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Apparatus for Torsional Braid Analysis by a Digital Data Analyzer

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Synopsis

As apparatus for torsional braid analysis (TBA), which is a variant of the torsional pendulum technique, a new method for recording the damped wave electrically, using an electric meter as a transducer, is described. In order to process a large volume of data, an instrument to print out digitally the amplitude and period of oscillation has been developed. The thermomechanical behavior of poly(vinyl chloride) is presented so as to demonstrate the use of the present TBA apparatus.

INTRODUCTION

Torsional braid analysis (TBA), a variant of the torsional pendulum technique, has been widely used for thermal analysis of polymers developed by Lewis and Gillham.[1]

In traditional torsional pendulum or TBA techniques, the amplitude and period of oscillation were measured using a stopwatch and graduated scale, but this method is obsolete and limited in accuracy, so that now various other methods for recording the damped wave are under consideration. For example, Gillham[2] used a circular glass disc on which chromium was deposited by vacuum evaporation, and recorded the damped wave with a vacuum phototube and light source circuits. Bender[3] directed ultraviolet light onto a mirror under an inertia bar and which in turn was reflected to UV light-sensitive paper for recording the damped wave. Takahashi[4] used a differential transformer, and Gillham[5] used a pair of polarizers as the transducer to record the damped wave. However, even by these recording methods, considerable data must be processed manually.

Recently, Bell, Gillham, and Benci[6] have described a fully automated TBA apparatus interfaced with a data analyzer to print out digitally logarithmic decrement and period of the oscillation. Hazony, Stadnicki, and Gillham[7] also describe computerized TBA experiments for fully automated data acquisition, reduction, presentation, and data processing. Dalal et al.[9] developed a TBA apparatus with automated data acquisition/reduction which provides low-scatter, high-resolution data. Takahashi[4] also developed a fully automated TBA apparatus using an analog computer.

The present paper describes the TBA apparatus using an electric meter as transducer to record the damped wave of oscillation and also describes a data analyzer to print out digitally the amplitude and period of the damped wave.

The thermomechanical behavior of poly(vinyl chloride) (PVC) is presented so as to demonstrate the use of the instrument.

INSTRUMENTATION

Auto Recording of Damped Wave

In order to record the wave of the damped oscillation, an electric meter such as a voltmeter, an ammeter, or a volt–ohm–milliammeter can be used as a transducer. In the present work, the volt–ohm–milliammeter is used as the transducer.

The schematic diagram of the TBA apparatus is shown in Figure 1. From a rod extending under an inertia mass, a lever protrudes and holds the indicator of the meter whose coil is rotating in constant magnetic field. Asymmetry of the protruded lever shown in Figure 1 is impossible, since the weight of the lever (0.45 g) is smaller than that of the inertia (60 g), so that unwanted irregular oscillation does not occur.

The electromotive force, which is generated by rotating the coil in the constant magnetic field accompanied by torsional oscillation of the specimen, is amplified and recorded on a strip chart recorder as successive waves of oscillation, shown in Figure 3.

Figure 2 shows that a linear relationship exists between the angular displacement and the analog output from the transducer. With this type of the transducer, the mechanical oscillation of the specimen cannot be recorded directly,

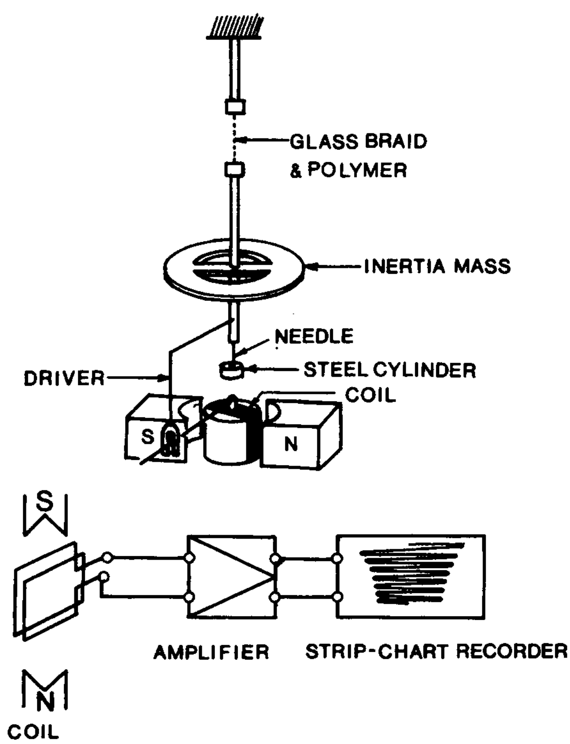

Fig. 1. Schematic diagram of TBA apparatus.

