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DYNAMIC MECHANICAL MATERIALS CHARACTERIZATION AND ANALYSIS USING A DYNAMIC MECHANICAL THERMAL ANALYZER

BY DR. WALTER M. MADIGOSKY
RESEARCH AND TECHNOLOGY DEPARTMENT

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NAVAL SURFACE WARFARE CENTER

Dahlgren, Virginia 22448-5000 • Silver Spring, Maryland 20903-5000

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FOREWORD

The purpose of this report is to document two of the dynamic viscoelastic properties (shear modulus and loss factor) of viscoelastic compounds. They are determined as a function of frequency and temperature. Applying the time-temperature superposition principle to the data, master curves are constructed and Williams-Landel-Ferry (WLF) shift constants are determined.

This work was done during FY88-89 with funds and materials provided by David Taylor Research Center (Code 1908), Bethesda, Maryland.

Approved by:

Carl E. Mueller

CARL E. MUELLER, Head
Materials Division

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INTRODUCTION

Knowledge of the dynamic mechanical properties of materials over a wide range of frequencies and temperatures is absolutely necessary in order to select materials and design systems which provide the greatest possible vibration control and noise reduction. Although there are many systems that have been designed for vibration damping and noise suppression, these systems generally have not been optimized in the strict mathematical sense. A systematic study of the problem is needed that demands not only a detailed knowledge of the dynamic mechanical properties but also a realistic mathematical model of the frequency and temperature dependence of these properties. The model parameters may then be used as input variables in a mathematical optimization computer code and, thus, both the design and the material can be chosen to guarantee optimal performance. The alternative to this is a trial and error approach which is time consuming, uncertain, and potentially expensive.

With the advent of new materials, chemicals, and processing, the need for a systematic approach is clear. Accordingly, this report describes the initial phases of a study which uses a commercial Dynamic Mechanical Thermal Analyzer (DMTA) to gather dynamic data on a number of materials as a function of temperature from the glassy to the rubbery region. Data were collected on different polymers to study the effect of polymer selection and on the polymers compounded with different loadings of a specific reinforcing filler, Durez, to study the effect of changing the hardness of the composition. The data were then analyzed according to the Williams-Landel-Ferry (WLF) equation[1] for time-temperature superposition and shifted into the frequency domain. The complex moduli were then fitted to the fractional operator model[2] which describes the moduli, storage modulus, and loss factor with a single complex equation. The simplest form of this model, used here, has only four independent parameters, all of which have a physical interpretation. Finally, an additional advantage of this approach is that these parameters, along with geometrical design parameters, may then be used as variables in a computer code that optimizes the material selection and geometry in order to achieve the maximum performance. Thus, the vibration damping or noise suppression properties of a design will be truly optimized, and no other similar design or material exists that can achieve a higher performance.

EXPERIMENTAL

Initially, two methods were used to obtain the dynamic mechanical properties as a function of temperature and frequency. The first method used a commercial DMTA from Polymer Laboratories.[3] In this method, small samples are mounted in a temperature controlled fixture, and the sample is driven at a constant frequency in either a single or double cantilevered mode. The stress necessary to achieve a given strain level and the phase angle between stress and strain is measured; the analysis of this information gives the Young's modulus and loss factor. The DMTA and its Universal Temperature Programmer (UTP) were controlled by a Hewlett-Packard Model 216 microcomputer using the software provided. Temperature control was achieved by using liquid nitrogen as a coolant and programmed heating to provide temperature scanning at low rates for good measurement accuracy. Samples to be tested were mounted using screw-on clamping bars inside the temperature enclosure and cooled to -60°C. The chamber was then momentarily opened and the clamps retightened with a torque screwdriver set to 5-10 N/cm. The DMTA was then programed to record data automatically at a ramp rate of 1 degree per minute over the temperature range of -50 to +50 degrees centigrade.

The second method of data collection used a Naval Surface Warfare Center (NAVSWC) developed resonance apparatus.[4] In this apparatus, samples were mounted vertically onto a miniature shaker while lightweight accelerometers were mounted on each end. The samples were driven with white noise and the transfer function measured as a function of frequency using a spectrum analyzer. The resulting spectrum of amplitude and phase was analyzed to yield the complex Young's modulus.

The first method had the advantage of fairly rapid and automatic data collection over a wide range of temperature but limited frequency range. The second method took longer, needed larger samples than ASTM test sheets, but provided a much larger frequency range and more accurate data over a smaller temperature interval. In the beginning, both methods were used to cross check the accuracy of the instruments and measurement procedure, especially the procedure of mounting samples in the DMTA. In addition, the greater frequency range of the resonance apparatus provided a cross check of the WLF temperature shifting procedure used in transferring the temperature data collected by the DMTA into the frequency domain. Once these procedures were established, then the bulk of the data collected (all that shown here) were collected using the DMTA and analyzed using the WLF shifting procedure.

MATERIALS AND COMPOUNDING

The materials chosen for this initial study included rubber compounds using natural, nitrile, and neoprene polymers and a commercially available urethane. In addition to the study of the effect of polymer type, the present work studied the effect of a changing the rubber modulus. In this case, the Shore "A" hardness was varied through the addition of a phenolic resin filler, Durez (made by Occidental Chemical Corp.). The formulations are shown in Table 1. The urethanes studied also varied in hardness from a Shore "A" of 60 to over 100 and are available from Conap, Inc., Olean, New York.

