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High temperature ultrasonic sensor for the simultaneous measurement of viscosity and temperature of melts

Krishnan Balasubramaniam[a)]
Department of Aerospace Engineering and Mechanics, Mississippi State University, Mississippi State, Mississippi 39762

Vimal V. Shah
Diagnostic Instrumentation and Analysis Laboratory, Mississippi State University, Mississippi State, Mississippi 39762

R. Daniel Costley
Miltec Incorporated, National Center for Physical Acoustics, University, Mississippi 38677

Gary Boudreaux and Jagdish P. Singh
Diagnostic Instrumentation and Analysis Laboratory, Mississippi State University, Mississippi State, Mississippi 39762

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An ultrasonic sensor that simultaneously measures temperature and viscosity of molten materials at very high temperature is described. This sensor has applications as a process monitor in melters. The sensor is based on ultrasonic shear reflectance at the solid–melt interface. A delay line probe is constructed using refractory materials. A change in the time of flight within the delay line is used to measure the temperature. The results obtained from this sensor on various calibration glass samples demonstrate a measurement range of 100–20 000 P for the viscosity and 25–1500 °C for the temperature. © *1999 American Institute of Physics.* [S0034-6748(99)01912-7]

I. INTRODUCTION

Measurement of the viscosity and temperature of fluids using conventional and/or laser ultrasound techniques has been reported by several of the authors[1–5] as well as by others.[7–11] In this article, the utility of an *in situ*/on-line ultrasonic delay line based technique for the simultaneous measurement of viscosity and temperature of molten materials at very high temperatures (between 800 and 1500 °C) is demonstrated. The repeatability of the data under laboratory conditions has also been studied. One of the potential applications of this technology will be to measure the viscosity and temperature of glass-like molten material just before or as it is being poured out of a vitrification melter. Therefore, the work described in this article has concentrated on measuring the viscosity of glass under similar conditions.

Ultrasonic shear waves are affected by viscosity of the fluid.[1] At a solid-to-fluid interface, the amount of ultrasonic shear wave energy reflected back into the solid (the reflection coefficient) depends on the operating frequency, the physical properties of the fluid (viscosity and density), and the solid (density, shear modulus). The effects of each of these parameters on the complex reflection coefficient of an ultrasonic wave incident on a solid–liquid interface have been discussed previously.[1,2] It was also shown earlier that the influence of density on the reflection coefficient (also referred to as the reflection factor) was relatively very small compared with the influence of the change in viscosity. This conclusion was based on typical changes in the physical properties (density and viscosity) of a glass melting process.

A delay line capable of withstanding hostile environments acts as a conveyer of ultrasonic waves between the transducer (generator) and the liquid medium. The delay line also acts as the solid elastic medium in the solid–liquid interface. Earlier experiments[1–5] were carried out using delay lines made from Plexiglas, graphite, and aluminum. These materials have low acoustic shear impedance and are readily available and easy to machine. However, they are not suitable for applications that require viscosity and temperature measurements at high temperatures (those beyond 1000 °C) in a potentially oxidizing environment.

II. BACKGROUND

Ultrasonic methods have been used to measure the density, viscosity, and temperature in liquids.[6–10] Adequate methods are not presently available for measurements at high temperatures. Currently, process control in high temperature melters is achieved by inferring the viscosity from a temperature measurement, usually by using a thermocouple. This assumes a predetermined relationship between the viscosity and the temperature. Any slight change in composition and/or errors in temperature measurement leads to significant errors in the viscosity measurement. As an alternative, off-line sampling techniques have also been employed. However, there are difficulties in obtaining samples and analyzing off-line causes delays.

Few sensor technologies[11–15] are commercially available for on-line (*in situ*) viscosity measurement of melts at very high temperatures (above 1000 °C). Roger[11] has demon-

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[a)] Electronic mail: balas@ae.msstate.edu strated continuous monitoring of glass viscosity in a plant over an extended period of time using a vibrational ceramic rod that is immersed into the melt from the top. This sensor uses additional information on the glass melt level and internal temperature, both provided by other sensors, to measure the average viscosity over the depth of the immersed rod. The dependence of this technique on density and the influence of the glass viscosity variation profile near the upper surface of the melt have not been discussed. Faber *et al.*[12] discussed methods of measuring glass flow velocity, detection of bubbles greater than 0.2 mm, and also measuring glass temperatures using a 100 kHz wave transmitted between two immersed ceramic buffer rods in a glass melt. A noncontact laser based technique for measuring the flow quantity of melt, which has been poured through a fixed diameter orifice, and subsequently relating this to the viscosity of the melt has been demonstrated in a glass melting plant.[13,14] This technique may require calibration of each type of glass, and may depend on the density and the variation in the constitution of the glass. Also, high temperature rotating viscometers[15] and an oscillating-cup viscometer[16] have found application in high temperature metal processes.

