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DIRECT MEASUREMENT OF THE DYNAMIC MATERIAL PROPERTIES OF POLYMERS FOR LOW FREQUENCIES

Presenting Author

Paul J. Macioce
Anatrol Corporation

Co-Authors

Ahid D. Nashif, Tom M. Lewis
Anatrol Corporation

ABSTRACT

This paper documents recent advances relative to the characterization of the dynamic material properties of polymers over the frequency range of 1e-4 to 1000 Hz. Discussion of the technique is included as well as data taken on a familiar material. In addition, datasets have been generated for the above material using other techniques over a wide frequency and temperature range, and are presented in a reduced frequency format for correlation of this new technique with other methods.

Anatrol Corporation, 4120 Birch Street, Newport Beach, CA, 92660
Tel: (714) 660-7051

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I. INTRODUCTION

There are a number of measurement techniques which are currently used to obtain the dynamic properties of viscoelastic materials. It is commonly accepted that no one technique offers the range necessary to overcome the widely varying nature in material properties of viscoelastics. These properties vary as a function of such parameters as temperature, frequency, dynamic strain amplitude, and static preload for multiple states-of-stress. For this reason, multiple sources are needed to completely characterize a given material. In addition, confidence is established in the accuracy of the test data through correlation of the various techniques over a wide range of temperatures and frequencies. This in turn provides an assurance that the temperature-frequency superposition principle holds within the range of measurements.

Therefore, it is the goal of the data acquisition system to gather dynamic properties over as wide a test range as possible to establish an accurate characterization of materials with a high degree of confidence. The vibrating beam technique and the low-frequency impedance method, presented in this paper, allow for such a range in measurements.

It is the purpose of this paper to discuss the details of the low-frequency impedance technique developed by Anatrol Corporation. Though the development of this system is an ongoing process, this system is currently capable of performing direct material evaluations over a frequency range of 1e-4 to 100 Hz (6 decades of frequency coverage).

A brief description of the measurement techniques used to generate the material properties presented in this paper, including discussion of the merits and limitations of each, is provided with special emphasis on the impedance technique. To demonstrate the effectiveness of this new test system, measurements were made on the "Round Robin" TTPC material [1] using the vibrating beam technique to provide high frequency correlation. A sandwich-type beam specimen was used to obtain the shear properties over a temperature and frequency range of -70° to 150°F and 300 to 3000 Hz, respectively. Preliminary measurements using the impedance technique include data taken at 65°, 75°, and 85°F for frequencies ranging from 5e-4 to 100 Hz.

II. VIBRATING BEAM TECHNIQUE

The vibrating beam technique has become an industry standard for obtaining material properties of viscoelastics over a wide temperature and frequency range (ASTM E-756-80). This technique lends itself well to automated control, and is used to provide a quick characterization of a material with accurate results.

The vibrating beam technique allows for the use of a number of different types of test specimens to calculate either shear or Young's modulus. Some of these specimen configurations are presented in Figure 1.

The complete set of equations which govern this technique for the various configurations shown in Figure 1 can be found from many sources [2,3,6]. However, the sandwich beam equations used to generate the shear vibrating beam data are presented below:

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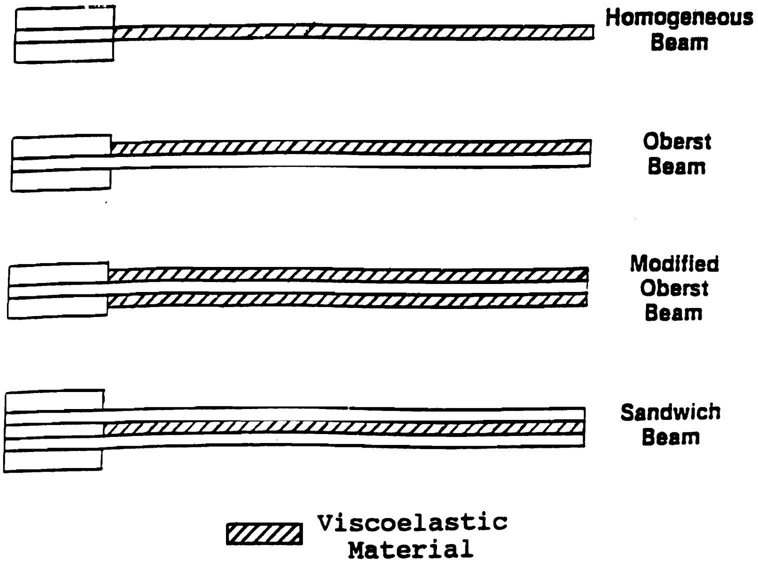

Figure 1: Various Test Specimen Configurations for the Vibrating Beam Technique

<u>Sandwich Beam</u>

$$G'(\omega) = \frac{(E_1 H_1 H_2 a_n)\left[(A - B) - 2(A - B)^2 - 2(A\eta_c)^2\right]}{L^2\left[(1 - 2A + 2B)^2 + 4(A\eta_c)^2\right]} \qquad (1)$$

$$\eta = \frac{A\eta_c}{A - B - 2(A - B)^2 - 2(A\eta_c)^2} \qquad (2)$$

where

$$A = \left(\frac{\omega_n}{\omega_{1n}}\right)^2\left(2 + \frac{\rho_2}{\rho_1}\frac{H_2}{H_1}\right)\left(\frac{B}{2}\right) \qquad (3)$$

$$B = \frac{1}{6\left(1 + \frac{H_2}{H_1}\right)^2} \qquad (4)$$

$$\eta_c = \frac{\Delta f_n}{f_n} \qquad (5)$$

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Though the vibrating beam method allows for measurements in both shear and tension/compression, it is extremely difficult to generate scatter-free data necessary to provide a precise estimate of Poisson's ratio. In addition, this technique is unable to monitor the effect of such things as dynamic non-linearities or static preload on the material's dynamic properties.

