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MECHANICAL PROPERTIES OF BIS-PHENOL-A POLYSULFONE
AND ACETYLENE TERMINATED BIS-PHENOL-A BLEND

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Polymer Branch
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June 1983

Final Report for Period June 1982 to January 1983

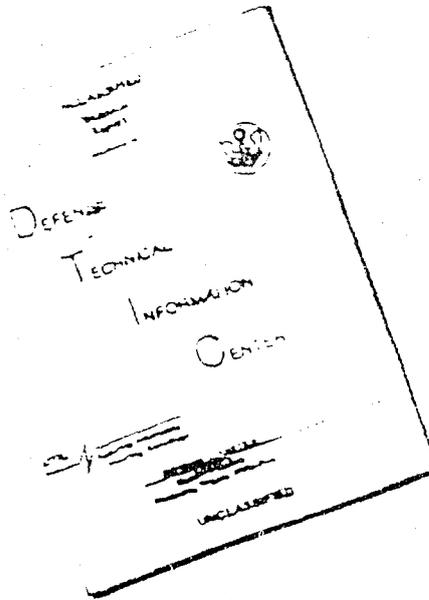
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This technical report has been reviewed and is approved for publication.



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FOR THE COMMANDER



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Torsion Impreganted Cloth Analysis (TICA) has been used as a technique to study the compatibility of two high temperature polysulfone thermoplastics with three acetylene terminated oligomers. Detailed property evaluations were made for blends of bisphenol A polysulfone (PSF) and 4,4'-(3-ethynylphenoxy)phenyl dimethylmethane (ATB). TICA and DSC measurements have shown the uncured blends to be compatible by the usual criterion of a single composition dependent glass transition over the range of compositions from 0 to 30 wt % ATB. A blend		

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containing 30% ATB was shown to be easily processed at 150°C, a temperature more than 100° lower than required to melt fabricate PSF alone. Tensile testing of the molded blends indicated embrittlement and moderate break strength. Nearly total cure of the ATB in these blends could be achieved by heating molded films to higher temperature (e.g., 225°C) for several hours. The curing process apparently leads to phase separation of the PSF component, but results in an overall improvement in tensile properties and suggestion of improved resistance to solvent attack.

FOREWORD

This report was prepared by the Polymer Branch, Nonmetallic Materials Division. The work was initiated under Project No. 2419, "Nonmetallic and Composite Materials," Task No. 241904, Work Unit Directive 24190415, "Structural Resins." It was administered under the direction of the Materials Laboratory, Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio with T. E. Helminiak as the AFWAL/ML Project Scientist. Dr. J. R. Fried, a visiting scientist of the Laboratory from the University of Cincinnati, and Dr. C.Y-C. Lee of AFWAL/MLBP are the authors.

This report covers research conducted from June 1982 to January 1983.

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SECTION I
INTRODUCTION

High temperature thermoplastic composites are potential substitutes for traditional epoxy based composites for use in structural aircraft components (Reference 1). Advantages of thermoplastic composites include high damage threshold, low moisture sensitivity, and fast fabrication time due to the absence of a separate cure stage. To satisfy a 350°F use temperature, candidates for thermoplastic composite matrices include the class of high temperature engineering thermoplastics with heat deflection temperatures in excess of 350°F (177°C). These include the polysulfones, principally bisphenol A polysulfone (PSF), polyphenylsulfone (PPS), and polyethersulfone (PES).

Among disadvantages of thermoplastic compared to thermoset composites are high processing temperatures required to form a prepreg which is often boardy and vulnerable to stress crazing and cracking in the presence of deleterious liquids such as kerosene and hydrocarbon based fuels. Potentially, some of these disadvantages can be corrected by blending the thermoplastic with a compatible reactive plasticizer (References 2, 3) such as the acetylene terminated (AT) oligomers developed at Wright-Patterson AFB. These materials have low glass transition temperatures (T_g 's) compared to the thermoplastic and can be reacted by thermal treatment to form a network. The resulting lower T_g of the thermoplastic/oligomer mixture has the potential of prepreg fabrication at substantially reduced temperatures and the subsequent network formation provides a basis for improved solvent resistance.

Several thermoplastic/reactive plasticizer systems have been or are in process of being studied by WPAFB personnel. Wereta et al (Reference 2) have shown that blends of PSF and 4,4'-bis-(3-ethynylphenoxy) diphenyl sulfone (ATS) are compatible by the common criterion of a single T_g . Blends containing 30 wt % ATS could be molded at 325°F (163°C) well below typical molding temperatures for unblended PSF. Goldfarb and Sikka (Reference 3) have reported similar improvement in processing of compatible blends of PPS and ATS. In addition, fully cured PPS/ATS blends

were found to show improved resistance to stress failure in solvent environment. Properties of blends of PES and ATS are currently being investigated (Reference 4). The present study reports preliminary observations of the compatibility of PSF and PES with three AT oligomers (structures given in Figure 1) and detailed thermal, mechanical, and rheological properties of blends of PSF and 4,4'-bis-(3-ethynylphenoxy) phenyl dimethylmethane (ATB).

SECTION II

EXPERIMENTAL

Samples for torsion impregnated cloth analysis (TICA) were prepared by immersing a 4" wide strip of Stevens (Reference 5) heat cleaned fiberglass cloth (S/7781) into a 10% (W/V) polymer/methylene chloride solution. The soaked cloth was then hung to dry in a hood for about two hours and placed in a vacuum oven for at least six days at room temperature for further solvent removal. Two strips, cut and folded, as illustrated in Figure 2, were used to prepare a single TICA sample by procedures described elsewhere (Reference 6).

Polymers used in this study were Union Carbide P-1700 PSF pellets and ICI 200P PES reactor powder. Both were dried at 100-120°C under vacuum prior to use. A thick film of PSF was molded at 300°C by placing 6.5 gm of the dried polymer in a 3" x 3" cutout of a 6" x 6" x 0.030" stainless steel shim. The shim was sandwiched between two 3/8" steel plates and placed in a hydraulic press whose platens were heated to 572°F (300°C). After 13 minutes, the heaters were turned off and the mold was allowed to cool to 392°F (200°C) at a pressure of 4,400 lbs before removal. Two disks were cut from the resulting film by use of a 1-1/8" hole cutter. These were then pressed together at 343°F (173°C) to give a single disk, 1.88 mm thick and about 2.50 cm in diameter for parallel plate rheological measurements.

A molding resin of PSF blended with 30 wt % ATB (PSF/30-ATB) was prepared by casting clear films from a 10% solution in methylene chloride onto glass plates with a Gardner casting knife. These were dried one week in vacuum at room temperature, stripped in water, and ground to a powder with dry ice in a CRC micro-mill. The resulting powder was dried an additional 11 days in vacuum. Prior to molding, the powder was heated to 212°F (100°C) for two hours under vacuum (5.5 wt % loss). About 6 gms were molded at 324°F (162°C) in the stainless steel shim for 17 minutes. The resulting film contained many small voids (probably due to residual solvent). A film suitable for rheological measurements was made by stacking small pieces of the voided film and molding at 284°F (140°C) for 17 minutes.

Microtensile (ASTM-1708) specimens of the PSF/30-ATB blend were prepared by pressing the powder in a break-away mold at 302°F (150°C). Some samples were cured at 437°F (225°C) in a circulating air oven.

Dynamic melt properties were determined by means of a Rheometrics Dynamic Spectrometer (RDS) equipped with a TC-2,000 transducer, EC-3000 environmental chamber, and operating in the parallel plate (2.50 cm disk) mode. TICA measurements were obtained by use of the RDS or Rheometrics Mechanical Spectrometer (RMS-7200) with an EC-3000 environmental chamber and TC-10,000 transducer. Differential Scanning Calorimetry (DSC) measurements were made with a Dupont 990 instrument at a heating rate of 20°/min through the efforts of Mr. Edward J. Soloski. Tensile specimens were prepared and tested through the courtesy of Capt. Phil Mykytiuk of MLCB:

SECTION III

RESULTS AND DISCUSSION

1. COMPATIBILITY STUDIES

A preliminary study of the compatibility of PSF and PES with the AT oligomers identified in Figure 1 was made by inspection of the TICA glass transition(s) of each blend. TICA curves (loss and storage moduli) for unblended PSF and PES are shown in Figure 3. Glass transition temperatures (T_g 's) are summarized in Table 1. TICA spectra of blends containing 30 wt % of the AT oligomers are shown in Figures 4 and 5. Both PSF/30-ATS and PSF/30-ATB exhibit a single although broadened glass transition whose temperature at the maximum of the loss curve (b) is indicated by arrows in Figure 4. For the case of PSF/30-ATP, two glass transitions appear merged. These may represent transitions of an ATP-rich and PSF-rich phase at low (ca. 47°C) and high (ca. 102°C) temperatures, respectively.

Results of TICA measurements of the PES blends are shown in Figure 5. The loss modulus curve of PES/30-ATS shows two merged transitions suggestive of the same partial miscibility observed for PSF/30-ATP. Only the glass transition of unblended ATB is evident for PES/30-ATB. This is indicative of macrophase separation which is also suggested by opacity and extreme brittleness of cast films of PES/30-ATB. Insufficient ATP was available to investigate the blend properties of PES and ATP. General conclusions of blend capability are summarized in Table 2.

In addition to the loss peaks corresponding to the glass transition of the uncured blend, each blend exhibits a high temperature vitrification peak. These are very broad for the PSF blends (Figure 4) but relatively narrow for the PES blends (Figure 5). Discussion of the effect of blending on the cure process will be discussed later.

