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Dynamic Elastic Moduli of Iron, Aluminum, and Fused Quartz*

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The dynamic elastic constants of Armco iron, pure aluminum, and fused quartz have been measured with a supersonic pulse technique as functions of pressure and temperature over the range 1 to 9000 bars and 25°C to 300°C (200°C for quartz).

Within the experimental accuracy the dynamic elastic moduli of these materials can be represented by linear functions of pressure and temperature.

The isothermal bulk moduli of other observers have been used to compute the equivalent adiabatic moduli by use of the thermodynamic relations. Comparison shows that at high pressures the dynamic moduli agree with the adiabatic moduli within the experimental accuracy for iron and aluminum. At low pressure the dynamic modulus of Armco iron is less than the adiabatic. The deviation is small but is about four times the estimated probable error.

INTRODUCTION

ELASTIC wave velocities in small samples can be measured with excellent accuracy with modern electronic equipment and piezoelectric transducers. A wide variety of experimental arrangements and apparatus have been employed.[1–3] The pulse method is very convenient for measuring velocities as a function of hydrostatic pressure and temperature[2] or of nonhydrostatic stress.[4] Since the moduli are effectively measured directly and not by differentiating the data on volume or length as a function of stress the accuracy should be great. A study of several materials, however showed discrepancies greater than the experimental error of either method. A very careful investigation has therefore been undertaken to determine the source of these discrepancies.

EXPERIMENTAL PROCEDURE

The apparatus for subjecting the sample to hydrostatic pressure and timing the pulses through the samples has been described.[5] A few slight modifications have been made. A second intensifier has been added so that the upper pressure limit is 10 000 bars; a thyratron pulser has been substituted for the pulse amplifier; and an internal furnace has been constructed for the pressure cylinder. The temperatures are measured by thermocouples placed directly on the sample and the temperature is controlled by a Foxboro Controller. Kerosene has been used as the pressure fluid for most of the work; carbon dioxide and argon have been used occasionally.

The samples were cylindrical rods 1 to 3 in. long and $\frac{3}{4}$ to 1 in. in diameter. Elastic pulses were generated by an x-cut quartz crystal on one end of the sample and detected by a second x-cut crystal on the opposite end.

The authors are indebted to The Aluminum Company of America for the aluminum samples. Mr. L. A. Willey of the Aluminum Company kindly furnished the following analysis: aluminum 99.99+%, copper 0.002%, iron 0.001%, silicon 0.001%, and others 0.000%. He also provided very accurate values of the thermal expansion over the range 20° to 500°C which are required in the calculations. The value of the specific heat, C_p, was taken from a publication of the Dow Chemical Company.[6] The measured density was 2.698 g/cm³ at 25°C. Annealing produced no detectable change in its properties.

The fused quartz was obtained from the General Electric Company. The measured density was 2.202 g/cm³. The measured density of the Armco iron was 7.872 g/cm³.

A dilatational pulse traveling axially along a cylinder generates secondary pulses[1] delayed with respect to the primary pulse by a time which is a function of the dilatational wave velocity, rotational wave velocity, and the diameter of the rod. These secondary pulses together with the main pulse permit the dilatational and rotational wave velocities to be determined simultaneously.[2] The dynamic bulk modulus can thus be computed from the equation

$$K_D=\rho(V_D^2-4/3V_R^2), \tag{1}$$

where ρ is the sample density, V_D is the dilatational wave velocity, and V_R is the rotational wave velocity. The quantities ρ, V_D, and V_R are computed for the sample length and volume under the particular pressure and temperature of the observation.

There are several sources of error which must be watched carefully. If the rise time of the driving pulse is not very short or if the sample attenuates the high frequencies, the received pulse will have no readily identifiable beginning and the arrival time will be

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[2] Hughes, Pondrom, and Mims, Phys. Rev. 75, 1552 (1949).

[3] D. Lazarus, Phys. Rev. 76, 545 (1949).

[4] D. S. Hughes and J. T. Kelly, Phys. Rev. 92, 1145 (1953).

[5] D. S. Hughes and J. H. Cross, Geophysics 16, 577 (1951).

[6] D. R. Stull and G. C. Sinke, "The thermodynamic properties of the elements in their standard state," Dow Chemical Company, Midland, Michigan (1955).

TABLE I. Armco iron. V_D longitudinal wave velocity (m/sec), V_R rotational wave velocity (m/sec).

