RESONANT ACOUSTIC DETERMINATION OF COMPLEX ELASTIC MODULI

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ABSTRACT

A simple, inexpensive, yet accurate method for measuring the dynamic complex modulus of elasticity is described. Using a "free-free" bar selectively excited in three independent vibrational modes, the shear modulus is obtained by measuring the frequency of the torsional resonant mode and the Young's modulus is determined from measurement of either the longitudinal or flexural mode. The damping properties are obtained by measuring the quality factor (Q) for each mode. The Q is inversely proportional to the loss tangent (tan δ). The viscoelastic behavior of the sample can be obtained by tracking a particular resonant mode (and thus a particular modulus) using a phase-locked-loop (PLL) and by changing the temperature of the sample. The change in the damping properties is obtained by measuring the in-phase amplitude of the PLL which is proportional to the Q of the material. The real and imaginary parts or, in general, the complex modulus can be obtained continuously as a function of parameters such as temperature, pressure, or humidity. For homogeneous and isotropic samples only two independent moduli are needed in order to characterize the complete set of elastic constants, thus, values can be obtained for the dynamic Poisson's ratio, bulk modulus, Lamé constants, etc.

INTRODUCTION

The accurate measurement of the elastic constants of materials and their dependence on temperature, static pressure and other ambient parameters is important in many fields of science and engineering research as well as in product design and quality control. The elastic modulus relates the strains to the applied stresses, so its value and its temperature dependence are important design parameters. Manufacturer's specifications for elastic constants of castable polymers and epoxies usually are determined by static techniques, if at all, and rarely contain more than one modulus. The data also varies widely depending upon sample preparation and cure temperature, and never contain information about the temperature dependance of the moduli or their dynamic properties. Since it is the dynamic modulus which is needed for engineering applications involving noise, shock, vibration, transducer design, and acoustics, it is important that the dynamic properties are available to the designer.

Due to the number of variations in the preparation and large numbers of epoxies and polymers that are available from manufacturers, it is desirable to have a convenient technique for measuring the moduli of material samples. In the following sections a technique for measuring the dynamic properties of materials is described which is convenient, accurate, precise, and economical. The method relies on the measurement of the frequencies of the longitudinal, flexural, and torsional resonant modes of a single rod shaped sample using the same two transducers to excite and detect all three modes.

The fact that the technique is resonant insures high signal-to-noise ratio while the fundamental measurement being a frequency means that one can obtain extremely high precision with inexpensive instrumentation (i.e. a frequency counter). The technique can be used with both insulating or conducting samples which are not ferromagnetic. An additional attractive feature of this technique is the fact that in addition to the shear modulus, the Young's modulus can be measured independently using either or both the longitudinal and flexural modes.

The technique for measurement of the torsional mode is a refinement of one developed first by Barone and Giacomini [1] to study the modes of vibration of bars having variable cross-section. A similar arrangement was used later by Leonard [2] to disprove the existence of the "Fitzgerald Effect" [3-5] by measuring the attenuation of torsional waves in Teflon™. The technique was modified by Professor Isadore Rudnick at UCLA in order to excite and detect the flexural and longitudinal modes in addition to the torsional mode while still using the same transducer. He then incorporated the free-free bar technique as a teaching laboratory experiment in an upper level undergraduate acoustics class at UCLA. The technique has been extended to measure the complex modulus in a manner similar to that used by Barmatz, et. al.[6], by measuring the quality factor, Q, or free decay time. We have also included a phase-locked-loop which allows the continuous tracking of the moduli and loss tangent as a function of an external variable such as temperature [7-9].

MEASUREMENT TECHNIQUE

Modes of a Bar: A Rod of Circular Cross-Section

A uniform, bar-shaped sample of a homogeneous, isotropic solid having a circular cross-section of diameter, d, and length, L, will propagate three independent waves (torsional, flexural, and longitudinal) provided the wavelength of vibration, λ, is much greater than the bar diameter. The vibrational modes will exhibit characteristic resonances at particular frequencies which depend upon the dimensions of the sample, the density, and the elastic modulus. Of course, the resonant frequency will depend on the boundary conditions imposed on the ends of the rod. The simplest and most reproducible boundary condition to impose on the ends of the rod is that of zero stress and zero moment (i.e. a free-free boundary condition).