III IMPEDANCE TECHNIQUE

III.A Overview

The impedance (or forced-oscillation) technique is a direct measurement method which is not limited to the resonance of the test sample and therefore can make measurements over a wide temperature and frequency range. Of the many available systems, the test apparatus described in this paper was developed to provide measurements at extremely low frequencies. This system uses a single-degree-of-freedom model and operates at frequencies below resonance. As with all other techniques, the test apparatus is placed in an environmental chamber to monitor the effect of temperature. This system is currently able to perform automated measurements at frequencies from 1e-4 to 100 Hz and temperatures from -100° to 300°F.

Among the features of this technique, is that the effect of static preload and dynamic strain amplitude can be monitored. In addition, measurements can be made on the same sample in both shear and tension/compression states-of-stress.

III.B Test Specimens

The test specimen design used in the low frequency impedance device allows for measurements to be made in both shear and tension/compression on the same test sample in order to reduce the scatter associated with sample to sample variations. This is done by rotating the sample 90° in the fixture and applying the load from a perpendicular direction. A conceptual sketch of the test specimen is provided in Figure 2. Static preloading of the material in compression is provided by inserting spacers between the box frame of the specimen and the material sample as seen in Figure 2.

III.C Test Procedure

Once a test specimen has been fabricated, it is placed within the temperature controlled portion of the system and is oriented in a position to provide the desired state-of-stress. The box frame of the specimen is bonded in place, a temperature thermocouple is embedded within the viscoelastic material itself, and the center block spacer is attached to the mechanical actuator via a rigid stinger connection. The force and displacement transducers, as well as the actuator, are located outside of the environmental chamber. A sinusoidal input is applied to the specimen which is monitored with the force transducer connected in-line with the actuator. The resulting sinusoidal response of the sample is measured using a displacement transducer.

An FFT analyzer with programming capabilities is used to control the automated data acquisition. The user selects the frequency and temperature ranges to test over, as well as the dynamic strain level to input to the sample. The input force is varied to maintain this strain level for all measurements.

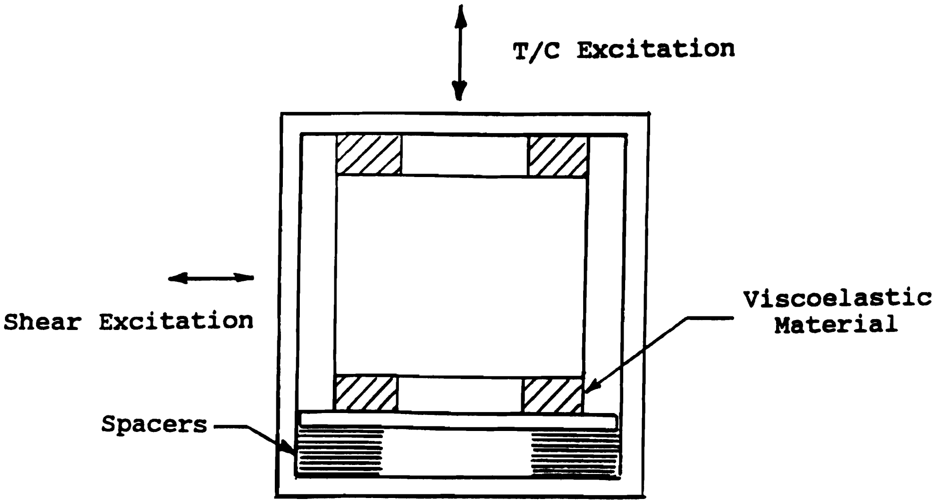

Figure 2: Schematic Drawing of the Test Specimen Used for the Impedance Technique

III.D Data Reduction

The impedance technique is based on a single-degree-of-freedom model in which a complex spring element is used. The imaginary portion of the modulus is calculated from the area within the hysteresis loop generated for the input stress on the sample versus the resulting strain of the material as a function of time. The specific equations used are given below:

Shear

$$G'(\omega) = \sqrt{\left(\frac{\sigma_o}{\epsilon_o}\right)^2 - G''(\omega)^2} + \frac{m\omega^2 t}{A_L} \qquad (6)$$

$$G''(\omega) = \frac{A_H}{\pi \epsilon_o^2} \qquad (7)$$

$$\eta = \frac{G''(\omega)}{G'(\omega)} \qquad (8)$$

Tension/Compression

$$E'(\omega) = \sqrt{\left(\frac{\sigma_o}{\epsilon_o}\right)^2 \frac{1}{(1 + \beta s^2)^2} - E''(\omega)^2} + \frac{m\omega^2 t}{A_L(1 + \beta s^2)} \qquad (9)$$

$$E''(\omega) = \frac{A_H}{\pi \epsilon_\circ^2} \tag{10}$$

$$\eta = \frac{E''(\omega)}{E'(\omega)} \tag{11}$$

III.E Precautions

As is the case with all test techniques, special care must be taken in fabricating the test sample, not only to ensure that proper adhesion is obtained, but also that the pad geometry of the sample is properly designed.