2. PSF/ATB BLENDS

Based on the conclusions concerning blend compatibility cited previously, availability of the AT oligomers, and in recognition of previous studies of PSF/ATS blends and present studies of PES/ATS, a decision was made to investigate in detail the properties of the PSF/ATB

system. Results of TICA measurements of the uncured (first heating) blends are shown in Figures 6 and 7. These show a single although broadened glass transition loss peak for each blend. Broadening increases with increasing ATB composition. This suggests decreasing compatibility with increasing ATB content. Location of the temperature at the maximum of each loss peak is plotted as a function of wt % ATB in Figure 8. Results of DSC T_g measurements ($1/2 \Delta C_p$ at $20^\circ/\text{min}$) for vacuum dried films are illustrated in Figure 9. Comparison is made with values of T_g predicted from the inverse rule of mixtures or Fox Equation (Reference 7) given as

$$\frac{1}{T_g} = \frac{W_1}{T_{g,1}} + \frac{W_2}{T_{g,2}} \quad (1)$$

where W represents the weight fraction of component 1 or 2. This relation has been shown, both experimentally and theoretically, to yield poor agreement with experimental values of T_g for polymer/plasticizer mixtures as evident by both dynamic mechanical and DSC data for the PSF/ATB system (Figures 8 and 9, respectively). Better agreement is afforded by use of the Fried Equation (Reference 8)

$$\ln(T_g/T_{g,1}) = \frac{W_2 \ln(T_{g,2}/T_{g,1})}{W_1 (T_{g,2}/T_{g,1}) + W_2} \quad (2)$$

which is obtained from the more general, theoretically derived Couchman-Karasz relation (Reference 9) when the Simha-Boyer rule (Reference 10) for the constancy of the product $T_g \Delta C_p$ is valid. As shown by thermal data given in Table 3, $T_g \Delta C_p$ is approximately constant for PSF and ATB and agree with values given by Boyer (Reference 11) and Fried (Reference 8) for other polymers and plasticizers. It is noted that although agreement is improved by use of Equation 2 over Equation 1, experimental values of T_g are overestimated by theoretical or empirical predictions at increasing ATB composition. Similar observations have been made for other reactive plasticizer blends

(References 2, 3). One possible reason for this observation is the extreme broadening and skewness of the glass transitions of these blends which may arise from a state of partial immiscibility or phase separation and makes assignment of a single T_g value more tenuous. Equation 2 is derived for a state of thermodynamic miscibility for which T_g is calorimetrically defined. Better agreement between experimental and predicted T_g values is obtained by use of $\tan \delta$ maximum values (Figure 8) or if the midpoint rather than the maximum of the loss modulus peaks is used to determine T_g . Values of predicted and experimental values of T_g obtained by different methods are summarized in Table 4.

TICA spectra for PSF, PSF blends, and ATB after TICA heating to 350°C (N_2) are shown in Figures 10 and 11. As shown by the loss modulus data appearing at the bottom of Figure 10, cured ATB exhibits a broad glass transition with a maximum near 280°C. The blends exhibit a prominent sharp loss maximum near 193°C corresponding in temperature with that of unblended PSF and a broad, weak loss maximum near 237°C. This maximum increases in intensity with increasing ATB composition. It is noted that unblended PSF also shows a broad, somewhat more pronounced peak loss at slightly higher temperature (243°C). This has been associated with a liquid-liquid transition (T_{ll}) (Reference 12) which is reported to be suppressed in a crosslinked system (Reference 13). The fact that the high temperature relaxation of the blends increases with increasing ATB composition, *i.e.*, with increasing network formation, would suggest that this peak probably cannot be associated with a T_{ll} . More likely, the low temperature peak can be identified with a PSF phase and the high temperature peak with an ATB phase formed during cure. One method of evaluating this contention is to determine the apparent activation energy (ΔH) of each relaxation according to the Arrhenius relation

$$f = A \exp(\Delta H/RT) \quad (3)$$

where f is frequency (in Hz), A is a constant, R is the ideal gas constant, and T is absolute temperature. Representative PSF TICA loss modulus curves at several frequencies are shown in Figure 12. As shown

by Figure 13, the frequency dependence of both $T_{\ell\ell}$ and T_g can be represented by straight lines in an Arrhenius plot of frequency versus reciprocal temperature (K^{-1}) at the loss maxima over the range of experimental frequencies (0.1 to 100 rad/sec). Activation energies were calculated from the slopes to be 82.9 and 226 kcal/mole for $T_{\ell\ell}$ and T_g , respectively. A high temperature loss maximum also is evident at high frequencies from dynamic parallel plate measurements of PSF melt as shown by represented curves in Figure 14. Activation energy of this peak (Figure 13) is 77.3 kcal/mole which agrees well with that obtained for the TICA $T_{\ell\ell}$ relaxation although the locations of the melt relaxations occur at consistently lower temperatures. This result suggests a common basis for both relaxations - a contention which will not be further pursued here.

The frequency dependence of the loss modulus of cured PSF/30-ATB blend is illustrated in Figure 15. The apparent activation of the low temperature relaxation is 241 kcal/mole which is comparable to the value of 226 kcal/mole found for unblended PSF. In contrast, the high temperature relaxation is highly broadened and temperature sensitive. Activation energy was calculated to be 73.9 kcal/mole. Both its temperature location and frequency dependence is comparable to the $T_{\ell\ell}$ relaxation of unblended PSF in opposition to the earlier contention that it may be associated with a separate ATB or ATB-rich phase glass transition. Future study of the frequency dependence of the loss maximum of cured ATB may help to clarify the significance of this high temperature relaxation.

The effect of blending ATB on the melt rheology of PSF is illustrated in Figure 16. As shown, a previously melt pressed PSF/30-ATB disk slowly cures at the melt temperature of 150°C over a one hour period, but its viscosity is still comparable to that of unblended PSF at its normal processing temperature of 260 to 270°C. During heating from 150 to 223°C (region B), viscosity first decreases and then increases as additional, more rapid cure occurs.

Preliminary results of tensile testing uncured and cured PSF/30-ATB microtensile specimens are summarized in Table 5. Most specimens contained small voids which can act as stress concentrators leading to premature failure. Blending thoroughly degassed resins without the use of a solvent (dry blending) should eliminate all voiding problems. For this reason, maximum values of blend tensile properties for small sample populations (5 uncured; 2 cured) are given for comparison with results for unblended PSF. Both uncured and cured samples failed in a brittle mode unlike unblended PSF which yields and cold draws. Results suggest that the uncured (compatible) blends act as antiplasticized systems which display elevated modulus and small elongation to break. Cure, which leads to phase separation suggested by the TICA studies cited previously, results in restoration of some of the properties of the matrix (PSF) including high break strength and respectable elongation to break.

Although resistance to solvent attack was not studied in any detail, some observations were made. A piece of cured PSF/30-ATB resin was placed in a beaker with methylene chloride, a good solvent for both PSF and uncured ATB. There was no immediate signs of dissolution over a 24-hour period although there was evidence for swelling and some leaching of PSF which could be recovered by casting the clear solvent after sample immersion.

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TABLE 1

TICA TRANSITION TEMPERATURES OF PSF AND PES

	<u>PSF</u>	<u>PES</u>
RMS No.	8291	8292
T_g , °C (loss modulus)	192	234
T_g , °C (tan δ)	198	240

TABLE 2

COMPATIBILITY OF BLENDS

	<u>PSF</u>	<u>PES</u>
ATS	yes	marginal
ATB	yes	no
ATP	marginal	unknown

TABLE 3

DSC THERMAL DATA

	T_g (°C)(K)	ΔC_p (cal/gm-K)	$T_g \Delta C_p$ (cal/gm)
ATB	-13 260	0.1119	29.1
ATB*	- 3 270	0.1003	27.1
PSF	186 459	0.0645	29.6
PSF/10-ATB	139 412	-	-
PSF/30-ATB	62 335	-	-

*Degassed 1 hr. at 83-85°C in vacuum.

TABLE 4
GLASS TRANSITION TEMPERATURES (K)

<u>W</u> <u>ATB</u>	DSC		b peak max.			tan δ max			
	<u>exp.</u>	<u>Eq. 1</u>	<u>Eq. 2</u>	<u>exp.</u>	<u>Eq. 1</u>	<u>Eq. 2</u>	<u>exp.</u>	<u>Eq. 1</u>	<u>Eq. 2</u>
0	459	-	-	467	-	-	469	-	-
0.1	412	426	418	422	439	432	437	442	437
0.2	-	-	-	382	414	404	405	419	410
0.3	335	373	359	355	391	380	375	397	387
1.0	260	-	-	284	-	-	293	-	-

TABLE 5
TENSILE PROPERTIES OF PSF/30-ATB

<u>Property</u>	<u>PSF^a</u>	<u>Uncured Resin^b</u>	<u>Cured Resin^c</u>
Young's Modulus (psi)	352,000	500,000	363,000
Break Strength (psi)	6,500	6,700	10,140
Yield Strength (psi)	8,500	-	-
Elongation to Break (%)	45	1.4	4.6
Elongation to Yield (%)	5.3	-	-

a. J. R. Fried and H. M. Kalkanoglu, Polym. Eng. Sci. (submitted).