The torsional resonances of a bar having a free-free boundary condition are given by

$$f^T{}_n = \frac{n}{2L}\sqrt{\frac{G}{\rho}}\ ; \quad n = 1, 2, 3... \ , \tag{1}$$

where G is the shear modulus, ρ is the mass density, and L is the length of the rod. The dynamic shear modulus of the bar material can then be easily expressed in terms of its density, length, and the ratio of the frequency of its n-th mode of vibration to its mode number, n as follows:

$$G = 4\rho L^2\left(\frac{f^T{}_n}{n}\right)^2 . \tag{2}$$

This result, in general, is dependent on the cross-sectional shape of the rod.

The longitudinal resonances of a bar having a free-free boundary condition are given by

$$f^L{}_n = \frac{n}{2L}\sqrt{\frac{E}{\rho}}\ ; \quad n = 1, 2, 3... \ , \tag{3}$$

where E is the Young's modulus of the rod material. This result, in general, is also independent of the cross-sectional shape of the rod as long as the initial assumptions ($\lambda >> d << L$, homogeneous, isotropic) are met. The dynamic Young's modulus of the bar can then be easily expressed in terms of its density, length, and the ratio of the frequency of the n-th mode of vibration to its mode number, n as follows:

$$E = 4\rho L^2\left(\frac{f^L{}_n}{n}\right)^2 . \tag{4}$$

Unlike the torsional and longitudinal modes, the flexural waves of the bar obey a fourth-order differential equation and the flexural wave phase speed, c_F, is dispersive. The application of free-free boundary condition for flexural vibrations leads to a series of modes, $f^F{}_n$, given by

$$f^F{}_n = \frac{\pi B_n{}^2 c_F \kappa}{8L^2}\ ; \quad B_n = 3.0112,\ 4.9994,\ 7,\ 9,\ 11..., \tag{5}$$

This result is accurate at low frequencies where the effects of rotary inertia and shear deformations associated with the flexure can be neglected. The flexural wave phase speed, c_F, can be expressed as

$$c_F = \sqrt{2\pi f \kappa c_L}\ . \tag{6}$$

where κ, the radius of gyration, is given by

$$\kappa^2 = \left(\frac{1}{S}\right)\int z^2\, dS \tag{7}$$

Here S is the cross-sectional area of the rod and z is the distance of an element above the neutral axis in the direction of flexure. For a rod of circular cross-section $\kappa = d/4$. The Young's modulus of a bar in terms of its flexural resonances can be expressed as

$$E = \frac{1024}{\pi^2}\frac{\rho L^4}{d^2}\left(\frac{f^F{}_n}{B_n{}^2}\right)^2 . \tag{8}$$

The redundancy provided by the fact that Young's modulus can be determined by the measurement of either the longitudinal or flexural modes provides a self-consistency check on the results and extends the frequency range over which the Young's modulus can be determined. This is a result of the flexural resonances occurring at frequencies which are typically an order-of-magnitude lower than the longitudinal resonances.

In using the ideal free-free boundary condition, the theoretical predictions do not take into account the added mass of the transducers which are placed on the bar to excite and detect the characteristic bar motion. However, the added mass is generally a small effect, typically a few percent of the entire mass of the rod. Also, the additional mass loading can easily be accounted for by the introduction of an effective length, L_{eff}, which provides a first-order correction if accuracy of greater than a few per cent is required. For the longitudinal and flexural modes, the additional mass loading of the transducers at the end of the bar has the same effect as lengthening the bar in the ratio of M : M + dM, where M is the mass of the "bare" rod and dM is the added mass of the transducer coils and their adhesive [10,7]. For the torsional mode, the effective correction is twice as large since the coils, mounted on the surface of the rod, make a proportionately greater contribution to the moment of inertia [7].

