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A FACILITY FOR STUDYING ELASTIC MODULI
AS A FUNCTION OF TEMPERATURE
BY AN ULTRASONIC METHOD

Measurements on Vanadium-20 w/o Titanium

by

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Metallurgy Division

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ABSTRACT

A facility used to determine ultrasound velocities in materials at temperatures up to around 1000°C is described. It is shown how to compute the elastic constants of a material from these velocities. Results of measurements made upon V-20w/oTi are given.

I. THE MEASUREMENT FACILITY

A. Introduction

The elastic moduli, e.g., Young's modulus, Poisson's ratio, and shear modulus, of materials that are used in components of high-temperature nuclear reactors must be known so that designers of these reactors may allow for length changes, stress distribution changes, and other physical effects of mechanical and thermal stresses. A facility to study the behavior of moduli as a function of temperature has been assembled in the Metallurgy Division of ANL. An ultrasonic, pulse-echo technique is used to determine the velocity of sound waves propagating in a heated region of rod-shaped specimens. A vacuum furnace is used to heat the specimens.

B. Propagation of Sound in Rods

The velocities of sound waves in any medium are directly related to the elastic properties of the medium^{1,2} since both parameters ultimately depend upon the interatomic forces in the medium. The relations between them are

$$V_{\ell} = \left[\frac{E}{\rho} \frac{1 - \sigma}{(1 + \sigma)(1 - 2\sigma)} \right]^{1/2}, \quad (1)$$

$$V_s = \left[\frac{\mu}{\rho} \right]^{1/2}, \quad (2)$$

and

$$\frac{V_\ell}{V_s} = \left[\frac{1 - \sigma}{\frac{1}{2} - \sigma} \right]^{1/2}, \quad (3)$$

where

V_ℓ = velocity of longitudinal waves in the medium,

V_s = velocity of shear waves in the medium,

E = Young's modulus of the medium,

μ = shear (rigidity) modulus of the medium,

σ = Poisson's ratio in the medium,

and

ρ = density of the medium.

These equations apply strictly only to isotropic, unbounded media; a polycrystalline sample that is fine-grained and has no preferred orientation or texture is such an isotropic medium. However, a solid bar whose length is much longer than its diameter obviously cannot be considered to be an unbounded medium, and care must be taken to determine under what conditions the above equations apply.^{3,4} It is known that the above equations hold for rods when short-duration ultrasonic pulses in the megacycle-frequency range are used; that is, the velocity measured in a rod of a given material is the same, to within experimental error, as the velocity measured in a large piece with the lateral dimensions much larger than the dimension in the direction of propagation.^{5,6,7} The fact that the rod is laterally bounded does alter the propagation of sound; as a longitudinal sound-wave pulse travels axially along the bar, part of its energy is continuously changed into shear-wave energy. These shear waves not only progress axially along the rod, they have a radial velocity component as well. They eventually reach the opposite side of the rod, and part of their energy is reflected while the rest of the shear-wave energy is converted back to longitudinal waves that again move axially down the rod. Each time a pulse of shear-wave energy strikes the surface of the rod, a longitudinal pulse is generated that generates more shear waves, etc. The result of this is that one longitudinal wave pulse sent into a bar gives rise to many longitudinal pulses and many shear pulses. This process is described elsewhere;^{8,9} it is found that the longitudinal wave pulses, called "trailing pulses," will be separated from each other by a time Δt given by

$$\Delta t = D(V_s^{-2} - V_l^{-2})^{1/2}, \quad (4)$$

where D is the diameter of the rod.

It is also known that threading the bar will greatly reduce the generation of trailing pulses.¹⁰ We have found that flat-bottomed threads distort the shapes and reduce the amplitudes of these pulses, and that threads with sharp bottoms reduce their sizes to the point where the trailing pulses are very small in comparison with pulses that might be produced by reflection from an end face of a bar. It has also been determined that the tops of the threads do not have to be sharp, as long as the threads are deep enough and sharp-bottomed. In no case have we found that the trailing pulses disappeared entirely.

