A CW INTERFEROMETER FOR ELASTIC CONSTANT MEASUREMENTS

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ABSTRACT

A continuous wave ultrasonic interferometer is described which makes possible measurement of both absolute values of and small changes in the elastic constants of solids using commercially available subsystems.

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INTRODUCTION

Although continuous wave velocity measuring techniques were among the first dynamic methods employed to obtain values of the elastic moduli ¹⁻⁵, these early low frequency cw methods had been almost entirely replaced by pulse techniques because of the higher precision of the latter. In recent years, as research has extended into areas where the pulse methods are difficult to apply, renewed use has been made of continuous wave methods.

Bolef and coworkers ⁶⁻¹¹ as well as others ¹²⁻¹⁵ have successfully applied MHz frequency continuous wave methods to the measurement of absolute elastic constants and elastic constant changes. The basic cw advantages are that thin samples can be used and that high attenuation can be tolerated. The present article describes a system which can be used both for absolute elastic constant measurement and elastic constant change measurement. The principle advantage of the present system is that it can be readily constructed with ease using commercial subsystems.

ABSOLUTE ELASTIC CONSTANT MEASUREMENT

Wide variations in the temperature, pressure and magnetic field environments, under which present and future research in our laboratory would be carried out, and limitations on the physical size of the device over whose entire volume the parameter of interest could be held constant, necessitated the development of an elastic constant measuring apparatus which minimized the volume whose environment was to be controlled. This requirement led directly to the investigation of continuous wave methods whose advantage over standard pulse techniques is readily seen in the

following calculation. Assuming for the pulse technique that the minimum pulse width is 10^{-5} sec and the sample velocity is 10^5 cm/sec, then the minimum sample length would be 0.5 cm; whereas for a cw system where the nominal frequency is 10^7 Hz and the sample velocity is 10^5 cm/sec, the minimum sample length would be 0.5×10^{-2} cm.

A block diagram of the electronic system which was constructed for the measurement of absolute elastic constants is seen in Figure 1. The interferometer simply consists of a Hewlett-Packard 8405A Vector Voltmeter and a Hewlett-Packard 606B Signal Generator with the necessary 50 ohm terminations. Figure 2a is a trace of the output transducer amplitude and Figure 2b of the phase difference between the input and output waves of a lithium single crystal, as the frequency is swept from 9.5 MHz to 10.5 MHz.

Bolef and Menes⁸ have shown that, if the frequencies are known at which amplitude maxima occur, the elastic constant can be found using the following formulas:

$$f_s^n = f_c^n \left(1 + k_t - \frac{k_b}{2}\right) - k_t f_t, \quad (1)$$

$$\Delta f_s = \Delta f_c \left(1 + k_t - \frac{k_b}{2}\right), \quad (2)$$

$$v_s = 2\ell \Delta f_s, \quad (3)$$

$$C = \rho_s v_s^2, \quad (4)$$

where f = frequency,

n designates the integer associated with the resonance frequency f ,

$$k_t = \rho_t v_t / \rho_s v_s,$$

$$k_b = \rho_b v_b / \rho_s v_s,$$

v = velocity,

ρ = density,

C = elastic constant, and

t , b , and s designate the transducer, bond, and specimen values respectively.

Figure 3 shows the variation of C' with temperature and pressure for a lithium single crystal. The error in the determination of C' at any temperature and pressure is fitted to within $\pm .1\%$ using a two parameter curve fit. Similar data for other elastic constants indicate that the percentage error is a function of the magnitude of the elastic constant as follows:

At room temperature the elastic constants C' and C_{11} of lithium are 11×10^9 dynes/cm² and 130×10^9 dynes/cm² respectively. The density is .533 gm/cm³. Assuming a sample length of 0.5 cm and using Equations (3) and (4), Δf is found to be as follows for the two elastic moduli

$$\Delta f = \frac{1}{2\ell} \sqrt{\frac{C}{\rho}}, \quad \Delta f(C') = 143.7 \text{ KHz},$$

$$\Delta f(C_{11}) = 493.9 \text{ KHz}.$$

With a working range of approximately 1 MHz around a nominal 10 MHz transducer resonance frequency, 8 resonance peaks are available for the C' wave, whereas 3 are obtainable for the C_{11} wave. Because the number of points involved in the latter case is reduced greatly, the accuracy with which the larger elastic constant can be obtained is decreased considerably relative to the accuracy with which the smaller of the elastic constants can be measured.