b. Molded at 150°C

c. Cured at 225°C for one hour in a circulating air oven

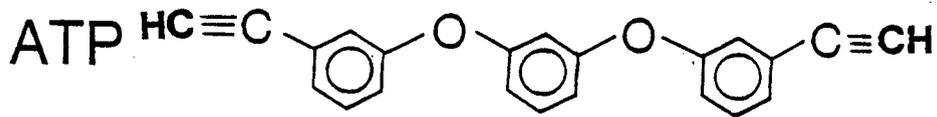
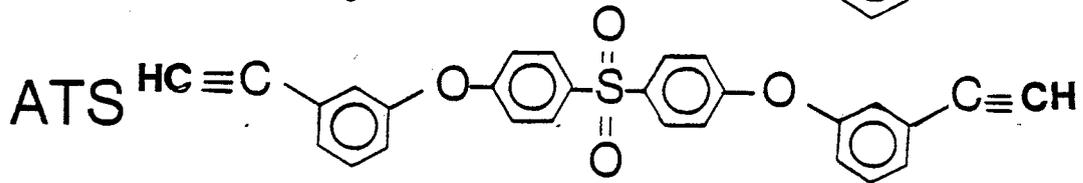
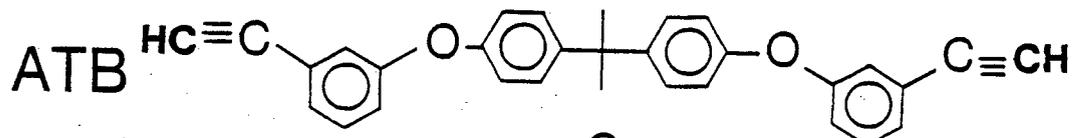
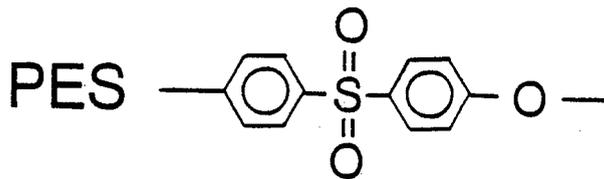
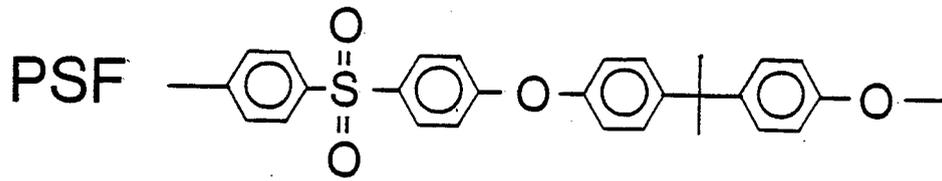


Figure 1. Structures of Repeat Units of Bisphenol A Polysulfone (PSF), Polyethersulfone (PES), and Three AT Oligomers.

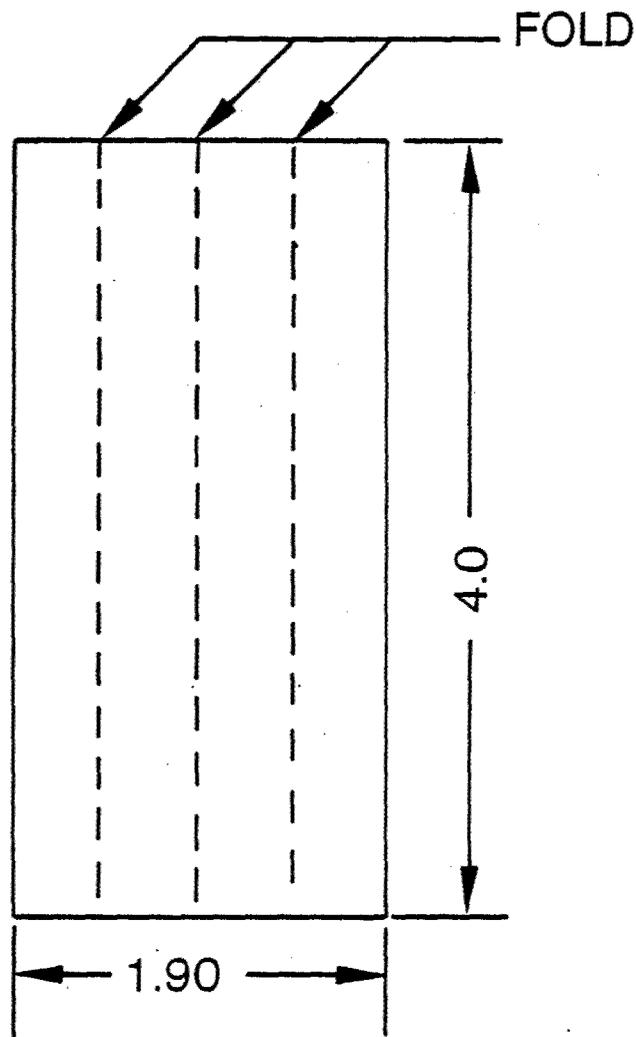


Figure 2. TICA Sample Dimensions (Inches).

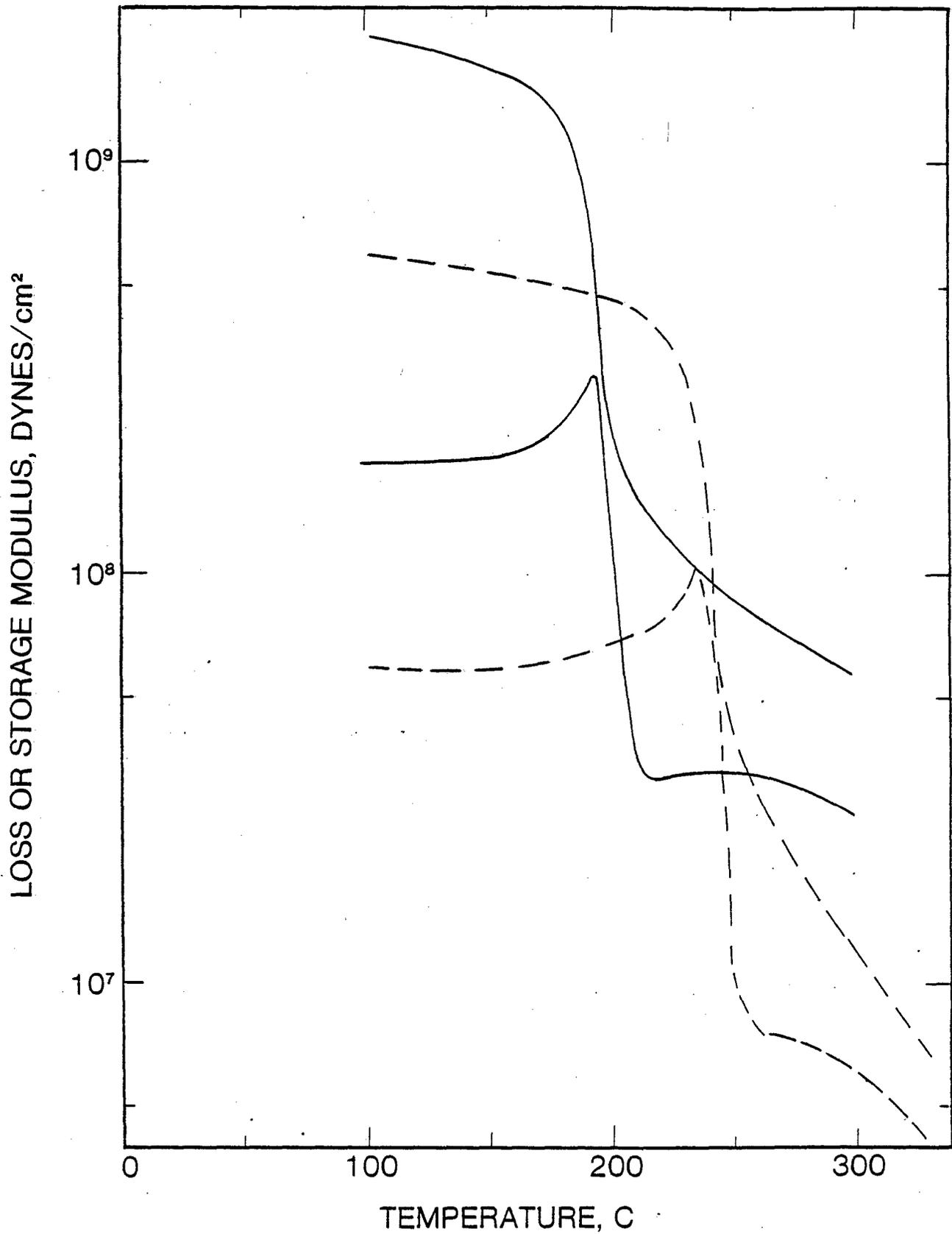


Figure 3. Plots of Storage (a) and Loss (b) Moduli of PSF (-) and PES (--) Obtained During Second Heating at 10 sec/rad (1.6 Hz) from RMS Measurements. Curves have been arbitrarily shifted along ordinate.

