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ULTRASONIC MEASUREMENT OF ELASTIC
CONSTANTS AT TEMPERATURES FROM
20 TO 1100° C

M. W. Moyer
J. P. Hammond

September 1976

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OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

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**ULTRASONIC MEASUREMENT OF ELASTIC CONSTANTS
AT TEMPERATURES FROM 20 TO 1100° C**

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ABSTRACT

Techniques have been developed that are necessary to determine the elastic moduli of structural materials of interest to the Liquid Metal Fast Breeder Reactor and the High Temperature Gas Reactor. Measurements were made at temperatures from 20 to 1100° C. A detailed summary has been made of the sources of error and a calculation of the precision of the measurements is given.

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SUMMARY

Measurements of the elastic moduli at temperatures from 20 to 1100° C of a number of materials of interest in the Liquid Metal Fast Breeder Reactor and the High Temperature Gas Reactor were accomplished using acoustic techniques. A two-step process was required to complete the measurements. First, the acoustic velocities were measured accurately on bulk samples at room temperature, then a wire sample was used to make elevated-temperature measurements. A computer was used to calculate the moduli and plot the data. A detailed summary has been made of the sources of error and a calculation of the precision of the measurements is given.

INTRODUCTION

This report describes the techniques used at the Oak Ridge Y-12 Plant^(a) to measure the ultrasonic velocities necessary to determine moduli and Poisson's ratio. This project supports the Oak Ridge National Laboratory's effort to specify materials for certain reactor systems. Systems of current interest include the Liquid Metal Fast Breeder Reactor (LMFBR) and the High Temperature Gas Reactor (HTGR). It is important to characterize the mechanical properties of the materials which will be used in these reactor systems. One set of properties of interest is the elastic moduli as a function of temperature. The moduli of a material can be inferred from the sonic velocities.

Included in this document is a discussion of the experimental arrangement and procedure for calculating the moduli, a sample of the results obtained on representative materials, and an analysis of the errors-which permits an estimate on the accuracy of the moduli calculations.

(a) Operated by the Union Carbide Corporation's Nuclear Division for the US Energy Research and Development Administration.

ULTRASONIC MEASUREMENT OF ELASTIC CONSTANTS

MEASUREMENT OF VELOCITY

To determine the sonic velocity, room-temperature velocity measurements are made using conventional transducers on bulk samples and then measurements are made at elevated temperatures on thin rods. To make velocity measurements at temperatures to 1100° C, the sound must be transmitted through a buffer rod. A magnetostrictive transducer was used to generate sonic waves in a thin wire buffer rod and a sample rod. The magnetostrictive technique does not have as high an absolute accuracy as the room-temperature measurement since transducer limitations require that a lower frequency be used, but the technique can accurately measure small changes in velocity. Thus, the velocity measurements as a function of temperature are normalized to the room-temperature wire measurement and multiplied by the room-temperature bulk-sample measurement.

Room-Temperature Velocity Measurement on Bulk Samples

Measurement of the ultrasonic velocity, V , involves the measurement of length, X , and time, t , since $V = X/t$. In a pulse-echo measurement, the time must be measured for a round trip in a specimen of length, L , so:

$$V = 2L/t. \quad (1)$$

The sample length can be measured using gage blocks or a micrometer. Accuracy in the determination of the length (L) will depend upon how flat and parallel are the sample faces. Measurement of time must be made electronically and is subject to certain systematic errors for which there must be an accounting.

The technique used to measure the transit time is a double-pulse method which is similar to the pulse-echo overlap method described by Papadakis.⁽¹⁾ The double-pulse technique consists of an oscillator and gating circuitry which allows two successive oscillations to trigger the transducer. The frequency of the oscillator is adjusted to obtain superposition of the multiple reflections in the sample caused by the two excitation pulses. The transducer response adds the two wave trains to generate a combined waveform. A block diagram and the associated time traces are presented in Figure 1. The time traces show the signals at each point in the system. When the superposition of two successive multiple reflections is obtained, a counter enables the oscillator period and thus the transit time in the sample to be measured.

Analysis of Errors on Bulk-Sample Measurements

Since the measurement of ultrasonic velocity requires that both the sample length, L , and transit time, t , be measured, an estimate of each is required. Papadakis⁽²⁾ describes some of the errors involved in measuring the transit time in the sample: (1) the precision in overlapping the multiple echoes; (2) phase shift associated with bond thickness between the transducer and sample; (3) phase shift associated with diffraction; (4) counting errors, and (5) vagaries in the electronics. Two additional sources of error must be considered, namely: (6) the anisotropy of a sample, and (7) variations between samples of the same material.

Figure 1. DOUBLE-PULSE SYSTEM TO MEASURE SONIC VELOCITIES.

These seven sources of error will be considered one at a time.

1. An estimate of the ability to overlap echoes can be obtained by repeating each measurement several times. The average time, t , and a standard deviation, δt , can then be calculated.
2. Measurement of the phase shift associated with bond thickness is more difficult. McSkimin⁽³⁾ has shown that the combination of a transducer plus a couplant against one surface of a sample causes a phase shift of a signal reflected from the surface. (This behavior implies that the transducer plus couplant represents a complex acoustic impedance.) This phase shift will occur for each reflection. McSkimin presents a technique to measure the bond-thickness error which requires measurements to be made with a tuned system. Since the velocity measurements in this study were made on a wide-bandwidth system, a different technique was used to eliminate the bond-thickness error. Incidentally, this technique was also useful for eliminating delays in the timing system caused by the cabling and amplifiers.

