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Measurement of viscosity and shear wave velocity of a liquid or slurry for on-line process control

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Abstract

An on-line sensor to measure the density of a liquid or slurry, based on longitudinal wave reflection at the solid–fluid interface, has been developed by the staff at Pacific Northwest National Laboratory. The objective of this research is to employ shear wave reflection at the solid–fluid interface to provide an on-line measurement of viscosity as well. Both measurements are of great interest for process control in many industries. Shear wave reflection measurements were conducted for a variety of liquids. By analyzing multiple reflections within the solid (only 0.63 cm thick—similar to pipe wall thickness) we increased the sensitivity of the measurement. At the sixth echo, sensitivity was increased sufficiently and this echo was used for fluid interrogation. Shear wave propagation of ultrasound in liquids is dependent upon the viscosity and the shear modulus. The data are analyzed using the theory for light liquids (such as water and sugar water solutions) and also using the theory for highly viscous liquids (such as silicone oils). The results show that, for light liquids, the shear wave reflection measurements interrogate the viscosity. However, for highly viscous liquids, it is the shear wave modulus that dominates the shear wave reflection. Since the density is known, the shear wave velocity in the liquid can be determined from the shear wave modulus. The results show that shear wave velocities in silicone oils are very small and range from 315 to 2389 cm/s. Shear wave reflection measurements are perhaps the only way that shear wave velocity in liquids can be determined, because the shear waves in liquids are highly attenuated. These results show that, depending on the fluid characteristics, either the viscosity or the shear wave velocity can be used for process control. There are several novel features of this sensor: (1) The sensor can be mounted as part of the wall of a pipeline or tank or submerged in a tank. (2) The sensor is very compact and can be located within the process stream. (3) The sensor can interrogate and characterize very attenuative liquids or slurries because the sensor operation depends upon reflection at the interface between the solid and the fluid, rather than on transmission through a liquid. (4) The sensor performance is not affected by fluid flow rate, entrained air, or vibration.
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1. Introduction

An on-line sensor to measure the density of a liquid or slurry has been developed by the staff at Pacific Northwest National Laboratory [1–9]. The objective of the research presented here is to extend the sensor's capability to measure viscosity as well. There are many laboratory instruments to measure viscosity, but few to make the measurement on line and in real time. Such an instrument is needed for process control in many different industries. It is well known that the reflection of a shear wave at the interface between a solid and a liquid provides information about the viscosity. In this research multiple echoes within the solid are used to increase the sensitivity of the measurement.

Shear wave reflection measurements were carried out for both water-based fluids and oils: (1) water and sugar water solutions and (2) silicone oils. These data are analyzed using the theory for light liquids and the theory for highly viscous liquids. While measurements of

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viscosity for slurries are not reported here, extensive measurements for the density of slurries using reflection techniques show the equivalence of liquids and slurries for such measurements [5,8,9].

2. Measuring density

An on-line ultrasonic sensor to measure the density of a liquid or slurry is shown schematically in Fig. 1. The sensor consists of longitudinal (B, C, D, E, and F) and shear wave (A) transducers mounted upon a Rexolite™ wedge. The transducers have a center frequency of 2.25 MHz. When ultrasound from transducer F (operating in pulse–echo mode) strikes the wedge–liquid interface, part of it is reflected back toward F and the rest is transmitted into the liquid. Similarly, when ultrasound from transducer D (or B) (operating in pitch-catch mode) strikes the interface, some of it is reflected toward transducer E (or C), some mode converts to a shear wave in the wedge, and part is transmitted into the liquid. The reflection coefficient, which describes the amount of ultrasound reflected to the receive transducer, is dependent upon the densities and speed of sound in the liquid and the wedge material. The reflection coefficient is measured by comparing the voltage on the receive transducer when the base is immersed in the liquid with that when the base is immersed in a reference liquid, usually water. The experimental measurements are described in detail in Refs. [3–9]. The sensor operates by measuring the reflection coefficient at two angles of incidence and then solving for the density of the liquid and speed of sound in the liquid. In the original design shown in Fig. 1, the shear wave transducer A was used to measure the velocity of the shear wave as a function of temperature. In order to measure viscosity, this design was modified by placing transducer A at a distance of only 0.63 cm from the base of the wedge so that multiple echoes could be observed, as will be described.