One limitation that is true with all impedance techniques is that testing can not be performed when the sample stiffness approaches the fixture stiffness. This happens at higher frequencies and cold temperatures, or when testing in tension/compression with pads having large shape factors. On the other end of the spectrum, if the test specimen is designed to be too weak, a poor signal-to-noise ratio problem will result with the force transducer when testing in shear at extremely low frequencies and high temperatures. Since this technique covers such a wide range of modulus values, it is difficult to design one sample that will provide clean and accurate data for both extremes. For this reason it may be necessary to design multiple samples of differing geometries.

However, a good rule of thumb is to define the maximum allowable stiffness of the test apparatus, and design the sample to approach this stiffness at the highest test frequency and coldest temperature desired. The vibrating beam technique can be used to provide initial dynamic modulus values needed to calculate the sample stiffness at a given temperature and frequency.

Once the sample has been designed, it is important to ensure that good adhesion is obtained throughout the data acquisition. For those materials which are not self-adhering, a thin layer of structural epoxy is used to bond the material in place without the adhesive "short circuiting" the viscoelastic links or contaminating the edge of the pad. It is favorable to cure the epoxy at room temperature when time will allow to ensure that the material's dynamic properties are not altered in any way due to exposure at high curing temperatures.

It is also good practice to perform measurements at room temperature three times during the testing process; once at the beginning of the test, and again after both the lowest and highest test temperatures. If the same material properties are not obtained after the cold temperature leg of the testing, it is a good indication that the sample has become disbonded. If differences are seen after high temperature testing, it is likely that the material has been heat aged, and irreversible damage has been done to the sample.

In addition to the above precautions, it is important to ensure that proper alignment of the sample with the stinger is achieved, and that all unwanted static preloads are removed before testing is commenced. To obtain accurate results, the sample should be allowed much time to reach an equilibrium temperature, and that the sample is allowed several loading cycles to reach a steady-state condition. An additional concern is to be aware of where the sample resonance occurs and to restrict testing to frequencies well away from it.

IV. PRESENTATION OF DATA

To demonstrate the effectiveness of this technique, a familiar material was selected, namely the "Round Robin" TTPC [1], and tested over the entire range of frequencies currently available. Measurements were restricted to only a few temperatures, emphasizing the advantage of a wide frequency testing capability. To supplement this low frequency data, the vibrating beam technique was used over a wide temperature range.

Figure 3 represents a plot of temperature versus frequency for the two datasets showing the range in measurements conducted. It can be seen from this plot that the combined dataset spans 7 decades of frequency coverage over a temperature range of -70° to 155°F. Since the beam test is restricted to the linear region of the material, measurements made using the impedance technique were also tested in the linear range. To ensure that this was the case, a linearity check of the material was performed by measuring the dynamic properties for various dynamic strain levels input to the sample. Figures 4 and 5 are the direct shear storage modulus and loss factor values as a function of strain amplitude.

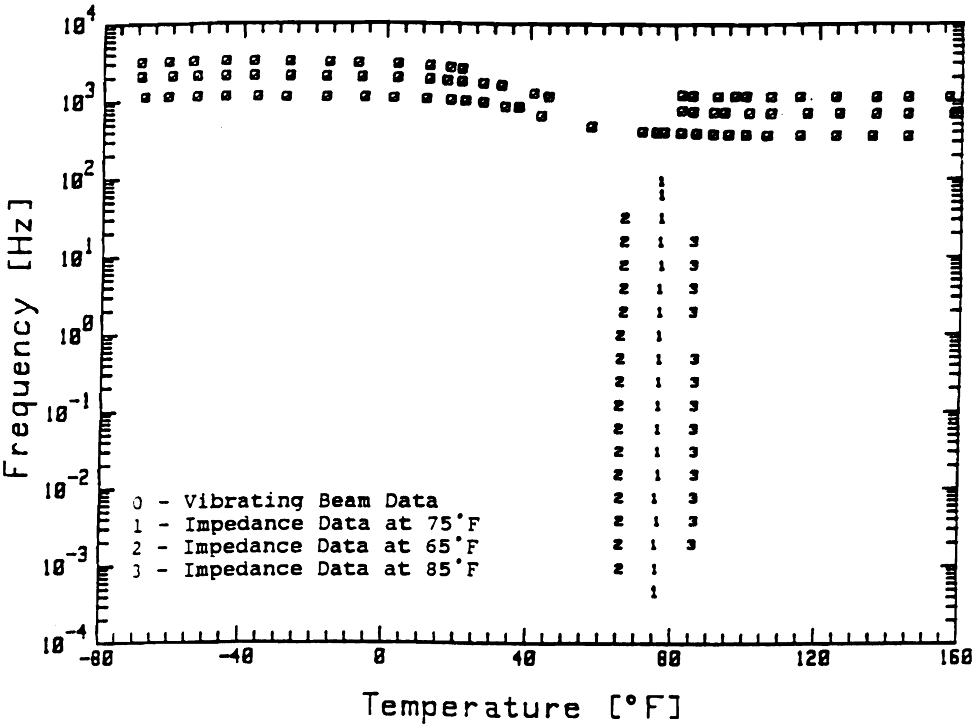

Figure 3: **Range of Testing Performed in Shear Using the Impedance and Beam Techniques**

