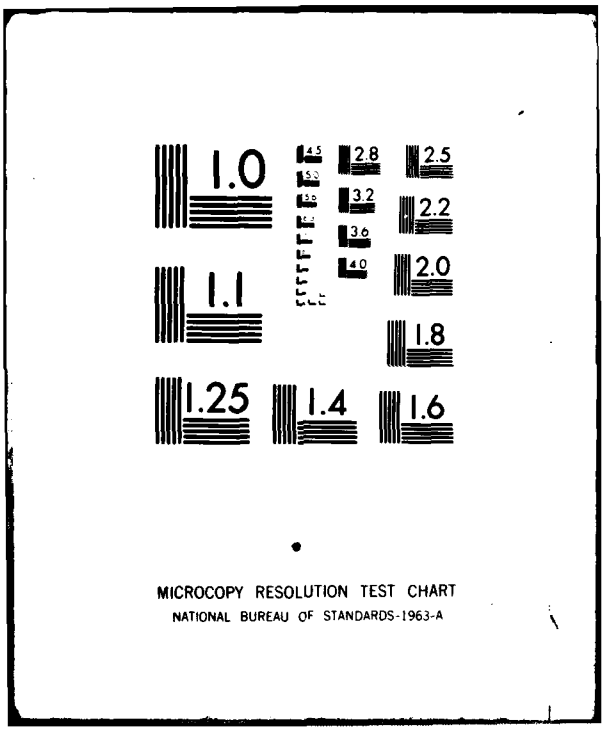
AD-A083 681 RUTGERS - THE STATE UNIV PISCATAWAY NJ HIGH PRESSURE--ETC F/G 11/9
PRESSURE DEPENDENCE OF THE GLASS TRANSITION AND RELATED PROPERT--ETC(U)
APR 80 D L GUESTAD, K D PAE, B A NEWMAN N00014-75-C-0840
UNCLASSIFIED TR-17 NL

END
DATE FILMED
6-80
DTIC


1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0
4.5
5.0
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

ADA083681

(24) LEVEL II

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: Technical Report # 17
2. GOVT ACCESSION NO.: AD-A083 681
3. RECIPIENT'S CATALOG NUMBER:
4. TITLE (and Subtitle): PRESSURE DEPENDENCE OF THE GLASS TRANSITION AND RELATED PROPERTIES OF AN ELASTOMER-SOLITHANE 113
5. TYPE OF REPORT & PERIOD COVERED: Technical Report Interim
6. PERFORMING ORG. REPORT NUMBER: (14) TR-17
7. AUTHOR(s): (10) D. L. Questad, K. D. Pae, B. A. Newman, and J. I. Scheinbeim
8. CONTRACT OR GRANT NUMBER(s): (15) N00014-75-C-0540
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Rutgers University, High Pressure Research Lab. & Dept. of Mechanics & Materials Science, College of Engineering, Piscataway, NJ 08854
10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: NR 356-564
11. CONTROLLING OFFICE NAME AND ADDRESS: Office of Naval Research (Code 472) Arlington, Virginia 22217
12. REPORT DATE: April 1, 1980
13. NUMBER OF PAGES:
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office):
15. SECURITY CLASS. (of this report):
15a. DECLASSIFICATION/DOWNGRADING SCHEDULE:
16. DISTRIBUTION STATEMENT (of this Report): Approved for public release; distribution unlimited. Reproduction in whole or in part is permitted for any purpose of the United States Government.
(9) Interim technical rpt.
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report): (11) 1 Apr 80
18. SUPPLEMENTARY NOTES: (12) 27

DTIC ELECTE S MAY 1 1980 D A

19. KEY WORDS (Continue on reverse side if necessary and identify by block number): High pressure, glass transition, dielectric measurements, pressure-volume-temperature measurements, Young's Modulus.

20. ABSTRACT (Continue on reverse side if necessary and identify by block number): Several studies have been carried out on a polyurethane elastomer (Solithane 113). At atmospheric pressure dielectric methods were used to study ε" over the temperature range from -180°C to +20°C. Three peaks in ε" were observed: an α peak associated with the glass transition and two low temperature peaks (β and γ). Mechanical tests were performed to observe the effect of the pressure induced glass transition, P_g, on the Young's Modulus. The glass transition was studied as a function of pressure from 1 bar to 6.5 kilobar

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE S/N 0102-014-6601

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

411647

DTIC COPY

by observing the shift of the dielectric α-peak with pressure and also by volumetric methods. A densification of the glass and a resultant shift in the glass transition temperature could be achieved by forming the glass at high pressures (T_g) as opposed to pressuring the glassed formed at 1 bar (T_g^*). It was found that $\Delta\beta/\Delta\alpha > dT_g/dP$, but $\Delta\beta/\Delta\alpha \approx dT_g^*/dP$. At high pressures dT_g/dP reached a limiting value of 10.4°C/kb.

OFFICE OF NAVAL RESEARCH

Contract N00014-75-C-0540

Task No. NR 356-564

TECHNICAL REPORT NO. 17

PRESSURE DEPENDENCE OF THE GLASS TRANSITION AND RELATED PROPERTIES OF AN ELASTOMER-SOLITHANE 113

by

D. L. Questad, K. D. Pae, B. A. Newman, and J. I. Scheinbeim

Prepared for Publication

in the

Journal of Applied Physics

High Pressure Materials Research Laboratory
Department of Mechanics and Materials Science
College of Engineering
Rutgers University
P. O. Box 909
Piscataway, New Jersey 08854

April 1, 1980

Accession For
NTIS GRA&I
DDC TAB
Unannounced
Justification
By
Distribution/
Availability Codes
Dist | Avail and/or Special
A

Reproduction in whole or in part is permitted for any purpose of the United States Government

This document has been approved for public release and sale; its distribution is unlimited

80 4 24 052

PRESSURE DEPENDENCE OF THE GLASS TRANSITION AND RELATED PROPERTIES OF AN ELASTOMER - SOLITHANE 113

D. L. Questad, K. D. Pae, B. A. Newman and J. I. Scheinbeim

High Pressure Materials Research Laboratory

Department of Mechanics and Materials Science

College of Engineering, Rutgers University

P. O. Box 909, Piscataway, New Jersey 08854

Abstract

Several studies have been carried out on a polyurethane elastomer (Solithane 113). At atmospheric pressure dielectric methods were used to study ε'' over the temperature range from -180°C to +20°C. Three peaks in ε'' were observed: an α peak associated with the glass transition and two low temperature peaks (β and γ). Mechanical tests were performed to observe the effect of the pressure induced glass transition, P_g, on the Young's modulus. The glass transition was studied as a function of pressure from 1 bar to 6.5 kilobar by observing the shift of the dielectric α-peak with pressure and also by volumetric methods. A densification of the glass and a resultant shift in the glass transition temperature could be achieved by forming the glass at high pressures (T_g) as opposed to pressuring the glass formed at 1 bar (T_g^*). It was found that $\Delta\beta/\Delta\alpha > dT_g/dP$, but $\Delta\beta/\Delta\alpha \simeq dT_g^*/dP$. At high pressures dT_g/dP reached a limiting value of 10.4°C/kb.

I. INTRODUCTION

The research studies investigated the glass transition behavior of a polyurethane elastomer (Solithane 113) and other related properties as a function of pressure and temperature. Two methods have been employed in determining the glass transition temperature: dielectric and volumetric measurements. In addition, characterization of two low temperature transitions in the elastomer has also been made.

The study of the glass transition as a function of pressure has had its share of controversy and confusion, and often theoretical predictions have gotten ahead of clear experimental results. The small amount of experimental data has been unable to establish any one of several theories as clearly superior to the others.[1,2,3] Conclusions based on experimental results have been varied owing to the variety of ways in which the glass can be formed in pressure-volume-temperature space and also due to the difficulty of measuring certain quantities accurately, especially the change in compressibility across the transition. The kinetic nature of the observed glass transition also complicates any thermodynamic understanding of the phenomenon.