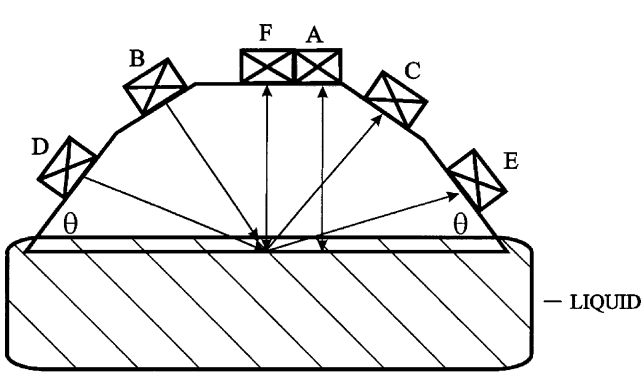

Fig. 1. Ultrasonic wedge immersed in fluid showing transducer configuration and ultrasonic signal paths.

2.1. Bulk measurement

An important question about the reflection methods for density and viscosity measurements is the following: Is the reflection method a bulk measurement or a surface measurement? We consider the experimental evidence. When the density sensor was being developed, a very important step was making the measurements with slurries. The data gave reasonable agreement with the *average* bulk density of the slurry *and not* just the density of the suspending fluid. That is, if the reflection measurement were a surface measurement, then one would expect the reflection to depend upon the suspending fluid because the number of particles in contact with the interface would be quite small. Furthermore, slurries mixed, say, with water, would all give the same density—that of water. This did not happen. Therefore, we conclude that the measurement is a bulk measurement. One is reminded of total internal reflection, when a wave traveling in a solid is transmitted into another material, where it travels at angle θ. When θ is 90°, a surface wave along the interface is produced that decays exponentially with the distance from the interface. This is called an evanescent wave [10,11]. In fact, the characteristic length of the exponential decay approaches infinity, when θ is exactly equal to 90° for a non-absorbing material. Total internal reflection can also occur for an ultrasonic wave traveling from a solid into a liquid. It *may be* that the reflection at a solid–liquid interface has some properties akin to total internal reflection. Additional study is needed on this point.

3. Theoretical considerations

In 1867 Maxwell proposed that the behavior of a liquid is governed by both the viscosity η and the shear modulus G [12] and the relationship is given by:

$$-\frac{\partial u}{\partial y} = \frac{p_y}{\eta} + \frac{\dot{p}_y}{G} \tag{1}$$

where u denotes the velocity of a particle in the liquid. The first term on the right-hand side of Eq. (1) results from the basic definition of viscosity, in which the shearing stress is proportional to the rate of change of the shearing strain. For a solid the shearing stress is proportional to the shearing strain, where the shear modulus G is the proportionality constant. For a liquid, the second term on the right-hand side is essentially the time derivative of the stress–strain relationship for a solid.

Eq. (1) is analogous to the differential equation for the charging of a capacitor in series with a resistor with a time constant RC. In this case the time constant τ is given by

$$\tau = \frac{\eta}{G} \tag{2}$$

3.1. Theoretical calculation of the reflection coefficient and viscosity for light liquids

For water, G is 10^{10} dynes/cm^2; the viscosity is 1 cP; and the relaxation time τ is 10^{-12} s. For light liquids, like water, where the relaxation time is so small, the second term on the right-hand side of Eq. (1) can be neglected [12]. In this case, the propagation of the shear wave in the liquid is determined entirely by the viscosity. This yields:

$$p_y = -\eta \frac{\partial u}{\partial y} \tag{3}$$

A shear wave transducer is placed in a liquid, in which the waves travel in the y-direction with radial frequency ω. Consider a layer of fluid lying in the x–z plane, where the boundaries of this layer are separated by a distance δy. The *change* in pressure δp_y between the two boundaries is given by:

$$-\delta p_y = \eta \frac{\partial^2 u}{\partial y^2} \delta y \tag{4}$$

From Newton's second law, one obtains the following:

$$\delta p_y = \rho \delta y \frac{\partial u}{\partial t} \tag{5}$$

Equating δp from Eqs. (4) and (5), we find:

$$\frac{\partial^2 u}{\partial y^2} + \frac{\rho}{\eta} \frac{\partial u}{\partial t} = 0 \tag{6}$$

