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**Millimeter-Wave Measurements of High Level and
Low Level Activity Glass Melts**

Introduction

EMSP supported research of millimeter-wave technology for nuclear waste glass melter monitoring has been very productive in establishing this field and showing great progress. This work has garnered significant recognition, winning an R&D 100 Award for viscosity monitoring, a Best Paper Award by the American Ceramic Society for nuclear waste glass monitoring, investment by the Glass Plus industry consortium to test this technology for glass fiber manufacture, investment by Savannah River Technology Center in purchasing key hardware components for additional tests, and Japanese initiated exchange visits between MIT and the vitrification facilities at Japanese Atomic Energy Research Institute (JAERI) in Tokai to review this technology. There are also potentially important spin offs to other areas including nuclear and fossil fuel power production, and National Institutes of Health sponsored research as indicated below. Consequently, this work has the potential of becoming a major internationally recognized EMSP success story. A summary of the main accomplishments follows. The readers are referred to the cited reference publications for more details, many of which were EMSP supported by this work.

A. Millimeter-Wave Instrumentation

1. Sensor Configuration

Extensive laboratory work has established the basic configuration of a millimeter-wave sensor for multiple parameter measurements of the molten glass pool in a melter as shown in Figure 1. The basic building blocks are the millimeter-wave heterodyne receiver, a beamsplitter in the receiver field-of-view, a waveguide/optics transmission line to the melt pool, a window to seal the waveguide, and a thermal return reflection (TRR) mirror aligned with the split signal from the beamsplitter. Millimeter-wave signals are both received from, and transmitted to the molten glass. With this configuration, and slight modifications, it is possible to monitor all the melt pool parameters listed in Table 1 using only a single access point into the melter. The physical melt pool effect that is being exploited in the millimeter-wave range to obtain the measured parameter is listed in the second column. More details of the hardware and measurement basis are given in references [1, 2].

Figure 1. Basic millimeter-wave sensor configuration.

Table 1. MMW Measurements

Parameter	Measured Effect
Temperature (T)	Thermal emission
Emissivity (ϵ)	Reflection amplitude
Density (ρ)	Reflection phase
Viscosity (η)	Reflection phase rate
Foaming	Surface acceleration/emissivity
Salt Layer	Emissivity/turbulence

two sections, followed by the analytical modeling accomplishments and the laboratory/field measurements that were achieved. Also, it should be noted that Table 1 is not comprehensive. It shows only the parameter measurements that have been demonstrated so far by the millimeter-wave technology. Additional melt pool parameter measurement capability for liquidus, redox, noble metals, plenum temperature, and others are potentially possible with future development.

2. High Temperature Waveguides

Demonstrating efficient millimeter-wave waveguides that could be used at temperatures over 1000°C inside the melter and in the melt pool itself was a major accomplishment. It was found that efficient hollow millimeter-wave waveguides could be fabricated from almost any refractory material such as those used to fabricate melter crucibles. The key to this accomplishment is to make the waveguide diameter much larger than the wavelength and use the HE_{11} waveguide mode (transverse magnetic and electric fields) for propagation [3]. The HE_{11} mode is a natural mode inside smooth walled dielectric tubes (the same mode is used inside single mode fiber optic cables at light wavelengths) and can be achieved inside electrically conducting metallic tubes by circumferentially corrugating the inside wall with $\frac{1}{4} \lambda$ deep grooves at more than 2.5

Figure 2. MMW waveguide used for many measurements was a composite of room temperature corrugated brass and high temperature ceramic sections.

Achieving the sensor configuration as shown in Figure 1 required developing novel millimeter wave components suitable for use in the high temperature and industrial environment of a nuclear waste glass melter. These components are the high temperature waveguide that interfaces with the hot melt pool and the quasi-optical beamsplitter TRR mirror configuration that is both rugged and efficient. These two component developments are summarized in the next

grooves per λ of waveguide length. The HE_{11} mode is also ideally suited for monitoring because it launches as a free space Gaussian beam, which is optimum for achieving the smallest possible diffraction limited spot sizes for good spatial resolution.