C. Description of the Method

A method similar to the techniques described by Levitt and Martin¹¹ and by Dunegan¹² is used. The specimen configuration is shown in Fig. 1. The rod is about 2.5 cm in diameter, except for one end, which is about 2 cm in diameter. Its length is approximately 25 cm. The length of the section with the small diameter may vary from 2 to 3.8 cm. The end faces and the surface of the shoulder are made as nearly parallel as possible; the measured length between the shoulder and the end face of a given rod typically varies about 0.001 cm. The end faces and the shoulder are specified to be flat to within one fringe of sodium light as viewed against an optically flat glass plate. The flat surfaces are specified to have a 20- μ cm polish on them. An ultrasonic transducer is coupled to the large-diameter end of the bar, and this device generates sound-wave pulses that travel

AREAS A, B, AND C DENOTE UNTHREADED REGIONS OF THE SPECIMEN SURFACE. AREAS A AND B ARE THE LOCATIONS OF THE WATER JACKET O-RING SEALS; AREA C IS THE LOCATION OF THE VACUUM SEAL TO THE TOP COVER OF THE FURNACE.

Fig. 1. Typical Specimen Configuration

down the length of the bar. As a pulse reaches the shoulder, where the diameter of the bar changes, part of the sound energy is reflected back to the transducer. The rest of the pulse travels until it reaches the end of the rod, where it also is reflected back to the transducer. When the two pulses reach the transducer after they have been reflected, they can be detected by the transducer, and the time of arrival of each pulse can be measured. The difference in arrival times is the time required for a sound wave to make a round trip in the small-diameter section of the rod. The longitudinal- and shear-wave velocities may be determined by measuring the transit times of waves generated by two separate transducers, each generating its own characteristic type of wave. However, it is not necessary to use two transducers if it is possible to take advantage of the waveguide nature of the rod. Trailing pulses will be generated in the small-diameter section of the rod if longitudinal waves are initially generated at the other end of the rod. Since the delay time between successive trailing pulses is a function of the shear-wave velocity, measurements of both delay time and round-trip transit time in the small end of the rod enable both the longitudinal-wave velocity and the shear-wave velocity to be computed. Trailing pulses will be generated in the large-diameter part of the rod too, but these can be eliminated, for all practical purposes, by threading as much as possible of the large-diameter section.

The working equations for computation of velocities and moduli are then as follows:

$$V_l(T) = 2 \frac{L(T)}{2\tau(T)}, \quad (5)$$

and

$$V_s(T) = \left\{ \left[\frac{\Delta t(T)}{D(T)} \right]^2 + \frac{[2\tau(T)]^2}{4L^2(T)} \right\}^{-1/2}, \quad (6)$$

where

$L(T)$ = length of small-diameter section at temperature T ,

$2\tau(T)$ = round-trip transit time in small diameter section at T ,

and

$D(T)$ = diameter of small-diameter section at T .

Also,

$$\sigma(T) = \frac{\left(\frac{1}{2}\right) \left[\frac{V_{\ell}(T)}{V_s(T)} \right]^2 - 1}{\left[\frac{V_{\ell}(T)}{V_s(T)} \right]^2 - 1}, \quad (7)$$

$$\mu(T) = \rho(T) V_s^2(T), \quad (8)$$

and

$$E(T) = 2\mu(T)[1 + \sigma(T)]. \quad (9)$$

Let

$$L(T) = L_o \left[1 + \frac{\Delta L}{L_o}(T) \right], \quad (10)$$

where $(\Delta L/L_o)(T)$ represents the fractional length change at temperature T due to thermal expansion ($\Delta L = 0$ and $L(T_o) = L_o$ at $T = T_o$). We can say also that

$$\frac{\rho(T)}{\rho_o} = \frac{L_o^3}{L^3(T)} = \left[1 + \frac{\Delta L}{L_o}(T) \right]^{-3}. \quad (11)$$