Free volume theories are based on the assumption that the fractional free volume, $f = f_g + \alpha_f(T-Tg_o) - \beta_f(P)$ is constant at the glass transition. Here f_g is the fractional free volume at Tg_o, the glass transition temperature at atmospheric pressure, α_f and β_f are the thermal expansivity and compressibility of the free volume, respectively. Since the assumption is made that $f = f_g$ at the glass transition, then the free volume theory predicts a linear increase in

Tg with pressure according to $dTg/dP = \beta_f/\alpha_f$.[1] The free volume thermal expansivity, α_f, was originally determined by fitting data from relaxation studies to the WLF equation. This leads to a "universal" value of α_f which was generally about equal to $\Delta\alpha = \alpha_\ell - \alpha_g$ obtained from volumetric studies. The association of β_f with $\Delta\beta$, however, has not been as successful.[4] The discrepencies between measured values of $\Delta\beta/\Delta\alpha$ and dTg/dP imply that free volume is not the single controlling parameter of the glass transition.[3,5] Some have allowed for changes in free volume at the glass transition, but this is equivalent to saying that free volume is not the single ordering parameter governing the glass transition.[6]

Thermodynamic theories are based on the assumption that the glass transition is or reflects a true second order thermodynamic transition. The theory of DiMarzio, et al. obtains the transition temperature, T_2, as a function of pressure where T_2 is the temperature at which the configurational entropy becomes zero. This theory is based on equilibrium thermodynamics; therefore it predicts the Ehrenfest relations to hold (i.e. $dTg/dP = \Delta\beta/\Delta\alpha = TV\,\Delta\alpha/\Delta Cp$). The two parameters controlling the glass transition in this model are n_0, the number of holes in the lattice approximation of the polymer, and f, the number of bonds in the flexed or high energy conformation. The pressure is assumed not to affect f, and therefore as the pressure increases n_0 decreases causing dTg/dP to decrease. At high enough pressures n_0 becomes zero, and further increases in pressure will not increase Tg. So the theory predicts a levelling off of the Tg versus pressure curve as pressure is increased, and the authors claim this

effect will occur in the 10 Kb range.[2]

Experimental observations that $\Delta\beta/\Delta\alpha$ is usually greater than dTg/dP whereas $TV\Delta\alpha/\Delta Cp$ is usually about equal to dTg/dP has lead to the view that the glass transition is controlled by the excess entropy as opposed to the free volume.[7] The value of dTg/dP, however, can be made to equal $\Delta\beta/\Delta\alpha$ by measuring dTg/dP for glasses which are formed at only one pressure.[3,8] This has been associated with the freezing in of an ordering parameter at one glass formation pressure which would vary if the glass was formed at different pressures.[3]

Dielectric measurements have been used to determine dTg/dP for a number of polymers.[12] Values of dTg/dP measured by dielectric and volumetric methods have generally compared well.[9,10]

In this investigation we studied the glass transition as a function of pressure by dielectric and volumetric means. The results are compared with certain thermodynamic quantities for glasses formed by different methods. We also measured the Young's modulus below and above the glass transition pressure up to 7 Kb, and the specific volume as a function of pressure from which the bulk modulus can be calculated.

II. MATERIAL

The material used is Solithane 113 which is a polyurethane based elastomer produced by the Thiokol Chemical Corporation. It is formed by the reaction of a resin and catalyst. The resin is a trifunctional isocyanate formed from castor oil and tolylene diisocyanate. The catalyst consists essentially of castor oil. By combining different ratios of resin and catalyst the degree of cross-linking

can be changed. In this study we deal with the equivoluminal composition or a 50/50 volume ratio of resin and catalyst. For this composition the molecular weight between crosslinks is approximately 2000 g/mole.[9] The specific gravity is determined to be about 0.97 cm^3/gr.

III. EXPERIMENTAL

A. Dielectric Measurements

A thin sample (~.05 cm thick), electroded on both sides, was used for the dielectric measurements. This sample was placed between two brass electrodes which in turn were placed between ceramic heating plates in order to assure uniformity of temperature. The apparatus is shown schematically in Fig. 1. Temperature was measured by a chromel-alumel thermocouple embedded in the brass electrode and the temperature of the test was varied at a rate of 1^{o} C/min. A Piezotron-U (Toyo Seiki, Tokyo, Japan) was used to measure the dielectric constants the values of which were recorded continuously on a chart recorder. All measurements were made at a test frequency of 3 Hz.

B. PVT Measurement

A schematic drawing of the PVT apparatus is shown in Fig. 2. It consists essentially of a sample which is fixed on one end and free floating on the other end. The free end is connected to a small steel rod which rides in a linear variable differential transformer (LVDT) enabling changes in length to be measured electrically. The sample is surrounded by a cylindrical heater, and temperature is measured by a chromel-alumel thermocouple placed at the sample sur-

face. The entire apparatus was placed inside a pressure vessel filled with silicon oil as the pressure medium.

C. Method of Glass Formation and Tg Measurement

In order to study how glass formation pressure affects glass transition behavior, the tests were run in two different manners. First, the sample was cooled at atmospheric pressure below the Tg, forming the normal glass (NG). The pressure was then applied to the normal glass to the desired intensity of pressure after which the temperature was raised isobarically to record specific volume versus temperature through the glass transition region. In the second experiment, the sample was cooled from above the Tg which was achieved in the first experiment holding pressure constant, again recording specific volume versus temperature. This second method formed the equilibrium or pseudo equilibrium glass. The glass formed by lowering temperature at elevated pressures as in the second experiment is designated Tp-glass. Normal glass (NG) is a Tp-glass in which P is atmospheric pressure. Both glasses were studied in the dielectric tests.

D. Measurement of Young's Modulus

The apparatus for performing mechanical tests under high pressure is shown schematically in Fig. 3. The sample is mounted in a pressure vessel and is loaded by a hydraulically driven piston. The loading piston is mounted with strain gages to measure the applied load, while sample deformation is measured by a linear variable differential transformer (LVDT). The electrical signals from the strain gages and the LVDT are amplied and displayed on an X-Y recorder. By

use of special sample yokes both tension and compression tests can be performed, however in this study only compressive measurements were made. The Young's modulus has been identified with the initial slope of the compressive stress-strain curve.

IV. RESULTS

A. Dielectric Measurements

Fig. 4 shows the dielectric loss constant, ε'', versus temperature at atmospheric pressure over the temperature range from -180°C to +20°C. Three peaks in ε'' are observed. At 3 Hz the α-peak occurs at +2°C. This peak which is by far the most prominent of the three peaks is associated with the glass transition. At about -80°C, a small β-peak occurs, which has been related to the motion of small molecules, especially water, contained in the elastomer network,[10] and at about -160°C a very broad γ-peak occurs which may be associated with a local mode transition occurring in the glassy state.[10]

The application of hydrostatic pressure causes the α-peak to be shifted to higher temperatures. By choosing the maximum point in the α-peak to locate the transition, a measure of the pressure dependence of the glass transition temperature can be obtained. This behavior is shown in the upper solid curve in Fig. 5. The maximum in the α-peak shifts about 80°C with the application of a pressure of 6.5 kilobar. The rate of increase is constantly decreasing up to a pressure of about 2.5 kilobar after which the transition temperature increases at a constant rate of 10.4°C/kb. The α-peak decreases in height and broadens slightly due to the application of pressure, but this effect is small, so the error with which the maximum point can be determined is essentially unaffected by pressure over the entire pressure range.

The pressure dependence of the α-peak is not affected by how the glass was formed. In other words, the same location in temperature of the α-peak at a given pressure, is obtained when either the normal glass (NG) or Tp-glass is used for the test.