The technique involves measuring the transit time in several different blocks of the material with different thicknesses and determining the best straight-line fit to the data.

The phase shift due to the coupling thickness is constant for all sample thicknesses. Thus, if the velocity fit is extrapolated to a zero sample thickness, the offset time can be determined. In practice, only two thicknesses were available for each material. The zero-thickness offset was determined by averaging the offset measured on 22 different materials. This average offset was then used as a transit-time data point for zero thickness in the calculation of velocity for each material. This technique provides several additional benefits. Since more than one sample is measured, the velocity is an average between samples. Thus, the variation measured accounts not only for the ability to repeat a measurement on one sample but also for the uniformity between samples. Also, all the electronic delays are constant and, thus, the zero-thickness offset contains the electronic delays.

3. The diffraction-phase shift error must be estimated. The phase of a plane-wave radiation from a piston source changes by $\pi/2$ radians between the surface of the piston and infinity. If the transit time in a sample is measured between two multiple echoes, a phase shift will occur since the reflecting interface causing the two echoes are at different distances from the transducer. This phase change has been calculated by a number of authors^(4,5) as a function of a normalized distance, S , where:

$$S = Z\lambda/a^2 = ZV/a^2f. \quad (2)$$

In Equation 2:

- Z represents the propagation distance,
- λ the wave length,
- a the transducer radius,
- V the sonic velocity, and
- f the transmitted frequency.

Most calculations for diffraction phase shifts were made for narrow-bandwidth transducers; however, Papadakis⁽⁶⁾ extended the calculations to cover broad-bandwidth pulses and provided plots of the diffraction phase shift as a function of S_C and transducer bandwidth (where S_C is the normalized bandwidth at the center frequency of the transmitted pulse).

Papadakis computed a weighted average of the diffraction phase shift as a function of the transducer bandwidth. Once the diffraction phase shift is determined as a function of the normalized distance, S , an increment of time, Δt , can be determined which must be added to the measured travel time to obtain the true travel time with which to compute the velocity, thus:

$$\Delta t = [\phi(S_2) - \phi(S_1)] / 2\pi f_C, \quad (3)$$

where:

$\phi(S_1)$ represents the diffraction phase shift in radians for the first multiple at a normalized distance, S_1 , and

$\phi(S_2)$ the diffraction phase shift for the second multiple at a normalized distance, S_2 .

Normally, the $\phi(S_i)$ s would be determined from one of the curves generated by Papadakis. The transducers used in this study had approximately an 80% bandwidth, so a least-squares polynomial fit was made to the curve generated by Papadakis to facilitate the calculation of $\phi(S_i)$.

4. The counting statistics on the audio-frequency oscillator which drives the double-pulse system generates an error. By using a period-averaging counter to count for 10^3 to 10^4 periods, a 0.1 ns measurement can be made.
5. Vagaries in the electronics include delays caused by cabling and time jitter caused by triggering the audio oscillator. The electronic delays are corrected at the same time as the coupling-thickness phase shift is measured, by measuring several different thickness samples. The jitter is averaged by using a period-averaging counter.
6. Another error is due to the anisotropy of the sample. To get an estimate of the anisotropy of the samples, shear-velocity measurements were made with polarization directions at 0 and 90 degrees. An average shear-velocity value was used to calculate the moduli. If the anisotropy is excessive, the model used to calculate the moduli from the measured velocities becomes invalid and must be modified. The solution to this problem is beyond the scope of this report.
7. Variations in velocity between different samples of the same material are averaged at the same time as the coupling phase shift is measured by measuring several different thickness samples.

It should be mentioned that the use of broad band pulses can generate additional errors, particularly when the measurements are made on attenuating materials. Attenuation is frequency dependent so that higher frequencies are attenuated more than lower frequencies. This difference causes a pulse-shape change, resulting in errors in the transit time measurement. The materials measured in this study had relatively low attenuation, thus keeping this error to a minimum.

Sonic-Velocity Measurements at Elevated Temperatures

Measurement of the sample transit time at temperatures to 1100° C presents a whole new set of problems. No suitable transducers are available which operate at these elevated temperatures, so the transducer must be coupled to the sample through a buffer rod. Difficulties arise for longitudinal velocity measurements because beam reflections at the buffer-rod surface cause a mode conversion which makes it difficult to measure the sample velocity. In addition, long buffer rods are required, causing attenuation problems, and coupling the sample to the buffer rod at high temperatures presents another problem (silver solder will melt).

If a thin wire is used in which the diameter is less than 0.1 the wavelength of sound, extentional and torsional waves can be generated using a magnetostrictive transducer. Extentional waves are similar to longitudinal waves except that they have a radial component of particle motion and have a lower velocity than longitudinal waves in bulk material.

Torsional waves are shear waves with a circumferential particle motion and have the same velocity as shear waves in bulk material. With the magnetostrictive system, pulse frequencies are limited to 100 to 200 kHz. For typical materials, the wave length would range from 25 to 50 mm, and the maximum wire diameter would be limited to a range of 2.5 to 5.0 mm. Thus, samples approximately 2.5 mm in diameter were used. To obtain sufficient length to make a reasonably precise measurement, these samples needed to be longer than those used for the bulk-sample measurements. A sample length of about 100 mm was found to be satisfactory.