P bars	26°C V_D	26°C V_R	100°C V_D	100°C V_R	200°C V_D	200°C V_R	300°C V_D	300°C V_R
1	5928	3203	5876	3165	5801	3119	5748	3061
1000	5936	3219	5886	3174	5820	3148	5758	3072
2000	5951	3229	5901	3185	5836	3154	5778	3077
3000	5959	3233	5916	3189	5844	3168	5790	3082
5000	5966	3242	5925	3198	5859	3178	5801	3099
7000	5982	3248	5945	3200	5875	3179	5810	3120
9000	6001	3252	5957	3212	5882	3181	5827	3136

uncertain. The driving pulse has a rise time of 0.02 μsec followed by an exponential decay of about 3 μsec. The maximum amplitude of the driving pulse is about 400 v. The received pulse is usually a damped oscillatory wave. If a quartz sample is used and quartz backers placed on the crystals the received pulse is almost identical with the driving pulse.

If the main pulse contains many oscillations the true beginning of the secondary pulse may be unreadable. Since V_R is measured by taking the time of the secondary pulse following the main pulse, great care must be used in reading this pulse. In the present work the shear velocities have been checked for the room temperature run to 9000 bars using y-cut crystals with argon as the pressure fluid and Dow Corning V-9 resin for coupling the crystals to the sample. This material becomes too fluid to provide coupling at high temperatures.

This method gave V_R of Armco iron as 3213 m/sec at 25°C and atmospheric pressure. Resonance methods[7] show V_R of Armco to be 3220 m/sec in the frequency range 0.5 to 1.5 mc and to decrease from this value to about 3210 m/sec at 3 mc.

By using samples of various lengths and crystals of various thicknesses it has been found that the observed time through the sample depends upon the thickness of the crystal. It is necessary to subtract $\frac{1}{4}f$ μsec from the observed travel time where f is the fundamental frequency of the crystals in Mc/sec to obtain the correct travel time.

At each temperature, readings were taken at atmospheric pressure, 1, 2, 3, 5, 7, and 9 thousand bars. Readings were taken with both increasing and decreasing pressure. Most runs were repeated. The times were consistent on repetition to about ± 0.01 μsec for the direct pulse and about ± 0.02 μsec for the secondary pulse. The computed probable error in K_D for the materials studied is 0.3%. All three materials were measured at 25, 100, and 200°C. Armco iron and aluminum were also measured at 300°C.

Table I shows the observed velocity data on Armco iron for all pressures and temperatures. These velocities are for an infinite media. At atmospheric pressure and temperature we have used samples with diameters varying from $\frac{1}{4}$ in. to $2\frac{1}{2}$ in. and lengths varying from $\frac{1}{2}$ in. to 48 in. All these samples have shown the same velocity within the accuracy of reading the arrivals.

The dynamic bulk modulus of aluminum and Armco iron was thus measured at 28 pressure and temperature points and quartz at 21 points. It was found possible to fit all data with a linear relation of the type

$$K_d = K_0 + aT + bP. \quad (2)$$

In Eq. (2) K_d, K_0, and P are measured in bars, a in bars/°C, and T in °C. Table II shows the best value of these constants determined from the observations.

The last column is the mean difference without regard to sign between the observed values and the values computed with these constants. It indicates a probable error of about 0.4%.

TABLE II. Constants for computing K_d.

Material	10^{-5} K_0 bars	a, bars/°C	b	Mean deviation
Armco iron	16.96	−330	+4.3	±0.05
Aluminum	7.64	−200	+4.0	±0.03
Fused quartz	3.66	+140	−5.4	±0.02

TABLE III. Comparison of our results with Professor Bridgman's.

P bars	t°C	10^{-5} K_d, bars	10^{-5} K_s (Bridgman's)	Difference
		Armco iron		
1	30	16.86	17.11	−0.25
1	100	16.63	16.97	−0.34
9000	30	17.35	17.33	+0.02
9000	100	17.12	17.21	−0.09
		Aluminum		
1	30	7.57	7.68	−0.10
1	100	7.43	7.44	−0.01
9000	30	7.93	7.93	0.00
9000	100	7.79	7.70	+0.09

The adiabatic and isothermal bulk moduli are related by the equation

$$K_s = K_t + \frac{T\beta^2 K_s}{\rho C_P},$$

where K_s is the adiabatic modulus, K_t the isothermal, T the absolute temperature, β the volume coefficient of thermal expansion, ρ the density, and C_p the specific heat at constant pressure. As a comparison, Professor Bridgman's results for the isothermal compressibilities[8] of Armco iron and aluminum have been used to compute the adiabatic moduli. Table III shows a comparison of those adiabatic moduli with the dynamic moduli.