RESULTS

The dynamic data were recorded on the Polymer Laboratories' DMTA as a function of temperature from -50 to +50 degrees centigrade at four frequencies: 1, 3, 10, and 30 Hz. It was found that this frequency range provided sufficient data to WLF shift into the frequency domain. As mentioned above, this was checked by comparing data with that obtained by the resonance technique at higher frequencies. The advantage of the DMTA was that samples were cut directly from the ASTM sample test plates for use in the DMTA, whereas special samples had to be prepared for the resonance tester.

Figure 1 shows a typical set of data as obtained directly from the DMTA instrument. By noting the glass transition temperature, -50°C, and adjusting the two WLF shift constants, C1 and C2, where both constants are referenced to the glass transition temperature, the frequency data were converted into a single shifted curve which spans 14 decades in frequency at a reference temperature of 10°C. The results are shown in Figure 2. Note that in both the temperature and frequency presentation, the glassy and rubbery moduli and plateaus are clearly defined. Also note that the data in Figure 2 collapses into a single curve as it should if the WLF formula is applicable. Figure 3 shows similar results for four other urethane materials available from Conap which vary in hardness (Shore "A" = 60, 70, 80, 90, and 95). In each case, the glassy modulus and the glass transition temperature, Tg = 50°C, remains constant but the rubbery modulus increases as the hardness increases. Also, a single transition is observed in each of the materials. Figure 4 shows the results for another Conap material EN-4 and, in this case, a double transition is observed indicating possibly a transition in both the soft and hard segments of the urethane. Finally, the data in Figure 3 were WLF shifted and the results at 10°C are shown in Figure 5. All of the materials were found to shift satisfactorily with shift constants very near the universal constants proposed by WLF, C1 = 21 and C2 = 65, when those constants are referred to the glass transition temperature.

Similar results were found for the natural, neoprene, and nitrile rubbers; these are shown in Figures 6 through 8. In the case of these rubbers, the hardness effect was achieved through the addition of Durez. Again note that the glass transition temperature is essentially independent of the Durez as is the glassy modulus, but the rubbery modulus increases with the Durez level and the Shore hardness. Also, as in the urethane samples, the loss factor decreases as the rubbery modulus increases. The WLF shift constants were again close to the universal constants and were C1 = 19 and C2 = 51 at Tg = -60°C for the natural rubber compounds, C1 = 22 and C2 = 51 at Tg = -30°C for the nitrile rubber compounds (except compound #NBR-0 which had a Tg = -40°C) and C1 = 18 and C2 = 51 at Tg = -37°C for the neoprene rubber compounds. Finally note that the addition of Durez to the nitrile compound has a different effect on the loss factor frequency dependence in that it appears to be much broader than in the neoprene and natural compounds.

The frequency data were then fitted to the fractional derivative model which in its simplest form is:

$$E^* = Eg + (Er - Eg)/[1 + (iF/Fo)^{\alpha}]$$

where E*, Eg, and Er are the complex, glassy, and rubbery values of Young's modulus, F is the frequency, and Fo and α are two constants that are the frequency at which the imaginary part of Young's modulus is a maximum and the slope of the real part of Young's modulus at the inflection point. Fo determines where the transition occurs in frequency space, and α is a measure of the width of the transition. Thus, all four constants have physical meaning.

Each of the frequency curves were fitted to this model, and typical results for the nitrile compounds are summarized in Table 2. Note the decreasing value of α which corresponds to the increased broadening of the transition with increasing hardness.

Finally, it is clear that the four fractional derivative parameters are a realistic set of constants that maybe used as variables in a computer code which optimizes the material response to obtain a desired level of performance. Knowledge of how these constants vary with each of the ingredients in a rubber compound will make it possible to define the compound formula that has the optimal dynamic Young's modulus properties predicted by the computer code.

