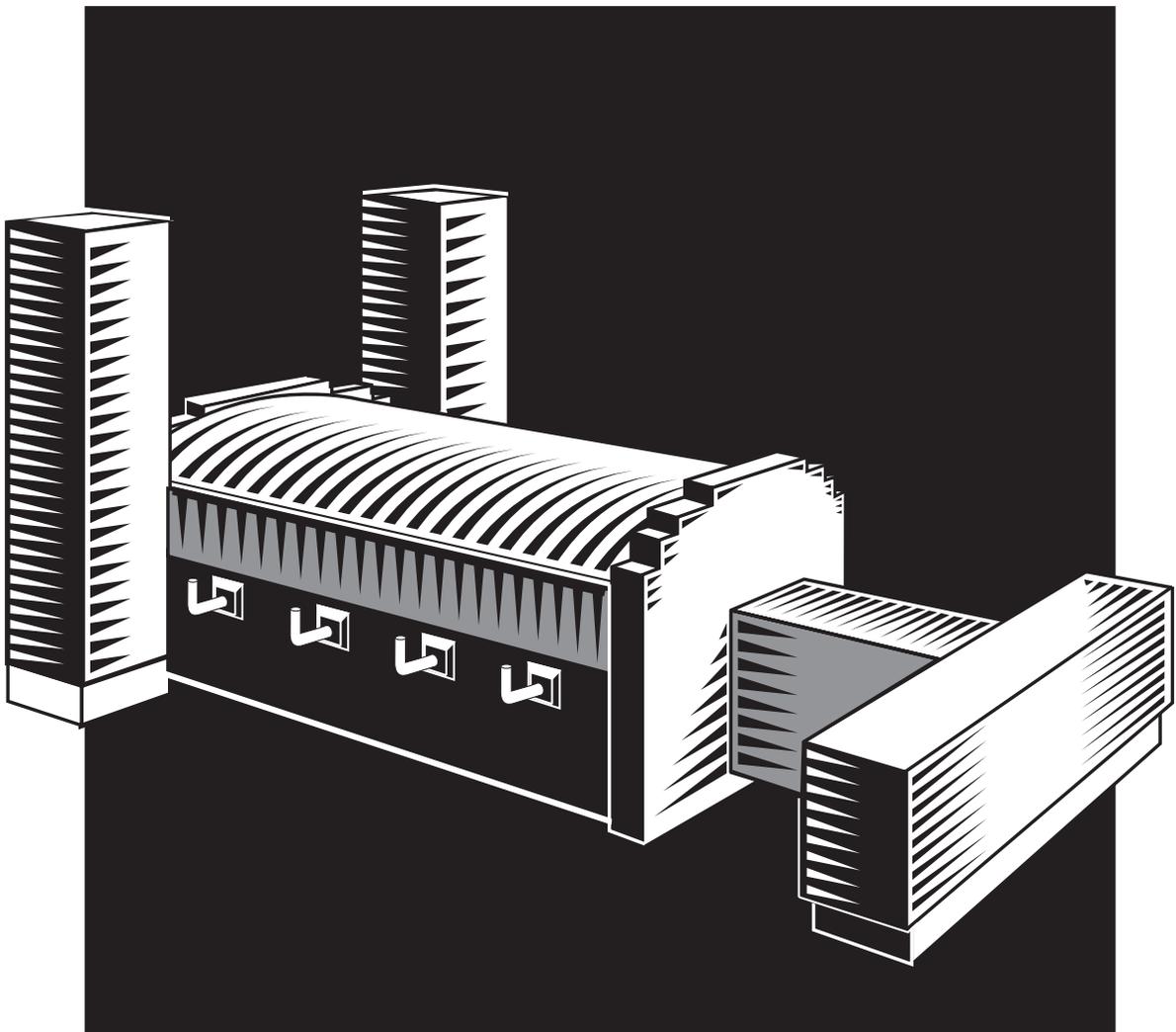


Glass Industry of the Future

Quarterly Status Reports

As of September 30, 2007



U.S. DEPARTMENT OF ENERGY

02-GA50113-03

Glass
Industry of the Future

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Glass Industry of the Future