Let us define

$$V_{\ell o}(T) \equiv 2 \frac{L_o}{2\tau(T)}, \quad (12)$$

$$V_{so}(T) \equiv \left\{ \left[\frac{\Delta t(T)}{D_o} \right]^2 + \frac{[2\tau(T)]^2}{4L_o^2} \right\}^{-1/2}, \quad (13)$$

$$\sigma_o(T) \equiv \frac{\left(\frac{1}{2}\right) \left[\frac{V_{\ell o}(T)}{V_{so}(T)} \right]^2 - 1}{\left[\frac{V_{\ell o}(T)}{V_{so}(T)} \right]^2 - 1}, \quad (14)$$

$$\mu_o(T) \equiv \rho_o V_{so}^2(T), \quad (15)$$

and

$$E_o(T) \equiv 2\mu_o(T)[1 + \sigma_o(T)]. \quad (16)$$

Then it can be seen that

$$V_l(T) = V_{l_0}(T) \left[1 + \frac{\Delta L}{L_0}(T) \right], \quad (17)$$

$$V_s(T) = V_{s_0}(T) \left[1 + \frac{\Delta L}{L_0}(T) \right], \quad (18)$$

$$\sigma(T) = \sigma_0(T), \quad (19)$$

$$\mu(T) = \mu_0(T) \left[1 - \frac{\Delta L}{L_0}(T) \right], \quad (20)$$

and

$$E(T) = E_0(T) \left[1 - \frac{\Delta L}{L_0}(T) \right]. \quad (21)$$

We have here used the approximation that

$$\left[1 + \frac{\Delta L}{L_0}(T) \right]^{-1} \approx \left[1 - \frac{\Delta L}{L_0}(T) \right]. \quad (22)$$

The quantities V_{l_0} , V_{s_0} , μ_0 , σ_0 , E_0 , which are all functions of temperature, can be considered as the first approximations to the actual variables. The addition of the factor involving length change with temperature adds a correction that is an order of magnitude less than the first approximations.

D. Experimental Apparatus

The experimental apparatus (see Fig. 2) includes the ultrasonic equipment and the high-temperature vacuum furnace.

Fig. 2
Overall View of
the Facility

1. Ultrasonic Equipment and Data Taking

Figure 3 is a block diagram of the ultrasound generating and detecting equipment used for these measurements. The source of 5-Mc/sec pulses, nominally $0.5 \mu\text{sec}$ long, is a Sperry Products Division Ultrasonic Attenuation Comparator. A detailed description of it is contained in the literature.¹³ An Arenberg Ultrasonic Laboratory Model PA-620 tuned pre-amplifier, working in conjunction with an Arenberg WA-600 wide-band amplifier,¹⁴ received the returning sound echoes, which were finally viewed and measured on a Tektronix Type 535A oscilloscope with a Type B preamplifier.¹⁵ X-cut quartz crystals were used as transducers; their fundamental frequencies were 5 Mc/sec, and their diameters were either 1.27 or 1.88 cm. Nonaq¹⁶ stopcock grease was used to couple the ultrasonic vibrations of the transducers to the metal rods. One face of the quartz crystal was plated with a thin layer of silver, which acted as one electrode; the flat face of the specimen was the grounded electrode.

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Fig. 3. Block Diagram of Ultrasound Equipment

Figure 4 is a photograph of the face of the oscilloscope showing a typical echo train that is received from a sample like that shown in Fig. 1. The first pulse, labelled A, is the reflection from the shoulder in the bar. Pulse B is the end reflection, and the succeeding echoes are the trailing pulses generated in the small-diameter section. The scale of the photograph is such that each major division along the horizontal axis is $5 \mu\text{sec}$.

By the use of the time-delay circuits of the oscilloscope, the elapsed time between the leading edges of pulses A and B is measured, and this is the value of $2\tau(T)$ needed in Equations (5) and (6). The time intervals between the leading edges of the trailing pulses are then measured, and the average of these measurements (usually two or three trailing pulses are suitable for measurement) is the value of $\Delta t(T)$ used in Equation (6).