The magnetostrictive transducer consists of a section of magnetostrictive wire (wire which undergoes strain when placed in a magnetic field) and an exciting coil. The magnetostrictive wire was coiled and drawn through a die to produce a helical structure. This structure enables both extentional and torsional waves to be generated simultaneously. These waves can be separated because of their different velocities. The helical structure of the magnetostrictive wire is annealed at a fairly low temperature so that it is necessary to weld a 304 SS buffer wire to the transducer wire. The samples are then welded to the 304 SS buffer wire and inserted into a furnace.

The double-pulse technique used on the bulk samples requires an operator adjustment to determine the transit time in the sample. Since many measurements were required on each of the wire samples to determine the velocity as a function of temperature, an automatic system was set up to make time measurements. Figure 2 is a block diagram of this system. The furnace has an automatic controller to heat and cool to the desired temperature at a constant heating rate.

A heating rate of 10°C/min was used for all elevated-temperature measurements. A Chromel/Alumel thermocouple was attached to the center of the sample to measure its temperature; an argon gas purge was used to reduce sample oxidation.

The ultrasonic excitation system consists of a transducer and a pulser. The time traces show the signals found at points throughout the system. Echoes from the sample are fed through a limiter to clip the excitation pulse, a preamplifier, and then to two parallel amplifiers. When two reflections are chosen to measure the time, these parallel amplifiers are used to adjust the amplitude of the two pulses to be equal. An inverter is included to make the two pulses of the same polarity. The amplifier outputs are supplied to two constant-fraction-timing, single-channel analyzers (CFTSCA). The CFTSCA provides a trigger pulse at a time which is a constant fraction of the peak pulse height. Thus, changes of the pulse amplitude do not cause the time location of the trigger pulse to change. Two gate generators are provided to set a gate on the pulses of interest. The signals from the gates and the CFTSCA are routed to coincidence circuits to provide timing signals which are sent to a time interval meter.

A timer was used to take readings of the time intervals and thermocouple voltage at 100-second intervals (equivalent to 17°C increments) and punch the data on paper tape for later analysis on a computer. Four runs were taken on each sample—two extentional runs and two torsional runs. The timing gates had to be moved between the extentional and torsional runs.

Figure 2. ELEVATED-TEMPERATURE SONIC-VELOCITY MEASUREMENT SYSTEM.

CALCULATION OF MODULI

Evaluation of Room-Temperature Data

Once transit times for the bulk samples were measured and corrected for the diffraction phase shift, the thickness was measured and the velocity was calculated using a special least-squares fit. The thickness of each sample was measured ten times with a micrometer, and the average thickness and standard deviation were calculated. Precision-ground gage blocks were also measured to determine the micrometer accuracy. The transit time was measured a number of times so that the average and standard deviation could be calculated. Thus, for each material at least three data sets (two thicknesses plus the zero thickness offset time) were available which contained an independent measurement of thickness and time with an estimate of the repeatability of each measurement. An adaptation of a technique described by Graybill⁽⁷⁾ which is used to calculate the slope and offset of a least-squares straight-line fit to data of the form $x_i, \delta x_i, y_i, \delta y_i$ and to obtain an estimate of the standard deviation of the slope and offset is given in the Appendix. A computer program using this technique was written to calculate the room-temperature velocities for each sample.

Following are listed the equations necessary to calculate all the moduli and Poisson's ratios for a bulk sample knowing the longitudinal and shear velocities, and the density:

$$\sigma = \frac{1 - 2(V_S/V_L)^2}{2 - 2(V_S/V_L)^2} \quad (4)$$

$$V_E = V_L \sqrt{\frac{(1 + \sigma)(1 - 2\sigma)}{(1 - \sigma)}} \quad (5)$$

$$Y = V_L^2 \rho \frac{(1 + \sigma)(1 - 2\sigma)}{(1 - \sigma)} \quad (6)$$

$$S = V_S^2 \rho, \text{ and} \quad (7)$$

$$K = \frac{Y}{3(1 - 2\sigma)} \quad (8)$$

Also, it is necessary to calculate an estimate of the standard deviation of each variable. An estimate of the standard deviation of a function of more than one variable can be obtained by taking the partial derivative with respect to each variable as follows:

$$\delta F(a,b) = \left[\left(\frac{\partial(F)}{\partial a} \right)^2 (\delta a)^2 + \left(\frac{\partial(F)}{\partial b} \right)^2 (\delta b)^2 \right]^{1/2} \quad (9)$$