Selective Excitation and Detection of the Resonant Modes

The simplicity, speed, and economy of the free-free resonance bar method of modulus measurement is due to the ability to selectively and strongly excite the three resonant modes independently using the same inexpensive transducers. An electrodynamic transduction scheme is used to detect and excite all three modes using a pair of continuous coils of wire attached to each end of the sample bar. The sample is placed in various orientations within the field of a permanent magnet in order to selectively excite a particular mode. A typical apparatus for making these measurements is pictured in Figure 1, which shows the two magnets into which the coils are placed and an adjustable support structure in the foreground. The electronic interface (driver and signal amplifiers) is shown in the background.

The direction of the resulting differential Lorentz force, $\vec{dF}$, produced on a segment of wire, $\vec{dl}$, carrying a current, I, in a static magnetic field, $\vec{B}$, is given by

$$\vec{dF} = I\,\vec{dl} \times \vec{B}. \qquad (9)$$

It is the cleaver placement of the transducer wire coils, themselves attached to the bar, that allows for the excitation of one particular mode of vibration. Figure 2 illustrates the arrangement of the magnet and coil for excitation and transduction of the torsional modes. Here, the bar is placed with its axis at the center of the pole-piece faces which are aligned along the bar axis. The normal to the plane of coil is perpendicular to the magnetic field lines.

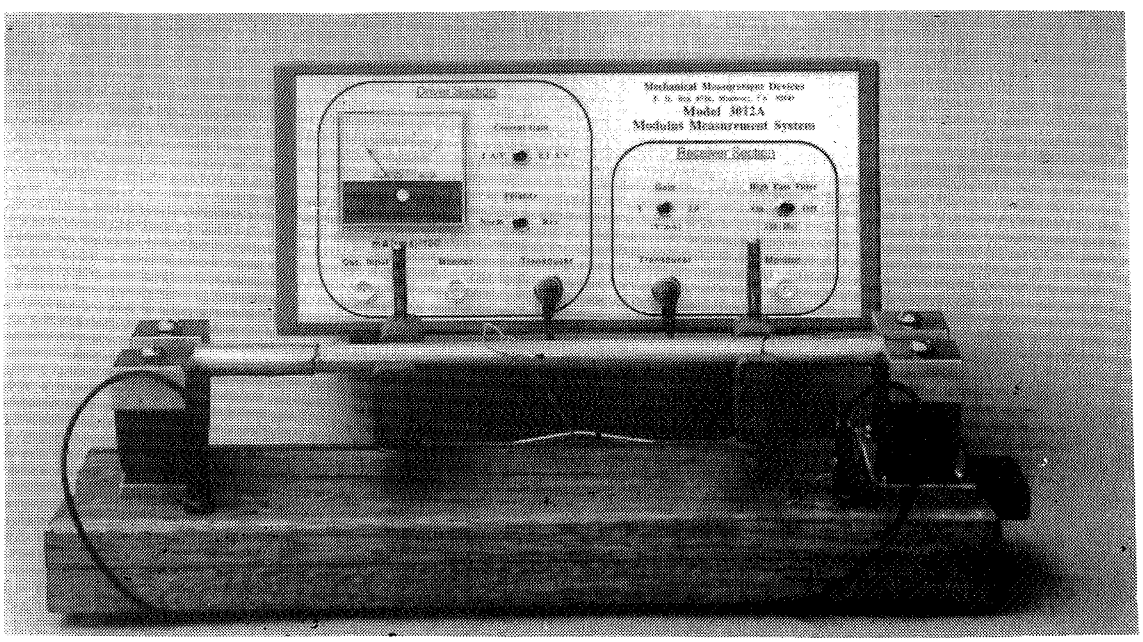

Figure 1. A measurement apparatus [11] used to electrodynamically excite and detect the flexural, longitudinal, and torsional modes of a free-free bar for determination of the shear and Young's elastic moduli.

