

# IMMERSION APPARATUS FOR ULTRASONIC MEASUREMENTS

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ABSTRACT: An immersion apparatus for making ultrasonic measurements was constructed. This apparatus is used to measure longitudinal and shear sound speeds in polymeric materials as a function of temperature as well as sound absorption measurements. Seven polymers were evaluated and the measurements all compared favorably with available literature values. Using the immersion apparatus, sound speed measurements are accurate to ±2 percent while absorption measurements are accurate to ±10 percent.

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IMMERSION APPARATUS FOR ULTRASONIC MEASUREMENTS

This report describes experimental work done in constructing an apparatus for measuring ultrasonic properties and illustrative measurements using this apparatus. The purpose of such measurements is to evaluate materials for potential acoustic applications such as underwater sound damping.

Most of this report was taken from a dissertation submitted by Bruce Hartmann to the faculty of the American University in partial fulfillment of the requirements for the degree of Doctor of Philos phy in physics.

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> ROBERT WILLIAMSON II Captain, USN Commander

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#### INTRODUCTION

The work reported here is a continuation of our previous investigations<sup>1-3</sup> of the ultrasonic properties of polymers. The goal of this work is to evaluate materials for potential acoustic applications such as underwater sound damping. Of particular interest are the ultrasonic properties of partly crystalline polymers through the melting point.

Cur earlier work using an immersion apparatus<sup>2</sup> demonstrated the usefulness of such equipment but was limited to longituinal wave measurements. Shear wave measurements are also of interest not only in themselves but also because knowledge of both shear and longitudinal sound speeds (and density) allows one to calculate the elastic constants of a material. Therefore, it was decided to construct an immersion apparatus with a capability for both longitudinal and shear wave measurements.

The work on absorption at the molting point  $^{1}$ ,  $^{3}$  was done by using delay rods. These measurements were difficult because the acoustic bond between the specimen and the delay rods changed as the specimen temperature was raised to the melting point. Immersion techniques avoid this difficulty since the acoustic coupling comes from the intimate contact of the immersion liquid and is very reproducible. For this reason as well as those mentioned in the previous paragraph, an immersion apparatus is preferred.

The principal conclusions of this work are that the immersion apparatus is convenient for measuring both longitudinal and shear sound speeds and absorptions as functions of temperature in polymers; sound speeds are accurate to  $\pm 2$  percent while absorption measurements are accurate to  $\pm 10\%$ . The remainder of this report describes the immersion apparatus, its operation, and typical data obtained by its use.

#### DESCRIPTION OF APPARATUS AND EXPERIMENTAL TECHNIQUE

Ultrasonic absorption and sound speed measurements are made by converting an electrical signal into an ultrasonic signal of the same frequency and measuring the decrease in amplitude and time of flight of this signal as it passes through the specimen of interest. The data presented here was obtained using an immersion apparatus where the specimen was immersed in a bath of silicone liquid. A major advantage of the immersion method is that it avoids the unknown acoustic losses in the bonds which couple the ultrasound to the specimen. This source of error is present in most other methods.

## A. Electronics.

The purpose of the electronics is to generate electrical pulses of a given frequency, which are then converted to ultrasonic pulses, and to detect the pulses which have gone through the specimen. Three different electronic arrangements were used, depending on the frequency desired.

At 0.6 MHz, the setup used is shown in Figure 1a. A continuous sinusoidal signal, 20 volts peak, is generated by the oscillator (Hewlett-Packard 200 CD). The output of the oscillator is applied to a transmission gate. The gate is a circuit in which the output is an exact reproduction of the input waveform during a selected time interval and is zero otherwise. The time interval for transmission is selected by an externally impressed signal which is called the gating signal and is usually rectangular in wave-shape. Thus, the output from the gate is a pulsed sinusoidal signal which is then amplified by the power amplifier (General Radio 1233 A) and applied to the transmitting transducer which converts the electrical signal to an ultrasonic signal of similar wave-shape.

After passing through the immersion device, the ultrasound is converted to an electrical signal by the receiving transducer. This electrical signal is then amplified by the preamplifier (Tektronix Type L Plug-In Unit) and displayed on the oscilloscope (Tektronix 535 A).

