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WIDE-TEMPERATURE-RANGE FREE-LAYER DAMPING MATERIALS

F. S. OWENS

TECHNICAL REPORT AFML-TR-70-242

DECEMBER 1970

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AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
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Techniques are described for extending and altering the temperature range over which viscoelastic materials are useful for free-layer damping. The approach is based upon the phenomenon that viscoelastic polymers exhibit a high level of structural damping only within a few degrees of their glass transition temperature and that certain types of polymer blends exhibit more than one glass transition temperature. The dynamic modulus and loss factor values of several vulcanized blends of mutually insoluble polymers containing selected amounts and/or kinds of polymers, fillers, plasticizers, and curing agents were measured over temperature ranges including all of the (apparent) glass transitions of the polymers used in the blends. Several very effective formulations for free-layer damping over wide temperature ranges were developed and the structural damping behavior of selected materials were investigated in simulated applications. The results show that polymer blends are versatile materials for wide-temperature-range free-layer damping treatments and can be formulated to meet specific needs of a particular vibration damping problem requiring free-layer damping; i.e., the properties of the material can be matched to the requirements by judicious selection of polymers, fillers, curing agents, and plasticizers.

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Modulus vs Temperature						

AFML-TR-70-242

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F. S. OWENS

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Distribution of this report is limited to protect technical information relating to critical products and manufacturing processes.

FOREWORD

This report was prepared by the Elastomers and Coatings Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work was initiated under Project No. 7340, "Nonmetallic and Composite Materials," Task No. 734005, "Elastomeric and Compliant Materials," and was administered by the Air Force Materials Laboratory, with Mr. F. S. Owens (LNE) serving as Project Engineer.

This report covers that portion of work conducted from 15 July 1966 to 13 October 1969, and was submitted by the author in November 1969.

The author gratefully acknowledges the assistance of personnel of the Strength and Dynamics Branch, Metals and Ceramics Division, who evaluated the dynamic mechanical properties of some of the experimental formulations.

Some of the materials compared in this report were commercial materials that were not developed or manufactured to meet Government specifications, to withstand the tests to which they were subjected, or to operate as applied during this investigation. Any failure to meet the objectives of this research program is no reflection on any of the commercial materials discussed herein or on any manufacturer.

This technical report has been reviewed and is approved.

Warren P. Johnson

WARREN P. JOHNSON, Chief
Elastomers and Coatings Branch
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Air Force Materials Laboratory

ABSTRACT

Techniques are described for extending and altering the temperature range over which viscoelastic materials are useful for free-layer damping. The approach is based upon the phenomenon that viscoelastic polymers exhibit a high level of structural damping only within a few degrees of their glass transition temperature and that certain types of polymer blends exhibit more than one glass transition temperature. The dynamic modulus and loss factor values of several vulcanized blends of mutually insoluble polymers containing selected amounts and/or kinds of polymers, fillers, plasticizers, and curing agents were measured over temperature ranges including all of the (apparent) glass transitions of the polymers used in the blends. Several very effective formulations for free-layer damping over wide temperature ranges were developed and the structural damping behavior of selected materials was investigated in simulated applications. The results show that polymer blends are versatile materials for wide-temperature-range free-layer damping treatments and can be formulated to meet specific needs of a particular vibration damping problem requiring free-layer damping; i.e., the properties of the material can be matched to the requirements by judicious selection of polymers, fillers, curing agents, and plasticizers.

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SECTION I

INTRODUCTION

Vibrations in flight vehicles are caused by unstable dynamic conditions. Transient vibrations persist for brief periods of time and generally cause little damage to the structure unless the stresses and strains are excessive or the vibrations occur repeatedly. Persistent vibrations, however, last for long time intervals and cause considerable damage to the structure and its components even though stress and strain levels may be low. Resonant or near resonant vibrations result in excessive noise, structural fatigue, component failure, instrument inaccuracies, etc.

Viscoelastic materials are used to isolate, insulate, and attenuate unwanted and harmful oscillations. These materials protect delicate instruments from vibrations, structural components from fatigue failure, and personnel from undesirable noise. Ideally, the materials used for damping should be insensitive to variations in temperature, frequency, strain, and the manner of applying the materials to obtain damping. Damping materials for use on flight vehicles should also have low density. Viscoelastic materials are almost ideal for damping flight vehicle vibrations because they are capable of dissipating several hundred times more energy than other typical structural materials (References 1, 2, and 3) and can withstand higher strain levels for a larger number of vibration cycles.

This report is concerned with the development of a versatile viscoelastic material formulation for use in a special kind of structural damping — unconstrained or free-layer damping treatments. A free-layer damping treatment is one in which the damping material is subjected to extensional deformation cycles, such as would occur when a single layer of viscoelastic material adheres directly to one side of a flat plate or bar or to a large diameter shell. Free-layer damping treatments are highly effective in reducing the stresses and amplitudes (strains) of longitudinal flexural vibrations (References 4, 5, 6, and 7). The effectiveness of unconstrained layer treatments in attenuating resonant frequencies and spurious oscillations depends on the dynamic and physical properties of both the viscoelastic layer and the base material (References 8, 9, 10, and 11). Generally, a single-layer damping treatment is most effective when both the dynamic modulus of elasticity and the loss factor or the internal hysteresis of the damping layer are large (Reference 4). The modulus should be high enough to afford a significant amount of resistance to flexural bending motions, but should not exceed the bending stiffness of the primary structural material (assumed to be a metal) to which it is attached. In addition, the loss factor, a measure of the damping ability of the viscoelastic layer, should be high so as to rapidly dissipate the mechanical energy associated with flexural undulations or, perhaps, even the energy pulses tending to cause such peregrinations.

One of the most difficult problems to overcome with free-layer damping treatments has been to maintain high modulus and loss factor values over wide temperature or wide frequency ranges. This problem has been partially resolved at the Air Force Materials Laboratory by the use of immiscible polymer blends (References 12, 13, 14, 15, 16, and 17). This development was based on the fact that physical blends of dissimilar high polymers which do not chemically or physically interact with one another exist in separate phases, and each separate phase exhibits its own characteristic phase transformations which can be used for damping purposes (References 18, 19, and 20).

The objective of this research was to improve the damping ability of the previously developed vibration damping material (control blend) with special emphasis toward developing more versatile formulations for free-layer damping treatments. The "control" compound is a three-polymer blend consisting of equal parts by weight of a very high acrylonitrile containing acrylonitrile-butadiene rubber (Paracril-D*), polyvinyl acetate, and polystyrene (see Figures 19 and 20). This three-phase material is a candidate for free-layer treatment of Air Force damping problems because: (1) its high modulus and loss factor values are maintained throughout the temperature range of about 30 to 230°F within the frequency range of 100 to 1000 Hz, where many flight vehicle vibration problems occur; (2) the density is low (about 1 gm/cc); and (3) the material is resistant to gasoline, oils, and hydraulic fluids.

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*Uniroyal Trademark

The manner in which the modulus, E_2, and the loss factor, η_2, of the polymer blend change with temperature indicates that the effective temperature range can be increased if the qualitative compounding rules (see Chapter 7, Reference 18) for single polymer compositions extend to multiphase formulations. Thus, the experimental compounding will emphasize extending or altering the effective temperature range for adequate damping, testing qualitative rules for designing multiphase damping treatments, and showing that multiphase damping formulations can be produced in suitable form for application to any reasonable vibration problem where free-layer damping is needed.

SECTION II

EXPERIMENTAL EQUIPMENT AND MEASURING TECHNIQUES

1. EQUIPMENT

The dynamic mechanical properties of cantilever beam structures were measured using the Bruel & Kjaer Complex Modulus Apparatus and auxiliary equipment. The equipment, shown schematically in Figure 1, consists of a temperature chamber, a beat frequency oscillator, a signal amplifier, a level recorder, two magnetic discs, and the complex modulus apparatus. The test chamber is capable of maintaining well controlled temperatures from -100° to +500°F and is equipped with sealed electrical connections for the complex modulus apparatus. This apparatus is a massive nonmagnetic structure used to support the beam-type specimen and two electromagnetic transducers. The driving transducer, located near one end of the specimen, is excited by the beat

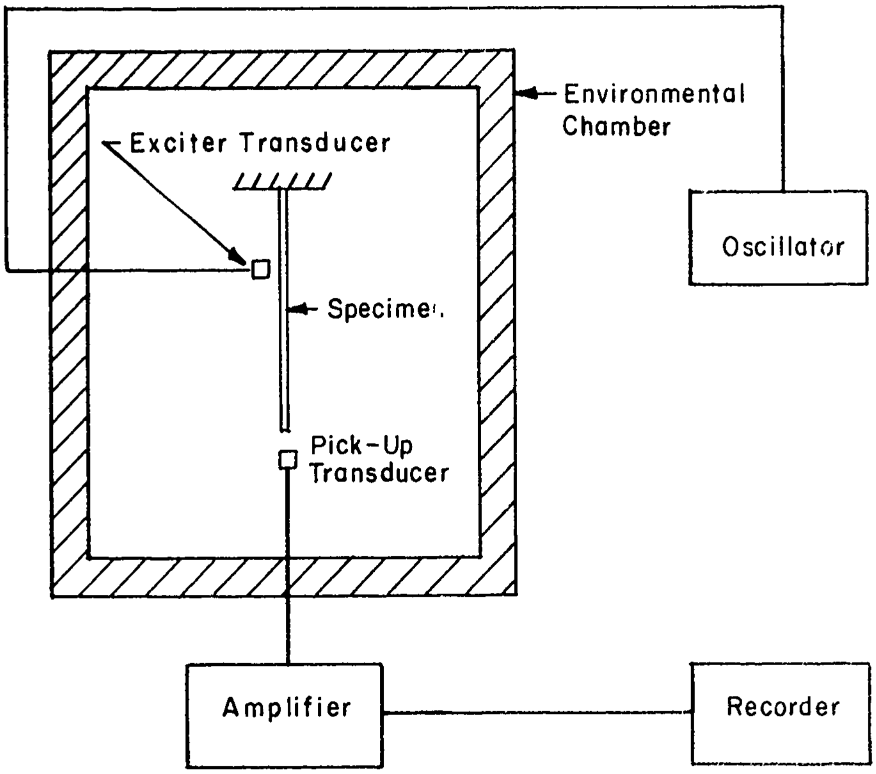


Figure 1. Schematic of Test Equipment

frequency oscillator with a sinusoidal voltage. This oscillating voltage causes the magnetic disc which is bonded to the test specimen to react in a sinusoidal fashion and, consequently, the beam reacts sinusoidally. The second transducer picks up the response through the second magnetic disc located on the other end of the specimen, and the electrical response is sent to the amplifier. The amplifier output signal is sent into the level recorder. Both the amplifier and recorder indicate the peak, average, or root-mean-squared input signal in decibels. The level recorder can be used to drive the oscillator, thus resulting in an automatic sweep through the entire frequency range of the oscillator, 20 to 20,000 H_z. The amplitude of the beam response as a function of frequency is recorded on precalibrated chart paper during the automatic sweep.

2. MEASURING PROCEDURES

All dynamic measurements were made in the temperature test chamber within the temperature range -100^o to $+480^oF$. The physical and geometrical measurements of the test specimens were made at ambient temperatures (near 75^oF) and were assumed to remain constant with temperature changes. While this assumption is not entirely valid, it is sufficiently accurate for the comparative data to be used in adjusting the composition of polymer blends to improve damping. Error analysis (Reference 21) was used as a basis for accepting or rejecting the results.

Experiments at ambient temperature (near 75^oF) were run first, followed by tests at successively lower temperatures. Low-temperature experiments were discontinued at -100^oF, or at higher temperatures when the results were unacceptable because of possible errors. Next, the higher-temperature experiments were run, starting at ambient temperature again. Specimens were soaked for a 30-minute period at each experimental temperature. The oscillator was calibrated just prior to making the test. At each experimental temperature, the measurements consisted of determining the resonant frequencies, f_n, and the width, Δf_n, of each resonant peak, $n = 1, 2, 3, \ldots$, from a graphical display of the amplitude vs. frequency curve of a specimen. f_n and Δf_n are related to the dynamic and physical properties of the experimental beams.

For a fixed-free homogeneous beam with uniform rectangular cross section, the real part of Young's complex modulus, E, and the loss factor, η, are given (Reference 22) by:

$$E = 48\pi^2\rho\left(\frac{\ell^2}{h} \times \frac{f_n}{k_n}\right)^2 \qquad (1)$$

and

$$\eta = \frac{\Delta f_n}{f_n} \qquad (2)$$

where

π = 3.1416

ρ = density of material in the beam

ℓ = free length of beam

h = thickness of beam

f_n = resonant frequency of the n-th mode

n = 1, 2, 3, . . .

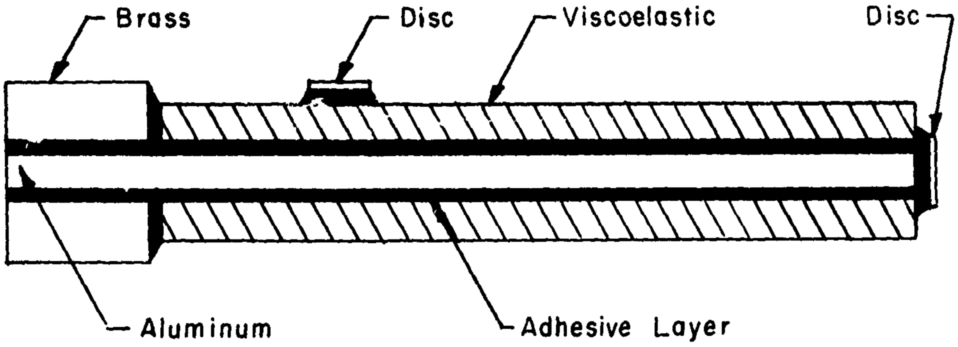
k_n = 3.52, 22.0, 61.7, 121, 200, 299, 217, 555, 713, and 891, respectively, for n = 1, 2, 3, . . ., 10

Δf_n = distance between the frequencies above and below the resonant frequency at which $A^2 = \frac{1}{2}(A_{max})^2$, with A_{max} being the amplitude at resonance.

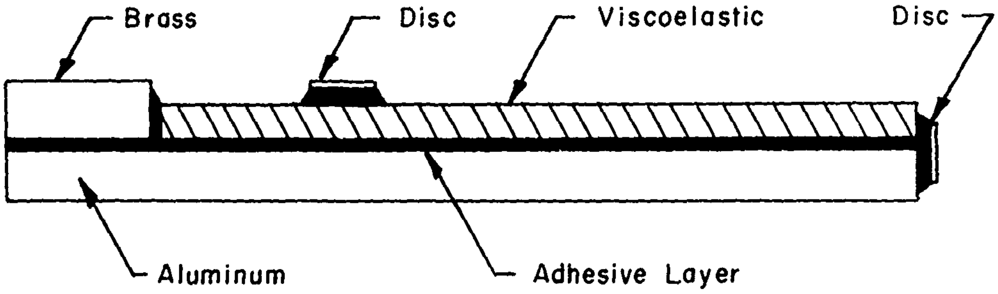
The test equipment was designed to measure E and η values within the approximate range where $E \geq 10^4$ psi and $10^{-3} \leq \eta \leq 0.2$. Thus, the equipment and Equations 1 and 2 are more applicable to metals than to viscoelastic materials when large temperature variations are involved (References 22, 23, and 24). The equipment, however, can be used to measure the dynamic response of composite beams consisting of a metal strip, beam, or bar; a thin rigid adhesive layer; and one layer (Reference 22) or two (Reference 21) layers of viscoelastic material of equal thickness on opposite sides of the metal beam (Figure 2). The resonant frequencies, f_n and Δf_n, and the bandwidths of the resonant frequencies, are related to the dynamic and physical properties of the materials used to construct the composite beams as well as to the type of construction. Three-layer (symmetrically or doubly coated) beams (Figure 2a) were used for measuring E_2 and η_2 at discrete temperatures throughout the frequency range 20 to 20,000 Hz. Two-layer (unsymmetrically or singly coated) beams (Figure 2b) were used to simulate a practical structural application of the materials for free-layer damping. The derivation of the equations and the details of data reduction are adequately documented elsewhere (Reference 21) and are not discussed here. The following results were used:

$$E_2 = \frac{E_1}{k}\left[\left(\frac{f_n}{f_{1n}}\right)^2\left(1 + 2\frac{h_2\rho_2}{h_1\rho_1}\right) - 1\right] \tag{3}$$

$$\eta_2 = \eta\left(1 + \frac{E_1}{kE_2}\right) \tag{4}$$



a. Three-Layer Specimen



b. Two-Layer Specimen

Figure 2. Test Specimens

where

$$k = 8\left(\frac{h_2}{h_1}\right)^3 + 12\left(\frac{h_2}{h_1}\right)^2 + 6\left(\frac{h_2}{h_1}\right)$$

$\eta = \frac{\Delta f_n}{f_n}$, the loss factor of the composite beam

E_1 = real part of Young's complex modulus for the metal

E_2 = real part of Young's complex modulus for the viscoelastic layer

h_1 = thickness of the metal

h_2 = thickness of viscoelastic layer on either side

f_n = resonant frequency of n-th mode of the composite beam

f_{1n} = resonant frequency of the n-th mode of the metal beam

ρ_1 = density of metal

ρ_2 = density of viscoelastic material.

3. SPECIMEN PREPARATION

Aluminum strips 8 inches long, 0.450 inch wide, and up to 0.125 inch thick were used in constructing the experimental specimens. One reason for using aluminum was because many if not most Air Force vibration problems involve aluminum structures. Usually, the viscoelastic layer was simultaneously press-cured and bonded to the aluminum. A thin layer of a high-temperature adhesive system (Chemlock*-203 primer and Chemlok-220 adhesive) was used when the modulus, E_2, and the loss factor, η_2, were being determined. Epoxy adhesives were used in constructing model structures (Figure 2b) or when the viscoelastic material had been cured prior to making the specimens. Specimens were also prepared from polymer solutions and aqueous suspensions by brush coating, spraying, troweling, caulking gun, casting, and dipping techniques.

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* Hughson Chemical Company trademark.

4. COMPOUNDING POLYMER FORMULATIONS

Experimental formulations consisted of physical mixtures of one, two, three, or four viscoelastic high polymers and selected additives or compounding ingredients. Three-polymer mixtures were the primary concern. The polymers and compounding ingredients were usually mixed together on a two-roll rubber mill but sometimes were mixed by solution or latex techniques. Compounding ingredients were added to improve the damping capability as well as to enhance the environmental resistance of the "control" three-polymer blend (Reference 14). The objective was to increase the modulus of a blend of a very high acrylonitrile rubber (Paracril-D*), polyvinyl acetate, and polystyrene without significantly decreasing the loss factor values. This was accomplished by altering the relative proportions of the polymers used in the blend and adding more curing agent and mineral fillers such as carbon black, graphite, and mica. Plasticizers were added to shift the temperature range of effective damping.

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* Uniroyal Trademark.

SECTION III

TEST RESULTS

1. COMPOUNDING INVESTIGATIONS

The 'Control" blend under development, a lightly crosslinked polymer blend consisting of equal parts by weight of acrylonitrile rubber, polyvinyl acetate, and polystyrene, along with 10 parts zinc oxide and 3 parts dicumyl peroxide per hundred parts rubber (phr), has been shown to have potential for wide-temperature-range damping applications (Reference 14). Theoretically, this formulation will give a high level of damping as a free-layer treatment when used on aluminum structures (References 8, 9, and 10). The modulus of this formulation was greater than 10^4 psi up to about 200°F, and the loss factor was greater than 0.1 from about 30 to 250°F. The loss factor curves had three distinct peaks, one for each polymer in the blend. The particular shape of the modulus vs temperature and the loss factor vs temperature curves suggests that the width of the temperature range where adequate damping can be obtained might be increased by changing the relative proportions and/or the kind of ingredients in the formulation.

a. Four-Component Blends

The formulation was first modified by adding another polymer having a glass transition temperature about 75°F below that for the nitrile rubber. This mixture consisted of 100 parts butyl rubber (Enjay 268)*, acrylonitrile rubber (Paracril-D), polyvinyl acetate, and polystyrene along with 10 parts zinc oxide and the curing agent SP-1055 resin.** The polyvinyl acetate and polystyrene were prepared according to the polymerization recipes in Table I (Reference 26). Adding butyl rubber and changing the curing agent resulted in maintaining $E_2 \geq 10^4$ psi at a temperature of up to about 110°F only, as shown in Figure 3, which was about 90°F lower than was obtained with the control blend. The loss factor curves, Figure 4, exhibited only three peaks when four

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* Enjay Chemical Company Trademark

** Schenectady Chemicals, Inc. Trademark were expected. This was because all measurements at and above 125°F were taken in an error region (as discussed in Reference 21) and the results were unreliable. Reliable data could have been obtained, but was not because of the relatively low modulus values, particularly at the higher temperatures. The low modulus values were attributed to the diluting effect of having equal amounts of four polymers with widely separated glass transition temperatures in the blend. Since the three-polymer blend has higher modulus values at higher temperatures, it would be better for free-layer damping applications. Therefore, the experimental effort was directed toward developing it into a practical formulation for Air Force applications.

TABLE I

POLYMERIZATION RECIPES

Polyvinyl Acetate

Water	500 ml
Vinyl Acetate	340 ml
Soap	5.0 gm
Potassium Persulfate	1.5 gm

Ran for 3 hours at 65°C; air dried reaction mixture.

Polystyrene

Water	500 ml
Styrene	300 ml
Soap	5.0 gm
Potassium Persulfate	1.5 gm

Ran for 4 to 8 hours at 50°C; acid precipitated, filtered, and dried in vacuum oven at 50°C.

b. Three-Component Blend Modifications

One practical objective of the experiments was to alter the proportions of the polymers in the three-polymer blend so that all three loss factor peaks would be nearly the same height. Since the acrylonitrile rubber and polystyrene peaks were already about the same height, and the polyvinyl acetate peak was higher (Reference 14), we decided to vary the amount of polyvinyl acetate.

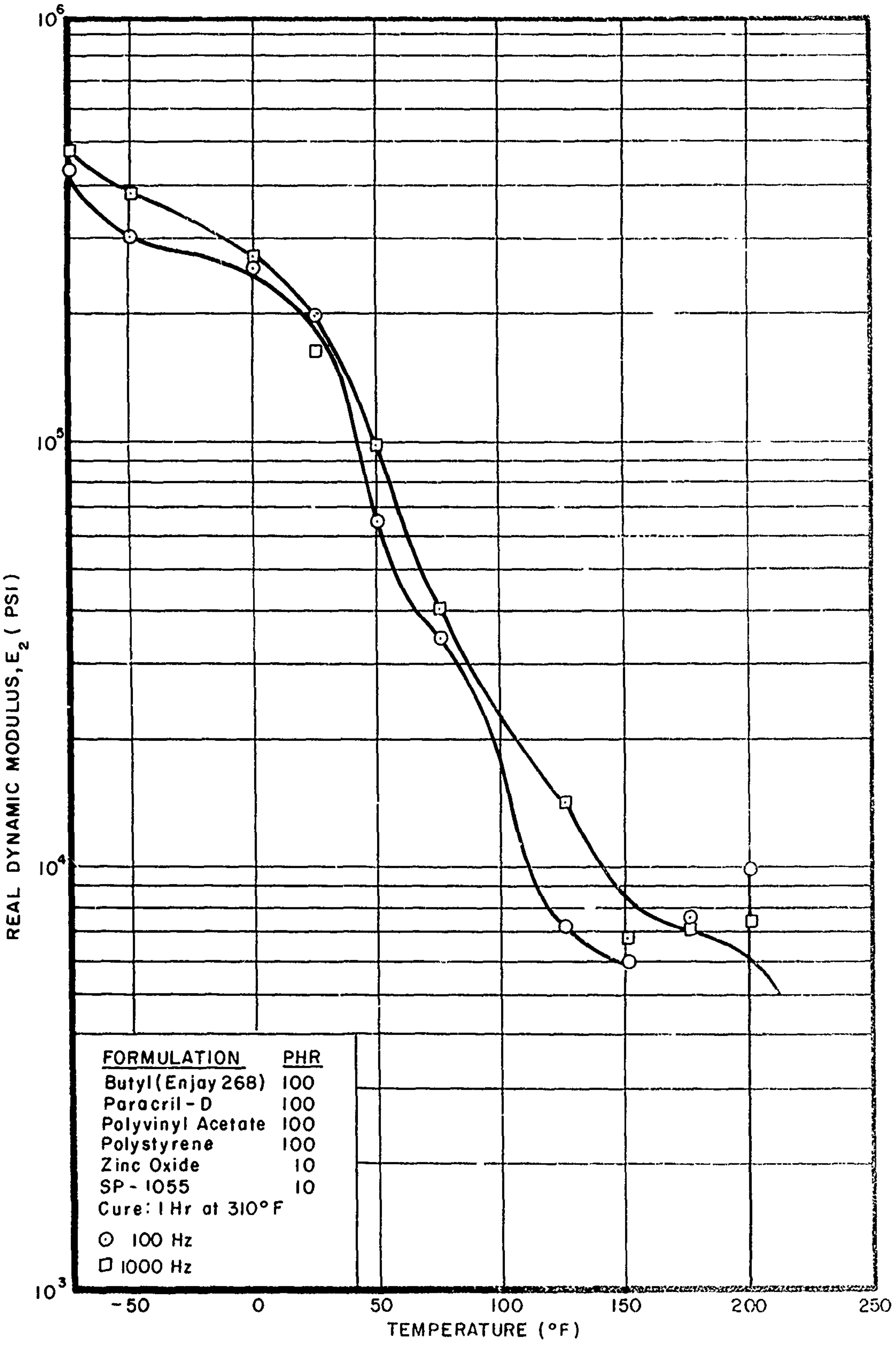

Figure 3. Real Dynamic Modulus vs Temperature of a Four-Polymer Blend

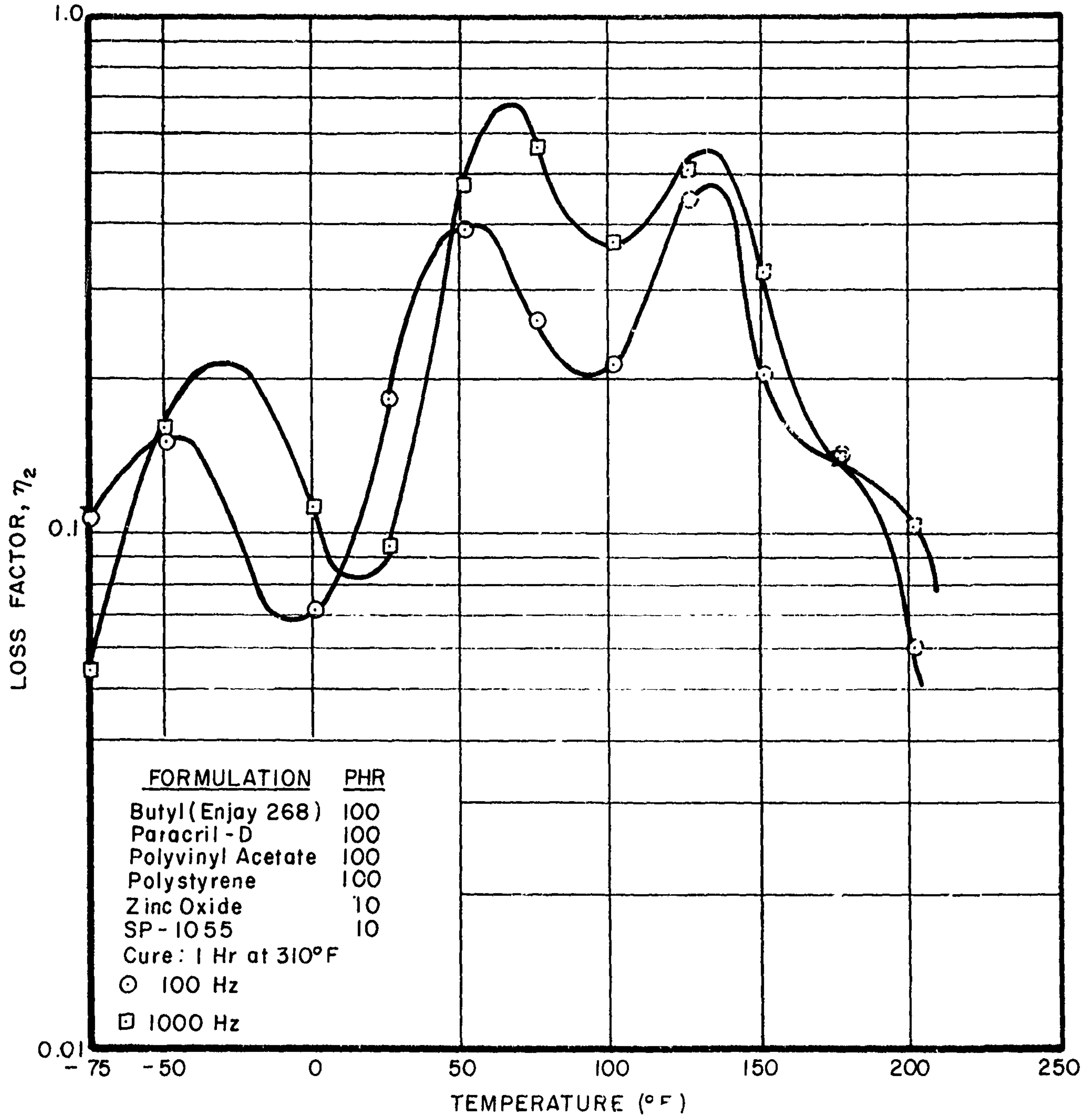

Figure 4. Loss Factor vs Temperature of a Four-Polymer Blend

Formulations consisting of 100 parts (in parts by weight) of acrylonitrile rubber (Paracril-D) and polystyrene, 10 parts zinc oxide, and 3 parts dicumyl peroxide were mixed with 0, 10, 20, 40, 50, 60, 80, and 100 parts polyvinyl acetate. The modulus and loss factor values measured at 100 and 1000 Hz are shown in Figures 5 through 20.

Generally, the results agree with linear viscoelastic theory (References 18 and 25) even though the modulus and loss factor values at temperatures above 180°F do not appear to be consistent with the amount of polyvinyl acetate. The modulus and loss factor values at temperatures above 180°F, however, are due primarily to the polystyrene rather than to the polyvinyl acetate. Thus, the variations in the high-temperature modulus values must be due to either the the polystyrene preparation or the experimental method. A careful analysis of the results showed the high-temperature modulus values varied with variations in conditions used in preparing the relatively small batches of polystyrene from monomer in the laboratory. Conditions that led to low molecular weight and highly branched polymer chains produced high-temperature modulus values that were consistently low, and conditions that led to linear polystyrene of high molecular weight produced high-temperature modulus values that were consistently high. These large variations in the modulus values were later avoided by using a commercial grade of polystyrene.

The loss factor curves show that the relative height of the polyvinyl acetate peaks occurring at about 125°F varies with the relative amount of polyvinyl used in the blend and that these variations alter the relative heights of the acrylonitrile rubber and polystyrene peaks occurring at about 50°F and 230°F, respectively. As expected, increasing the amount of polyvinyl acetate resulted in reducing the peak loss factor values of both the polystyrene and acrylonitrile rubber and produced a third peak. All three loss factor peaks were about the same height for formulations containing 40, 50, or 60 parts polyvinyl acetate (Figures 12, 14, and 16.) The formulation containing 50 parts polyvinyl acetate was considered to be the most desirable for practical free-layer damping formulations, partly because of the high-temperature modulus values (Figures 11, 13, and 15) and partly because slight variations in the amount of polyvinyl acetate would not greatly influence the internal losses of the damping treatment.