If the pressure from the transducer is a sinusoidal traveling wave, then one assumes that u has the form $A \exp \mathrm{j}(\omega t - k' y)$. This is a solution of Eq. (6) only if

$$k' = \left(\frac{\omega \rho}{2\eta} \right)^{0.5} (1 + \mathrm{j}) \tag{7}$$

The basic definition of the acoustic impedance Z is given by p/u. Eq. (3) provides a definition of p, when the quantity $\partial u/\partial y$ is calculated with the restriction given in Eq. (7). The acoustic impedance for a shear wave traveling in a liquid is therefore given by:

$$Z = \left(\frac{\omega \rho \eta}{2} \right)^{0.5} (1 + \mathrm{j}) \tag{8}$$

The reflection of the shear wave at the wedge–liquid interface is due to the difference in the acoustic impedance of the liquid and the wedge material. The following summary of the theory is adapted from Harrison and Barlow [13] and Sheen et al. [14]. The reflection coefficient is given by

$$R^* = \frac{(Z_{\mathrm{liq}} - Z_{\mathrm{s}})}{(Z_{\mathrm{liq}} + Z_{\mathrm{s}})} \tag{9}$$

where Z_{liq} is the acoustic impedance of the liquid and Z_{s}, of the solid. For a viscous liquid, the acoustic impedance is complex and the reflection coefficient R^* is therefore also complex. Defining R^* as

$$R^* = -R \cos\theta + \mathrm{j} R \sin\theta \tag{10}$$

and solving Eq. (9) for Z_{liq}, we find

$$Z_{\mathrm{liq}} = \frac{Z_{\mathrm{s}}(1 - R^2 + \mathrm{j}2R \sin\theta)}{(1 + R^2 + 2R\cos\theta)} \tag{11}$$

If θ is assumed to be less than a few degrees, then $\cos\theta$ is $\sim$1 and Eq. (11) becomes

$$Z_{\mathrm{liq}} = Z_{\mathrm{s}} \frac{(1 - R)}{(1 + R)} + \mathrm{j} Z_{\mathrm{s}} \frac{(2R \sin\theta)}{(1 + R)^2} \tag{12}$$

Equating the real parts of Eqs. (8) and (12) and solving for the density–viscosity product, we find

$$(\rho\eta)^{0.5} = \rho_{\mathrm{s}} c_{\mathrm{TS}} \left(\frac{2}{\omega} \right)^{0.5} \left(\frac{1 - R}{1 + R} \right) \tag{13}$$

where ρ_{S} is the density of the solid and c_{TS} is the shear wave velocity in the solid. This shows how the viscosity is determined from the reflection coefficient R and the density ρ of the liquid.

Later, we shall want to predict the reflection coefficient when the viscosity is known. Solving Eq. (13) for R:

$$R = \frac{1 - K}{1 + K} \tag{14}$$

where K is given by:

$$K = \frac{\left(\frac{\rho\eta\omega}{2} \right)^{0.5}}{\rho_{\mathrm{s}} c_{\mathrm{TS}}} \tag{15}$$

3.2. Considerations for highly viscous liquids

For highly viscous liquids the relaxation time is much larger. Mason [15] and Mason et al. [16] measured a relaxation time for castor oil at 20 °C of 1.6×10^{-6} s. In this case, the term involving the shear modulus G in the differential equation cannot be neglected. The solution to the differential equation is dependent upon the radial frequency, ω (rad/s), of the ultrasound. In this research, the frequency is 2.25 MHz. For castor oil, for example, the product of the radial frequency and the relaxation time is 22.6. When $\omega\tau \gg 1$, the second term on the right-hand side of Eq. (1) dominates and we obtain:

$$\dot{p}_y = -G \frac{\partial u}{\partial y} \tag{16}$$

By following the same steps leading to Eq. (6) (or simply by analogy to Eq. (6)), we obtain:

$$\frac{\partial^2 u}{\partial y^2} + \frac{\rho}{G} \frac{\partial^2 u}{\partial t^2} = 0 \tag{17}$$

The solution for u has the form $A \exp(\omega t - ky)$, where $k = (\rho/G)^{0.5}\omega$.