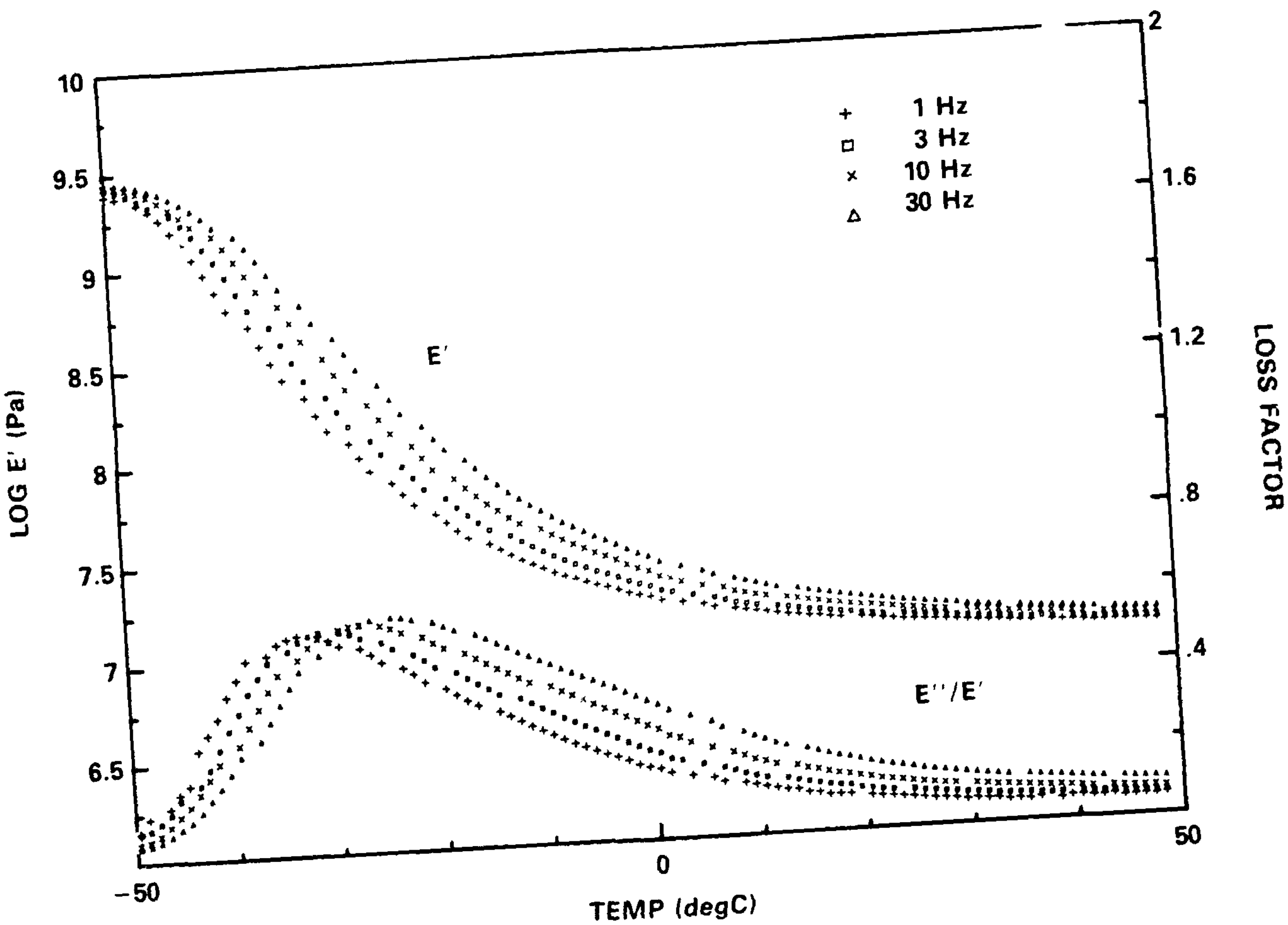

FIGURE 1. YOUNG'S MODULUS AND LOSS FACTOR FOR URETHANE POLYMER TU-700 (CONAP, INC., OLEAN, NEW YORK) AS A FUNCTION OF TEMPERATURE MEASURED BY THE DMTA