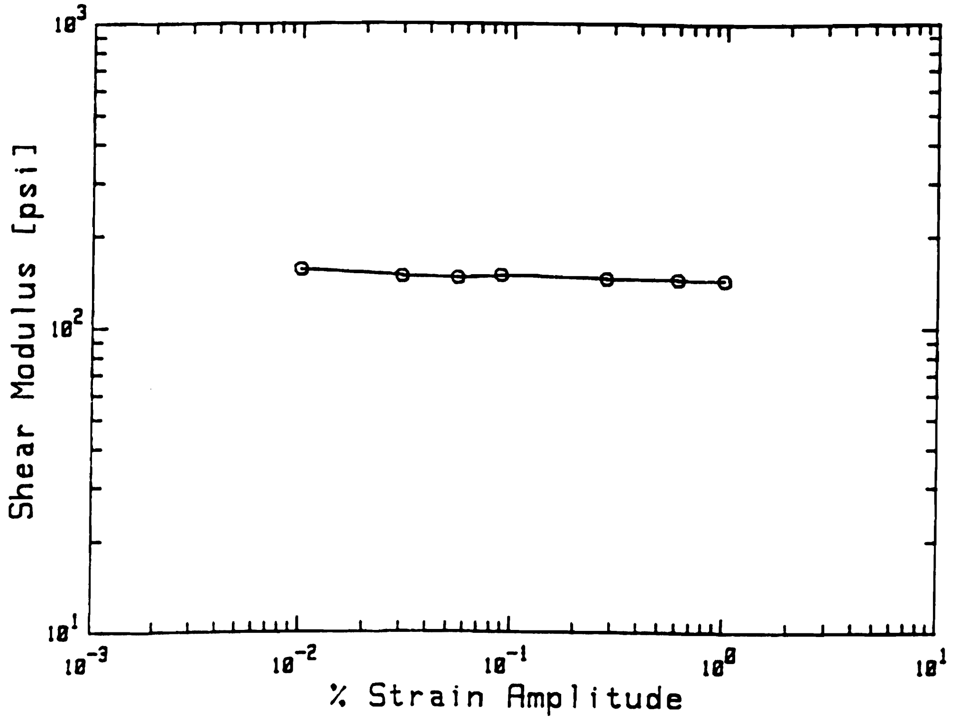

Figure 4: Effect of Various Dynamic Strain Levels on Shear Storage Modulus of the Material

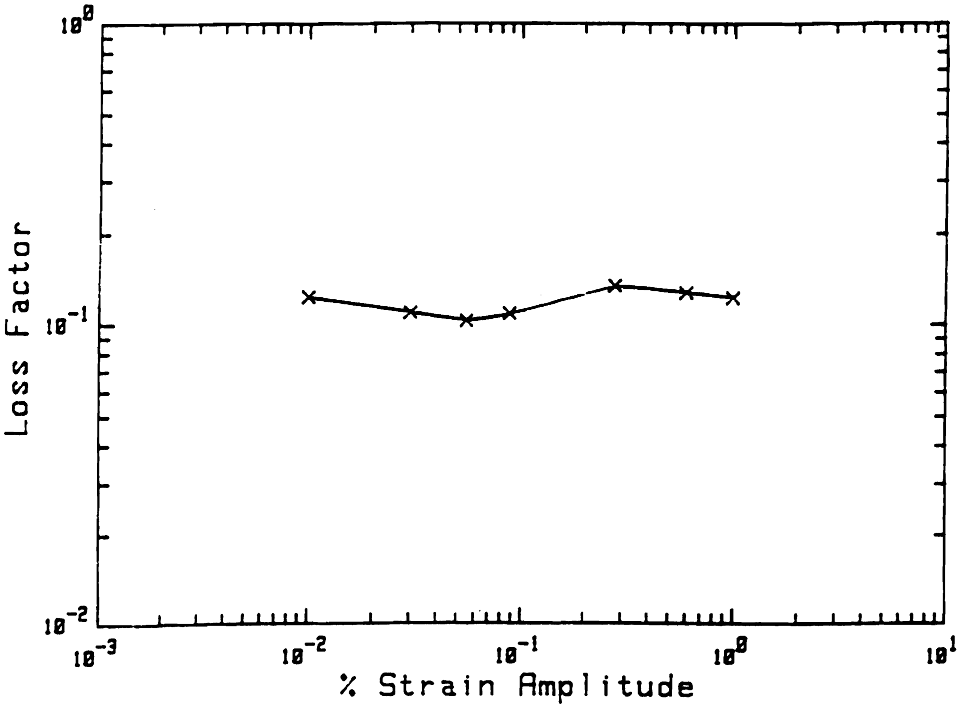

Figure 5: Effect of Various Dynamic Strain Levels on Loss Factor of the Material

In order to display all the material test results in one format, the reduced frequency nomogram is used by applying a shift factor equation to the data. More specifically, the Arrhenius shift factor equation was employed, and found to provide a good representation of the combined datasets of the vibrating beam and impedance techniques. The reduced frequency nomograms are provided for these datasets both separately and combined in Figures 6 through 8.