In order to determine the acoustic impedance, the quantity $\partial u/\partial y$ is calculated and substituted into

Eq. (16). When dp_y/dt is integrated to give p_y, the result is given by:

$$p_y = (\rho G)^{0.5} u \tag{18}$$

$$Z = (\rho G)^{0.5} \tag{19}$$

The shear wave velocity c_T of the liquid can be determined from the solution for u, since

$$c_T = \frac{\omega}{k} = \left(\frac{G}{\rho}\right)^{0.5} \tag{20}$$

The following is obtained by combining the last two equations:

$$Z = \rho c_T \tag{21}$$

Eq. (21) is very similar to that for the impedance of a longitudinal wave. Eq. (20) is the same as that for shear waves in solids.

The reflection coefficient at the solid–liquid interface is used to determine the acoustic impedance of the liquid, which is given by:

$$Z_{liq} = Z_{solid}\left(\frac{1-R}{1+R}\right) \tag{22}$$

Substituting the acoustic impedance into Eq. (19), the shear modulus, G, can be determined, provided the density is also known.

4. Measurements with sugar water solutions

The measurement of viscosity is accomplished by placing the shear wave transducer a distance of 0.63 cm from the base of the Rexolite wedge. This permits multiple echoes to be observed, as shown by the oscilloscope trace in Fig. 2. When shear waves strike the Rexolite–liquid interface, the energy of the shear waves transmitted into the liquid is very small. The reason is that, while liquids easily support a longitudinal wave, liquids do not easily support a shear wave. Thus, the (large) remainder of the energy is reflected back to the shear wave transducer. However, the ability of a liquid to support a shear wave increases with its viscosity. This means that the reflection coefficient, defined as the ratio of reflected ultrasound to incident ultrasound, decreases as the viscosity increases.

By using multiple echoes, small viscosities as well as small changes in the viscosity can be observed. For example, compare two liquids having reflection coefficients of 0.970 and 0.975, differing only by 0.5%. If the sixth echo is observed, then the voltage response is proportional to $0.97^6 = 0.833$ and $0.975^6 = 0.859$, respectively, differing by 3%. The difference between these two voltages is easily observed.

Measurements using the shear wave transducer were carried out using sugar water solutions of different weight percentages. The data acquisition system is the same as that described in Ref. [5]. A tone burst signal of 10 cycles with a frequency of 2.25 MHz was sent to the shear wave transducer. The sixth echo was observed and the maximum amplitude between two cursors was determined for each signal. The amplitude signal was averaged 150 times and recorded. These values are shown in Table 1. The standard deviation of the amplitude signal is very small, less than 0.5%.

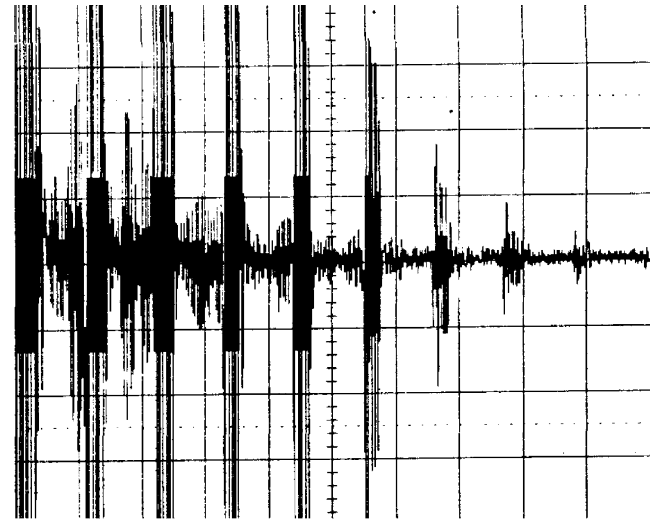

Fig. 2. Oscilloscope trace showing voltage versus time for multiple reflections. The units on the horizontal scale is 10 μs per division and on the vertical scale, 200 mV per division.

