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A Marx Three Component Oscillator for Internal Friction Measurements at Low and High Temperatures in High Vacuum

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An apparatus for the measurement of internal friction in the kilohertz frequency range is described. The Marx three component piezoelectric technique is used and the apparatus is designed for measurements at low and high temperatures in high vacuum. A graphical technique for calculating the damping from the measured voltages is presented and is used in a manner that improves the accuracy with which amplitude independent and amplitude dependent damping can be separated.

I. GENERAL

ONE of the methods widely used for the measurement of the internal friction and elastic moduli of solids involves the excitation of specimens by piezoelectric crystals.[1] A common variation of this general technique, first introduced by Marx,[2] is shown in Fig. 1. The Marx oscillator consists of three basic components (a drive crystal, a gauge crystal, and the specimen) each of which is cut to resonate at a specified frequency, typically in the range 20–100 kHz for longitudinal vibration. Often a dummy rod (generally of fused silica) with a length equal to an integral number of half-wavelengths of the specified frequency is inserted between the specimen and the driver crystal so that temperature of the specimen may be changed while the crystals are maintained at ambient temperature.

The main feature of the Marx system is that the internal friction, modulus, modulus defect, and strain amplitude can be determined from the measurement of three quantities—the drive voltage V_d, the gauge voltage V_g, and resonant frequency f_r.

The internal friction of the system δ_t is given by

$$\delta_t=\alpha(V_d/V_g), \tag{1}$$

where α, the proportionality constant, may be calculated[2] or determined experimentally from the width of the resonance curve in the gauge circuit. For example, if Δf is the difference between frequencies at -3 dB from the output of the system at resonance, then $\delta_t=\Delta f/f_r$ if the internal friction is amplitude independent. The internal friction of the specimen δ_s is obtained from the total decrement and the decrement of the crystal–dummy composite δ_c by the relationship

$$m_t\delta_t=m_s\delta_s+m_c\delta_c. \tag{2}$$

Here m_s, m_c, m_t are the masses of the specimen, the crystal–dummy composite, and the total mass m_s+m_c of the system, respectively.

The modulus of the specimen M_s is determined directly from the resonant frequency f_s. In longitudinal vibration Young's modulus may be given by $E_s=\rho_s(2l_sf_s)^2$ where ρ_s is the density and l_s is the length of the specimen. The modulus defect is simply $2(f_s-f_{s,r})/f_{s,r}$ where $f_{s,r}$ is the resonant frequency corresponding to the relaxed modulus. f_s (and thus M_s) is determined from the resonant frequency of the system f_t through a relationship comparable to Eq. (2).

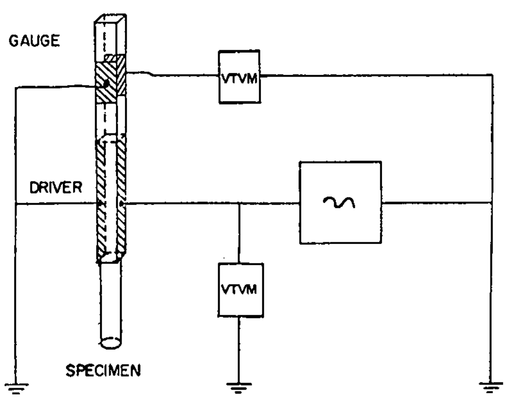

FIG. 1. The Marx[2] three component piezoelectric oscillator for measurement of internal friction.

[1] W. P. Mason, *Physical Acoustics and the Properties of Solids* (D. Van Nostrand, New York, 1958).
[2] J. Marx, Rev. Sci. Instrum. 22, 503 (1951).

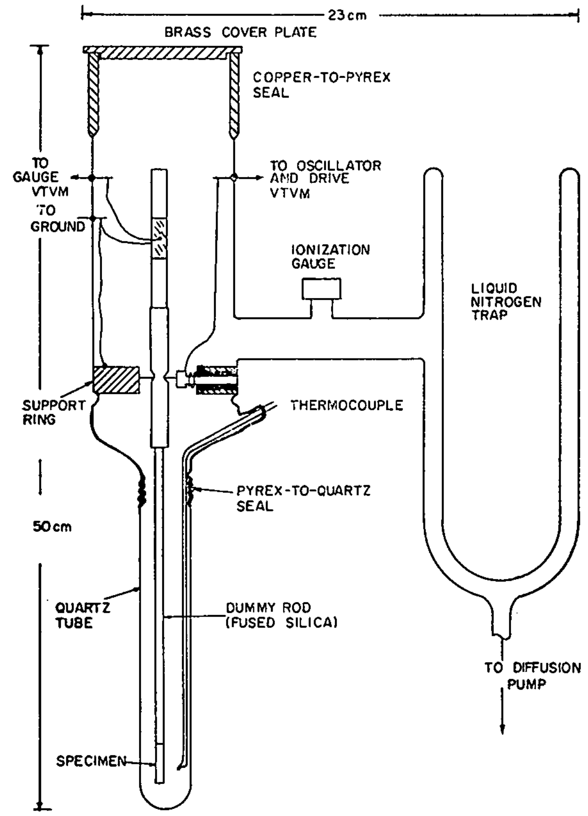

FIG. 2. The three component oscillator system for measurement of internal friction at high or low temperatures in high vacuum.