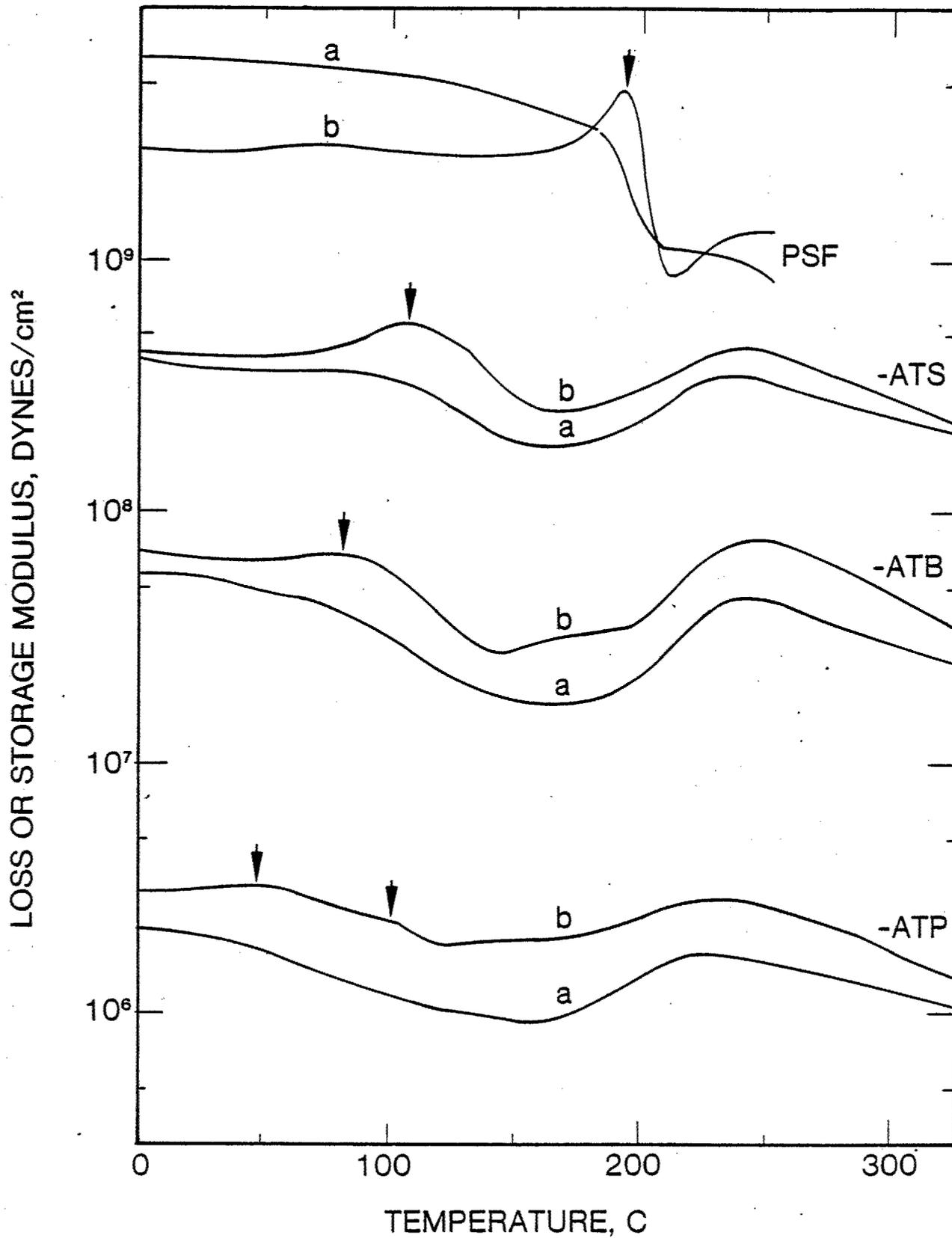


Figure 4. Plots of Storage (a) and Loss (b) Moduli of PSF and Three PSF Blends Containing 30 wt % of an AT Oligomer (All First Heat). Each curve is arbitrarily shifted along the ordinate. Arrows indicate approximate location of the loss maximum or maxima at the glass transition.

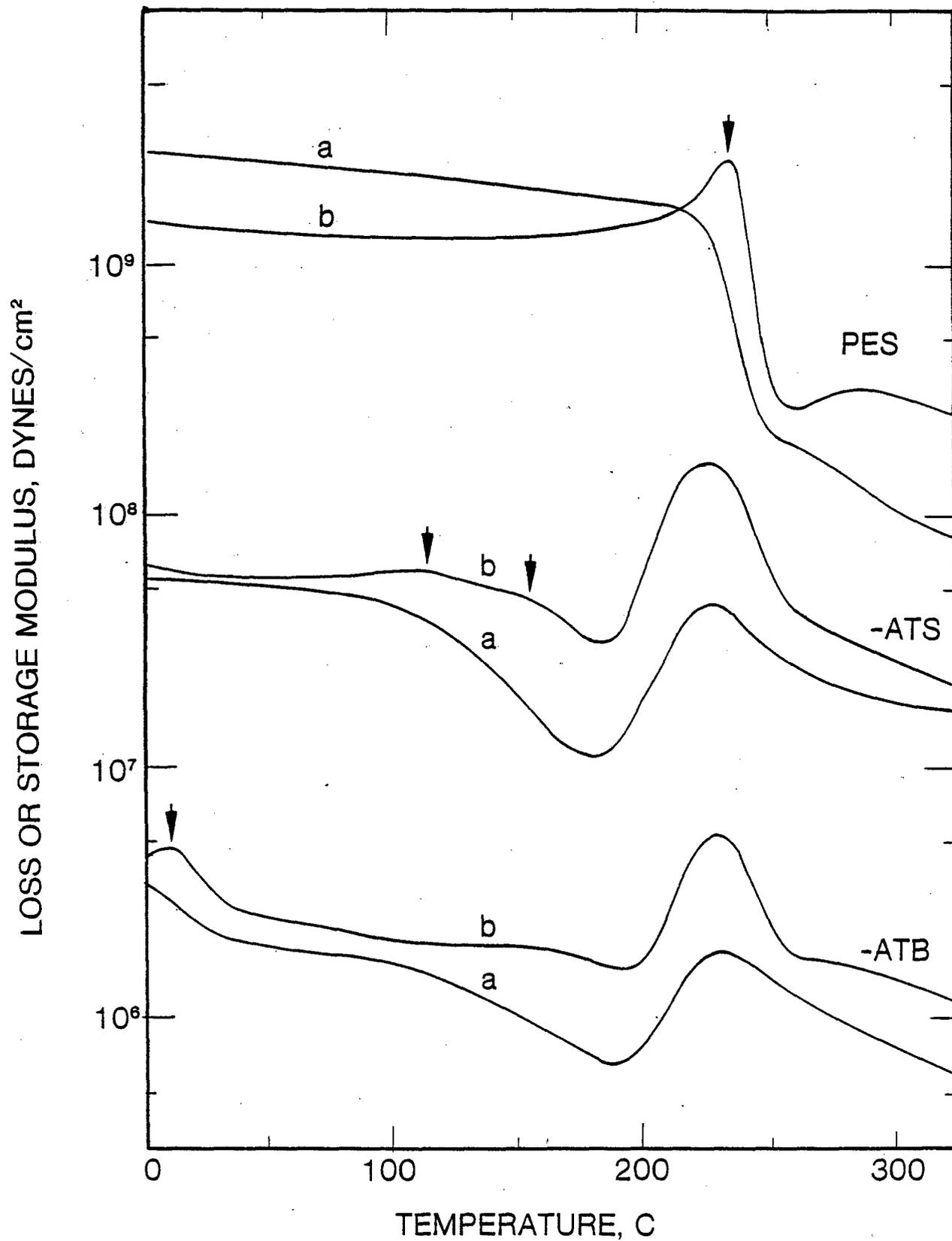


Figure 5. Plots of Storage (a) and Loss (b) Moduli of PES and Two Blends. Legend is same as in Figure 4.

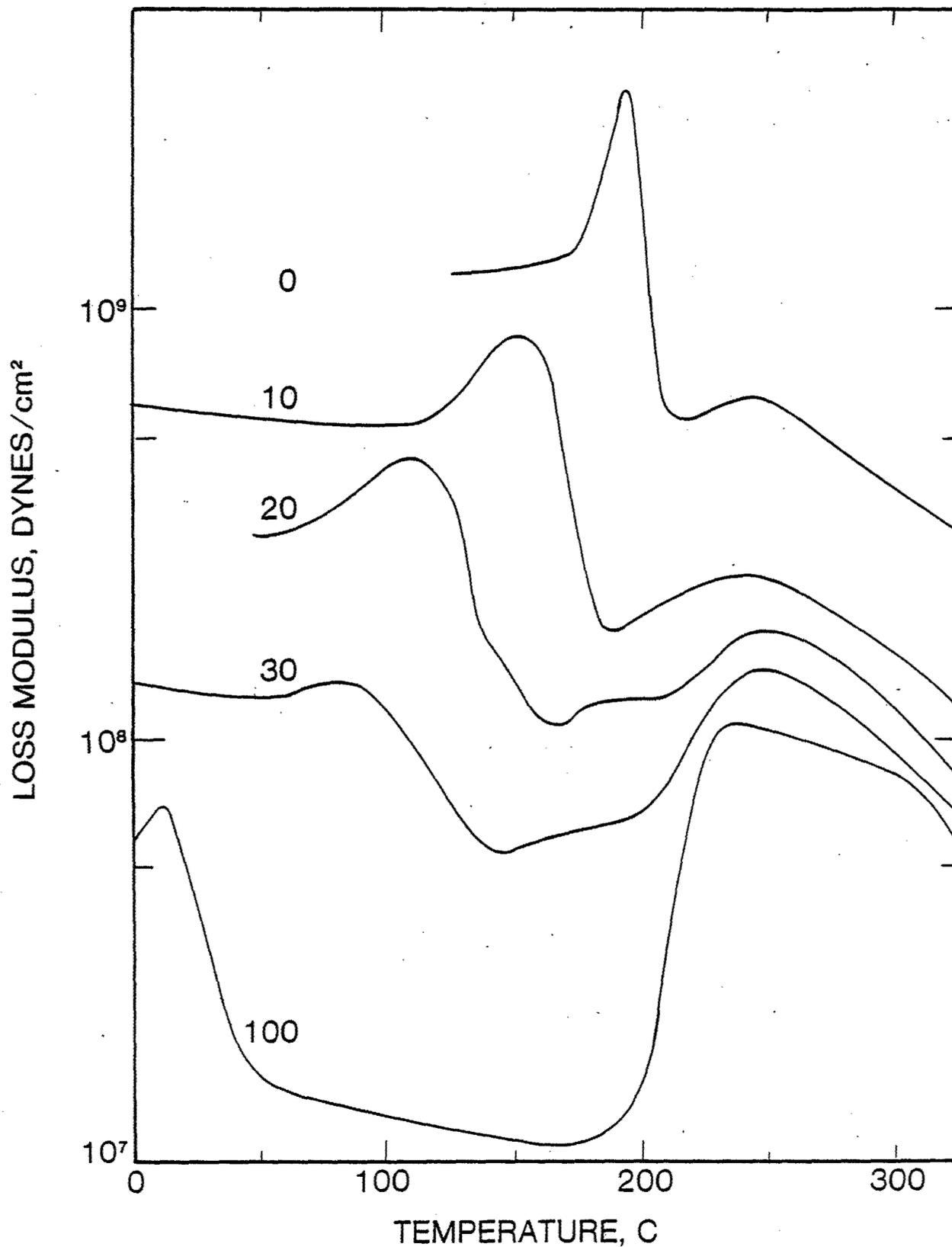


Figure 6.. TICA Loss Modulus Spectra for PSF, PSF/10-ATB, PSF/20-ATB, PSF/30-ATB, and ATB at 10 rad/sec. Curves have been arbitrarily shifted along the ordinate. Numbers represent wt % ATB. All spectra obtained during initial heating to 350°C.