The viscosity of a melt must be qualified with the corresponding temperature at which the measurement is carried out. Although thermocouples have been used for the temperature measurement, simultaneous measurement of temperature and viscosity from the same local region in the melt is sometimes required. The authors are not aware of any sensor that can do this. Current methods for measuring temperatures using immersed thermocouples are sometimes not desirable due to frequent breakage and inconsistent readings at high temperatures.

The velocity and the attenuation of an ultrasonic wave traveling in a viscous fluid are affected by the viscosity of the fluid[9,10] and hence can be a very significant tool in measuring viscosity. For Newtonian fluids, the relationship between viscosity and the ultrasonic velocity and attenuation is provided by the classical relationship[12]

$$\eta = \frac{3}{2} \frac{\rho c^2}{\omega^2} \alpha, \tag{1}$$

where η is the viscosity in Pa s, ρ is the density in kg/m^3, c is the velocity of sound in m/s, ω is the circular frequency in rad/s, and α is the attenuation in nepers. The use of attenuation for the measurement of viscosity has been well demonstrated. These methods have traditionally been used to measure the viscosity of small quantities of samples in laboratory experiments. However, due to the very high attenuation of the sound waves in molten materials (especially at high temperatures) such as molten glass, the scope of using the velocity or the attenuation to measure viscosity is limited for *in situ* measurements during material processing. For instance, for a 1 MHz ultrasonic frequency, the attenuation is 1000 dB/m at 1000 °C.[12] Also, the attenuation and velocity based methods measure average physical properties over the wave travel path. Due to the presence of temperature gradients and consequently viscosity gradients, a localized measurement technique is valuable.

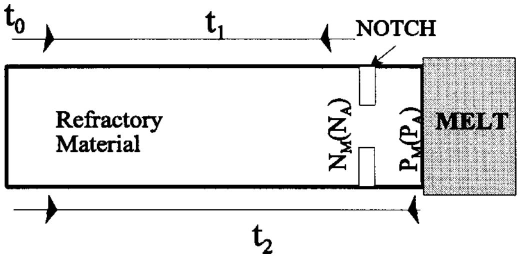

FIG. 1. Buffer rod sensor diagram with the notch as the reflecting reference mechanism.

In contrast, at a solid–fluid interface, the amount of ultrasonic shear wave energy reflected back into the solid is independent of the attenuation and velocity of the fluid. Also, the wave does not have to travel inside the molten material for the measurement to be made. Hence, by using a well characterized solid, the local fluid properties can be measured using the shear wave reflection characteristics at the solid–liquid boundary.

III. METHOD

The shear wave reflectance approach is used in this article. This technique employs a delay line waveguide. A piezo-electric transducer generates a shear wave ultrasonic signal into one end of the delay line (see Fig. 1). This shear wave is reflected from two reflecting surfaces near the other end of the delay line: (a) a notch or slit or an interface between two materials embedded into the probe and (b) the other end of the probe which is in contact with the hot liquid or hot melt. The reflected signals are detected by the same transducer at the signal generating end, both after specific time delays. Time and amplitude information from (a) is the reference data and the time and amplitude from (b) represent the parameters measured. The time delay between the two reflected signals will provide information on the temperature, whereas the reflected amplitude ratio will provide measurement of the viscosity.