The first objective was to simply see if a voltage change could be observed for these liquids because they are not very viscous. Because there are six echoes, the voltage is proportional to the reflection coefficient raised to the sixth power. Using air as a reference liquid with a reflection coefficient of 1.0, the reflection coefficient R for the liquid is given by

$$R_{liq} = R_{air}\left(\frac{V_{liq}}{V_{air}}\right)^{(1/6)} \tag{23}$$

Table 1 shows the density of the liquid obtained by weighing a known volume of the sample and the viscosity measured independently using a Haake viscometer. The Haake viscometer operates by placing a liquid between two coaxial cylinders—one stationary and one rotating—and determining the torque required to turn the cylinder at a given rotational speed. For Newtonian fluids, such as sugar water solutions, the shear stress is proportional to the shear rate du/dy. The viscosity is independent of the shear rate.

The reflection coefficient, the most basic measurement, is obtained from the voltages using Eq. (23). These values, called the experimental values, are given in sixth column of Table 1. Eqs. (14) and (15) show that reflec-

Table 1
Comparison of experimental and predicted reflection coefficients for water and sugar water mixtures. The density and viscosity measurements were obtained independently

Fluid ID	Sixth echo voltage (volts)	Density (g/cm^3)	Viscosity (cP)	$(\rho\eta)^{0.5}$ (g/cm^3 cP)$^{0.5}$	Experimental reflection coefficient, R	Predicted reflection coefficient, R
Air	0.3125					
Water	0.3032	1.000	1.000	1.000	0.995	0.996
10% SW	0.3002	1.038	1.332	1.176	0.993	0.995
20% SW	0.3013	1.097	1.873	1.433	0.994	0.994
30% SW	0.2996	1.121	3.165	1.884	0.993	0.992
40% SW	0.2910	1.196	7.145	2.923	0.988	0.987
50% SW	0.2829	1.232	12.385	3.890	0.984	0.983
60% SW	0.2047	1.287	93.85	10.990	0.932	0.952

tion coefficient can be predicted, if basic properties of the liquid and solid (Rexolite) are known. The objective is to compare the experimental value of the reflection coefficient with these predicted values, shown in the last column of Table 1. For this calculation the density of Rexolite is 1.049 g/cm^3, the longitudinal speed is 2.337E+05 cm/s, the shear speed is 1.157E+05 cm/s, and the frequency is 1.414E+07 rad/s (2.25 MHz). Excellent agreement is obtained between the experimental and predicted values of the reflection coefficient. Unfortunately, when the Haake viscometer measured the viscosity of the 60% sugar water solution, the fluid temperature was higher than the fluid temperature when obtaining the ultrasonic value, so there is a 2% error associated with this measurement. Fig. 3 shows a graph of the reflection coefficient versus the density viscosity product, $(\rho\eta)^{0.5}$. The squares are the experimental data points and the solid line is the predicted value.

5. Reflection coefficient measurements for silicone oils

Data were also obtained for silicone oils that are used as viscosity standards. Two standard fluids were mixed to produce those labeled "A", "B", and "C". The results are shown in Table 2. The experimental value of the reflection coefficient was obtained using the sixth echo, as has been described previously, and compared with the voltage obtained for air. The measurements of density were obtained by weighing and the independent measurement of the viscosity was obtained using the Haake viscometer. The silicone oils are non-Newtonian fluids, which means that the viscosity is dependent upon the shear rate. Since outdated viscosity standards were being used for these measurements, the shear rate for the viscosity measurement was chosen to be in approximate agreement with the labeled viscosity, as indicated by the Fluid ID in Table 2.