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Glass Projects

Rapid Conditioning for the Next Generation Melting System 1
Gas Technology Institute
GO16010

***Rapid Conditioning for the Next Generation
Melting System***

Gas Technology Institute

GO16010

QUARTERLY PROGRESS REPORT

Project Title Rapid Conditioning for the Next Generation Melting System

Covering Period July 1, 2007 through September 30, 2007

Date of Report November 16, 2007

Recipient Gas Technology Institute
1700 S. Mt. Prospect Rd.
Des Plaines, IL 60018

Award Number DE-FC36-06GO16010

Other Partners NYSERDA – project sponsor
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Project Objective

The objectives of this project are 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. Techniques to produce higher quality glass from the melter will be investigated, and external fast conditioning approaches will be analyzed. The add-on rapid conditioning techniques evaluated through modeling and limited testing will rely on sonic energy, steam, and time discharge to speed conditioning

Statement of Objectives

The objectives of Budget Period One are to:

- Evaluate furnace operating parameters on glass quality. These parameters include burner firing patterns, melt temperature, glass residence time, etc.
- Conduct initial tests to evaluate the improvements achieved in glass quality using sonic refining
- Analyze product glass quality using advanced techniques for both on-line and off-line measurement of seed count and size
- Model other promising rapid conditioning techniques adaptable to the SCM
- Develop the engineering requirements for a Next Generation Melting System designed to recycle scrap fiberglass.

All work will be carried out using a single E glass composition. This approach will provide the largest impact of changing melter parameters and rapid conditioning conditions to be compared and analyzed.

Project Schedule

The project timeline is shown below. The first task will include the design and construction of the short heated discharge section and preparation of the sonic refining test setup and sampling methods. Task 1 will be nine months in duration. Task 2 will include all testing and analysis of samples and will also include modeling of other rapid refining approaches. It is anticipated that there will be up to 6 total melter tests performed over the 12 months of Budget Period One. Each melt will be 12 to 72 hours long and will include multiple experimental tests. These tests will incorporate evaluation of the impacts of changes in melter operation (pull rate, burner firing rate, burner patterns, etc.) along with evaluation of the sonic rapid conditioning method. The third task will be used to prepare for an engineering design and CFD model for a demonstration-scale NGMS process to melt scrap fiberglass. Task 3 will last 3 months. Quarterly reports and a Final Technical report will be prepared for all sponsors.

Task	Title	Budget Period One, months											
		1	2	3	4	5	6	7	8	9	10	11	12
1	Preparation for SCM Glass Quality Tests	█											
2	Rapid Conditioning Tests and Analyses	█											
3	Engineering Design of NGMS for Scrap Fiberglass Recycle												

Performance Measures

Performance measures and go/no-go decision metrics are placed at the end of Year 1 of the project. Performance measures include the following:

Performance Measures

- Design, fabrication, and shake-down pilot SCM modifications including short refractory discharge zone and sonic refining equipment
- Completion of SCM tests with variations in operating conditions and with sonic refining following by glass quality analyses
- Initial modeling of other rapid refining techniques for the Next Generation Melting System
- Preparation of engineering design parameters for a Next Generation Melting System for

The performance measures for Budget Period One will lead directly into the go/no-go decision points for continued project work in Budget Period Two. The go/no-go decision points will refer to all activities as well as to which quality improvement approaches will be evaluated further or discontinued from evaluation. Go/no-go decisions are:

Go/No-Go Decision Points - at the end of Year 1

- Completed modifications to SCM pilot unit for testing with multiple approaches to glass refining and to glass quality measurement
- Conducted tests with sonic refining and shown significant glass quality

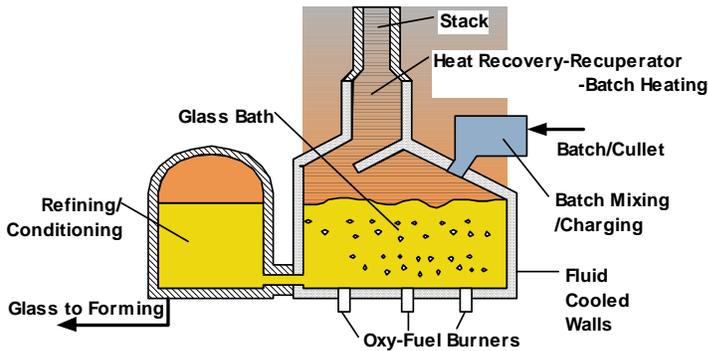
Background

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, with means temperature must usually be controlled to within several degrees. Along with all these requirements, each industry segment specifies molten glass must have the number and size of bubbles (seeds) for their product. In general terms, seeds must be below 0.1 mm in diameter, although optical glass demands are even stricter.