A. Probe description

This method employs an acoustical waveguide (delay line) made of an appropriate high temperature material (such as alumina, molybdenum, inconel, tantalum, etc.). This delay line was externally cooled, thus allowing the use of traditional piezoelectric crystals for both the generation and reception of shear waves. Conventional off-the-shelf piezoelectric transducers were used to launch and detect shear waves. These transducers have significant advantages over other means of generating had detecting ultrasound such as laser ultrasound or electromagnetic acoustic transduction (EMAT). They are more efficient, sensitive, reliable, and less expensive. The shear transducer used was Y-cut type with broadband specification with a central specified frequency of 10 MHz. However, these transducers cannot function at elevated temperatures and therefore the dooled delay line conduit was employed to convey the shear waves from the transducer to the melt and back. Cooling the delay line leads to simultaneous temperature, velocity, and attenuation gradients, which are difficult to characterize quantitatively.

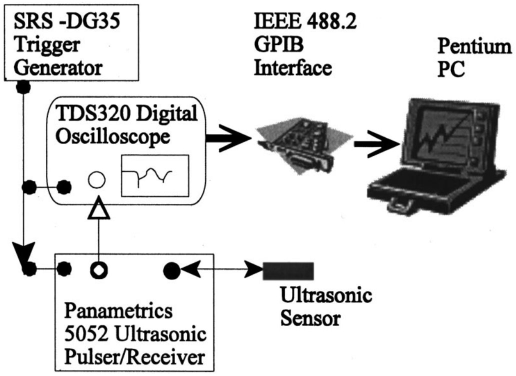

FIG. 2. Schematic representation of the instrumentation used for data collection.

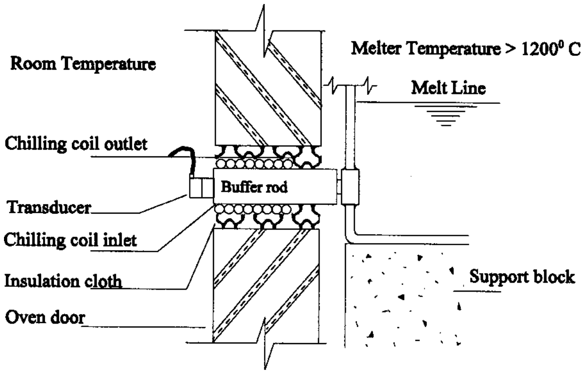

FIG. 3. Laboratory based experimental setup to simulate a melter environment.

Hence, the sensor was calibrated to compensate for thermal gradients in the delay line. The experimental setup is shown in Fig. 2 where an alumina (Al_2O_3) delay line acts as a solid substrate. A copper cooling coil was bonded to the delay line using high temperature bonding paste (Omegatherm 201, Omega Engineering Inc.). The flow rate of the coolant (water) was regulated in order to determine the flow rate that would bring down the temperature at transducer contact to room temperature. It was determined that a flow rate of 120 ℓ/h was sufficient for melt temperatures up to 1500 °C. This flow rate is available from most common faucets.

This method requires that the other end (the probing end) of the delay line be wetted by the viscous liquid that is being examined. A notch was milled in the solid, close to the probing end, to provide a constant reference (see Fig. 1). The two faces (the transducer contact surface and the delay line–liquid surface) as well as the reference notches are precision machined so that they are all parallel to each other in order to achieve normal incidence. Surface roughness was minimized by diamond grinding all the surfaces.

The shear wave transducer is connected to the instrumentation that generates the electrical signal to excite the ultrasonic piezoelectric transducer and to receive and condition the received ultrasonic signal. The instrumentation involves a pulse generator and a rf power amplifier. The signals received from the piezoelectric transducer are filtered and amplified by a high-fidelity amplifier. The amplified signal is then digitized using an analog-to-digital converter. Alternatively, the amplified signal can be processed in analog mode using peak detector and time detection circuits. Several off-the-shelf ultrasonic nondestructive evaluation instruments can be used for this measurement. The Panametrics 5052PR instrument was used to pulse the transducer, and it received and amplified the signal. A high precision time delay trigger (SRS-DG35) was utilized to reduce jitter problems. Analog-to-digital conversion was performed using a Tektronix TDS320 digital oscilloscope and the data were transferred to a PC using a IEEE488.2 general purpose interface bus (GPIB) interface. A schematic representation of the instrumentation is illustrated in Fig. 2. The digital data were analyzed and the travel time and amplitude data were extracted for further processing by the algorithm that will be described below.

B. Probe calibration

For a given delay line material and geometry, calibration depends only on the operating frequency and needs to be carried out only once for a particular configuration. The calibration process consists of two steps: one for temperature and another for viscosity.