The waveguide configuration developed for the millimeter-wave sensor measurements is shown in Figure 2. Unlike earlier work with graphite [4], this one could be used in an oxidizing environment. It consists of a room temperature corrugated waveguide section and a ceramic section, which for most measurements was a mullite ($3Al_2O_3 \cdot 2SiO_2$) tube 1 1/8 inch (28.6 mm)

in internal diameter. The MMW receiver frequency used in the tests was 137 GHz ($\lambda=2.19$ mm) and the corrugation dimensions shown for the brass section are optimum for that frequency. Theoretically, transmission losses in the corrugated brass waveguide are very small and it can be made as long as necessary to transmit the millimeter-wave signal outside the biological shield of a nuclear waste glass melter (10's of meters). The mullite waveguide was measured to have a loss of about 20% per 0.5 meter. Therefore, it is only used where necessary to access the high temperature environment. Tests were carried out at temperatures of up to 1500°C and with waveguide glass immersions for up to one week in nuclear waste glass melts. Good millimeter-wave performance results were achieved. Some penciling of the immersed waveguide was observed, but did not affect the millimeter-wave performance significantly in the tests so far. Advanced refractories could be used to make the waveguide last as long as the melter crucible. This waveguide technology also has important spin off applications to monitoring in nuclear fusion experiments, nuclear power plants, fossil fuel power plants, and refining manufacturing where diagnostic access to high temperature environments is extremely challenging [5].

3. Compact Quasi-Optical Beamsplitter/TRR Mirror

At high millimeter-wave frequencies optics are the most efficient way of achieving beam splitting and redirection of signal, but millimeter-wave optics are large and very sensitive to alignment. In an industrial scale nuclear waste melter environment it is not desirable to have sensitive optics that take up a lot of space. Therefore, one of our achievements has been to invent a quasi-optical corrugated waveguide and beamsplitter device that achieves the thermal return reflection function shown in Figure 1 in a compact, rugged component. A cross-section of this device is shown in Figure 3. Crossed corrugated waveguides are machined in an aluminum block with a diagonal split as shown to accept a beam splitter. In the experiments carried out so far at 137 GHz the waveguide block was only 2.5 inches (6.4 cm) square by 1.5 inches (3.8 cm) deep with a quartz beamsplitter. The millimeter-wave signal from the melt and receiver field-of-view transverses the block in one direction and the split signal is directed in an orthogonal direction to a TRR mirror (not shown). Insertion losses were measured to be only 0.5%. This component has been very successful in the EMSP sponsored work and has been copied by an NIH sponsored experiment to monitor a 250 GHz gyrotron beam for dynamic nuclear polarization (DNP) measurements [6]. At frequencies above 100 GHz there is no other way to efficiently and compactly monitor millimeter-wave beams.

Figure 3. Quasi-optical beamsplitter.

B. Analytic Modeling

1. Temperature and Emissivity

Thermal emission signals are proportional to the product of the emissivity and temperature (εT) of the material that is viewed. Methods that can resolve the emissivity and temperature are of general interest to materials analysis. An important accomplishment has been to derive the analytical basis for determining these parameters using the thermal return reflection method. The basic concept was to use the thermal radiation from the viewed source as a probe of its emissivity. There is a significant advantage to using incoherent thermal radiation for probing versus coherent probe beam as demonstrated in an earlier work [7], and use of the viewed objects own emission as a probe greatly simplifies the instrumentation hardware requirements.

The reader is referred to the publication [8] for details of the derivation. Here we just state the results with the aid of Figure 4. The receiver views a sample (S) through a beamsplitter (BS) and waveguide system (WG). The analysis requires taking into account all of the sources and losses of signal in the field of view of the receiver. Each component has associated with it an emissivity (ε), temperature (T), transmission factor (τ), and reflectivity (r). If a component is at room temperature, then its emissivity and temperature will cancel out as in the case of the beamsplitter.

Figure 4. Illustration for defining the terms in the analytical results for the TRR method.

The analytical results for the measured temperature at the receiver for the two TRR cases are given by:

$$T_{eff} = \varepsilon_{wg} T_{wg} + \tau_{wg} \varepsilon_s T_s + r_s \tau_\kappa \tau_{wg} \varepsilon_{wg} T_{wg} \quad (1)$$

$$T'_{eff} = \frac{T_{eff}}{1 - r_s \tau_\kappa r_{bs}^2 \tau_{wg}^2} \quad (2)$$

where T_{eff} and T'_{eff} are the MMW temperatures measured at the receiver without and with the thermal return reflection, respectively, T_{wg} and T_s are the waveguide and melt surface temperatures, ε_{wg} and τ_{wg} are the waveguide emissivity and transmission related by $\varepsilon_{wg} = (1 - \tau_{wg})$, ε_s and r_s are the emissivity and reflectivity of the viewed surface related by $\varepsilon_s = (1 - r_s)$, r_{bs} is the beamsplitter reflectivity, and τ_κ is the viewed surface coupling factor. These two equations can be solved to obtain the emissivity and temperature of the viewed

sample. If surface roughness is a factor then a thermocouple measurement of temperature can be used to solve these two equations for emissivity and coupling factor.