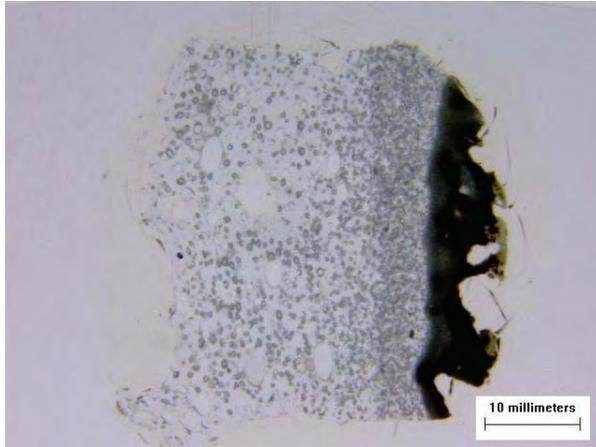
The U.S. Department of Energy is supporting collaborative efforts to develop a Next Generation Melting System (NGMS) that will produce glass of needed quality to the forming step of each glass industry segment. The submerged combustion melting technology, licensed and controlled by the Gas Technology 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 filed for a patent in this area. 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 will be worn away by moving glass, so walls must be built from externally cooled walls 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 and E glass compositions have been melted to date in a lab-scale SCM unit (shown below), and work is underway to design a pilot-scale 1 ton/h melter.



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



Results to date with SCM are highly encouraging. The product glass, however, must be of higher quality to meet many industrial needs. There are multiple means to improve the glass quality, and the proposed project will focus entirely 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 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). All of the techniques mentioned will be tested and evaluate in this project.

External Conditioning Approaches

Along with improved SCM operation, external, rapid conditioning methods can improve glass quality. These techniques will be modeled in this project, and the most promising will be tested in a bench-top lab apparatus, and potentially on the pilot SCM unit. 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.² The upward velocity of small bubbles in a liquid is characterized by Stokes Law.

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

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 is inversely proportional to the

viscosity of the glass (μ). All refining approaches modify one or more variable 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. The most promising approaches will be modeled and analyzed in this project. The project team includes experts in water refining (Tomazawa), sonic refining (Spinosa), and other approaches such as vacuum (Wooley). Each rapid refining approach takes advantage of one or more variables in Stokes Law to accelerate the conditioning process. A list of the rapid conditioning approaches to be modeled and analyzed in this project is shown in the table below.

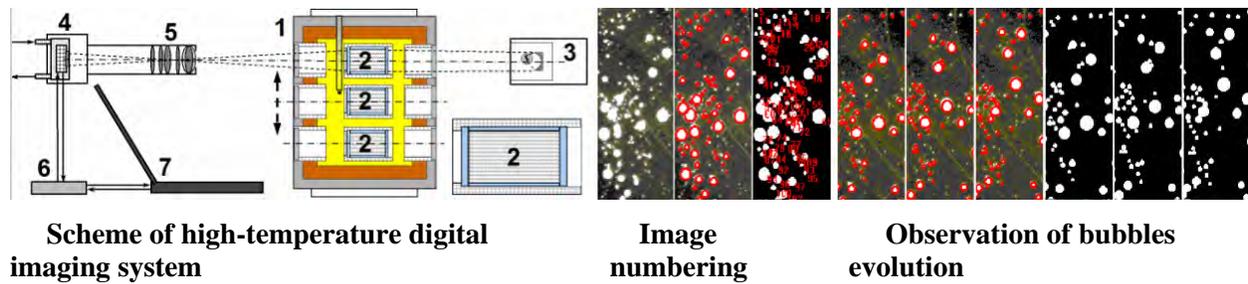
Rapid Conditioning	Basis for Accelerated Conditioning
Helium (inert gas) ³	High solubility and high diffusion leads to scavenging of small bubbles into larger bubbles and fast bubble rise to surface (d_b is larger)
Water (steam) ⁴	High solubility and high diffusion leads to scavenging of small bubbles into larger bubbles and fast bubble rise to surface (d_b is larger)
Sonic ⁵	Vibrations lead to bubble consolidation and faster rise to the surface
Vacuum ⁶	Decrease pressure leads to bubble expansion and fast rise to the surface
Centrifugal ⁷	Gravitational constant changed from g to ω^2 which can greatly increase velocity

