up)	14.5	gpm	10,191	ccf/y	$10,300	1.01	$/ccf				industrial U.S.:	search.oecd.org/officialdocuments/displaydocumentpdf/?doclanguage=en&cote=env/epoc/geei(98)10/final
Chemicals for CT					Site Dependent						3	gpm/100 ton	www.gorhamschaffler.com/tower_makeup.htm
Bags for Baghouse					Equipment Dependent						0.1	%	www.jutzi.com/usercontent/documents/CoolingTowerCalculations.p
											7	CR	www.eng-tips.com/faqs.cfm?fid=1152

A number of subsystem costs have not been established for various reasons. All steel framing is listed as Contractor Bid and would be priced based on bids received during the engineering and fabrication periods. The exhaust duct from the melter to the dilution air fan and the flue from the dilution air fan to the baghouse cannot be priced because their lengths would be plant specific. Likewise, the stack may or may not be needed. The melter may be tied into an existing stack, and the stack specifications will be site specific. Costs of the piping and electrical (wiring and conduit) would be bid competitively and would be plant specific, so these costs also cannot be estimated accurately at this time. Overall, the largest cost items have been priced either through vendors or by GTI estimated where necessary.

All glass melters are engineered for identified sites with and specific capacities, types of glass, product glass compositions, and fraction of scrap or cullet in the feed. This engineering design analysis is meant to provide a basis for engineers to assess the benefits of the NGMS for insulation fiberglass production from both raw batch and batch containing up to 50 percent scrap fiberglass. Results from this engineering analysis would serve as the starting point for the design of a working melter at a specific location and plant.

Publications, Conference Papers, Presentations, and Patents

Presentations were made in person in mid-2009 and March 2011 to Mr. Glenn Strahs at DOE Headquarters in Washington, DC. These presentations covered the technology concept and history as well as the work that has been supported by DOE. The goal of these presentations was to provide Mr. Strahs with background on the SCM and NGMS technologies and to bring him up to date regarding the key role DOE is playing in their development. Mr. Strahs at DOE was brought up to date with project progress during a meeting this quarter to DOE headquarters.

The first conference presentation related to this project was presented in October, 2009 at the 70th Glass Problems Conference. The paper was authored by David Rue, Walter Kunc, and John Wagner (GTI), Bruno Purnode (Owens Corning) and Aaron Huber (Johns Manville) and titled “Rapid Refining of Submerged Combustion Melter Product Glass”. This was the first public presentation on project approach and status. Data from tests conducted to date and on refining approaches was discussed. Non-proprietary results were included that showed the short residence refining approach can remove 99% of bubbles to lower the void fraction of SCM product glass from 30% to under 0.5%. Conference papers will be published in spring 2010 by the American Ceramic Society.

A paper was submitted for review to the International Journal of Applied Glass Science. The paper was published in the December, 2011 issue of the journal (Rue, D., Brown, J.T., “Submerged Combustion Melting of Glass”, Int. J. Appl. Glass Sci., 2(4), 262-74, Dec. 2011.

A paper on submerged combustion melting general applications including glass and other products was presented at the 73rd Conference on Glass Problems. GTI co-authored this paper with the primary authors being the original developers of this configuration of submerged combustion melting, Leonard and Igor Pioro.

A presentation was made the DOE AMO peer review meeting on May 6 and 7, 2014. This 10-minute presentation will be made public through DOE.

A patent application was submitted to the U.S. Patent Office covering the filtration developed for refining of glass from high-intensity melters such as the SCM. The patent office awarded the patent as U.S. Patent 7,874,179, “Method of Removal of Gaseous Inclusions From Viscous Liquids”.

An invention disclosure was prepared in early 2010 covering the second approach to rapid refining developed in this project (thermal convective rapid refining). This second approach has been evaluated with model fluids and mathematically and was tested with batch and continuous molten glass trials. The invention disclosure was reviewed by the GTI patent review committee and was re-written and re-submitted in a stronger form. The new version of this invention disclosure covers the findings from the model fluid and CFD modeling work. The re-written invention disclosure was converted into a patent application and was formally submitted to the patent office in February, 2011 (US2012/0210751). GTI is attempting to secure this patent by responding through appeals to patent office objections. The Patent Office Appeals Committee has not yet made a final ruling on the patent application. The application is still active and awaiting a final decision.

Conclusions

The primary objective of this project was to evaluate, test, and develop a rapid refining approach for a Next Generation Melting System using a submerged combustion melter (SCM) for the melting and homogenizing step. This study assumed that the glass entering a refining step from the SCM contained a large number of bubbles constituting 20-30% of the glass by volume. The table below summarizes presents the conclusions from consideration of nine approaches to rapid refining.

Table 11. Rapid Refining Conclusions

Refining Approach	Work performed	Conclusions
Temperature/time	Testing with SCM glass	Effective at removing large bubbles. Residence times of 60 to 120 minutes can remove 99% of bubbles. Unable to meet most glass quality requirements with residence times shorter than several hours.
Helium (inert gas)	Evaluation	Determined to be impractical because of large volume of helium required and the inability to recover and recycle the helium
Water (steam)	Evaluation	Determined ineffective because of the large amount of water already present from oxy-gs SCM firing
Sonic	Evaluation	Determined impractical because no material is available that can survive in service as an ultrasonic horn in contact with molten glass
Vacuum	Testing with simulated fluids	Proven commercially. The challenge is developing an approach compatible with industrial-scale glass production because operation with high levels of vacuum are challenging when working with high temperature molten glass.
Centrifugal	Testing with simulation fluids	Promising approach but requires rotational speeds greater than 1500 rpm. Ineffective at removal of small bubbles that must be removed to meet quality requirements for most commercial glass products. Other configurations of centrifugal refining may be an effective rapid refining technique.