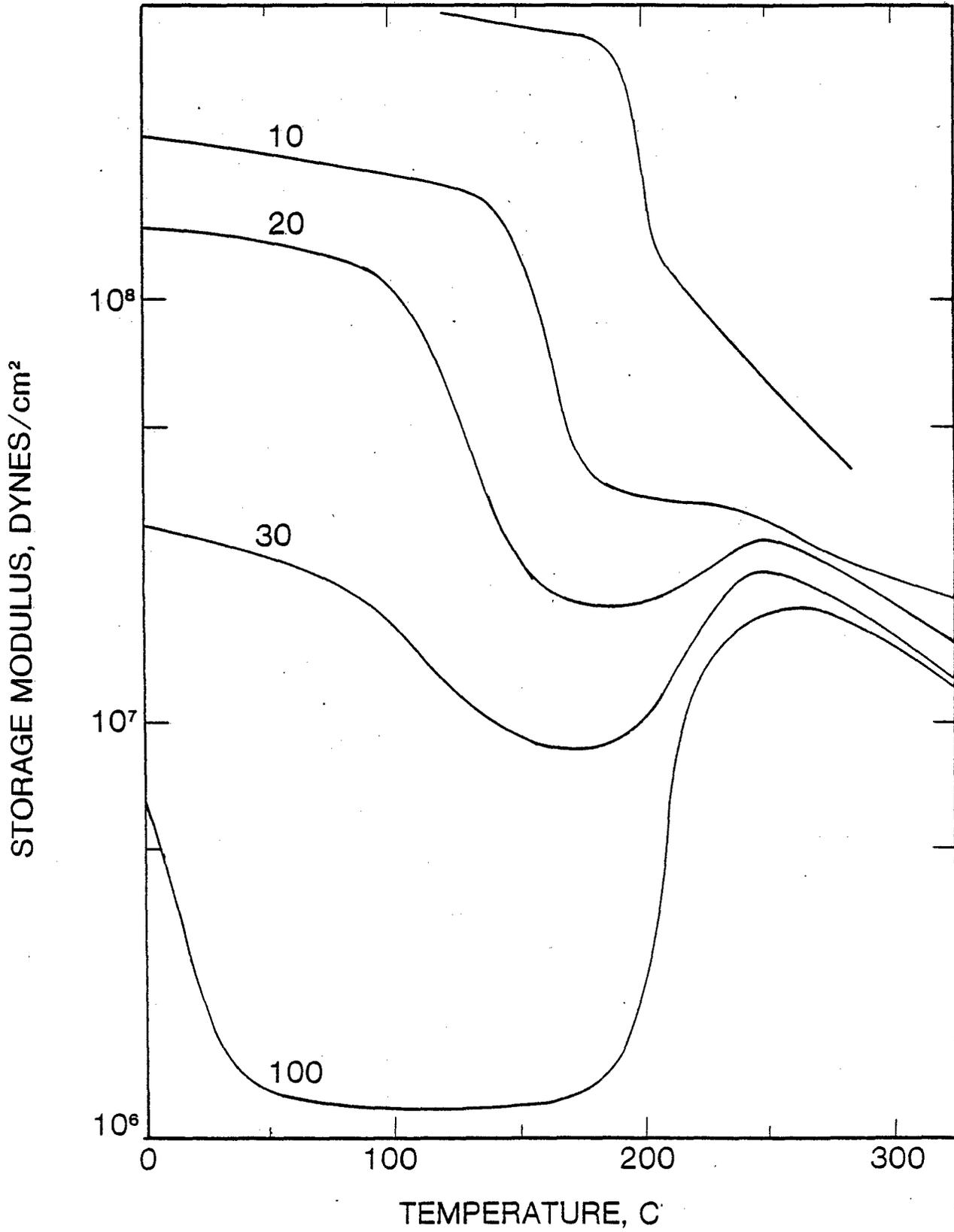


Figure 7. Storage Modulus Spectra for TICA Samples Identified in Figure 6.

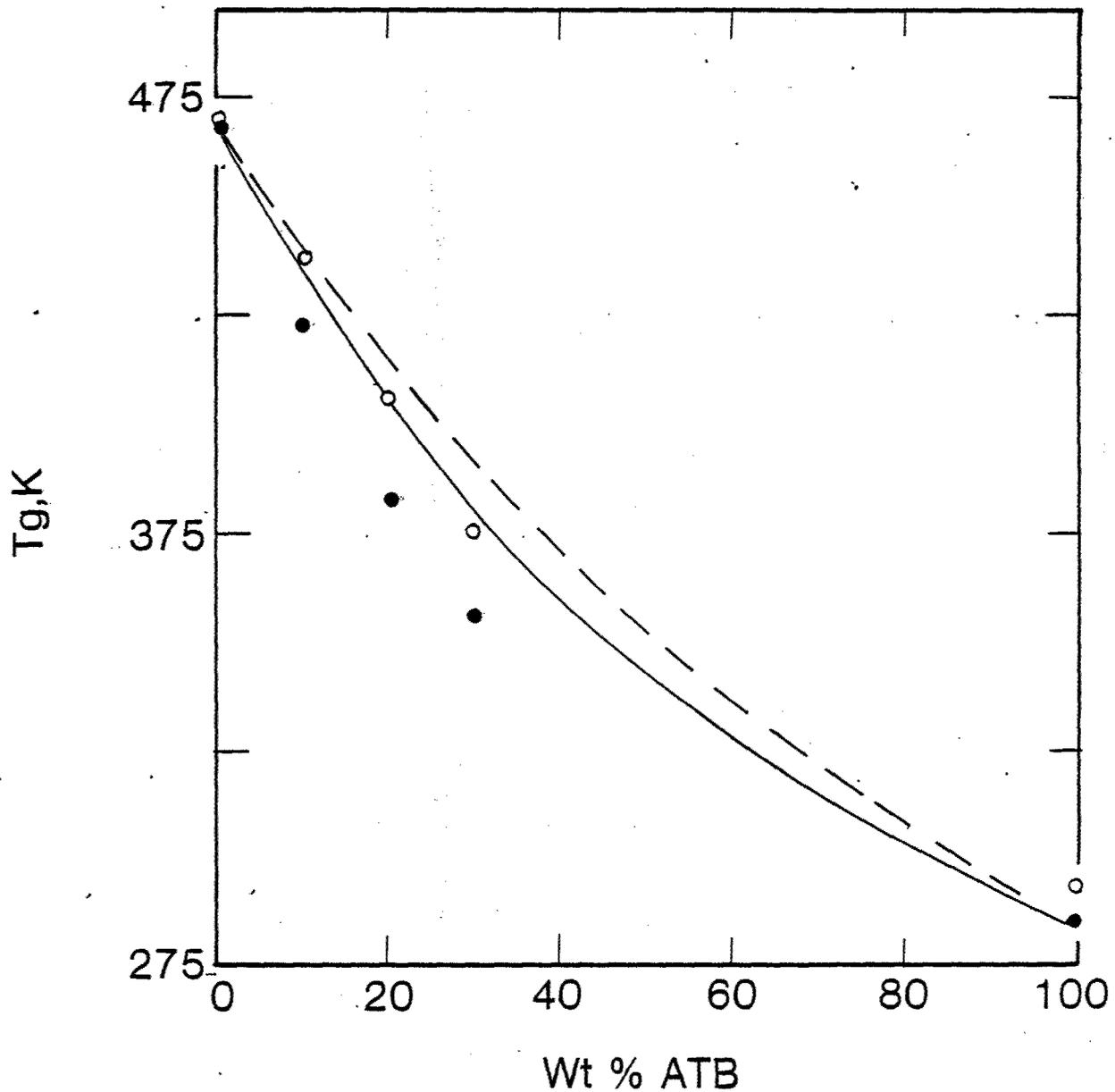


Figure 8. Plot of TICA Determined T_g of Uncured PSF/ATB Blends. ●, Temperature at Maximum of Loss Peak; ○, Temperature at Maximum of Tan δ Peak. (---) Fox Equation (Equation 1); (—) Fried Equation (Equation 2). Curves are drawn from T_g values calculated by use of loss maximum determined T_g 's of the unblended components.