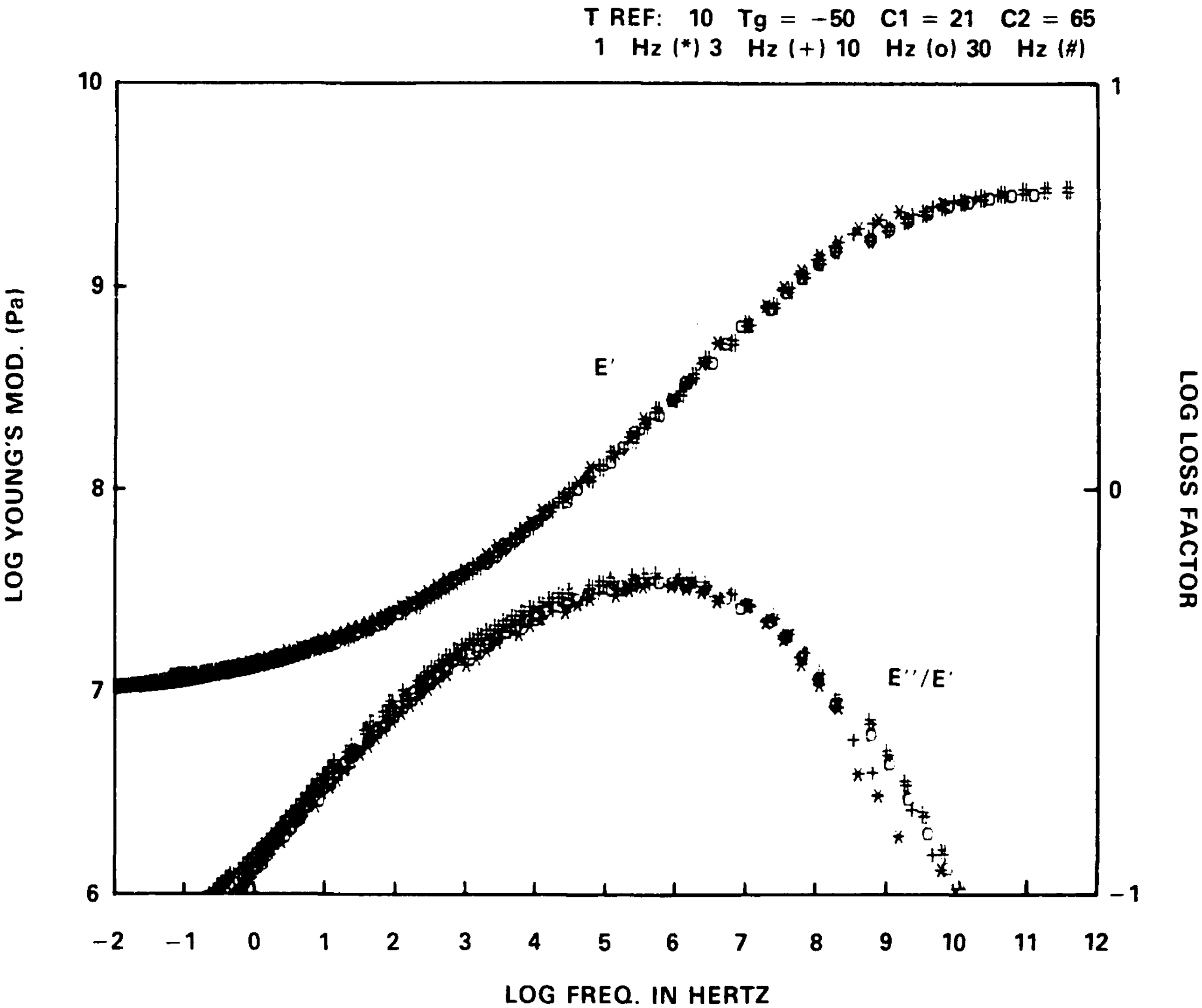

FIGURE 2. WLF SHIFTED DMTA DATA (FIGURE 1) VERSUS FREQUENCY FOR TU-700 AT 10°C

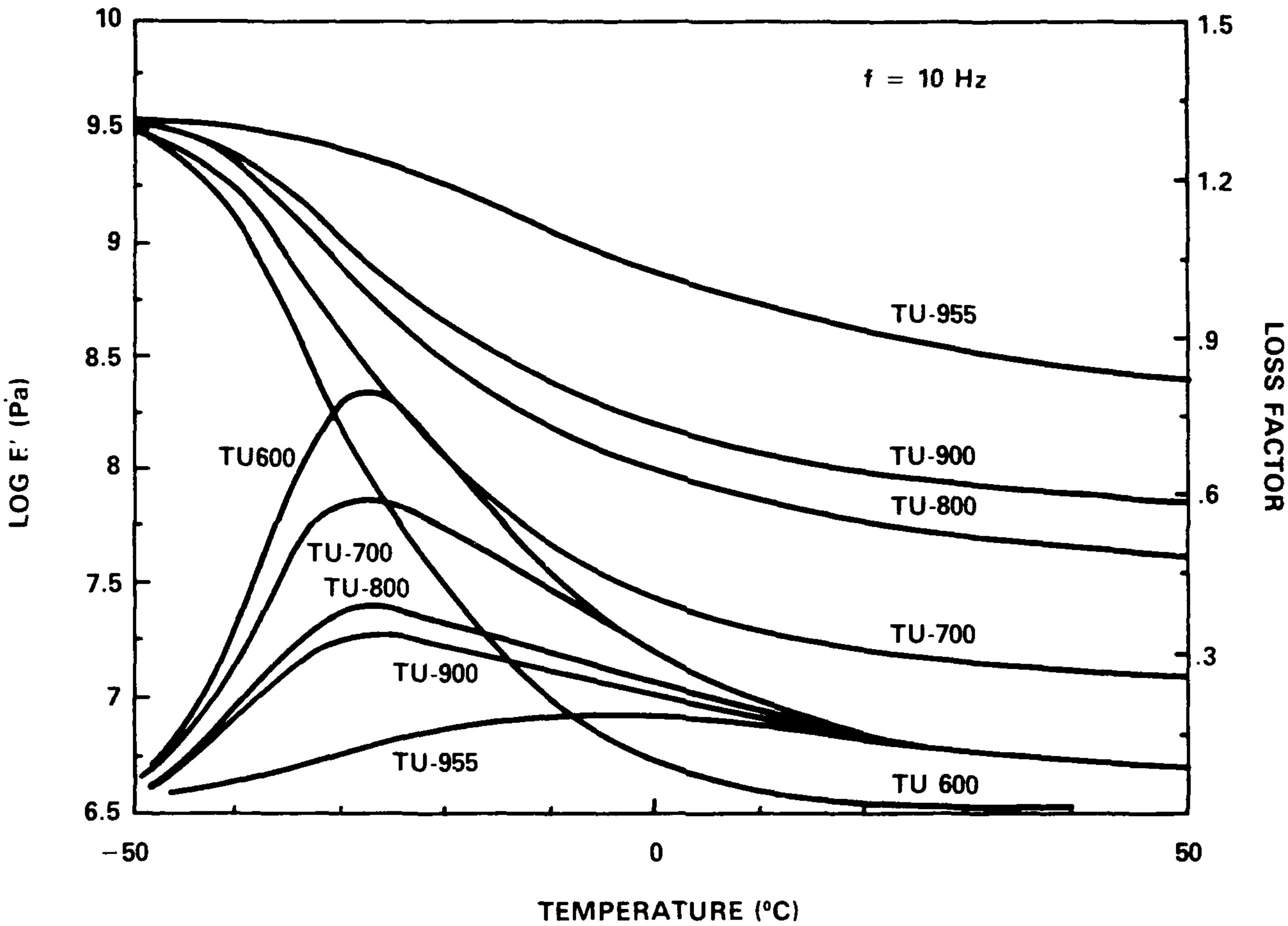

FIGURE 3. YOUNG'S MODULUS AND LOSS FACTOR FOR A SERIES OF URETHANE POLYMERS WITH INCREASING SHORE "A" HARDNESS

