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Automated measurement system for dynamic mechanical properties of viscoelastic materials

Edwin R. Fitzgerald and Roger E. Fitzgerald

Johns Hopkins University, Baltimore, MD 21218, USA

Summary

A method and automated system for measurements of complex dynamic shear compliance and modulus from 2 to 10,000 Hz, at temperatures between -50 and 150°C is described. Representative results for several materials at room temperatures and for a polyurethane rubber from 0 to 50° are presented to demonstrate the success of the measurement system in providing quantitative descriptions of dynamic mechanical behavior for viscoelastic materials.

Introduction

A rapid means for getting precise values of the complex shear compliance, $J^* = J' - iJ''$, (and/or modulus $G^*=1/J^*$) and its frequency dependence at various temperatures is needed for both using and understanding viscoelastic materials such as rubbers, plastics, and composites. These data are also necessary to establish the molecular and microstructural bases for the dynamic mechanical behavior of materials. In particular, the ability to get values of dynamic shear compliance over extended frequency ranges in short time intervals is of importance in studying polymerization and cross linking processes, and, in biomaterials, the effects of chemical or physiological processes on the mechanical properties of animal tissues (FITZGERALD, 1960, 1961, 1975, 1977; FITZGERALD, FREELAND, 1971). An electromagnetic transducer apparatus and forced vibration method for dynamic mechanical measurements has been described previously, but requires 5 to 10 minutes to get electrical impedance bridge balances and to record the data needed for calculations of complex compliance at each frequency in the range 10 to 5000 Hz at a temperature between -50 and 150°C. An additional 5 to 10 minutes is needed for calculations at each frequency (FITZGERALD, 1957; FITZGERALD, FERRY, 1953).

By contrast, the forced vibration measurement system described here, at any temperature within the same range, yields complex compliance and mechanical loss tangent (J''/J') values at 50 to 100 operator-selected frequencies between 2 and 10,000 Hz in 5 to 10 minutes. Linear or semi-logarithmic plots of complex compliance or loss tangent can be displayed on a monitor screen in less than a minute after the data have been taken; immediate evaluations of results are thus possible. Complete data print-outs and plots are also available within 2 to 5 minutes. Results at fewer frequencies in the 2 to 10,000 Hz range require less time; integration times for reliable results (±3%) at each frequency vary from 20 seconds below 10 Hz to only 3 seconds above 1000 Hz.

Measurement Method

The method is one in which two flat coils with their planes perpendicular are rigidly connected and suspended by fine wires so the coils can oscillate in their planar directions when placed in crossed magnetic fields of separate permanent magnet arrays. A diagram of the crossed coils with the magnetic fields indicated is presented in Figure 1. The coils are embedded and cemented in 1/8 in. thick plates of an insulating, thermal-setting material or "hardboard" which has beyond the coils an extension to which disk-shaped samples are clamped by moveable jaws of a sample holder as shown.

The coils 1, 2, with winding lengths ℓ_1, ℓ_2 are 1/16 in. thick x 7/8 in. wide x 1-1/4 in. long, and their opposite sides are separated by a 5/16 in. wide core. Small neodymium-iron-boron permanent magnets produce magnetic flux densities, B_1, B_2 of 6000 gauss in 3/16 in. gaps in which the coils are placed so that an alternating electrical current $\vec{I}_1$, through coil 1 produces an alternating force, in RMS vector notation, of $\vec{F} = B_1\ell_1\vec{I}_1$. In response to this force, the driving plate vibrates with a velocity, $\vec{v}$, of the same frequency, ν, as the current (and force), but with a phase shift. As a result, a motional emf is produced in coil 2 according to $\vec{E}_2 = B_2\ell_2\vec{v}$; because of their perpendicular and symmetric arrangement the non-motional, inductive coupling between coils is very small. Then expressions for the driving plate-sample system mechanical admittance, $\vec{Y}_M \equiv \vec{v}/\vec{F}$, or the inverse, mechanical impedance, $\vec{Z}_M \equiv \vec{F}/\vec{v}$, can be given in terms of an electrical transfer impedance, $\vec{Z}_{12}$, or an electrical transfer admittance, $\vec{Y}_{12}$ respectively.
That is,

$$\vec{Y}_M \equiv \vec{v}/\vec{F} = (1/K^2)\vec{E}_2/\vec{I}_1 = (1/K^2)\vec{Z}_{12} \quad \ldots\ldots\ldots (1)$$

$$\vec{Z}_M \equiv \vec{F}/\vec{v} = K^2\,\vec{I}_1/\vec{E}_2 = K^2\vec{Y}_{12} \quad \ldots\ldots\ldots (2)$$

where $K^2 = B_1\ell_1B_2\ell_2$ is the electromagnetic transducer constant.