B. Volumetric Measurements

Specific volume of the normal glass (NG) and the Tp-glass was measured as described in the Experimental section with increasing temperature at various pressures up to 6 Kbar as shown in Fig. 6. The glass transition temperature was then located by the intersection of straight lines drawn through the points below and above the Tg. The upper curves labelled NG in the glassy region for tests conducted at 1 and 2 Kilobar correspond to heating the normal glass (NG). All other curves corrrspond to cooling from the liquid state through Tg at various pressures. The glass transition temperature determined for the NG glass will be referred to as Tg^* while the glass transition for Tp-glass will be called Tg.

The normal glass has a structure which is found to be less dense at subsequently applied higher pressures than that of Tp-glass at the same pressure. Consequently its specific volume is higher as shown at 1 and 2 Kilobars in Fig. 6 The intersection of these glass curves with the liquid curve will therefore be located at higher temperatures than the intersection of the glass curve for Tp-glass. This produces a Tg^* which is greater than Tg at all pressures except atmospheric pressure where the glasses were formed identically. The slight flattening of the normal glass curves near the transition indicates volumetric relaxation as Tg^* is approached from below. This

is an indication of the kinetic nature of the observed glass transition.

The thermal expansivities of the glass and the liquid, α_g and α_ℓ, respectively, measured from the slopes of the curves in Fig. 6 both decrease with increasing pressure. The value of α_ℓ, however, decreases much more rapidly than α_g making determination of the glass transition more difficult as the pressure is raised.

Plots of Tg and Tg^* versus pressure are shown in Fig. 5. The lower solid curve is Tg (for Tp-glass), and the dashed curve is Tg^* (for NG). As noted earlier Tg^* has a higher value and increases more rapidly than Tg. The curve for Tg versus pressure is observed to parallel the curve obtained from dielectric measurements quite well. The volumetric glass transition being at all pressures about 20°C below the dielectric curve obtained with the frequency 3 Hz.

The slopes of the curves in Fig. 5 give values of dTg/dP (or dTg^*/dP) versus pressure. The slope of the dielectric and Tg curves are essentially the same. The values of dTg/dP obtained in this manner can be compared with $\Delta\beta/\Delta\alpha$. Values of $\Delta\alpha$ are obtained by taking the difference in thermal expansivities above and below the glass transition: $\Delta\alpha = \alpha_\ell - \alpha_g$. To obtain $\Delta\beta$, the change in compressibility across the transition, the following method was used.[11] At a certain test pressure, as the temperature is lowered through the Tg, the pressure is perturbed slightly to measure $\beta = 1/V(\partial V/\partial P)_T \approx (\Delta V/V_0)/\Delta P$. This value of β goes through a step decrease as the temperature is lowered through the transition region. From this step $\Delta\beta$ is obtained.

A plot of dTg/dP, dTg^*/dP and $\Delta\beta/\Delta\alpha$ are shown in Fig. 7. The values of dTg^*/dP are seen to be nearly twice the values of dTg/dP, and the $\Delta\beta/\Delta\alpha$'s are approximately equal to dTg^*/dP within experimental errors. Similar results have been observed by other authors on uncrosslinked systems.[3,8]

C. Comparison of Young's Modulus and Bulk Modulus

A pressure induced glass transition (or the glass transition pressure, Pg) occurs at room temperature within the seven Kilobar range as shown in Fig. 8. In this figure the Young's Modulus is seen to undergo an increase of more than two orders of magnitude in the 2 - 3 Kilobar range. Prior to the transition the modulus is typical of a rubber: $\sim 2 \times 10^7$ dyne/cm^2, and after the transition it is typical of a glass: $\sim 10^{10}$ dyne/cm^2. From a plot of specific volume versus pressure as shown in Fig. 9 the bulk modulus, B, can also be obtained. The bulk modulus is taken as the inverse of the slope of the curve in Fig. (9). B increases from $\sim 2 \times 10^{10}$ dynes/cm^2 to $\sim 8 \times 10^{10}$ dynes/cm^2 with the application of seven kilobars of pressure, but no abrupt change occurs at the glass transition as was the case for the Young's Modulus.

V. DISCUSSION

When a polymer is cooled through its glass transition, relaxation times of segments in the polymer go from very short to very long. There is actually a very broad distribution of relaxation times which is reflected in the breadth of the α-peak in Fig. 4. Although the peak maximum occurs at $+2^oC$, there are relaxations still occurring

well below -20°C. The maximum point in the α-peak corresponds to the condition where the average relaxation time equals the inverse of the test frequency, in this case 3 Hz. For this reason the maximum point in the α-peak must be in the liquid state (i.e. where large scale motions of the chain are possible). The fact that the maximum in the α-peak does not depend upon glass-formation conditions (see III-C) may be attributed to the fact that, although different glassy states might have been frozen in below Tg, by the time the temperature of the maximum in the α-peak is reached, the different initial structures will have relaxed to the same structure. Thus the frequency of test (3 Hz) becomes the overriding factor in determining the location of the α-peak.

The observation that dTg/dP is the same for the dielectric test and for the volumetric test cooled from above Tg may be understood as follows. On cooling the sample from above Tg at a given pressure a pseudo equilibrium structure is frozen in. The glass has assumed its pseudo equilibrium structure for the particular pressure and rate of test, just as it will assume its pseudo equilibrium structure in the dielectric test causing the α-peak to depend on frequency. The normal glass is by no means an equilibrium structure, so the dielectric test and the volumetric test cooled through Tg reflect the same pseudo equilibrium transition.

Although no published data has actually shown a levelling off of the Tg versus pressure curve (i.e. $dTg/dP = 0$) at high pressures, the curvature in some published Tg versus P data has made it appear that eventually it might level off. As mentioned earlier, this level-

ling off would be in agreement with certain thermodynamic theories of the glass transition. The data in Fig. 5 and Fig. 7, however, show dTg/dP reaches a constant non-zero value. Although nothing can be said about the dTg/dP behavior at higher pressures, it does not appear that it will become zero in the 10 Kilibar range as has been predicted.

As mentioned earlier and as shown in Fig. 7, $\Delta\beta/\Delta\alpha$ is approximately equal to dTg^*/dP as has been observed by others as well. $\Delta\beta$ is usually obtained by measuring compressibilities between isobars. It can be seen, however, that the perturbation method of measuring compressibility is equivalent to measuring compressibility between isobars for the normal glass. It is, however, important to point out that $dTg^*/dP > dTg/dP$ which is also implied from the densification shown in Fig. 6. This means that a single glass surface is not sufficient to describe the PVT behavior of the elastomer.

The Young's Modulus has been shown to undergo a large increase as the pressure is raised through the Pg, whereas the bulk modulus shows very little change. This implies that it is the deviatoric component of the stress which reflects the abrupt change at the glass transition and not the hydrostatic component. From elasticity theory $E = 9B/(1 + 3B/G)$, and according to Fig. 8 the abrupt change in E is due only to an abrupt change in the shear modulus, G. The calculated values of G at various pressures are listed in Table 1 along with the values of E and B.

VI. CONCLUSIONS

Study of the glass transition behavior of Solithane 113 by dielectric and volumetric methods has produced several interesting results. The transition temperature by dielectric measurements did not change with glass formation conditions and over the pressure range studied the dTg/dP values for dielectric measurements were the same as those for the volumetric method for the glass cooled from above Tg at various pressures.

The value of dTg/dP at high pressures (up to 6.5 Kilobar) did not approach zero but instead reached a limiting value of 10.4°C/Kb. The fact that $dTg^*/dP > dTg/dP$ indicates that, although there is a single equilibrium liquid surface in PVT space, there are many glass surfaces depending on the method of glass formation.

Acknowledgment

The authors wish to thank the Chemistry Program, Office of Naval Research (Contract No. N00014-75-C-0540 Task No. Ne 356-564) for financial support for this work. We would also like to thank Dr. W. G. Knauss at the California Institute of Technology for supplying the Solithane samples.

