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A Vibrating Reed Apparatus for Measuring the Dynamic Mechanical Properties of Polymers

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I. INTRODUCTION

Forced resonance vibrations are commonly used for the measurement of dynamic properties of viscoelastic bodies. The vibrating reed technique which involves steady state response of a reed strained in flexure (or tension) appears to be a particularly simple method when one is primarily interested in the temperature dependence of dynamic modulus. It should be kept in mind that the instrument allows one to calculate the dynamic Young's modulus in the direction of the long axis of the sample. The method consists essentially of a determination of the amplitude response of the free end of a cantilever specimen subjected to a small sinusoidal displacement of the clamped end.

The vibration is usually excited electromagnetically. The amplitude may be measured by visual observation with a microscope[1,2] or from an oscilloscope trace produced by a phototube activated by a beam of light interrupted by the moving sample.[3,4] Capacitance[5] and inductance[6] methods have also been employed. Use of a microscope, however, is tedious and slow; photoelectric devices require that the sample be enclosed in glass vessels to permit light transmission. Capacitance and inductance techniques, where developed, have generally required that relatively massive metallic objects be affixed to some part of the specimen. Such inertial masses require that large corrections be applied to the observations unless the specimen is made correspondingly large. In addition, convenient means for controlling the temperature of both sample and detection device over wide temperature ranges do not seem to have been developed.

In the apparatus described below, a capacitance method is used in such a manner that the specimen may be placed in conventional temperature-controlled chambers operating over a wide range of temperatures. In addition, the technique requires that only a negligibly small amount of metal be added to the specimen, even for relatively thin samples.

II. DESIGN OF THE APPARATUS

The electromechanical vibrating unit (Fig. 1) is an adapted phonograph speaker and consists of a wire coil on a paper cylinder free to move inside of a permanent magnet. An audio oscilllator (Hewlitt-Packard Model 200J) which can deliver signals in the frequency range of 6–6000 cycles/sec. drives the coil. To increase the signal strength to the coil, it was found necessary to use an amplifier (General Radio Type 1206B) and appropriate matching transformer.

The coil in turn drives a rigid member suspended at its ends by two strips of spring steel. Projecting down from the rigid member is a long rod

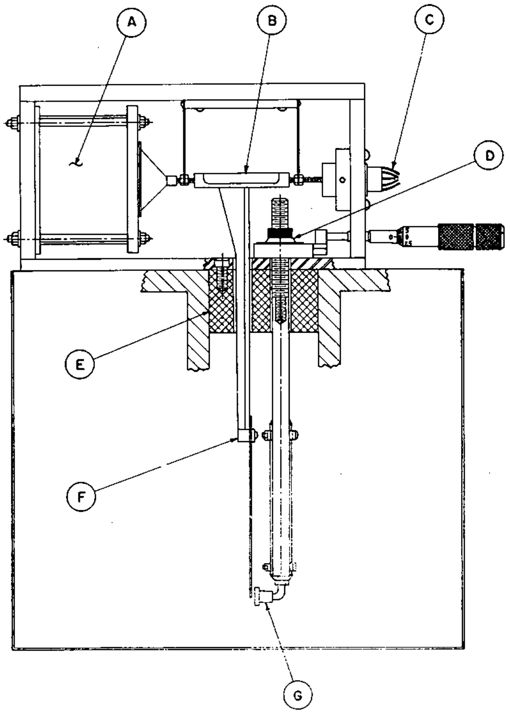

Fig. 1. Schematic of vibrating reed apparatus. Salient features are (*A*) adapted phono speaker, (*B*) horizontal driver, (*C*) linear variable differential transformer, (*D*) screw-driven slide for electrode (*G*), (*E*) transite block, (*F*) clamp for reed.

which terminates in a clamp. In order to achieve maximum rigidity, both of these members were fabricated as a single unit and have the shape of a "T" beam. The design of the clamp support permits insertion of the clamp and specimen in small temperature-controlled chambers and prevents the propagation of spurious motions which would otherwise result from the flexure of less rigid components.