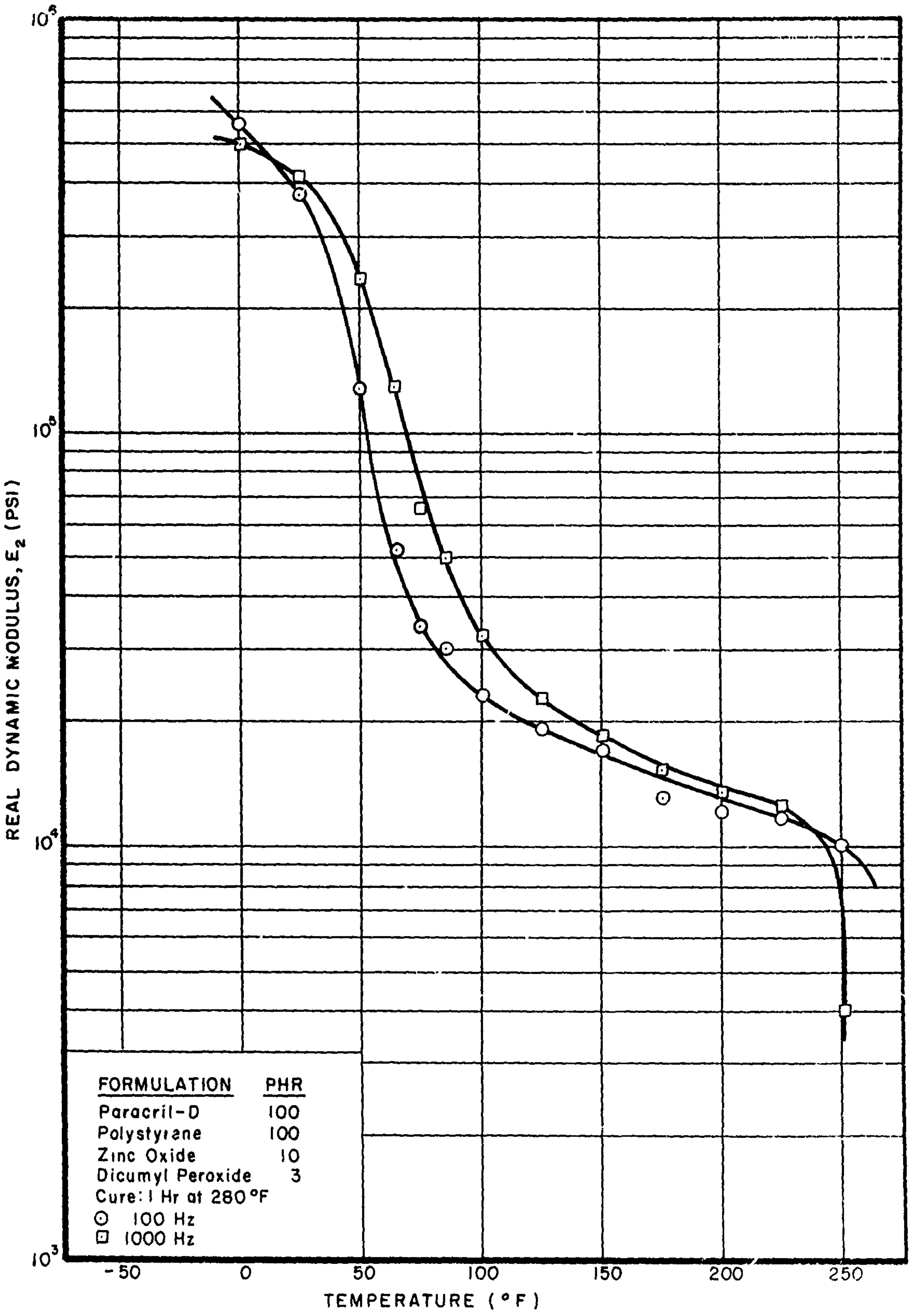

Figure 5. Real Dynamic Modulus vs Temperature of a Two-Polymer Blend

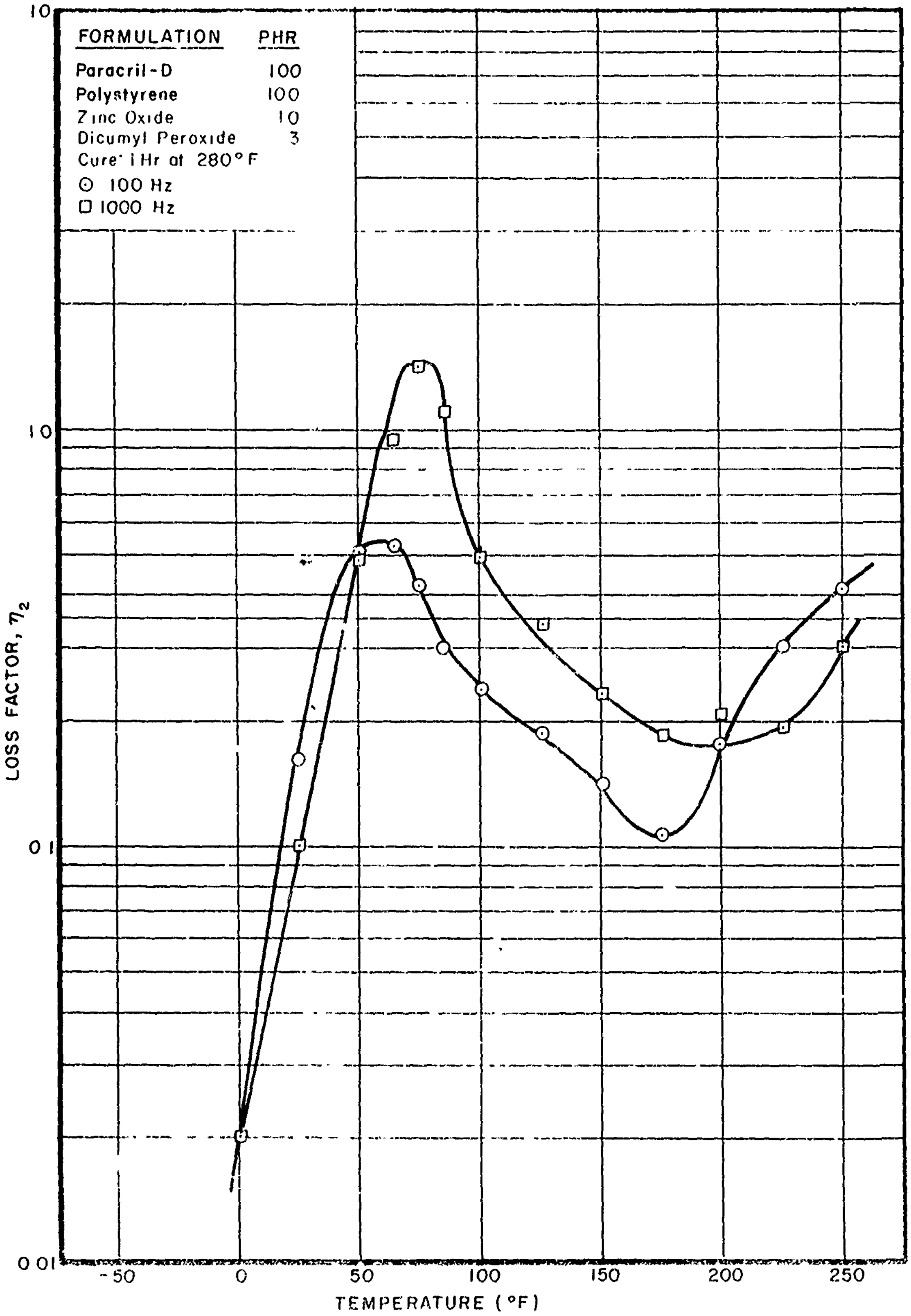

Figure 6. Loss Factor vs Temperature of a Two-Polymer Blend

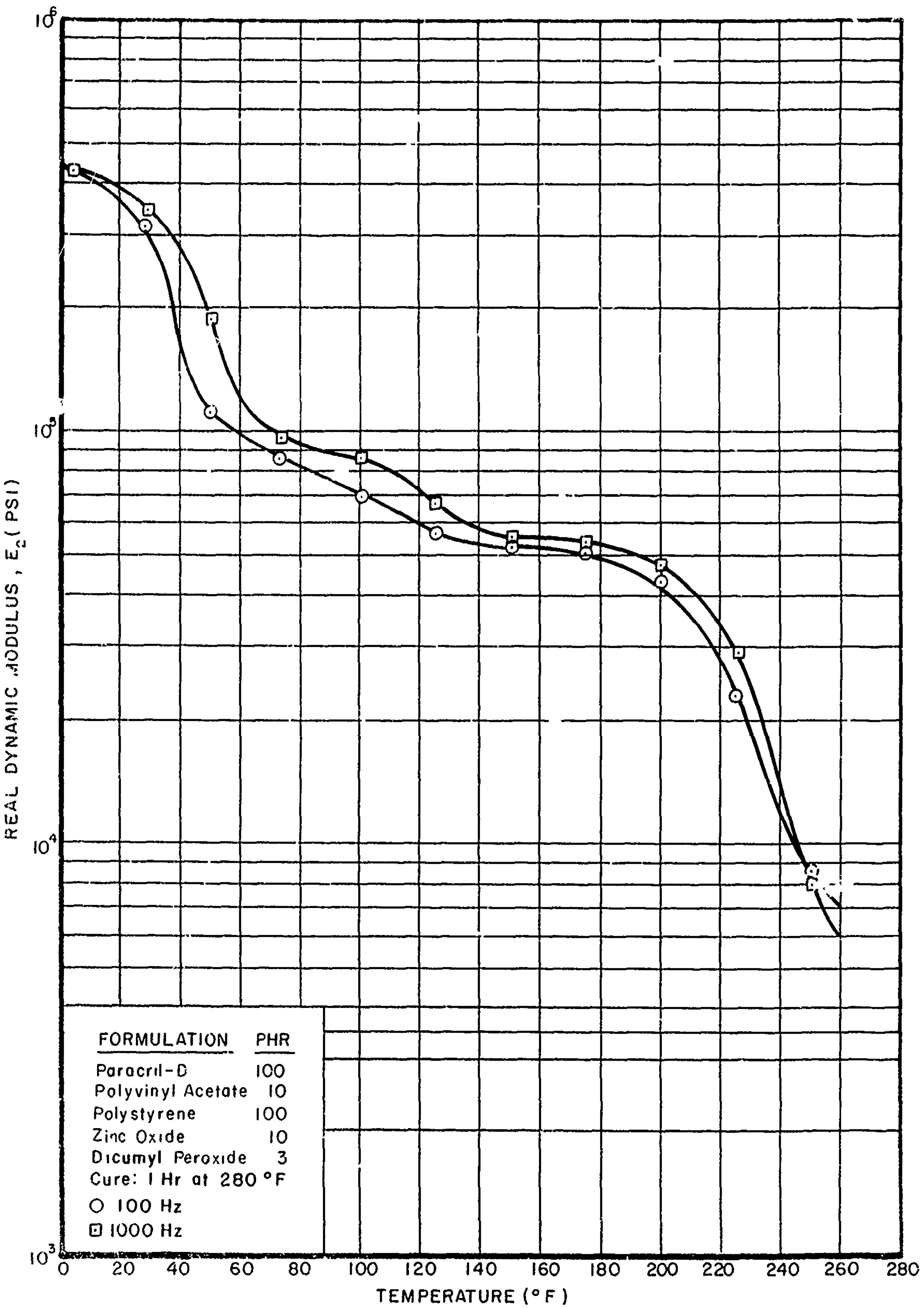

Figure 7. Real Dynamic Modulus vs Temperature of the Two-Polymer Blend With 10 Parts Polyvinyl Acetate Added

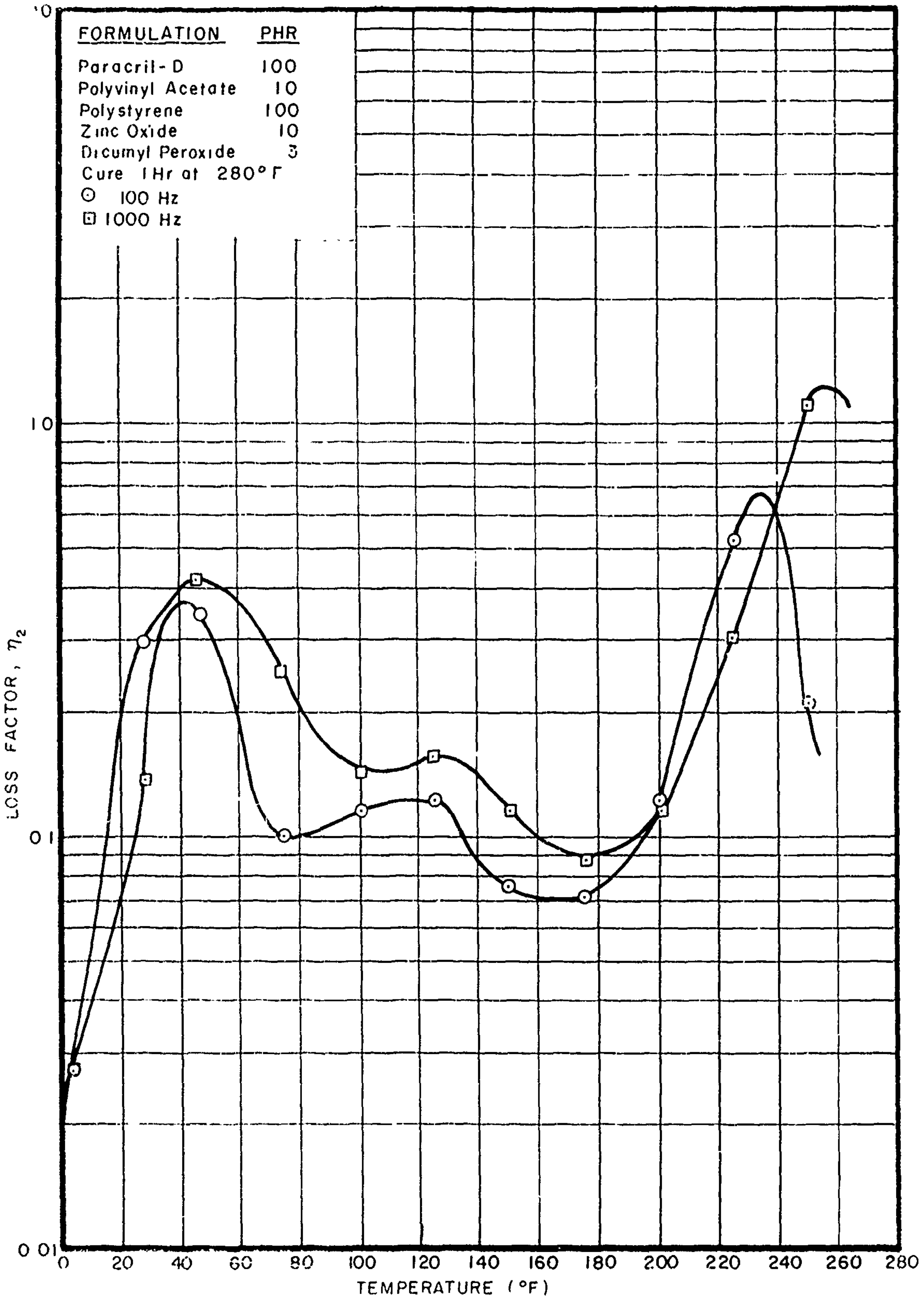

Figure 8. Loss Factor vs Temperature of the Two-Polymer Blend With 10 Parts Polyvinyl Acetate Added

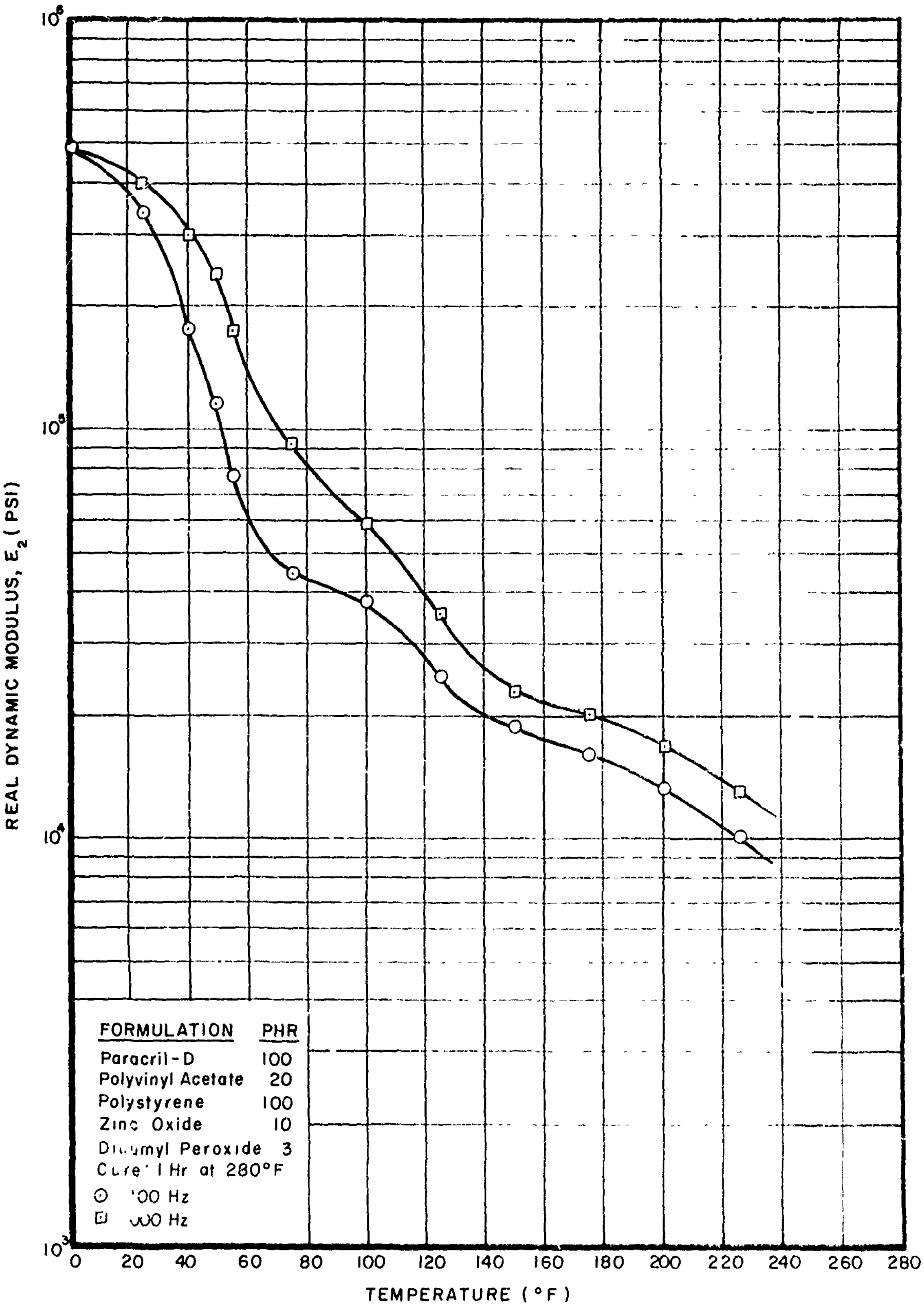

Figure 9. Real Dynamic Modulus vs Temperature of the Two-Polymer Blend With 20 Parts Polyvinyl Acetate Added

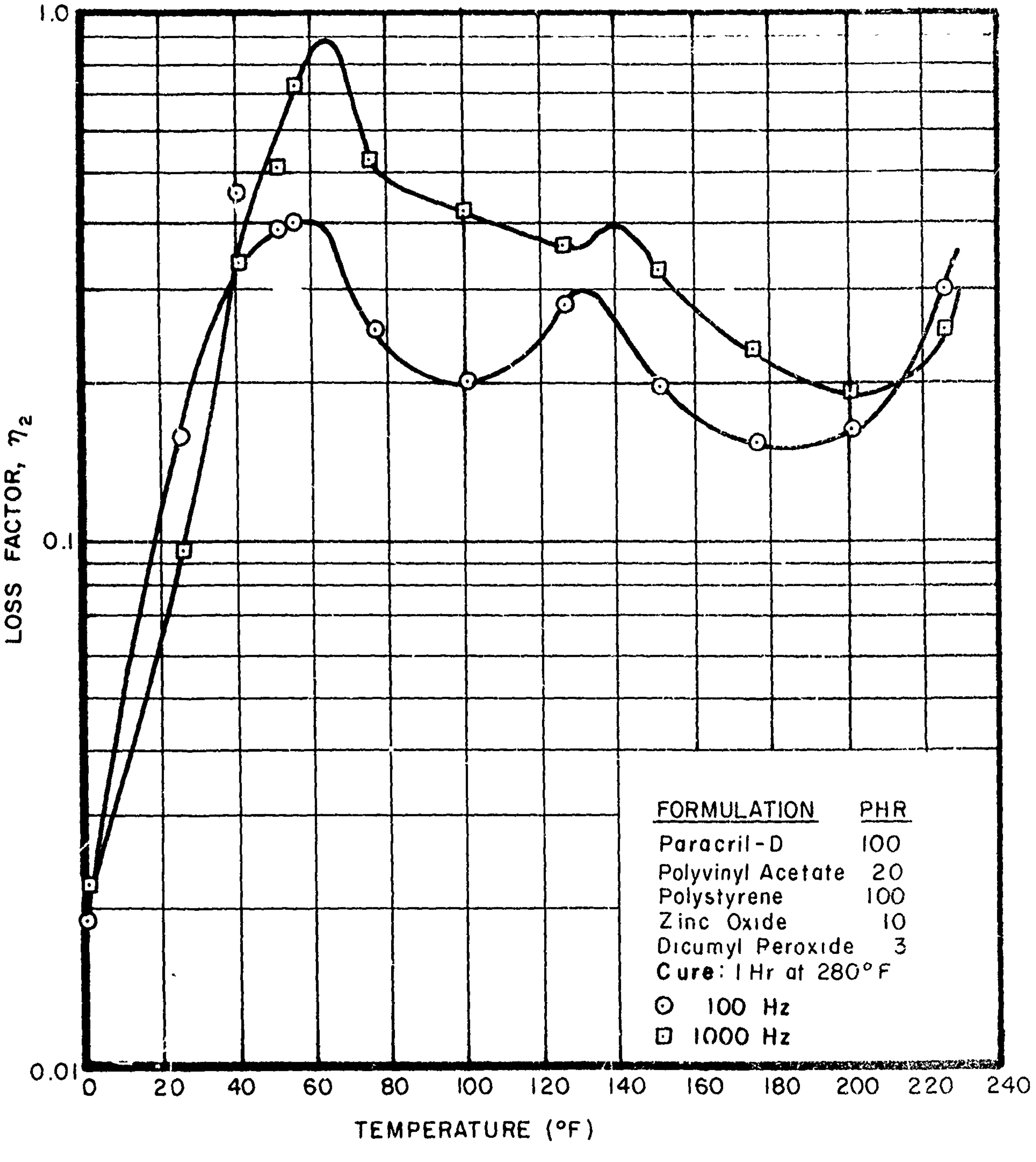

Figure 10. Loss Factor vs Temperature of the Two-Polymer Blend With 20 Parts Polyvinyl Acetate Added

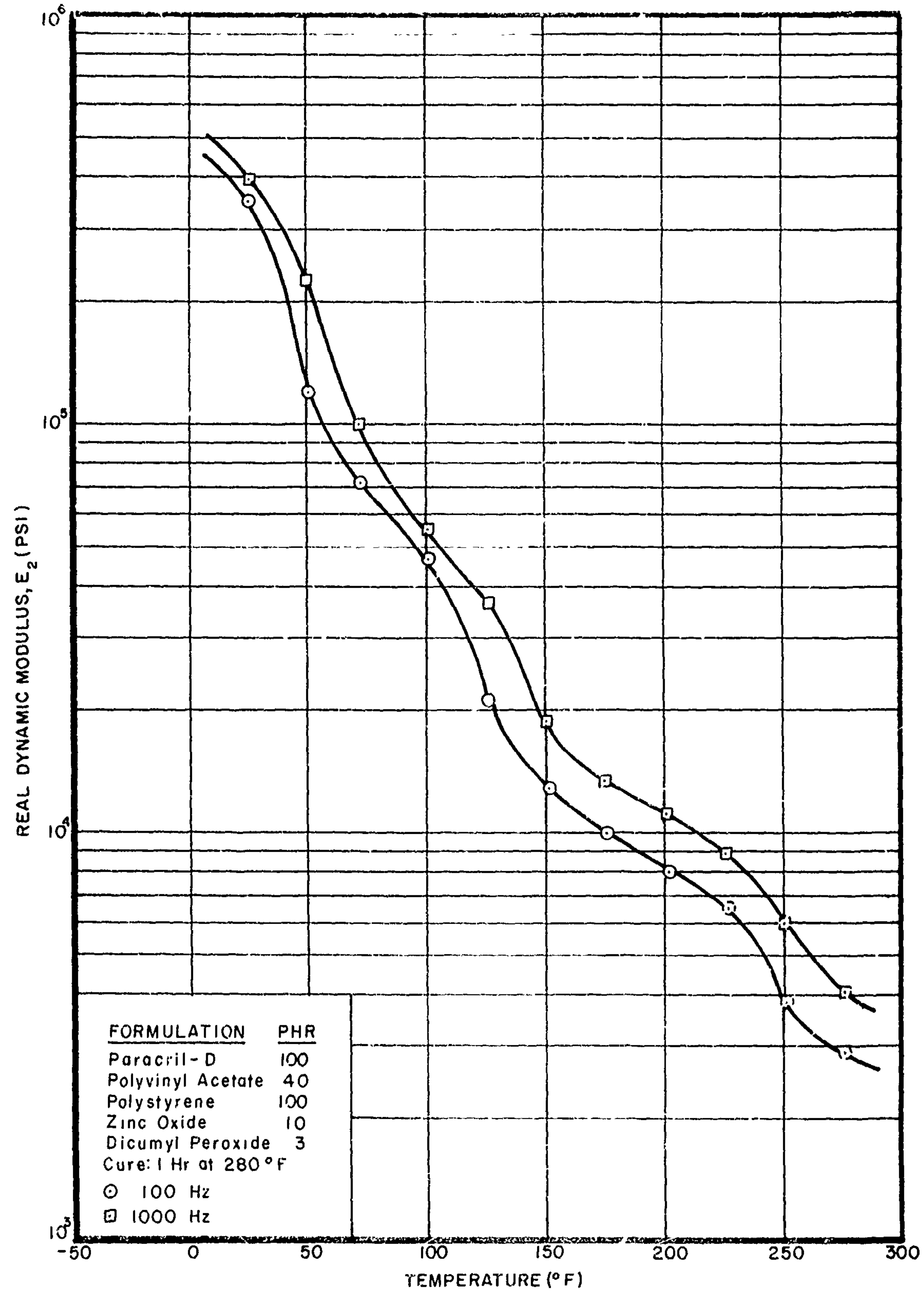

Figure 11. Real Dynamic Modulus vs Temperature of the Two-Polymer Blend With 40 Parts Polyvinyl Acetate Added

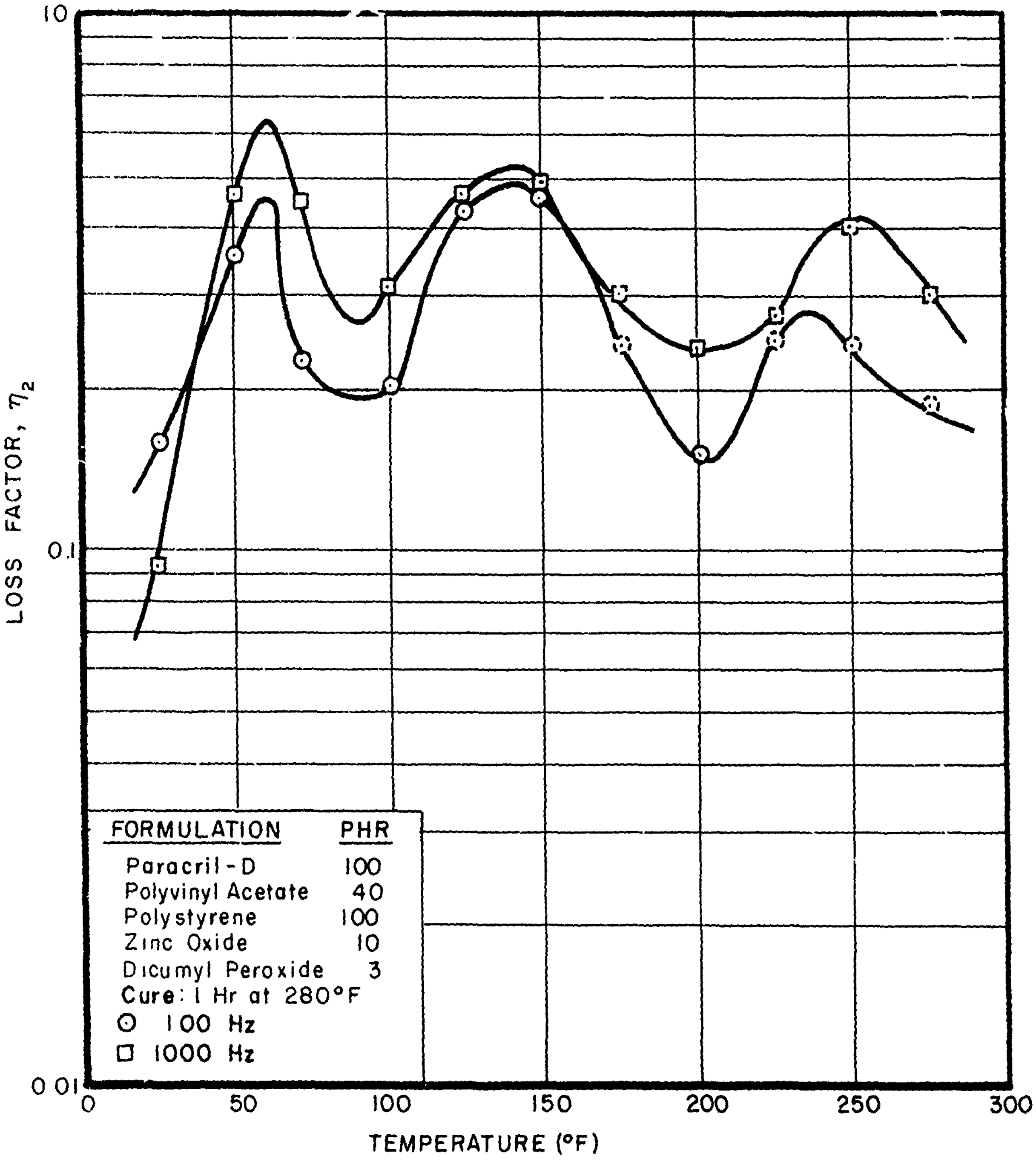

Figure 12. Loss Factor vs Temperature of the Two-Polymer Blend With 40 Parts Polyvinyl Acetate Added