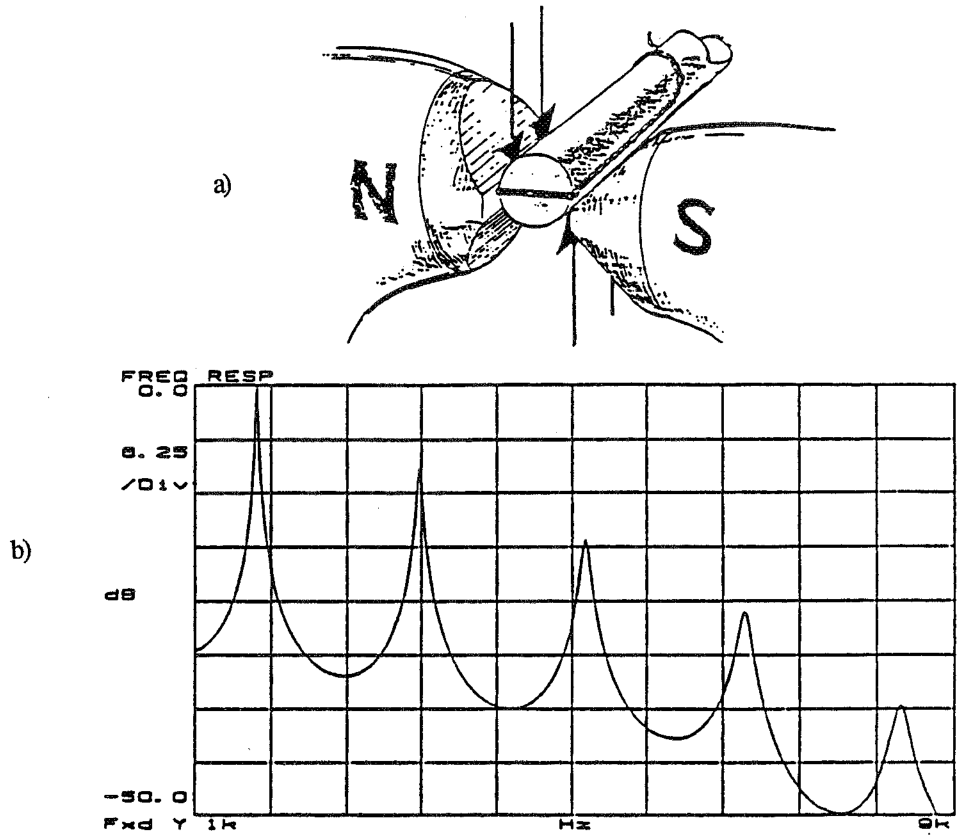

Figure 2. Torsional mode coil orientation and frequency response. (a) Orientation of the coils in the gap of the magnet pole pieces used to excite and detect the torsional mode. The arrows indicate the direction of the electromagnetic torque on the coil for a particular phase of the alternating electric current. (b) Frequency response output (log amplitude vs. frequency) for the five lowest torsional modes displayed on a HP 3562A Dual Channel Dynamic Signal Analyzer for an epoxy sample E-CAST™ F-28.

At the opposite end of the free-free bar, the receiver generates its "emf" as a result of the plane of the coil "rocking" back and forth in a region of strong, fairly uniform magnetic field. This change in angle modulates the flux through the coil since the projection of the area which the coil presents in the direction of the magnetic field is varying harmonically in time. Mathematically, we can express this induced voltage as

$$V = -\frac{d}{dt}\int_S \vec{B}\cdot\vec{n}\,dA \ , \tag{10}$$

where dA is in an incremental area subtended by the wire loop. For a small segment of wire moving with velocity, $\vec{u}$, in a magnetic field, $\vec{B}$, the induced emf is given by

$$\mathrm{emf} = \vec{B}\cdot\vec{l}\times\vec{u} \tag{11}$$

The flexural mode can be observed by rotating the bar by 90° and translating it up or down by a distance approximately equal to the diameter of the bar. This places one of the long coil sections near the top of the rectangular pole faces while the other section is above (or below) the pole face and hence in a position of weaker magnetic field. The difference in the two opposing forces (the gradient) causes the bar to flex. Likewise, the receiver coil is then raised and lowered through the field gradient inducing a change in the flux through the coil and generating an emf.