Using Equation 9, an estimate of the standard deviations of the moduli and Poisson's ratio can be made by taking the partial derivatives of Equations 4 through 8 and evaluating them according to Equation 9. Doing this calculation, other equations can be derived:

$$\delta\sigma = \left[\frac{V_L^2 V_S^4 (\delta V_L)^2 + V_S^2 V_L^4 (\delta V_S)^2}{(V_L^2 - V_S^2)^4} \right]^{1/2} \quad (10)$$

$$\delta V_E = \left[\frac{(1 + \sigma)(1 - 2\sigma)(\delta V_L)^2}{(1 - \sigma)} + \frac{V_L^2 \sigma^2 (\sigma - 2)^2 (\delta \sigma)^2}{(1 - \sigma)^3 (1 + \sigma)(1 - 2\sigma)} \right]^{1/2} \quad (11)$$

$$\delta Y = \left[\frac{4V_L^2 \rho^2 (1 + \sigma)^2 (1 - 2\sigma)^2 (\delta V_L)^2}{(1 - \sigma)^2} + \frac{V_L^4 (1 + \sigma)^2 (1 - 2\sigma)^2 (\delta \rho)^2}{(1 - \sigma)^2} + \frac{4\sigma^2 (\sigma - 2)^2 V_L^4 \rho^2 (\delta \sigma)^2}{(1 - \sigma)^4} \right]^{1/2} \quad (12)$$

$$\delta S = \left[4V_S^2 \rho^2 (\delta V_S)^2 + V_S^4 (\delta \rho)^2 \right]^{1/2} \text{, and} \quad (13)$$

$$\delta K = \left[\frac{(\delta Y)^2}{9(1 - 2\sigma)^2} + \frac{4Y^2 (\delta \sigma)^2}{9(1 - 2\sigma)^4} \right]^{1/2} \quad (14)$$

where:

- F is a function of a and b,
- δF represents the standard deviation of F,
- δa the standard deviation of a,
- δb the standard deviation of b,
- V_L the longitudinal velocity,
- δV_L the standard deviation of V_L ,
- V_S the shear velocity,
- δV_S the standard deviation of V_S ,
- V_E the extentional velocity,
- δV_E the standard deviation of V_E ,
- σ Poisson's ratio,
- $\delta \sigma$ the standard deviation of σ ,
- Y Young's modulus,
- δY the standard deviation of Y,
- S the shear modulus,
- δS the standard deviation of S,

- K the bulk modulus,
 δK the standard deviation of K ,
 ρ the sample density, and
 $\delta \rho$ the standard deviation of ρ .

For several materials, wire samples were received without bulk samples. Thus, room-temperature velocity measurements were required on the wire samples. To make good contact with the sensor wire, the sample needs to be welded to the sensor, which presents a problem in knowing the length of the sample with sufficient accuracy. In addition, the double-pulse system used for the bulk samples did not have sufficient delay to measure the wire samples. Thus, another double-pulse system had to be used in which the time interval between two trigger pulses was measured. This system introduced additional electronic delays which could produce an offset. To overcome these problems, a procedure was developed in which a reflector was clamped to the wire, and the transit time from the weld to the reflector was measured. The distance from the end of the wire to the reflector was then measured, and the reflector moved toward the weld. This process was repeated four or five times. Thus, data were obtained for the transit time versus the reflector position. The velocity could then be determined from the slope of a straight-line fit to the data using the least-squares technique described in the Appendix.

The equations used to calculate the moduli and Poisson's ratio from wire data are different from the equations used for bulk samples since the extentional velocity, V_E , and torsional velocity, V_S , are measured (instead of longitudinal and shear). These equations are:

$$Y = V_E^2 \rho, \quad (15)$$

$$S = V_S^2 \rho, \quad (16)$$

$$\sigma = \frac{Y}{2S} - 1, \quad (17)$$

$$K = \frac{Y}{3(1 - 2\sigma)}, \quad (18)$$

$$V_L = \frac{V_E}{\sqrt{\frac{(1 + \sigma)(1 - 2\sigma)}{(1 - \sigma)}}}, \quad (19)$$

$$\delta Y = \left[4V_E^2 \rho^2 (\delta V_E)^2 + V_E^4 (\delta \rho)^2 \right]^{1/2}, \quad (20)$$

$$\delta S = \left[4V_S^2 \rho^2 (\delta V_S)^2 + V_S^4 (\delta \rho)^2 \right]^{1/2}, \quad (21)$$

$$\delta \sigma = \left[\frac{(\delta Y)^2}{4S^2} + \frac{Y^2 (\delta S)^2}{4S^2} \right]^{1/2}, \quad (22)$$

$$\delta K = \left[\frac{(\delta Y)^2}{9(1-2\sigma)^2} + \frac{4Y^2(\delta\sigma)^2}{9(1-2\sigma)^4} \right]^{1/2}, \text{ and} \quad (23)$$

$$\delta V_L = \left[\frac{(1-\sigma)(\delta V_E)^2}{(1+\sigma)(1-2\sigma)} + \frac{V_E^2 \sigma^2 (\sigma-2)^2 (\delta\sigma)^2}{(1-\sigma)(1+\sigma)^3 (1-2\sigma)} \right]^{1/2} \quad (24)$$

Evaluation of Elevated-Temperature Data

Once the room-temperature data were obtained for the samples, elevated-temperature data were taken. A normalizing technique was used to limit the number of parameters which needed to be measured.

If a sample of length, L , is heated, the length changes as a result of thermal expansion. If the temperature changes from some initial temperature to some other temperature, T , then the new length, L' , is given by:

$$L' = L(1 + \alpha\Delta T), \quad (25)$$

where $\Delta T = T - T_0$.

Then, at temperature, T , the velocity is given by:

$$V = 2L'/t = \frac{2L(1 + \alpha\Delta T)}{t}, \quad (26)$$

where t represents the two-way transit time in the sample.