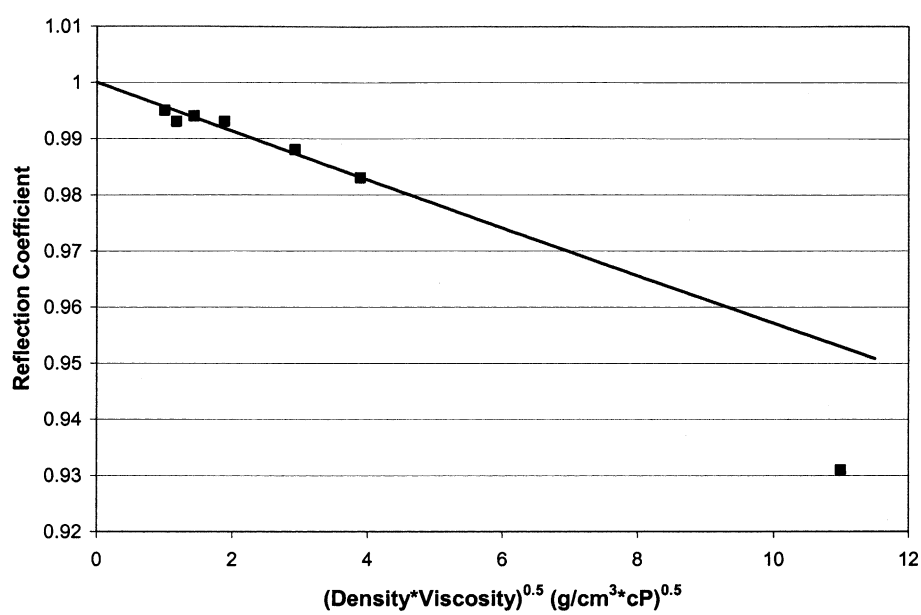

Fig. 3. Comparison of experimental reflection coefficients for water and sugar water solutions with theory for light liquids.

Table 2
Comparison between experimental and predicted reflection coefficients for silicone oils

Fluid ID	V_{liq}/V_{air}	Density (g/cm^3)	Viscosity (cP)	$(\rho\eta)^{0.5}$ (g/cm^3 cP)$^{0.5}$	Experimental reflection coefficient, R	Predicted reflection coefficient R for light liquids
Fluid 5	0.9723	0.9075	4.21	1.955	0.9953	0.9915
C	0.9513	0.9314	7.04	2.561	0.9917	0.9888
Fluid 10	0.9434	0.9409	9.026	2.941	0.9903	0.9873
A	0.8924	0.9580	27.92	5.172	0.9812	0.9776
B	0.8660	0.9606	34.18	5.730	0.9763	0.9752
Fluid 50	0.8440	0.9657	46.80	6.723	0.9721	0.9710
Fluid 100	0.8362	0.9666	92.52	9.457	0.9706	0.9594
Fluid 500	0.8004	0.9777	458.9	21.182	0.9636	0.9113
Fluid 1000	0.7934	0.9786	934.0	30.233	0.9622	0.8758

5.1. Comparison of data with theory for light liquids

As the data were being obtained for the reflection coefficient, some very interesting—and puzzling—results were observed, as shown in Table 2. Note that for Fluid 50 and Fluid 100, the experimental reflection coefficients are the same (within experimental error) even though the viscosities are different by a factor of 2! A similar statement can be made for Fluid 500 and Fluid 1000. When the density was measured for the samples, the densities for Fluid 50 and Fluid 100 are very close in value and similarly, for Fluid 500 and Fluid 1000. This suggests that the reflection coefficient is dependent upon the density of the fluid, rather than upon the product of density and viscosity. More will be said about this shortly. First, however, let us compare the experimental values of the reflection coefficient with the predicted value of R obtained from Eq. (14) for light liquids. The independently measured values of the density and viscosity are used to calculate the predicted value of R. The results are shown in Fig. 4. The experimental and predicted values are in good agreement for samples having a viscosity less than 50 cP. However, the agreement is *very* poor for samples having a viscosity greater than approximately 50 cP. We now consider the observation that the reflection coefficient depends upon density alone, rather than on the product of density and viscosity.

5.2. Highly viscous liquids and measurement of the shear wave velocity in a liquid

The theory of highly viscous liquids is now applied to the data obtained for silicone oils, as shown in Table 3. The viscosity and density values are given in Table 2. Table 3 shows the impedance of the liquid Z_{liq} obtained from Eq. (22), using the experimental value of the reflection coefficient R and the acoustic impedance of the solid (1.214E+05 g/cm^2 s). The shear velocity c_{Tliq} is obtained from the acoustic impedance of the liquid and density of the liquid, as shown in Eq. (21). The shear modulus G is obtained from Eq. (19).