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Fig. 4. Echo Train after One Round Trip through Specimen

2. Vacuum Furnace and Temperature Measurements

Figure 5 shows the vacuum furnace in which the specimens are mounted, the water jacket, and the electrode cap in which electrical contact

Fig. 5
Vacuum Furnace, Cooling
Jacket, Electrode Cap with
Mounted Specimen

was made to the quartz transducer. The small-diameter end of the rod is suspended inside the ceramic tube, which acts as a support for the 1-mm-diam tantalum heater wire. The top cover of the furnace is removable; the rod is sealed into the cover through a port that contains two O rings. The O rings are compressed against the specimen and against the wall of the port by a threaded nut. This compression makes the port vacuum-tight and also supports the full weight of the specimen as well as the force of the external atmospheric pressure acting upon the top of the rod. The top cover is hollow and is kept filled with circulating water so that the O rings in the port are maintained at a sufficiently low temperature. Enough heat comes up along the rod so that another cooling jacket is required to keep the rod cool enough to touch. In this device, the water actually bathes the specimen itself. The surface of the specimen must be smooth where the vacuum and water seals are located, but generally these areas are small enough so that the amplitudes of trailing pulses generated in them are very low.

The furnace draws about 18 Amp at 130 V (2.3 kW), and the maximum temperature attainable ranges from 900 to 1200°C, depending upon the thermal conductivity of the rod. The temperatures at two points on the surface of the small-diameter section of the rod are measured by means of thermocouples. With most specimens, Chromel-Alumel couples can be used. If possible, each thermocouple junction and its contact to the rod are made simultaneously by means of a resistance welder. The thermocouple wires are 0.25 mm thick, so a small junction can be made. The junctions are usually made to lie in the same vertical plane on the same side of the specimen; one junction is placed about 3 mm below the shoulder, and the other about 3 mm from the end of the rod. The bare-wire thermocouples are brought out of the furnace through compression seals, and the voltage outputs are read on a portable potentiometer.

The temperature differences between the two thermocouple junctions vary from zero to as much as 50°C. This thermal gradient depends upon the length of the sample inside the heat zone, upon the sample composition, and mostly upon the temperature. The furnace, as it exists in its present form, is not suitable for studying the elastic properties in regions in which these parameters are rapidly varying with temperature, such as in the vicinity of phase changes. In most materials of current interest, however, these parameters are slowly-varying functions of temperature, at least up to 1000°C.

The furnace is supported upon a liquid-nitrogen cold trap, which is connected to a 4-in. oil-diffusion pump and backed by a mechanical pump. Pressure inside the furnace is measured by a Bayard-Alpert type ionization gauge tube and control and by thermocouple gauges. With liquid nitrogen in the cold trap, the pressure in the furnace at room temperature reaches 2×10^{-7} Torr after a day of pumping and after the walls of the furnace have been outgassed by applying external heat. The pressure in the heated furnace

depends upon the rate of outgassing of the specimen. It is usually in the 10^{-5} -Torr range, but, if allowed to remain at 1000°C for an hour or more, generally it comes down into the 10^{-6} -Torr range. Considerable outgassing is noticed whenever the current in the furnace winding is increased.

E. Accuracy and Experimental Error

1. Accuracy

Transit-time measurements are made by using the time-delay circuits of the oscilloscope, which are calibrated, right at the time of measurement, by a Hewlett-Packard Model 524B Electronic Counter,¹⁷ whose accuracy is given by the manufacturer as five parts in 10^8 . Length and diameter are measured with laboratory verniers and micrometers. The accuracy of the length-measuring device is ± 0.0003 cm; the accuracy of the instrument used to measure diameters is better than ± 0.0001 cm. Variations in the flatness or effects due to nonparallel faces are not expected to produce any noticeable effects on the accuracy of the transit-time measurements. Since the sound pulses are guided along the bar by its boundaries, the waves will not "wander" away from the transducer if the end faces or shoulder are not exactly perpendicular to the sound beam. The only noticeable effect would be a widening of the echo pulses; variations in flatness or parallelism of 0.001 cm might be expected to widen the pulse by about 4×10^{-3} μsec , or about 0.02 cycle. Temperature readings above 260°C are within 0.75% of the nominal values as determined by the thermocouples at their actual points of contact. However, the temperature profiles inside the specimens are not known. Based on studies performed by Dunegan,¹² temperature differences of about 25 Centigrade degrees could exist inside the sample.