REFERENCES

1. D. H. Kaelble, Free Volume and Polymer Rheology, in Rheology, Theory and Applications, Vol. 5, Ed. F. R. Eirich, Academic Press, N.Y. & London, 1969.
2. E. A. DiMarzio, J. H. Gibbs, P. D. Fleming, III and I. C. Sanchez, Macromoleucles, Vol. 9 #5, 763, 1976.
3. A. Quach and R. Simha, J. Appl. Phys., Vol. 42 #12, 4592, 1971.
4. S. Matsuoka and T. K. Kwei, Physical Behavior of Macromolecules, Filing Case #37940-666, Internal Memorandum - Bell Laboratories, 1977.
5. U. Bianchi, J. Phys. Chem., 43, 139, 1965.
6. M. Goldstein, J. Phys. Chem., 77 #5, 667, 1973.
7. M. Goldstein, J. Phys. Chem., 39 #12, 3369, 1963.
8. J. E. McKinney and M. Goldstein, J. Res. Nat. Bur. Stad.-A, 78A #3, 331, 1974.
9. J. D. Ferry, Viscoelastic Properties of Polymers, John Wiley & Sons, N.Y. - Toronto, 1970.
10. J. M. O'Reilly, J. Poly. Sci., 57, 429, 1962.
11. W. G. Knauss and H. K. Mueller, The Mechanical Characterization of Solithane 113 in the Swollen and Unswollen State, AFRPL-TR-68-125, 1967.
12. N. G. McCrum, B. E. Read, G. Williams, Anelastic and Dielectric Effects in Polymeric Solids, John Wiley & Sons, London-New York-Sidney, 497, 1967.
13. H. J. Oels and G. Rehage, Macromolecules, 10 #5, 1036, 1977.

FIGURE CAPTIONS

Figure 1 - Dielectric Apparatus

Figure 2 - PVT Apparatus

Figure 3 - Mechanical Testing Apparatus

Figure 4 - ϵ'' vs. Temperature at 1 bar

Figure 5 - T_g vs. Pressure

Figure 6 - Specific Volume vs. Temperature at Various Pressures

Figure 7 - dT_g/dP, dT_g^*/dP and $\Delta\beta/\Delta\alpha$ vs. Pressure

Figure 8 - Young's Modulus and Bulk Modulus vs. Pressure

Figure 9 - Specific Volume vs. Pressure at 25°C

TABLE I

Bulk, Young's and Shear Moduli Versus Pressure

P(kb)	B($\times 10^{-10}$ dyne/cm^2)	E($\times 10^{-7}$ dyne/cm^2)	G($\times 10^{-7}$ dyne/cm^2)
0	2.19	2.21	0.74
1.10	3.16	2.63	0.88
2.13	4.27	3.33	1.11
2.38	4.59	6.25	2.08
2.57	4.81	24.3	8.10
2.67	4.97	46.7	15.6
3.11	5.52	175.	58.5
3.67	6.03	403.	135.
4.11	6.35	516.	174.
5.00	6.71	728.	246.
6.50	8.13	891.	301.

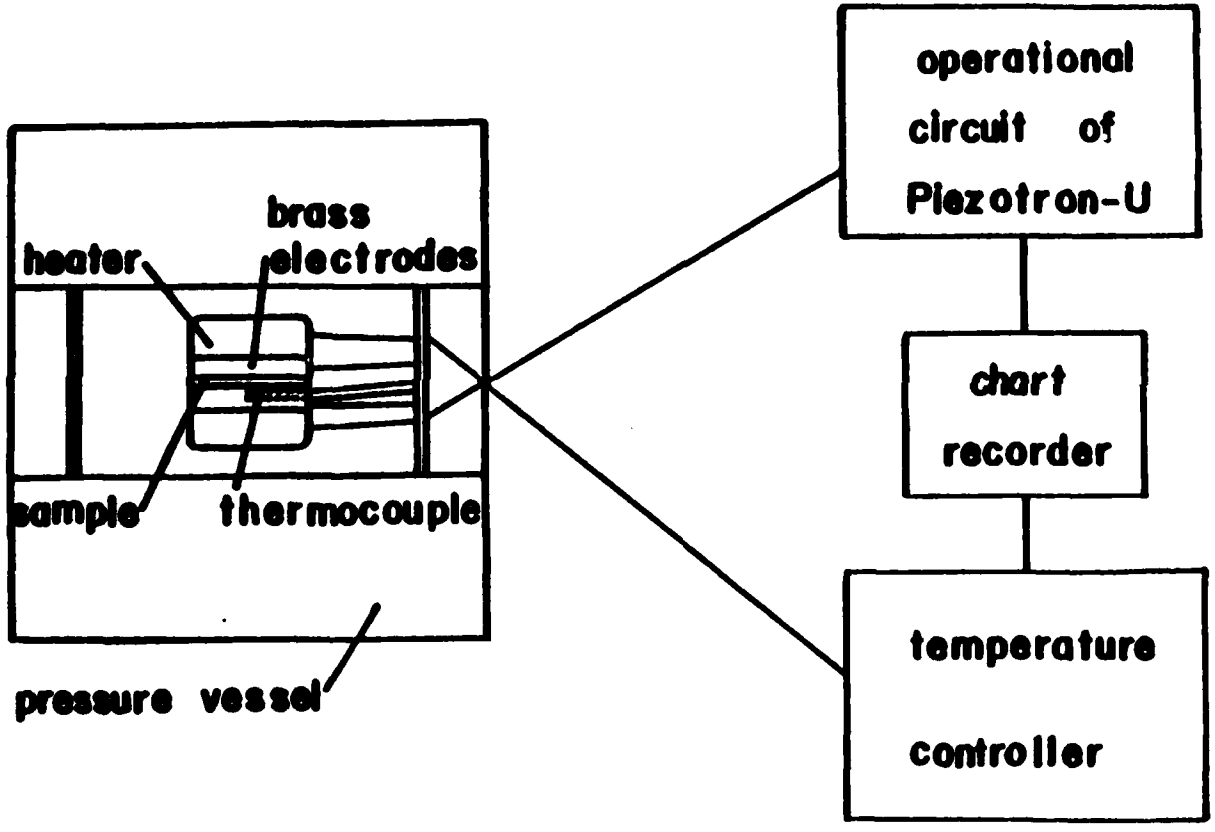

operational
circuit of
Piezotron-U
brass
electrodes
heater
chart
recorder
sample
thermocouple
temperature
controller
pressure vessel