2. Viscosity

The analytic basis for millimeter-wave viscosity measurements was also derived. Viscosity is related to melt glass flow, which can be measured with the setup shown in Figure 1 by immersing the waveguide into the molten glass and pressurizing the waveguide to displace the glass. In practice, the waveguide pressure is suddenly released by an electrically operated solenoid valve and the rate of the glass flow back to refill the waveguide is measured and related to viscosity. This is analogous to the standard room temperature laboratory capillary flow technique for viscosity measurement, but implemented at high temperatures using the refractory waveguide as the “capillary” and the millimeter wave receiver instead of a stopwatch to determine rate of flow.

The actual molten glass surface displacement is determined by monitoring the reflection of the leak local oscillator (LO) from the MMW receiver. The phase of the coherent LO reflection depends on the path length between the receiver and melt surface. Figure 5 illustrates how this signal will appear for an example displacement shown by the top curve. For every $\frac{1}{4}\lambda$ displacement of the melt level, the video signal will cycle through a minimum–maximum period (fringe) corresponding to a round trip reflection phase shift of π . For a linear displacement the video signal will appear as a rectified cosine function as shown in the beginning of the lower plot in Figure 5. For a non-linear level change, the video signal fluctuations will be distorted from a cosine function as shown in the latter part of the plot in Figure 5. In the case of molten glass refilling the waveguide, the flow observed is typically a gradually slowing rate.

Figure 5. Calculated illustration of the relationship between melt level height change and a millimeter-wave video reflection signal at a frequency of 137 GHz ($\lambda = 2.2$ mm).

Figure 6. Definition of terms used for analytic basis of viscosity determination.

A simplified Navier-Stokes analysis for laminar flow of a Newtonian fluid in a pipe has been used to derive a relationship between the observed measured average waveguide refilling

velocity and the molten glass viscosity. With the aid of the definition of terms in shown in Figure 6 this equation is given as:

$$v_{ave} = A\rho \frac{z_o}{(L - z_o)\eta} \quad (3)$$

where A is an instrumentation constant, ρ is the molten glass density, z_o is the initial glass displacement, L is the waveguide immersion depth, and η is the glass viscosity.

C. Laboratory Measurements

1. Advanced Refractory Measurements

Several advance refractory crucible materials were studied at high temperature to evaluate the possibility of using these as alternative materials for MMW waveguide fabrication to increase molten glass immersion survivability. The materials studied were chromium oxide (Cr_2O_3), alumina-zirconia-silica (AZS), and Monofax K3. Flat samples were put inside an electric furnace opposite the mullite MMW waveguide and heated up to 1500°C . The emissivity was monitored by the TRR method and by reference to a thermocouple. One material, AZS, was quickly identified as being unacceptable as shown in Figure 7. The top trace shows the furnace thermocouple and the bottom trace is the millimeter-wave signal product of emissivity and temperature (εT). At each pedestal on the millimeter-wave signal the TRR method is implemented as shown by the upward transients. At the temperature transition form 1400 to 1500°C the millimeter-wave signal makes a sudden increase greater that that expected by the thermocouple reading, indicating that the emissivity is changing as well as the temperature. Examination of the AZS sample after the furnace cooled down showed that glass from inside the sample had leaked out, changing the surface chemistry and figure. This would be unacceptable in a waveguide. Measurements of the chromium oxide and K3 samples did not reveal such behavior. The data of Figure 9 demonstrates the power of millimeter-wave measurements for high temperature thermal analysis of materials [9].

Figure 7. Measurements of alumina-zirconia-silica (AZS) slowing glass bleeding above 1400°C

2. Viscosity

Laboratory glass flow data as a function of temperature was taken for a number of glass compositions that included two Hanford glass mixes, DWPF black frit #265, and a commercial E glass. The connection of molten glass flow time in the waveguide with viscosity was empirically

Figure 8. Glass viscosity versus the flow time in a millimeter-wave waveguide.

established. It was also found that surface tension effects appear to dominate at the end of the flow time when the surface is relaxing to equilibrium and when the displacements are small in the waveguide. Also the surface curvature could be seen to be changing from the amplitude of the reflected signal. To achieve large dynamic range for viscosity measurement, the difference flow time between two pressure displacements was used to subtract out the common starting and ending surface motions. Consequently, the resulting time difference corresponds only to the laminar flow time in the waveguide.