For transduction of the longitudinal mode, the separation between the two magnet structures at either end of the bar is increased so that the strong region of magnetic field is concentrated primarily near the short section of coil which crosses the end of the bar along its diameter. The currents in the short section of the coil generate longitudinal forces on the end of the bar which excite the longitudinal resonance modes. Similarly, at the receiver, the coil is moved in and out of the strong field region by the wave-induced motion which generates the observed emf.

VISCOELASTICITY AND CONTINUOUS TRACKING OF THE STORAGE AND LOSS MODULUS

Dynamic (Storage) Modulus as a Function of Temperature

The dynamic modulus of elasticity of materials is important to engineers and scientists. These properties depend dramatically on temperature and it is their temperature dependance that often becomes critically important. The resonant bar technique described in this paper can be used for tracking both the storage and loss components of either the complex dynamic shear or Young's modulus. The tracking of the storage modulus is accomplished by tracking a particular resonant mode with a phase-locked-loop (PLL). The PLL is used to keep the measurement system "closed" and locked on resonance as the temperature of the sample is changed. If the modulus is a function of temperature, the resonance frequency of the bar will necessarily change. Figure 3 is a block diagram of the typical instrumentation used for such a automated tracking system.

After identifying the modes of the bar at room temperature a particular mode is selected for automatic tracking as a function of temperature. One adjusts the voltage controlled oscillator (VCO) manually to resonance with the error signal feedback path open so that there is no error signal presented to the voltage control (feedback) input. The phase shifter is then adjusted so that there is zero output from the quadrature signal channel of the lock-in amplifier at the preliminary setup temperature. As the temperature is then changed, a shift in the resonance frequency of the bar occurs, and the lock-in amplifier registers a quadrature output voltage that is used as a feedback signal to adjust the VCO. The quadrature signal changes the frequency output of the VCO to the same frequency and phase of the new resonance of the bar. It is this VCO output frequency that is proportional to the square of the appropriate modulus. The resonance frequency can be read from a suitable "bus compatible" VCO or frequency counter and the temperature of the samples with a small thermistor attached to a "bus compatible" multimeter so that the entire set of temperature and frequency measurement data can be acquired, displayed, and analyzed by computer. A program which controls the acquisition, analysis, and display of the data using an HP 9836 computer is included as an appendix to Reference [12] with modifications to track the loss modulus included in an appendix to Reference [9]. A sample data set for PR1592 is provided in Figure 4.

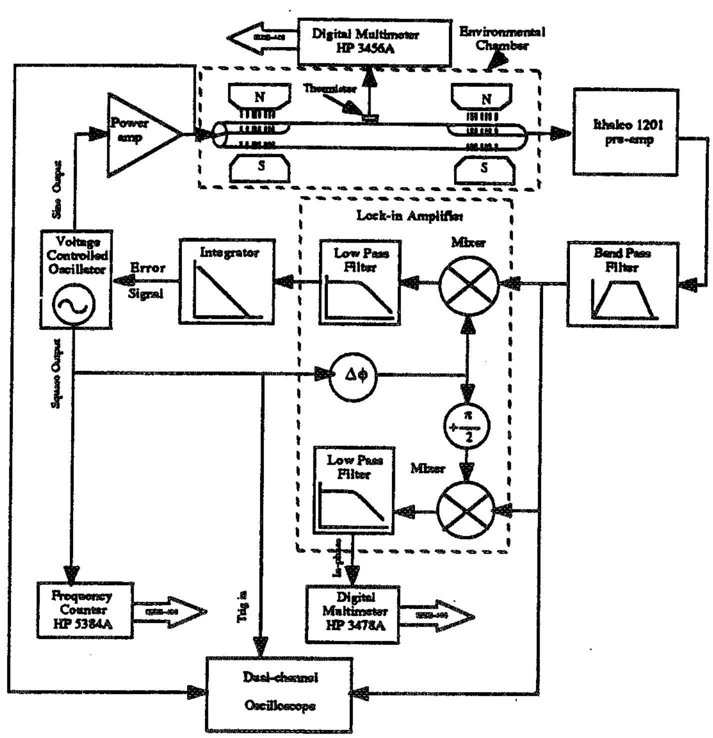

Figure 3. Block diagram of the phase-lock-loop used to automatically track the change in the resonance frequency (and thus modulus) of a particular mode as a function of temperature. The change in the loss tangent as a function of temperature can also be tracked by monitoring the in-phase output of the (lock-in) detector.