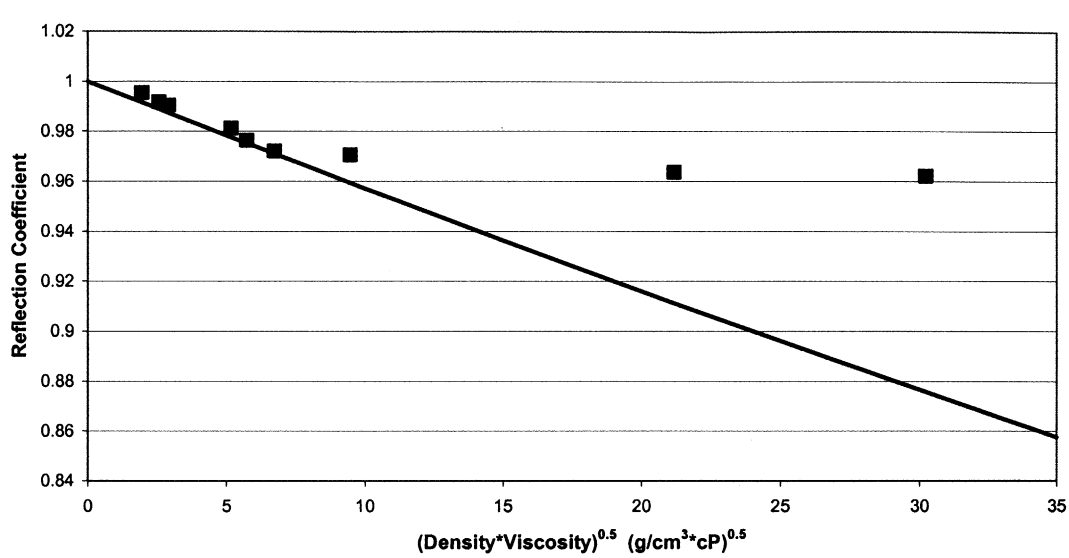

Fig. 4. Comparison of experimental reflection coefficients for silicone oils with theory for light liquids.

Table 3
Comparison of theory for highly viscous liquids with silicone oil data

Fluid ID	Experimental reflection coefficient, R	Z_{liq} (g/cm^2 s)	Shear speed of liquid (cm/s)	Shear modulus G (dyne/cm^2)	Relaxation time (s)	$\omega\tau$
Fluid 5	0.9953	285.9	315	90 064	4.67E−07	6.6
C	0.9917	505.8	543	274 656	2.56E−07	3.6
Fluid 10	0.9903	591.5	629	371 861	2.43E−07	3.4
A	0.9812	1151.7	1202	1 384 558	2.02E−07	2.9
B	0.9763	1455.5	1515	2 205 292	1.55E−07	2.2
Fluid 50	0.9721	1717.1	1778	3 052 994	1.53E−07	2.2
Fluid 100	0.9706	1810.7	1873	3 392 100	2.73E−07	3.9
Fluid 500	0.9636	2249.9	2301	5 177 365	8.86E−07	12.5
Fluid 1000	0.9622	2338.1	2389	5 586 111	1.67E−06	23.6