To qualify the accuracy and consistency of the data, the wicket plots of Figures 9 and 10 were used, plotted in log(storage modulus) versus log(loss modulus) and log(modulus magnitude) versus log(loss factor), respectively. It can be seen from these plots that the data points fall into one smooth and continuous curve, either hook-shaped (Figure 9) of bell-shaped (Figure 10), which is characteristic of the wicket plot.

The remaining nomogram shown in Figure 11 represents a combined display of both shear and tension/compression data. Contained in this plot is the vibrating beam data tested in both states-of-stress, and the impedance data in shear. The obvious missing piece of information is the impedance data in tension/compression. These measurements are currently being performed, and once completed, will allow accurate estimates of Poisson's ratio to be made. However, even with this incomplete dataset, it is interesting to note that Poisson's ratio for this material appears to be a real quantity given by the overlap in the loss factor between the two states-of-stress.

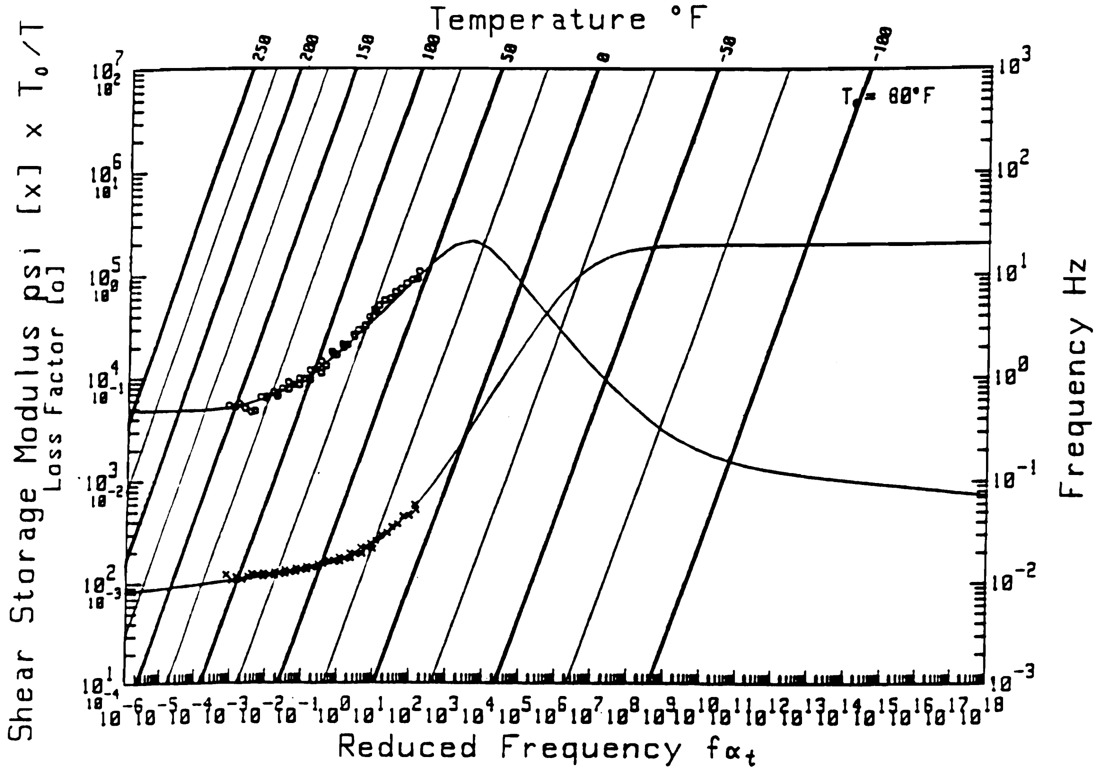

Figure 6: **Reduced Frequency Nomogram for the Material Tested in Shear Using the Impedance Technique**