Example data is shown in Figure 8 for Hanford #8 glass (target composition 56.8% SiO₂, 20% Na₂O, 12% Al₂O₃, 9% B₂O₃, and several other trace oxides). The measured waveguide flow time between 0.5 inch and 1.2 inch water pressure displacement is plotted as open squares against viscosity as determined by Corning Services Laboratory. Varying the temperature of the glass from 850 to 1200 °C varied the viscosity over the range of 2000 to 20 Poise. The data is plotted on a log-log plot because of the wide dynamic range of the measurements. The line through the experimental points is a linear fit to the experimental points. The measured flow time tracks the viscosity perfectly over a 100:1 dynamic range of 20 - 2000 Poise. Accurate viscosity measurement over such a large dynamic range is a unique capability of millimeter-wave viscometer in addition to the high temperature operation. These laboratory viscosity measurements not only established the viability of millimeter-wave viscosity measurements, but also revealed interesting surface dynamics that would be of interest for future study [1].

3. Foaming

Vitrification operations at DWPF have encountered sudden glass foaming events that seriously disrupt the operations. On-line monitoring that can alert operators to the onset of foaming would be of great value. We have accomplished a laboratory measurement with hi-foaming glass that shows the capability of millimeter-wave measurements not only to monitor a foaming event, but also to provide a precursor indication long before the foaming gases break the melt pool surface [10].

The data for a 3-inch (7.6 cm) diameter crucible melt is shown in Figure 9. The top trace shows the furnace thermocouple signal, the next trace below shows the millimeter-wave thermal signal (ϵT), and the bottom trace is the surface position video signal. The events of the foaming unfold as follows: 1) at about 65 minutes on the x-axis the surface begins to accelerate as evident by the rapid fringe shifts on the video signal, 2) shortly after the onset of surface acceleration the MMW thermal signal begins to approach the thermocouple temperature signal (emissivity increasing), it peaks at about 80 minutes on the x-axis, 3) after peaking the MMW thermal emission decreases, gradually at first and then abruptly at about 100 minutes on the x-axis. When the MMW thermal signal increases the video reflection signal from the melt surface decreases. At the peak MMW emission the video signal is approximately zero as would be expected for a surface that has gone to an emissivity of one ($\epsilon = 1$).

Figure 9. MMW signal record of hi-foaming glass as it is melted down.

A possible interpretation of these measurements is as follows: 1) gases come out of the melt pool solution and begin pushing on the surface, 2) it takes about 15 minutes for the gases to reach the melt surface in the 3-inch crucible, 3) when the bubbles break the surface they initially roughen the surface on MMW scale lengths to prevent reflections and then gradually coalesce to reform a uniform surface, eventually forming a large bubble that pops. If this interpretation is accurate, it will be a great advance for nuclear waste glass monitoring. In the large scale dimensions of a nuclear waste glass melter operators may have hour warnings before the advent of foaming by monitoring for millimeter-wave surface acceleration. This would give them plenty of time to take corrective action. However, the results in Figure 9 are only preliminary and more research is needed to establish this monitoring technique for foaming and to exploit this technique for a better understanding of the physics and chemistry of a melter foaming event.

D. Field Tests

A major accomplishment has been the carrying out of two field tests of the millimeter-wave monitoring technology on an engineering pilot scale glass melter. These field tests achieve two goals: 1) they demonstrate to the melter community that millimeter-wave monitoring technology is not a laboratory curiosity, but fully capable of functioning reliably in a 24 hour, 7 day a week manufacturing environment and 2) they provide access for research with full scale melt pools having continuous top feeding with a cold cap, deep melts with temperature gradients, and glass pour events that can not be easily simulated in the laboratory. Both field tests were carried out on the EnVitco (EV-16) joule heated melter at the Clemson Environmental Engineering Technology Laboratory (CETL). This melter has a melt pool size of 18"x 18"x 14" and is

powered typically at 70 kW in two zones by four molybdenum electrodes, one centered in each wall below the melt surface.

1. Cold Cap Measurements

The goal of the first field test was to demonstrate two dimensional millimeter-wave temperature measurements of the melt surface with and without a cold cap. The cold cap is a solid crust in top fed joule heated melters, playing a key roll in feeding and stabilizing the vitrification process. On-line monitoring of the cold cap would result in better process control, increased production, and reduced costs for waste vitrification. For these measurements the millimeter-waveguide was modified as shown in Figure 10.

An inconel miter mirror cap was attached to the end of a mullite waveguide, which was inserted horizontally into the melter plenum area above the melt pool. The waveguide could be rotated and retracted in and out to scan the millimeter-wave receiver field-of-view across the surface of the melt pool.