Extremely small movements of the free end of the reed are converted into changes of electrical capacity by measurement of the change of electrostatic field around a fixed electrode. Capacity changes are detected by using a sensitive a.c. capacitance-bridge instrument (Fielden Proximity Meter Capacitance Gauge, Series 95). To increase the magnitude of the effect, a small amount of conductive silver is painted on the end of the reed and on an edge so that it becomes grounded in the clamp. A milligram of silver has been found adequate to provide high sensitivity; smaller amounts may be used. Comparative tests carried out without the silver and by measuring amplitude with a microscope indicate that this low mass of silver has a negligible effect on the calculated modulus and damping when the specimen weight exceeds about 150 mg.

The wave form of the capacitance change due to motion of the free end of the reed may be displayed directly on an oscilloscope for which high impedance terminals on the instrument are provided. Alternatively, one may use an indicating meter provided with the instrument. Except at the lowest frequencies, the meter provides a static reading proportional to the capacitance change after the instrument is balanced to a null condition with the reed at rest.

The stationary probe or electrode is connected through a double shielded cable to one side of the capacitance bridge. Since the cable insulation is the only heat-sensitive part of the apparatus, an all-Teflon-insulated cable has been fabricated to assure adequate heat stability when the apparatus is immersed in an oven at temperatures above 100°C.

III. EXPERIMENTAL PROCEDURE

The polymer specimen is cut so as to have a rectangular cross section of about 0.3 in. in width and a thickness in the range of 0.005–0.05 in. A length is chosen according to the particular frequency range desired, generally 1–3 in. The reed is mounted vertically and the electrode face adjusted to be directly opposite the tip of the reed which has been previously painted with conductive silver. A distance is chosen which gives the desired sensitivity of amplitude detection. The sensitivity of the proximity meter depends on the gain of the instrument. As an illustration: with an initial spacing of 0.05 in., maximum gain and an area of 1 in.^{2}, full-scale displacement is obtained on the meter with 100 microinch displacement of the reed from its rest position.

At an appropriate gain setting and after being balanced to a null condition at some initial spacing, e.g., 0.05 in., the audio oscillator frequency is adjusted until maximum reed amplitude or capacitance change is noted; it is necessary, of course, to identify the fundamental mode of vibration. The real part of the dynamic Young's modulus, E', may then be calculated directly with the following expression:[1,7]

$$E' = (48\pi^2/a_0^4)(\rho l^4/t^2)f_r^2$$

where ρ is the density of the sample, l is length, t is the thickness of the specimen in the plane of the vibration, f_r is the resonance frequency, and a_0 is a constant depending on the mode of the vibration. For the fundamental vibration $a_0 = 1.875$.

It is important that the specimen possess uniform thickness. However, in preparing polymer specimens by molding, a wedge effect is often encountered, the thickness gradually changing from one end to the other. Where the effect is small, a more accurate value of E' is obtained if one uses the average square of the thickness in the region of the clamp where the greatest flexure occurs rather than over the entire sample. In this manner the values obtained are independent of whether the thick or thin end of the reed is placed in the clamp.

The damping may be calculated[2] from the frequency values at which the amplitude falls to half of its maximum value. Unfortunately, with the capacitance method, capacitance change is inversely proportional to distance. Nevertheless, there are a number of avenues available to facilitate determination of the half-amplitude frequency. The most precise, of course, is to calibrate the reed amplitude, that is, to determine the capacitance change as the distance of reed to probe is varied. However, a simpler but less accurate method, described below, is available for rapid measurements.