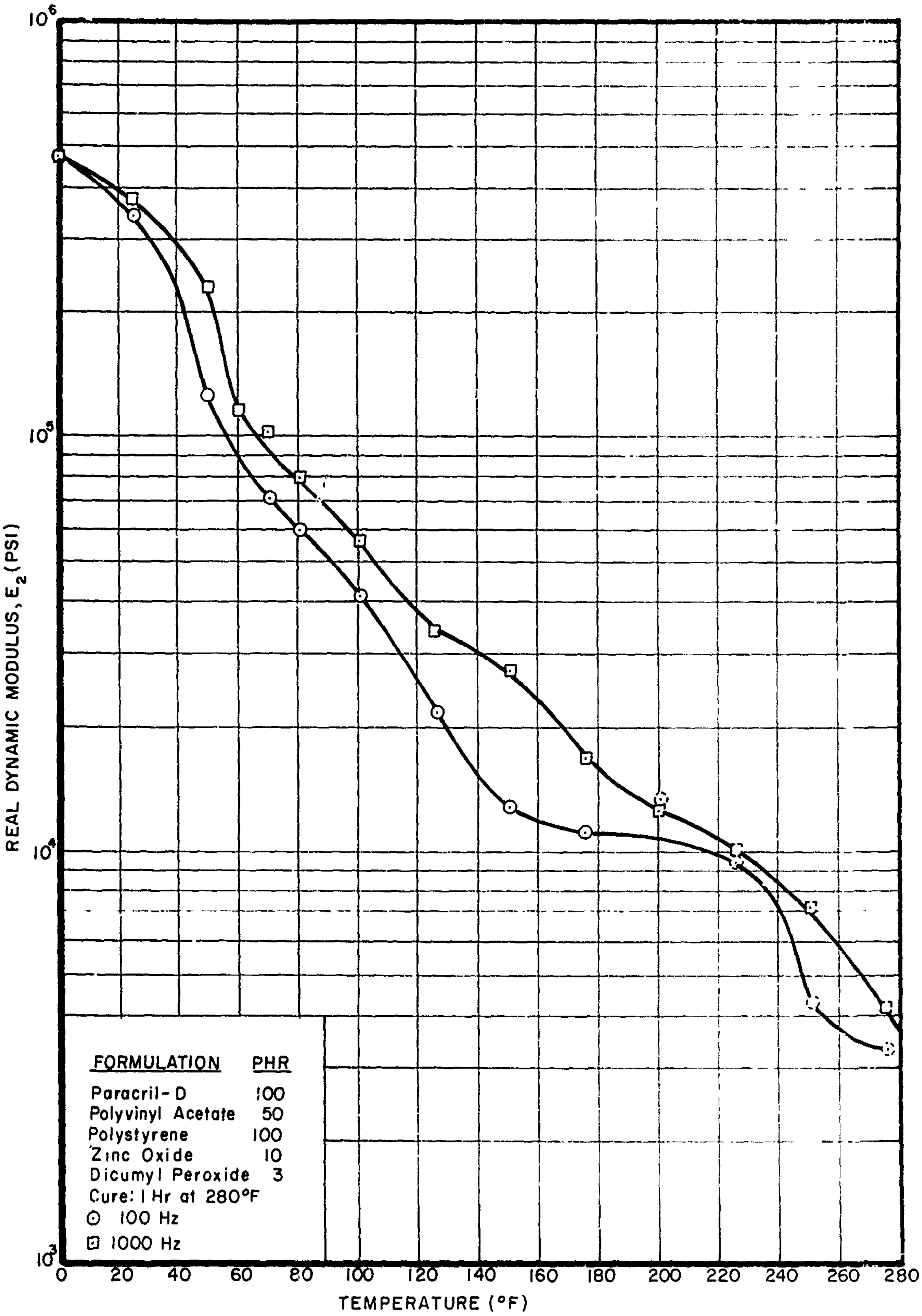

Figure 13. Real Dynamic Modulus vs Temperature of the Two-Polymer Blend With 50 Parts Polyvinyl Acetate Added

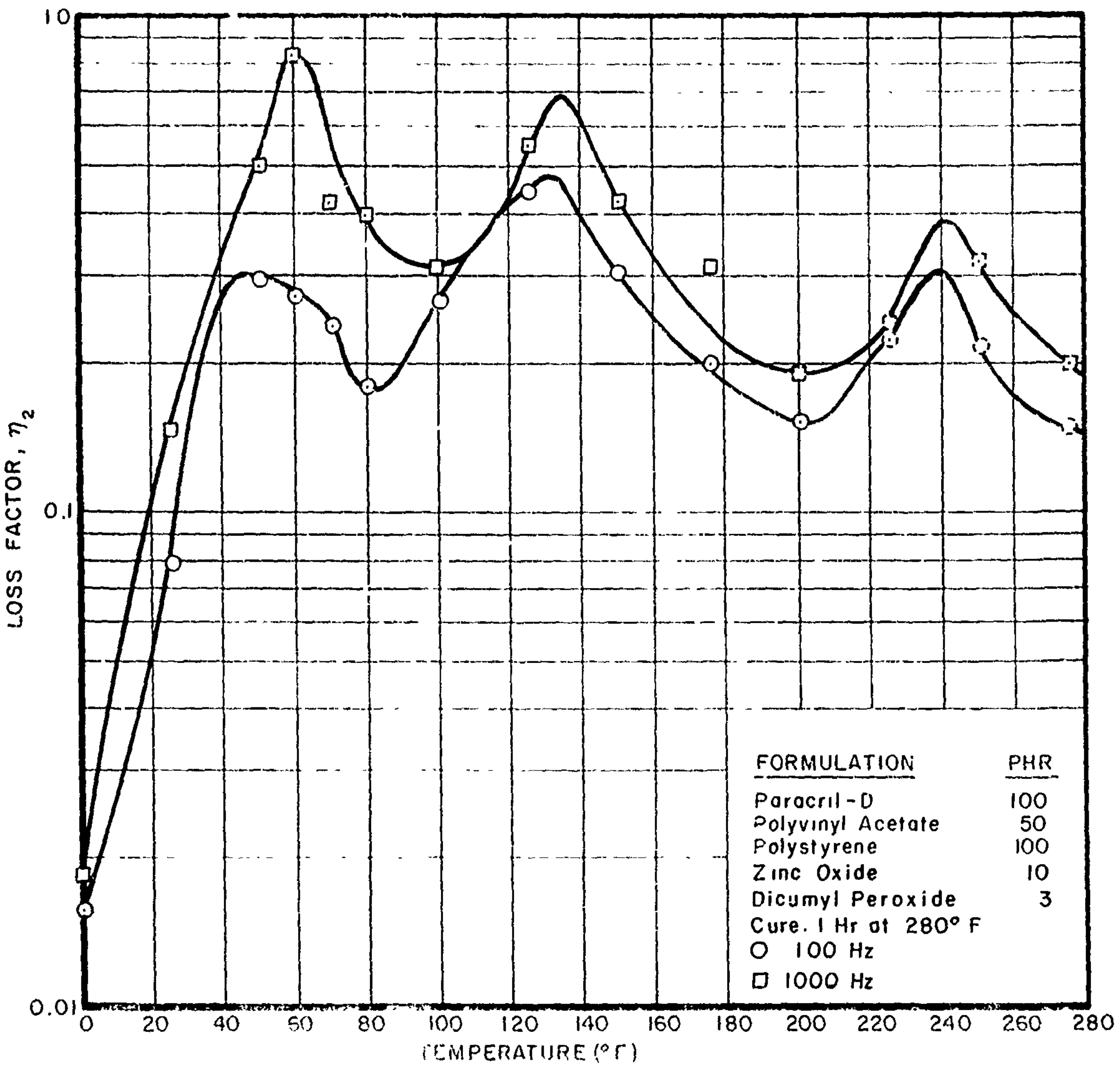

Figure Loss Factor vs Temperature of the Two-Polymer Blend With 50 Parts Polyvinyl Acetate Added

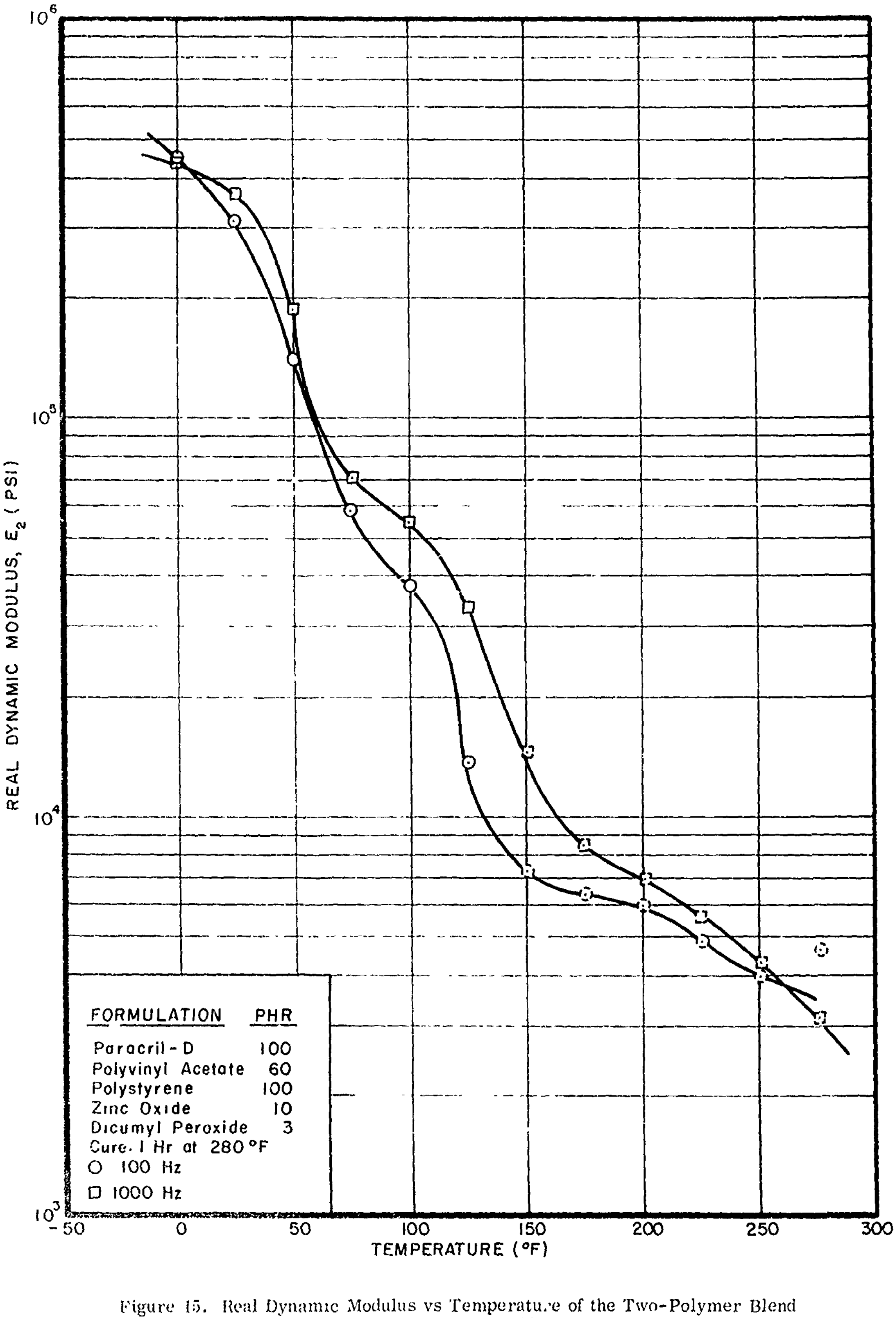

Figure 15. Real Dynamic Modulus vs Temperature of the Two-Polymer Blend With 60 Parts Polyvinyl Acetate Added

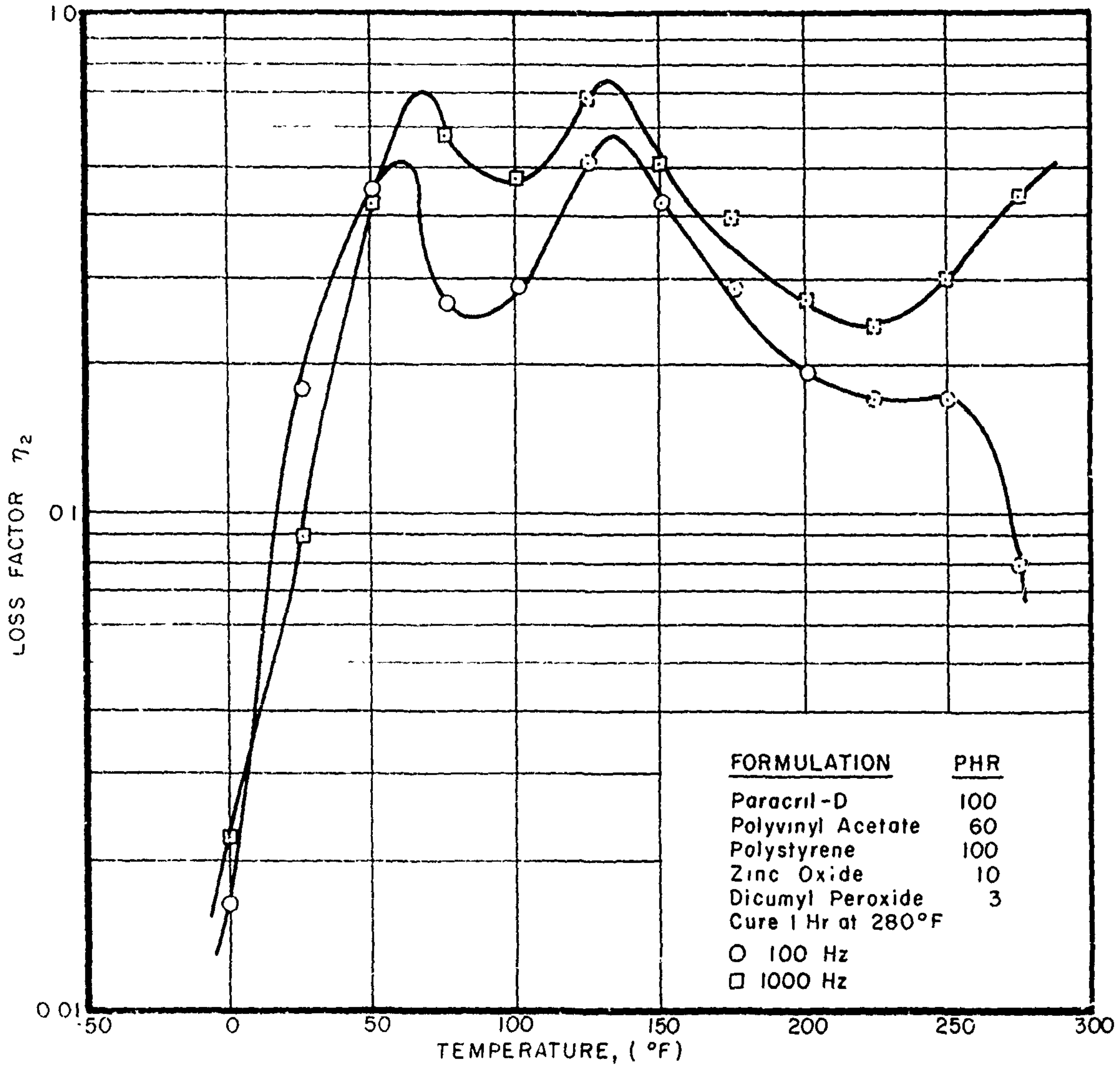

Figure 16. Loss Factor vs Temperature of the Two-Polymer Blend With 60 Parts Polyvinyl Acetate Added

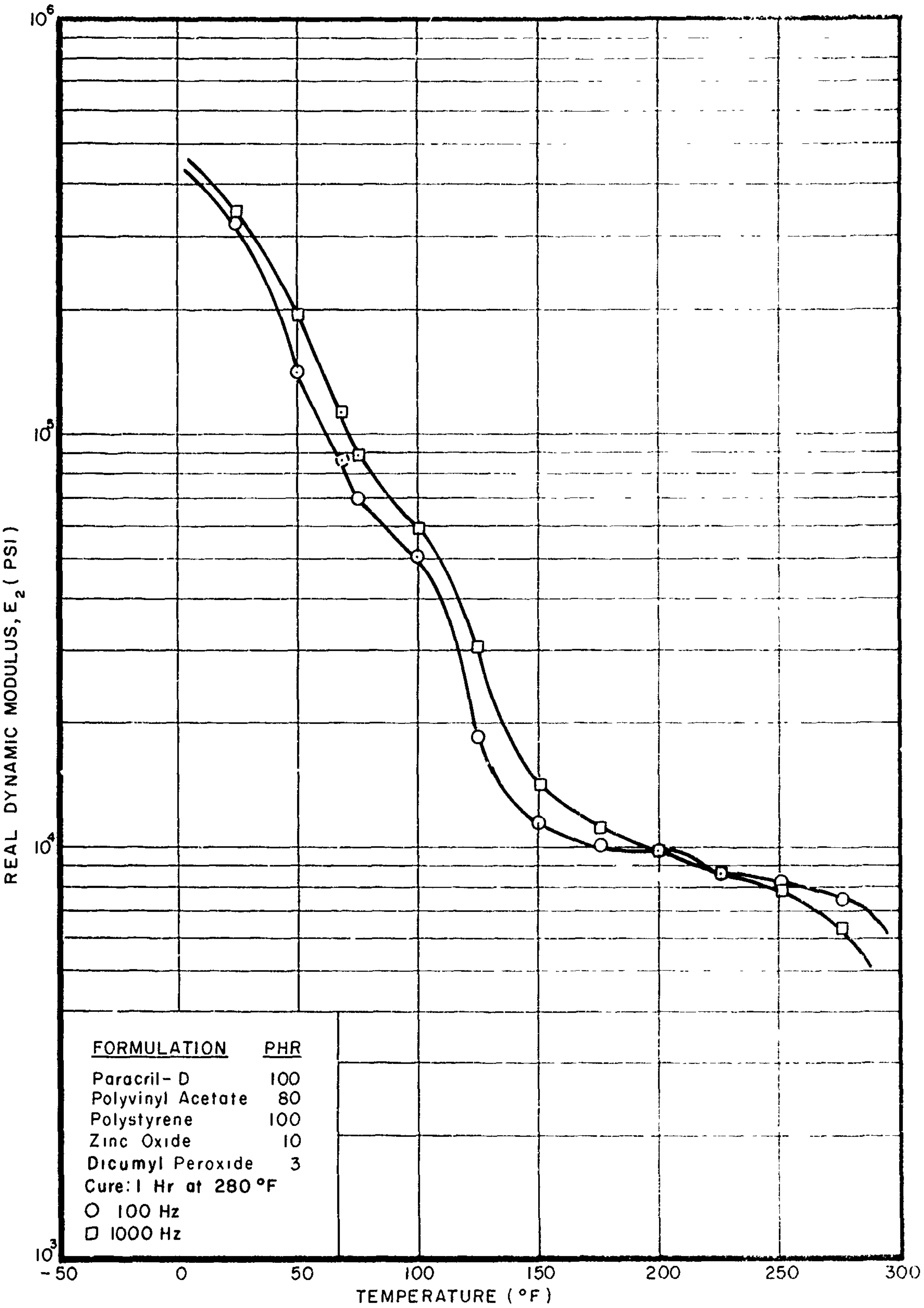

Figure 17. Real Dynamic Modulus vs Temperature of the Two-Polymer Blend With 80 Parts Polyvinyl Acetate Added

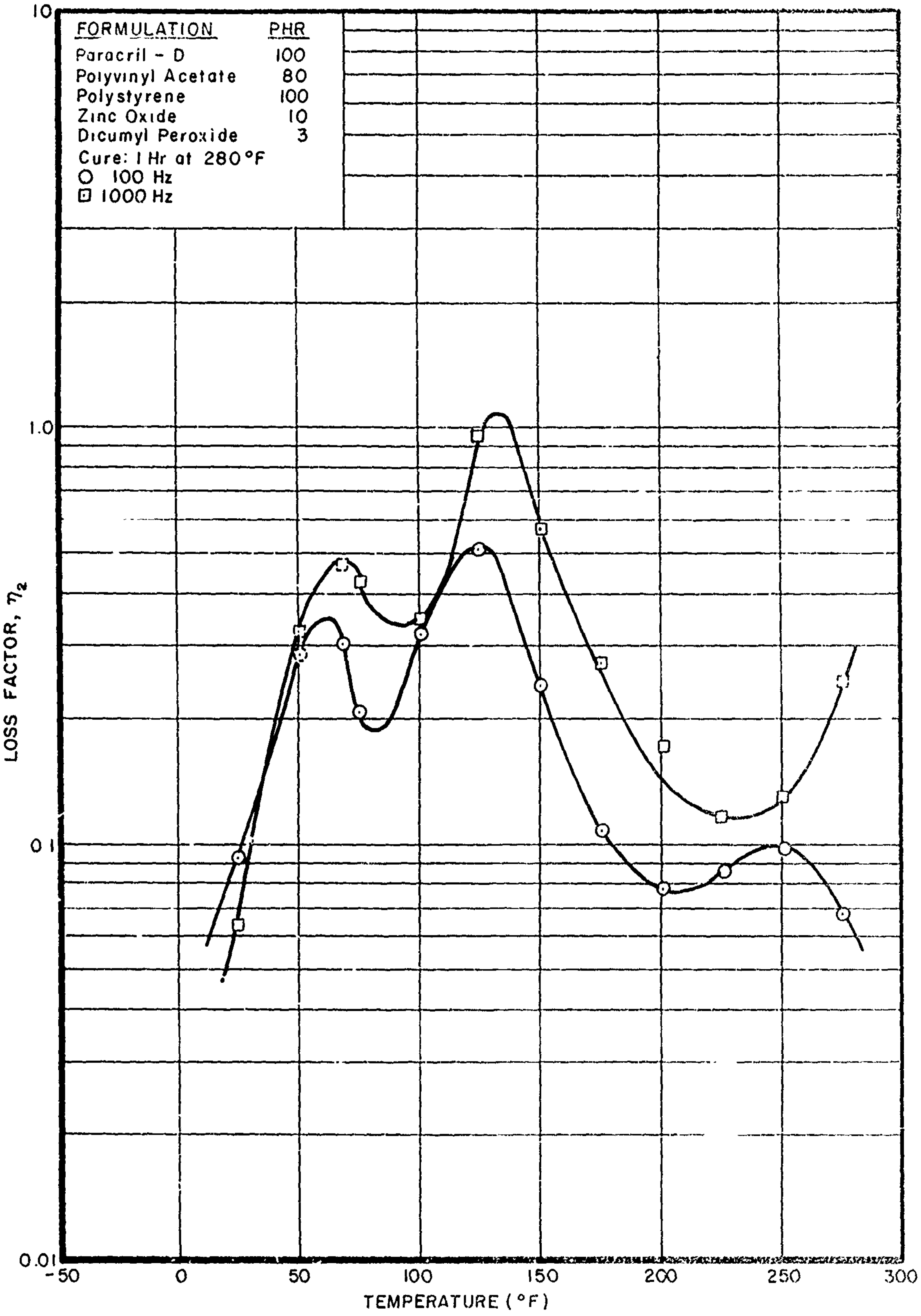

Figure 18. Loss Factor vs Temperature of the Two-Polymer Blend With 80 Parts Polyvinyl Acetate Added

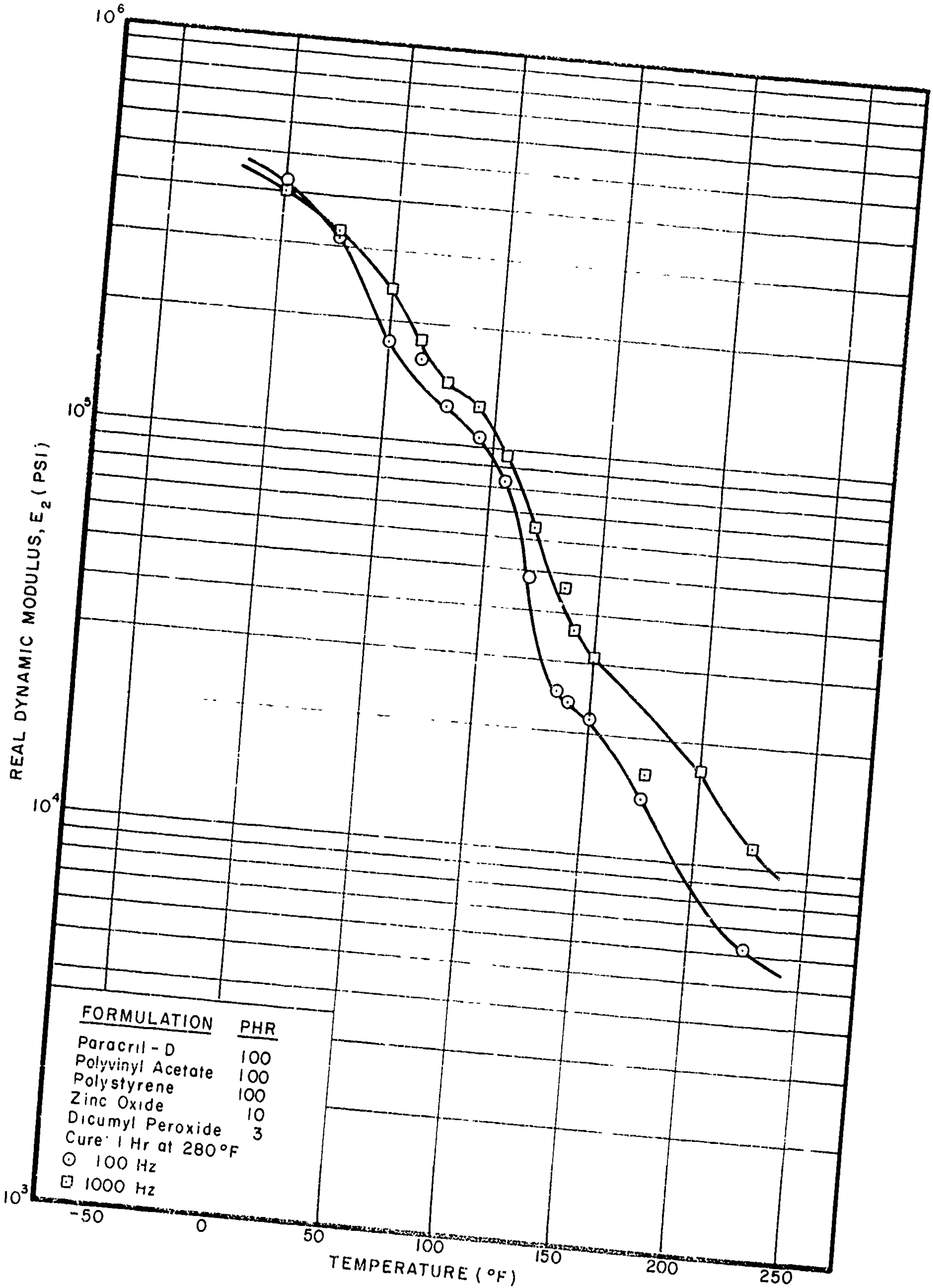

Figure 19. Real Dynamic Modulus vs Temperature of the "Control" Blend

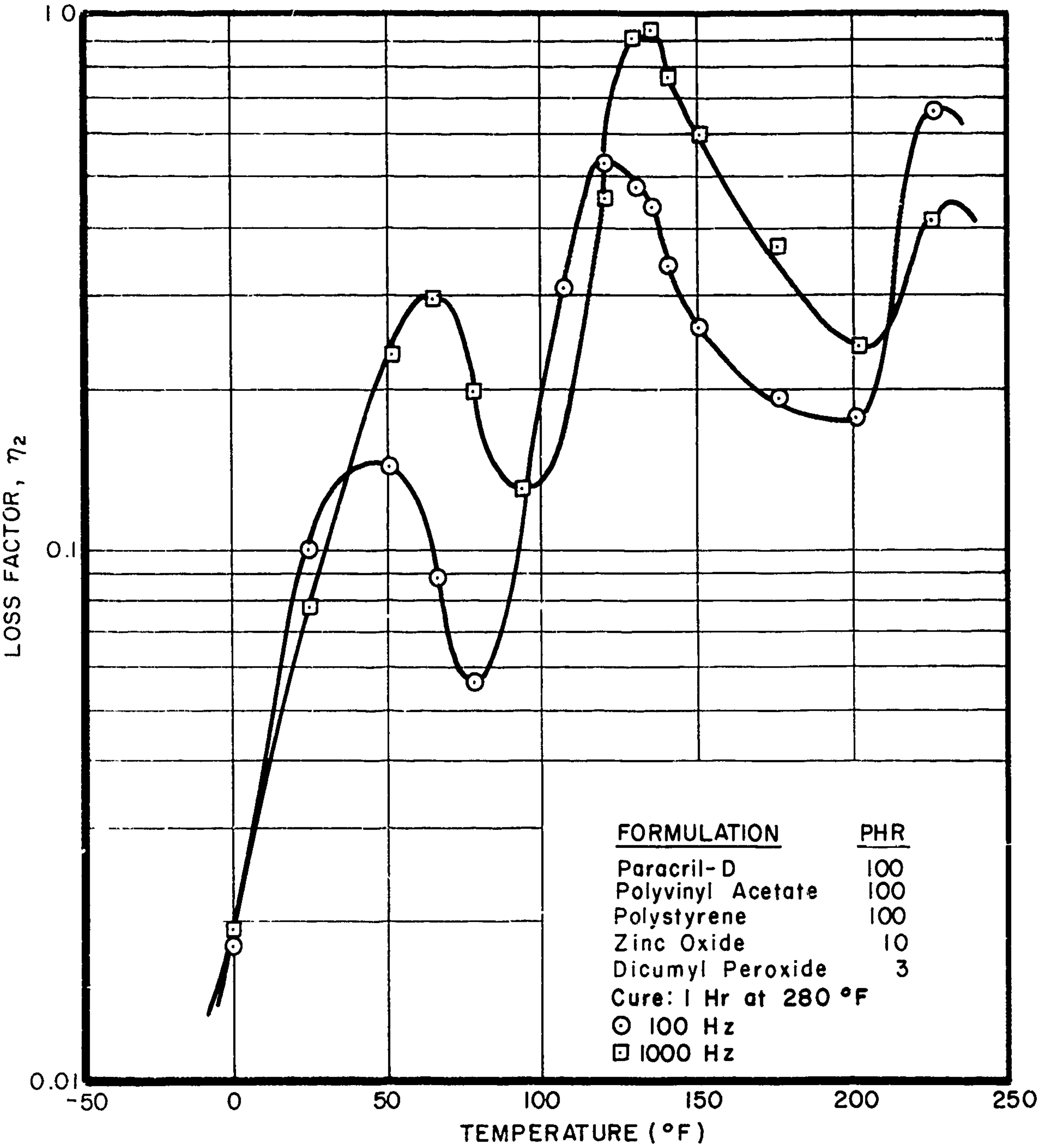

Figure 20. Loss Factor vs Temperature of the "Control" Blend

Aside from finding a very appropriate three-polymer blend for free-layer damping, these experiments also showed that the relative height of a particular loss factor peak depends, though not perfectly, on the relative amount of polymer giving rise to the particular peak. Of course, this applies only when the polymers are mutually insoluble and exist in separate intermingling phases (Reference 18).

c. Carbon Black Variations

Once the desired heights in loss factor peaks were obtained, the next step involved adjusting the modulus values, particularly at the higher temperatures, without significantly reducing the loss factor values. One way of increasing the modulus of a material exhibiting rubber-like behavior at a given fixed frequency and temperature is to add reinforcing mineral fillers (References 18, 25, and 27). The amount by which the modulus can be increased is usually very limited. SAF (super abrasion furnace) carbon black was used because it is one of the more efficient fillers for nitrile rubber (Reference 27). Antioxidant 2246* was added first to prevent oxidative degradation of the acrylonitrile rubber at the higher temperatures. Adding 1 phr, as shown in Figures 21 and 22, had little or no effect on the dynamic mechanical properties of the three-polymer blend, but it prevented high-temperature discoloration, which had been observed previously.