Sample densities are measured by the buoyancy method; here, the factor limiting the accuracy is knowledge of the density of the immersing fluid. For carbon tetrachloride, which is normally used, the density given by the manufacturers is good to 0.12%; thus, the accuracy of the sample-density measurement is that same figure. Inaccuracies in the balances used to weigh the samples are much less than this.

Thermal expansions are not measured in conjunction with these experiments; data based on the work of others must be used. Thus the accuracy of our final results will depend upon the accuracy of the expansion data; however, the thermal expansion has only a small effect on the results, as was shown in Equations (17) through (21).

McSkimin¹⁸ has shown how velocities measured in long rods may be in error because of diffraction effects. Using his criteria, we estimate that our velocity measurements would not be in error by more than 0.025% due to these effects.

Considering all the above sources of systematic error, we have calculated that the accuracy of the method should be within 0.2% for Young's modulus and for the shear modulus, and within 0.03% for Poisson's ratio. However, another source of error may seriously reduce the accuracy of the method.

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Fig. 6. Leading Edge of an Echo Train

Figure 6 shows a portion of the oscilloscope trace; seen are the base line and the leading edge of an echo pulse. Notice that the base line is made up partly of small oscillations of the same frequency as the pulse. These oscillations are not strictly noise, since they are not random in time. When the amplitude of the echo becomes very low, it may be difficult to determine exactly where the leading edge of the pulse is located. In such cases, it is possible to be in error by an amount of time equivalent to a multiple of the period of the 5-Mc oscillations. If the transit time, 2τ , and the delay time, Δt , were both in error in the amount of $0.2n \mu\text{sec}$, where n is the number of cycles "off" from the true leading edge, then the values of σ could be in error by $2n\%$; the values of E and μ would then be in error by $6n\%$. The longitudinal- and shear-wave velocities would be off by $2n\%$ and $3n\%$, respectively. These figures are based on a transit time of $10 \mu\text{sec}$ and a delay time of $5 \mu\text{sec}$. This situation would generally arise only under conditions of poor echo trains, but might be expected to occur at high temperatures when the attenuation of the sound generally increases.

2. Experimental Error

We have previously discussed the inherent accuracy of the method based on a knowledge of the systematic errors present. Nothing has been said about the precision of the method based on experimental errors, which are reflected in the reproducibility of the measurements. We have made repeated measurements of all the necessary parameters on actual samples at several different temperatures to determine what the repeatability was. Using standard systems of statistical analysis,¹⁹ we calculated the standard deviations in measurements of length, transit time, density, etc., and from these computed the standard deviations in the results. For all the moduli, the standard deviations range from 0.11 to 3.3% of their absolute values. I believe that, in general, considering the improvement in the measuring technique that has occurred as we have become more familiar with the equipment, our results can be considered to have a precision or reproducibility of 1% for all three calculated moduli.

It thus appears that the overall error in determining the moduli is mostly due to experimental error, except when the leading edges of pulses cannot definitely be ascertained, in which case the measurement may have a large systematic error.

II. RESULTS ON TV-20

A. Material

An arc-melted ingot of vanadium-20 w/o titanium alloy was obtained from Union Carbide Corporation (ingot number UC-761-1). It was canned in Type 304 stainless steel and extruded through a die at 1100°C (ANL extrusion number 290), and the extrusion ratio was 6.5:1. After extrusion the bar was sectioned and the 304 SS jacket removed by mechanical peeling. A 22.9-cm section (No. 290-1) was turned down to a 3.3-cm diam and cold-rolled to a diameter of 2.6 cm, which resulted in a total reduction of approximately 37%. The rolled bar was press-straightened and vacuum-annealed for 1 hr at 900°C.