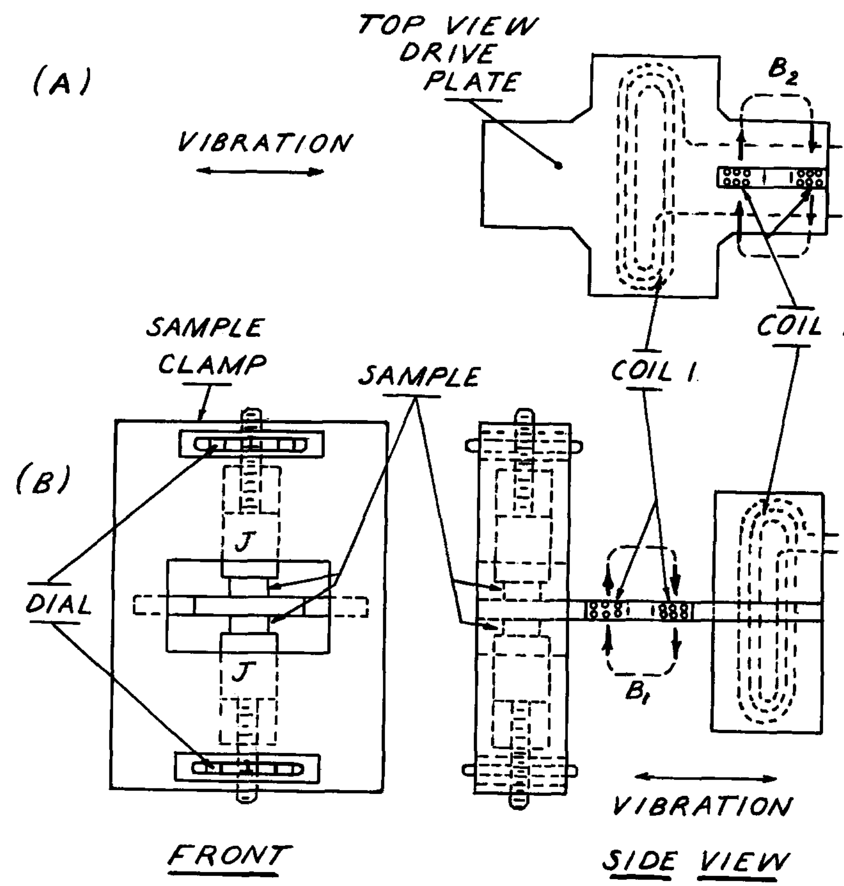

Figure 1 (A) Top view of drive plate and (B) front and side views of sample clamp and drive plate. Sample clamp jaws, J, are moved in or out by graduated captive dials threaded on the screws; force coil 1 and velocity coil 2 are shown schematically by dashed lines. Magnetic fields of permanent magnets (not shown) are indicated by paths B_1, B_2. The drive plate is suspended by fine wires (not shown) so that it can vibrate as noted when alternating currents are passed through the force coil 1.

With samples pressed against it, the measured mechanical impedance, $\vec{Z}_M$, of the driving plate consists of a sample impedance, $\vec{Z}_{Ms}$, in a series with an intrinsic driving plate impedance, $\vec{Z}_{Mp}$. Therefore, it is necessary to find calibration values of mechanical impedance for the driving plate alone and the corresponding values of electrical transfer admittance, $Y^o_{12} = G^o_{12} - iB^o_{12}$, at each frequency in order to get the sample mechanical impedance

$$\vec{Z}_{Ms} = \vec{Z}_M - \vec{Z}_{Mp} = K^2\{(G_{12} - G^o_{12}) - i(B_{12} - B^o_{12})\} \quad ..(3)$$