The experimental formulations consisted of 100 parts each (by weight) of acrylonitrile rubber and polystyrene, 50 parts of polyvinyl acetate, 10 parts of zinc oxide, 3 parts of dicumyl peroxide, and 1 part of Antioxidant 2246 mixed with 0, 10, 20, 30, 40, 50, and 60 parts of SAF Carbon black. Figures 21 through 34 show the measured modulus and loss factor values. The emulsion polymerization of the vinyl acetate and styrene (References 14 and 26) were carefully controlled, so that the high-temperature modulus values were consistently high with little variation. The modulus and loss factor values did not vary greatly with changes in the SAF carbon black content. The high-temperature modulus increased in increments consistent with the amount of carbon black up to 30 parts, remained essentially constant for formulations containing 30, 40, and 50 parts, and

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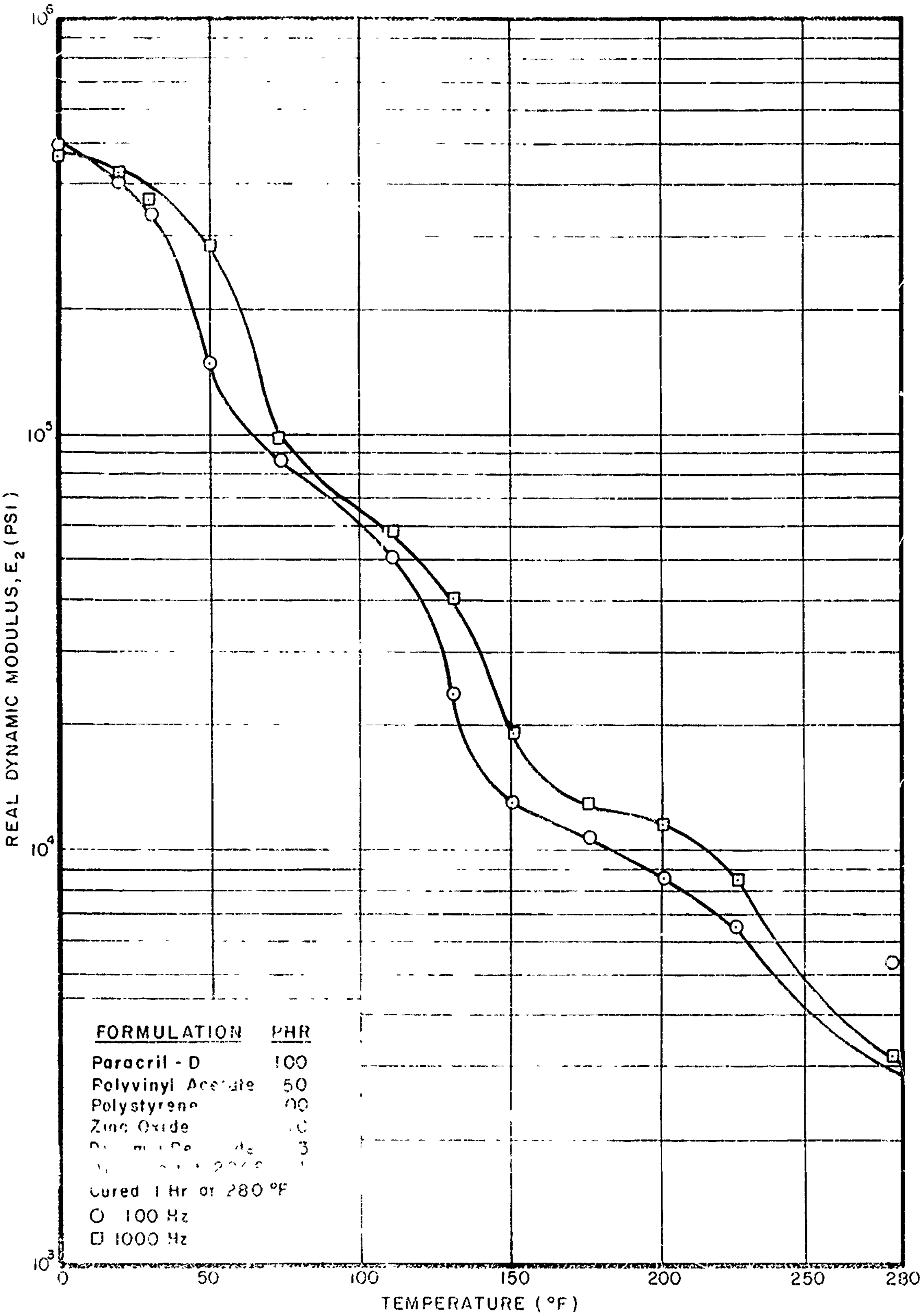


Figure 21. Real Dynamic Modulus vs Temperature of a Potential Damping Material With Antioxidant Added

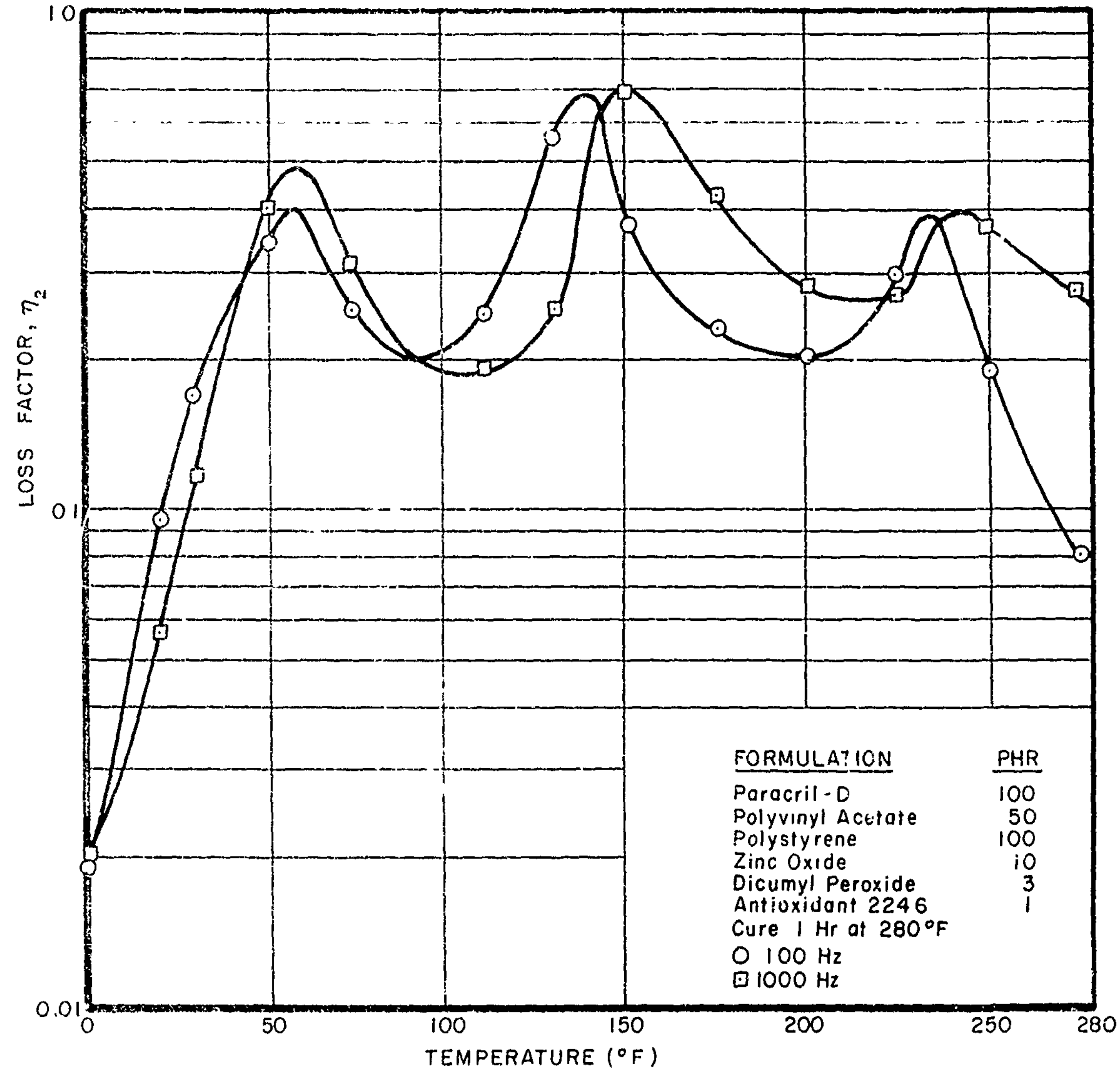


Figure 22. Loss Factor vs Temperature of a Potential Damping Material With Antioxidant Added

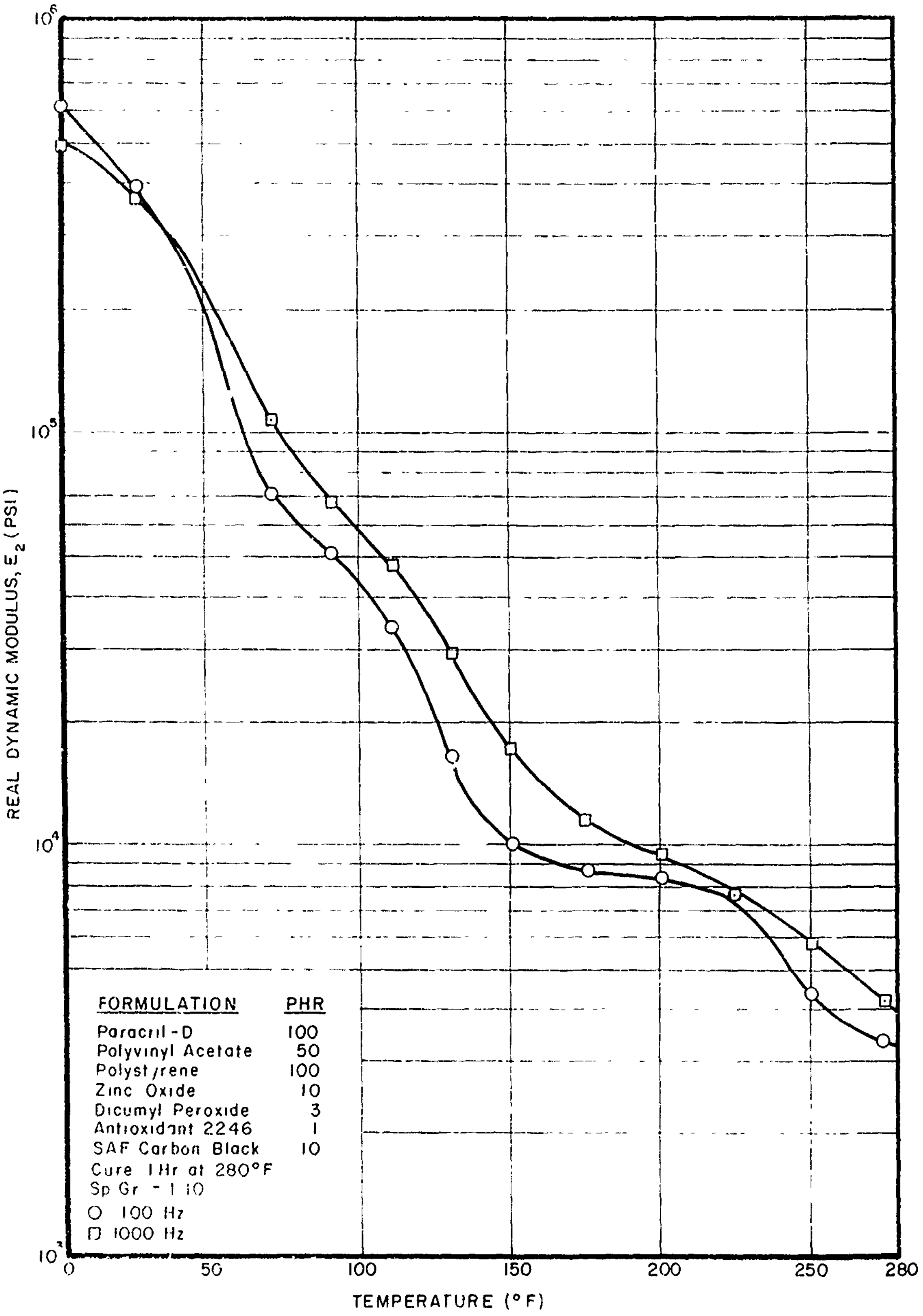


Figure 23. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 10 Parts SAF Carbon Black Added

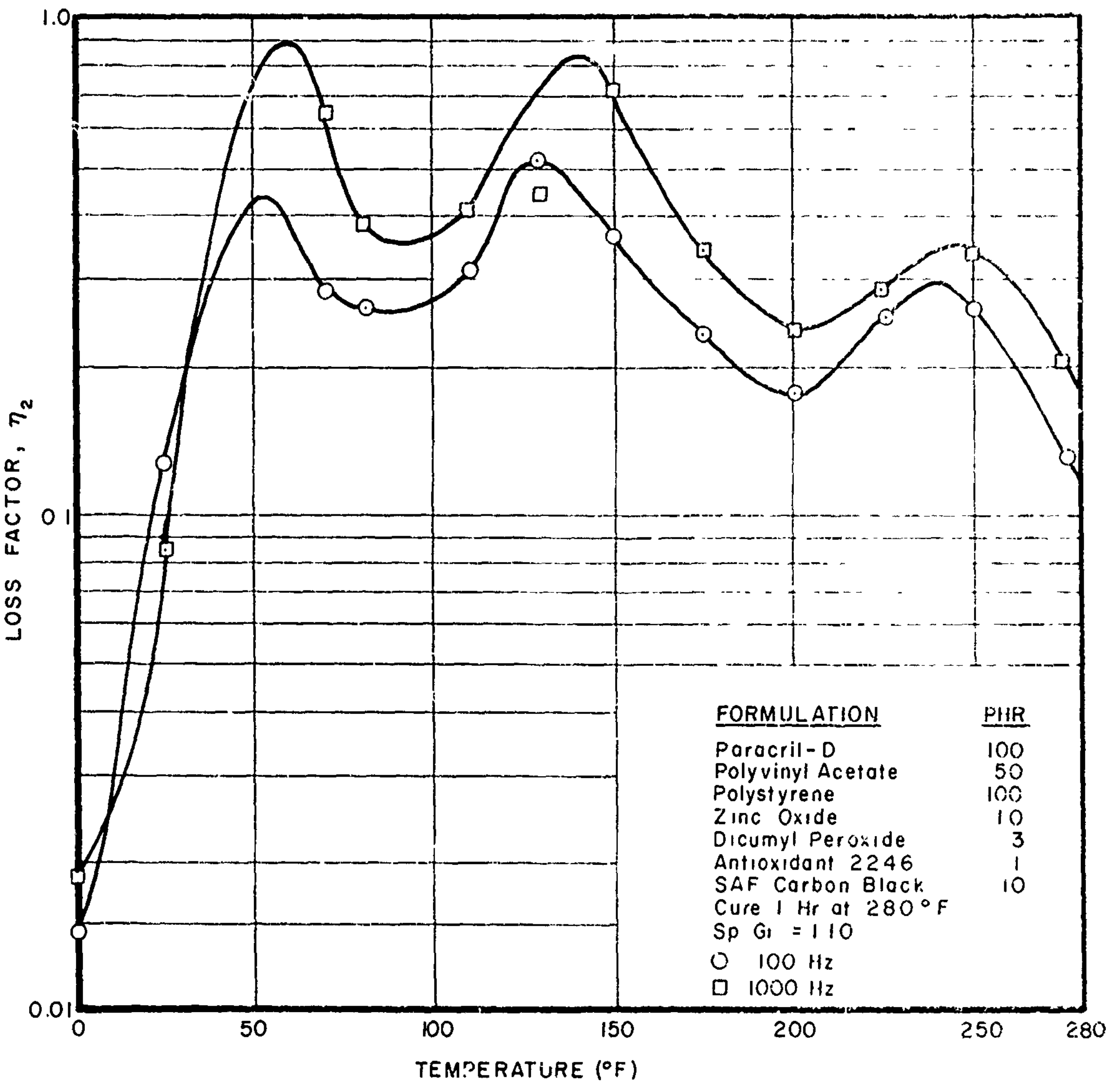


Figure 21. Loss Factor vs Temperature of a Potential Damping Material With 10 Parts SAF Carbon Black Added

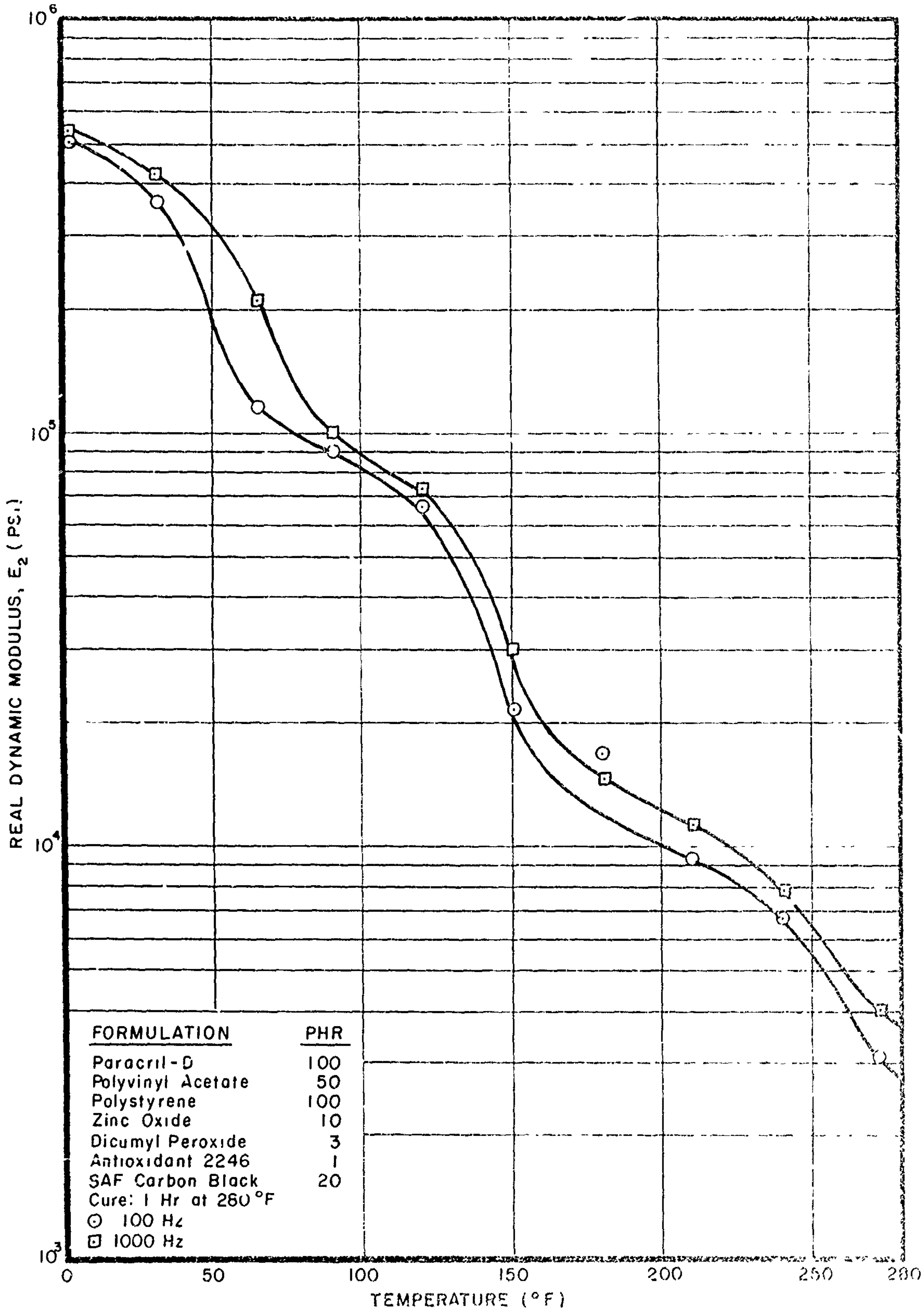


Figure 25. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 20 Parts SAF Carbon Black Added

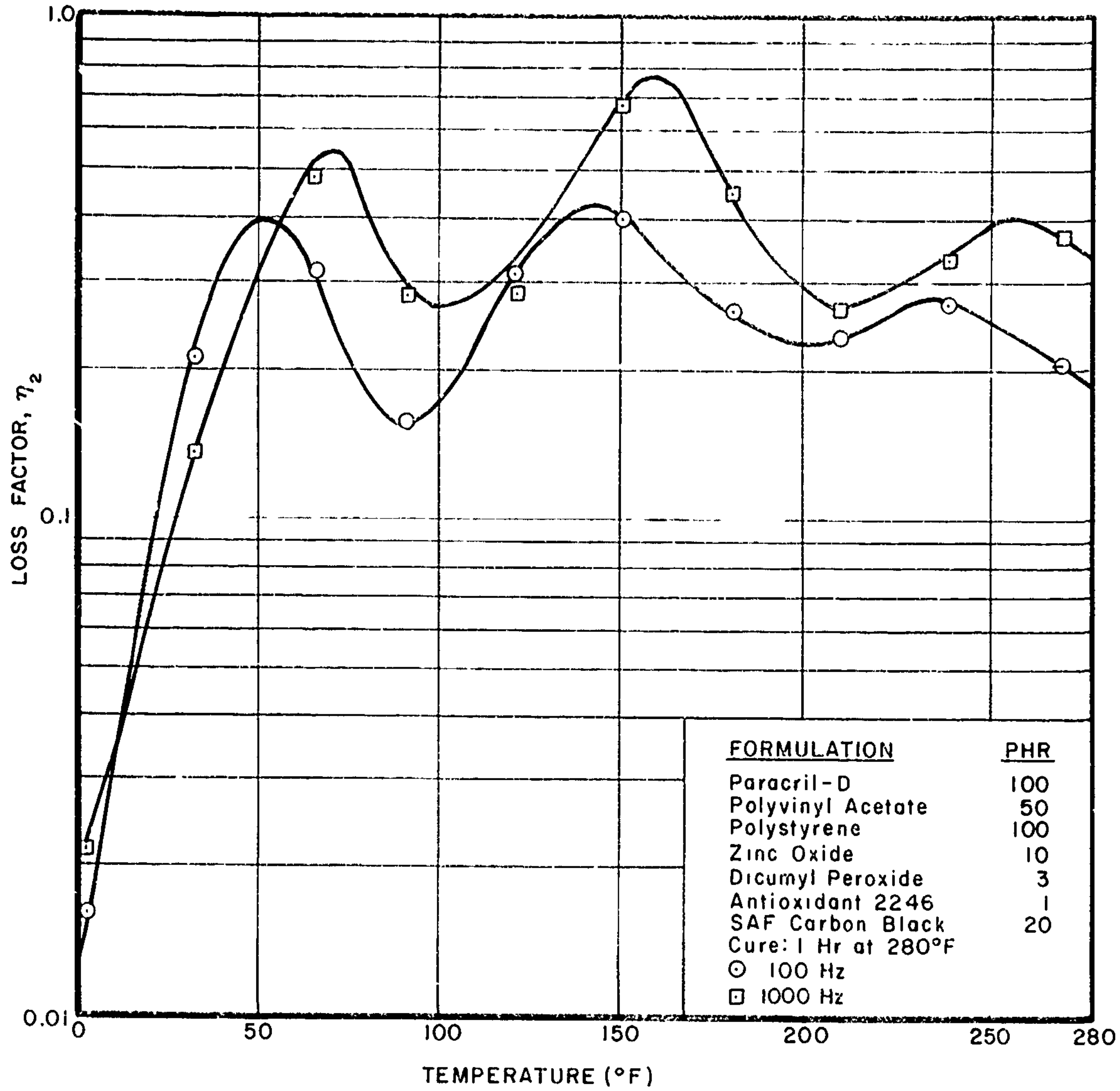
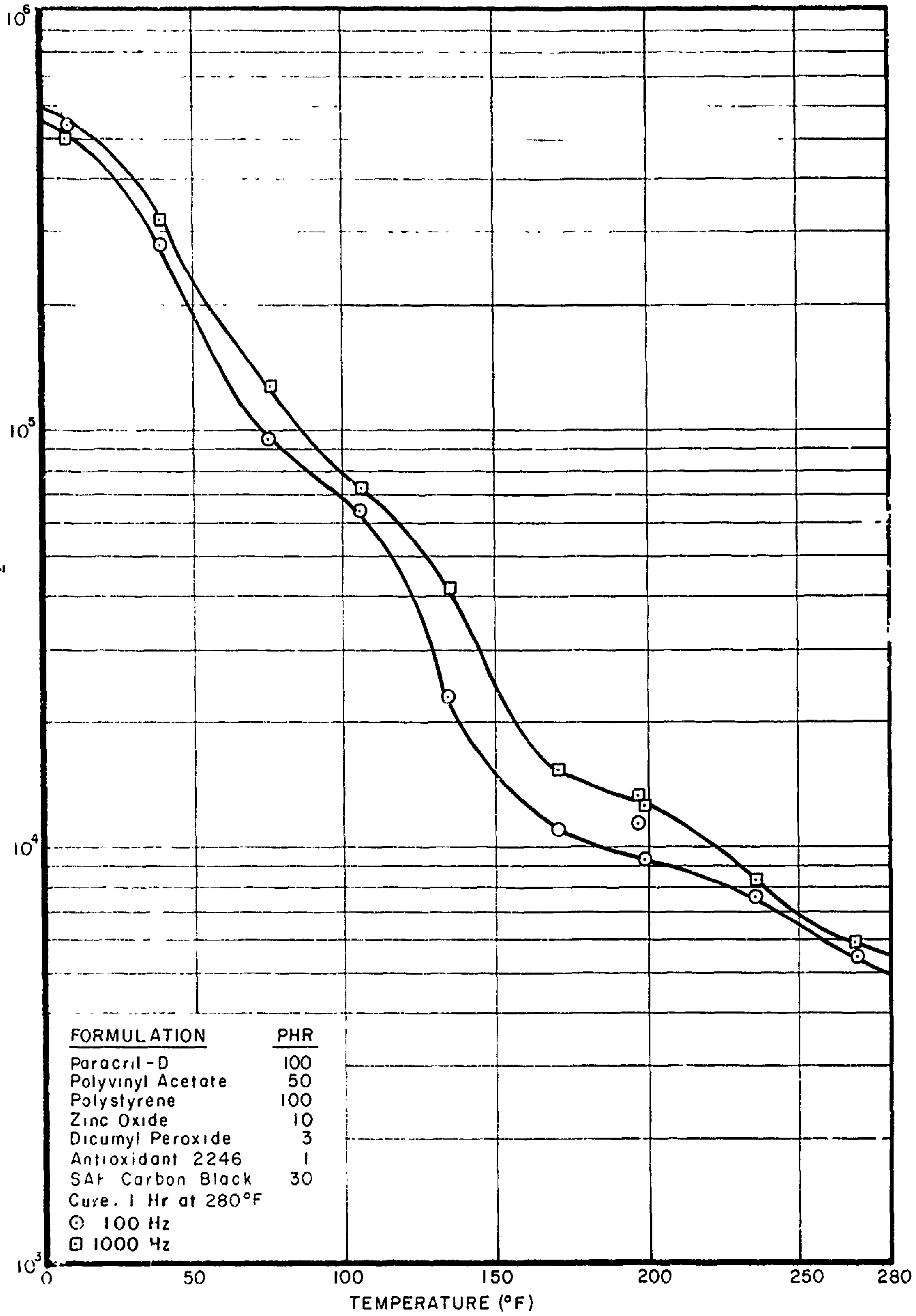


Figure 26. Loss Factor vs Temperature of a Potential Damping Material With 20 Parts SAF Carbon Black Added



FORMULATION	PHR
Paracril-D	100
Polyvinyl Acetate	50
Polystyrene	100
Zinc Oxide	10
Dicumyl Peroxide	3
Antioxidant 2246	1
SAF Carbon Black	30

Figure 27. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 30 Parts SAF Carbon Black Added

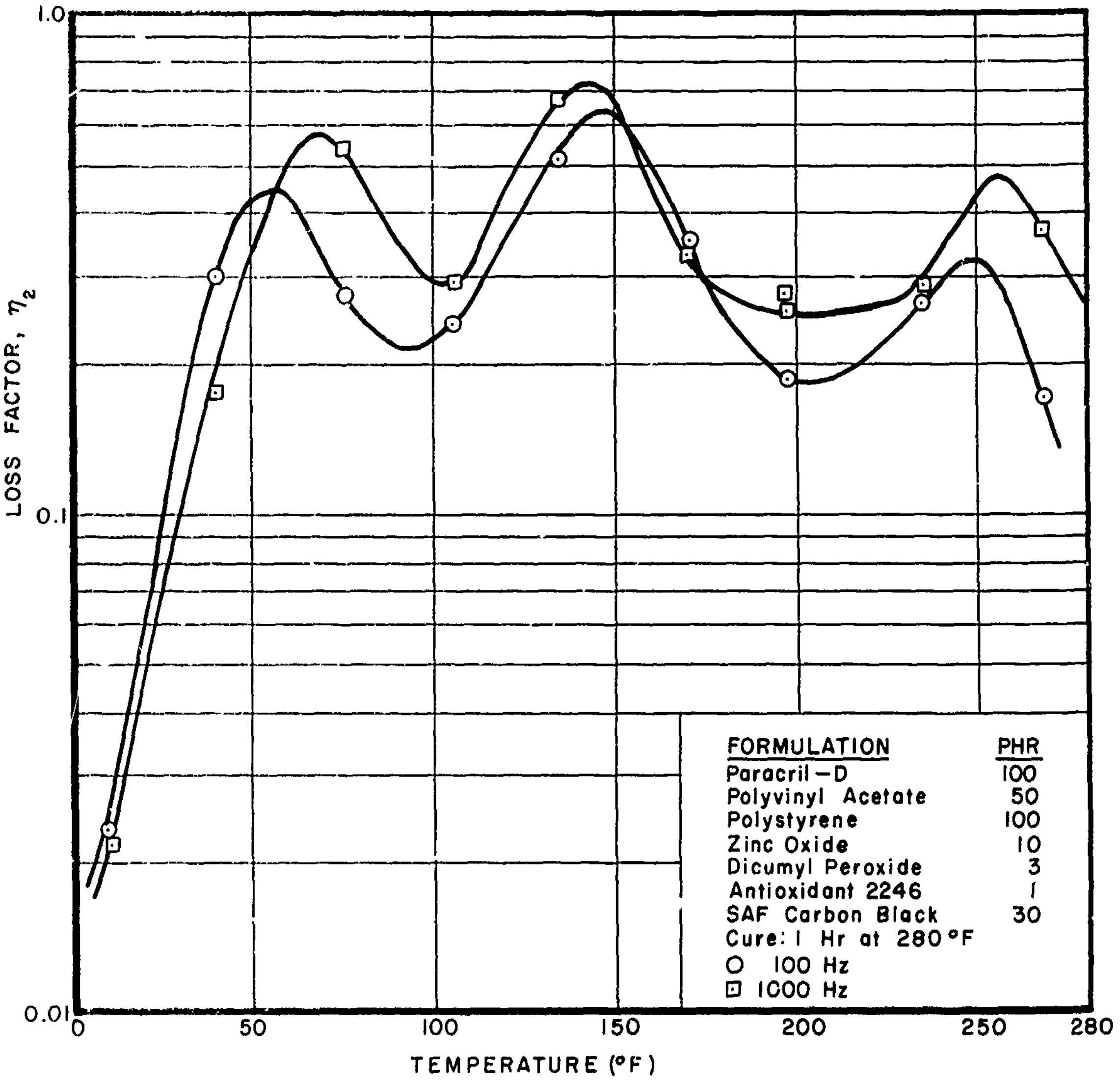


Figure 28. Loss Factor vs Temperature of a Potential Damping Material With 30 Parts SAF Carbon Black Added