A normalized velocity can then be calculated by:

$$N_V = \frac{V}{V_0} = \frac{\frac{2L(1 + \alpha\Delta T)}{t}}{\frac{2L}{t_0}} = \frac{t_0}{t} (1 + \alpha\Delta T), \quad (27)$$

where:

N_V represents the normalized velocity, and
 t_0 the two-way transit time in the sample at T_0 .

It can be seen that the length of the sample is not required. In a like manner, the Young's and shear moduli can be calculated. Repeating Equation 15:

$$Y = V_E^2 \rho, \quad (15)$$

then, at some temperature, T

$$\rho \cong \rho_0 / (1 + 3\alpha\Delta T), \quad (28)$$

where:

ρ_0 represents the density at T_0 , and
 ρ density at T .

Thus, a normalized modulus can be calculated from:

$$N_Y = \frac{Y}{Y_0} = \frac{V_E^2 \rho}{V_{E_0}^2 \rho_0} = N_{V_E}^2 / (1 + 3 \alpha \Delta T), \quad (29)$$

where:

N_Y represents the normalized Young's modulus, and
 Y_0 the Young's modulus at T_0 .

Likewise, the normalized shear modulus is given by:

$$N_S = \frac{S}{S_0} = N_{V_S}^2 / (1 + 3 \alpha \Delta T), \quad (30)$$

where:

N_S represents the normalized shear modulus, and
 S_0 the shear modulus at T_0 .

The actual velocities and moduli can be calculated by multiplying the normalized values by the room-temperature values. This fact is the reason accurate room-temperature values of sonic velocity are required.

Computer Calculations of High-Temperature Moduli

Four sets of data are taken on each wire sample—two extentional runs and two torsional runs. Each run can have as many as 75 sets of data (transit time and thermocouple voltage). Figure 3 is a block diagram of the computer program. A two-program package was written to do the data analysis and data plotting on a PDP-15/20 and a PDP-15/35 computer. The first program reads the data tapes from the four runs and calculates the normalized velocities correcting for the coefficient of thermal expansion (Equation 25) and temperature. These data are plotted to check for errors and written in data files on magnetic tape in a standard format. The second program combines the two extensional runs and the two torsional runs. The room-temperature velocities and moduli are supplied, and the program calculates the actual velocities and moduli as a function of temperature.

A least-squares program fits an equation of the form:

$$M = M_0 \left[1 + A(T - T_0) + B(T - T_0)^2 + C(T - T_0)^3 \right] \quad (31)$$

to both the Young's and shear moduli data. An estimate of the standard deviation of the fit is also made. These equations are required to calculate the Young's and shear moduli at equal temperature increments to use in the calculation of the bulk modulus and Poisson's

ratio since the extentional and torsional velocities were not measured at the same temperatures. Once Poisson's ratio is calculated, a least-squares routine is used to fit an equation of the same form as Equation 31 to Poisson's ratio. Finally, a table of moduli versus temperature is generated; and all the velocity, moduli, and Poisson's ratio data are plotted along with the fitted curves.

RESULTS AND CONCLUSIONS

Table 1 is a list of the samples tested in this program using the system described in this report. Compilation of all the data is the subject of another report.⁽⁸⁾ In this report, some typical results will be shown and some observations made.

Typical results from Sample 9 MA (a 2.25 CR-1 Mo steel) are listed in Tables 2 through 4 and outlined in Figures 4 through 10. Table 2 gives the calculation of the room-temperature velocities and moduli. The velocities were calculated using the least-squares fit routine described

earlier and in the Appendix. Shear-velocity measurements were taken twice with the polarization direction rotated 90 degrees between measurements. This measurement is taken to obtain an estimate of the anisotropy of the sample. The standard deviation of the average shear velocity is calculated from:

$$\delta V_S = \left[\delta V_{S0}^2 + \delta V_{S90}^2 \right]^{1/2} \quad (32)$$

the anisotropy is calculated from:

$$A = \left[\frac{V_{S0} - V_{S90}}{2} \right] \quad (33)$$

As mentioned earlier, a measure of the anisotropy of a sample is important. The equations used (Equations 5 - 9 and 15 - 19) assume a homogeneous isotropic material. Calculations of the moduli of a sample may be invalid if the sample has excessive anisotropy. The moduli were calculated using the average shear velocity.

It should be pointed out that the variations given are standard deviations on the repeatability of the measurements; ie, precision. Since no velocity standards are available it is difficult to know how accurate these measurements are. As described earlier, an attempt has been made to eliminate all identifiable errors.

Figure 3. COMPUTER PROGRAM TO CALCULATE ELEVATED-TEMPERATURE SONIC MODULI.