Here, the subscript ''*A*'' represents the reference data set with no melt material in contact with the solid (air), ''*C*'' is the data set using a well characterized viscosity calibration melt material, and ''*M*'' is the actual measurement data set of an unknown melt. The symbol ''*P*'' represents the signal reflected from the delay line–melt interface and ''*N*'' is the signal reflected from the notch.

First the sensor was calibrated for temperature with the delay line in contact with air, inside an induction oven (as shown in Fig. 3). The temperature in the oven was slowly raised, allowing the delay line to achieve steady state. The temperature of the air inside the oven was measured using a calibrated thermocouple. The temperature inside the oven was slowly increased from room temperature to 1500 °C and the echo arrival times in the shear ultrasound transducer were measured. The travel time difference between the two reflections from the reference notch and the solid–air interface, $t_A=(t_1-t_0)_A-(t_2-t_0)_A$, where t_0, t_1, and t_2 are the time measurements shown in Fig. 1. The difference in time between the t_A at current temperature and the t_A at room temperature (denoted by δt_A) is plotted as a function of the oven air temperature in Fig. 4. A polynomial fit of these data provides the temperature calibration curve. The peak-to-peak amplitudes of the echoes reflected from the notch $[N_A(\delta t_A)]$ and the probe interface $[P_A(\delta t_A)]$ in contact with air are also recorded as a function of oven temperature.

In the second step, the delay line was calibrated for viscosity. This was accomplished by repeating the above experiments with the probe interface in contact with a calibrated glass (Fig. 3). The peak-to-peak amplitudes of the echoes reflected from the probe surface (P_C) and the notch (N_C) were measured along with the time difference (δt_C). The calibration reflection coefficient (R_C) was calculated from

$$R_C=\frac{P_C(\delta t_C)N_A(\delta t_C)}{N_C(\delta t_C)P_A(\delta t_C)}, \tag{2}$$

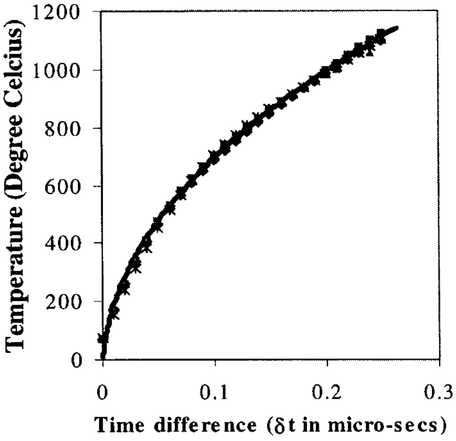

FIG. 4. Repeatability tests for temperature measurement using different buffer rods made of the same material and geometry.

where $P_A(\delta t_C)$ and $N_A(\delta t_c)$ were obtained from the first set of calibration curves. The relation between the glass viscosity and the temperature for the calibrated glass was obtained from the manufacturer. This relation was used along with the temperature calibration data and the reflection coefficient data to obtain a relation between the calibration reflection coefficient $[R_C(\delta t_C)]$ and viscosity $[\eta(\delta t_C)]$.

These calibration curves depend only on the delay line selection. Hence, the calibration curves remained unchanged for a specific material (Coors AD-94 alumina in this case) and for a particular geometry (length, diameter, notch location, and notch depth) and was used to obtain all the results presented in this article.

In a typical experiment on a sample with unknown viscosity, the delay line sensor measured the time difference (δt_M) and reflected amplitudes from the notch (N_M) and the contact surface (P_M). The time difference is related to the temperature of the fluid using the first set of calibration curves and the reflection coefficient is then related to the viscosity of the melt using both of the calibration curve sets. The measured reflection coefficient $[R_M(\delta t_M)]$ is

$$R_M(\delta t_M)=\frac{P_M(\delta t_M)N_A(\delta t_M)}{N_M(\delta t_M)P_A(\delta t_M)}, \tag{3}$$

where $P_A(\delta t_M)$ and $N_A(\delta t_M)$ are obtained from the calibration data set at (δt_M). Subsequently, using the viscosity versus the reflection coefficient relationship obtained in the second step, the viscosity $[\eta(\delta t_M)]$ of the unknown sample is determined.