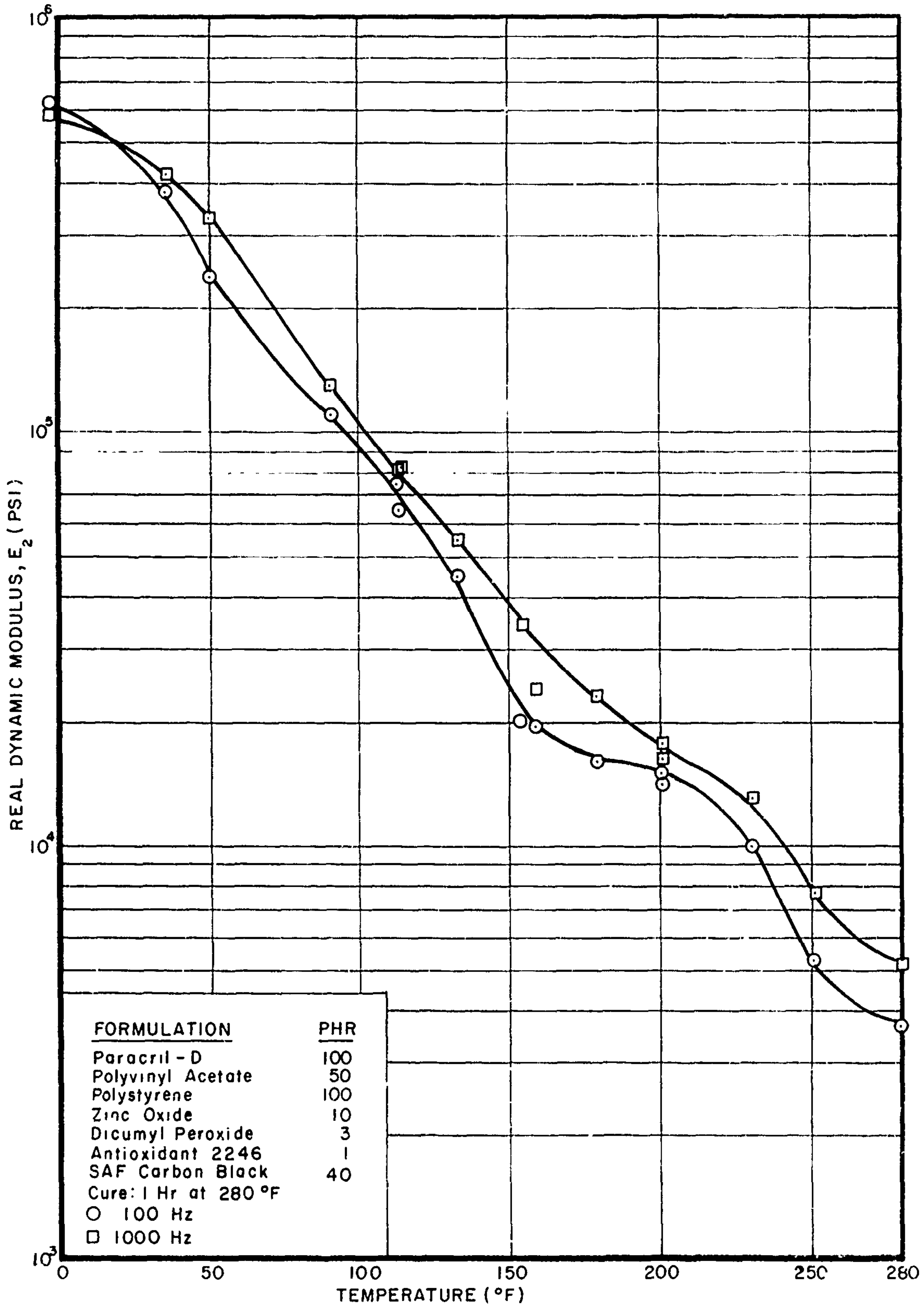


Figure 29. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 40 Parts SAF Carbon Black Added

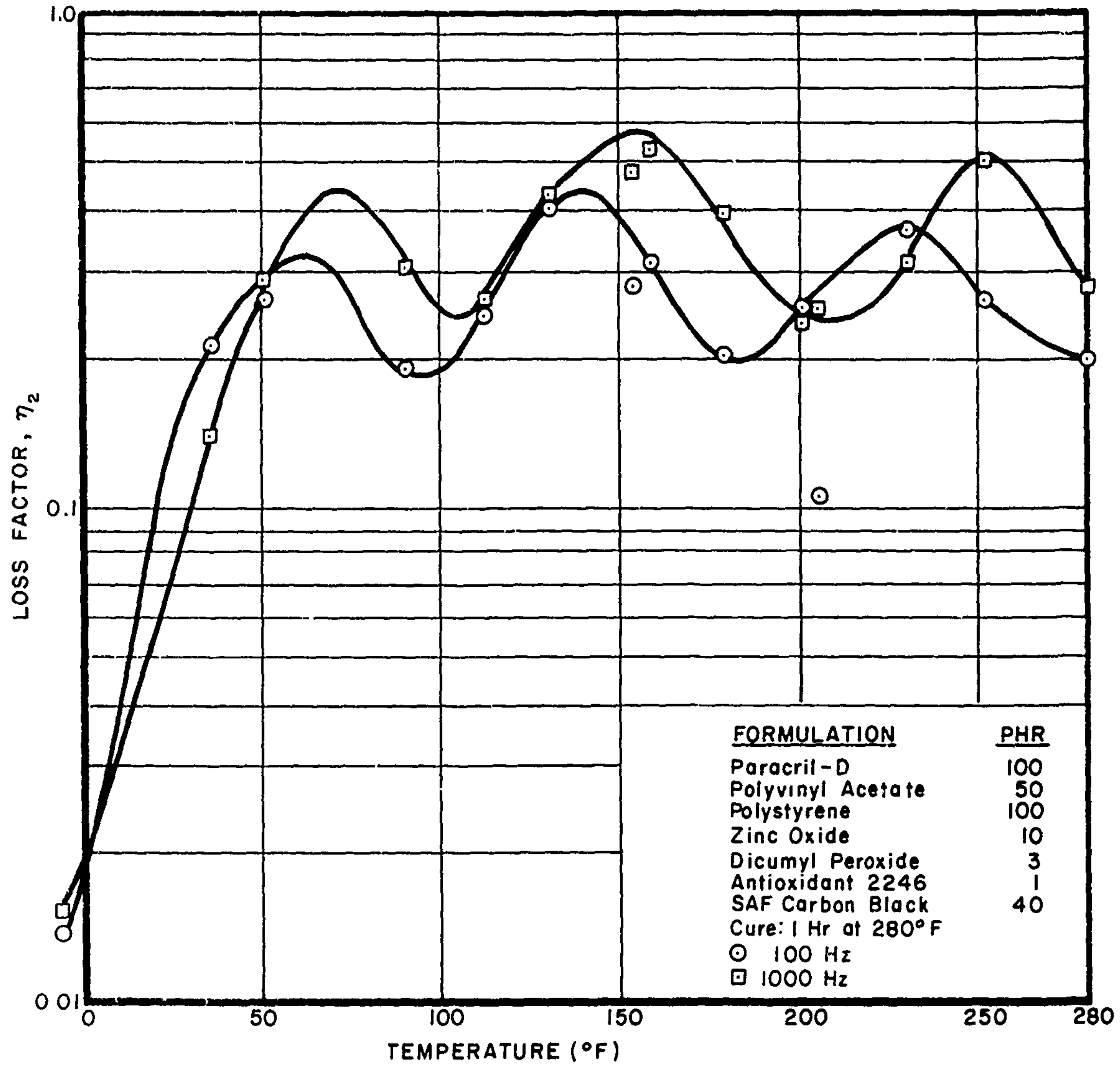


Figure 30. Loss Factor vs Temperature of a Potential Damping Material With 40 Parts SAF Carbon Black Added

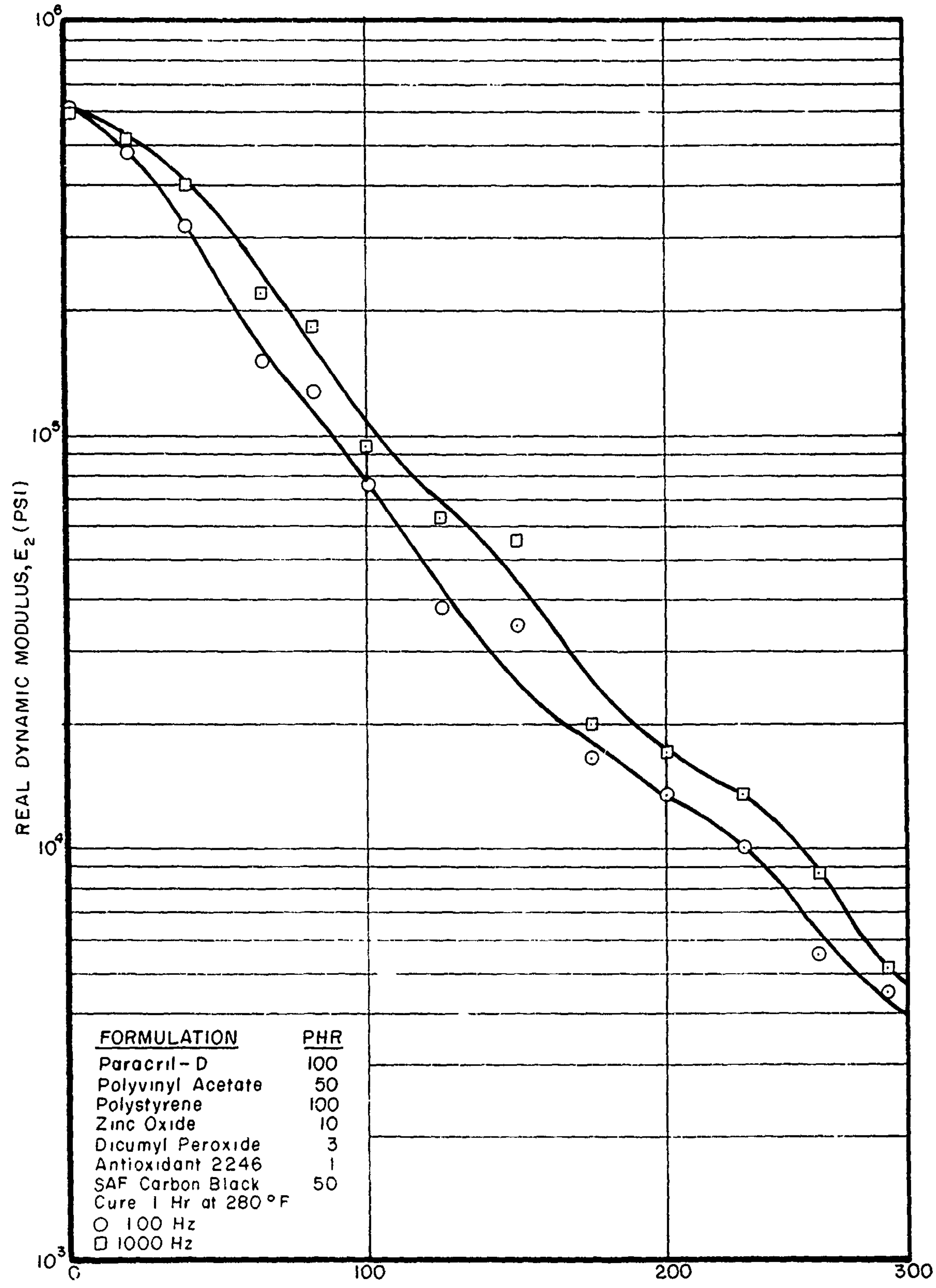


Figure 31. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 50 Parts SAF Carbon Black Added

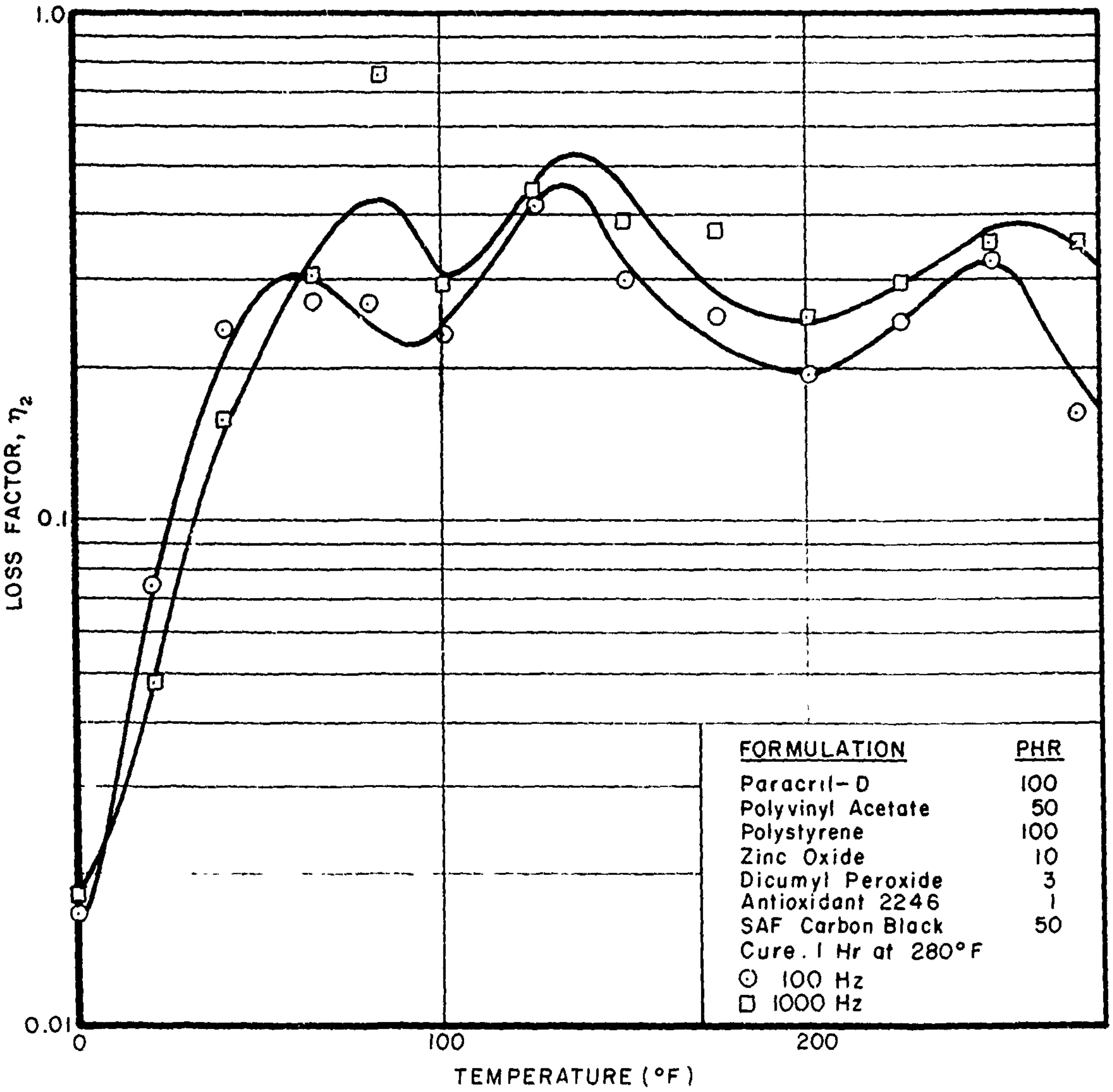


Figure 32. Loss Factor vs Temperature of a Potential Damping Material With 50 Parts SAF Carbon Black Added

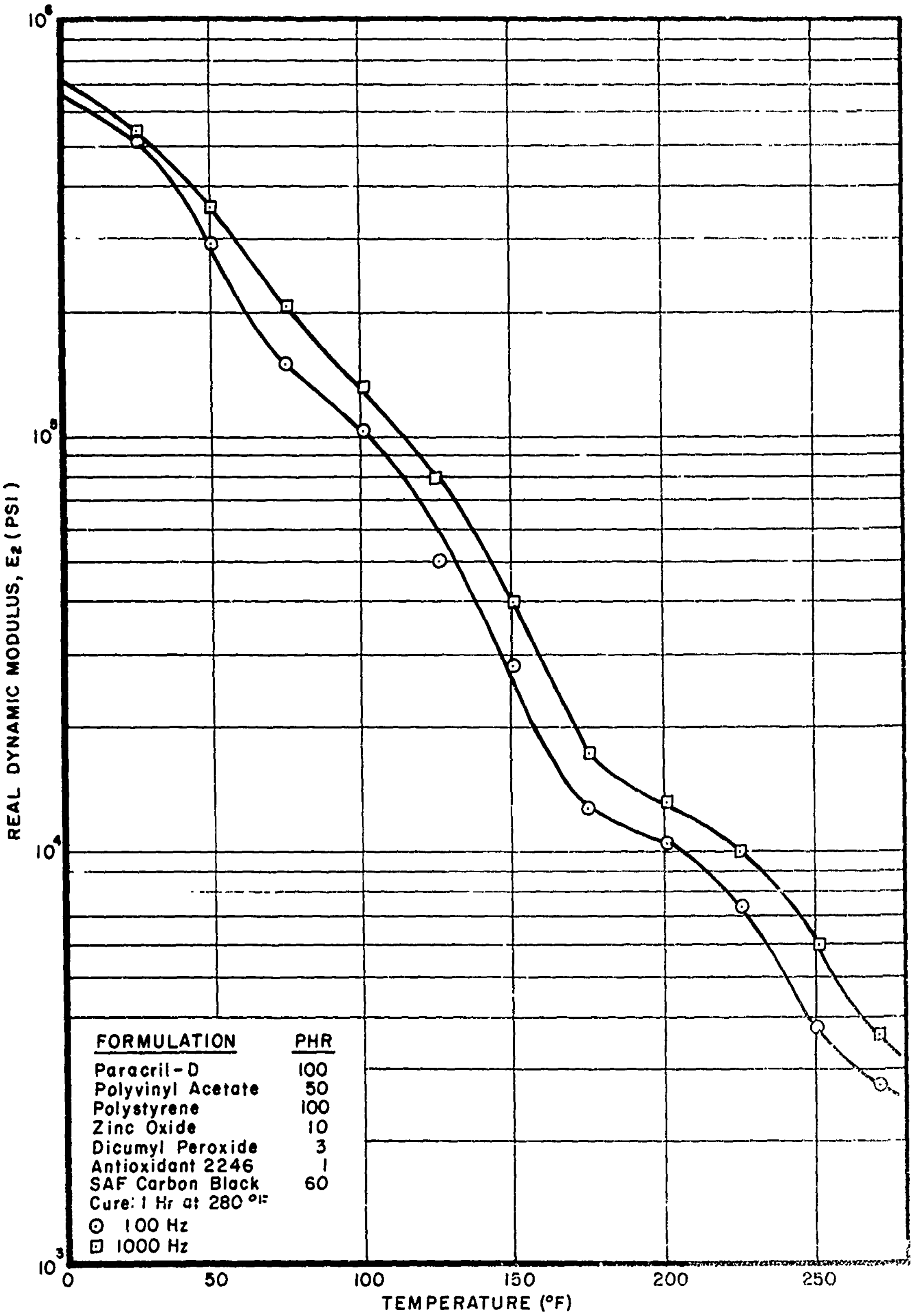


Figure 33. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 60 Parts SAF Carbon Black Added

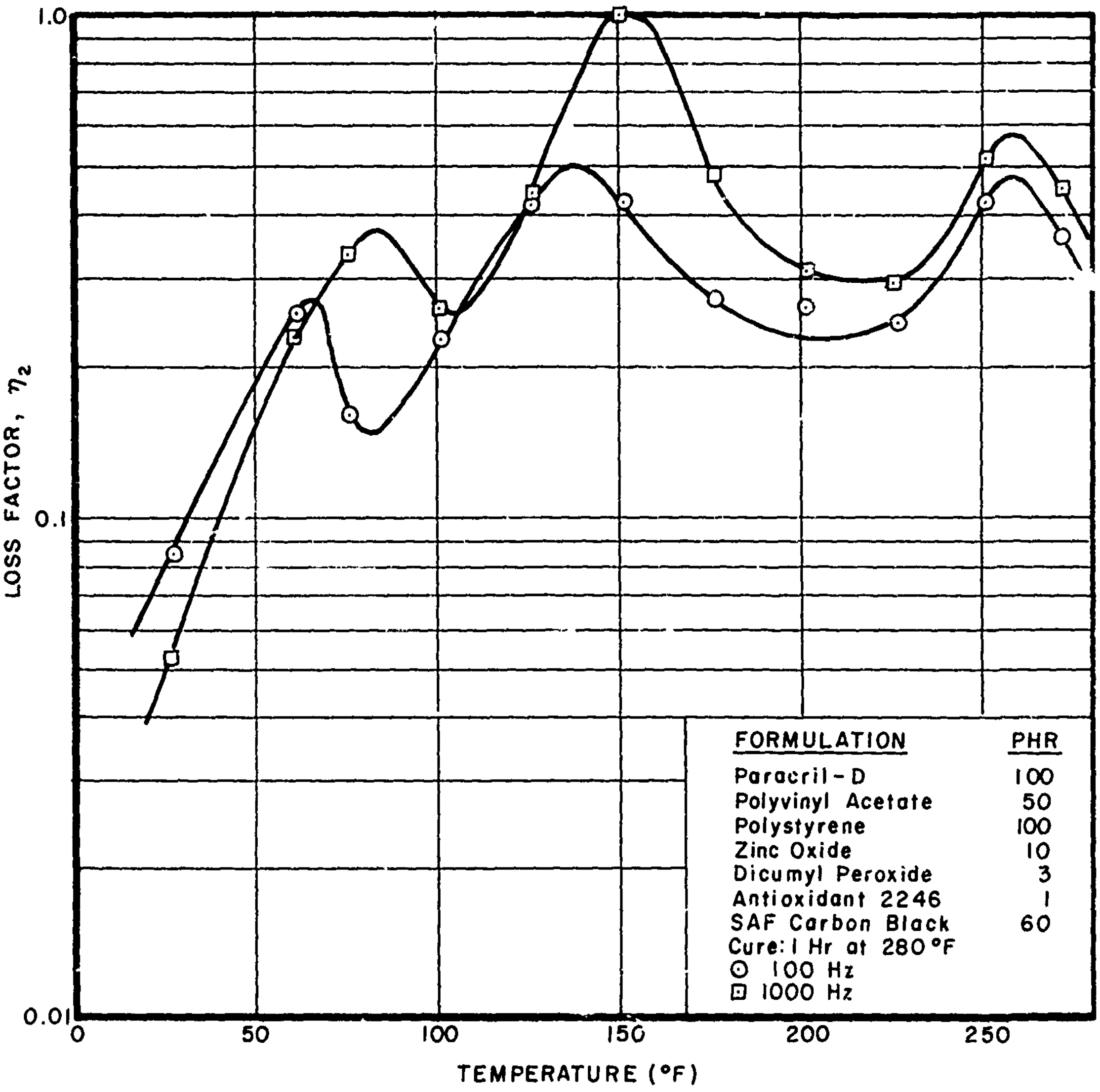


Figure 34. Loss Factor vs Temperature of a Potential Damping Material With 60 Parts SAF Carbon Black Added

decreased for a formulation containing 60 parts. The loss factor values remained essentially unchanged except, possibly, for slight increases in the trough values and a slight increase in the temperature at which the peak values occurred. The formulation containing 60 parts SAF black was not considered desirable because it was very difficult to process and did not flow well in the mold during press cure. The modulus and loss factor curves of the formulations containing 30, 40, and 50 parts carbon black were superposable within experimental error and would be equally effective for damping purposes. Based on overall physical properties shown in Figures 35, 36, and 37, the compound containing 50 parts of carbon black was selected as having optimum damping characteristics.

These results indicate that SAF carbon black reinforces the acrylonitrile rubber but not the polystyrene. Thus, the modulus values of the three-polymer blend at temperatures above 200°F are largely due to the polystyrene, and its modulus can be increased slightly by increasing the molecular weight of the polymer, cross-linking, and/or adding selective reinforcing fillers for the polystyrene (Reference 18).

d. Curing Agent Variations

The level of curing agent (recrystallized dicumyl peroxide) used in the blends was investigated to determine the effect on the mechanical characteristics of the blend. One objective was to increase the high-temperature modulus without reducing the loss factor. The experimental compounds consisted of 3, 5, 7, and 9 parts (by weight) dicumyl peroxide along with 40 and 50 parts SAF carbon black mixed with 100 parts acrylonitrile rubber, 50 parts polyvinyl acetate, 100 parts polystyrene, 10 parts zinc oxide, and 1 part antioxidant 2246 (an improved damping material). The formulations containing 9 parts dicumyl peroxide could not be mixed on a two-roll rubber mill. The modulus was increased somewhat, but the changes were considered insignificant (all data is not shown). The formulation containing 7 parts dicumyl peroxide and 50 parts SAF carbon black (Figures 38 and 39) was selected as having the best balance of physical properties, including tensile strength and elongation, as well as good damping ability.