Values of the transducer constant, K^2, are found from equating the reactive parts of the driving plate mechanical impedance without samples and the corresponding electrical transfer admittance after multiplying both by $\omega = 2\pi\nu$, viz (cf Eq. 1),

$$\omega B^o_{12} = -S_M/K^2 + (m/K^2)\omega^2 \quad \ldots\ldots\ldots\ldots..(4)$$

where m = 33.67 grams is the driving plate mass and S_M is the mechanical elastance of its support wires. A plot of ωB^o_{12} vs ω^2 is a straight line of slope m/K^2 and ωB^o_{12} intercept, $-S_M/K^2$, so that K^2 is determined from measured values of B^o_{12} vs frequency at any temperature.

From the mechanical admittance, $\vec{Y}_{Ms}$, the complex shear compliance is found in terms of the total cross-sectional area, A, of the sample pair, and thickness, h, of each,

$$(A/h)\vec{Y}_{Ms} = \frac{d(\vec{x}/h)/dt}{\vec{F}/A} = \frac{d\vec{a}/dt}{\vec{s}} \equiv \mu^* \quad \ldots\ldots.(5)$$

where $\vec{a}$ is the shear strain produced by displacement, $\vec{x}$, of the inner face of a sample relative to its outer face, and $\vec{s}$ is the shear stress on the sample pair. The ratio of rate of shear strain to shear stress defines a complex shear fluidity (reciprocal complex viscosity), μ^*, as indicated. For sinusoidal stress and strain of frequency, ν, the complex shear compliance is then,

$$J^* = J' - iJ'' = -i\mu^*/2\pi\nu \quad \ldots\ldots\ldots\ldots.(6)$$

Data Collection and Calculations

A function diagram of the measurement system is adduced in Figure 2. A Solartron model 1250 two-channel frequency response analyzer generates, at frequencies from 1 to 10,000 Hz, the sinusoidal driving current, $\vec{I}_1$, in coil 1 of the electromagnetic transducer through a standard resistance, R_c, of 100 ohms in the resistor box. At each frequency, the complex ratio of the voltage $\vec{E}_2$, in coil 2 of the transducer to the voltage, $\vec{I}_1 R_c$, is also measured by the frequency analyzer; these data are collected, stored and used for calculations of mechanical impedance and complex shear compliance by the computer control which consists of a Zenith Z 158 computer and monitor with IEEE 488 controller and temperature measurement boards.

The data printer is an Okidata 192 printer while the data plotter is a Hewlett Packard 7470A plotter. Software programs provide for calibrations of the driving plate and filing of these data; with calibration data filed, sample measurements are automatically made on command at operator-selected frequencey intervals, source voltage amplitudes, and integration times. Values of the electrical transfer impedance, $\tilde{Z}_{12}$, proportional to the total mechanical admittance of the transducer-sample system are displayed on the monitor as they are found at each frequency. The temperature of the chamber and the sample clamp are also shown during the measurements, and the beginning and ending times and temperatures for each run are printed on the data sheets. Operating instructions are displayed on the monitor as needed.

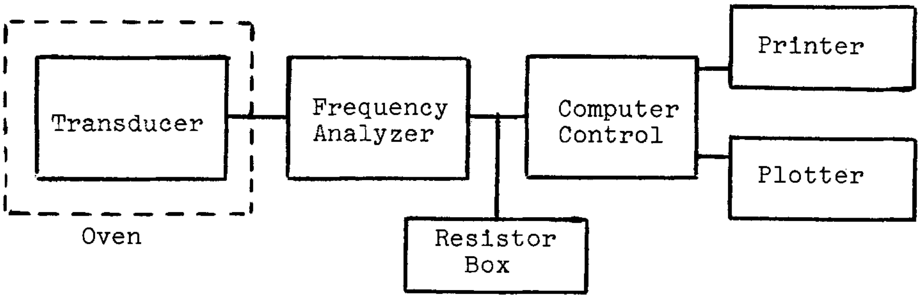

Figure 2 Functional diagram of the measurement system.