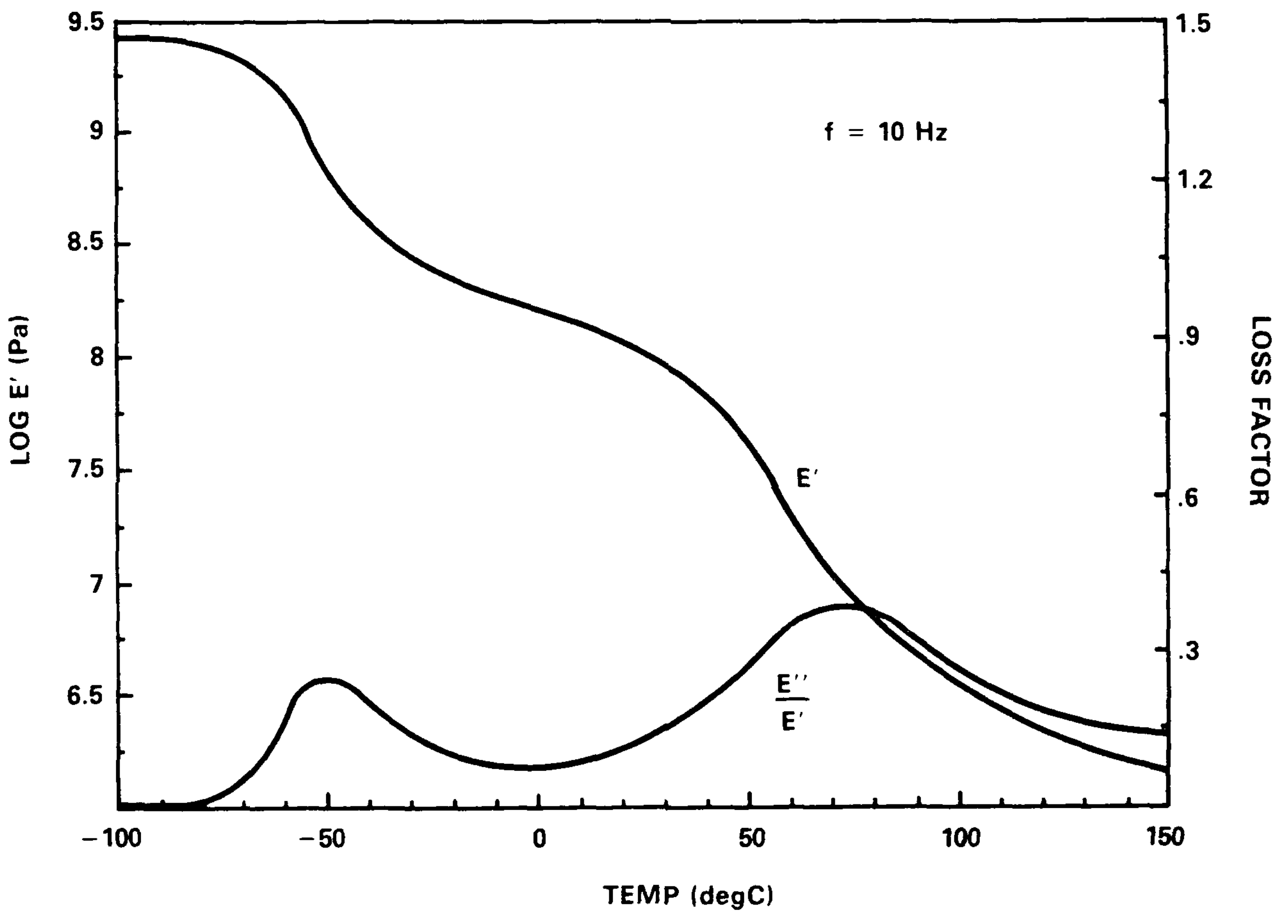

FIGURE 4. YOUNG'S MODULUS AND LOSS FACTOR FOR CONAP EN-4 AT 10 HZ AS A FUNCTION OF TEMPERATURE MEASURED BY THE DMTA

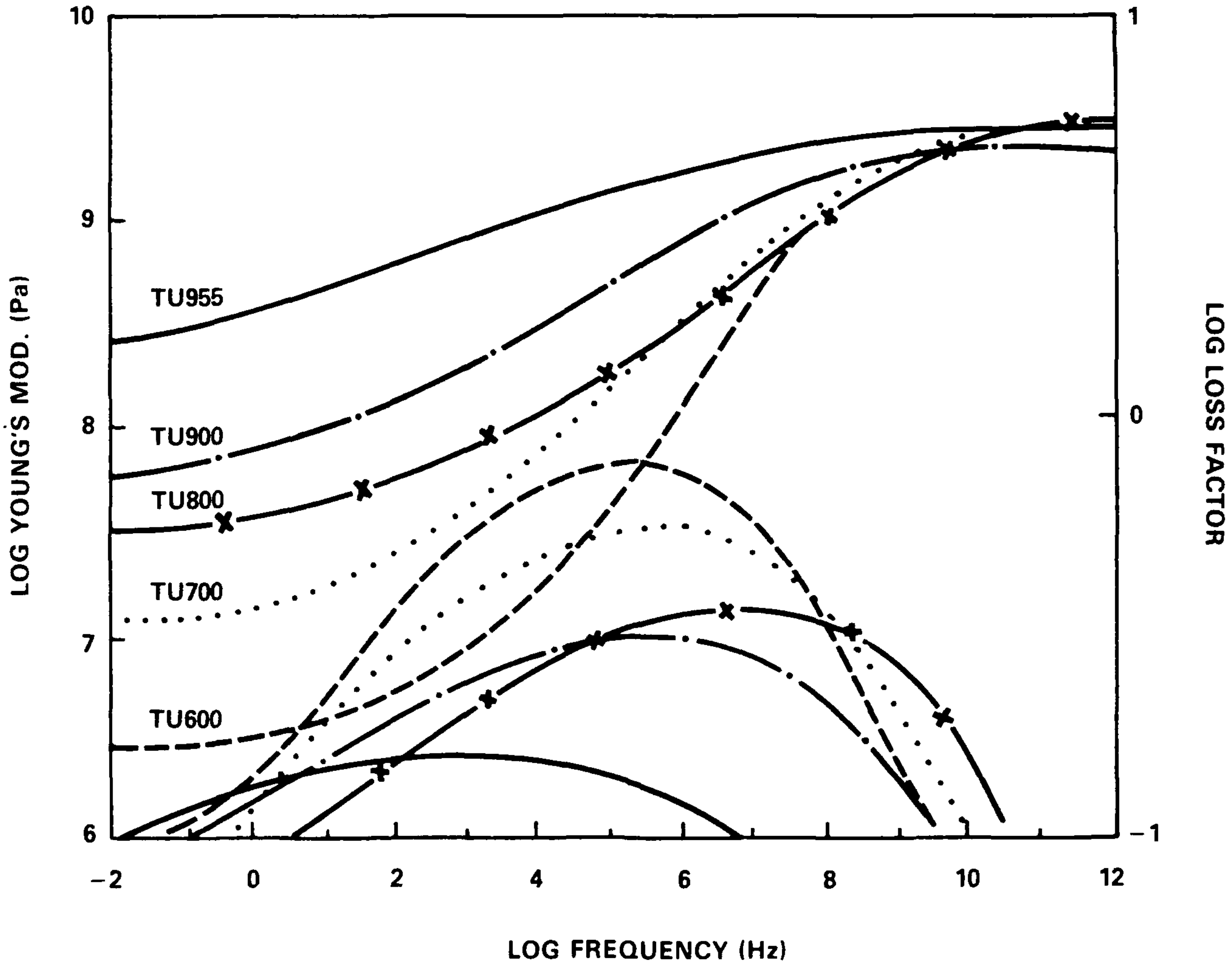

FIGURE 5. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND LOSS