In Figure 2 we have plotted empirical values of the capacitance change when sample to electrode

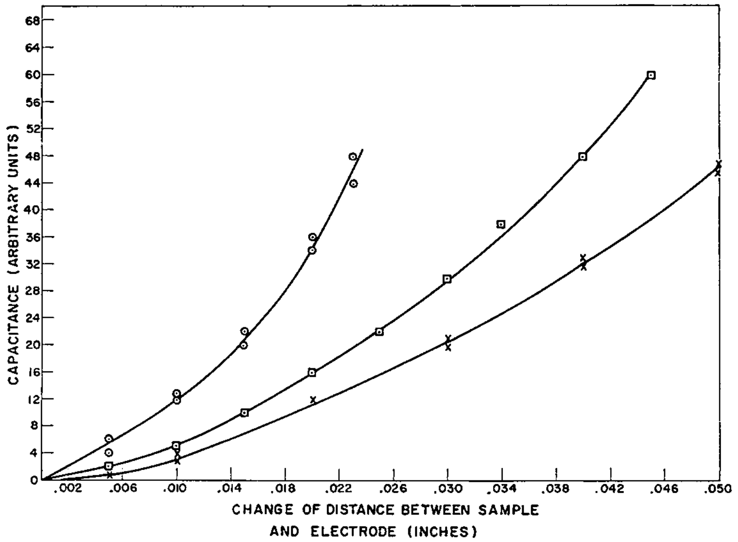

Fig. 2. Plot of capacitance change vs. distance moved for three different null conditions: (⊙) $D_0 = 0.025$; (⊡) $D_0 = 0.050$; (X) $D_0 = 0.065$.

distance is changed from an initial balance distance, D_0. In the neighborhood of distances $(D_0/2)$ where the reed has moved a distance ΔD from its initial position, it is seen that a capacitance decrease of two thirds corresponds closely to a change of distance equivalent to $\Delta D/2$. In practice, therefore, one need only adjust the resonance amplitude so that the tip of the reed traverses about one half the initial sample-electrode spacing and then adjust the oscillator frequency until the meter reading (proportional to capacitance) decreases to one third its value at the resonance amplitude. These frequencies represent the half-amplitude frequencies. While the calibration in Figure 2 was obtained statically, it is applicable to dynamic measurements, since similar curves were obtained when a microscope was used to measure vibration amplitude; further, the meter reading is found to be proportional to the peak amplitude of the capacitance change as measured with an oscilloscope.

The quantity $(f_2 - f_1)/f_r$, where f_2 and f_1 are the half-amplitude frequencies, is equal to $\sqrt{3}E''/E'$, where E'' is the imaginary part of the complex modulus.

Since the apparatus itself possesses resonance conditions which depend on the characteristics of the coil, rigidity of the springs, inertial effects, and so forth, it is occasionally necessary, particularly when near the apparatus resonance frequencies or when measuring materials of high damping over a wide band width, to adjust the driver amplitude so as to make it independent of frequency. For this purpose, a linear variable differential transformer (LVDT) has been located at the end of the rigid member with the core of the transformer fixed to the driver rod. We have used a 1000-cycle/sec. input to the primary of the transformer

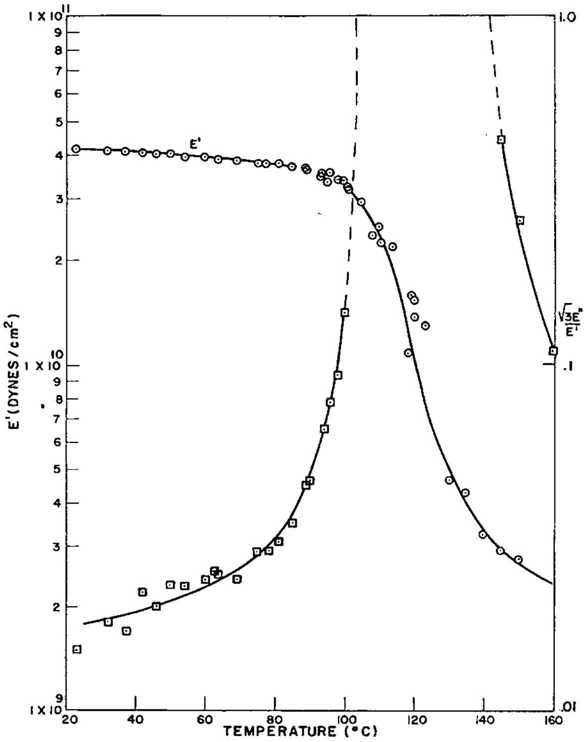

Fig. 3. Plot of (⊙) dynamic Young's modulus and (⊡) damping vs. temperature for crystalline polystyrene.