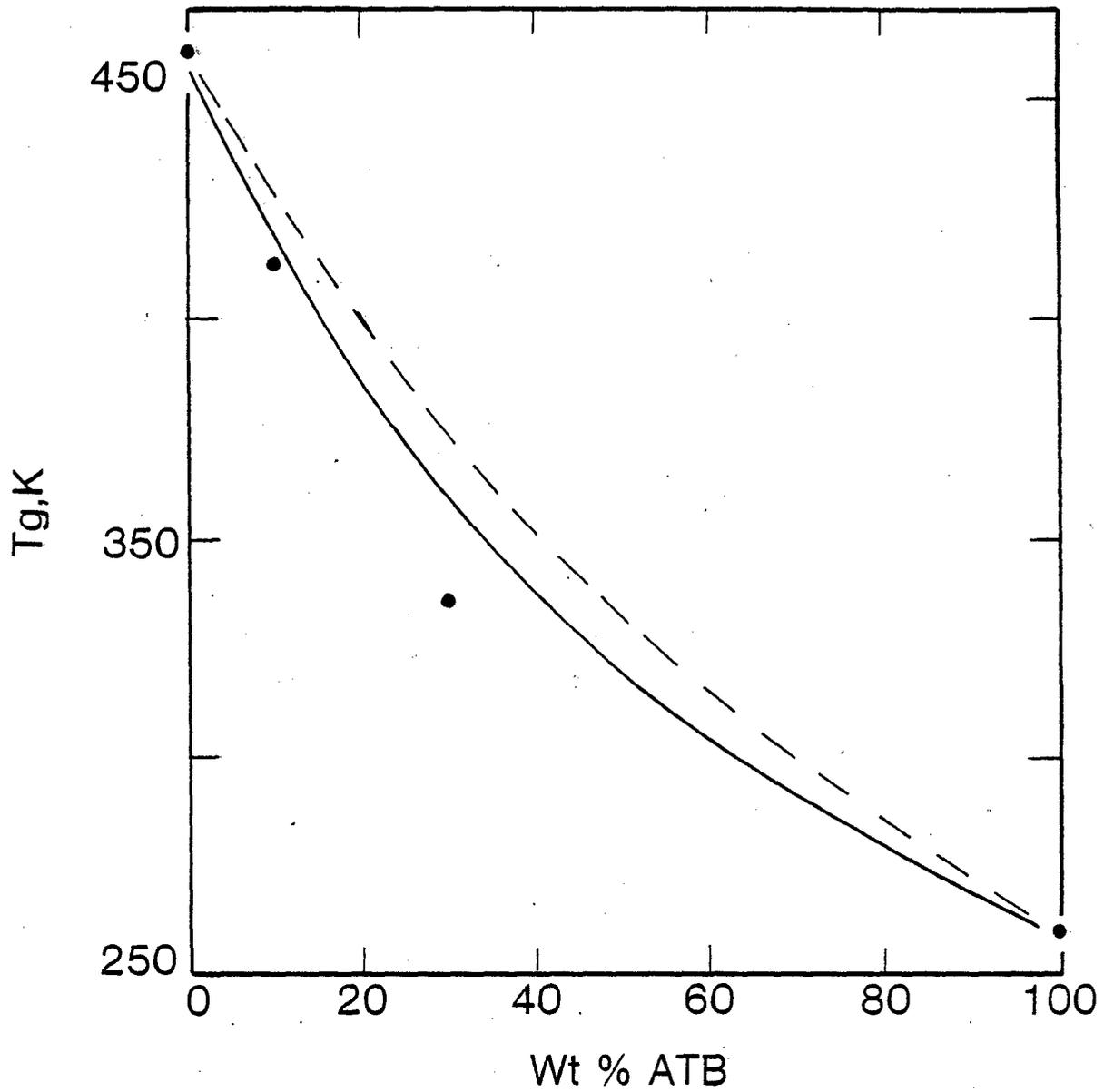


Figure 9. Plot of DSC T_g of Uncured PSF/ATB Blends as a Function of wt % ATB. Solid curve, Fried Equation (Equation 2); Broken Curve, Fox Equation (Equation 1).

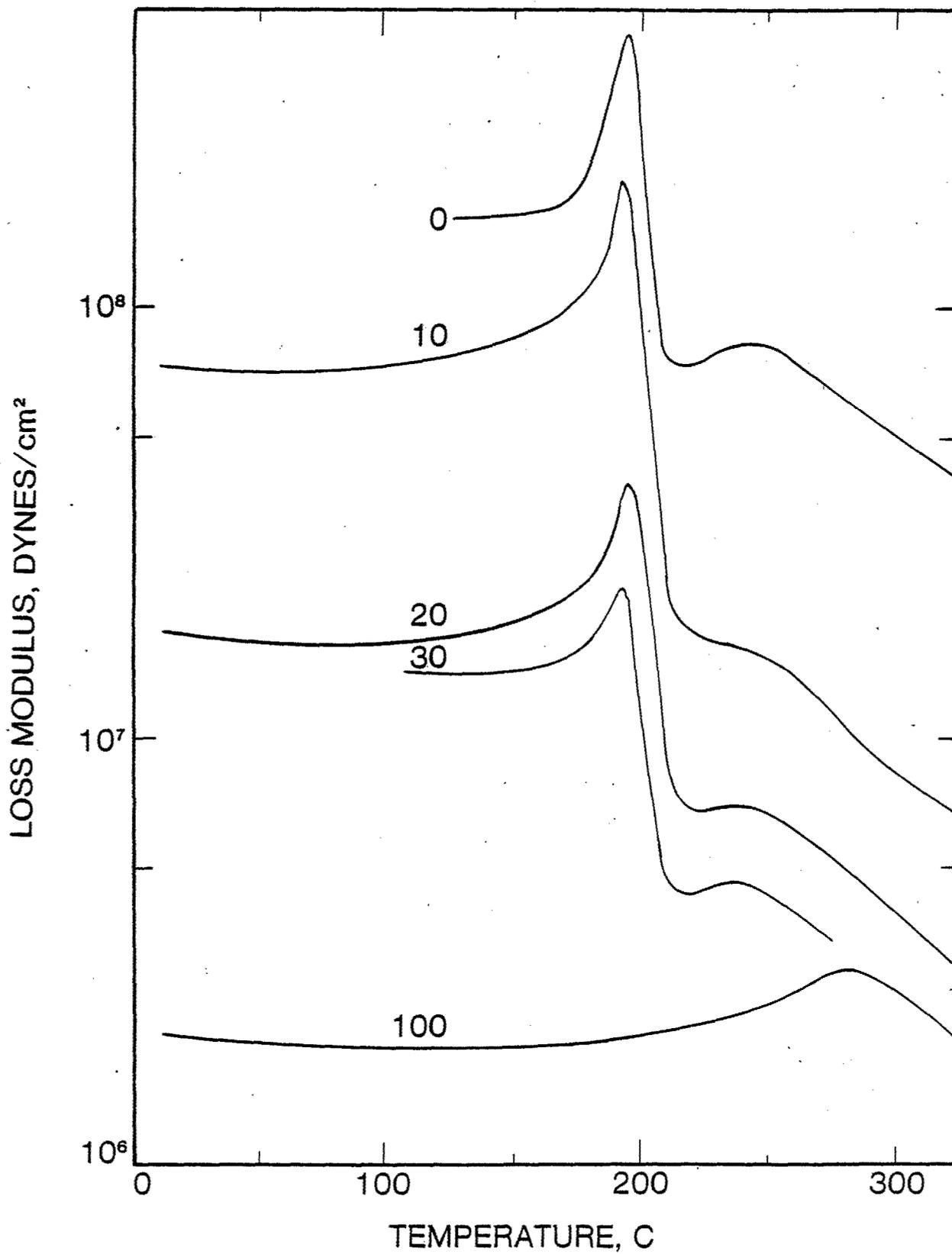


Figure 10. TICA Loss Modulus for PSF, PSF/10-ATB, PSF/20-ATB, PSF/30-ATB, and ATB at 10 rad/sec after Initial Heating to 350°C. Curves have been arbitrarily shifted along the ordinate.

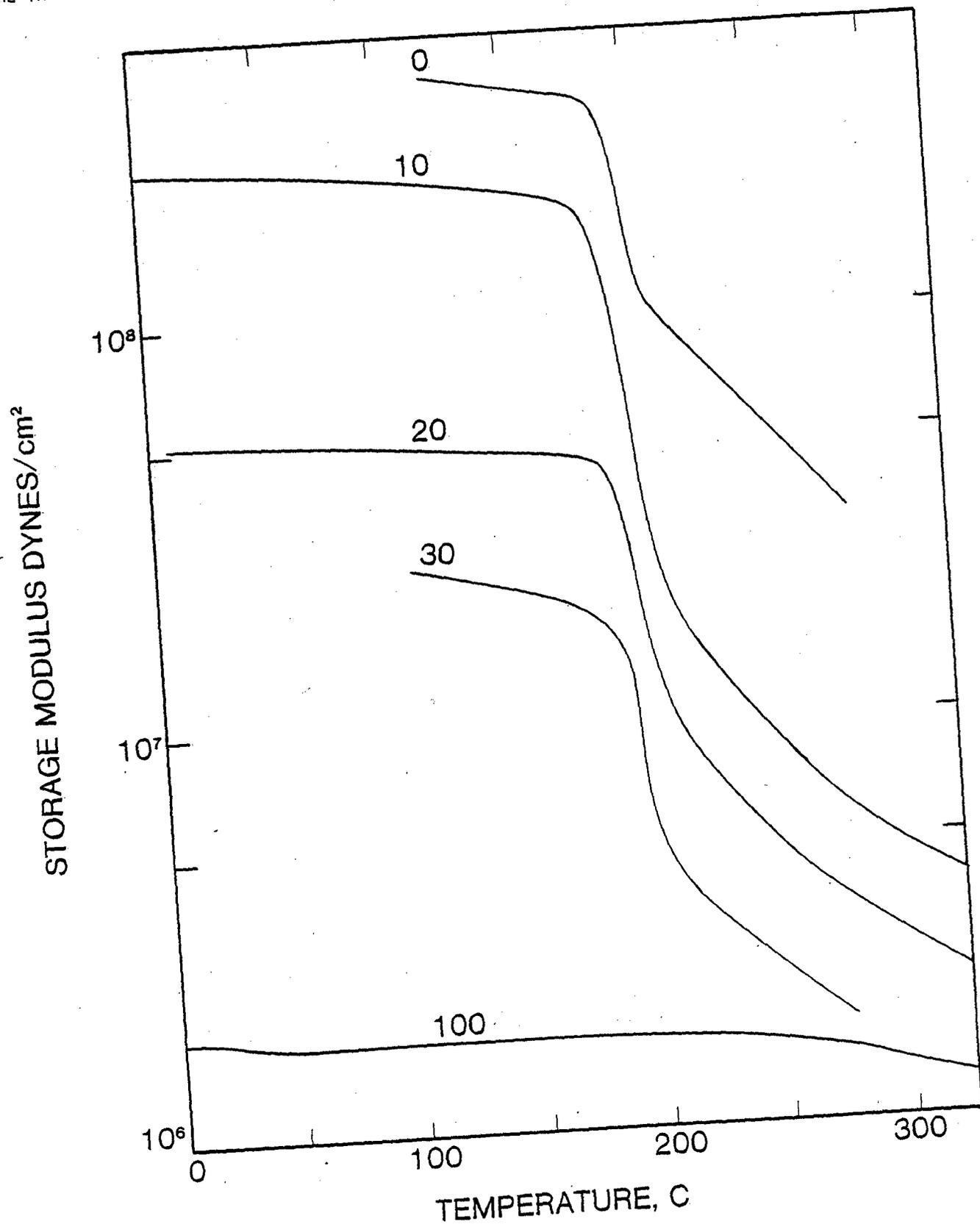


Figure 11. Storage Modulus of TICA Samples Identified in Figure 10.