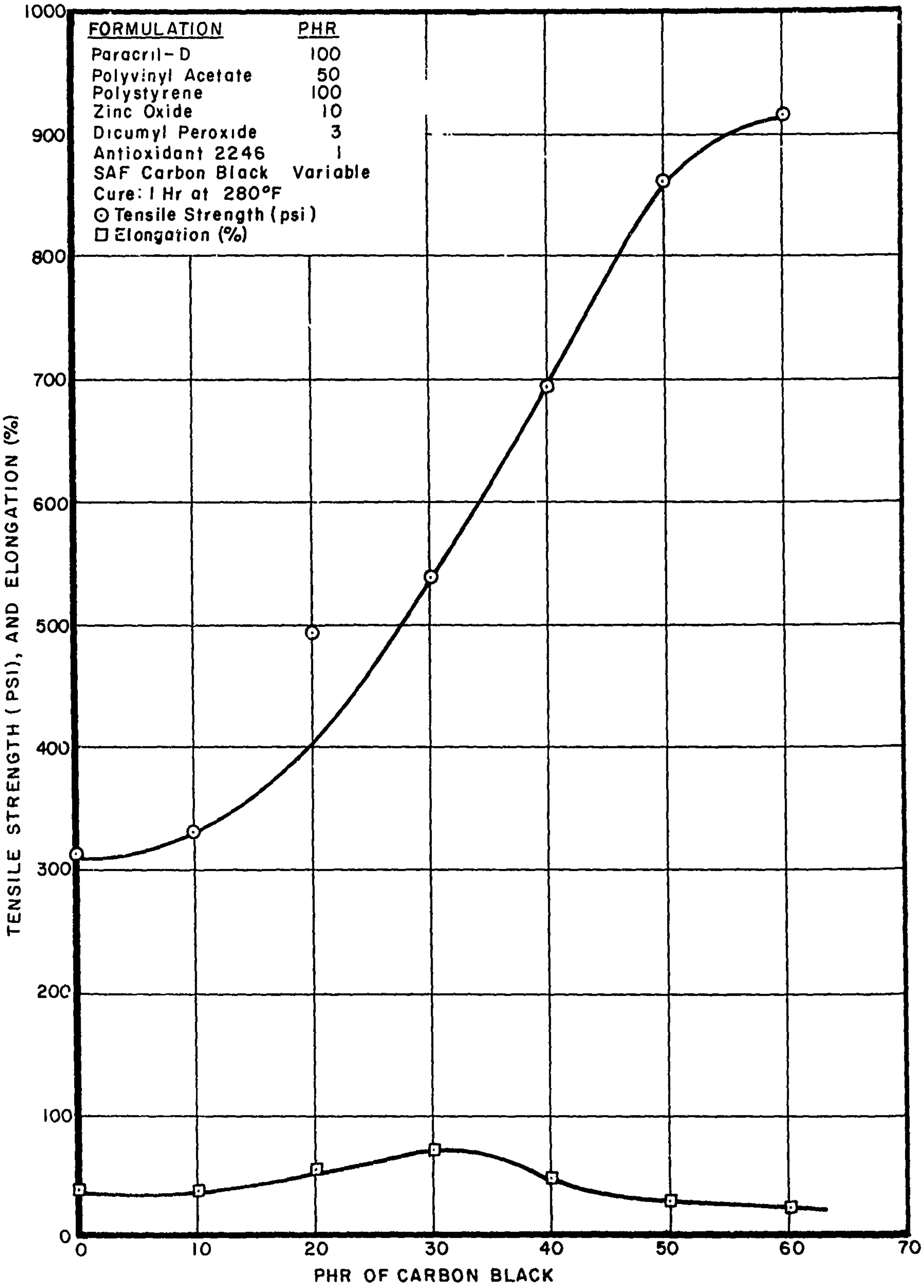


Figure 35. Tensile Strength and Elongation at 78°F vs PHR Carbon Black

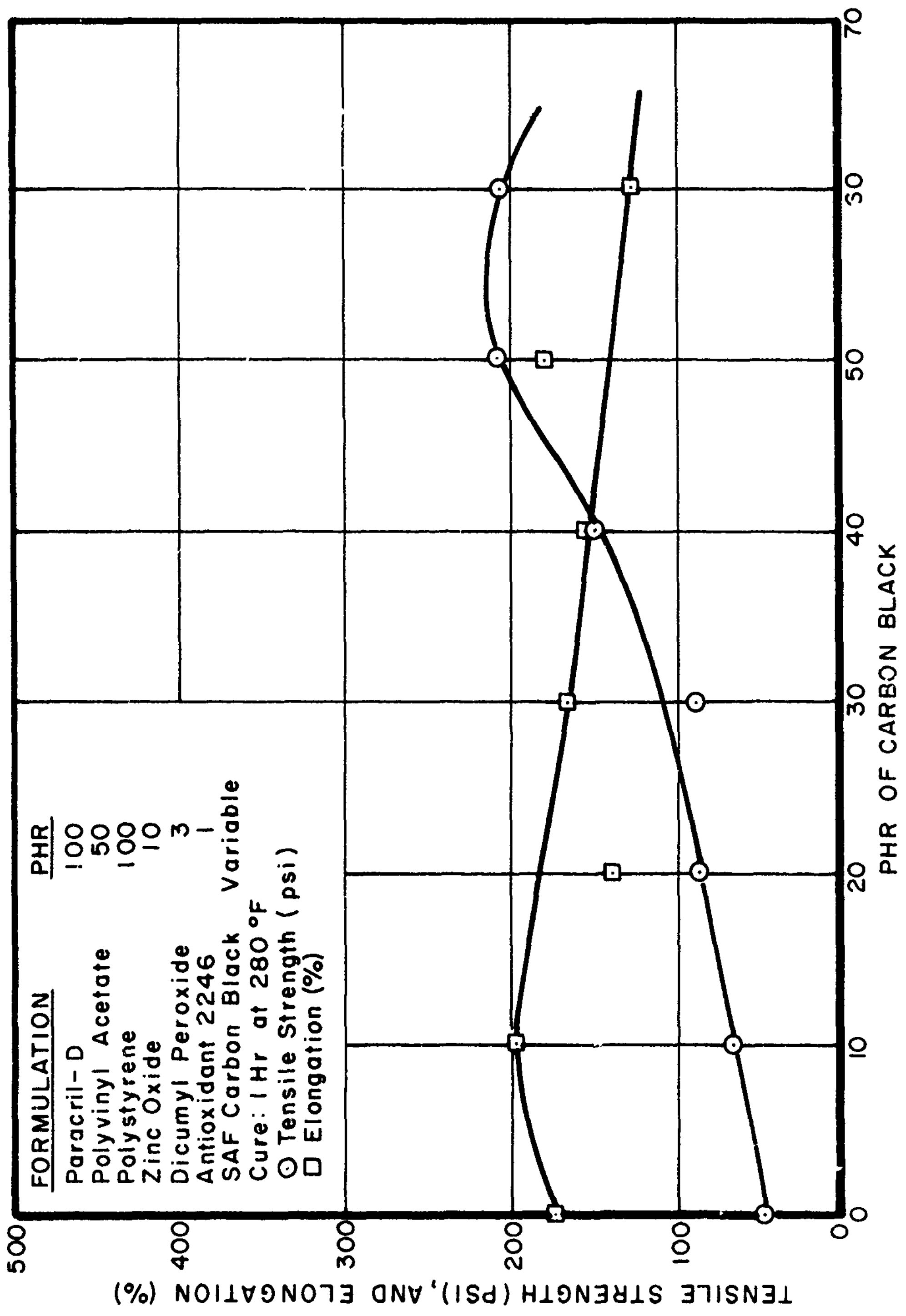


Figure 36. Tensile Strength and Elongation at 200°F vs PHR Carbon Black

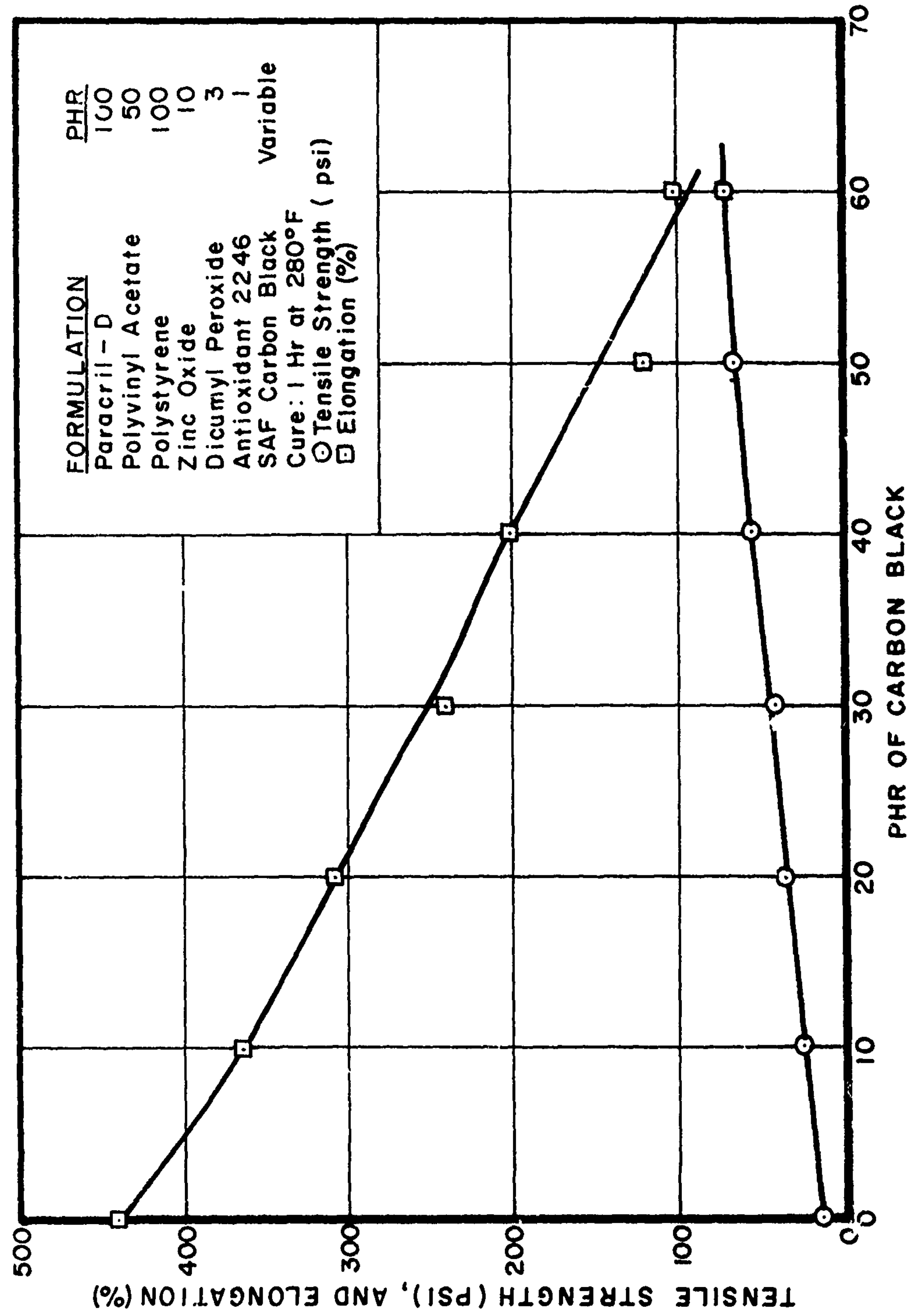


Figure 37. Tensile Strength and Elongation at 300°F vs PHR Carbon Black

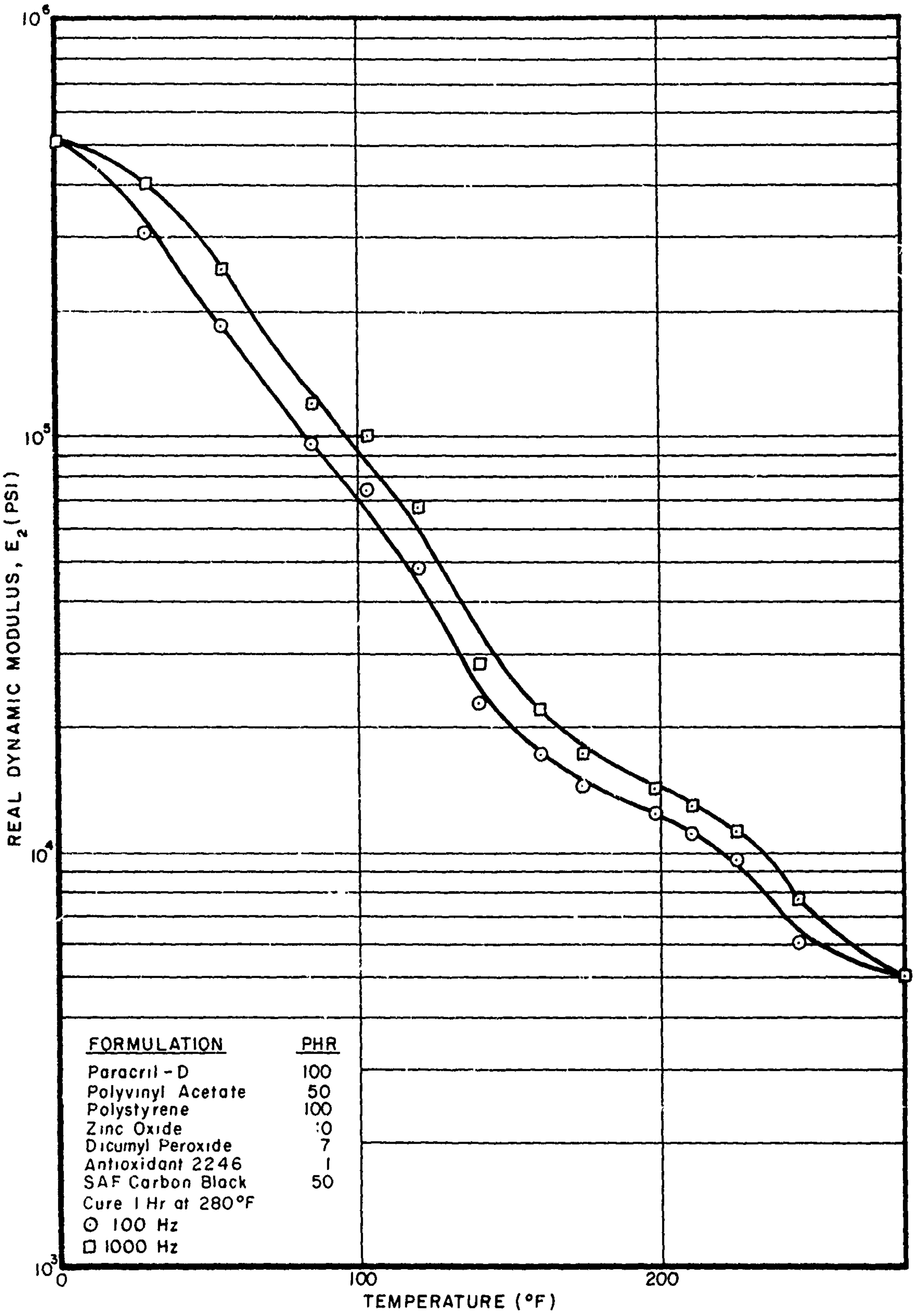


Figure 38. Real Dynamic Modulus vs Temperature of a Potential Damping Material With Additional Curing Agent Added

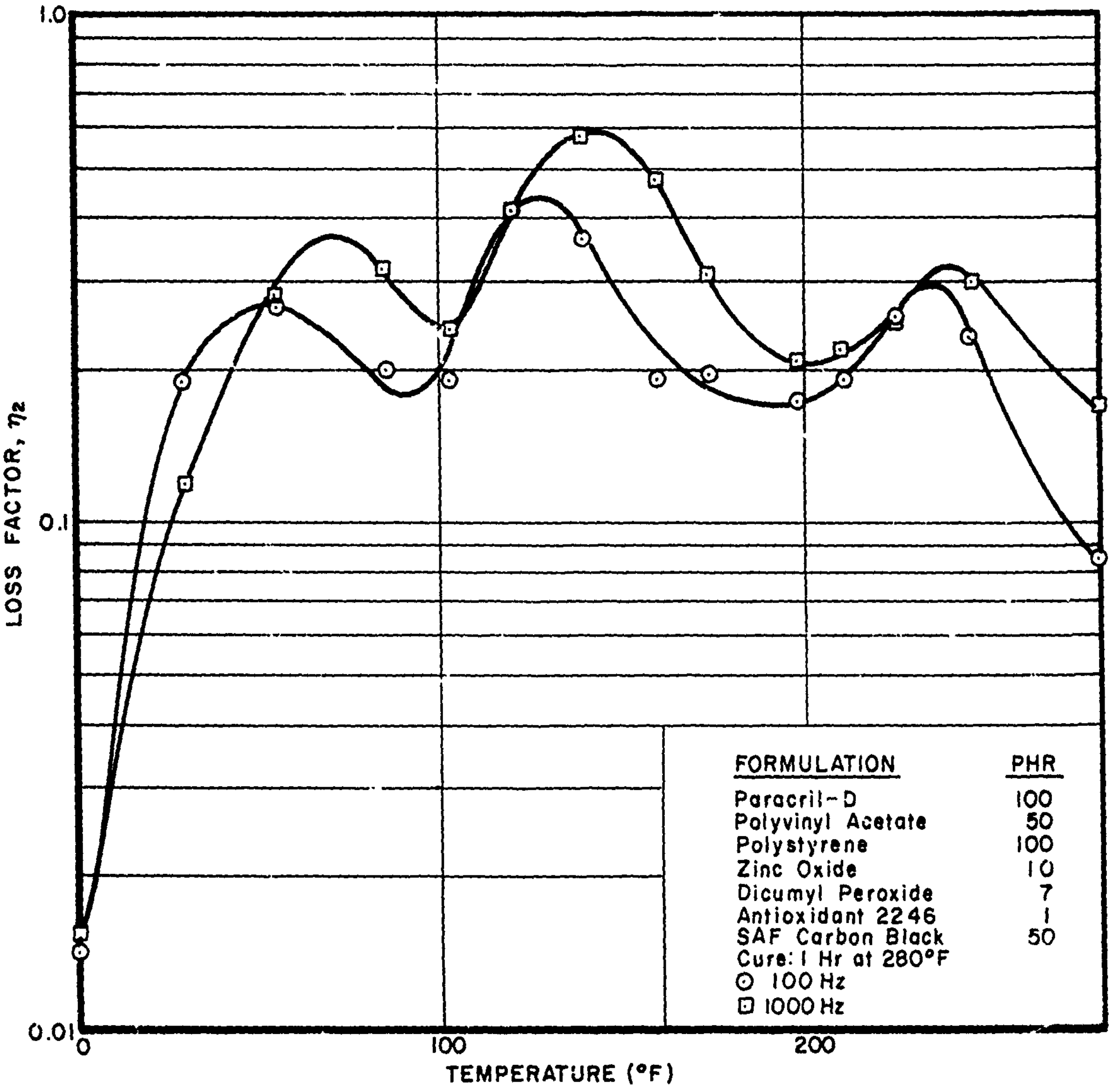


Figure 39. Loss Factor vs Temperature of a Potential Damping Material With Additional Curing Agent Added

e. Effect of Polystyrene Molecular Weight

The large variations of the high-temperature modulus values observed initially were attributed to nonuniformity in the molecular weight between various batches of the laboratory-prepared polystyrene. By using a commerically available polystyrene, namely Dylene 8*, the modulus was increased to 7 x 10^3 psi at 300°F, as shown in Figure 38. In addition, the peak loss factor values for both the polyvinyl acetate and polystyrene, which occurred at about 140° and 240°F, respectively, were slightly higher than usual, but the peak for the nitrile rubber was slightly reduced. Thus, the commercial-grade polystyrene was used in subsequent formulations because it produced slightly better damping properties. The modulus was at least 7 x 10^3 psi and the loss factor at least 0.1 over the temperature range 30° to 300°F at frequencies of 100 and 1000 Hz (Figures 38 and 39). This increase in the high-temperature modulus is considered to be significant for a wide-temperature-range damping material. The increase was attributed to the possible purity, molecular, and/or the glass transition temperature of the commercial grade polystyrene but not to the effect of filler reinforcement or the presence of chemical crosslinks in the polystyrene.

f. Investigation of Polystyrene Reinforcement

The above conclusions concerning the importance of the polystyrene, along with the previous negative results, imply that selective curing and reinforcing of the polystyrene, rather than of the acrylonitrile rubber in the formulation, will increase the high-temperature modulus values. The experiments involving polyvinyl acetate variations, however, imply that increasing the relative proportion of polystyrene in the formulation has the same effect. In either case, there will be concomitant changes in the loss factor and the modulus curves, since loss factor values are high only when the modulus is changing rapidly. Thus, insoluble polymers can be blended to have reasonably high modulus values by sacrificing the loss factor values, or to have reasonably high loss factor values by sacrificing the modulus values.

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*Koppers Company, Inc. Trademark

The relatively high loss factor values shown in Figure 39 were obtained at the expense of higher modulus values. This formulation was thought to have very adequate modulus and loss factor values for a high level of damping on aluminum structures and, because of the presence of three loss factor peaks of equal value, it would be effective over a wider temperature range than a single polymer damping material.

The contribution of the relatively high peak loss factor values on the effectiveness of the material was less than anticipated, particularly at temperatures above 140°F (Figure 40). This was attributed to the relatively low modulus values of the polymer blend at temperatures above 220°F (Figure 39) and implies that the polymer blend would be more effective for damping aluminum structures (Figure 2b) if the modulus values were increased, even at the expense of reducing the loss factor values. Adding 50 parts of flake graphite, a reinforcing filler for polystyrene (Reference 18), increased the modulus of the improved damping material without significantly reducing the loss factor values (Reference 17). The blend then provided better structural damping at the higher temperatures, as shown in Figure 41. This shows, as noted elsewhere (References 18 and 27), that adding properly selected mineral fillers to increase the modulus can also increase the damping effectiveness.

The effect of the flake graphite appeared to be more pronounced at temperatures where the polystyrene exhibits glass-like behavior, i.e., below 240°F. Thus, the modulus and the damping effectiveness of the polymer blend depends more on the modulus of the polystyrene than on that of the other two polymers. Generally, this conclusion implies that the temperature range where multiphase polymer blends exhibit adequate damping can be altered by essentially the same techniques as are used for single-polymer compositions (Reference 18).

2. COMPOUND VERSATILITY

The above compounding investigations provided an optimum basic formulation for wide-temperature-range damping. Now we will demonstrate how the basic formulation can be modified for specific damping problems and discuss different methods of applying the material to flat aluminum structures.

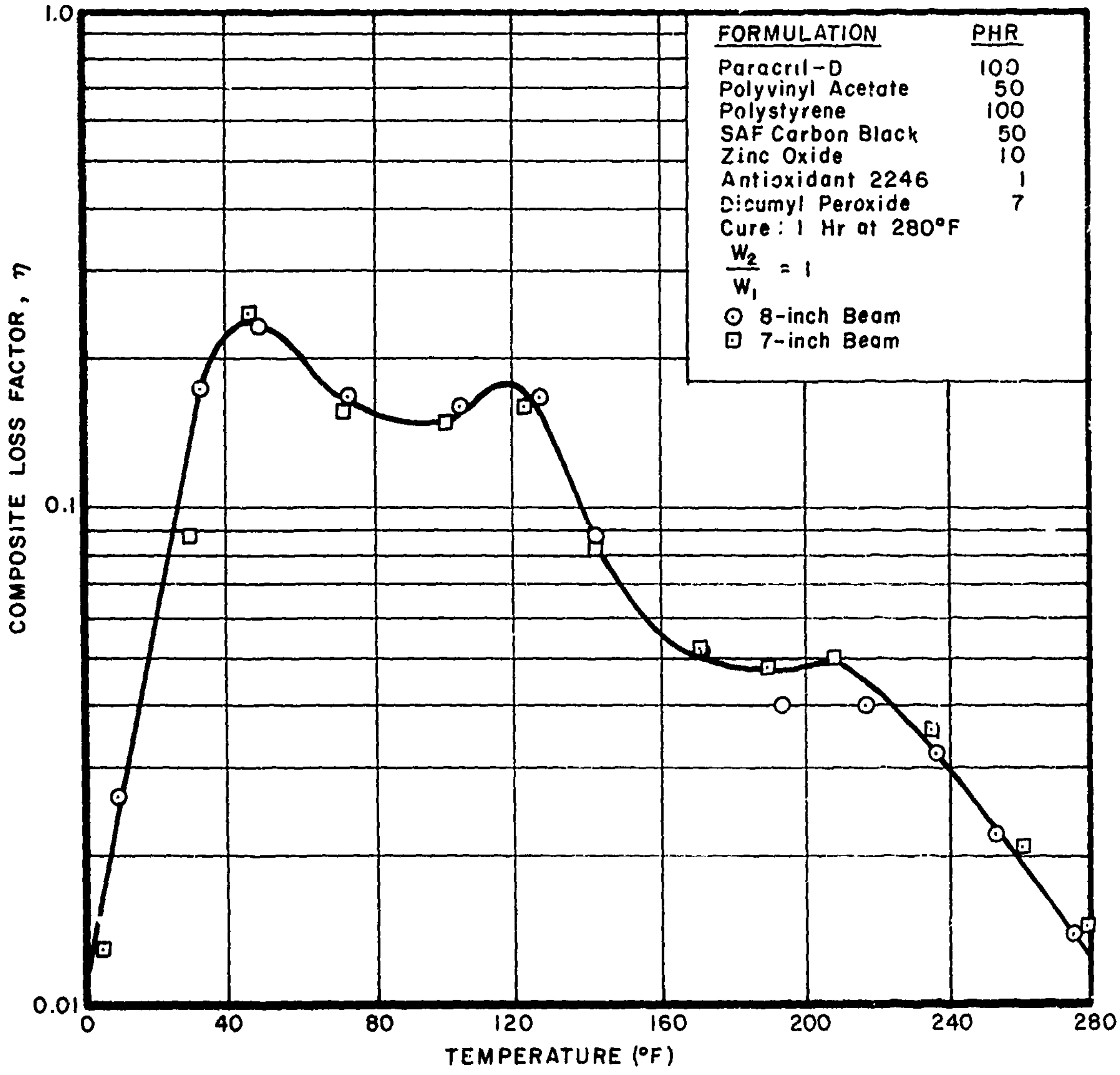

Figure 40. Measured Composite Loss Factor vs Temperature at 100 Hz of a Blend Containing 50 Parts SAF Carbon Black

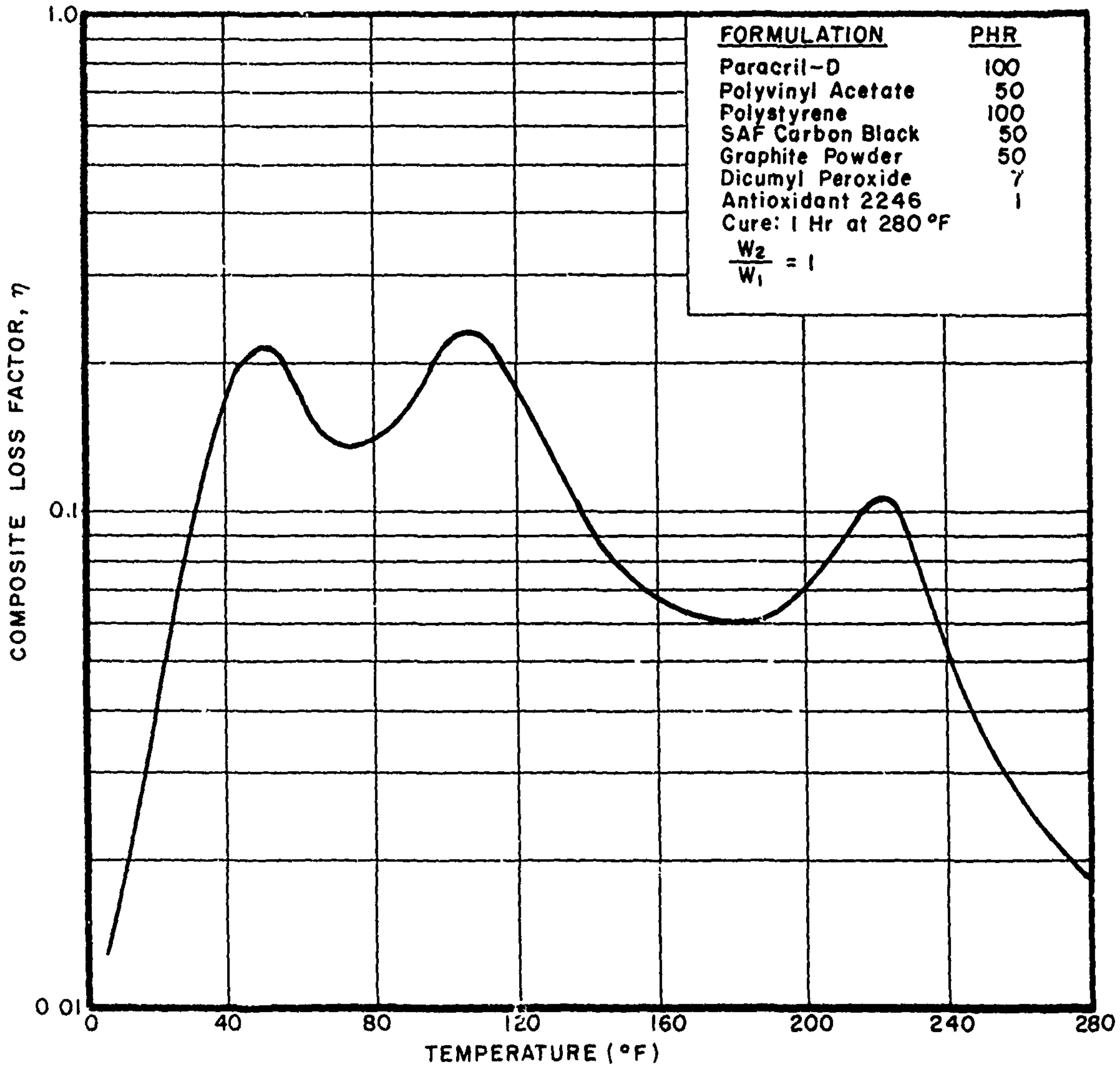

Figure 41. Measured Composite Loss Factor vs Temperature at 100 Hz of a Blend Containing 50 Parts Each of Carbon Black and Graphite Powder

The height of the composite loss factor curves in Figures 40 and 41, (i.e., the measured structural damping) can be varied between the limits of the loss factor curve for the uncoated aluminum beams (with losses less than 0.01) and that for the polymer blend by varying the relative weight of the coating (Reference 4). Comparing Figure 40 with Figure 39 shows the measured losses of the 1:1 weight ratio composite beam to be very close to the material losses of the polymer blend, particularly at temperatures from about 0° to 140°F. Thus, increasing the coating weight would be of doubtful value in this temperature range. At temperatures above 140°F, the composite losses are far removed from the material losses (i.e., the damping is not saturated, Reference 4), and the composite losses will increase as the relative coating weight is increased. Thus, the polymer blend can be used as an effective wide-temperature-range damping treatment. Another practical approach to increasing the structural damping at temperatures above 140°F would be to alter the formulation in such a way as to extend the width of the temperature range of the polystyrene modulus change or, equivalently, to compress the temperature range of the acrylonitrile rubber and polyvinyl acetate modulus change. This objective was accomplished by applying well-established compounding technology (Reference 18) along with experimental verification to increase the structural damping efficiency over a narrower temperature range, to increase the width of the effective temperature range, and to show polymer blends can be supplied in suitable form for almost any kind of field application.

A plasticizer, Thiokol TP-95* was selected from many candidates because it appeared to swell aliquot proportions of the three polymers used in the blend by about the same amount. It was anticipated that adding successively larger amounts of Thiokol TP-95 to the polymer blend would shift the effective temperature range to successively lower temperatures while the width of the temperature range remained about the same. The expected results were not obtained (Figures 42 through 47), but the results were very interesting. Adding 30 parts (by weight) of plasticizer caused the loss factor values of the polymer blend to increase (compare Figures 39 and 43), and adding 45 parts of plasticizer caused the loss factor values to increase to the highest level over the widest connected

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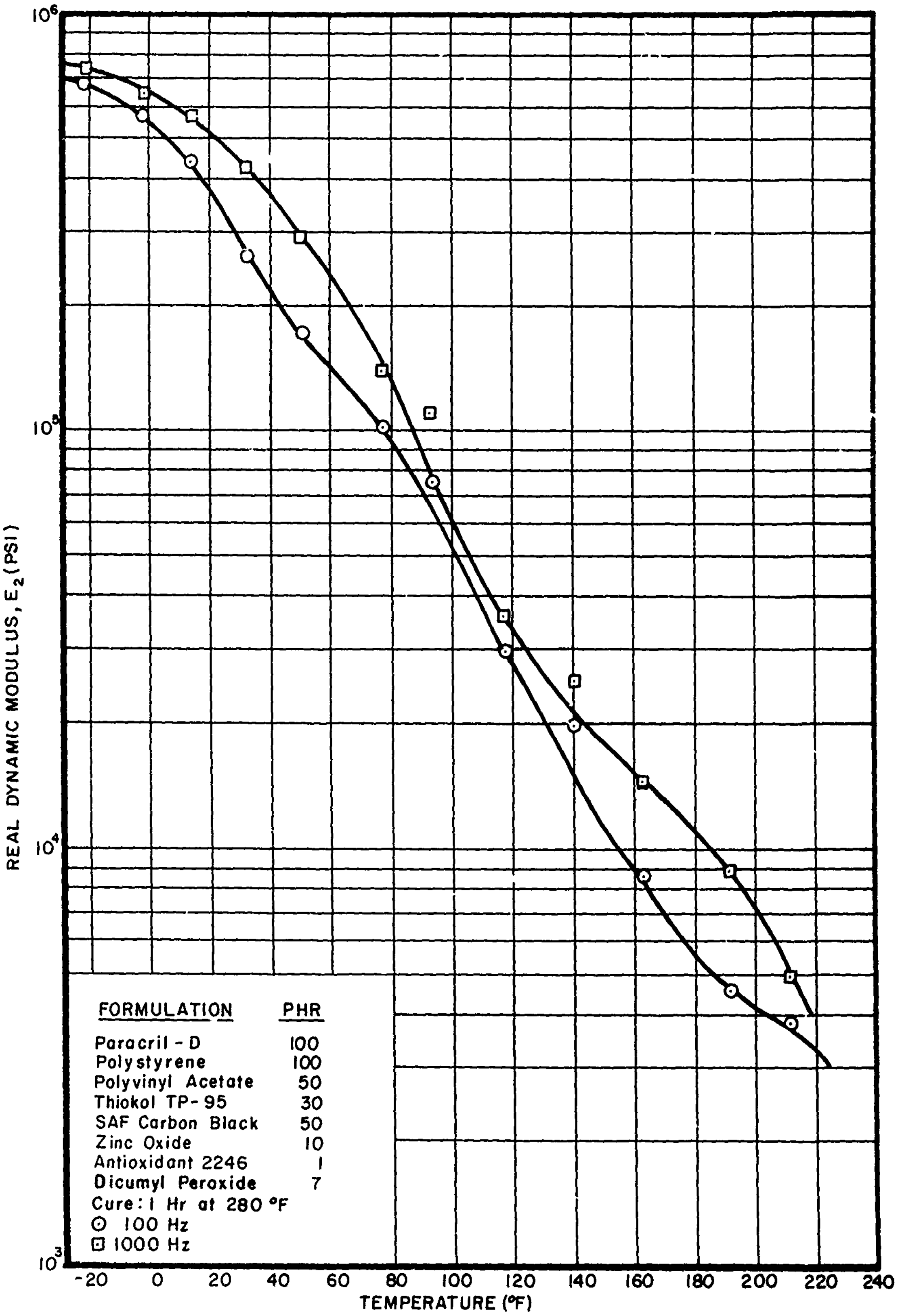

Figure 42. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 50 Parts Plasticizer Added