Table 1
SPECIMENS FOR SONIC DYNAMIC PROPERTIES

Code	Alloy	Material		Form	Test Range		Orientation of the Specimen Axis ⁽¹⁾	Specimen Condition ⁽¹⁾		Remarks
		Dimension			Room Temperature	To:		Block for Room Temperature	Wire for Elevated Temperature	
		(in)	(mm)		(° F)					
9	2.25 Cr-1 Mo	1	25	Plate	1200	649	Parallel to the RD	MA	MA	
9	2.25 Cr-1 Mo	1	25	Plate	1200	649	Parallel to the RD	AT	AT	
9	2.25 Cr-1 Mo	1	25	Plate	995	538	Parallel to the RD	CT	CT	
9	2.25 Cr-1 Mo	1	25	Plate	-	-	In Plane Perpendicular to the RD	Annealed ⁽²⁾		
9	2.25 Cr-1 Mo	1	25	Plate	-	-	Perpendicular to the RD	Annealed		
17	9 Cr-1 Mo	0.5	13	Plate	1200	649	Parallel to the RD	MA	MA	
15	Incoloy 800H	1	25	Plate	1650	899	Parallel to the RD	SA	SA	
1	Type 304 SS	1	25	Plate	1600	871	Parallel to the RD	SA	SA	
1	Type 304 SS	1	25	Plate	1000	538	Parallel to the RD	CT	CT	
1	Type 304 SS	1	25	Plate	1600	871	Parallel to the RD	AT	AT	
2	Type 304 SS	1	25	Plate	-	-	In Plane, 45° to the RD	SA	-	
3	Type 304 SS	1	25	Plate	-	-	In Plane, Perpendicular to the RD	SA	-	
4	Type 304 SS	1	25	Plate	-	-	Perpendicular to the Plane	SA	-	
5	Type 304 SS	2.75	70	Plate	-	-	Parallel to the RD	SA MA	-	
6	Type 304 SS	2	51	Plate	1600	871	Parallel to the RD	SA MA	SA	
7	Type 304 SS	2.5	64	Plate	-	-	Parallel to the RD	SA MA	-	
8	Type 304 SS	3	76	Bar	1600	871	Parallel to the RD	SA MA	SA	
10	Type 308 SS	-	-	FFTF Weld	1600	871	Parallel to the WD	AW	AW	Room-temperature data invalid due to anisotropy.
13	Type 316 SS	1	25	Plate	1600	871	Parallel to the RD	SA	SA	
16	Hastelloy X	0.75	19	Rod	2000	1093	Parallel to the RD	SA	SA	
19	Hastelloy N	1	25	Plate	2000	1093	Parallel to the RD	SA	SA	
11	Inconel 718	0.75	19	Plate	1000	538	Parallel to the RD	AH MA	AH	
12	Inconel 718	-	-	Weld Deposit	1000	538	Parallel to the WD	-	AH ⁽³⁾	
18	Inconel 617	1	25	Plate	1800	982	Parallel to the RD	SA	SA	
21	Inconel 82	-	-	Weld Deposit	1300	704	Parallel to the WD	-	SR ⁽³⁾	
21	Inconel 82	-	-	Wrought	1300	704	Parallel to the RD	-	SA ⁽³⁾	Room-temperature data used with 21SR.
22	Type 16-8-2	-	-	Weld Deposit	1300	704	Parallel to the WD	-	SA ⁽³⁾	
20	Pt-30 Rh-0 W	-	-	Sheet	1975	1079	Parallel to the RD	-	SA ⁽³⁾	

(1) The following notations are used:

AH = Age Hardened
 AT = Treated to Give Recrystallized Fiber Texture
 AW = As Welded
 CT = Treated to Give Cold-Worked Fiber Texture
 MA = Mill Annealed
 RD = Rolling Direction
 SA = Solution Annealed
 SR = Stress Relieved
 WD = Welding Direction

(2) No elevated-temperature data.

(3) Room-temperature properties taken on wire samples.

Table 2

**ROOM-TEMPERATURE ULTRASONIC VELOCITY
AND MODULUS MEASUREMENTS**

Sample - 9MA, 2.25 Cr-1 Mo, Heat Treat - 20017, II RD, Mill Annealed
Sample Configuration - Bulk

Ultrasonic Velocity Calculations, 9MA				
Longitudinal Velocity	=	5929.747	±	0.822 m/sec
0-Degree Shear Velocity	=	3267.996	±	1.318 m/sec
90-Degree Shear Velocity	=	3268.141	±	0.574 m/sec
Average Shear Velocity	=	3268.068	±	1.438 m/sec
Anisotropy of Shear Velocity	=	0.073		

Ultrasonic Moduli Calculations, 9MA				
Longitudinal Velocity	=	5929.747	±	0.822 m/sec
		233454.61	±	32.36 in/sec
Shear Velocity	=	3268.068	±	1.438 m/sec
		128664.09	±	56.61 in/sec
Extentional Velocity	=	5232.731	±	1.963 m/sec
		206013.02	±	77.28 in/sec
Density	=	7.83320	±	0.00050 g/cm ³
Poisson's Ratio	=	0.281872	±	0.000289
Young's Modulus	=	214.4845	±	0.1615 GPa
		0.3110832E+08	±	0.2342410E+05 psi
Shear Modulus	=	83.6607	±	0.0738 GPa
		0.1213394E+08	±	0.1070629E+05 psi
Bulk Modulus	=	163.8826	±	0.2498 GPa
		0.2376914E+08	±	0.3622838E+05 psi

Coefficient of Thermal Expansion = $14.18 \times 10^{-6}/^{\circ}\text{C}$.