Figure 1

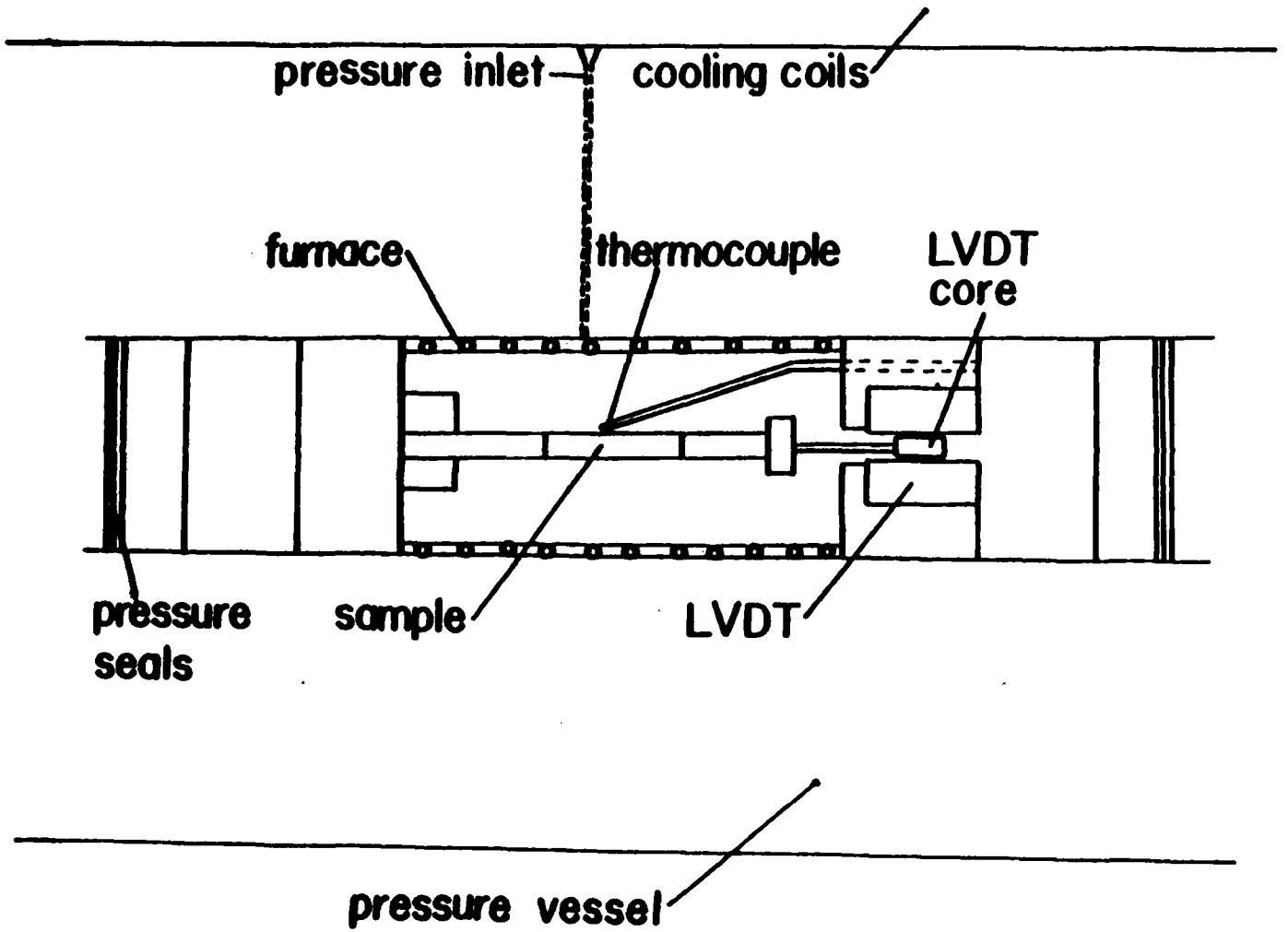

pressure inlet
cooling coils
furnace
thermocouple
LVDT
core
pressure
seals
sample
LVDT
pressure vessel

Figure 2

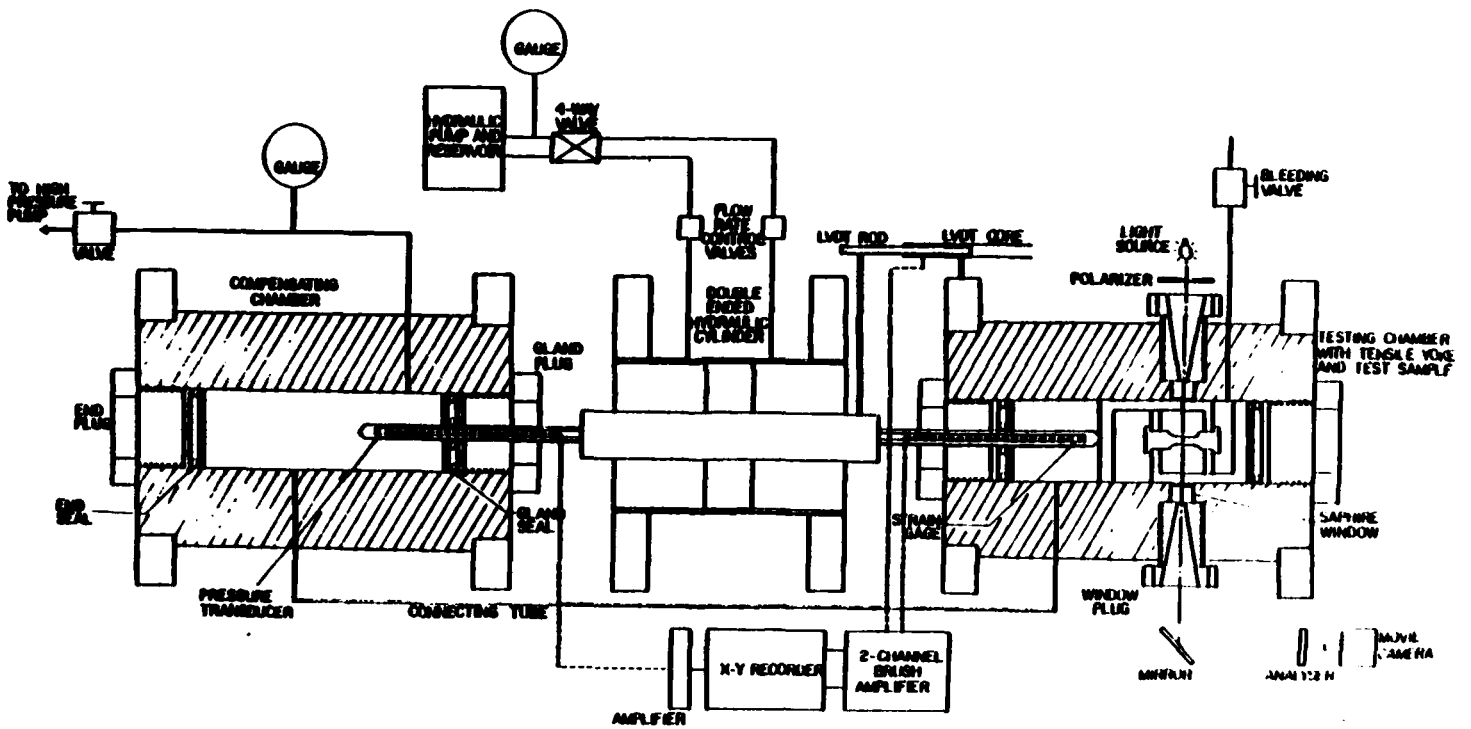

GAUGE
GAUGE
4-WAY VALVE
BLEEDING VALVE
VALVE
COMPENSATING CHAMBER
FLOW RATE CONTROL VALVES
LVDT ROD
LVDT CORE
LIGHT SOURCE
POLARIZER
GLAND PLUG
TESTING CHAMBER WITH TENSILE YOKE AND TEST SAMPLE
END PLUG
GLAND SEAL
STRAIN GAUGE
SAPPHIRE WINDOW
PRESSURE TRANSDUCER
CONNECTING TUBE
WINDOW PLUG
X-Y RECORDER
2-CHANNEL BRUSH AMPLIFIER
AMPLIFIER