IV. RESULTS

Calibration experiments to evaluate the effects of temperature gradients and the applicability of temperature compensation at very high temperatures were carried out using an empty crucible to observe the effect of temperature gradients on the ultrasound properties. The temperature (δT) can be computed as the positive root of the second order polynomial expression represented as

$$(t_r\beta_2)\delta T^2+(t_r\beta_1-\alpha)\delta T+[t_r(1+\beta_0)]=0,$$

where

$$t_r=\frac{\delta t}{t_s}, \tag{4}$$

where β_i are the coefficients describing the rate of change of the shear wave velocity $[V(\delta T)=\beta_2\delta T^2+\beta_1\delta T+\beta_0]$ as a function of temperature assuming a second order relationship, and δT is the difference between the current temperature (T) and the initial ambient temperature (T_0). Also, t_s is the travel time between the notch and the probing surface $[t_s=(t_1-t_0)_s]$ at T_0.

Figure 4 shows the experimental ultrasonic time difference (δt_A) data plotted against the temperature of air inside the oven measured by a thermocouple. The data represent several repetitions using different delay lines of the same geometry and made from the same material. This test was conducted to examine the repeatability of the temperature measurement. The data demonstrate precision of greater than 2% in the temperature measurement. The solid line represents the computed temperature using Eq. (4). This was calculated using the measured properties for alumina AD-94 $(\beta_2=-3.9711e-8,\ \beta_1=2.6394e-6,$ and $\beta_0=4.3882e-6,\ \alpha=8.20\times10^{-6}/{}^\circ\mathrm{C}$, density at room temperature$=3700$ kg/m^3, and shear wave velocity at room temperature$=5788$ m/s). The nonlinear relationship observed in this relationship is representative of two phenomena which simultaneously influence the time measurement. The first effect is the change in distance of ultrasonic wave travel (δL) between the reference notch and the probing interface and should represent a linear relationship if the coefficient of thermal expansion (α) of the delay line material is assumed to be linear with temperature. The nonlinear behavior can be explained as due to the nonlinear change in the ultrasonic velocity of the delay line material represented by the coefficients β_i. It was observed that the effect of a change in velocity due to temperature on the measured time difference (δt) was more significant than the effect of the change in length due to the coefficient of expansion.

Figure 5 compares the measurement of temperature, in a melt glass, using ultrasonic delay line and a RTD temperature sensor immersed in the melt. The range of measurement in this case was 25–1200 °C. The data represent a heat-up and a cool-down cycle. The two measurements compare well. The resolution of the time measurement was determined to be 1 ns which corresponds to a temperature resolution of 5 °C. Recently, affordable instruments that can measure arrival time with less than 0.1 ns resolution have become available. Hence, the accuracy in the temperature measurement of 0.5 °C may in practice be feasible.

Figure 6 plots the reflection coefficient versus temperature of the melt for several heat-up and cool-down cycles using a glass sample manufactured by Ferro (IP-745). According to theory,[1] the reflection coefficient should be ~1.0 for low viscosities (0.01 P or 0.001 Pa s) or for the solid–air interface. It is observed from this plot that there is almost perfect reflection (~1.0) during the initial portion (before softening) of the heating cycle. A possible explanation for this is that there is no transfer of shear waves from the delay line to the solid phase of glass. This was found to be true in using the initial frit form of glass in the first metal cycle and

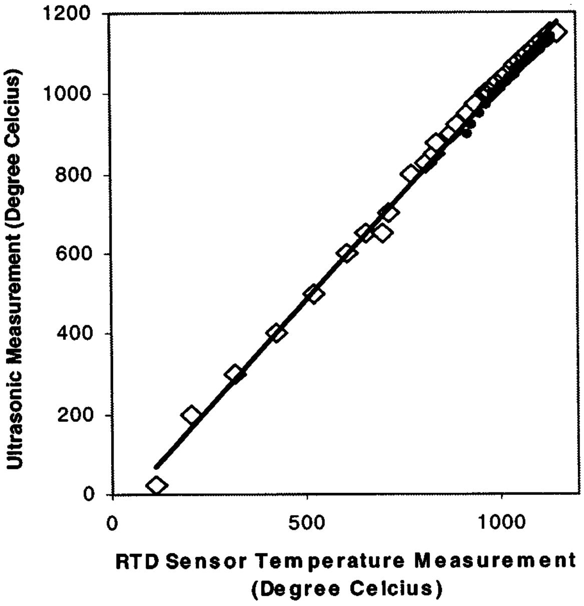

FIG. 5. Temperature measurement comparison between the RTD sensor and the ultrasonic buffer-rod sensor based on measurement in air. The open points represent the heat-up cycle, the closed points are the cool-down cycle, and the solid line represents a one-to-one correspondence.

during subsequent melting-solidification cycles of the same sample. This also indicates that the glass, upon cooling after a melting process, does not bond with the alumina waveguide.