Table 3

ELEVATED-TEMPERATURE ULTRASONIC MODULUS MEASUREMENTS

Sample - 9MA, 2.25 Cr-1 Mo, Heat Treat - 20017, II RD, Mill Annealed
Temperature Measurement Range - 24 - 663° C

Least Square Fit:

$$X = X_0(1 + A_1\Delta T + A_2\Delta T^2 + A_3\Delta T^3),$$

$$\text{or } y = A_1x + A_2x^2 + A_3x^3 \quad \text{where: } y = (X/X_0) - 1 \quad \text{and } x = \Delta T$$

	X_0	A_1	A_2	A_3	Standard Deviation of Fit
Young's Modulus	3.110836E-7	-3.05032E-4	-6.22360E-9	-1.71081E-10	6.87155E-4 psi
Shear Modulus	1.213396E-7	-2.09753E-4	-4.53940E-7	2.72752E-10	9.35099E-4 psi
Poisson's Ratio	0.281872	-4.66649E-4	2.19339E-6	-2.13668E-9	2.79229E-4

Table 3 contains the coefficients of the least-squares fit to the elevated-temperature data for Sample 9 MA, a 2.25 Cr-1 Mo steel sample. The elevated-temperature data were fitted to an equation of the form:

Table 4
MODULI AND POISSON'S RATIO AT ELEVATED TEMPERATURES

Sample - 9MA, 2.25 Cr-1 Mo, Heat Treat - 20017, II RD, Mill Annealed

Temperature (° C)	Temperature (° F)	Young's Modulus (X 10 ⁶ psi)	Shear Modulus (X 10 ⁶ psi)	Bulk Modulus (X 10 ⁶ psi)	Poisson's Ratio
24	75	31.12	12.14	23.79	0.282
38	100	30.99	12.10	23.52	0.280
52	125	30.86	12.06	23.27	0.279
66	150	30.72	12.02	23.04	0.278
79	175	30.59	11.98	22.84	0.277
93	200	30.46	11.94	22.65	0.276
107	225	30.32	11.89	22.49	0.275
121	250	30.19	11.84	22.34	0.275
135	275	30.06	11.79	22.21	0.274
149	300	29.92	11.74	22.09	0.274
163	325	29.78	11.69	21.98	0.274
177	350	29.65	11.63	21.89	0.274
191	375	29.51	11.58	21.81	0.274
204	400	29.37	11.52	21.73	0.275
218	425	29.23	11.46	21.67	0.275
232	450	29.09	11.40	21.62	0.276
246	475	28.94	11.34	21.58	0.276
260	500	28.80	11.27	21.54	0.277
274	525	28.65	11.21	21.51	0.278
288	550	28.50	11.14	21.48	0.279
302	575	28.36	11.08	21.46	0.280
316	600	28.20	11.01	21.44	0.281
329	625	28.05	10.94	21.43	0.282
343	650	27.90	10.87	21.42	0.283
357	675	27.74	10.80	21.41	0.284
371	700	27.58	10.73	21.39	0.285
385	725	27.42	10.66	21.38	0.286
399	750	27.26	10.59	21.37	0.287
413	775	27.09	10.51	21.35	0.289
427	800	26.92	10.44	21.33	0.290
441	825	26.75	10.36	21.30	0.291
454	850	26.58	10.29	21.26	0.292
468	875	26.40	10.21	21.22	0.293
482	900	26.22	10.14	21.17	0.294
496	925	26.04	10.06	21.10	0.294
510	950	25.85	9.98	21.03	0.295
524	975	25.67	9.90	20.93	0.296
538	1000	25.47	9.83	20.83	0.296
552	1025	25.28	9.75	20.70	0.296
566	1050	25.08	9.67	20.56	0.297
579	1075	24.88	9.59	20.40	0.297
593	1100	24.68	9.52	20.22	0.297
607	1125	24.47	9.44	20.01	0.296
621	1150	24.26	9.36	19.70	0.296
635	1175	24.04	9.28	19.53	0.295
649	1200	23.82	9.21	19.25	0.294
663	1225	23.60	9.13	18.95	0.292

$$x = x_0(1 + A_1\Delta T + A_2\Delta T^2 + A_3\Delta T^3) \text{ , or} \quad (34)$$

$$y = A_1x + A_2x^2 + A_3x^3 \text{ ,} \quad (35)$$

where:

$$y = \left(\frac{x}{x_0} \right) - 1, \text{ and}$$

$$x = \Delta T.$$

The standard deviation of the fit was then calculated from the equation:

$$\sigma = \left\{ \frac{\sum_{i=1}^n [(A_1 x_i + A_2 x_i^2 + A_3 x_i^3) - y_i]^2}{n - 1} \right\}^{1/2} \quad (36)$$

where:

$$y_i = \left(\frac{x_i}{x_0} \right) - 1.$$

The fitted equations were then used to calculate the data reported in Table 4.

Finally, the data and calculated curves were plotted, as seen in Figures 4 through 10. One of the samples (Sample 1CT-75, a 2.25 Cr-1 Mo steel with 70% cold work) was mistakenly tested above the annealing temperature. The normalized extentional velocity data are shown in Figure 11 for two thermal cycles. The first run shows the effect of annealing on the velocity after the sample reached approximately 550° C. (The offset between the start and finish of the test is indicative of the modulus change due to annealing.) The second run then shows the results for an annealed sample.

Figure 4. NORMALIZED EXTENSIONAL VELOCITY OF SAMPLE 9 MA.

Figure 5. NORMALIZED TORSIONAL VELOCITY OF SAMPLE 9 MA.