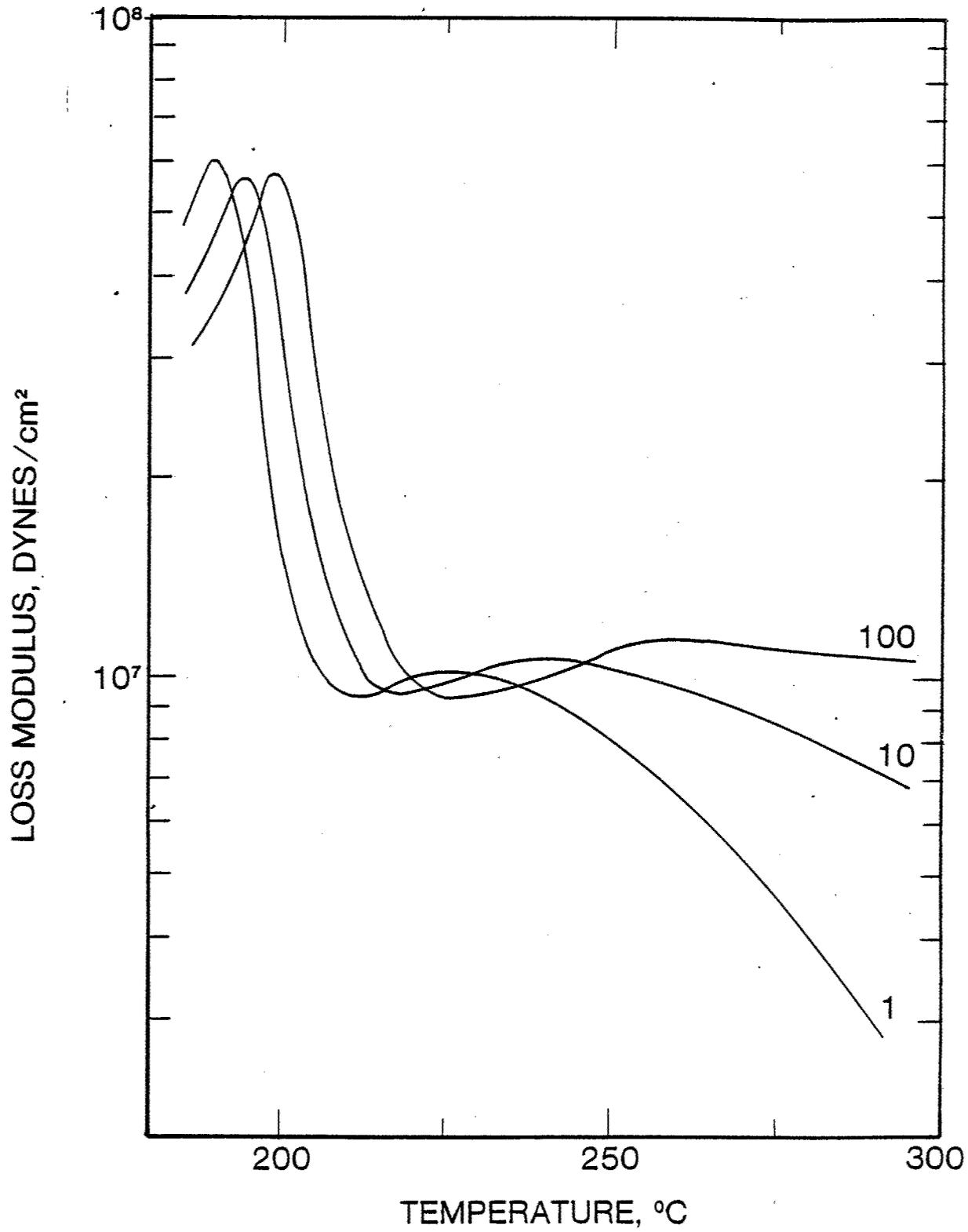


Figure 12. TICA Loss Modulus of PSF as a Function of Temperature and Frequency. Numerals represent frequency in rad/sec.

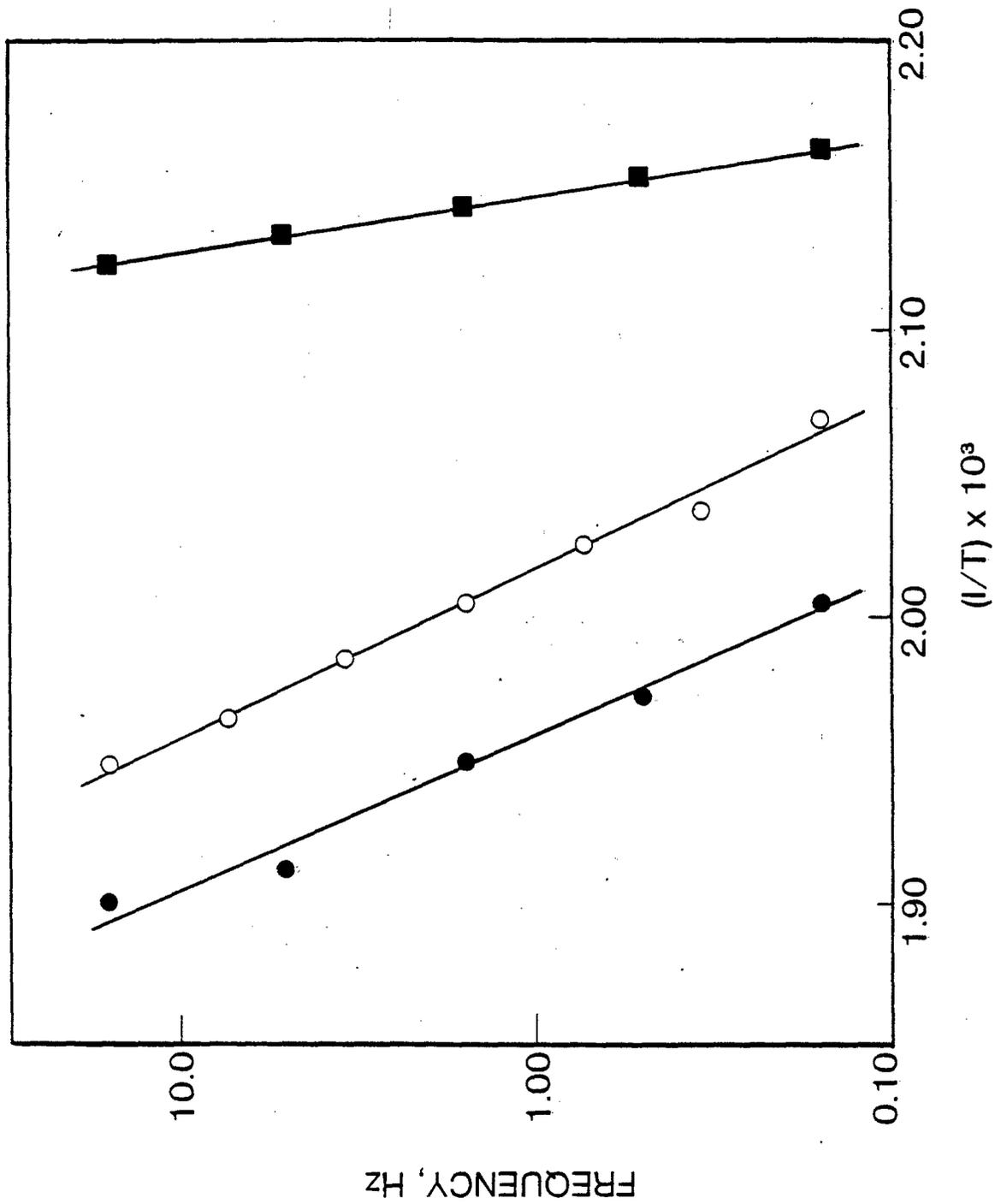


Figure 13. Arrhenius Plots of T_g Relaxation from Loss Modulus Data for TICA (●) and Parallel Plate Melt Rheology (○); TICA Glass Transition data (■). All for unblended PSF.