The rigidity moduli for these materials can also be represented by a linear relation

$$\mu_d = \mu_0 + cT + dP.$$

[7] D. S. Hughes and J. M. Kennel, J. Appl. Phys. 26, 1307 (1955).

[8] F. Birch, "Handbook of physical constants," Special Paper No. 36, Geological Society of America (1946), pp. 43–44.

The values of the constants are shown in Table IV. The value obtained for quartz is in fair agreement with other investigators[8] but it appears that different samples of fused quartz are physically different and precise agreement is not to be expected.

Dynamic moduli have been measured at extreme high pressures[9] for steel and aluminum. The observed dynamic bulk modulus of steel was 16.90×10^5 bars in very close agreement with our value of Table III. However, our values for the change in modulus with pressure is in much closer agreement with Professor Bridgman's value. The computed bulk modulus[9] for pure aluminum, 7.53×10^5 bars is in good agreement with our value. The rates of change of the bulk modulus with pressure are in fair agreement.

The agreement in Table II between K_d and K_s at high pressure for iron and for all pressures and temperatures for aluminum would seem to indicate that the experimental accuracy and method are adequate. The discrepancy for iron at atmospheric pressure is believed to be real. The sign of the discrepancy is somewhat surprising. It indicates that iron yields more to a rapidly applied stress than to a slowly applied stress. In other words, the stress-strain relations involve also the rates of change of the stresses and strains as postulated by Zener.[10] The data of Table III indicate that the stress relaxation time is about 4 μsec greater than the strain relaxation time if both are small and that with an increase in pressure these times decrease or become equal. A relaxation time of 7.7×10^{-4} μsec has been observed in crystalline quartz.[11]

TABLE IV. Constants for computing μ_d.

Material	$10^{-5}\,\mu_0$ bars	c, bars/°C	d
Armco iron	8.21	−294	3.4
Aluminum	2.65	−156	2.6
Fused quartz	3.05	+46	−3.0

[9] Goranson *et al.*, J. Appl. Phys. **26**, 1472 (1955).

[10] C. Zener, *Elasticity and Anelasticity of Metals* (University of Chicago Press, Chicago, 1948), first edition, p. 41.

[11] Bömmel, Mason, and Warner, Phys. Rev. **102**, 64 (1956).

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Diffusion of Gold in Single Crystals of Silver*†

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The diffusion of Au in Ag has been measured in the temperature range from 650 to 950°C, using Au^{198} as a tracer. The data indicate that the diffusion coefficient is given by the equation $D=0.26\exp(-45\,500/RT)$ cm²/sec. The fact that the activation energy for the diffusion of Au in Ag is identical to that for self-diffusion in Ag is discussed briefly.

INTRODUCTION

ONE of the basic needs in the field of diffusion is experimental data which can be used to check existing theories and to provide indications of the direction future theories should take. This means, first, that data of high accuracy are needed, and second, that there is a need for systematic data on the effect on diffusion rates of the valence, atomic size, and crystal structure (interatomic forces) of the solute. Tomizuka and Slifkin[1] have reported accurate data on the effect of valence using silver as the solvent metal. The present program is concerned with the diffusion of solute atoms of the same valence and crystal structure but of different atomic size. Recently, the data for diffusion of zinc in silver were reported[2] and it was found that the activation energy was identical to that for cadmium in silver but the frequency factor was different. The present report gives the data for diffusion of gold in silver, to be compared to the results for self-diffusion in silver.

EXPERIMENT

Single crystals of silver (99.99% pure, obtained from Handy and Harman) were grown by using a modified Bridgman technique. Particular attention was paid to obtaining crystals free of excessive substructure. Acceptable single crystals were cut into short cylinders, etched, formed accurately in the lathe on which they were to be sectioned later, lightly polished, etched, and annealed at 850°C for two hours to remove residual strains. After a final etch to check monocrystallinity, one end of each specimen was plated with Au^{198},

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[2] A. Sawatzky and F. E. Jaumot, Jr., Phys. Rev. **100**, 1627 (1955).