Figure 3

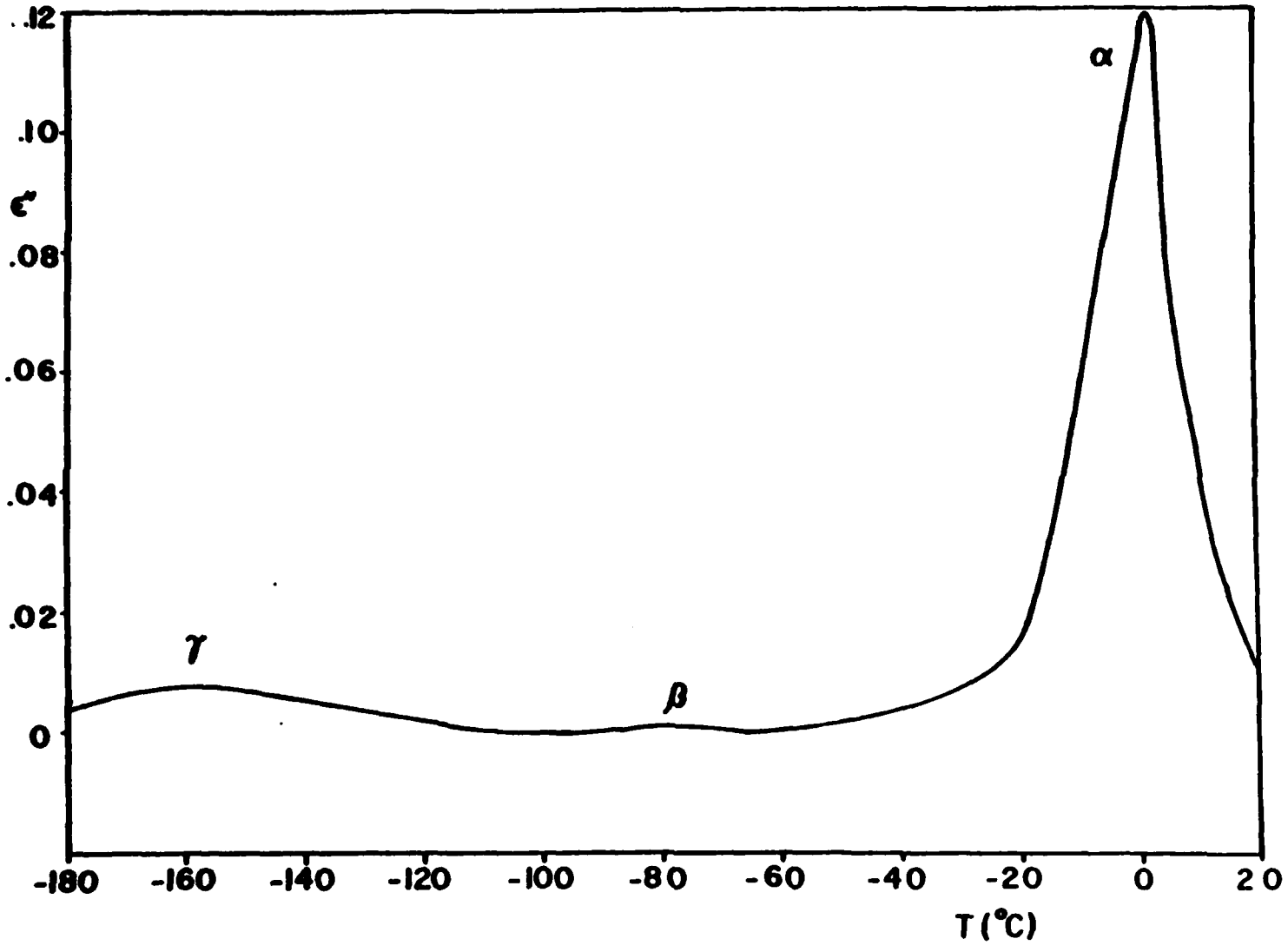


Figure 4

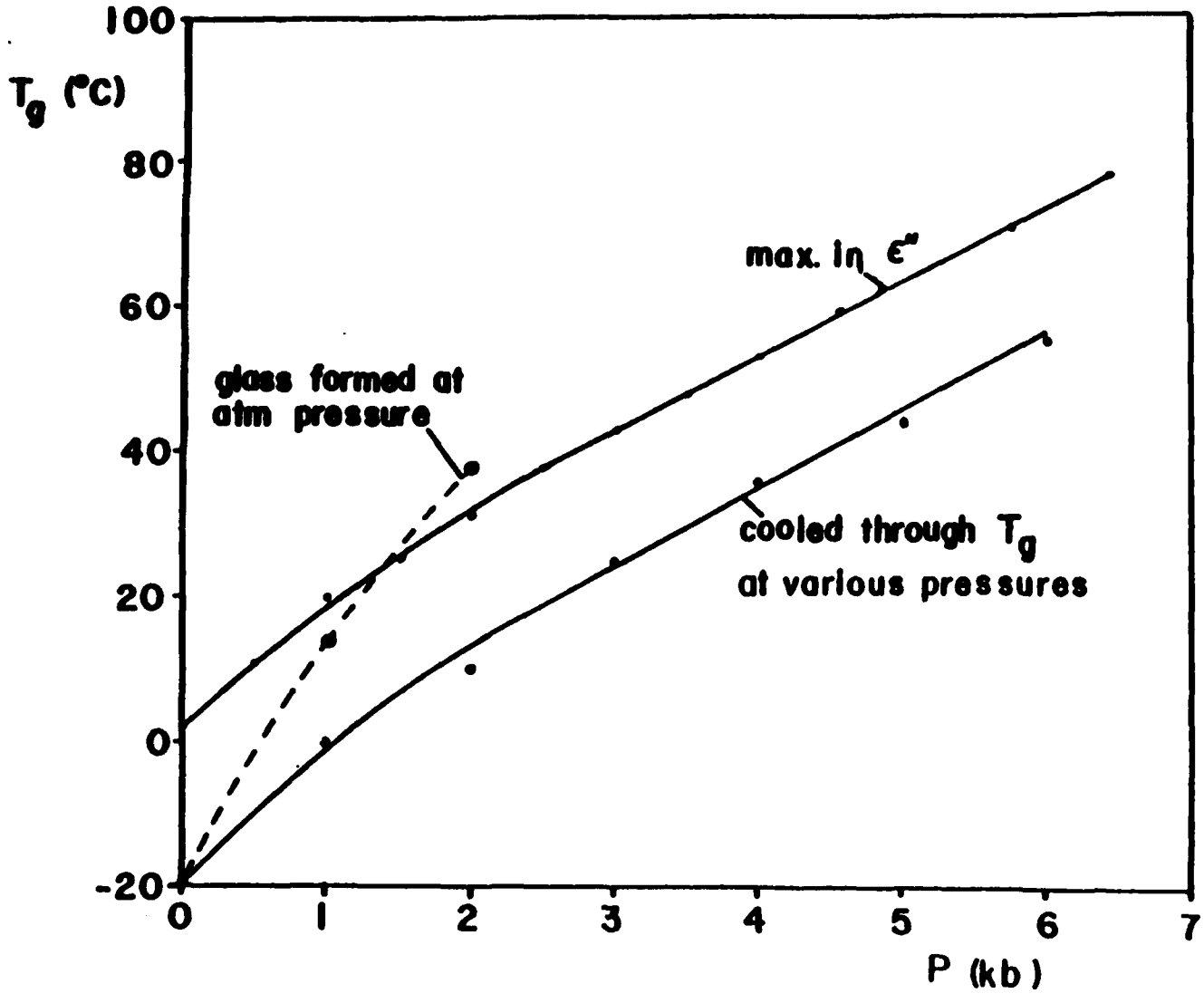

100
80
60
40
20
0
-20
T_g (°C)
0
1
2
3
4
5
6
7
P (kb)
max. in ϵ''
glass formed at
atm pressure
cooled through T_g
at various pressures