ELASTIC CONSTANT CHANGE MEASUREMENTS

Many interesting phenomena involve small changes in the the elastic moduli. These include magneto-acoustic oscillations,

temperature and pressure effects, chemical impurity effects, and some phase transitions such as the superconducting to normal transition.

Figure 4 is a block diagram of the cw interferometer used to make change measurements. As with the absolute measurement system, the cw signal originates with a Hewlett-Packard 606B Signal Generator. The frequency is stabilized to ± 2 Hz using a Hewlett-Packard 8708A Synchronizer. The stabilized signal is applied to the input piezoelectric transducer which is cemented to one sample face. The input signal and the signal generated by the output transducer are detected using a Hewlett-Packard 8405A Vector Voltmeter. A voltage, provided by the Vector Voltmeter and corresponding to a relative phase deviation between the input and output signals, is applied to the modulation input of the Synchronizer which automatically adjusts the signal generator frequency such that null phase difference is maintained. (In other words, the null phase shift condition is tracked by adjusting the signal frequency.) The response function of the Synchronizer depends upon the slope of the phase vs. frequency curve. The slope of the response function, and hence the sensitivity of the feedback loop, can be increased by inserting an amplifier between the Vector Voltmeter output and the Synchronizer deviation input. For this reason a PAR Boxcar Integrator was used which also served to filter out any white noise introduced through the Vector Voltmeter. The frequency is either monitored at discrete points using a digital printer or, via a digital to analog converter, plotted by an X-Y recorder as a function of the external parameter inducing the velocity change.

If the logarithmic derivative of Equation (4) is taken and $\frac{\Delta C}{C}$ is assumed small, the change in elastic constant due to a variation in an external parameter N is expressed by the formula

$$\frac{1}{C_s} \frac{\Delta C_s}{\Delta N} = \frac{1}{\rho_s} \frac{\Delta \rho_s}{\Delta N} + \frac{2}{f_s} \frac{\Delta f_s}{\Delta N} + \frac{2}{\ell_s} \frac{\Delta \ell_s}{\Delta N}, \quad (5)$$

where second order effects (i.e., change in correction terms with changing N) have been neglected.

Figure 5 illustrates the accuracy of the present system by presenting data on the magnetic field induced superconducting to normal transformation in single crystal niobium at 4.2°K and atmospheric pressure. The particular modulus is C_{44} , the (100) shear modulus. This transformation was chosen because the change was small and controllable and also because the length change portion of Equation (5) is negligible, thus offering a good test of the controllability and reproducibility of the electronic system. The three separate excursions of magnetic field contained in the curve are itemized in Table I. The maximum error occurs at 2944 Oersted and represents an error of 5 Oersted in the magnetic field setting. The estimated maximum error in the magnetic field is ± 8 Oersted. As the error in the low field frequency readings is ± 1 Hz, the apparent error in frequency (± 5 Hz) at higher fields is nearly entirely due to magnetic field reproducibility. In the present case the total change of elastic constant (in going from the superconducting to the normal phase) divided by the value of the constant is $\Delta C_{44}^{\text{total}} / C_{44} = 2.27 \times 10^{-4}$. The actual error in the change measurements owing to the frequency measurement system is $\delta C_{44} / C_{44} = \pm 2 \times 10^{-7}$.

TABLE I

Results of Magnetic Field Dependence of Nb Single Crystal Resonance Frequency. The corresponding elastic constant is C_{44} . Three magnetic field excursions were made.
 $f_0 = 10056756$ Hz.