Figure 10. Rotating waveguide that was horizontally inserted into the plenum area above the melt pool in the EV-16 melter.

The millimeter-wave receiver clearly identified hot and cold regions of the cold cap or melt surfaces and their dynamics during processing. Examples of the measurements are shown in Figure 11. Two temperature surface profiles crossing near the center of the melter are shown.

The top trace is with no cold cap and shows a surface temperature gradient that is indicative of how the power was distributed among the electrodes below. The lower trace shows the cold cap temperature profile during glass pour from a spout below the center of the melter. There is a clear drop in surface temperature by more than a factor of two above the location of the pour spout. These measurements demonstrate for the first time that on-line monitoring of melter melt pool surface dynamics are possible and that the millimeter-wave instrumentation works reliably in a glass manufacturing scale melter [11].

Figure 11. Melt pool surface temperature profiles.

2. Viscosity

In the second field test the goals were to demonstrate the feasibility of monitoring viscosity and salt layer formation in an engineering scale melter. For these tests the millimeter-wave waveguide was configured as originally shown in Figures 3 and 4 so that the mullite waveguide could be vertically lowered into the melt pool. Glass flow was measured over a 1" to 8" (2.5 to

20.3 cm) waveguide immersion range for a series of pressurizations from 0.5" (0.9 mm Hg) to a maximum of 12" (22.4 mm Hg) water pressure at the deepest immersion. The average waveguide refilling glass flow velocity measured for two of the initial waveguide pressurizations of 2" (2.8 mm Hg) and 4" (5.6 mm Hg) are plotted as inverted triangles and squares, respectively in Figure 12. Also plotted is a temperature profile measured with a thermocouple. Equation 3 is plotted as the two dashed curves assuming viscosity is inversely proportional to temperature ($\eta \propto 1/T$). Both calculated plots were fit to the same point at 2" (2.8 mm Hg) pressurization at a depth of 2" (5 cm) to obtain the instrumentation constant. The general trends of the measured flow velocities with depth and pressurization are reproduced reasonably well by the assumed model. The discrepancies could be explained by effects not modeled such as temperature gradients, drifts in temperature during the course of the measurements, and deviations from the laminar flow assumption, particularly at the shallow depths when immersion depth and displacement are similar. These results demonstrate for the first time in a glass melter a capability for not only on-line viscosity monitoring, but also for obtaining viscosity profiles within the melt [12].

Figure 12. Temperature and melt flow velocity measurements and comparison with simple viscosity theory (dashed curves).

3. Salt Layer Formation

Formation of salt pools in nuclear waste glass joule-heated melters is of considerable concern. Supersaturated salt coming out of the glass solution can compromise the integrity of the glass as a permanent nuclear waste storage medium (waste form), increase corrosion in the melter (due to corrosive nature of the molten salt), introduce power instabilities in joule-heated melters, and pose a serious hazard to the melter facility if a continuous layer is formed to short out the electrodes. At the end of the second field test at total of total of 4.2 lbs. (1.9 kG) of sodium sulfate (NaSO_4) was added to simulate a salt layer in a glass melter [12, 13]. The millimeter waveguide was not immersed, but positioned above the melt surface to monitor thermal emission. The dynamics of salt layer formation in a glass melter were observed for the first time. The salt forms small drops first that grow in size until a continuous surface layer is formed. Millimeter-wave surface fluctuations are coincident with the growth of the molten salt pools on the glass melt.

A quantitative measure of the emissivity of the molten glass and salt at 137 GHz was also obtained with the aid of the TRR method. Figure 13 shows the thermal return reflection data that was recorded. Not only is the MMW temperature of the glass melt higher than the molten salt, but also the ratio of the temperature without to that with the thermal return reflection is smaller for the glass melt as expected for a material having a higher emissivity. Using equations 1 and 2 and the thermocouple temperature the emissivity and surface coupling factor results in $\epsilon = 0.64 \pm 0.05$, $\tau_k = 0.46 \pm 0.05$ for the DWPF black frit glass and $\epsilon = 0.44 \pm 0.05$, $\tau_k = 0.60 \pm 0.05$ for the salt. The MMW emissivity of the molten salt is 32% lower than that for the glass. The difference in coupling factor suggests that the molten salt surface is a smoother, flatter surface (due to its low viscosity hence smooth covering) than the glass melt at these wavelengths.

Figure 13. MMW thermal return reflection measurements through an 80% transmitting warm waveguide ($\sim 360^\circ\text{C}$) of molten glass and salt at a thermocouple temperature of 950°C .

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