The pulse generator produces a rectangular pulse which is the gating signal controlling the transmission period of the gate. The generator is triggered by the line voltage and its repetition rate is 60 Hz. A synchronizing circuit is used to lock the gating signal to the output signal from the oscillator, so the start of the gating signal always coincides with a particular mase of the oscillator output. A delay circuit within the puls. generator enables one to adjust the start of the gating signal to coincide with any desired phase of the oscillator signal. Usually, the phase chosen is that corresponding to zero voltage level of the oscillator signal. Then the pulsed sinusoidal signal applied to the transmitting transducer starts from zero voltage level and does not contain any abrupt voltage steps which would generate higher frequency harmonics. The pulse generator also generates a pulse which triggers the horizontal sweep of the oscilloscope. This trigger signal occurs approximately 10 used before the gating pulse. Thus, it is possible to display on the oscilloscope both the signal applied to the transmitting transducer and the signal from the receiving transducer.

One of the main features of this electronic setup is the inclusion of a synchronizing circuit between the oscillator and the pulse generator. Therefore, both the trigger pulse to the oscilloscope and the gating pulse are phase-locked to the oscillator output. This eliminates any jitter in the ultrasonic

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signal displayed on the oscilloscope and allows amplitude and time-of-flight measurements to be made on the first few cycles of the ultrasonic signal. Detailed circuit analysis of the electronics used at 0.6 MHz has already been published.<sup>1</sup>

At 2.2 MHz, a very simple arrangement is used, as shown in Figure 1b. A continuous sinusoidal signal from the oscillator (Hewlett-Packard 650 A) is applied, along with a pulse from the pulse generator (Hewlett-Packard 212 A), to the gate. In this case, the gate is a mixer (Hewlett-Packard 10514 A) being used as a pulse modulator, and its output is a pulsed sinusoidal signal of the desired frequency and length. The pulse is amplified by the power amplifier (General Radio 1233 A) and applied to the immersion device. As before, the output from the immersion device is applied to the pre-amplifier and then displayed on the oscilloscope. Triggering is accomplished by the same pulse generator used to modulate the oscillator. The oscilloscope is triggered a few used before the gating action.

In distinction to the lower frequency apparatus the triggering does not occur at any specified phase of the signal, and there is some jitter in the beginning of the signal. This jitter leads to an uncertainty in determining the start of the pulse on the order of one cycle. However, this is of lesser importance at the higher frequencies.

At 5.5 MHz, the absorption is high and special amplification of the signal is required. A block diagram of the apparatus is shown in Figure 2. Triggering of the electronic circuits is accomplished by a pulse generator as before. This pulse generator is preceded by a Schmitt trigger and an isolation transformer. The Schmitt trigger converts the sinusoidal line voltage signal to a square wave of the same frequency. This signal is the external trigger to the pulse generator whose output is a pulse of variable length, with a repetition rate of 60 Hz. This repetition rate is used to minimize the jitter due to the 60 Hz ripple voltage which is present in the output of the various power supplies in the cir-The direct output of the pulse generator is applied to a cuit. transmitter whose tank circuit coil was picked to resonate at the test frequency. The pulses from the transmitter then pass through a matching unit which acts as a step-up transformer and also tunes out the static capacitance of the transducer used to generate the ultrasonic wave. The resulting signal is then applied to the immersion device.

After passing through the immersion device, the signal passes through another matching unit, identical to the first one. The signal is highly attenuated and needs considerable amplification before being displayed on the oscilloscope. For this purpose, the intermediate frequency strip of a radar receiver (Ostby Barton PR 202) is used. Since the intermediate frequency of the receiver is 60 MHz, the signal from the immersion device is added via a mixer to a signal from a VHF oscillator (Hewlett-Packard 3200 B) such that the sum of the two frequencies is 60 MHz. The resulting

sum is then amplified and demodulated by the receiver and displayed on the oscilloscope. The oscilloscope is triggered by the same signal that triggers the transmitter. There is, however, a time delay of the ultrasonic signal in passing through the immersion device. Photographs of some typical signals are shown in Figure 3.