Dial Setting	Field (Oersted)	$f - f_0$ (Hz)		
		1	2	3
0	0	0	0	0
.1	0	0	0	0
.2	46.0	0	0	0
.3	126.5	0	0	0
.4	207.0	0	0	0
.5	287.5	0	0	0
.6	368.0	0	0	0
.7	448.5	0	0	-1
.8	529.0	0	0	-1
.9	609.5	0	0	-1
1.0	690.0	-1	-1	-1
.1	770.5	-1	-1	-1
.2	851.0	-1	-1	-2
.3	931.5	-1	-1	-2
.4	1012.0	-1	-1	-1
.5	1092.5	0	-1	-1
.6	1173.0	0	0	1
.7	1253.5	2	1	1
.8	1334.0	3	4	5
.9	1414.5	7	6	7
2.0	1495.0	13	13	15
.1	1575.5	24	21	25
.2	1656.0	37	36	40
.3	1736.5	55	55	58
.4	1817.0	81	77	84
.5	1897.5	111	114	114
.6	1978.0	151	150	154
.7	2058.5	195	192	197
.8	2139.0	247	245	246
.9	2219.5	304	304	308
3.0	2300.0	368	368	372
.1	2380.5	432	435	437
.2	2461.0	511	509	512
.3	2541.5	586	585	583
.4	2622.0	660	663	663
.5	2702.5	745	745	745
.6	2783.0	821	825	824
.7	2863.5	904	905	905
.8	2944.0	1005	1016	1008
.9	3024.5	1122	1127	1131
4.0	3105.0	1137	1138	1139
.1	3185.5	1140	1138	1142
.2	3266.0	1140	1138	1142
.3	3346.5	1140	1138	1142
.4	3427.0	1140	1138	1142

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REFERENCES

1. J. Zacharias, Phys. Rev. 44, 116 (1933).
2. L. Balamuth, Phys. Rev. 45, 715 (1934).
3. W. T. Cooke, Phys. Rev. 50, 1158 (1936).
4. S. L. Quimby and S. Siegel, Phys. Rev. 54, 293 (1938).
5. S. Siegel and S. L. Quimby, Phys. Rev. 49, 663 (1936).
6. R. L. Melcher, D. I. Bolef and J. B. Merry, Rev. Sci. Instr. 39, 1613 (1968).
7. R. L. Melcher, D. I. Bolef and J. B. Merry, Rev. Sci. Instr. 39, 1618 (1968).
8. D. I. Bolef and M. Menes, J. Appl. Phys. 31, 1010 (1960).
9. D. I. Bolef and J. deKlerk, IEEE Trans. on Ultrasonic Eng. UE-10, 19 (1963).
10. J. G. Miller and D. I. Bolef, Rev. Sci. Instr. 40, 361 (1969).
11. R. G. Leisure and D. I. Bolef, Rev. Sci. Instr. 39, 199 (1968).
12. R. G. Leisure and R. W. Moss, Rev. Sci. Instr. 40, 946 (1969).
13. B. G. W. Yee and J. D. Gavenda, Phys. Rev. 175, 805 (1968).
14. A. G. Beattie and E. A. Uehling, Phys. Rev. 148, 657 (1966).
15. A. G. Beattie, Phys. Rev. 174, 721 (1968).

FIGURE CAPTIONS

- Figure 1. Block diagram of absolute value elastic constant measurement apparatus.
- Figure 2. X-Y recorder trace of (a) the amplitude and (b) the phase difference output of the Vector Voltmeter with a lithium single crystal as sample. $T = 200^{\circ}\text{K}$, $P = 1$ Kbar, trace center frequency is 10 MHz, trace width is 1 MHz, the corresponding elastic constant is $C' = (C_{11} - C_{12})/2$.
- Figure 3. Measured C' values of lithium plotted vs. temperature. Straight lines indicate best fit values.
- Figure 4. Block diagram of elastic constant change measurement apparatus.
- Figure 5. Resonance frequency change of a niobium single crystal vs. magnetic field dial setting. $T = 4.2^{\circ}\text{K}$, $P = 1$ bar, the corresponding elastic constant is C_{44} .

(a)

(b)