TABLE I
Period (T) and Logarithmic Decrement (Δ) for a Steel Wire 0.2 mm in Diameter and 100 mm in Length, Obtained from a Stopwatch and Graduated Scale, and Analog Output[a]

Experimental no.	A		B	
	Δ	T	Δ	T
1	0.01	2.67	0.01	2.48
2	0.01	2.43	0.01	2.48
3	0.01	2.43	0.01	2.48

[a] A: Values obtained from a stopwatch and graduated scale; B: values obtained from recorded wave.

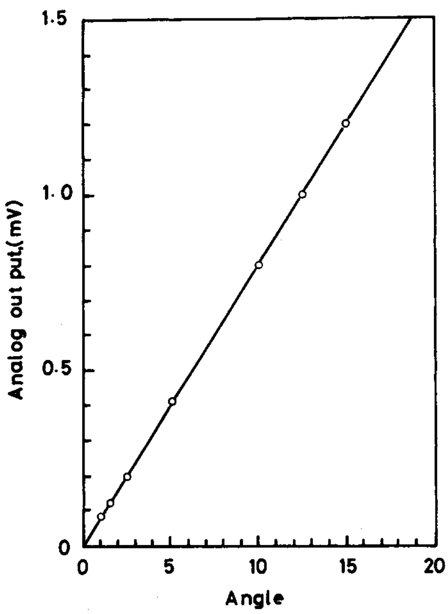

Fig. 2. Relationship between angular displacement and analog out-put.

but its derivative which corresponds to the angular velocity is recorded on a strip chart recorder. However, the damped wave and its derivative have identical logarithmic decrements and periods of oscillation as shown in the Appendix.

The logarithmic decrement and period for a steel wire 0.2 mm in diameter and 100 mm in length are listed in Table I for comparing the wave of mechanical oscillation obtained from a stopwatch and graduated scale technique with the derivative wave of mechanical oscillation obtained from the present apparatus. The values of (A) are well in agreement with those of (B).

The rest position on a strip chart recorder never changes if the rest position of the specimen changes during the experiment, since the angular velocity of the mechanical oscillation is measured.

To prevent unwanted irregular oscillation which occurs at the initial twist of the specimen, a needle 0.6 mm in diameter is attached to the lower end of the extended rod under an inertia mass and inserted into a steel cylinder of 1 mm inside diameter, so that the irregular oscillation can be damped out immediately.

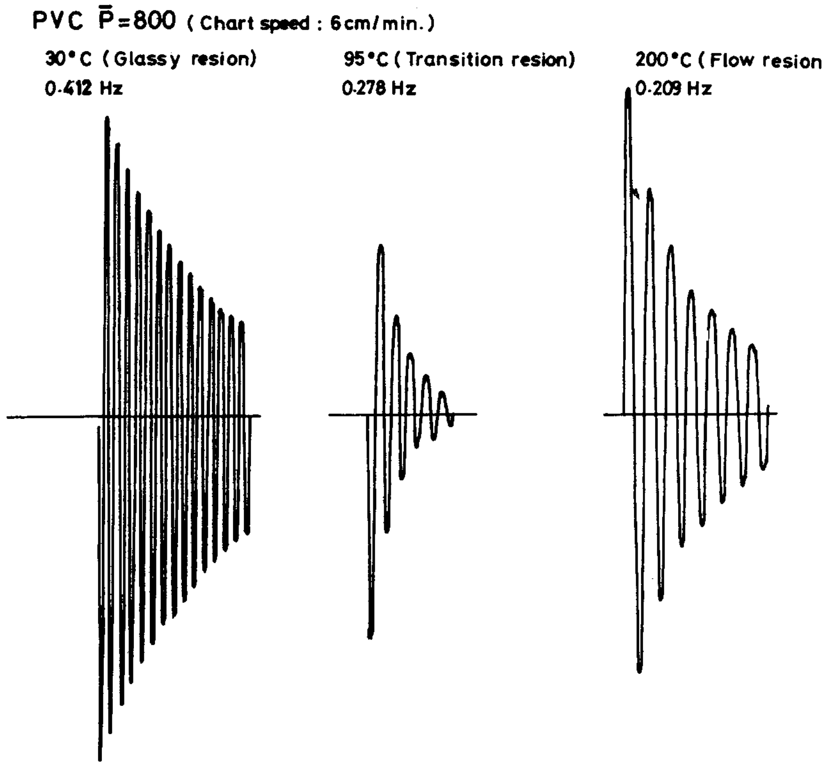

Fig. 3. Characteristic output wave for poly(vinyl chloride).