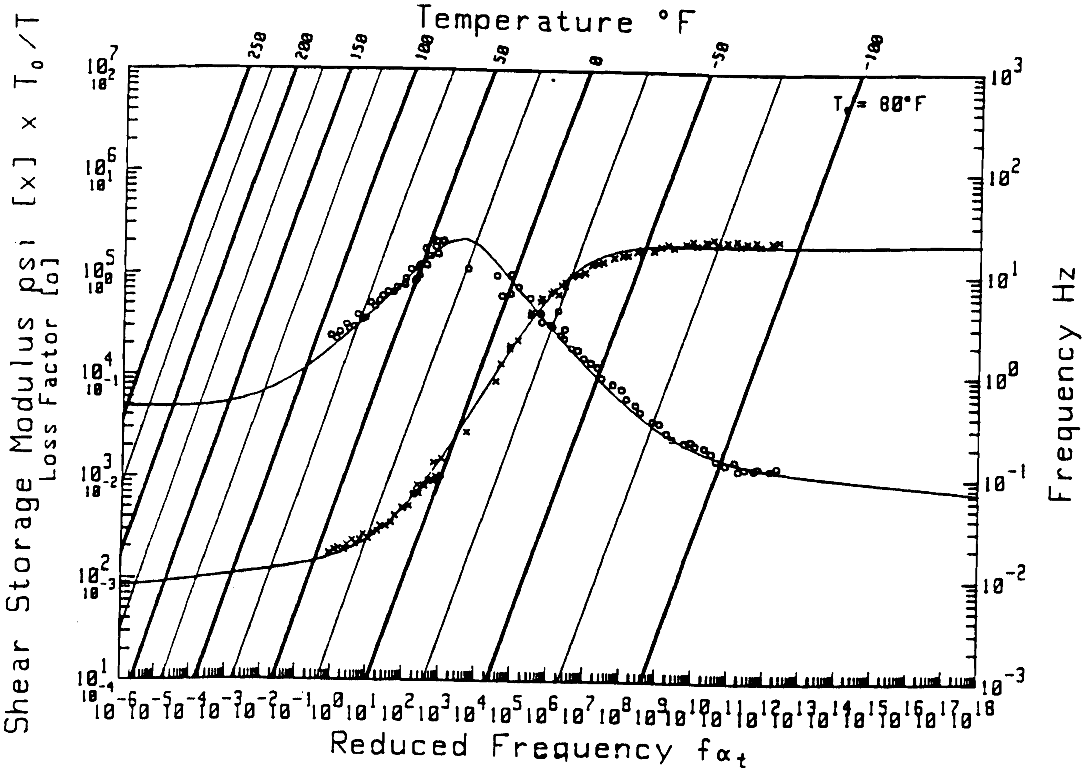

Figure 7: Reduced Frequency Nomogram for the Material Tested in Shear Using the Vibrating Beam Technique

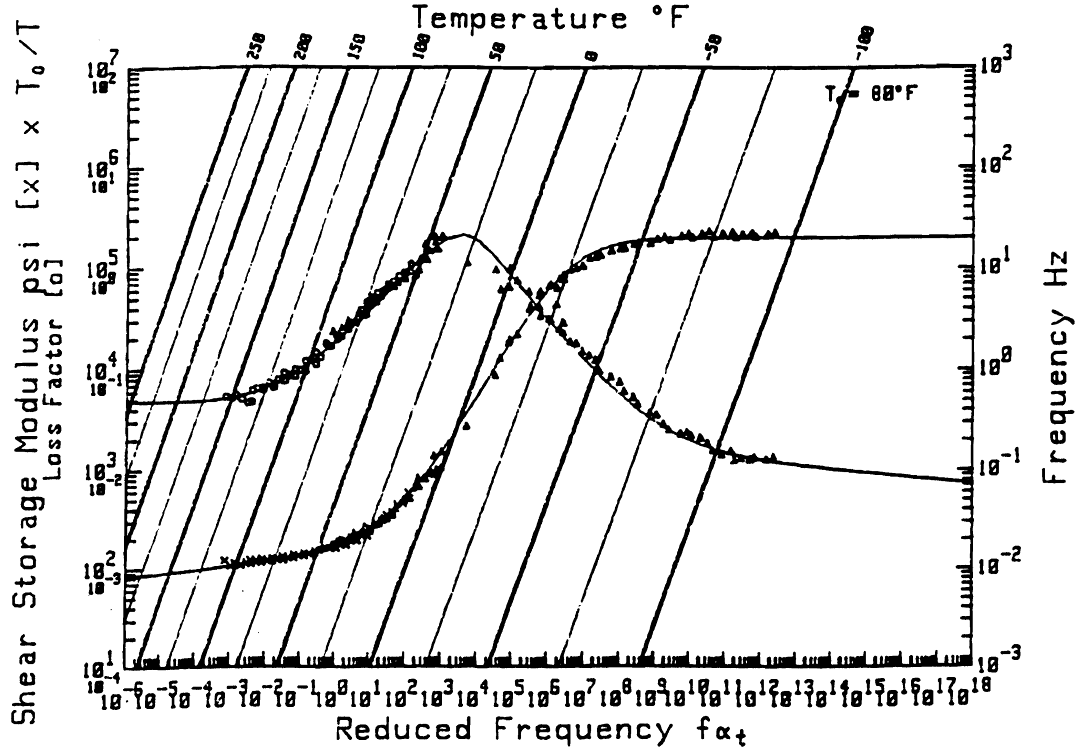

Figure 8: Reduced Frequency Nomogram for the Material Tested in Shear for Both the Impedance and Vibrating Beam Techniques