#### B. Acoustic System.

One of the major experimental problems in ultrasonic measurements is to find a reproducible way to introduce the sound wave into the specimen and detect the sound wave leaving the specimen. With either the transducers directly touching the specimen or delay rods between the specimen and the transducers, the specimen must be bonded to another material. This bond can be a grease or a more permanent bond like epoxy. The bond must provide good transmission of the sound wave and be reproducible. Further, it must maintain its characteristics over the temperature range of the measurements. Due to the number of variables involved, such as bond thickness, pressure used to form the bond, specimen parallelism, etc., it is difficult to make accurate absorption measurements with either delay rods or direct transducer contact. method which avoids these problems is the immersion technique. In this method, the transducers and specimen are all immersed in a liquid. Coupling between the transducers and the specimen is provided by the intimate contact of the liquid and is very reproducible.

Measurements are made with and without the specimen in the path of the sound beam and with specimens of different thickness. This method has been used with the specimen perpendicular to the path of the sound beam by several authors.<sup>4-7</sup>,<sup>2</sup> A variation of this method is to hold the specimen at an angle to the sound beam. In this manner, both longitudinal and shear waves are generated in the specimen at the first interface. If the angle at which the specimen is held is greater than the critical angle, the longitudinal wave is totally internally reflected and only the shear wave is converted at the second interface to a longitudinal wave in the immersion liquid. This technique has also been used by several authors.<sup>6-11</sup>

A typical immersion device is shown in Figure 4a. The specimen is held vertically and is rotated with respect to the transducers to obtain shear waves. A different arrangement was used in the present work. The specimen is held horizontally and the transducers are rotated to obtain shear waves, as shown in Figure 4b. This arrangement was used because it was desired to make measurements to above the melting point of polymers. At room temperature, the specimens are held on four pointed tip screws. This method of mounting is not usable at the melting point of a partly crystalline polymer. Some measurements were done in which the specimens were supported by a thin foil of aluminum. The foil was stretched tight between two hoops. The specimen was then rested on the foil

using either a thin layer of silicone liquid or silicone grease for coupling. The foil thickness was chosen so that, even at 5.5 MHz, the ultrasonic wavelength (0.05 cm) was ten times the foil thickness. Measurements showed that the foil had no effect on sound speed determinations but had a considerable effect on absorption measurements, especially at 5.5 MHz. For example, when a 1.903 cm thick specimen was inserted in the path of the sound beam, the amplitude of the ultrasonic signal decreased by  $\delta$ .9 db when the specimen was screw mounted but decreased by 18.3 dF when the specimen was foil mounted.

The problem of specimen support was solved by using a disk mounting in which a 5.% om diameter specimen rests on a flat aluminum disk with a 2.5 cm diameter hole in it. Over most of the area of the specimen, it is supported by the disk. Only the center portion is not supported. The hole in the aluminum is cut at an angle to enhance reflection of the sound energy at the edges. Only the sound that goes straight through the specimen is detected by the receiving transducer.

Photographs of the immersion device are shown in Figures 5 and 6. In Figure 5, the specimen is held perpendicular to the path of the sound beam, while in Figure 6, the transducers have been rotated. The entire device is immersed in a liquid to above the top transducer but below the control knobs. Measurements are made with different specimens or no specimen by turning the selector shaft. In a later modification of this apparatus, provision was made to hold eight specimens rather than just two.

The transducers used are lead zirconate titanate, 1.54 cm in diameter and 0.25 cm thick, with a resonant frequency of 0.75 MHz. The center wire of a miniature coaxial cable is soldered to the back of the transducer for one electrical lead. The ground lead is that part of the immersion device which presses against the front face of the transducers. The brass transducer holders are open to the atmosphere, thus providing an air backing for the transducers, which improves their operation and reduces multiple ultrasonic echoes.

Rotation of the transducers is accomplished by turning the rotation knob. There is a pointer on this knob and a scale attached to the instrument so that the angle of incidence of the sound wave can be read to the nearest degree from  $0^{\circ}$  to  $90^{\circ}$ .

One other feature is built into the device because of its use with shear waves. When a shear wave goes through a specimen, the emerging beam is displaced from the direction of the incident beam. (A similar effect is observed in optics when a light beam passes through a plate of glass, in air, at an angle.) If the transducers are directly facing each other, the displacement will cause some of the sound energy to miss the receiving transducer, causing a reduced amplitude and thus giving a false contribution to the measured absorption. To correct for this, the transducers are rotated in opposite directions about an axis perpendicular to the

main rotation axis. This rotation is accomplished with the displacement knob (Fig. 5). The knob is rotated until the observed signal is a maximum; hence no rotation scale is required.