and followed the output with a vacuum-tube voltmeter. The amplitude of the driver may be held constant by manually adjusting the gain of the oscillator so as to maintain a constant output voltage of the LVDT. In the instrument described here, apparatus resonances occurred in the regions of 12 and 200 cycles/sec., thereby leaving a wide frequency range over which little difficulty as regards the variation of driver amplitude with frequency was experienced.

The clamp assembly and capacitance pick-up may be set down into chambers with diameters as small as 2.0 in. A Transite block through which the clamp support rod passes serves to close off the upper portion of the chamber. The oven may be of metallic construction without interfering with the capacitance pick-up. For our initial studies we have used a chamber operable from −50 to +300°C. whose construction has already been described.[8]

Thermocouples placed at different locations in the oven indicated that there was a uniform temperature over the length of the sample, except that at elevated temperatures the clamp was a few degrees cooler, indicating heat loss up the clamp support rod. We presume that the sample may experience a very steep temperature gradient over a short portion of its length at the entrance to the clamp. No simple means are available to correct this situation except to immerse a larger portion of the clamp support in the oven than our oven allowed. In practice, we have not found this situation serious and have located a thermocouple at a fixed position from clamp and sample in order to obtain consistent results from one sample to another.

A typical result for the dependence of the dynamic modulus obtained on a sample of crystalline polystyrene over a frequency range of about 120–30 cycles/sec. is shown in Figure 3.

When the damping is low, we have found it possible to obtain values of E' which agree within about 3% for replicate samples. The major source of error arises from the length measurement. With increased damping, the error may be larger due to the difficulty of locating f_r. Calculating the mechanical loss from the difference $(f_2 - f_1)$ invariably leads to about 10% error when the difference is of the order of 1 cycle/sec. With increasing damping, the reproducibility goes through a maximum. At the condition, $(f_2 - f_1)/f_r \simeq 1.0$, the band width and relatively low reed amplitude relative to the driver amplitude, together with other factors, make it practically impossible to obtain the damping during routine measurements.

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Synopsis

A vibrating reed apparatus for the measurement of dynamic Young's modulus has been constructed. The apparatus utilizes (*a*) a sensitive capacitance pick-up to measure displacement of a reed having approximately 0.001 g. of silver painted on the free end of the reed, (*b*) a clamp assembly which permits placement of the specimen in small temperature-controlled chambers, and (*c*) a linear variable differential transformer to facilitate maintenance of constant amplitude (independent of frequency) of the clamped end of the reed.

Résumé

Un appareil à lame vibrante pour la mesure des modules dynamiques de Young a été construit. L'appareil utilise (*a*) une capacité sensible de pick-up, pour mesurer le déplacement de la lame avec 0,001 gr d'argent point sur l'extrémité libre de la lame, (*b*) un système d'attache qui permet de placer l'échantillon dans une petite chambre à température controlée, et (*c*) un transformateur linéaire à différentiel variable pour faciliter le maintien d'une amplitude constante (indépendante de la fréquence) de l'extrémité attachée de la lame.

Zusammenfassung

Es wurde eine Vibrationsapparatur zur Messung des dynamischen Young-Moduls gebaut. Der Apparat verwendet (*a*) eine empfindliche Kapazitätsanzeige zur Messung der Verschiebung eines Stäbchens, auf dessen freies Ende eine Menge von ungefähr 0,001 g Silber aufgebracht ist, (*b*) ein System von Klammern, das die Anbringung der Probe in kleinen, temperaturgeregelten Kammern gestattet, und (*c*) einen linear variierbaren Umwandler zur Erleichterung des Aufrechterhaltens einer konstanten Amplitude (unabhängig von der Frequenz) des festgeklammerten Endes der Probe.

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