Dynamic Loss Tangent as a Function of Temperature

The loss tangent is proportional to the ratio of the energy dissipated in a material to the energy stored in the material in a given cycle of applied stress. The loss tangent is also equal to the inverse of the quality factor, Q, for a particular vibrational resonance. The temperature dependance of the loss tangent is obtained by measuring the in-phase voltage from the lock-in analyzer. This is possible since the response (in-phase voltage measured by the lock-in) of the vibrating rod at resonance is proportional to the Q of the system. Thus, by locking into a resonance as explained in the previous section by using the quadrature signal output of the PLL as a feedback signal to a VCO, the system stays on resonance independent of the temperature. The in-phase voltage is then monitored as a function of changing temperature to yield the temperature dependance of the Q. The inverse of the in-phase voltage is proportional to the loss tangent. The measurement of Q introduces additional experimental complications since care must be taken to insure that losses through the suspension system used to support the free-free bar are not significant compared to those intrinsic to the sample material under study. Because the strain distribution is known for each mode, these suspension losses can usually be reduced to insignificant levels since the two support points for the bar can be adjusted to occur arbitrarily close to velocity nodes.

Mathematically, the relationship between loss tangent and in-phase voltage can be shown through the derivation that follows which is based on a lumped oscillator model. The mechanical impedance of the bar at resonance is equal to the resistance of the system (by definition the reactance goes to zero at resonance) and can be expressed as

$$\frac{F}{u} = R\,, \tag{12}$$

where F is an applied force, R is the resistance, and u is the resulting velocity. The Q of the system can be expressed as

$$Q = \frac{\omega_o m}{R}\ , \tag{13}$$

where ω_o is the radian resonance frequency of the system, and m is the mass. Combining these two equations we can express the Q as

$$Q = \frac{u\omega_o m}{F}\ . \tag{14}$$

Now turning our attention back to the bar, suppose we drive the bar at resonance with a constant amplitude force, the ratio of the Q to the resonance frequency and velocity product remains constant, and equal to

$$\frac{Q}{u\omega_o} = \frac{m}{F}\ . \tag{15}$$

Recall that in the previous section the electrodynamic detection of the wire coil attached to the bar sample and suspended in the magnetic field, an emf was produced that is proportional to the velocity of the end of the bar. Letting V_{in} equal the in-phase component of the output voltage of the lock-in analyzer for a particular frequency and temperature, there is a constant of proportionality, A, that can be measured for the transduction scheme,

$$\frac{Q}{V_{in}\,\omega_o} = A\ . \tag{16}$$

Thus, as soon as this constant has been measured the loss tangent, tan δ, can then be determined as a function of the change in temperature and/or frequency of the sample,

$$\tan\delta = \frac{1}{Q} = \frac{1}{A_{in} V\,\omega_o} \tag{17}$$

The loss tangent can be measured directly at a particular frequency and temperature by any conventional method such as the 3dB down frequency technique or by measuring the relaxation time constant in a free decay mode. After having obtained the loss tangent directly the technique described above can be use to track the relative change in the modulus as a function of some external parameter variation such as temperature. A typical plot of the loss tangent is provided in Figure 4b. It is conventional [13] to plot the modulus and loss tangent as a function of reduced frequency $a_t f$ in order to obtain a master curve that illustrates the viscoelastic behavior of the material. Such a plot is provided in Figure 5.