The needle must be set at the center of the cylinder, so that additional damping accompanying friction between the inner wall of the cylinder and surface of the needle can be avoided completely.

Characteristic output waves for a composite specimen of PVC and glass braid at 30°C (in glassy state), 95°C (in transition region), and 200°C (in liquid flow region) are shown in Figure 3.

Digital Representation of Amplitude and Period

To record the damped wave on a strip chart recorder to obtain accurate results is convenient but slow for determining the rigidity and logarithmic decrement for each damped wave, as large amounts of data must be processed to determine the thermomechanical spectra of polymers over a wide range of temperatures.

The digital data analyzer described in this section is such that each amplitude of successive oscillations is measured, converted to a digital signal, indicated by a digital voltmeter, and printed out by a digital printer. The total time required for measuring the amplitude by a preset number of cycles is also measured and printed out.

The block diagram and timing chart of the apparatus are shown in Figure 4. Generated voltage on both ends of the electric meter transducer accompanying the torsional oscillation of the specimen is dc amplified and sent to a level comparator. In the level comparator, the fed voltage level is inspected for adequacy and whether plus or minus, and then the result is sent to a controlling circuit. After the starting signal is sent, the peak holding command, the reading com-

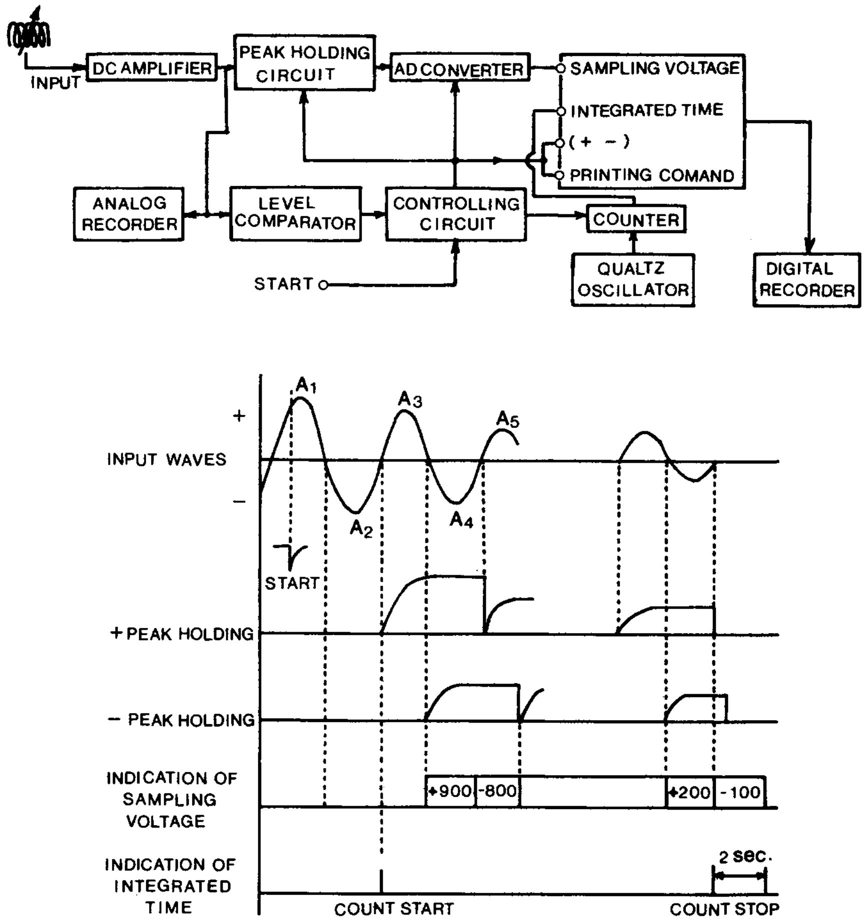

Fig. 4. Block diagram and timing chart of a digital data analyzer.

mand, and the gate opening command are sent to a peak holding circuit from the controlling circuit, and each sampling voltage (corresponding to amplitudes) is indicated by a digital voltmeter, whereupon the printing command is immediately sent to a digital printer which prints out each amplitude by the preset number of cycles. At the same time, the total time between measuring the amplitude by the present number of cycles is computed accurately by a counter with a quartz oscillator and indicated by a digital voltmeter, as well as printed out digitally. The analog output from the dc amplifier is connected to a strip chart recorder while monitoring analog output waves, and the starting button is pushed at the right moment, whereupon the analyzer commences to measure and print out the amplitude and the total time.