FACTOR FOR A SERIES OF URETHANE POLYMERS AS A FUNCTION OF FREQUENCY AT 10°C

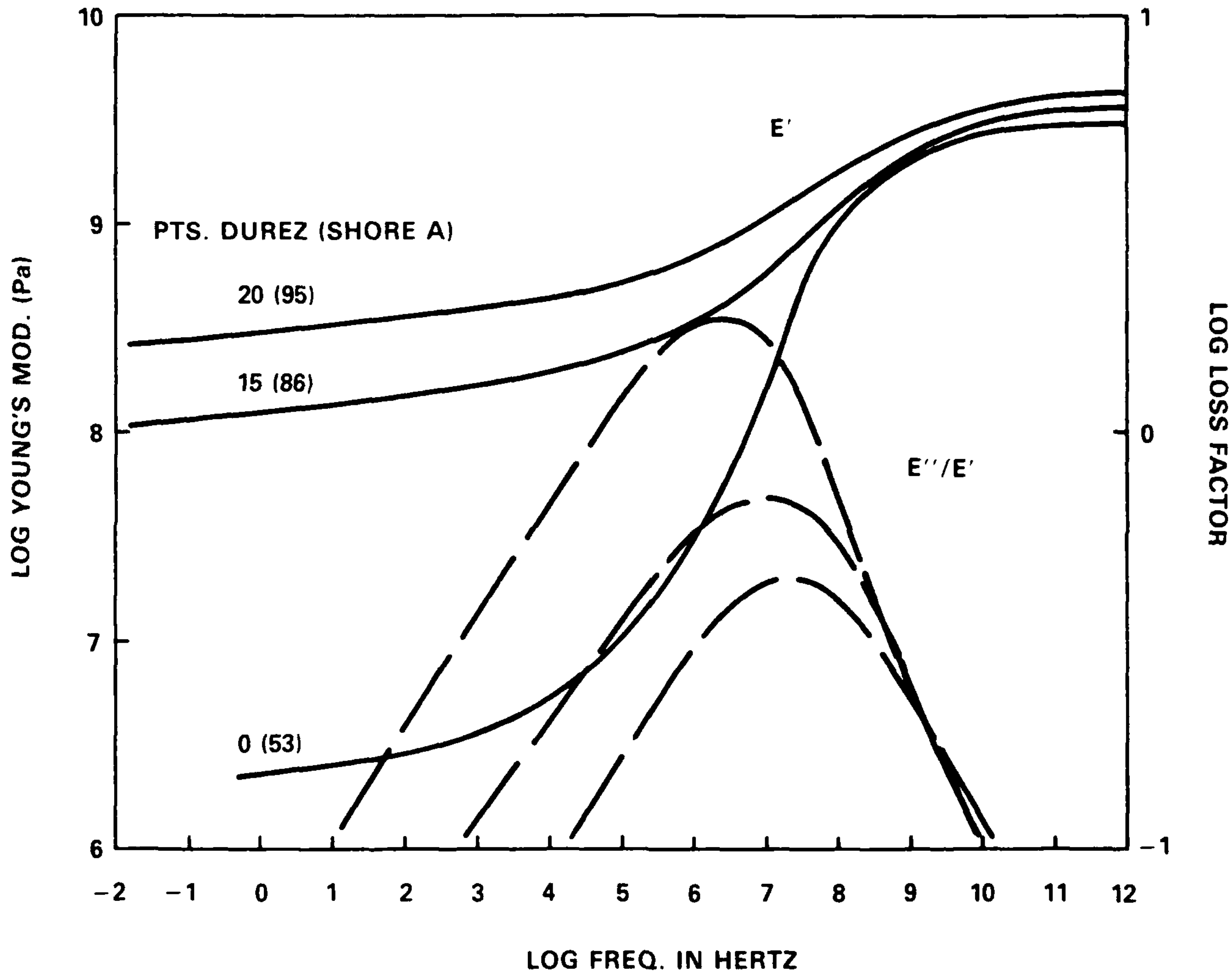

FIGURE 6. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND LOSS FACTOR FOR A SERIES OF NATURAL RUBBER COMPOUNDS AS A FUNCTION OF FREQUENCY AT 10°C

