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DYNAMIC HARDNESS TESTER AND CURE METER

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ABSTRACT

The Shore hardness tester is used extensively throughout industry to determine the static modulus of materials. The new apparatus described here extends the capability of an indentortype tester into the dynamic regime, and provides a measurement of the dynamic shear orYoung's modulus and loss factor as a function of frequency. The instrument, model and data of typical rubber samples are given and compared to other dynamic measurements.

INTRODUCTION

The standard durometer has been used to measure the static "hardness" or resistance of materials to indentation. Various static models have been developed to quantify these measurements by relating the static Young's modulus and Poisson ratio of a viscoelastic material to the force of indentation, the penetration depth, and size (radius) of the indentor.

To our knowledge, an equivalent satisfactory dynamic model in which the indentation force is time dependent has not been developed analytically. Various empirical models, however, have been examined to model the interaction of transducers placed in contact with a viscoelastic slab to excite shear and compressional waves in the material. R. von Gierke[1] e.g. has produced such a model to examine the acoustic properties of living tissue. He assumes the transducer behaves as a hemispherical radiator whose diameter equals that of the cylindrical transducers used in his experiments. In this model, the radiation impedance of a vibrating sphere is related to the complex dynamical shear and compressional moduli of the material. This model, under certain approximations, can be used to infer the dynamic Young's modulus, E', and loss factor, δ. To perform these measurements the amplitude and phase of the driving force and acceleration of the transducer which is in contact with the material are measured.

We are examining the validity and applicability of such models experimentally by comparing the predicted results for E' and δ to those obtained by more conventional methods such as those developed by Madigosky and Lee[2],or other type of dynamic mechanical testing apparatus such as the dynamic mechanical thermal analyser (DMTA).

The goal of this study is to develop a model and simple apparatus which will allow a dynamical measurement of E' and δ of a material in a manufacturing environment.

THEORY

The theoretical model we will first examine for measuring the dynamic response of a viscoelastic material to intrusion by a perturbing force is that developed by von Gierke to examine the acoustic properties of living tissue. The theory considers the medium to be homogeneous, isotropic, compressible, and viscoelastic. The probe perturbing the medium is a cylindrical transducer placed in contact with the surface of the medium. The transducer-medium interaction is modeled as a hemispherical radiator whose diameter equals that of the transducer.

The complex impedance of the medium which is defined as the ratio of force over velocity is related analytically to the complex shear and compressional moduli of the material which are defined as:

$$\mu = \mu_1 + j\omega\mu_2 = \mu_1(1 + \delta_S) \qquad (1)$$

$$\lambda = \lambda_1 + j\omega\lambda_2 = \lambda_1(1 + \delta_L) \qquad (2)$$

where $\omega\mu_2/\mu_1 \equiv \delta_S$ is the shear loss factor and $\omega\lambda_2/\lambda_1 \equiv \delta_L$ is the compressional loss factor.

The Young's Modulus is related to the shear modulus by

$$E \equiv E'(1 + j\delta_S) \cong 3)\mu \qquad (3)$$

so that

$$E' \cong 3\mu_1 \qquad (4)$$

The radiation impedance Z_S of the sphere is

$$Z_S = -4\pi\rho\omega a^3 j/3 \cdot \{(1 - 3j/ah - 3/a^2h^2) - 2(j/ah + 1/a^2h^2) \cdot [3 - a^2k^2/(jak + 1)]\}/\{(1/a^2h^2 + j/ah)a^2k^2/(jak + 1) + [2 - a^2k^2(jak + 1)]\} \qquad (5)$$

where a = radius of the sphere,

$$k^2 = \rho\omega^2/(2\mu + \lambda) \qquad (6)$$

and

$$h^2 = \rho\omega^2/\mu \qquad (7)$$

We have examined several limiting cases of Z_S

a. $\lambda_1 \gg \mu_2, \omega\lambda_2, \lambda_2 \approx 0$

This limit is achieved when $ak \ll 1$, then

$$Z_S \rightarrow Z_2 \equiv -2\pi\rho\omega a^3 j/3 \cdot (1 - 9j/ah - 9/a^2h^2) \qquad (8)$$

b. high loss materials ($\omega\mu_2 \gg \mu_1$)

$$Z_S \rightarrow Z_3 \equiv -2\pi\rho\omega a^3 j/3 \cdot (1 + 9(\mu_2/(2\rho\omega a^3)^{1/2} - 9j(\mu_2/2\rho\omega a^2)^{1/2}) \cdot [1 + (2\mu_2/\rho\omega a^2)^{1/2}] \qquad (9)$$

c. low loss materials ($\omega\mu_2 \ll \mu_1$)

$$Z_S \rightarrow Z_4 \equiv -2\pi\rho\omega a^3 j/3 \cdot (\omega - 9\mu_1/\rho\omega a^2 - 9j[(\mu_1/\rho a^2)^{1/2} + \mu_2/a^2]) \quad (10)$$

Z_2 and Z_4 will be of primary interest for the materials with properties in the frequency range of interest (50 -1000 Hz).

A comparison of the real part of Z_S, Z_2 and Z_4 as a function of frequency when the sphere radius a = 1 mm shows that the formulas for Z_2 and Z_4 are in excellent agreement with Z_S over a wide frequency range, and diverge from Z_S only at very high frequency.

Z_4 since it is straightforward to interpret and use it as the basis for an experimental method to obtain μ_1 and μ_2.