After the initial stage, at a particular temperature, a rapid change in the reflection factor is observed within a small temperature range. This phenomenon was repeatable and consistent for the various glass samples tested. It starts to occur at 800 °C for glass sample IP-745. It is believed that the apparent reduction in the reflection factor may be due to the glass phase transition. It could represent a key phase transition point during the softening of glass. The exact mechanism of the phase change in the glass and its relationship to the ultrasonic shear wave reflection coefficient is beyond the scope of this article.

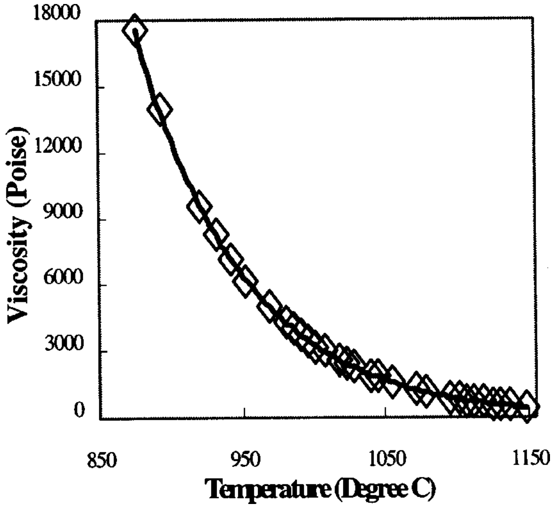

FIG. 7. Calibration viscosity data (the solid line is the best fit curve) as a function of the temperature of the melt, with both parameters measured simultaneously using the ultrasonic technique.

Once the glass is sufficiently softened and complete wetting of the alumina probe surface is accomplished, at a particular viscosity of the melt, a minimum in the reflection coefficient curve is observed. This occurs around 950 °C for IP-745. Here, the ratio of the shear impedance of the melt to the solid is 2.4142 and the minimum in the reflection factor curve is 0.41. It has been shown that the reflection factor of 0.4142 is a characteristic of a Newtonian fluid[17] based on the plane wave impedance model.

As the softening process continues and the viscosity of the melt decreases, there is a gradual decrease in the energy transmitted to the melt and the reflection coefficient increases. The relationship between the reflection coefficient and the melt temperature was found to be nearly linear at temperatures beyond the minimum. It is in this region that the sensor is capable of measuring viscosity.

Figure 7 shows the data for glass sample IP-745 in the temperature regime beyond the minimum in the reflection factor. The reflection coefficient data were used, along with

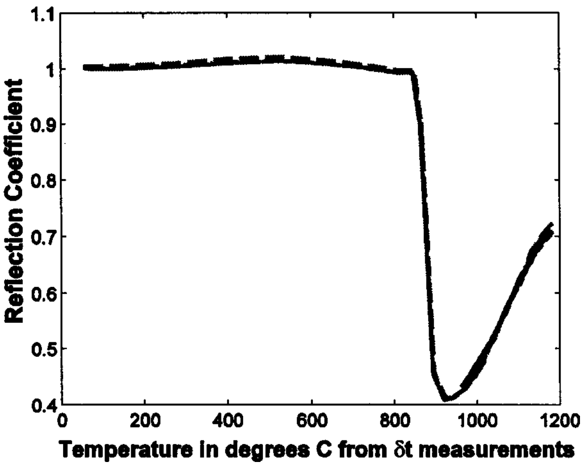

FIG. 6. Reflection coefficient vs ultrasonically measured temperature for IP-745 glass. The three different lines (solid and dashed) represent data collected on different samples on different days.