As mentioned above, the transducers are 2.54 cm in diameter. The specimens used were about 5 cm in diameter in case there was any beam spreading, especially at lower frequencies, and so that specimen alignment would not be critical. The specimen thickness was governed by the magnitude of the absorption and varied from 0.5 cm to 2.5 cm.

A silicone fluid (polydimethylsiloxane) is used as the immersion liquid. This liquid is made by the Dow Corning Corporation, Midland, Michigan, under the trade name DC 200. The grade used here has a room temperature viscosity of 100 cs. To prevent swelling, all rubber gaskets in contact with the liquid are made of a chlorinated silicone rubber. Most of the immersion device is aluminum and all these parts are anodyzed.

#### C. Temperature Control.

Specimen temperature control is obtained by using two 1-kilowatt continuous heaters immersed in the tank holding the immersion device. The current to these heaters is controlled by a variac, and in this manner the temperature can be controlled to about  $\pm 0.5^{\circ}$ C. A stirrer is used to promote good mixing and approximate isothermal conditions. The stirrer is run continuously. The temperature is read using a chromel-alumel thermocouple immersed near the specimen. The output of this thermocouple is measured using a precision potentiometer. The highest temperature that can be attained is determined by the gelation point of the silicone fluid and is about 150°C. The outside of the immersion tank is covered with a high-temperature polyurethane foam, 2.5 cm thick. (Some measurements were also done below room temperature by cooling the silicone liquid with dry ice.)

Thermal gradients in the silicone liquid were also found to be a problem. Partly because of the high viscosity of the silicone liquid, the temperature is not uniform throughout the tank. The temperature gradients cause changes in the signal amplitude as the thermal currents pass between the transducers, and this effect is particularly troublesome at the highest frequency used. The thermal currents were reduced by installing brass foil tubes over the transducer holders. The tubes go from one transducer to the other with the exception of the central region where the specimen is located.

Also, the silicone liquid was found to out-gas as the temperature is raised. Air bubbles then tended to collect under the specimen and the upper transducer interfering with the absorption measurement. These air bubbles are wiped away just prior to making measurements. (Above 704C, this problem no longer existed.)

#### D. Measurement Technique.

Sound speed measurements are made by measuring the change in the time of flight of the ultrasonic pulse when the specimen is removed from the path of the sound beam. The difference in time of flight is due to the difference in sound speed between that in the liquid and that in the specimen. Thus, the first step is to find the sound speed in the liquid over the temperature range used. This is done by using an aluminum specimen as a standard. For 6061-T6 aluminum, the longitudinal sound speed as a function of temperature is given by 12

$$v_{\ell}(A1) = 6428 - 0.91 t$$
 (1)

in m/sec with t in degrees centigrade. For a specimen of thickness L, the difference in time of flight,  $\lambda \theta$ , without and with the specimen perpendicular to the path of the sound beam is

$$\mathbf{a} = \frac{\mathbf{L}}{\mathbf{v}(11q)} - \frac{\mathbf{L}}{\mathbf{v}_{\ell}(\text{spec})}$$
(2)

where v(liq) is the sound speed in the liquid and  $v_{\ell}(spec)$  is the longitudinal sound speed in the specimen. For an aluminum specimen  $v_{\ell}(spec)$  is known, equation (1), and so v(liq) can be found from AP mean ements. In this manner, the results shown in Figure 7 were oblined. The data is given very accurately by

$$v(liq) = 976 - 2.5 (t - 25)$$
 (3)

which is in reasonable agreement with other measurements.<sup>13</sup> Knowing (3), the sound speed in the polymer can now be found from (2). This is the method used to find the longitudinal sound speed as a function of temperature in polymers.

For calculating shear sound speeds, the procedure is a little more complicated because the path length through the specimen is not just equal to the specimen thickness. Referring to Figure 8,

$$\Lambda \mathbf{a} = \frac{\mathbf{y}}{\mathbf{v}(\mathbf{liq})} - \frac{\mathbf{x}}{\mathbf{v}_{\mathbf{s}}(\mathbf{spec})} \tag{4}$$

where v<sub>c</sub>(spec) is the shear sound speed in the specimen. Since

7

$$\cos \varphi = L/x \tag{5}$$

$$\cos (\varphi - \Psi) = y/x \qquad (6)$$

and Snell's Law is just

$$\frac{\sin \psi}{\sin \varphi} = \frac{v(liq)}{v_{g}(spec)}$$
(7)

with a little manipulation, equation (4) can be put in the form

$$\mathbf{v}_{s}(\text{spec}) = \mathbf{v}(\text{liq}) \left[ (\cos \Psi - \mathbf{v}(\text{liq}) \wedge \theta / L)^{2} + \sin^{2} \Psi \right]^{-\frac{1}{2}} (8)$$