Bubble Characterization

Using optical techniques for bubble characterization, the project team will be able to routinely and rapidly determine the impact of changes in parameters on the quality of SCM product glass. By being able to see through a molten glass one is able to study the glass melting processes taking place. Consider two examples: light scattering connected with formation and evolution of bubbles at re-finishing, and refraction of light connected with dissolution of cords at homogenization. Both phenomena can be studied using a high-temperature imaging system similar to that developed by the Laboratory of Glass Properties in 1998–2001. By taking images of the molten glass layer through sapphire windows using its own near-infrared radiation, diffused or directed backlight, one obtains all of the information necessary for analysis of small (~0.005" seeds) or large (~0.5" bubbles and thick cords) objects visible in the observation area.⁸

LGP support in the proposed project is based on 12-years experience in development of equipment for optical studies of molten glass at temperatures up to 3000°F. Based on know-how developed in LGP, a stable and accurate high-temperature imaging system will be developed.

The proposed system works as a stand-alone accessory, compatible with different refiner modules. The system includes: a) stabilized light source (3); b) CCD module consisting of 1 MP VIS-NIR matrix sensor (4) supplied with frigistor and water-cooler; c) zoom lens (5); c) imaging processor (6); d) portable computer (7). The following components designed in LGP will be used as prototypes: high-temperature optical cell (offered in 1994 and used without design changes until now), stable light sources, original large-aperture optical schemes, data acquisition systems and software, and others. A scheme of the system is presented below. The light source, CCD sensor module and image processor are drawn schematically. Real components are high-tech portable units, which will be developed using modern advanced technologies. The system allows still images to be taken at 1 fps or video at 24 fps. Image analysis software recognizes and numbers individual bubbles and assemblies, does bubble measurement, count, and determines their size distribution. The system is also capable in determining degree of non-homogeneity.



Bubble #	X	Y	d
1	0 0010	0 0217	0 0022
2	0 0020	0 1260	0 0022
7	0 0213	0 2575	0 0054

Development of the system for on-line monitoring of bubbles and cords in steady-state or flowing melt includes:

- Assembling a working prototype of the system, consisting of full-size light source, CCD module and imaging processor with reduced (lab-scale) high-temperature chamber plus initial version of image analysis software;
- Performing an initial set of experiments with commercial glass samples;
- Adjusting, customizing, and finishing the prototype to full-value registration *system*;
- Developing the released version of image analysis software, to write user manual;
- Bringing the system, and all necessary software and documentation to an experimental facility of RC flexible laboratory melter, to install it and test with different types of glasses;
- Performing pilot-scale approbation of the system with the pilot-scale SCM unit;
- Providing technical support during the project period.

Status – Work This Quarter

The first active work on this project was conducted last quarter. Work this quarter focused on two major activities. The first effort was to conduct a pilot-scale SCM test and refining samples on controlled conditions in a high-temperature electric oven. This work was