The strain amplitude of all components of the system is a linear function of the gauge voltage. The strain in the specimen is

$$\epsilon_s = \beta V_g, \tag{3}$$

where β is given by[2]

$$\beta = \frac{V_d}{V_g} \frac{1}{f_{r,h} l_s} \left(\frac{1}{2hm_c f_{r,h} R_h \delta_c} \right)^{\frac{1}{2}} \tag{4}$$

if calibration is made with the specimen removed. Here $f_{r,h}$ is the resonant frequency for the mechanical overtone of order h ($=1$ for the fundamental mode), R_h is the Ohmic resistance of the system, and l_s is the length of the specimen.

II. DESCRIPTION OF THE APPARATUS

In our investigations, it was necessary to measure internal friction of reactive refractory metals at low temperatures, as well as temperatures as high as 1200 K. Consequently, a simple combination of a high resolution internal friction device, a high vacuum system, and a system adaptable to the wide range of temperature was desired. A Marx three component oscillator system and the vacuum chamber and system depicted in Fig. 2 were selected.

The three component oscillator system is top loaded into the vacuum chamber, a Pyrex tube with a quartz cold (or hot) finger. An oscillator support ring rests on a concentric constriction put into the Pyrex wall by glass blowing. The vacuum system is sealed by soldering a brass cover plate to a coppér-to-Pyrex seal with a low temperature lead–tin (solid core) solder. Electrical leads to ground, the driver oscillator (General Radio model 1162-A frequency synthesizer), vacuum tube voltmeters (Ballantine, models 310 and 317), and thermocouple leads are connected through glass-to-metal seals. The temperature of the specimen is easily changed over wide ranges by surrounding the quartz tube with a small electric furnace (300–1200 K) or a Dewar (77–300 K) and heating coil. For measurements in the high temperature range, sodium silicate is used to bond the specimen to the dummy rod. Similarly, A-12 epoxy resin (Techkits, Adhesive Kit No. 200A) has worked well as a bonding agent between 77 and 380 K.

The main features of the vacuum system are its valveless and glass construction, small volume, and greaseless joints beyond the diffusion pump. These characteristics minimize the outgassing and improve the final vacuum. Typically a vacuum of $\sim 10^{-8}$ Torr is maintained in the chamber, even at high temperatures ($\sim 800°C$), with the system pumped by a 10 cm oil diffusion pump which is liquid nitrogen trapped and backed by a mechanical forepump. For measurements below room temperatures, helium transfer gas can be added to the forepumped system from a valve placed between the diffusion pump and the forepump.

Figure 3 gives a detailed view of the ring that supports the oscillator system and transmits the drive voltage to the crystal. The razor blade mounting was found to give a more stable support and less electric contact resistance than the wire type or point mounting generally used.

III. CALCULATION OF THE DAMPING

A convenient and accurate graphical technique has been used to calculate the internal friction of a specimen excited

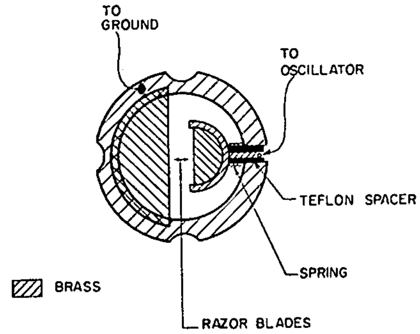

FIG. 3. Detailed view of support ring for the three component oscillator. The portions of the brass ring cut away at the outer surface are to permit the oscillator to be lowered past the glass-to-metal seals shown in Fig. 2.

in a three component oscillator system such as described above. The technique is particularly useful for measurements obtained on materials for which the internal friction has both strain amplitude independent and amplitude dependent components δ_I and δ_H, respectively, as in innumerable studies of dislocation damping of solids.[3]