Some Experimental Results

In order to demonstrate the results that can be obtained with this system, a comparison of the frequency dependence of elastic shear compliance, J', and mechanical loss tangent, J"/J', for various materials at room temperatures is presented in Figure 3. The effect of temperature on the elastic and loss components of dynamic shear compliance, J', J", for a polyurethane rubber is shown in Figure 4.

Detailed discussions of these and other data will follow in full-length journal articles, but they are useful to illustrate both the advantages and limitations of this measurement method. Because the samples are measured in shear while held between parallel, plane surfaces, a wide range of solids, gels, and even viscous liquids can be measured; the samples do not have to be stiff or self-supporting beams, plates, or rods. On the other hand, the sample mechanical impedance is found by subtracting the driving plate impedance from the total

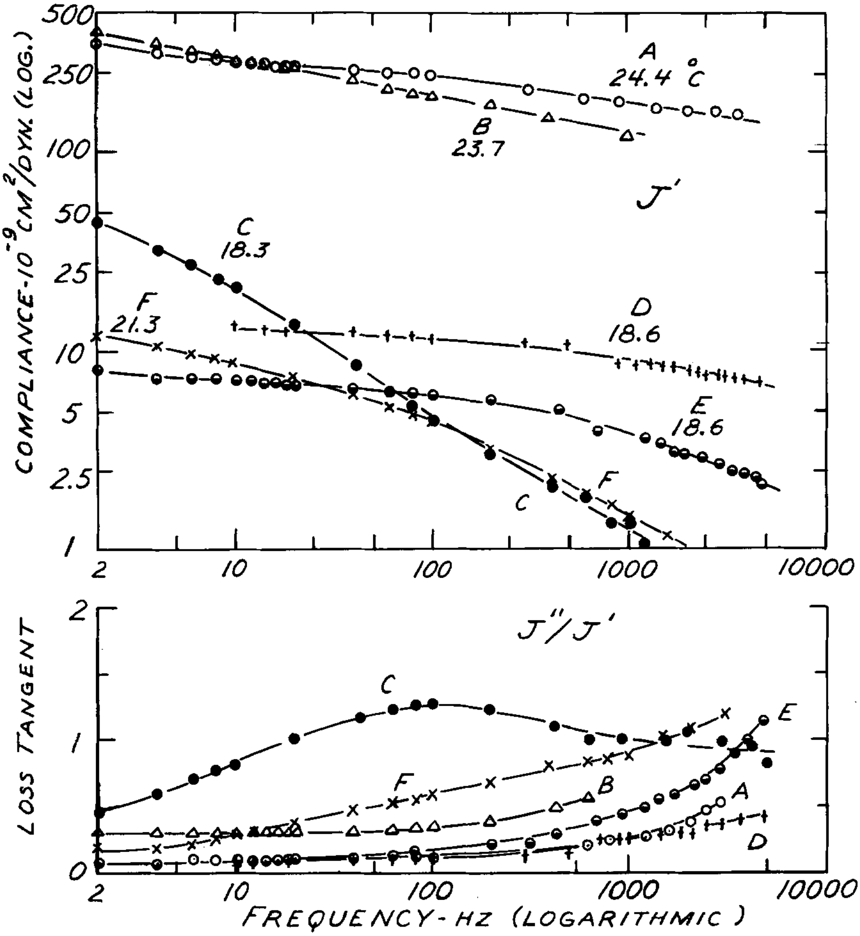

Figure 3 Logarithmic frequency dependence of elastic shear compliance, J', and mechanical loss tangent, J"/J', at temperatures indicated for (A) natural rubber (Hevea) gum stock, (B) swiss cheese (Kraft), (C) a polyurethane rubber (3% rebound), (D) a synthetic rubber tire stock (SBR, 80 parts carbon black), (E) a piezoelectric rubber (PZT-filled neoprene), and (F) a butadiene-acrylonitrile-styrene terpolymer (Hycar 1072 with 10 parts carbon black). The sample shape constants, A/h, varied from 92.5 cm. for (A) to 2.7 cm. for (F) as discussed in the text.