From the digital printed amplitude A_1, A_2, A_3, ... A_n and total time, the logarithmic decrement Δ and the relative modulus G_r for the composite specimen are calculated as follows:

$$\Delta = \ln(A_1/A_3) = \ln(A_3/A_5) = \ldots \quad \text{for plus wave} \tag{1}$$

$$\Delta = \ln(A_2/A_4) = \ln(A_4/A_6) = \ldots \quad \text{for minus wave} \tag{2}$$

$$G_r = (T_0/T)^2 \tag{3}$$

where numerical subscripts to A show plus amplitude for an odd number and minus amplitude for an even number, T is the period of oscillation converted by dividing the total time by the preset number of cycles, and the subscript 0 to T shows the period at the initial stage.

In the present experiment, Δ and G_r must still be calculated by manual data reduction techniques; but with the present digital data analyzer, all computations are fully automated.

APPLICATION

The thermomechanical behavior of a composite specimen of glass braid and PVC and the thermogravimetry (TG) curve for bulk PVC are shown in Figure 5.

The composite specimen was prepared from about a 10% solution of PVC of which the extent of polymerization was 800 in tetrahydrofuran, and the solvent was removed sufficiently by heating for three days at 80°C in vacuo (10^{-5} mm Hg). The clamp-to-clamp distance of the specimen was 8.0 cm. The rate of heating for TBA and TG was 1°C/min.

The decrease in relative modulus G_r and the peak of logarithmic decrement Δ at about 90°C occurs at the glass transition of PVC. In the vicinity of 200°C, a minimum in G_r and a peak in Δ occur, and this process seems to be associated with the melting transition in which the molecular chain entanglement becomes disconnected, or with the melting transition of the crystallite contained in the polymer. At about 210° and 260°C, G_r shows two stages of increase. The first gradual increase in G_r at about 210°C is concerned with the stiffening process of the molecular chain accompanied by the formation of double bonds owing to dehydrochlorination of the PVC, and at this temperature the TG curve shows a marked change. The second apparent increase in G_r at about 260°C is concerned with the crosslinking reaction among the double bonds formed by dehydrochlorination.

A peak in Δ at 295°C is concerned with the loss of mechanical energy when the polymer undergoes a rheological transition from the liquid state to the glassy state passing through a viscoelastic region during the process of the crosslinking reaction.

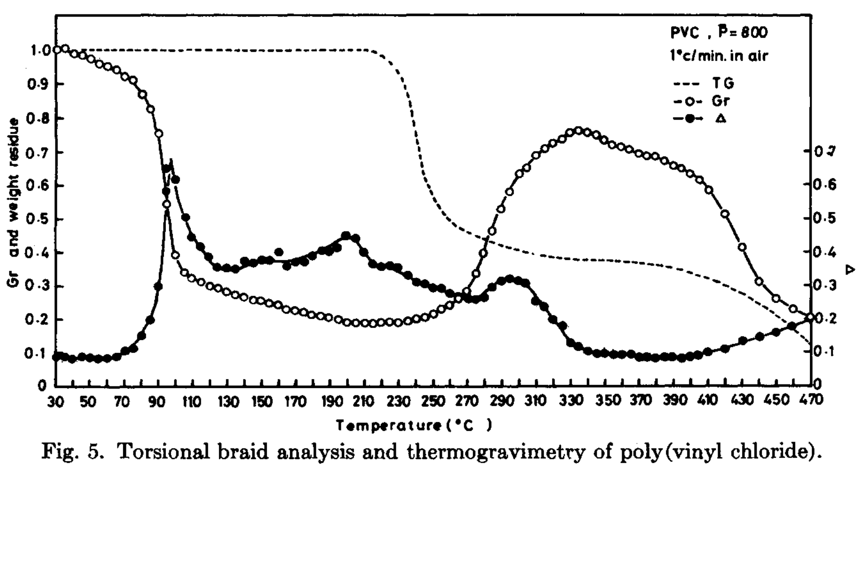

Fig. 5. Torsional braid analysis and thermogravimetry of poly(vinyl chloride).

At 330°C, G_r decreases gradually, then sharply drops at 410°C. These processes reflect respectively the formation of low molecular substance accompanied by chemical rupture of crosslinked networks and the vaporization of low molecular substance from the glass braid.