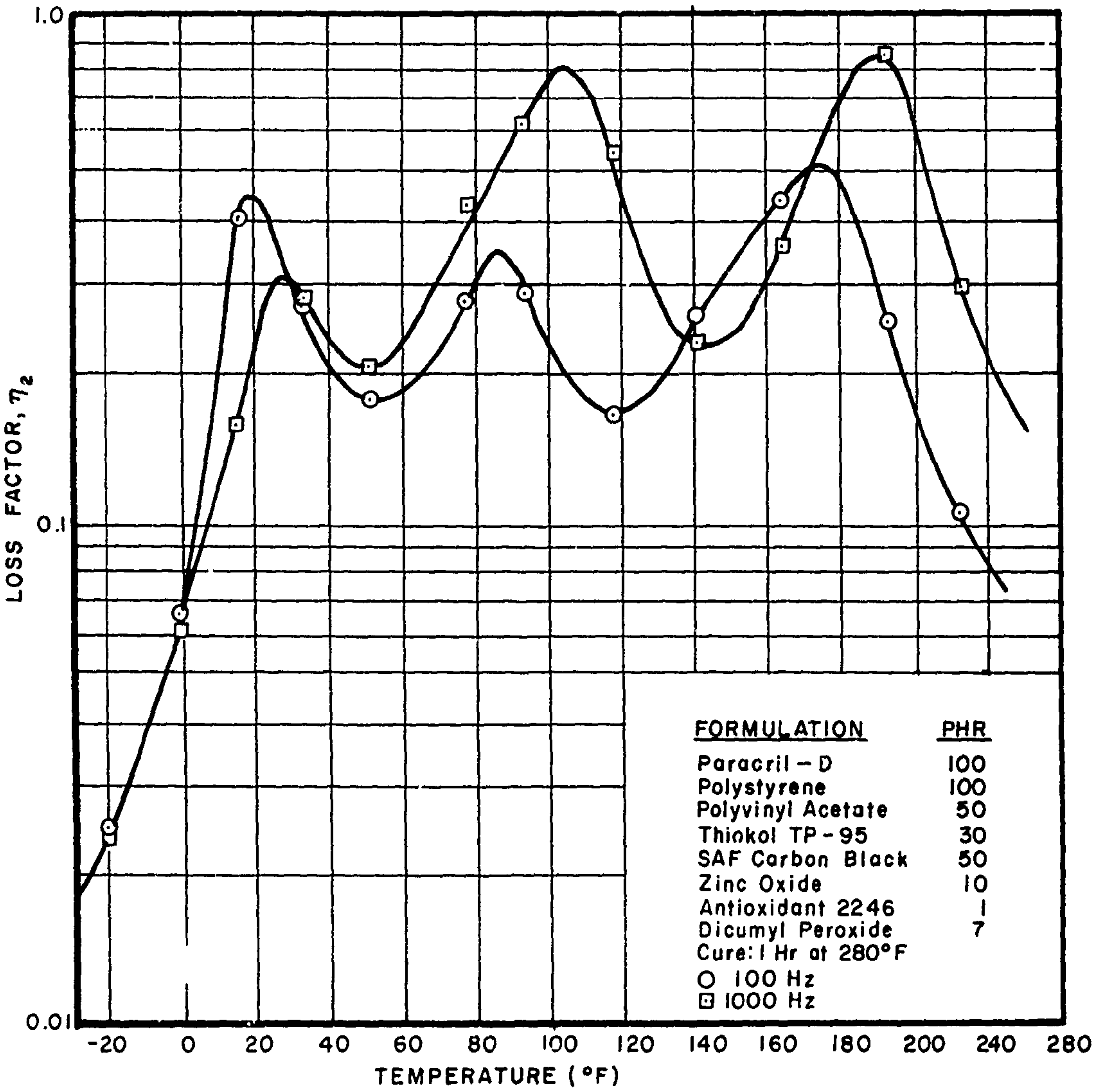

Figure 43. Loss Factor vs Temperature of a Potential Damping Material With 30 Parts Plasticizer Added

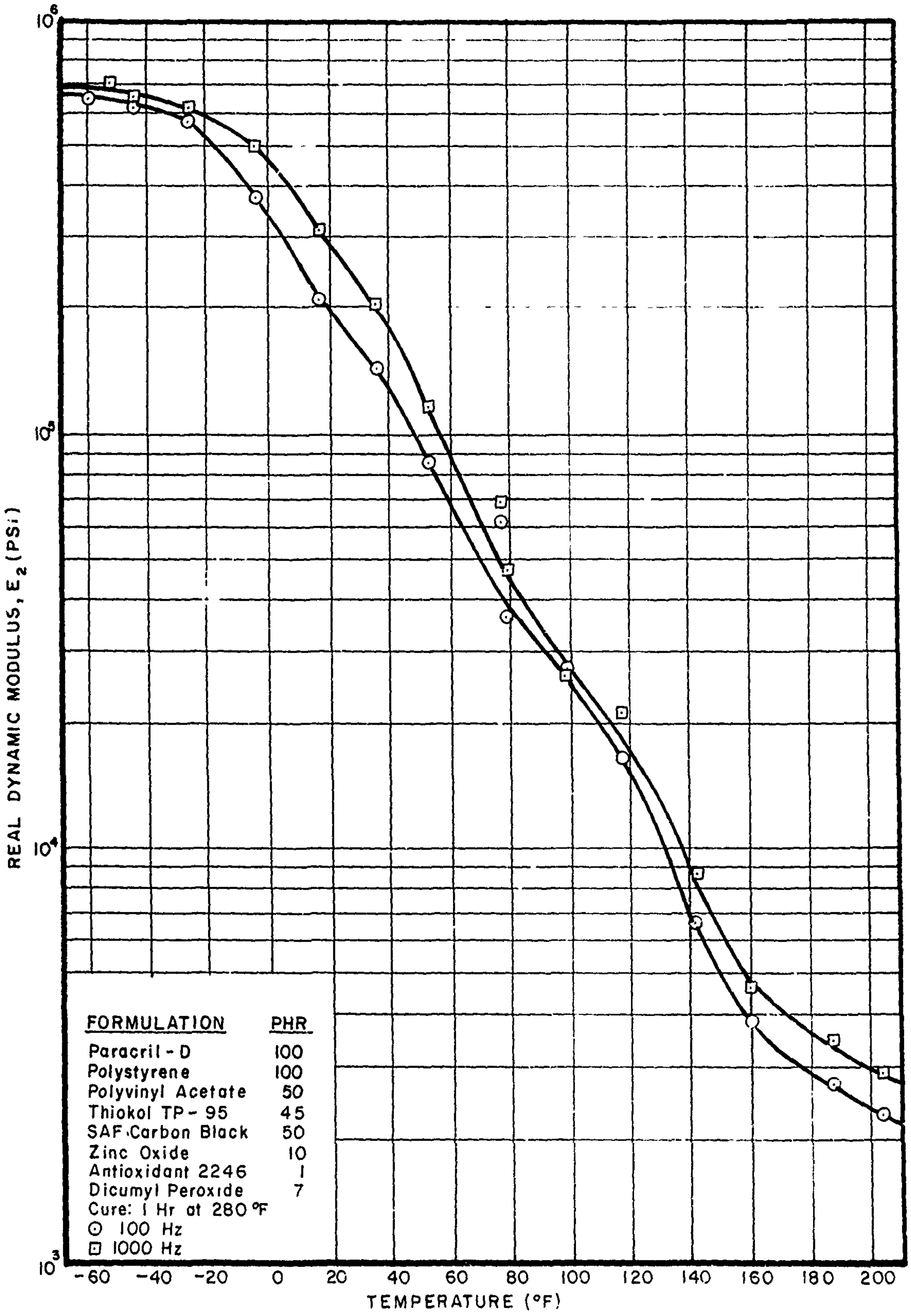

Figure 44. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 45 Parts Plasticizer Added

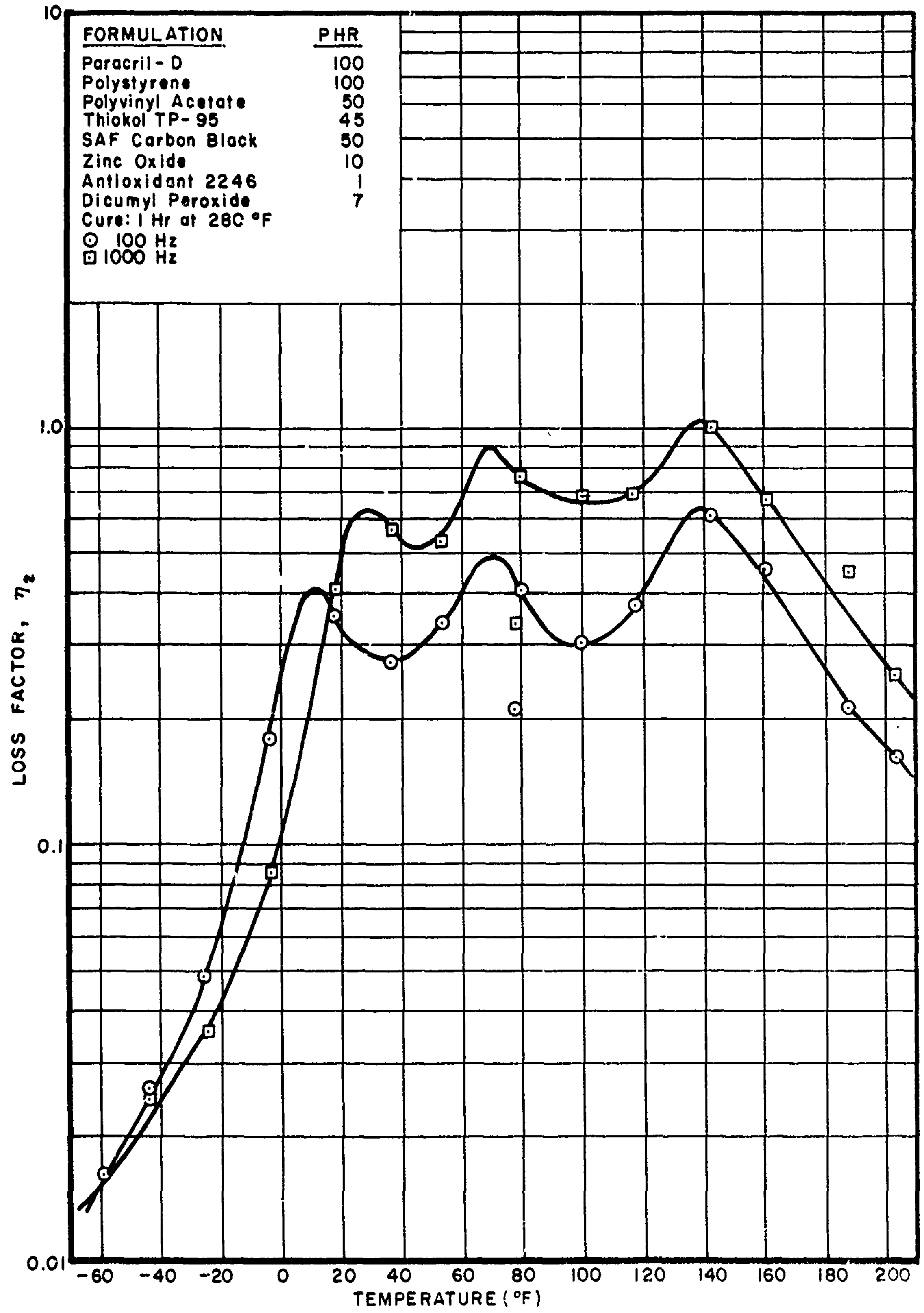

Figure 45. Loss Factor vs Temperature of a Potential Damping Material With 45 Parts Plasticizer Added

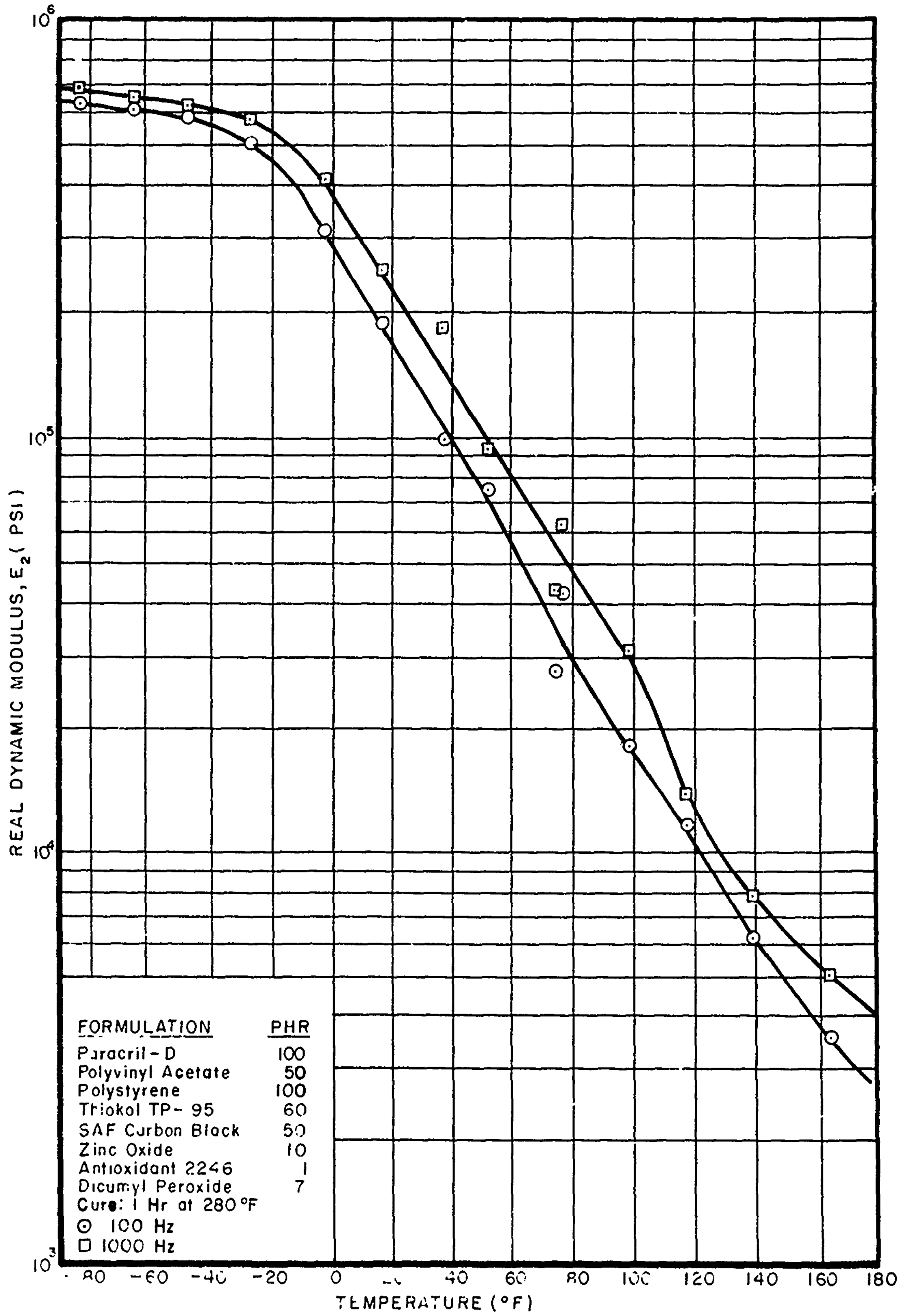

Figure 46. Real Dynamic Modulus vs Temperature of a Potential Damping Material With 60 Parts Plasticizer Added

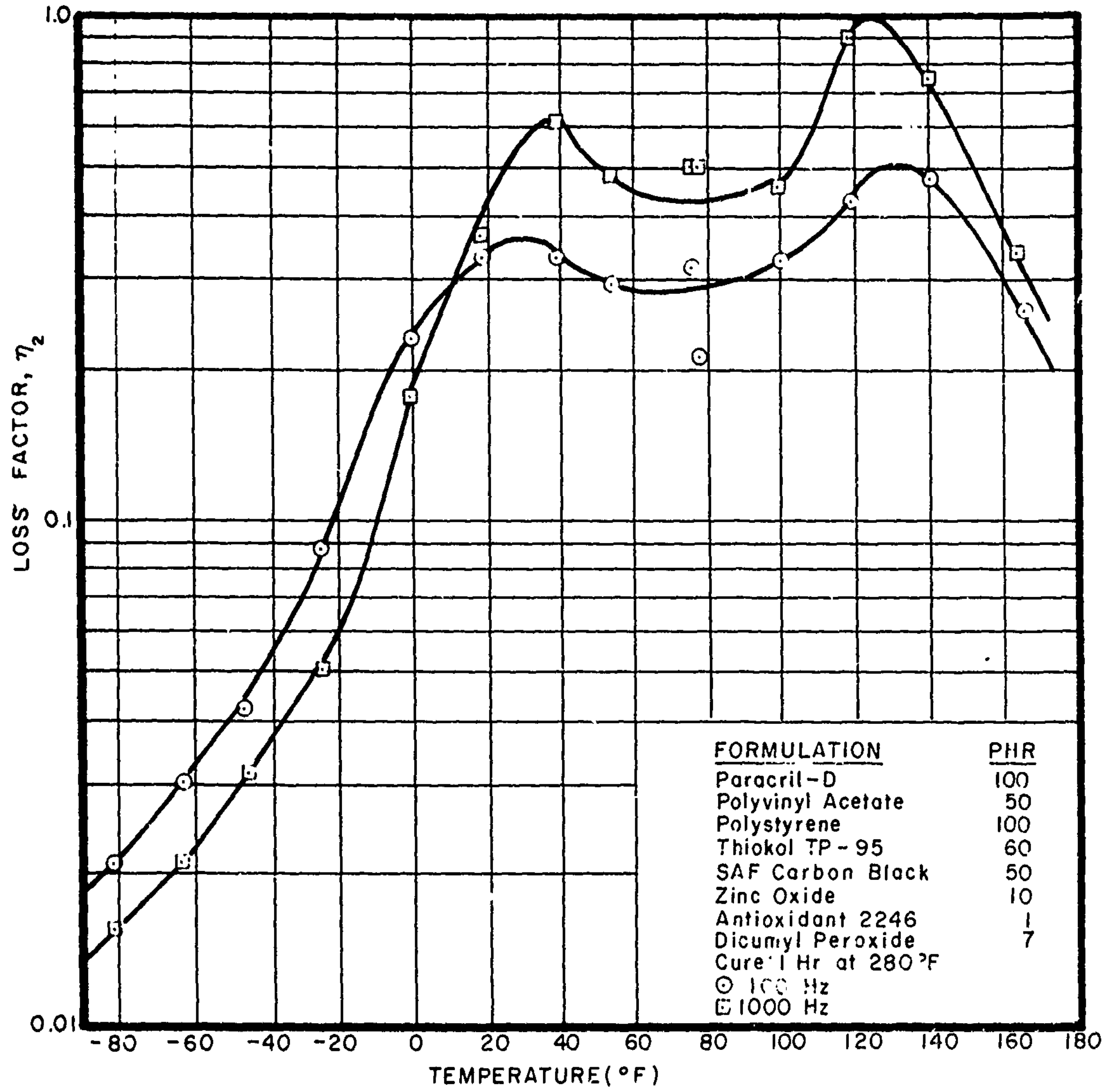

Figure 47. Loss Factor vs Temperature of a Potential Damping Material With 60 Parts Plasticizer Added

temperature range of any formulation studied (Figure 45). At 100 Hz the loss factor values were above 0.25 from 0° to 205°F, with peak values of 0.4, 0.5, and 0.65; at 1000 Hz the loss factor values were 0.5 from 20° to 170°F, with peak values of 0.6, 0.9, and 1.0. Increasing the amount of plasticizer to 60 parts resulted in somewhat lower loss factor values (Figure 47) and caused one loss factor peak to disappear. The width of the temperature range over which effective free-layer damping could be obtained was reduced by adding larger amounts of Thiokol TP-95. Thus, we concluded that Thiokol TP-95 is not equally effective in plasticizing all three polymers.

The polymer blend containing 45 parts plasticizer was a very good damping material because of its very high loss factor values. It was converted into a good material for free-layer damping over a low-temperature range (Figures 48 and 49) by replacing the Paracril-D with another nitrile rubber, namely Paracril-BJ*, having a lower glass transition temperature. This formulation, as is, would be an effective free-layer damping material over the temperature range -50° to 150°F and increasing the width of the temperature range would be an easy matter. These results, at least in principle, show the effects of adding a suitable plasticizer to a polymer blend are essentially the same as adding an appropriate plasticizer to a single polymer formulation (Reference 18).

One formulation (Figure 41) has been recommended (Reference 17) for applications where wide-temperature-range free-layer damping is required. The measured structural damping (Figure 41), or the composite losses, were very near 0.1 over the temperature range 30° to about 230°F for singly coated 1:1 weight ratio aluminum beams. This formulation was changed in two ways:

(1) The width of the temperature range where the measured composite losses were near 0.1 (Figure 52) was extended from somewhere below 0° to about 240°F; it should be noted that this was accomplished without increasing the width of the temperature range where the modulus and loss factor were above 10^4 psi and 0.1, respectively (Figures 50 and 51). These high composite losses over such a wide temperature range were attributed to the loss factor

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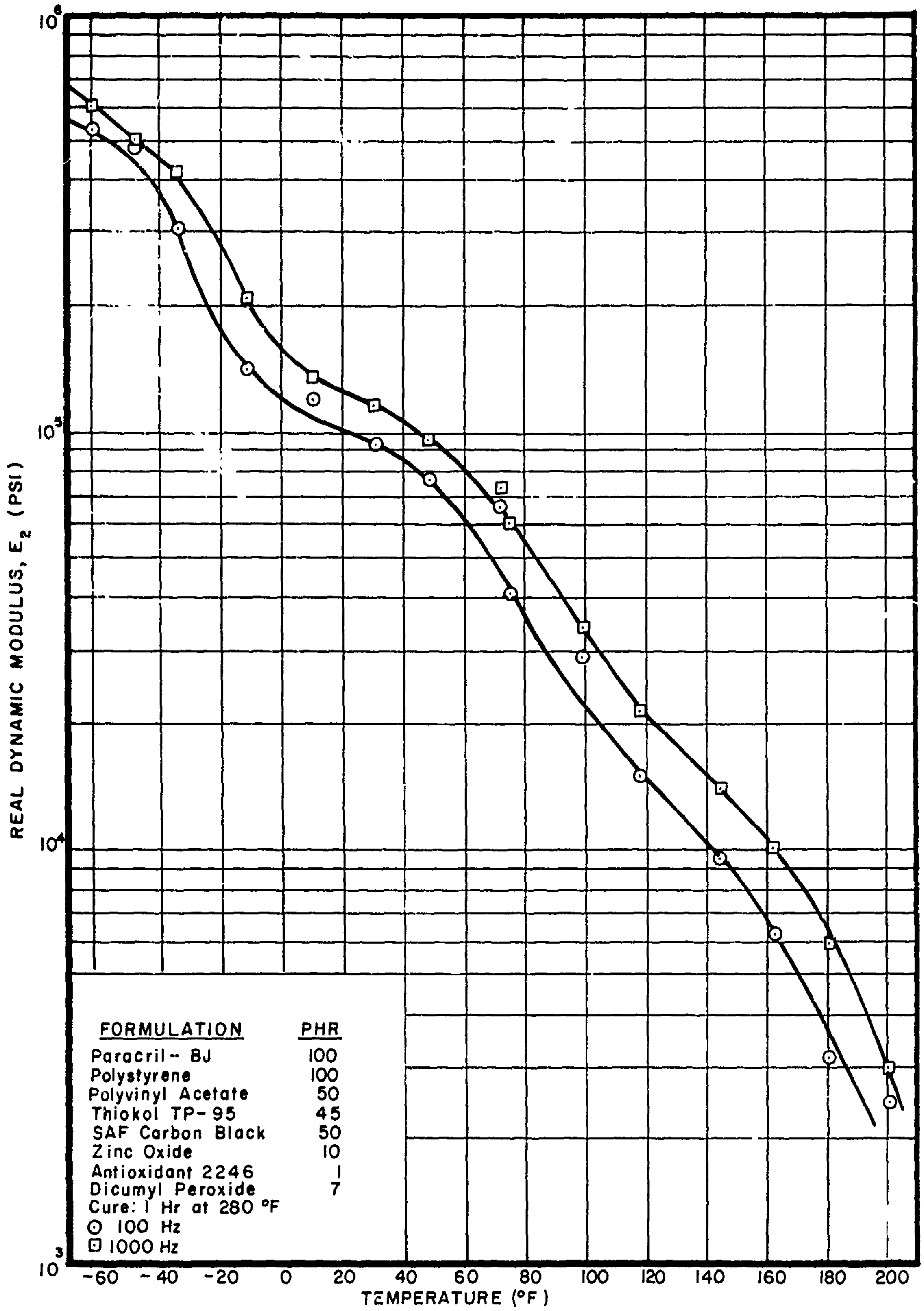

Figure 48. Real Dynamic Modulus vs Temperature of a Potential Low Temperature Damping Material

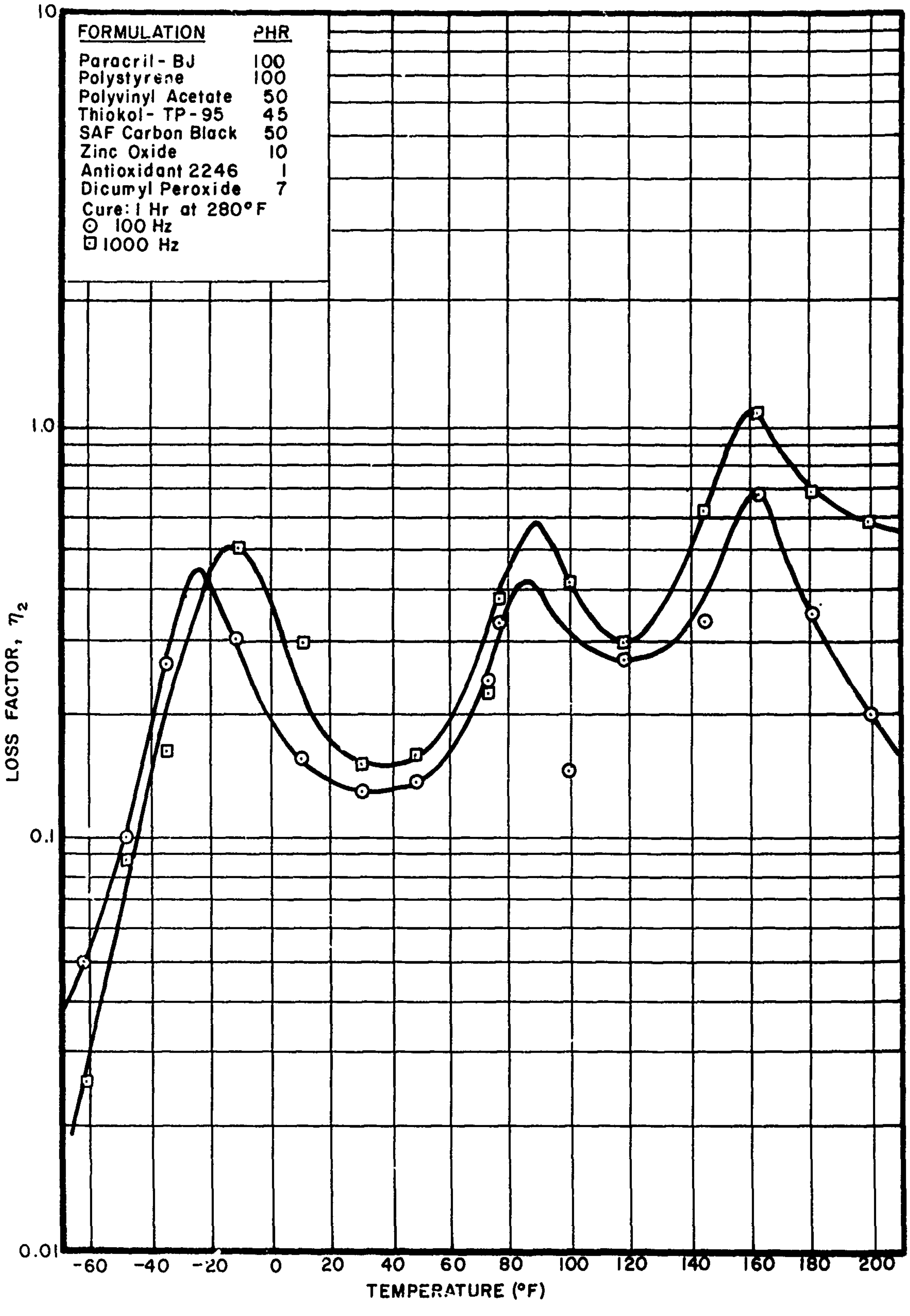

Figure 49. Loss Factor vs Temperature of a Potential Low Temperature Damping Material

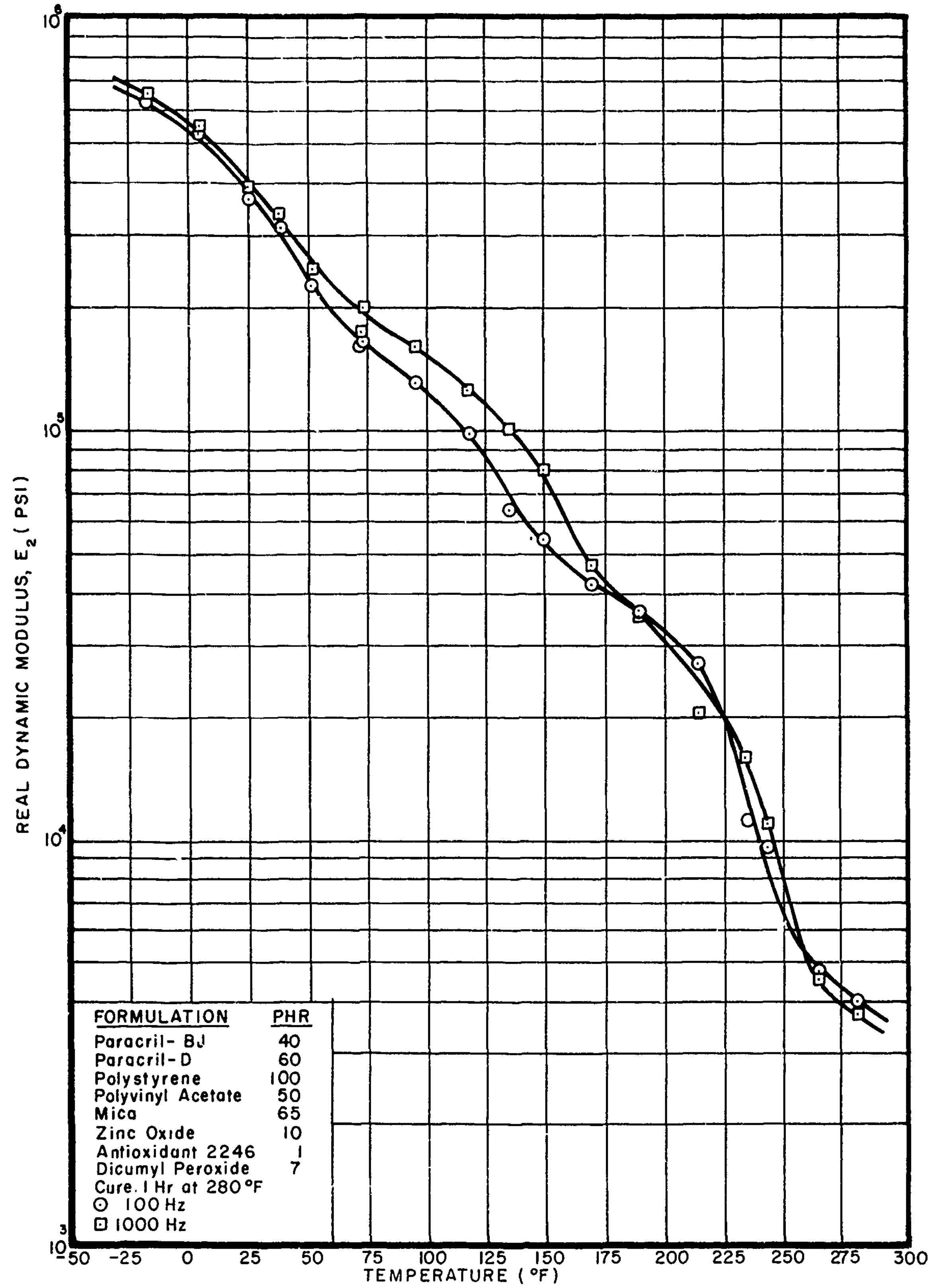

Figure 50. Real Dynamic Modulus vs Temperature of an Optimized Wide-Temperature-Range Damping Material Containing Mica