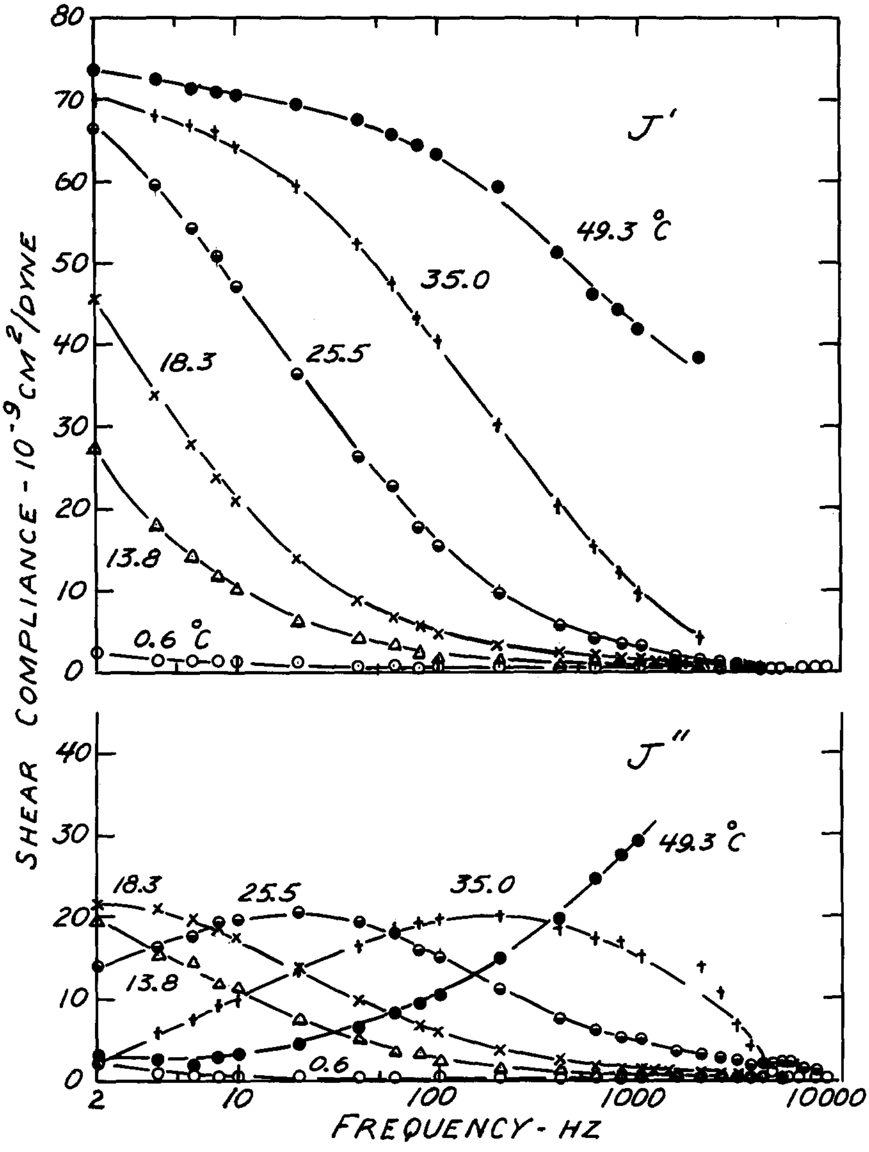

Figure 4 Frequency dependence of elastic (J') and loss (J") compliances for a polyurethane rubber between 0 and 50°C as indicated. For the sample shape constant (A/h = 8.0 cm) reliable measurements at 49 and 35°C extend only to 1500 Hz, but at 13.8 and 0.6°C results are valid to 8500 and 9500 Hz (cf text).

measured mechanical impedance, and when this difference, for a "soft" sample, is small the results become unreliable. Conversely, a very stiff sample will effectively clamp the driving plate, reduce its velocity and the motional emf in coil 2 so that the values of electrical transfer impedance, $\vec{Z}_{12}$, become too small for accurate measurement. These limitations can be extended by varying the sample disk dimensions to give A/h values from 2 to 200 cm, for example, but limit somewhat the useful compliance and frequency ranges for any particular sample pair.

Conclusions

An automated measurement system has been developed and used to give rapid, precise values of the dynamic complex shear compliance in the range from 2 to 10,000 Hz and between -50 and 150°C as shown by results obtained for various rubbers, plastics, and other materials.

Acknowledgements

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