Appendix

In general, the differential equation for motion in free vibration is given by

$$I(d^2\theta/dt^2) + (\eta'/K)(d\theta/dt) + G'\theta/K = 0 \tag{A-1}$$

where I is the moment of inertia, η' is the dynamic viscosity, G' is the dynamic modulus, θ is the angle of displacement, and K is the geometrical shape factor of the specimen.

The solution of eq. (A-1) is

$$\theta = \{A \cos(\omega t) + B \sin(\omega t)\} \exp(-\epsilon t) \tag{A-2}$$

with

$$\epsilon = \eta'/2IK$$

where A and B are constants, ω is the frequency, and ϵ is the damping coefficient.

The derivative of eq. (A-2) is

$$\dot{\theta} = \{(B\omega - A\epsilon)\cos(\omega t) - (A\omega + B\epsilon)\sin(\omega t)\} \exp(-\epsilon t). \tag{A-3}$$

From eqs. (A-2) and (A-3), eqs. (A-4) and (A-5) are obtained, since $\theta = \theta_0$ and $\dot{\theta} = 0$ at time $t = 0$:

$$\begin{aligned} \theta &= \theta_0\{\cos(\omega t) + (\epsilon/\omega)\sin(\omega t)\} \exp(-\epsilon t) \\ &= (n\theta_0/\omega)\cos(\omega t - \alpha)\exp(-\epsilon t) \end{aligned} \tag{A-4}$$

$$\dot{\theta} = -(n^2\theta_0/\omega)\sin(\omega t)\exp(-\epsilon t) \tag{A-5}$$

where $n^2 = \omega^2 + \epsilon^2$ and $\tan \alpha = \epsilon/\omega$.

The logarithmic decrement of mechanical oscillation (Δ) and that of the differentiated wave of the mechanical oscillation (Δ') are given as follows. In case of the wave of the mechanical oscillation,

$$\Delta = \ln(\theta_1/\theta_3) = \ln(\theta_3/\theta_5) = \ldots \text{ etc.} \tag{A-6}$$

$$\theta_1/\theta_3 = \frac{\theta_0\{\cos(\omega t) + (\epsilon/\omega)\sin(\omega t)\} \exp(-\epsilon t)}{\theta_0\{\cos \omega(t + T) + (\epsilon/\omega)\sin \omega(t + T)\} \exp\{-\epsilon(t + T)\}} = \exp(\epsilon T) \tag{A-7}$$

where T is the period of oscillation. Thus,

$$\Delta = \ln(\theta_1/\theta_3) = \ln(\theta_3/\theta_5) = \ldots = \epsilon T. \tag{A-8}$$

In case of the differentiated wave of mechanical oscillation,

$$\Delta' = \ln(\dot{\theta}_1/\dot{\theta}_3) = \ln(\dot{\theta}_3/\dot{\theta}_5) = \ldots \text{ etc.} \tag{A-9}$$

$$\dot{\theta}_1/\dot{\theta}_3 = \frac{-(n^2/\omega)\theta_0\sin(\omega t)\exp(-\epsilon t)}{-(n^2/\omega)\theta_0\sin \omega(t + T)\exp\{-\epsilon(t + T)\}} = \exp(\epsilon T). \tag{A-10}$$

From eqs. (A-9) and (A-10),

$$\Delta' = \epsilon T \equiv \Delta. \tag{A-11}$$

Consequently, the logarithmic decrement of mechanical oscillation is the same as that of the differentiated wave of mechanical oscillation which is recorded on a strip-chart recorder by using the present transducer. Similarly, the wave of mechanical oscillation and its derivative also have identical periods, since $\omega = 2\pi/T$.

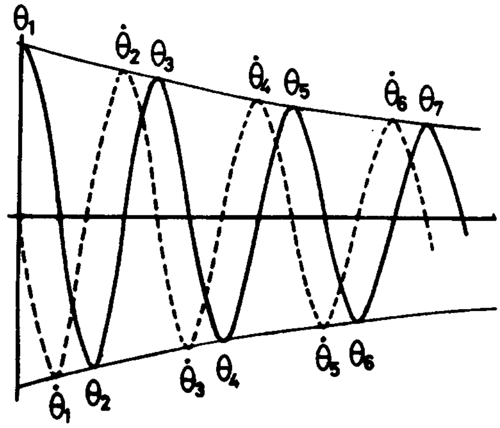

Fig. 6. Relationship between the waves of mechanical oscillation (solid line) and the differentiated waves.

The relation between the wave of mechanical oscillation and its derivative is shown in Figure 6. The differentiated wave (recorded wave) differs only in phase, but it has a logarithmic decrement and period identical to those of mechanical oscillation.

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