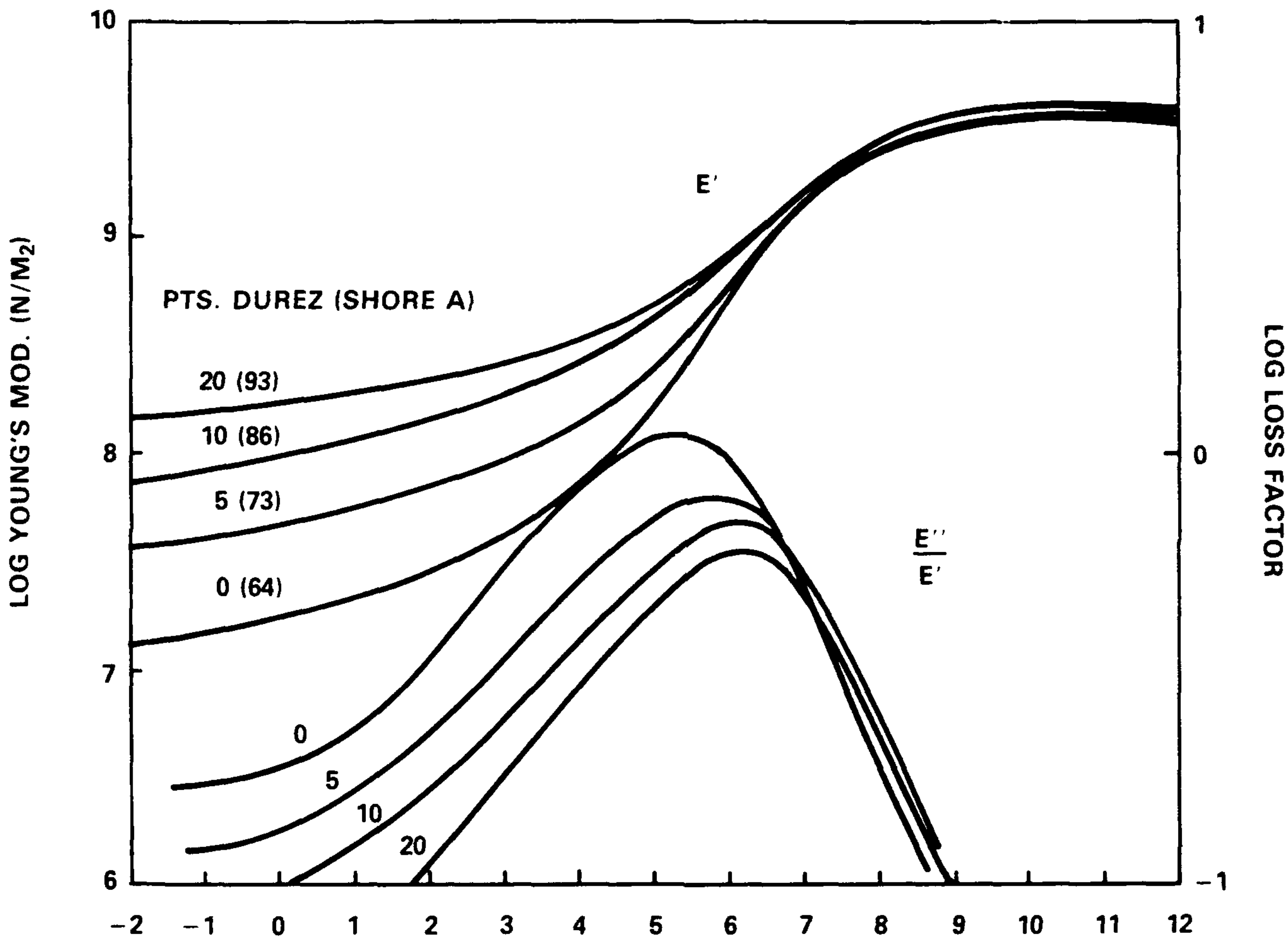

FIGURE 7. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND LOSS FACTOR FOR A SERIES OF NEOPRENE RUBBER COMPOUNDS AS A FUNCTION OF FREQUENCY AT 10°C