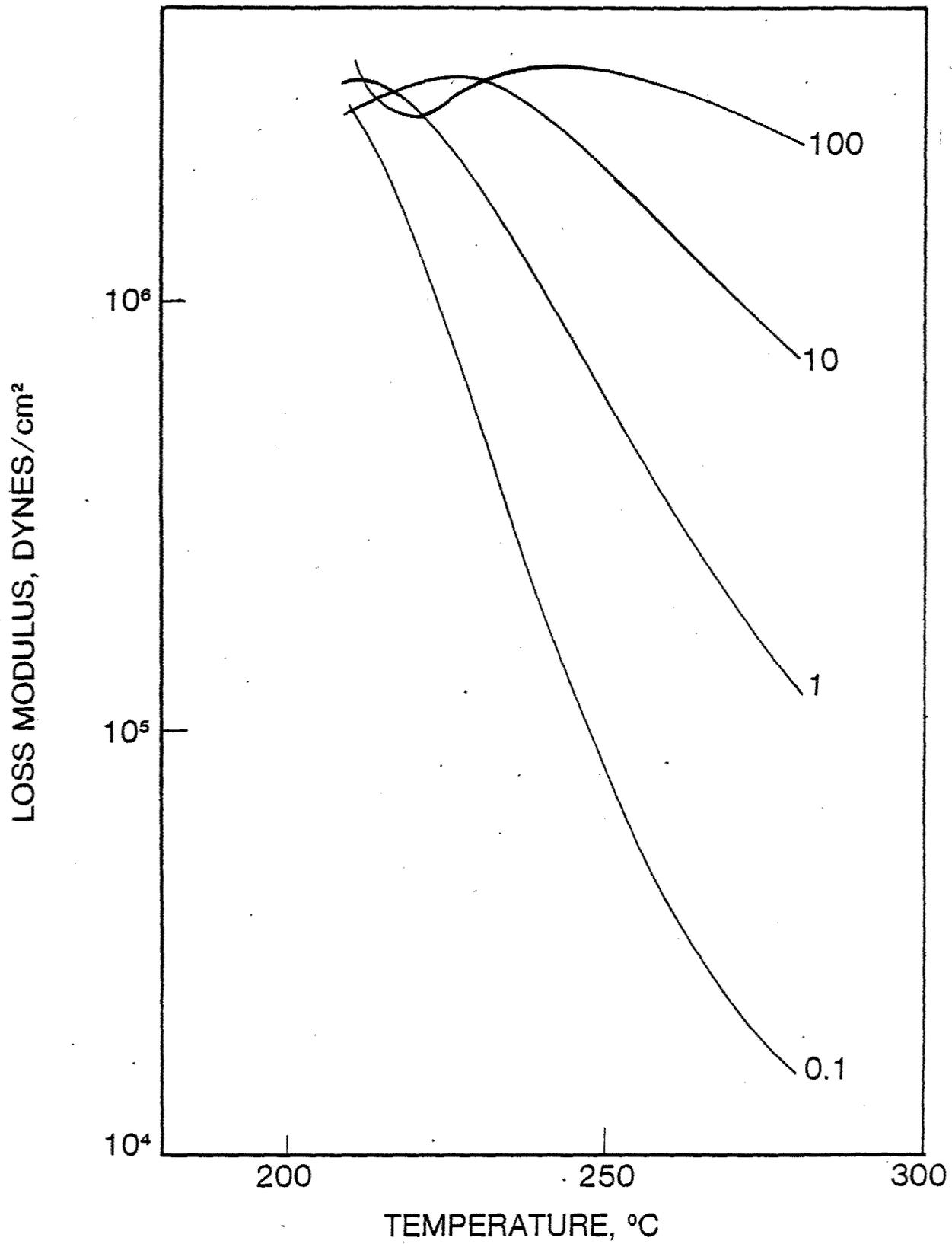


Figure 14. High Temperature Loss Modulus of PSF Melt at Different Frequencies (rad/sec).

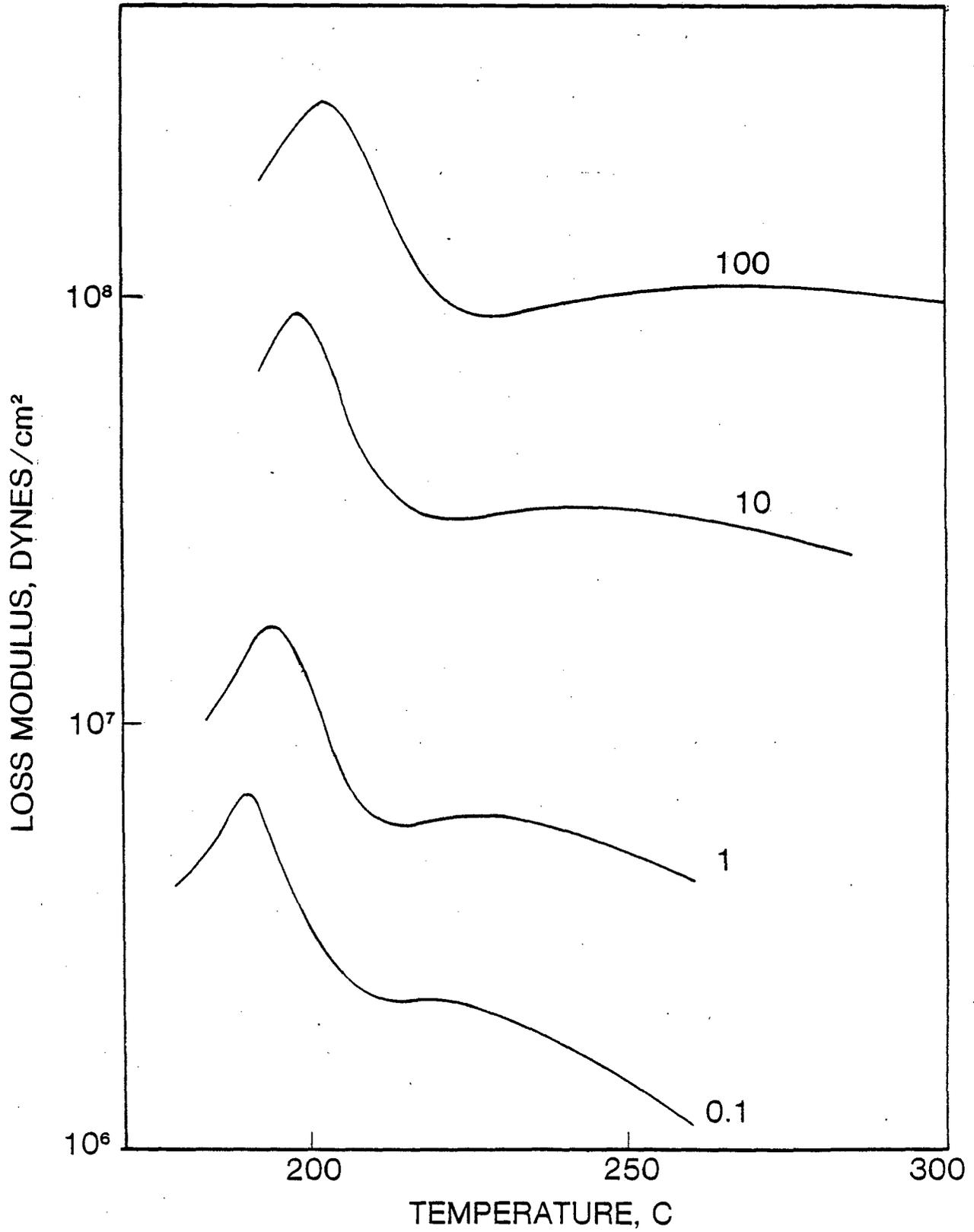


Figure 15. TICA Loss Modulus of Cured PSF/30-ATB as a Function of Temperature and Frequency (rad/sec).

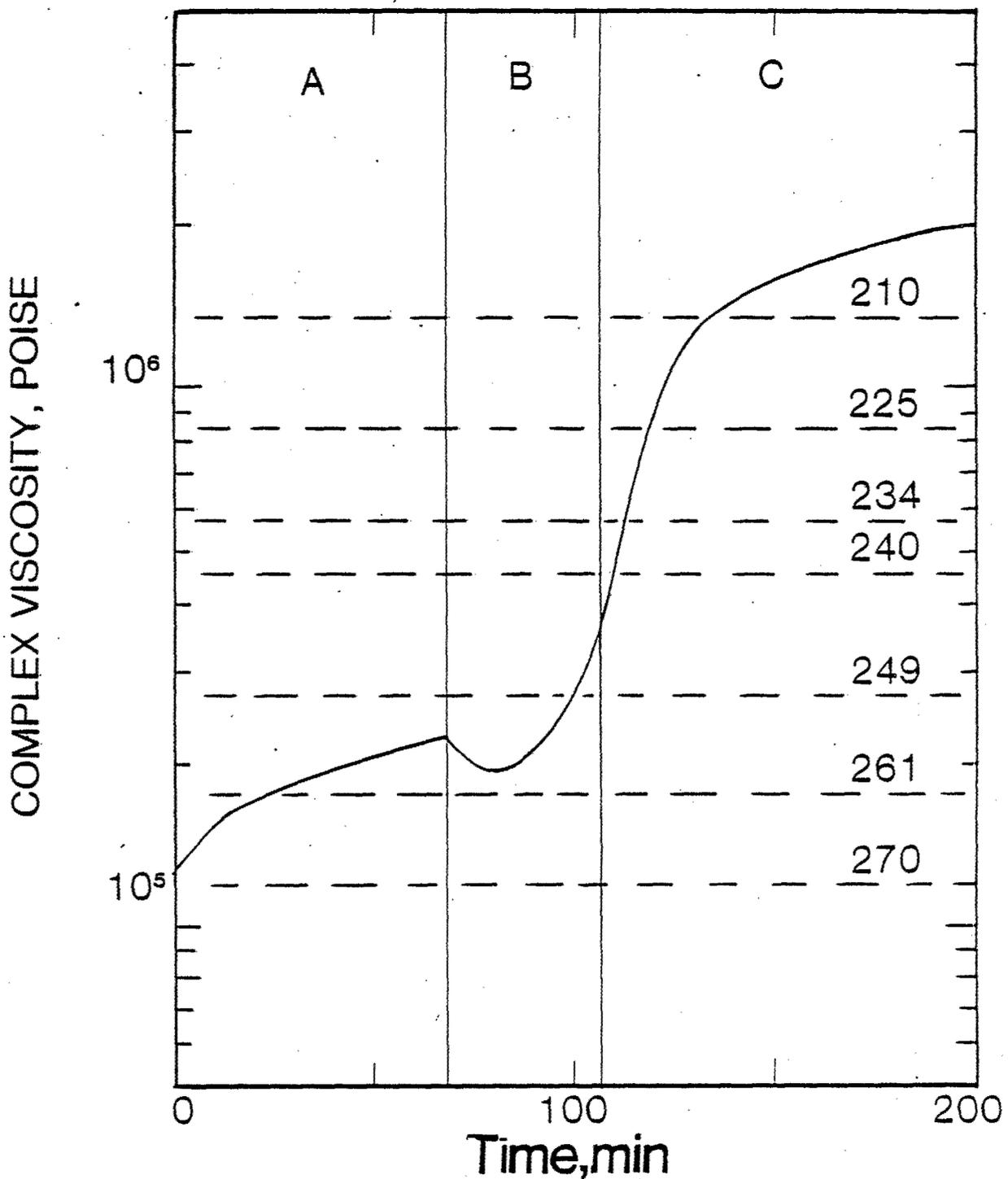


Figure 16. Plot of Complex Viscosity (η^*) of PSF/30-ATB Melt as a Function of Time at 10 rad/sec During Air Cure. A, Isothermal at 150°C; B, Temperature Raised from 150°C to 223°C at 2°/min; C, Isothermal at 223°C. Horizontal broken lines represent complex viscosity of unblended PSF at temperatures (°C) indicated at the right.