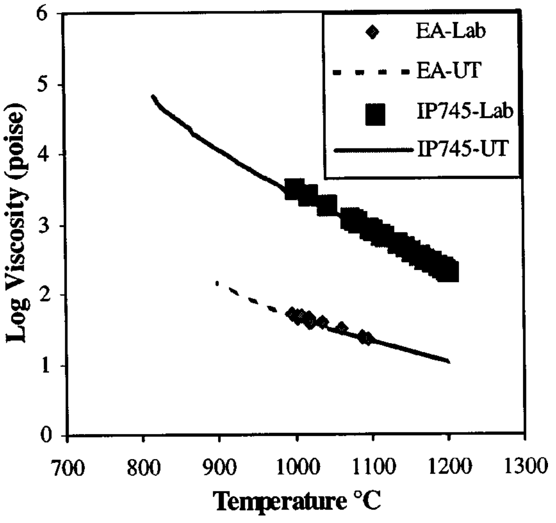

FIG. 8. Comparison between data obtained using ultrasonic buffer-rod data (UT) and independent laboratory data (Lab) for two glass samples, EA and IP-745.

the viscosity–temperature relationship provided by Ferro Glass (using a high temperature rotating type viscometer), to obtain a calibration curve of the viscosity. It must be noted that both the temperature (from δt_M) and the viscosity (from R_M) are both measured simultaneously using the ultrasonic delay line technique. The expected exponential relationship with temperature is observed in this calibration plot. The range of calibration viscosity is between 100 and 20 000 P.

Using the above calibration curve (Fig. 7) for the viscosity two glass samples were examined. The results are presented in Fig. 8. Another sample of IP-745 was used as an unknown glass sample. The second case was an environment assessment (EA) standard cement based borosilicate glass material obtained from Westinghouse Savannah River Corporation. The EA glass is a surrogate glass used in vitrification test melter systems. The results were then compared with the data supplied by an independent laboratory (Corning Glass Laboratories) using a rotating viscometer approach. Figure 8 shows an excellent comparison between the data obtained by the delay line technique and the independent measurements for both of the glass samples considered.

V. DISCUSSION

An *in situ*/on-line sensor to simultaneously measure the viscosity and temperature of molten glass at very high temperatures (up to 1500 °C) has been developed and the potential for application in the glass melt process has been demonstrated. The experiments using the sensor were found to be repeatable, and compared well with other independent measurements of viscosity and temperature. Although the sensor requires calibration for a given delay line type (material and geometry), it does not require re-calibration for different glass samples. The sensor is sensitive in a broad range of viscosities (10–20 000 P). It is believed that this sensor can also be used in lower temperature applications. The current work did not explore other melt processes involving metals and polymers, but it is anticipated that a similar process will be applicable and will have comparable results. For applications such as those in the viscosity measurements, in the very low viscosity ranges (0–10 P), the sensitivity of the technique may not compare favorably with other known techniques.

The key advantages of the technique, over existing methods for measuring viscosity and temperature can be summarized as follows:

(1) Prior on-line methods for viscosity measurements are limited to measurement at room temperature or at temperatures that are not very high (up to 190 °C).[6–8] The technique reported here has the capability to measure viscosity in very hot liquids such as molten metals and molten glass at high temperatures.

(2) This technique has the potential to be employed as an on-line and noninvasive device.

(3) This sensor will provide both temperature as well as viscosity measurements at a point location in the vicinity of the solid–liquid contact zone.

(4) The potential exists for using the reflection coefficient to determine other phase transition properties of the melt process.

It must also be noted that the delay line described in this article was designed for incorporation into the wall of a vitrification melter. Modifications to the geometry of the delay line will allow this technique to fit into other components of a melter such as stop rods, pour ports, etc. Other material configurations like graphite coated with high temperature materials such as platinum, rhenium, iridium, zirconia, etc. (using chemical vapor deposition methods) may yield delay lines which can be used at temperatures higher than 1500 °C and in high oxidation environments.

The technique may be modified for melters where the delay line may not have direct access to the melt. This can be accomplished by bonding the delay line (without the notch) onto the wall or onto the melter component (pour nozzle, stop rod, electrode, etc.). In this case, the refractory material in the melt will become a part of the sensor. Here, the reflected signal between the delay line and the melter component is used as the reference signal, for both time and amplitude measurement.

ACKNOWLEDGMENT

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