The impedance Z_4 can be interpreted as that of a simple oscillator. To see this, consider the equation of a forced harmonic oscillator

$$m\ddot{x} + k^*x = F \quad (11)$$

Here, k^* is complex to account for the case of viscous damping. The impedance of the oscillator $Z \equiv F/\dot{x}$ can be obtained by assuming $x = x_0 e^{j\omega t}$ and $F = F_0 e^{j\omega t}$ so that $\dot{x} = j\omega x$ and

$$Z = mj(\omega - k'/\omega m - jk''/\omega m) \quad (12)$$

where $k^* = k' + jk''$

Comparing Z to the equation for Z_4 we see that Z_4 behaves like an oscillator with mass $\equiv m = 2\pi a^3 \rho/3$ (which is half the mass of a sphere), stiffness $\equiv k' = 6\pi a\mu_1$ and frictional resistance $\equiv k'' = 6\pi a^2 \rho \cdot [(\mu_1/\rho)^{1/2} + \mu_2/a\rho]$ as observed by von Gierke.

If then, a hemispheric impedance head of real mass M is placed in contact with the viscoelastic material, and it is assumed that the impedance of the material is ideally modeled by that of the radiating sphere model under the approximation $Z_S \approx Z_4$ for the frequency range of interest, the acceleration measured by such an impedance head is given by:

$$F/\ddot{x}_{measured} = M + Z_4/j\omega = (M + m) - k^*/\omega^2) \quad (13)$$

Then, in view of the interpretation of k' and k" given above, we may write:

$$k' = 6\pi a\mu_1 = \omega_2(M + m - \mathrm{Re}(F/\ddot{x}_{meas})) \quad (14)$$

$$k'' = 6\pi a^2 \rho = \omega^2 [(\mu_1/\rho)^{1/2} + \mu_2/a\rho] = \omega^2 m\, \mathrm{Im}(F/\ddot{x}_{meas}) \quad (15)$$

We can then use these equations to calculate $E' = 3\mu_1$ and $\delta = \omega\mu_2/\mu_1$.

Similarly the expression for Z_2 can be inverted to produce these values by solving for the variable $1/h$ in equation (8).

EXPERIMENT

We have performed experiments on a variety of viscoelastic materials to test the validity and applicability of the model given above.

To do this we employ a compact Wilcoxon piezoelectric shaker- impedance head to which we can attach indentors of various diameters, a Hewlett-Packard noise generator / spectrum analyzer which drives the shaker with white noise and measures the real and imaginary parts of the acceleration (amplitude and phase of the driving force). With the help of a computer, the complex impedance of the excited material is calculated and the real and imaginary parts are used to produce the Young's modulus and loss factor as described above.

Tables I. and II. show a comparison of experimental results on generic nitrile and urethane rubber samples using the method described above and those obtained using a dynamic mechnaical thermal analyser (DMTA) made by Polymer Laboratories. The DMTA was used to measure the Young's modulus and loss factor as a function of temperature using the frequencies 0.3, 1, 3, 10 and 30 Hz. The data was then shifted to the frequency domain by using the standard Williams-Landel-Ferry (WLF) technique.

Table I. Comparison of Results on Nitrile Rubber

	Dynamic Durometer Method		DMTA Method	
F(Hz)	E (m Pa)	Loss Factor	E (m Pa)	Loss Factor
50	4.3	0.3	7.5	0.2
100	5.3	0.47	7.7	0.25
200	7.0	0.34	7.9	0.3
300	7.5	0.31	8.5	0.33
400	7.8	0.25	8.9	0.35
500	8.1	0.27	9.5	0.4

Table II. Comparison of Results on Urethane Rubber

	Dynamic Durometer Method		DMTA Method	
F (Hz)	E (m Pa)	Loss Factor	E (m Pa)	Loss Factor
200	54	0.15	63	0.085
300	59	0.16	66	0.090
400	61	0.10	68	0.094
500	62	0.11	70	0.097
600	70	0.17	74	0.100

These results indicate the good agreement between the two types of measurement. Further studies are being done to test the effect of indentor surface area and the effect of static contact pressure of the indentor.

In addition to the dynamic modulus measurements on cured materials, a study of the change in dynamic modulus as a function of time was made on a two component polyurethane (Techthane 13, Seaward International) as i t changed from a viscous mixture to a cured solid. The results are given in Table III. A small disc, 0.8 cm diameter, was used. As can be seen from the data, this technique may be used to accurately determine the rate of cure and the state of cure in materials.

Table III. Dynamic Cure Test of Polyurethane

Time (hours)	Young's Modulus (kPa)	Loss Factor
1	4	1.7
2	51	0.9
3	320	0.68
5	610	0.52
6	770	0.42
7	850	0.41
19	1150	0.38
31	1620	0.37
43	1850	0.37
53	1890	0.36
94	1980	0.34
182 (8 days)	2150	0.33

Similar rate and state of cure measurements can be obtained on epoxies and vulcanized rubbers, thus replacing current methods which provide only relative data.

REFERENCES

1. H. von Gierke et. al., Physics of Vibrations in Living Tissues, J.Appld. Physiology, 4, p. 886-900, 1952.

2. W. Madigosky and G. Lee, Improved Resonance Technique for Materials Characterization, J. Acoust. Soc. Am. 73, p. 1374-77, 1985.