Ultrasonic absorption is found in the following manner. The amplitude of the envelope of the ultrasonic pulse that has gone through a specimen of thickness  $L_1$  is measured and similarly for a specimen of thickness  $L_2$ . When the ultrasonic pulse impinges on the specimen, part of the sound energy is reflected due to the acoustic impedance mismatch. The amount of reflection is assumed to be the same for both specimens. Then the difference in measured amplitude is due to the difference in absorption between the specimen and the immersion liquid occurring in a thickness equal to the difference in thickness of the two specimens. The absorption, in dB/cm, is then

$$= \alpha(liq) + \frac{20}{L_2 - L_1} \log \frac{A_1}{A_2}$$
(9)

where  $\alpha(\text{liq})$  is the absorption in the liquid and A<sub>1</sub> and A<sub>2</sub> are the amplitudes measured for the specimens of thickness L<sub>1</sub> and L<sub>2</sub>. Greater accuracy will, of course, be obtained if more than two specimens are used. The absorption in the liquid was calculated from McSkimin's data.<sup>14</sup> At 5.5 MHz and 25°C, the absorption in the immersion liquid used here is 1.6 dB/cm. This absorption depends on the square of the frequency.

 $\alpha$ 

For shear waves, the distance traveled by the shear wave is x (Fig. 8) where

$$x = L[1 - (v_s(spec) \sin \frac{\psi}{v(liq)})^2]^{-\frac{1}{2}}$$
(10)

Thus, using the difference in distance traveled by the shear wave in going through the specimens of thicknesses  $L_1$  and  $L_2$ ,

$$\gamma_{\rm s} = \frac{20}{L_2 - L_1} \left[1 - (v_{\rm s}({\rm spec}) \sin \psi/v({\rm liq}))^2\right]^{\frac{1}{2}} \log A_1/A_2$$
 (11)

In some cases, the absorption is so high that a measurable signal can be propagated only through the thinnest specimen. With only one specimen, equation (9) for absorption cannot be used. An alternate approach is based on the fact that the difference in signal detected with and without the specimen in place is due to the reflection at the top of the specimen, the absorption in the specimen, and the reflection at the bottom of the specimen. (This neglects standing waves in the specimen, a good assumption when the absorption is high and the ultrasonic pulse is short enough that successive echoes in the specimen are well separated.) As pointed out by Maeda? and Asay et al, 12 the absorption is given by

$$\alpha = \alpha(\text{liq}) + (L_{I} - L_{R})/L \qquad (12)$$

8

where L is the thickness of the specimen,  $L_I$  is the insertion loss and  $L_R$  is the reflection loss. The insertion loss, in dB, is 20 times the (common) logarithm of the ratio of the received amplitude without and with the specimen in the path of the sound beam. The reflection loss is the dB loss caused by reflection at the two surfaces of the specimen.  $L_R$  can be calculated from the relation

$$L_{\rm R} = 20 \log \left[ \frac{(Z_1 + Z_2)^2}{4Z_1 Z_2} \right]$$
(13)

where  $Z = \rho v$  is the acoustic impedance, subscript 1 refers to the liquid and subscript 2 refers to the specimen.

#### E. Materials Used.

The materials used in this work were all commercial products for which sound speed measurements were available in the literature. They were machined to a nominal test size of 5.08 cm (2.000 in) diameter and 1.27 cm (0.500 in) thick, top and bottom parallel to within 0.003 cm (0.001 in). A description of the seven materials used is given in Appendix A.

#### ILLUSTRATIVE RESULTS

In order to check out the apparatus and to determine its experimental accuracy, measurements were made of sound speeds and absorptions in some common polymers. These measurements were then compared with literature values.