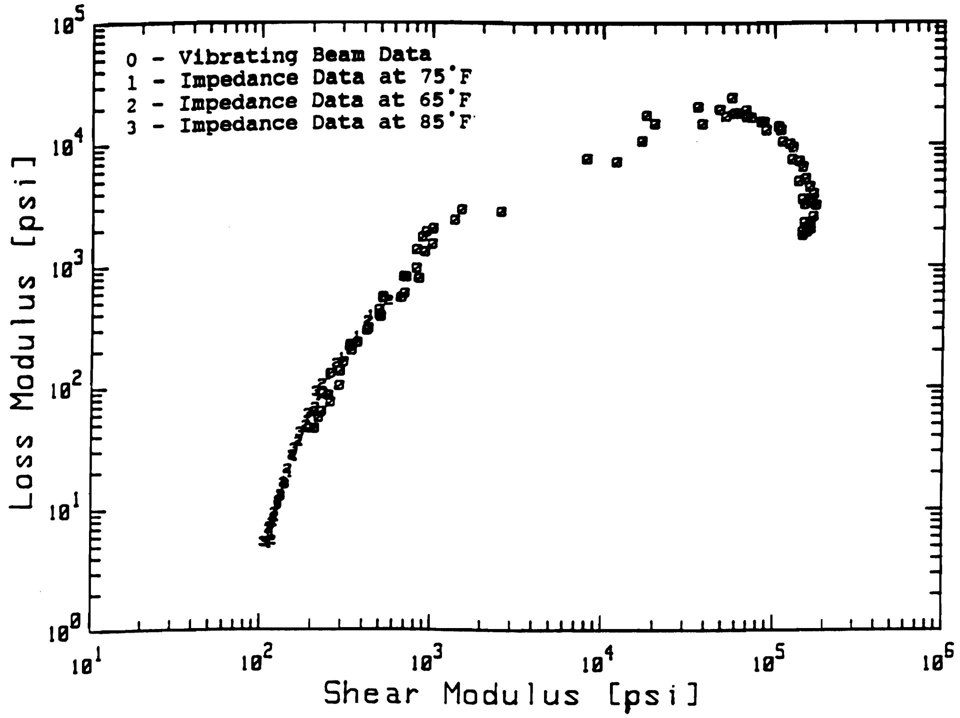

Figure 9: Qualification of Combined Shear Data Using the Wicket Plot in Loss Modulus vs. Storage Modulus Format

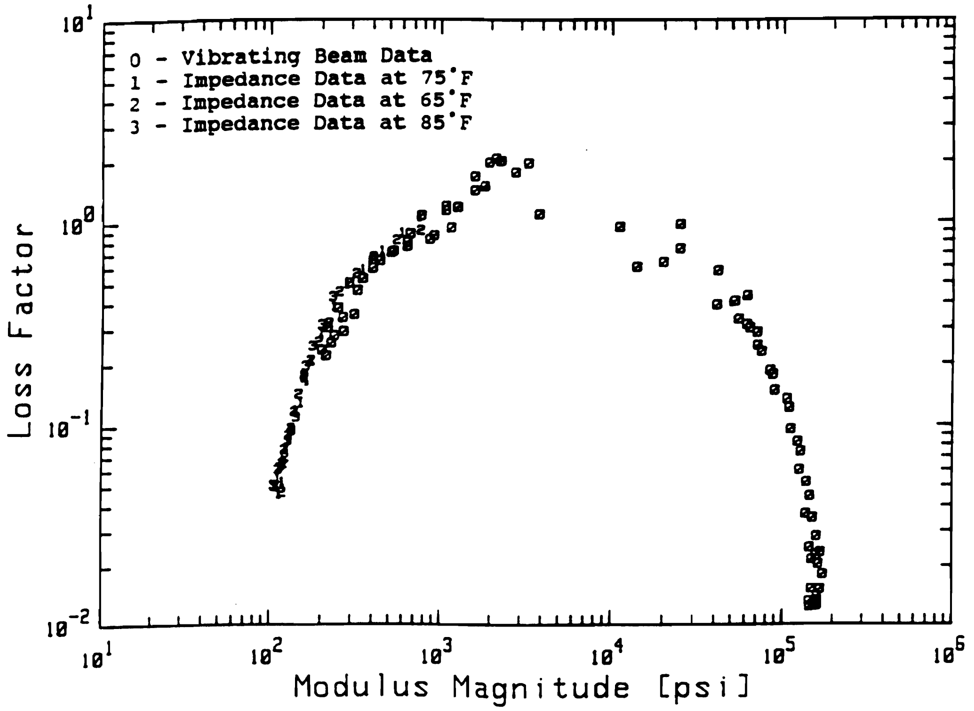

Figure 10: Qualification of Combined Shear Data Using the Wicket Plot in Loss Factor vs. Modulus Magnitude Format