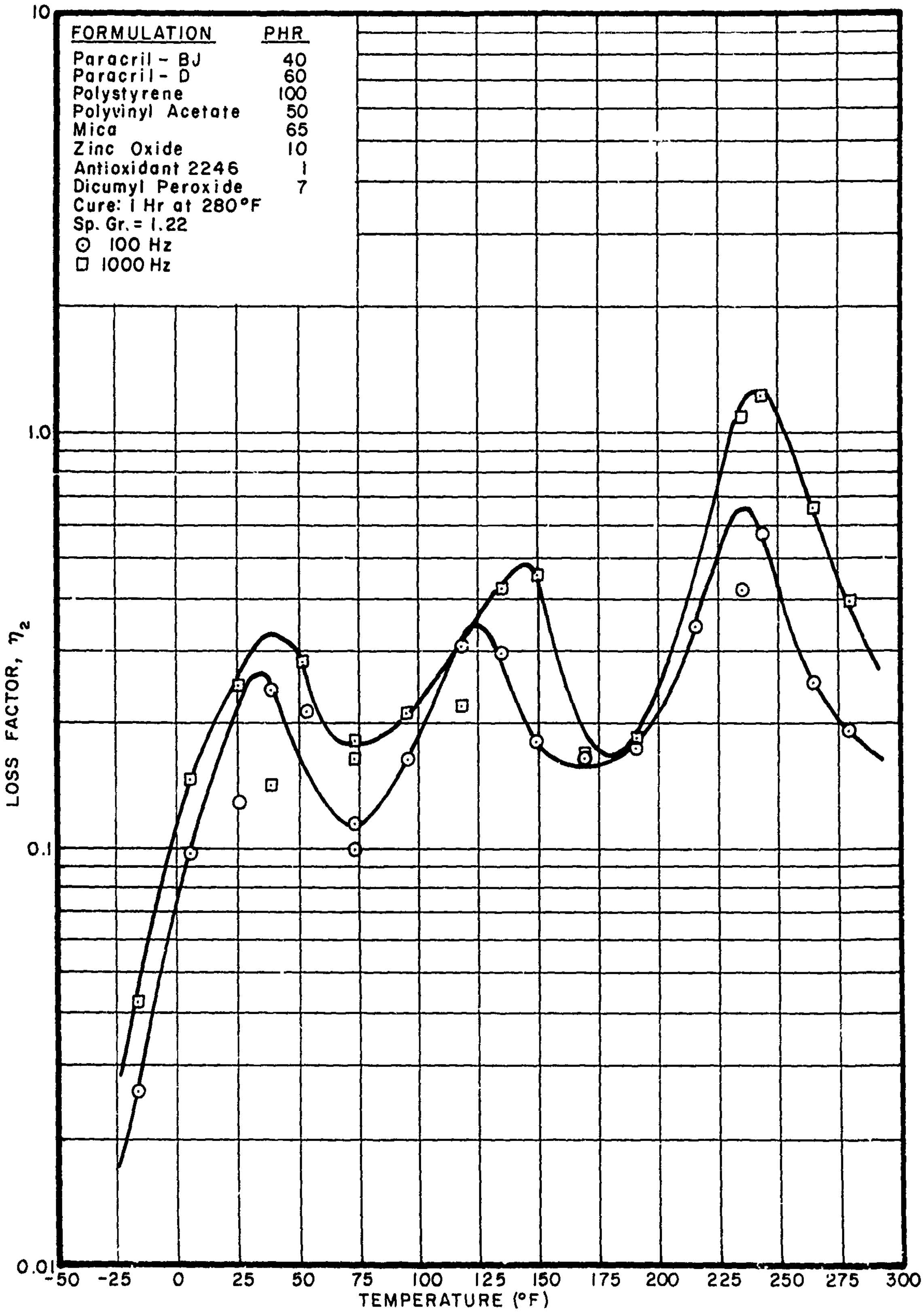

Figure 51. Loss Factor vs Temperature of an Optimized Wide-Temperature Range Damping Material Containing Mica

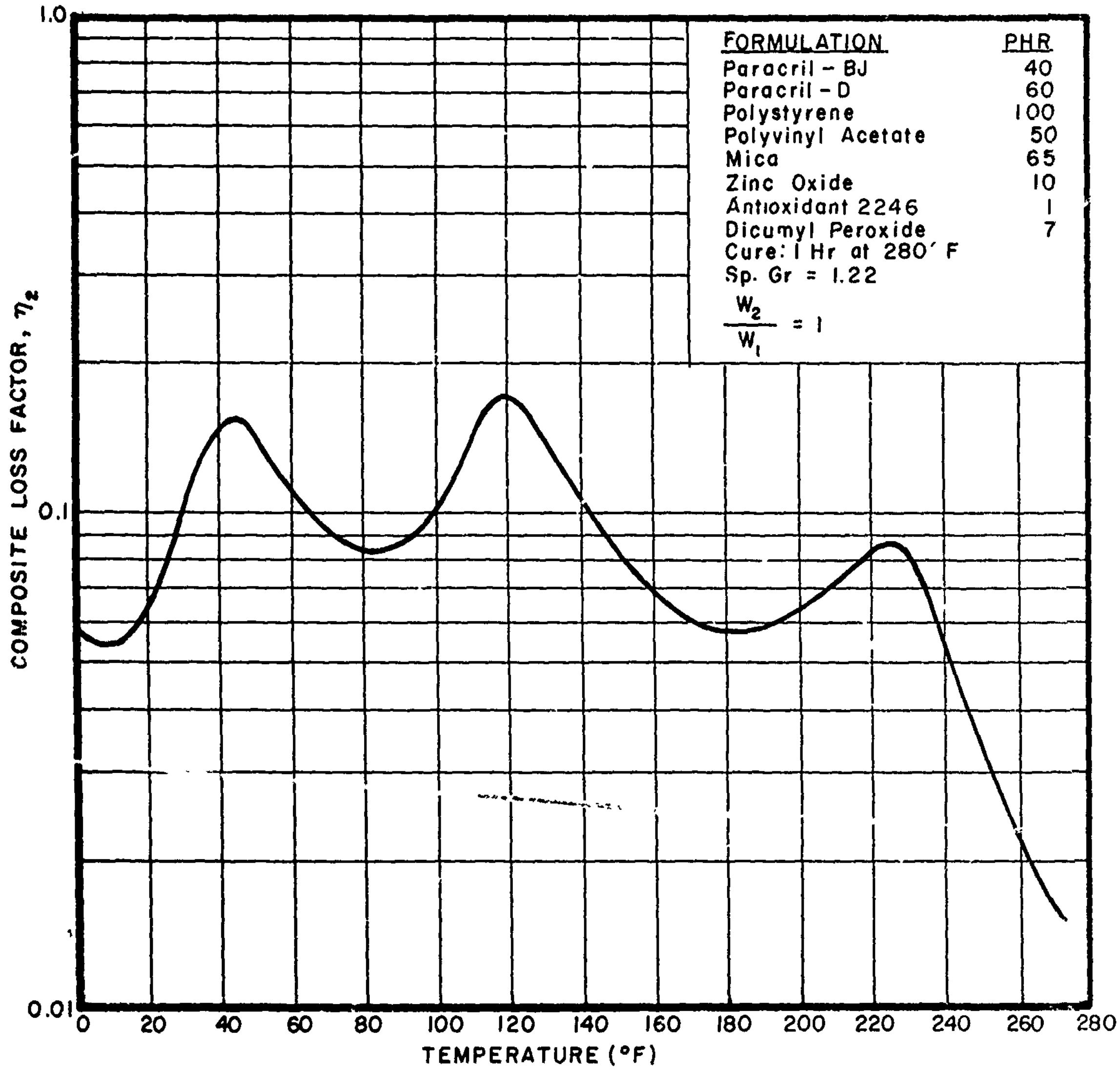

Figure 52. Measured Composite Loss Factor vs Temperature at 100 Hz of the Optimized Wide-Temperature-Range Damping Material

values being above 0.1 over the temperature range 0° to 300°F and to the temperature rate of the modulus change. Several other closely related formulations were evaluated, but their composite losses were not as good.

(2) The dynamic properties were improved (Figures 53 and 54), which, in turn, improved structural damping, particularly over the temperature range 20° to 240°F where the measured composite losses of a 1:1 weight-ratio, singly-coated beam averaged slightly more than 0.1 (Figure 55). The fact that all composite loss factor peaks were nearly the same height indicated the formulation had been optimized to give maximum damping efficiency over a wide temperature range. This fact was confirmed by evaluating formulations containing 55 and 75 parts mica. The formulation containing 75 parts mica did not process very well, while the formulation containing 55 parts mica (Figures 56 and 57) had less damping ability at the high-temperature end of the 0° to 300°F range.

The formulation (Figures 53, 54, and 55) containing 65 parts mica (3x Mineralite, Mineralite Sales Corp.) was selected for demonstrating that polymer blends can be produced in suitable form for any kind of field application requiring free-layer damping over a wide-temperature range. This particular polymer blend was cured with a peroxide curing system, but it could have been cured with a sulfur curing system (Figures 3 and 4); either can be used, but the sulfur curing system is preferred for field use of liquid-like room-temperature vulcanizing (RTV) damping treatments. The versatile polymer blend formulation presented in Table II has been mixed on a rubber mill in a solvent mixture of 1/3 (by weight) toluene and 2/3 methylethyl ketone, and in ammonia-stabilized aqueous suspensions containing about 30% total solids. Mill-mixed formulations have been dissolved or suspended in the mixed solvent having up to 50% total solids without gellation. These solutions have been used to coat horizontal and vertical plates by dipping, brushing, and trowelling, and with a caulking gun. Aqueous suspensions have been used for dipping and brush coating. All the liquid-like damping treatments were cured at room temperature (about 77°F) by adding 2 phr Accelerator 808* just prior to use. The shelf-life

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*E. I. duPont de Nemours & Co., Inc. Trademark

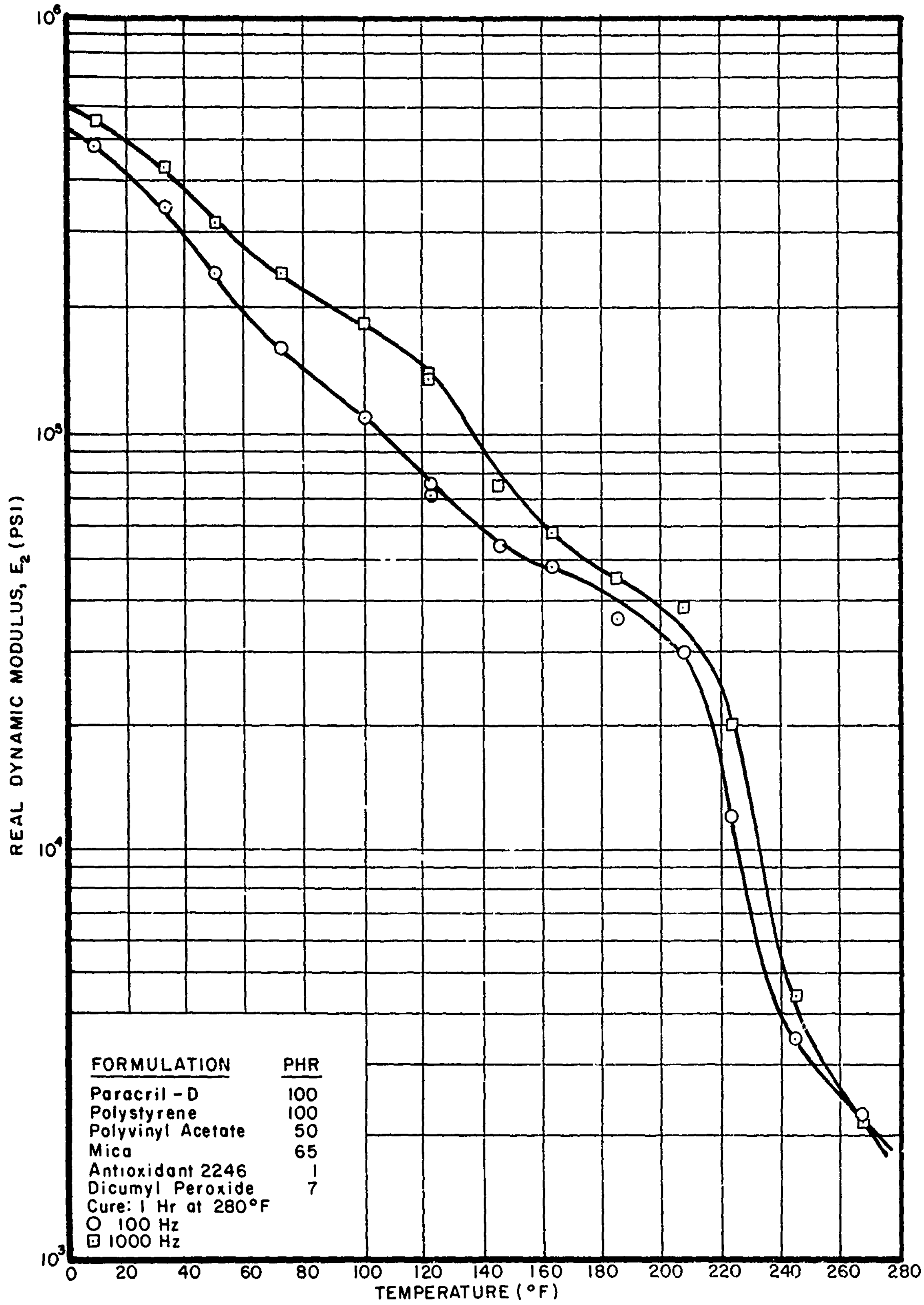

Figure 53. Real Dynamic Modulus vs Temperature of an Optimized Three-Polymer Blend

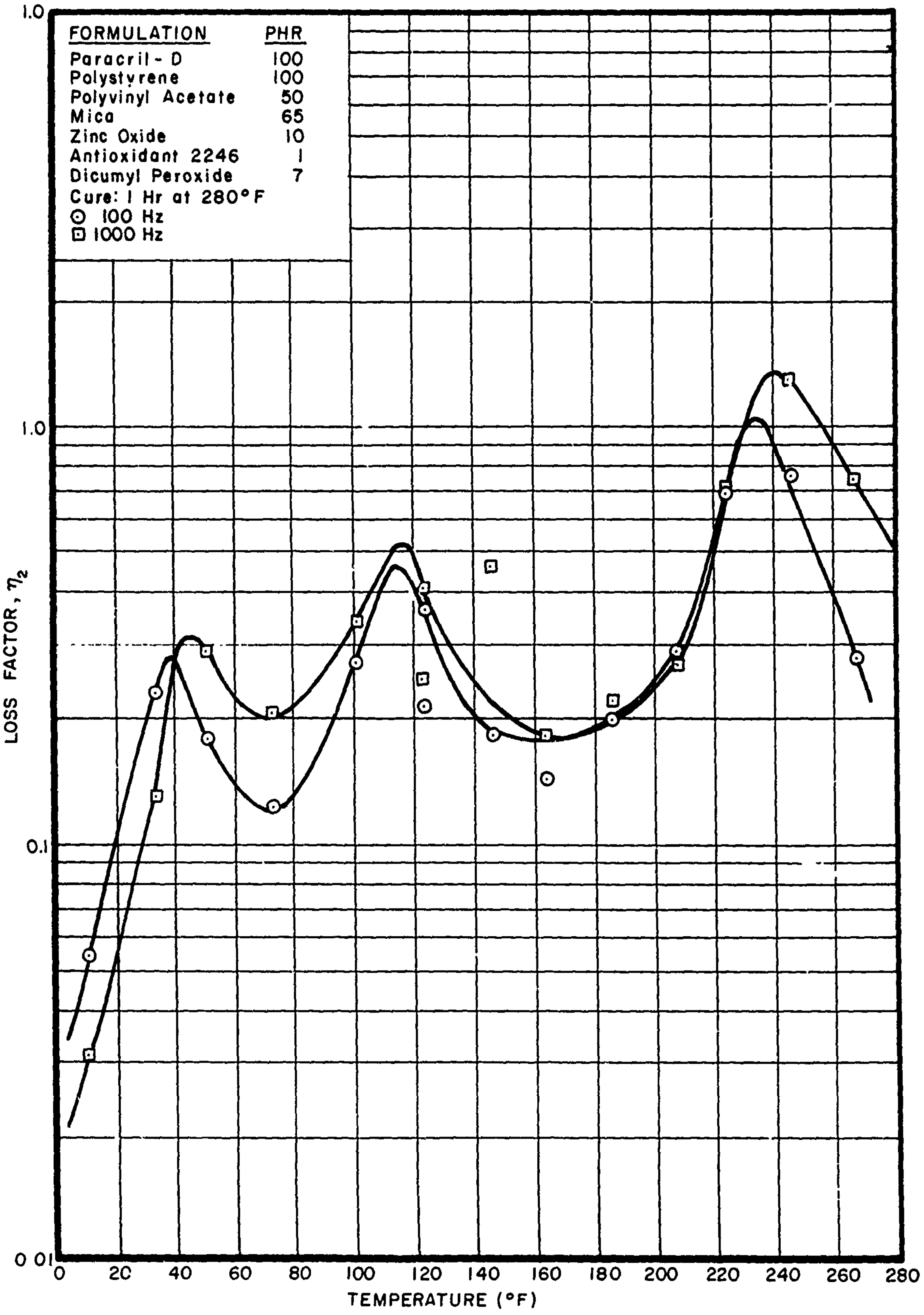

Figure 54. Loss Factor vs Temperature of an Optimized Three-Polymer Blend

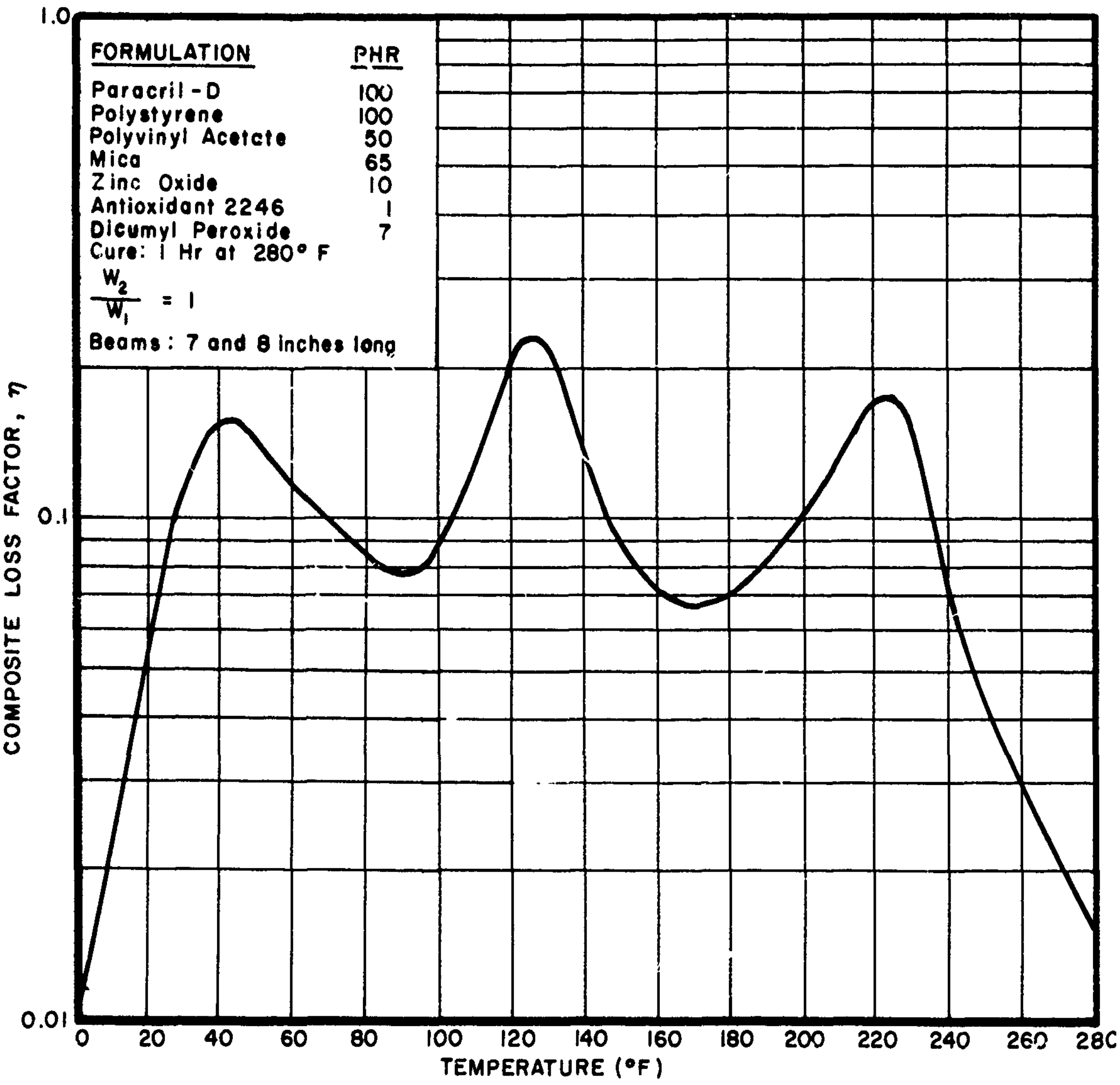

Figure 55. Measured Composite Loss Factor vs Temperature at 100 Hz of the Optimized Three-Polymer Blend

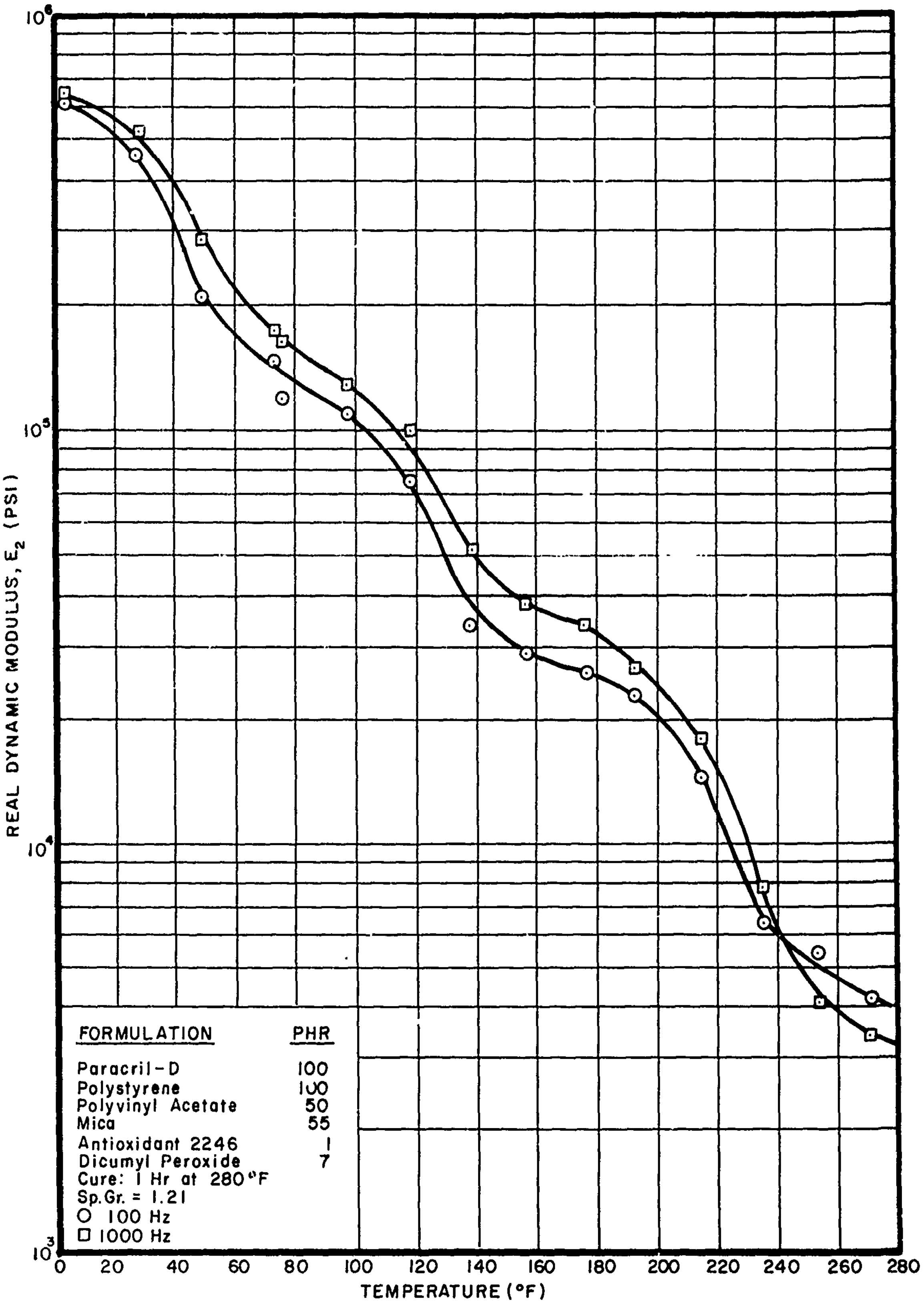

Figure 56. Real Dynamic Modulus vs Temperature of a Three-Polymer Blend With 55 Parts Mica Added

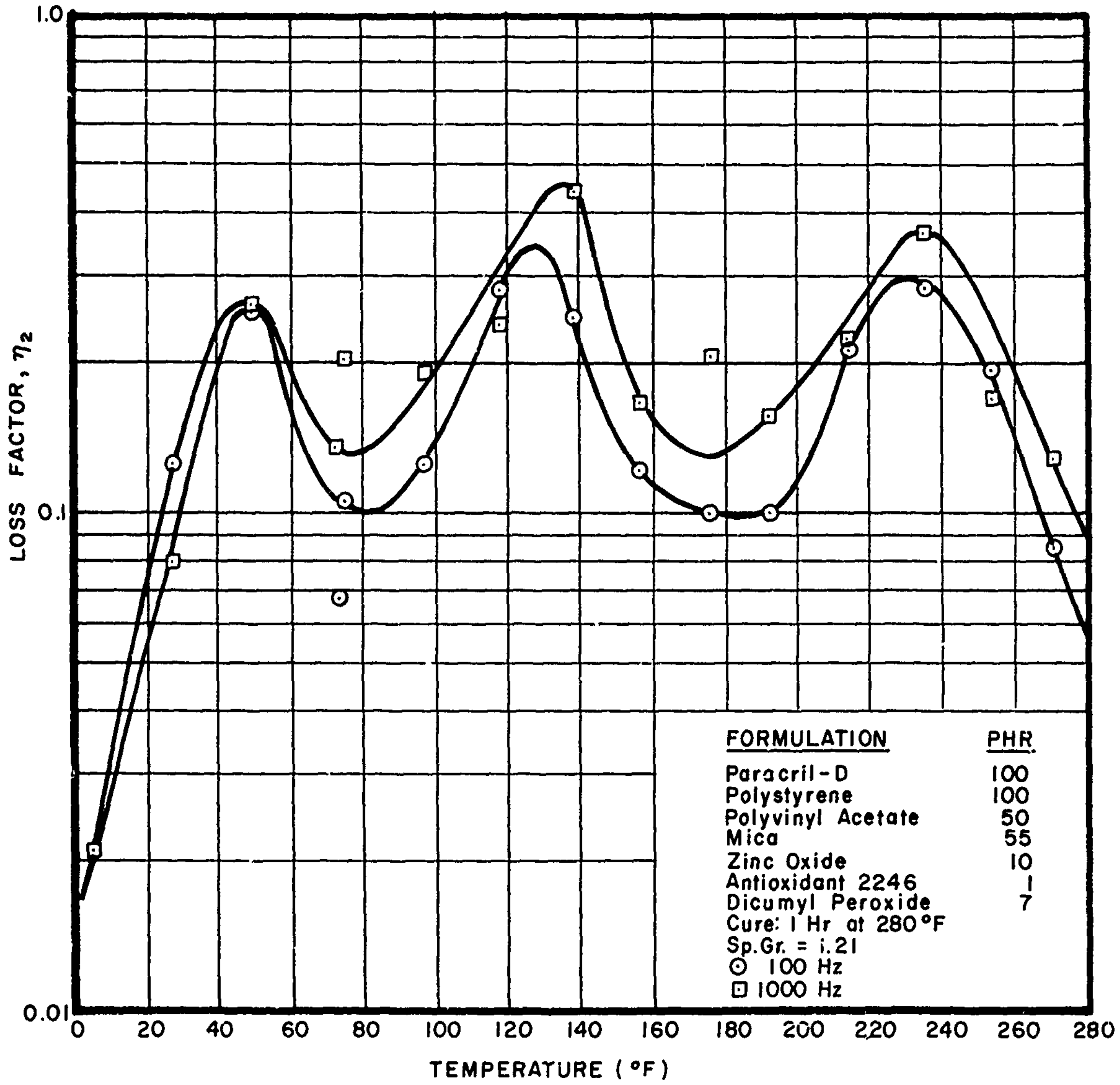

Figure 57. Loss Factor vs Temperature of a Three-Polymer Blend With 55 Parts Mica Added

TABLE II

A VERSATILE POLYMER BLEND

Component	PHR
Paracril-D	100
Polystyrene	100
Polyvinyl Acetate	50
Mica (3X Mineralite)	65
Antioxidant 2246	1
MBT (2-Mercaptobenzothiozole)	4
Sulfur	4

CURE: 20 minutes at 310°F in press or add 2 phr Accelerator 808 and cure at room temperature (77°F) for about 72 hours.

of liquid-like formulations not containing Accelerator 808 was found to be more than 6 months when stored at 77°F. It was also found that the liquid-like room-temperature-curing formulations would self-adhere to aluminum; good adhesion was obtained without additional primers or adhesives. These results show wide-temperature-range damping materials can be produced in a form which can be easily applied to almost any structure where damping may be required.

SECTION IV

CONCLUSIONS

1. The width of the temperature range over which an existing wide-temperature-range damping material, consisting of a lightly crosslinked physical blend of equal amounts by weight of polyacrylonitrile-butadiene (Paracril-D), polyvinyl acetate, and polystyrene has been substantially improved.

2. The damping ability of the experimental polymer blend, like that of material formulations containing only one polymer, depends on the modulus and loss factor values and how these values change with changes in the thermal environment. The modulus and loss factor values of the experimental polymer blends can be regulated somewhat like the modulus and loss factor values of single-polymer formulations, e.g., by incorporating reinforcing fillers, more curing agent, and/or plasticizers; in addition, damping characteristics may also be changed by varying the relative amounts or even the kinds of polymers in the blends.

3. The loss factor values of the experimental blends were varied within wide limits. For example, incorporating increasingly larger quantities of polyvinyl acetate into formulations containing equal weight amounts of polyacrylonitrile-butadiene and polystyrene resulted in the height of the loss factor peaks due to these two polymers being reduced, and a third peak appearing when sufficient polyvinyl acetate was added.

4. The modulus vs temperature values (fixed frequency conditions) of the polyacrylonitrile-butadiene, polyvinyl acetate, and polystyrene blends, as expected, underwent relatively drastic changes throughout the temperature range where each of the loss factor peaks occurred.

a. The temperature range over which each relatively drastic modulus change and/or loss factor peak occurred correlated, though not perfectly, with the glass transition temperatures of the polymers in the blends.

b. The extent of the modulus changes and/or the heights of the loss factor peaks correlated with the relative amounts of the polymers in the formulation.

c. The overall modulus of the blends was due primarily to the polystyrene, e.g., the highest Tg polymer.

5. The damping ability of free-layer damping treatments appears to correlate with the loss or imaginary modulus values (i.e., the product of the real modulus and loss factor values) of the formulations.

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