#### A. Sound Speeds.

Since the electronics at 0.6 MHz is designed so that the start of the pulse always occurs at the same phase of the signal, measurements of sound speed at this frequency are more accurate than at 2.2 MHz, where the beginning phase is constantly changing. The equipment at 5.5 MHz is not designed in such a way as to readily permit sound speed measurements. As accurately as can be determined, there is no difference in sound speed between the 0.6 MHz and 2.2 MHz measurements for the polymers examined.

Measured sound speeds at room temperature and a frequency of 0.6 MHz for seven common polymers are listed in Table 1 along with literature values. 10, 12, 15-20 Precise comparisons with this literature data are difficult because of the variations that exist in what is presumably the same polymer. There are also some differences in temperature and test frequency. Based on the reproducibility of repeated measurements on the same specimen, the estimated uncertainty of our measurements is  $\pm 2\%$ . Shear

measurements are more difficult to make (and less accurate) than longitudinal measurements. In fact, no reliable shear measurement was possible for polytetrafluoroethylene.

The above measurements were all made at room temperature. Sound speed measurements as a function of temperature were made on polymethylmethacrylate, polyethylene, and polypropylene. Results for polymethylmethacrylate are shown in Figures 9 and 10 along with literature values.<sup>15,10</sup> Results for polyethylene are shown in Figures 11 and 12 along with literature values.<sup>21,20</sup> Results for polypropylene are shown in Figure 13 along with literature values.<sup>17,10</sup> Again, the uncertainty of our measurements is estimated to be about ±2%.

#### B. Absorptions.

For polymethylmethacrylate and polyethylene, sound absorptions were measured at room temperature and 5.5 MHz. Four polymethylmethacrylate specimens were used, of thickness 0.632, 1.270, 1.903, and 2.540 cm respectively. The measured longitudinal absorption was 5.1 dB/cm and the shear absorption was 12.8 dE/cm. Using the sound speeds for polymethylmethacrylate from Table 1, the longitudinal wavelength is 0.049 cm while the shear wavelength is 0.024 cm. Then the longitudinal absorption per wavelength (loss per cycle) is 0.25 dB compared with 0.23 dB found by Asay et al.<sup>15</sup> Our measured shear absorption per wavelength is 0.31 dB compared with 0.32 dB found by Asay et al.<sup>15</sup> Comparisons of absorption data with literature values are even more difficult than with sound speed data because the sound absorption is much more sensitive to small differences in polymer density, preparation, purity, etc.

Three polyethylene specimens were used: of thickness 1.270, 2.032, and 2.526 cm respectively. The measured longitudinal absorption per wavelength was 0.5 dB compared with 0.7 dB found by  $Eby^{21}$  on a material of higher density. No shear absorption measurements on a comparable polyethylene were found.

Considering the variations in absorption to be expected from specimen to specimen variation, the above comparisons with literature values are considered good. The estimated uncertainty of the absorption measurements is  $\pm 10\%$ .

#### CONCLUSIONS AND RECOMMENDATIONS

An apparatus has been constructed to measure longitudinal and shear sound speeds and absorptions from room temperature to about  $150^{\circ}$ C. Measurements were made on a total of seven common polymers for which comparisons with literature values were possible. Sound speed measurements are estimated to be accurate to  $\pm 2\%$  while absorption measurements are accurate to  $\pm 10\%$ .

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It is recommended that this apparatus be used to determine the elastic constants of polymers, preferably as a function of temperature, and to determine the absorption in partly crystalline polymers at their melting point.

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	Reference	This work 12 16	<b>This work</b> 10 12 15	This work 18 17	This work 12	This work 12 20	This work 10 16	This work 16 12
TH LITERATURE VALUES	Shear speed,* m/sec	1120 1110 	1340 1320 1388 1401	1300 	1000 1067	950 1070 1000	1150 1100 	 501
SOUND SPEEDS WI	Longițudinal speed,* m/sec	2710 2700 2650	2690 2750 2756	2650 2500 2600	2440 2440	2430 2520 2580	2400 2350 2360	1380 1340 1400
COMPARISON OF MEASURED SOUND SPEEDS WITH LITERATURE VALUES	Polymer	Polyhexamethylene adipamide	Polymethylmethacrylate	Polypropylene	Polyoxymethylene	Polyethylene	Polystyrene	Polytetrafluoroethylene

\*At room temperature and various ultrasonic frequencies

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Table 1

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I a. APPARATUS USED AT 0.6 MHz