Figure 5

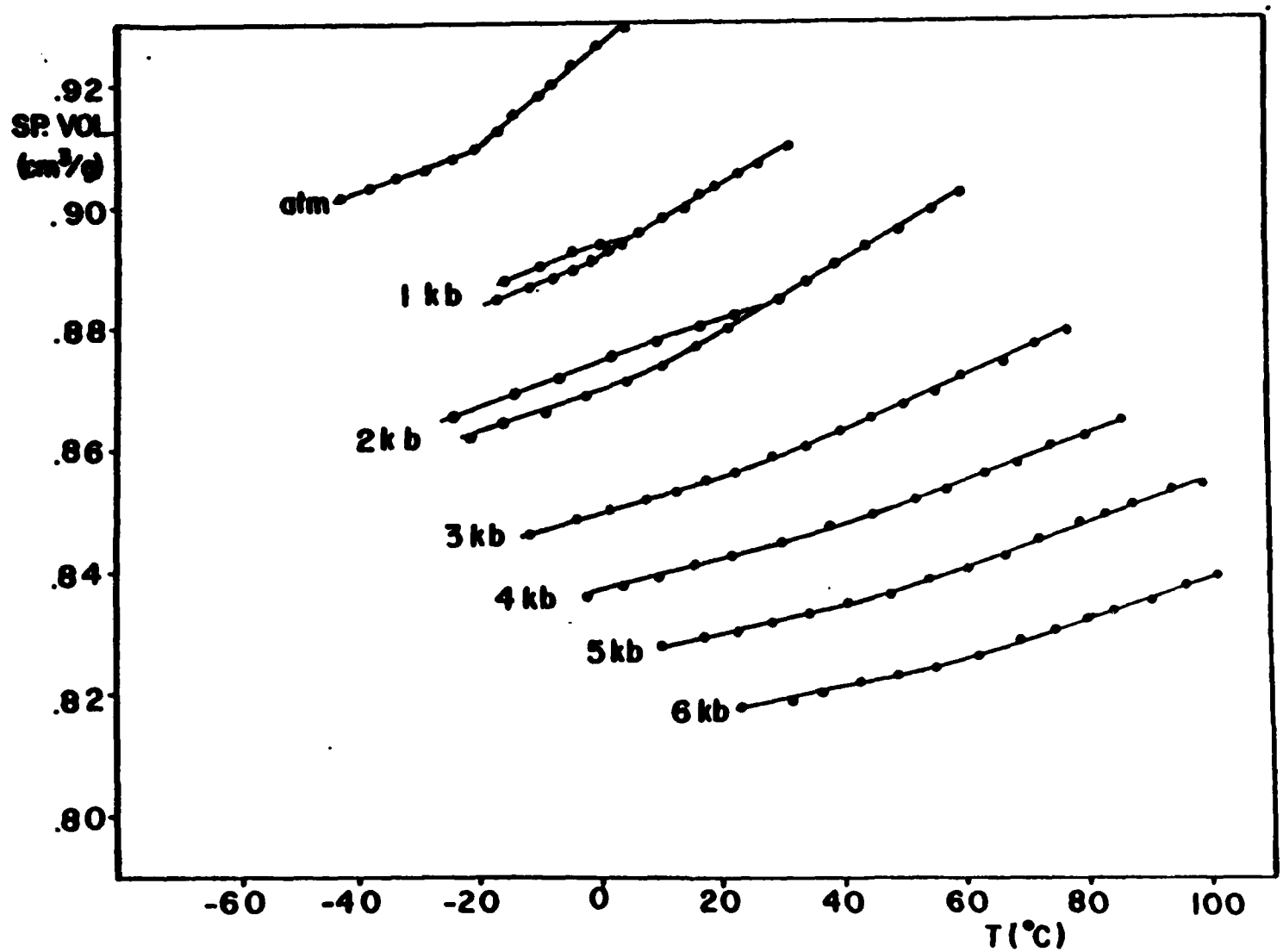

.92
SP. VOL
(cm³/g)
.90
.88
.86
.84
.82
.80
atm
1 kb
2 kb
3 kb
4 kb
5 kb
6 kb
-60
-40
-20
0
20
40
60
80
100
T(°C)

Figure 6

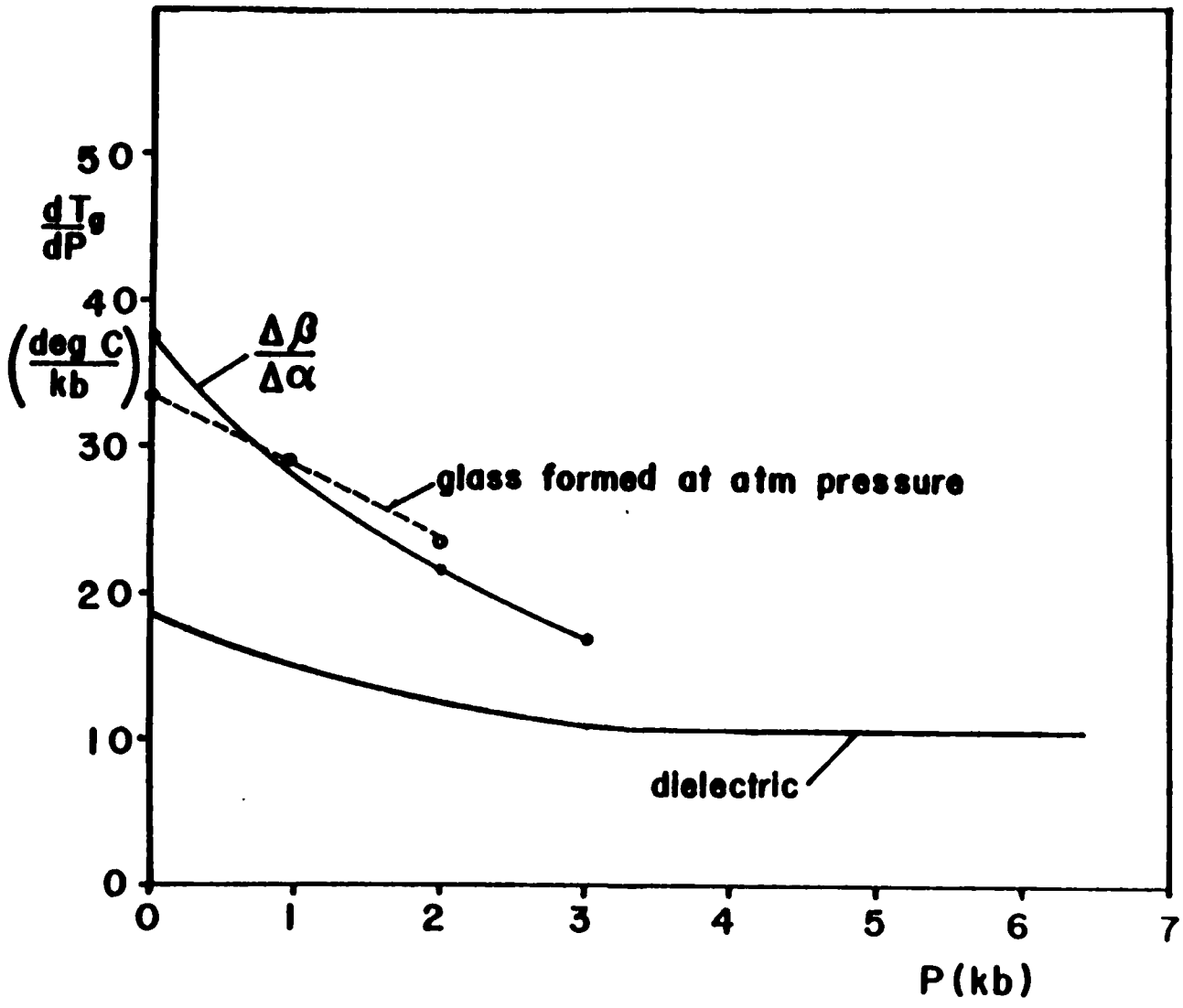

50
40
30
20
10
0
$\frac{dT_g}{dP}$
$\left(\frac{\text{deg C}}{\text{kb}}\right)$
$\frac{\Delta\beta}{\Delta\alpha}$
glass formed at atm pressure
dielectric
0
1
2
3
4
5
6
7
P(kb)