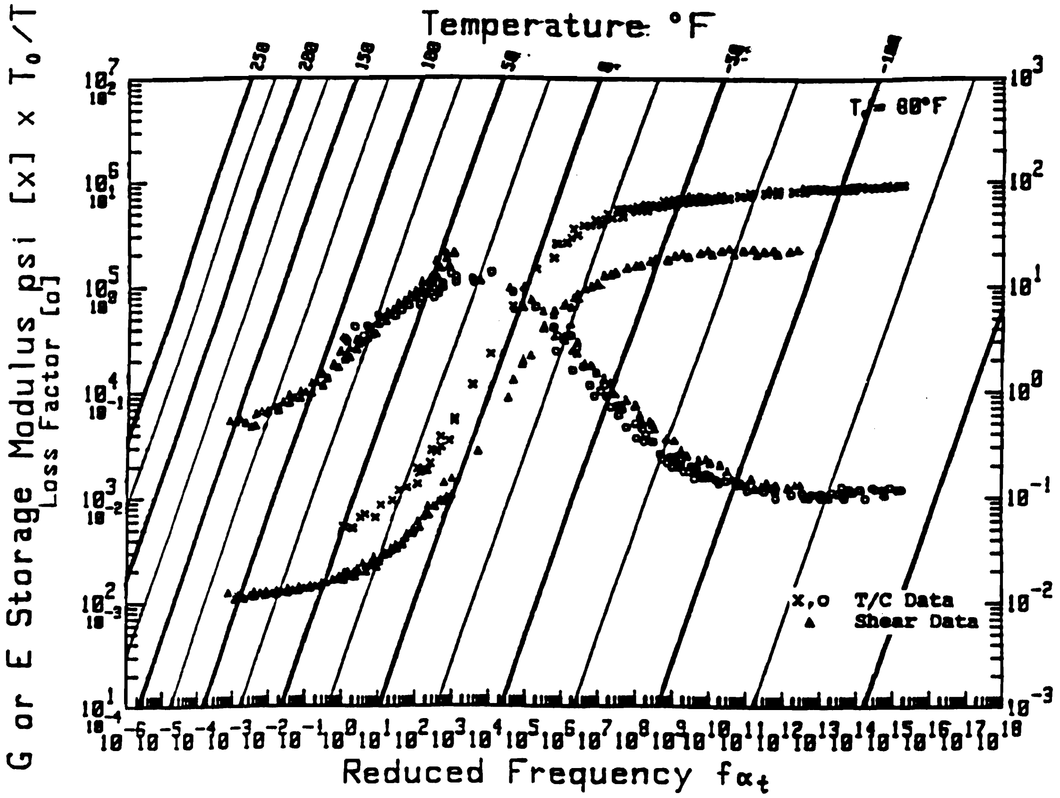

Figure 11: **Reduced Frequency Nomogram for the Material Tested in Shear and Tension/ Compression Using Various Techniques**

V. FUTURE INVESTIGATIONS

Some of the areas that will be focussed on to make improvements to the range and accuracy of this low-frequency impedance technique are discussed below:

(1) Expand the effective frequency range in which measurements can be performed, both on the high and low end of the spectrum. Theoretically, it should be possible to make measurements above the sample resonance, as well, assuming there are no other modes that interfere with the phase between the force and displacement. However, structural modes of the test fixture itself exist below 1000 Hz. In order to improve the performance on the high end, it will be necessary to increase the rigidity of the stinger and support structure, pushing the system resonances higher in frequency.

The actuator that is used is capable of producing low frequency sine waves down to DC. The limitations on the low frequency end lie in the source generator used, and the practical limits in allowable time which can be invested in making these measurements. Currently, it takes approximately 3 to 4 hours to produce clean waveforms at a frequency of 5e-4. This assumes there are no interruptions that contaminate the measurement traces. Frequencies much lower than this may be possible, but not practical. An achievable goal is however to consistently produce valid measurements down to 1e-4. This would require running the test at night over a 8 to 12 hour time period.

(2) Make estimates of Poisson's ratio by performing measurements in both shear

and tension/compression on the same test specimen.

(3) Select measurement transducers which cover a wider range of operation. At the present time, several transducers are used to cover the wide range of operation demanded by this technique. Displacement transducers have the capability to make measurements from 1e-5 to 4e-2 inches. Unfortunately, two force transducers are required to cover the wide range of allowable frequency measurements.

It is anticipated in the near future to have a system which is capable of generating extremely low scatter data for both shear and tension/compression over a frequency range of 1e-4 to 1000 Hz (7 decades), and at temperatures ranging from -100° to 300°F. However, as the data presented in this paper would indicate, this system is currently able to produce valid data over a wider frequency scale, and at lower frequencies than is available with other impedance systems on the market.

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LIST OF SYMBOLS

$G'(\omega)$	Shear Storage Modulus
$G''(\omega)$	Shear Loss Modulus
$E'(\omega)$	Young's Storage Modulus
$E''(\omega)$	Young's Loss Modulus
A_H	Area of Hysteresis Loop
A_L	Loaded Area of Damping Material
a_n	Eigenvalue of nth Mode
E_1	Young's Modulus of Metal Beam
f_n	nth Natural Frequency
Δf_n	Half-Power Bandwidth
H_1	Thickness of Metal Beam
H_2, t	Thickness of Damping Material
L	Length of Beam
m	Added Mass of Test Sample

s	Loaded Area-to-Unloaded Area Ratio
β	1.5 for Filled Elastomers/2.0 for Non-Filled Elastomers
ϵ_0	Maximum Strain Amplitude
σ_0	Maximum Stress Amplitude
η	Loss Factor of Damping Material
η_c	Loss Factor of Composite Beam
ω	Circular Frequency ($2\pi f$)
ω_n	Circular Frequency of nth Mode
ω_{1n}	Circular Frequency of nth Mode of Metal Beam
ρ_1	Density of Metal Beam
ρ_2	Density of Damping Material