Ib. APPARATUS USED AT 2.2 MHz

FIG. I BLOCK DIAGRAMS OF APPARATUS USED AT 0.6 MHz AND 2.2 MHz



FIG. 2 BLOCK DIAGRAM OF APPARATUS USED AT 5.5 MHz



3 d. 5.5 MHz OSCILLOSCOPE DISPLAY. COMPARISON PULSE ON LEFT, ULTRASONIC SIGNAL ON RIGHT. (TIME SCALE 10µsec/cm)



3 b. 2.2 MHz DISPLAY. (TIME SCALE 5µsec/cm)



3c. 0.6 MHz DISPLAY (TIME SCALE 20 µsec/cm)

FIG. 3 PHOTOGRAPHS OF TYPICAL ULTRASONIC SIGNALS



40. TYPICAL DEVICE WITH SPECIMEN ROTATED



4b. PRESENT DEVICE WITH TRANSDUCERS ROTATED

FIG. 4 SCHEMATIC DRAWINGS OF IMMERSION DEVICES





FIG. 5 PHOTOGRAPH OF IMMERSION DEVICE WITH TRANSDUCERS VERTICAL





FIG. 6 PHOTOGRAPH OF IMMERSION DEVICE WITH TRANSDUCERS ROTATED





# FIG. 8 GEOMETRY OF SHEAR WAVE PROPAGATION



FIG. 9 LONGITUDINAL SOUND SPEED VS TEMPERATURE FOR POLYMETHYLMETHACRYLATE

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FIG. 13 LONGITUDINAL SOUND SPEED VS TEMPERATURE FOR POLYPROPYLENE

and framework .....

#### APPENDIX A

#### DESCRIPTION OF MATERIALS USED

All of the materials used in this work will be described in this section. Trade names are given for identification purposes only.

Polyhexamethylene adipamide (nylon 6-6): a highly crystalline polymer. The material used here was made by the Dupont Co., Wilmington, Delaware, under the trade name Zytel 101 and received in the form of a rod. The measured density of the polymer was  $1.147 \text{ g/cm}^3$ .

<u>Polymethylmethacrylate</u>: this polymer has the structure  $[-CH_2C(CH_3)(COOCH_3)-J_n]$  and is (generally) amorphous. The glass transition temperature of polymethylmethacrylate is given<sup>22</sup> as 105°C. The material used here was made by the Rohm and Haas Co., Philadelphia, Pennsylvania, under the trade name Plexiglas II UVA, and received in the form of a cast block. The measured density of the polymer was 1.191 g/cm<sup>3</sup>.

<u>Polypropylene</u>: this polymer has the structure  $[-CH_2CH(CH_2)-]_n$ and is highly crystalline. The melting point of polypropylene is 176°C.<sup>23</sup> The material used here was made by the Avisun Corp., Marcus Hook, Pennsylvania, under the trade name Avisun 1010, and received in the form of an extruded sheet. The measured density of the polymer was 0.913 g/cm<sup>3</sup>.

Polyoxymethylene: this polymer has the structure  $[-CH_2O-]_n$ and is highly crystalline. The material used here was made by the Dupont Co., Wilmington, Delaware, under the trade name Delrin. The measured density of the polymer was 1.425 g/cm<sup>3</sup>.

Polyethylene: this polymer has the structure  $[-CH_2CH_2-j_n]$ and is highly crystalline. The melting point of polyethylene is taken<sup>23</sup> as 137°C though large single crystals may melt as high as 141°C. The material used here was made by the Allied Chemical Corp., Morristown, New Jersey, under the trade name FF-COl, and received in the form of an extruded block. The manufacturer gives a weight average molecular weight of 2 x 10<sup>5</sup> g/mole. The measured density of the polymer was 0.957 g/cm<sup>3</sup>.

<u>Polystyrene:</u> this polymer has the structure  $[-CH_2CH(C6H_5)-]_n$ and is amorphous. The material used here was made by the Dow Chemical Co., Midland, Michigan, under the trade name Styron 666. The measured density of the polymer was  $1.052 \text{ g/cm}^3$ .

Polytetrafluoroethylene: This polymer has the structure  $[-CF_2CF_2-J_n]$  and is highly crystalline. The material used here was made by the Dupont Co., Wilmington, Delaware, under the trade name Teflon. The measured density of the polymer was 2.177 g/cm<sup>3</sup>.