Figure 6. EXTENSIONAL VELOCITY OF SAMPLE 9 MA.

Figure 7. TORSIONAL VELOCITY OF SAMPLE 9 MA.

Figure 8. YOUNG'S MODULUS OF SAMPLE 9 MA.

Figure 9. SHEAR MODULUS OF SAMPLE 9 MA.

Figure 10. POISSON'S RATIO OF SAMPLE 9 MA.

Figure 11. NORMALIZED EXTENSIONAL VELOCITY OF SAMPLE 1 CT-75.

APPENDIX

TECHNIQUE TO DETERMINE A LEAST-SQUARES FIT

Herein, a technique is presented that can determine a linear least-squares fit to data in which both x and y are subject to error. A fit of the form:

$$y = \alpha + \beta x \quad (A-1)$$

is desired as well as an estimate of the standard deviation of both α and β .

A method is outlined by Graybill⁽⁷⁾ to obtain an estimate of σ_e^2 , α , and β for the functional relationship of Equation A-1. The technique requires that λ be known, where λ is defined as a ratio:

$$\lambda = \frac{\sigma_x^2}{\sigma_y^2} \quad (A-2)$$

An estimate of σ_x^2 is obtained from the equation:

$$\sigma_x^2 = \frac{[(n_i - 1)\sigma_{x_i}^2]}{n_i - N} \quad (A-3)$$

where:

n_i represents the number of samples used to determine σ_{x_i} , and
 N the number of data points used for the least-squares calculation.

Likewise:

$$\sigma_y^2 = \frac{[(n_i - 1)\sigma_{y_i}^2]}{n_i - N} \quad (A-4)$$

The maximum-likelihood estimates are given by:

$$\hat{\beta} = \pm (U^2 + \frac{1}{\lambda})^{1/2} + U \quad (A-5)$$

where:

$$U = \frac{\Sigma(y - \bar{y})^2 - \frac{1}{\lambda} \Sigma(x - \bar{x})^2}{2 \Sigma(y - \bar{y}) \Sigma(x - \bar{x})} \quad (A-6)$$

Also:

$$\hat{\alpha} = \bar{y} - \beta \bar{x} \quad , \text{ and} \quad (A-7)$$

$$\sigma_e^2 = \frac{1}{N-2} [\sum (y_i - \bar{y})^2 - \hat{\beta} \sum (y_i - \bar{y})(x_i - \bar{x})] \quad (A-8)$$

Using the following identities, these equations can be simplified for calculations:

$$\text{let } S = N \sum x_i y_i - \sum x_i \sum y_i = N [\sum (x_i - \bar{x})(y_i - \bar{y})] \quad (A-9)$$

$$C = N \sum y_i^2 - (\sum y_i)^2 = N \sum (y_i - \bar{y})^2 \quad , \text{ and} \quad (A-10)$$

$$D = N \sum x_i^2 - (\sum x_i)^2 = N \sum (x_i - \bar{x})^2 \quad (A-11)$$

where:

$$\bar{y} = \frac{\sum y_i}{N} \quad , \text{ and} \quad (A-12)$$

$$\bar{x} = \frac{\sum x_i}{N} \quad (A-13)$$

Then, simplifying Equations A-6 through A-8 gives:

$$U = \frac{C\lambda - D}{2\lambda S} \quad (A-14)$$

$$\hat{\alpha} = \frac{\sum y_i}{N} - \hat{\beta} \frac{\sum x_i}{N} \quad , \text{ and} \quad (A-15)$$

$$\sigma_e^2 = \frac{1}{N(N-2)} [C - \hat{\beta} S] \quad (A-16)$$

where $\hat{\beta}$ is determined by Equation A-5. The sign is chosen by the sign of S. Thus, if S is positive, $\hat{\beta} = U + \sqrt{U^2 + 1/\lambda}$; and, if S is negative, $\hat{\beta} = U - \sqrt{U^2 + 1/\lambda}$.

John Mandel⁽⁹⁾ then presents a method for calculating approximate estimates of $\hat{\sigma}_{\hat{\beta}}$ (standard deviation of the slope), and $\hat{\sigma}_{\hat{\alpha}}$ (standard deviation of the intercept). Assuming that errors in y are uncorrelated with errors in x, the estimates given by Mandel reduce to:

$$\hat{\sigma}_{\hat{\beta}} = T \hat{\sigma}_e \quad , \text{ and} \quad (A-17)$$

$$\hat{\sigma}_{\hat{\alpha}} = \sigma_e \sqrt{1/N + (T \cdot \bar{x})^2} \quad (A-18)$$

where:

$$T = \frac{(1 + \lambda \hat{\beta}^2)}{\left\{ \sum (x_i - \bar{x})^2 [1 + 2\lambda \hat{\beta}^2 + \sum (x_i - \bar{x})^2 \sum (y_i - \bar{y})^2 \cdot \lambda^2 \hat{\beta}^4] \right\}^{1/2}} \quad , \text{ and} \quad (A-19)$$

σ_e^2 is given by Equation A-16. Using Equations A-9 through A-13, T can be expressed as:

$$T = \frac{(1 + \lambda \hat{\beta}^2)}{D/N[1 + 2\lambda \hat{\beta}^2 + D \cdot C \lambda^2 \hat{\beta}^4/S]} \quad (A-20)$$

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