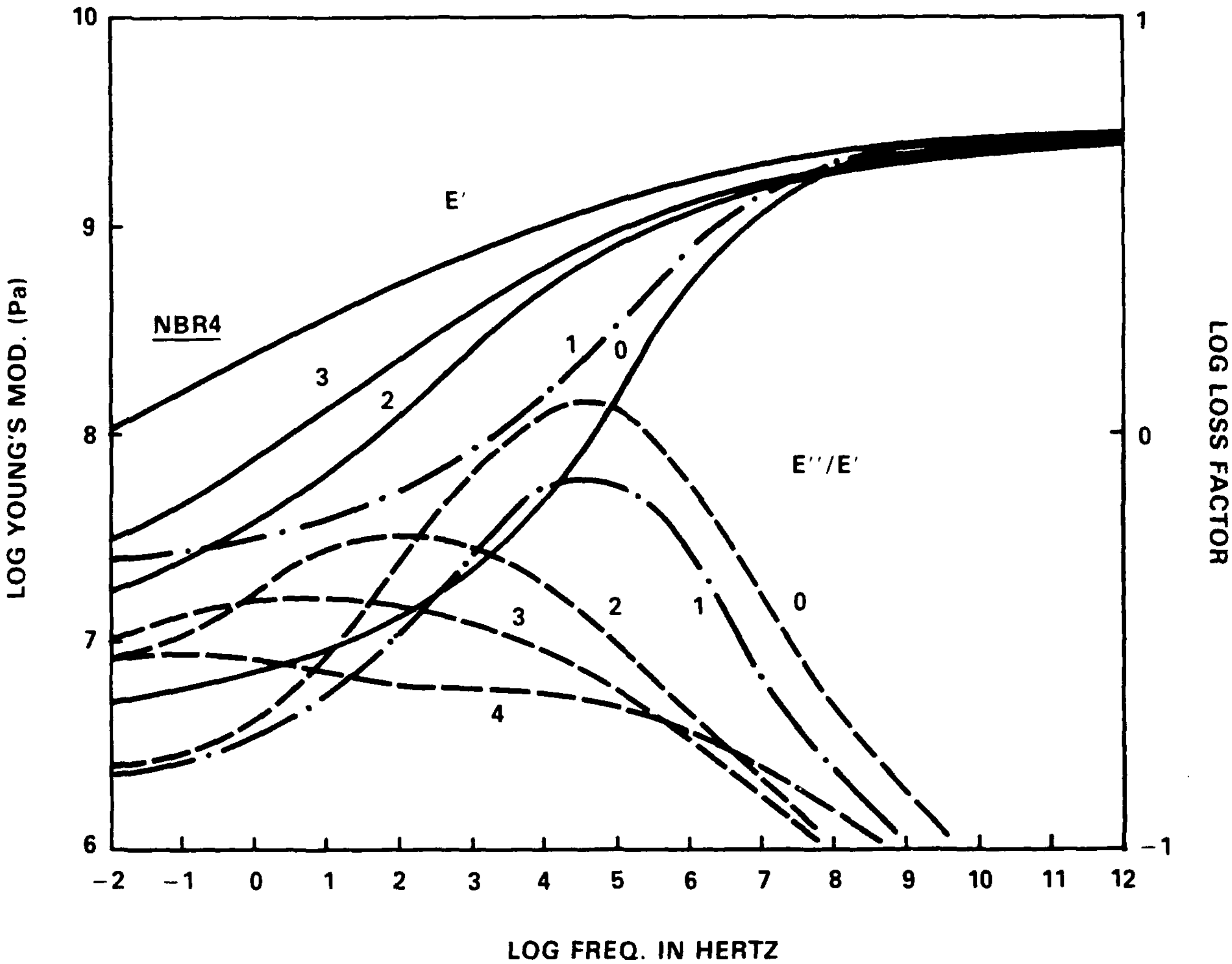

FIGURE 8. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND LOSS FACTOR FOR A SERIES OF NITRILE RUBBER COMPOUNDS AS A FUNCTION OF FREQUENCY AT 10°C

TABLE 1. RUBBER FORMULATIONS USED IN THIS STUDY

Compound #	NR-1, 2, 3	NBR-0, 1, 2, 3, 4	1, 2, 3, 4
Natural SMR-20	100	---	---
Paracril BLT	---	100	---
Neoprene NPR6398	---	---	100
Black N330	---	---	---
Black N660	20, 65, 65	0, 60, 40, 40, 40	---
Black N990	---	---	100
Protox 166	5	5	---
Steric acid	3	3	1
Dicup 40C	---	4.25	---
Red lead 95%	---	---	15
Sulfur	2.2	---	---
Altax	2.5	---	1.5
Circo light oil	3	---	---
Octamine	2	1	2
DOP	---	15, 0, 0, 0, 0	---
Califux	---	0, 5, 5, 5, 5	---
Age Rite Resin D	---	0.5	---
TE-70	---	---	2
Durez 12687	0, 15, 20	0, 0, 20, 30, 40	0, 5, 10, 20

TABLE 2. FRACTIONAL DERIVATIVE CONSTANTS FOR NITRILE COMPOUNDS

Compound #	LOG Er (Pa)	LOG Eg (Pa)	Fo (MHz)	α
NBR-0	6.85	9.40	2.3	.58
NBR-1	7.48	9.40	10.0	.42
NBR-2	6.60	9.40	0.17	.33
NBR-3	7.36	9.40	0.10	.30
NBR-4	7.95	9.48	1.0	.23

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Material characterization is essential both for design use and control. Specifically, materials designed for vibration damping and noise suppression precise values of the modulus and loss factor over a wide range of frequency and temperature. Until now, apparatus for making such measurements had very limited frequency and temperature ranges, were of questionable accuracy, and were usually very time consuming to operate. This report presents data as a function of temperature, from the glassy to the rubbery region, measured on a Polymer Laboratories' Dynamic Mechanical Thermal Analyzer (DMTA) and the same data transformed into the frequency domain using the Williams-Landel-Ferry (WLF) time-temperature shift equation. Data were collected on a number of natural, neoprene, nitrile, and polyurethane materials with different levels of reinforcement filler. The data clearly show the effect of polymer selection, filler type, and loading on the dynamic properties. Finally, the complex moduli may be analyzed in terms of the fractional operator model which describes both the storage and loss moduli with a single complex equation. The results suggest an analytic method for optimizing the dynamic response through detailed material selection.

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