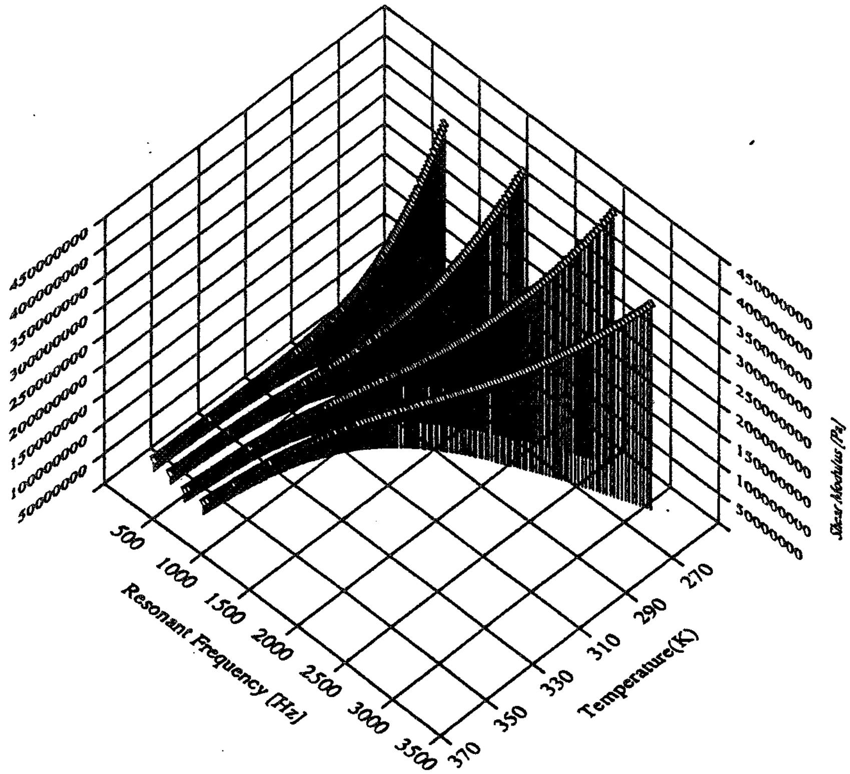

Figure 4a. Plot of the shear modulus of PR1592, as a function of temperature and frequency. Data was obtained for the shear modulus by tracking the first four torsional modes (separately) as the sample's temperature was changed. Only the first mode is shown for the loss tangent.

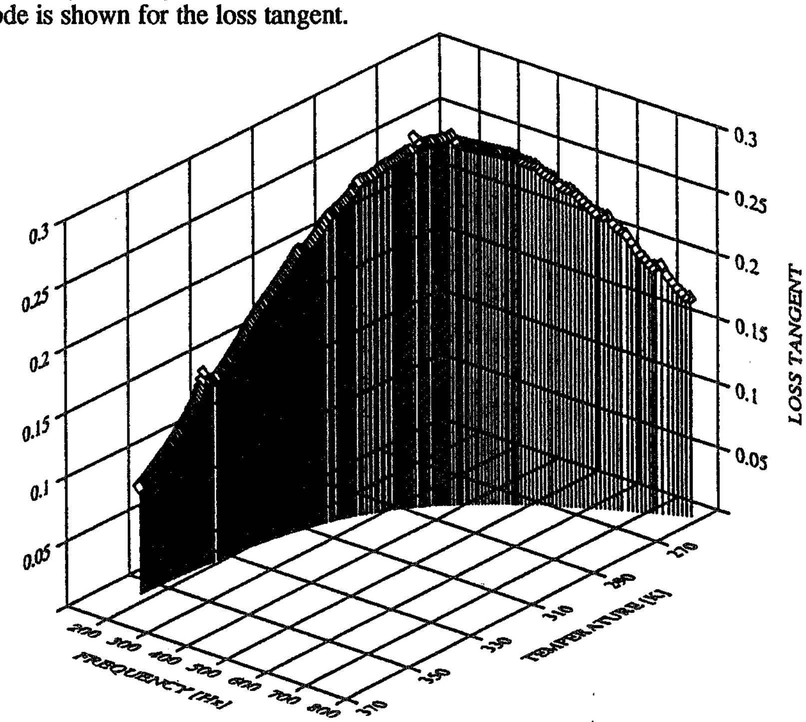

Figure 4b. Plot of the loss tangent of PR1592, as a function of temperature and frequency. Data was obtained as the sample's temperature was changed for the first torsional mode.