Figure 7

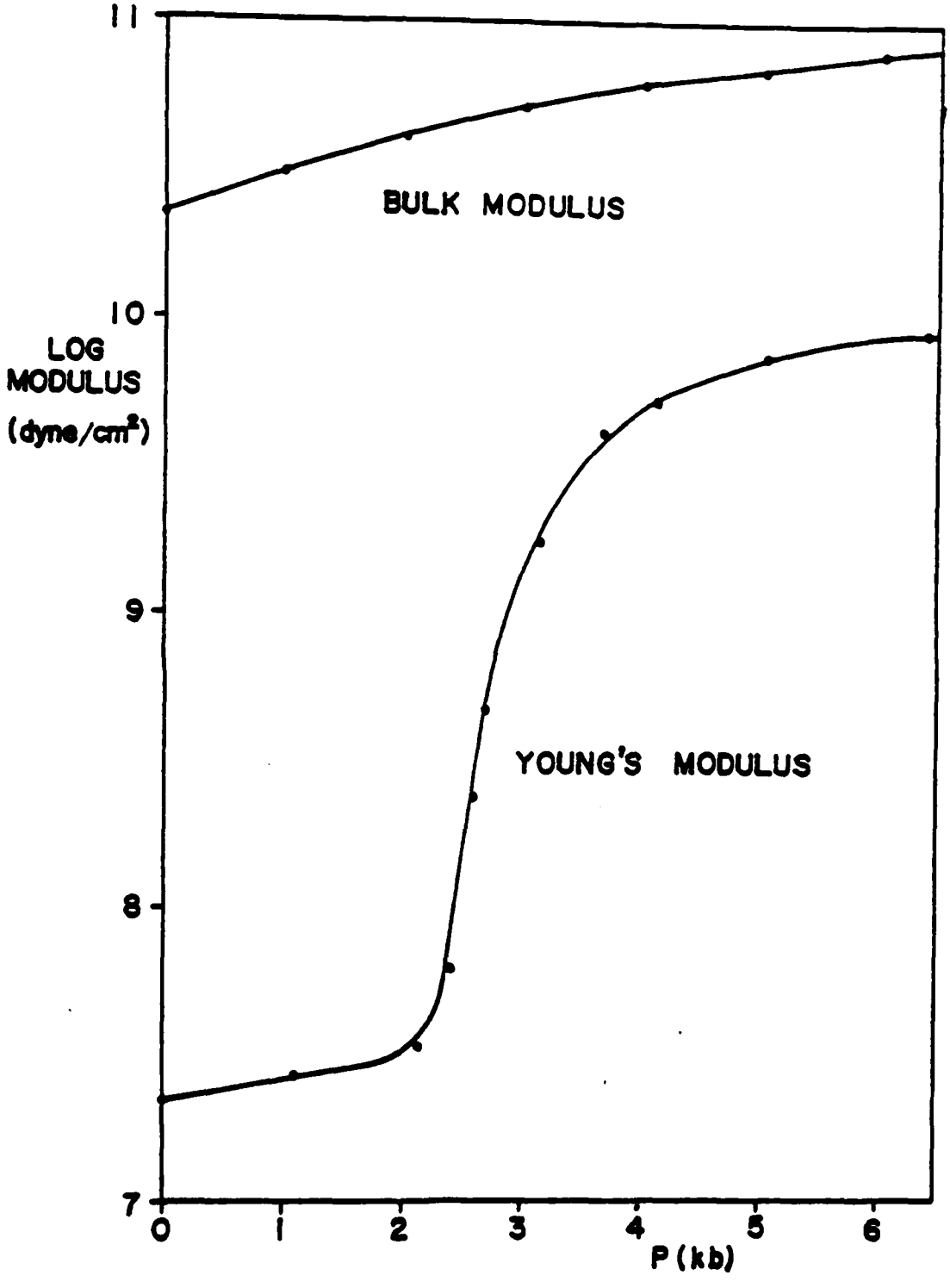

11
10
9
8
7
LOG
MODULUS
(dyne/cm²)
BULK MODULUS
YOUNG'S MODULUS
0
1
2
3
4
5
6
P (kb)

Figure 8

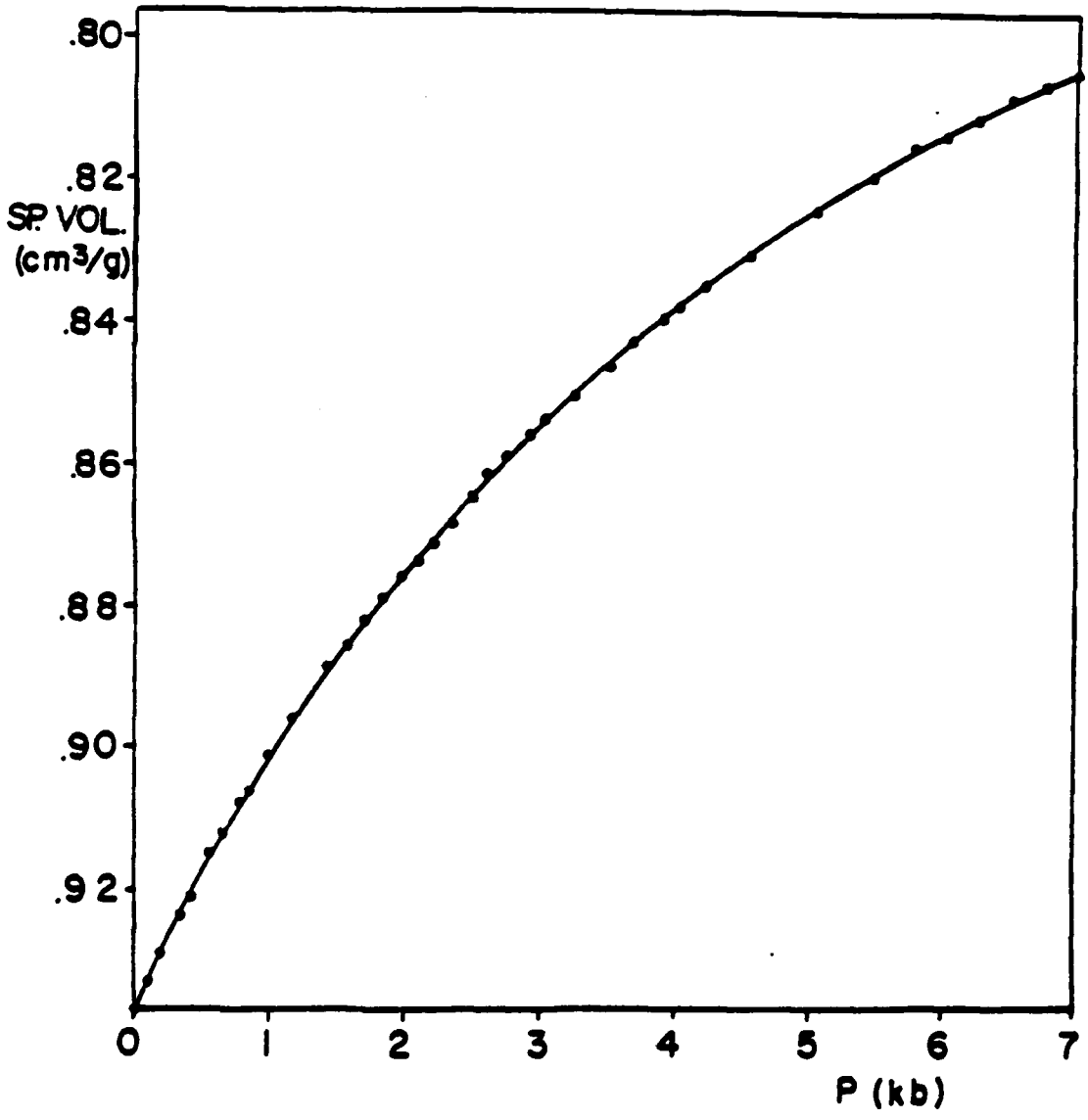


Figure 9