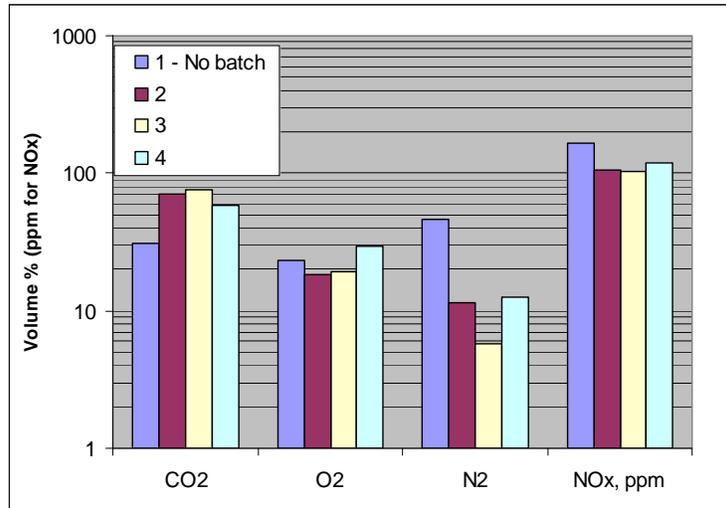
needed to provide baseline information for SCM refining requirements and to determine the quality of SCM product glass after initial refining. The second effort was to continue tests on potential refining approaches using ambient temperature surrogate liquids.

A pilot-scale SCM test was conducted this quarter 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 1250° and 1450°C. The photos below show the set-up used and the careful collection and movement of samples. Note that a second electric oven at 800°C 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.



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.



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

The second area of work this quarter involved testing of multiple refining approaches using ambient temperature surrogate liquids. New silicone oil with a viscosity of 100 poise was ordered and received so the liquid and bubbles would more closely simulate the SCM molten condition. A series of tests was conducted that confirmed that bubbles rise at rates similar to those observed for the SCM product borosilicate glass. A series of experiments were conducted to evaluate refining methods. Several of these tests provided positive results that will be followed up with more rigorous testing next quarter.

Plans for Next Quarter

Work will be carried out next quarter primarily on Task 1 activities. The primary focus will be on preparations for initial rapid conditioning testing. This will involve -

- Design of further tests with surrogate liquids such as high viscosity silicone oils to simulate glass refining conditions. This will help verify the potential for various refining approaches before they are conducted with molten glass from the pilot-scale SCM unit
- Completion of surrogate liquid refining tests. These tests will involve multiple approaches to refining including technologies such as ultrasonic refining and thin film refining
- Analysis of glass produced from the pilot-scale SCM unit to determine the suitability of this glass to typical long residence time refining
- Design of an add-on 'trough' piece for pilot-scale SCM tests. This will allow continuous operation while varying refining time and obtaining refining data. This test will involve collecting molten glass samples from the melter tap and the end of the 'trough', placing the samples in a temperature-controlled furnace for specified periods of time, annealing the samples, and then examining the samples to determine bubble size and count. This information will provide researchers needed insight into the level of refining required for SCM product glass. The data will also serve as a baseline for the effectiveness of various refining approaches as tested in later work.

In following quarters the selected rapid conditioning approaches will be assembled and tested in both surrogate liquids and in molten glass from the pilot-scale SCM unit. Product glass will be analyzed by the project team so that a complete understanding of the impacts of rapid conditioning approaches becomes available.

Publications/Presentations

There have been no publications or presentation yet related to this project. No patents have been filed, but later work is expected to lead to patents.

Milestone Status Table

This project is divided into three Tasks over a three-year period. A detailed schedule of project activities and completion dates will be prepared next quarter and included in the quarterly report.

Budget Data

The DOE contract was signed in June 2006 with an effective start date of July 1, 2006. The NYSERDA contract for co-funding will be finished in the next several quarters. Gas industry co-funding through GTI's SMP program is in place. The glass industry partners will be providing in-kind support to the project. The overall project budget, and spending to date, is shown below. Only cash funding is shown. In-kind cost-sharing by subcontractors and the two glass company partners is not shown.

Phase / Budget Period			Approved Spending, \$K			Actual Spending, \$K		
			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Year 1	7/06	8/07	109	100	209	92	0	92
Year 2	7/07	8/08	257	100	257	0	0	0
Year 3	7/08	8/09	6	150	156	0	0	0
Total			372	350	722	92	0	92

The first DOE spending was charged last quarter. Work will continue next quarter with refining tests with both surrogate liquids and molten glass. At that point, the pilot-scale SCM unit will be prepared for continuous melt tests focused on refining.

Index of Award CID Numbers

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