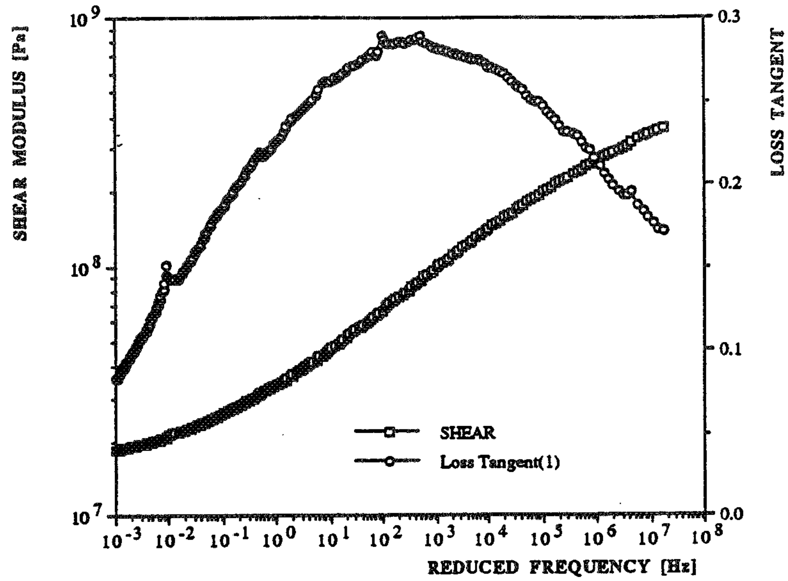

Figure 5. Master curve of loss tangent and storage shear modulus of PR1592 as a function of reduced frequency, $a_t f$, for a frequency shift parameter [13,14] $\log a_t = -12.9(T-283.15)/(107+T-283.15)$.

Comparison to the "Transfer Impedance" Technique

The advantages that this resonance bar technique has over the other conventional "transfer impedance" technique such as those discussed by Norris and Young [15] and Parsons, Yater, and Schloss [16] are briefly discussed here. The transfer impedance technique measures the amplitude and phase of the transfer function between typically two accelerometers mounted at either end of a vertically suspended rod-shaped sample which is driven from the upper end at fixed displacement by a shaker. One problem that this technique has is that the mass of the accelerometers is certainly not negligible as compared to the mass of the sample. What results is a difficult transcendental equation describing the motion of the bar. A computer is required to "invert" the frequency response information to account for this end-loading due to the accelerometer". In the Norris version, a Newton-Ralphson technique is used for this inversion. In both methods, transverse vibrations tend to be an undesirable problem and most importantly neither version of the transfer impedance method can measure the shear modulus. Thus, complete characterization of even the simplest materials, having only two independent moduli, is not possible. Other disadvantages include the fact that the sample must be clamped or otherwise bonded to the shaker at one end and the accelerometer must also be attached at the other end. Also, the shaker table and accelerometer is orders of magnitude more costly than the electrodynamic coil-magnet technique described in this paper.

Another important issue is reproducibility. The shaker table and accelerometer attachments can introduce gross ireproducibilities and misleading results due to the bonding and clamping of these fixtures. Since the cost of the accelerometers is not small, they are typically removed after each measurement. And, for accurate remeasurement one requires that the bonding be duplicated exactly. If one attempted to get a second independent moduli such as the shear modulus one would require, in addition to very accurate measurements, that the second (entirely different) apparatus would produce the same compensating error in order to obtain other elastic constants such as Poisson's ratio.

The transducers cost pennies and weigh less a penny (typically a few grams each, including the adhesive). Because the transducer is so cheap, there is no reason to remove them from samples hence re-testing, even years later, can resolve small or slow changes which would be masked by the remounting more massive accelerometers. Both the Young's and shear moduli can be measured using the same apparatus and the same pair of transducers. The ability to excite and detect both the longitudinal and flexural modes provides a redundancy in the determination on the Young's modulus that checks the method for self-consistency and/or extends the frequency range over which the measurement is possible. In addition to the storage modulus, the loss tangent is also easily accessible with this resonant bar technique.

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