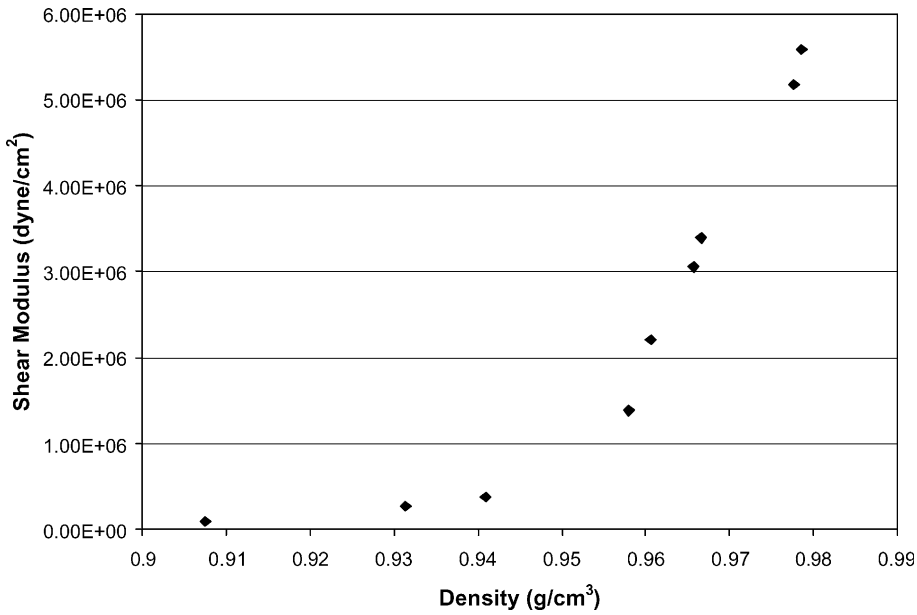

Fig. 5. Shear modulus for silicone oils, obtained using theory for highly viscous liquids, plotted versus density.

It is important to note that the shear wave velocities in Table 3 are *very* small. *This method is perhaps the only way in which the shear wave velocity in viscous liquids can be determined.* A time-of-flight measurement would be very difficult to implement because the shear wave is highly attenuated as it travels through the liquid.

As described earlier, highly viscous liquids have a large relaxation time τ. Also, for the acoustic impedance of the liquid to be defined by Eq. (19) the quantity $\omega\tau$ should be greater than 1. Since G has been determined and the viscosity has been determined using the Haake viscometer, the relaxation time τ can be determined from Eq. (2). Here we see that the relaxation times are much greater than that for water (1.6E−12 s). For Fluid 1000 the relaxation time is very close to that for castor oil, 1.6E−06 s. The quantity τ is shown in the last column of Table 3. All values of $\omega\tau$ are greater than 1, but only two are greater than 10. Thus, both the relaxation time and the quantity $\omega\tau$ indicate that the silicone oils are represented by the theory for highly viscous liquids.

Fig. 5 shows a graph of the shear modulus G (Table 3) versus the density (Table 2). This graph does show a smooth progression, rather than the abrupt change noted in Fig. 4. Therefore, for highly viscous liquids, these data indicate that the reflection coefficient is a measure of shear modulus G and not the viscosity, when the ultrasound frequency is 2.25 MHz. A theoretical curve has not been calculated to compare with the data in Fig. 5.

6. Conclusions

The results of these experiments at 2.25 MHz show that aqueous solutions, such as sugar water solutions and slurries, are best characterized by the theory for light liquids, in which the reflection coefficient is dependent upon the product of the density and viscosity of the liquid. For oils, the data are best characterized by the theory of highly viscous liquids where the reflection coefficient is dependent upon the shear modulus (or the

shear wave velocity). Therefore, this technique can be applied to measure the viscosity of water-based solutions and slurries and can be used for real-time process monitoring. For oil-based products, changes in the shear modulus (or shear wave velocity) can also provide a method for real-time process characterization and monitoring. An on-line sensor has been designed to measure density and viscosity. The base of the Rexolite wedge is part of the pipeline wall. There are several novel features of this sensor. An important one is its small size. Liquids or slurries that severely attenuate ultrasound are easily characterized because the sensor operation depends upon reflection at an interface, rather than transmission through a liquid. The sensor is not affected by fluid flow rate, entrained air, or vibrations.

7. Further research

We have found that the viscosity sensor is sensitive to changes in viscosity when operated at stable ambient temperature and that we can measure low values of the viscosity. With an increase in temperature, the shear wave attenuation increases in Rexolite. This change affects the reference voltage in air and thus the value of the experimental reflection coefficient. The shear wave velocity is also temperature dependent [5]. Therefore, calibration as a function of temperature is required. The advantage of using a plastic wedge for the sensor is its low acoustic impedance. Additional research is needed to carry out viscosity measurements using a sensor material with a reasonably low acoustic impedance that is also less affected by temperature. The objective is to develop a robust sensor to measure the density and viscosity/shear wave velocity.

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