Filtration	Testing with simulated fluids and molten glass	Effective at removing bubbles. Velocity must be very low making this technique impractical for refining the large volumes of glass in industrial operations.
Obstructed path	Testing with Simulated Fluids	Not effective at separating bubbles in simulated fluids. Obstructions do not prevent bubbles from traveling along streamlines created by fluid flow. Possible improvements could be obtained by reducing velocities near zones created by obstructions.
Thermal convective	CFD modeling, testing with molten glass in batch and continuous modes	Effective in CFD modeling and in batch tests with molten SCM glass. Continuous tests were inconclusive because the needed level of operational control was not successfully achieved. This approach is considered promising.

The study of rapid refining approaches has identified two approaches that are the most promising. Vacuum refining (also known as sub-atmospheric of low pressure refining) has been operated commercially with other melters and should be effective with SCM in a Next Generation Melting System. The proper configuration of a vacuum refining system most compatible with an SCM would be needed to optimize the overall NGMS process. A challenge of vacuum refining is lowering and then raising the pressure on glass while at the high temperatures needed to keep the viscosity at the needed value for refining.

The second promising rapid refining approach is thermal convective rapid refining (TCRR). TCRR is a refinement of traditional temperature-time refining in which controlled heating creates convective currents in the molten glass and then other controlled heating breaks bubbles on the surface of the glass. Modeling and batch testing with SCM glass indicate that fiberglass and potentially container glass can be refined in under 30 minutes with a well-controlled TCRR system. Continuous testing results in this project were inconclusive. The continuous tests were not able to achieve the refining chamber temperature and residence time profiles needed to sustain the TCRR effect. With careful TCRR process control, modeling indicates a reduction of residence time compared with temperature-time refining of at least a factor of four.

Along with the primary work on rapid refining, a number of other issues related to advancing the NGMS technology were addressed in this project and projects providing cost share to this work. Conclusions in these related areas include:

- Advanced oxy-gas burner for SCM – A new burner design was developed. This advance burner uses a different means of missing oxygen and natural gas in an effort to produce a shorter, stiffer flame. Testing found that the flame is shorter and stiffer than the flame from the original, patented SCM burner. Also, the burner is cooler and uses less water than the original SCM burner. This is expected to lead to longer burner service life. The burner was tested on an open test stand and in several SCM tests. The burner performed well, but is still under development. Issues related to pluggage of the burner top during unsteady period (start up, shut down, operational upsets) are still under investigation.

- Optical melt monitoring sensor – Active melt in the SCM coats all surfaces with glass, making any optical monitoring difficult. A sensor was developed and tested that uses a fiber optic bundle, a small diameter opening (possibly under 0.5 in. in diameter), and an active purge. The probe watched the active melt bed surface for several hours before being coated with glass. With improved purge gas flow patterns and preheated purge gas that does not instantly quench the glass, a working probe is likely possible. A reliable probe working for the life of a melter would be a valuable tool for long-term industrial SCM operation.
- Alternative graphite melt discharge tap – A graphite tap using a cooled sliding gate was designed and tested. The graphite survived for several hours in SCM operation, but the graphite eventually cracked. Graphite was found to not be an acceptable material for an SCM discharge gate.
- Refining channel improvements – The thermal convective rapid refining approach relies on heat applied through the floor of a channel into the molten glass above. This creates convective flow that slowly moles toward the surface, carrying the bubbles upward in a coalesced mass. One means of heating the floor is with an electrically conductive non-oxide ceramic. Researchers at the Missouri University of Science and Technology tested a ZrB2 ceramic in contact with several glass compositions under aggressive conditions and high temperature. The ZrB2 performed better that baseline ceramics and would appear to be an excellent choice for the floor of a thermal convective rapid refining channel. Design of plates for this purpose can now be carried out. Care must be taken to control plate thickness because heating is a function of ceramic cross-sectional area.
- Preheated air-firing – An SCM test was conducted with pre-heated air instead of oxygen as the oxidant. The test went well operationally. Preheated air was found to significantly reduce the possible pull rate. This reduced the production rate and efficiency of the melter. Data increased the range of operating conditions studied with the pilot-scale SCM and provided a deeper understanding of the characteristics of the SCM process. Overall, oxygen is preferred because oxygen
- Techno-economic analysis for a commercial scrap fiberglass SCM – A design was developed for a 5 ton/h SCM producing insulation fiberglass from raw materials and from scrap. The resin on the scrap was found to lower the natural gas requirement but not affect oxygen demand. Work included sizing the melter, carrying out parametric analyses, and pricing equipment. Utility requirements were determined. All major pieces of equipment were sized and priced from vendor catalogues or price quotes. Overall, the SCM looks fiscally attractive as a replacement melter for fiberglass made with or without scrap addition. The SCM capital cost is significantly lower while energy cost is similar to slightly lower. The SCM offers the ability to remelt scrap fiberglass, an operation that cannot be carried out in traditional glass melters.