From Eqs. (1) and (2), the damping of the specimen may be written in the form

$$\delta_s = A(V_d/V_g) - B, \tag{5}$$

where $A = \alpha(m_t/m_s)$ and $B = (m_c/m_s)\delta_c$ are constants at a given temperature and usually over wide ranges of temperature and strain amplitude. At constant temperature, δ_s may be expressed as the sum

$$\delta_s = \delta_I + \delta_H. \tag{6}$$

Equations (5) and (6) give

$$V_d = (B + \delta_I/A)V_g + (\delta_H/A)V_g \tag{7a}$$

or

$$V_d = V_{d1} + V_{d2}. \tag{7b}$$

Equation (7) suggests that a plot of V_d vs V_g is linear when the internal friction is amplitude independent and deviates (almost always positively) from this line when the internal friction becomes amplitude dependent. This is shown schematically in Fig. 4. From Eqs. (7a) and (7b) and Fig. 4 the amplitude independent damping is given by

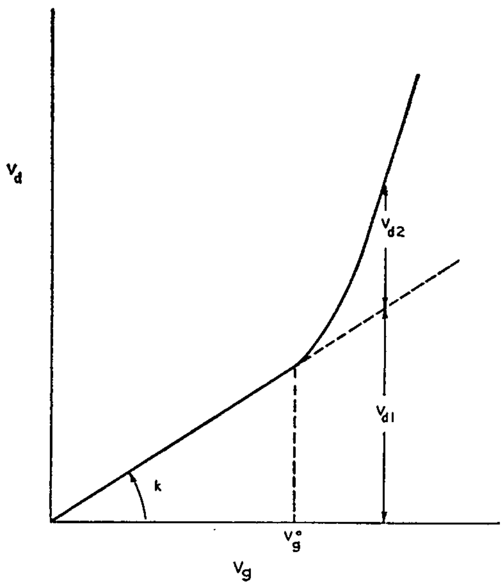

FIG. 4. Drive voltage vs gauge voltage for a three component oscillator in which the internal friction is amplitude dependent at high strain amplitudes.

[3] A. Granato and K. Lucke, *Physical Acoustics*, W. P. Mason, Ed. (Academic Press Inc., New York, 1967), Vol. IV.

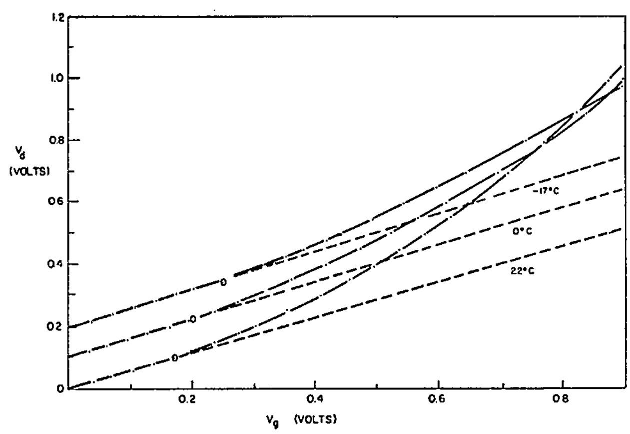

FIG. 5. Portions of typical curves of V_d vs V_g for a high purity single crystal of niobium. The curves are displaced by 0.1 V along the ordinate to prevent their overlap.

$$\delta_I = kA - B \tag{8}$$

and the amplitude dependent damping is

$$\delta_H = A(V_{d2}/V_g) = A(V_d/V_g - k). \tag{9}$$

Here k is the slope of the linear part of the curve of V_d vs V_g and is given by the ratio V_{d1}/V_g at any value of V_g.

As shown in Fig. 4, the strain at which the damping becomes amplitude dependent corresponds to the voltage at which the plot of V_d vs V_g becomes nonlinear. Notice also that the amplitude dependent damping of the specimen can be obtained without knowledge of the damping of the crystal–dummy composite, if desired. Finally, the amplitude independent damping and small changes in it may be determined very accurately from a least squares determination of the slope k of the initial straight line.

The calculation techniques suggested by Fig. 4 and Eq. (7) are simpler and more accurate than the usual method of computing damping and strain amplitude directly from Eqs. (1)–(3) and then attempting to analyze the data into components. The plot of V_d vs V_g gives the important additional condition that the slope V_{d1}/V_g must pass through the origin.

A typical set of data showing dislocation damping of a high purity single crystal of niobium tested at −17, 0, and 22°C with a 50 kHz oscillator system is given in Fig. 5. For this specimen one may easily trace the subtle change in "breakaway strain" (marked by the open circles -O-) from 3.8×10^{-6} at −17°C to 2.7×10^{-6} at 22°C, as well as to see that there are no measurable changes in the amplitude independent damping.

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