The analysis of the original arc-cast ingot is as follows:

<u>Element</u>	<u>Content</u>
Ti	20.2 w/o
C	480 ppm
O ₂	358 ppm
N ₂	138 ppm
H ₂	13 ppm
V	Remainder

The annealed rod was then machined to the configuration shown in Fig. 1. The dimensions of the small-diameter region were $L_0 = 3.808$ cm and $D_0 = 1.748$ cm; $T_0 \approx 25^\circ\text{C}$. The total length of the rod was 25.4 cm, and the diameter (measured at the smooth section of the rod) was 2.55 cm.

A small section of the rod had been cut off before the final machining. The density of this piece was determined to be

$$(5.728 \pm 0.004) \text{ g/cm}^3.$$

Rhude and Yaggee²⁰ measured the thermal expansion of TV-20. Their values were used in computing the final results of this study.

B. Sound Velocities

Figure 7 shows the variation of longitudinal sound-wave velocity and of shear-wave velocity in TV-20. The horizontal lines through some data points denote the temperature gradient present in the small-diameter end of the sample.

Fig. 7
TV-20 Sound Velocities as Functions of Temperature. Horizontal lines through each data point indicate temperature difference in small-diameter section of sample.

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C. Moduli

Figure 8 shows the variation of Young's modulus, the shear modulus, and Poisson's ratio for TV-20. Also shown on the figure are the spans of possible error due to an error in determining the exact leading edge of the pulses by one cycle. This error may have been made at the high-temperature end of the run. Figure 9 shows two determinations of Young's modulus that have been reported previously and a comparison with the data

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Fig. 8. TV-20 Elastic Moduli as Functions of Temperature. Horizontal lines through each data point indicate temperature difference in small-diameter section of sample.

Fig. 9
 Comparison of Data from This
 Experiment with Previously Pub-
 lished Data for Young's Modulus

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reported here. The previously reported data are by Hill and Wilcox,²¹ who measured the moduli for many different vanadium-titanium alloys (the values shown in the figure are interpolated between their values for V-17Ti and V-25Ti), and by Yaggee and Gilbert.²² Our data are essentially in agreement with their work. Table I lists the longitudinal-wave velocities, shear-wave velocities, and the three moduli as functions of temperature.

TABLE I. Sound-wave Velocities and Elastic Properties of TV-20

Average Temperature, °C	Temperature Difference, °C	Longitudinal- wave Velocity, 10^5 cm/sec	Shear-wave Velocity, 10^5 cm/sec	Young's Modulus, 10^{11} dyne/cm ²	Shear Modulus, 10^{11} dyne/cm ²	Poisson's Ratio
18.5	0.05	5.87	2.60	10.7	3.87	0.378
25	0.3	5.95	2.64	11.0	4.00	0.377
221	12.3	5.89	2.63	10.8	3.91	0.375
308	17.0	5.88	2.61	10.7	3.87	0.377
410	22.2	5.80	2.62	10.7	3.88	0.372
507	26.7	5.78	2.60	10.4	3.82	0.373
586	29.0	5.78	2.59	10.3	3.76	0.375
683	30.6	5.80	2.57	10.3	3.71	0.376
787	32.4	5.74	2.56	10.1	3.67	0.375
830	45.4	5.79	2.56	10.0	3.64	0.379
885	30.8	5.72	2.55	9.91	3.62	0.376
939	30.6	5.67	2.55	9.90	3.60	0.374
944	48.6	5.76	2.53	9.45	3.56	0.380
1024	49.7	5.73	2.50	9.59	3.47	0.382
1099	One thermo- couple broke	5.70	2.51	9.57	3.47	0.380

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