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THE EFFECT OF TIME AND TEMPERATURE ON THE MECHANICAL BEHAVIOR OF A 'PLASTICIZED' EPOXY RESIN UNDER DIFFERENT LOADING MODES

A. E. Moehlenpah, O. Ishai, and A. T. DiBenedetto

September 1968

PROGRAM MANAGER
ROLF BUCHDAHL

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FOREWORD

The research reported herein was conducted by the staff of the Monsanto/Washington University Association under the sponsorship of the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-67-C-0218 (formerly N00014-66-C-0045), ARPA Order No. 873, ONR contract authority NR 356-484/4-13-66, entitled "Development of High Performance Composites."

The prime contractor is Monsanto Research Corporation. The Program Manager is Dr. Rolf Buchdahl (phone 314-694-4721).

The contract is funded for $5,000,000 and expires 30 April 1970.

Abstract

Epoxy-Versamid specimens were loaded in tension, compression, and flexure at different strain rates and temperatures to determine mode of failure, yield stress and strain, and tangent and relaxation moduli. Stress-strain curves were used to define brittle, ductile, ductile-rubbery, and rubbery modes of behavior which prevailed in different temperature-strain rate regions. The time-temperature superposition principle was applied to yield stress, initial tangent moduli, and relaxation moduli data for all three types of loading.

The transition regions, tangent and relaxation moduli, and shift factors were the same in tension, compression, and flexure. Thus the most convenient mode of loading can be used to determine the general time-temperature dependence. The ratio of compressive to tensile yield stress was almost constant over the entire ductile region. Flexural yielding data were used to predict yield stress in tension and compression, and stress relaxation master curves were shown to be related to elastic modulus versus strain rate curves. The yielding phenomenon was interpreted using Eyring's theory of non-Newtonian viscoplastic flow. The apparent activation energy and activation volume were larger for tension than compression. A theory is offered to explain why yielding can occur in a cross-linked system.

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I. Introduction

In practical applications, most materials are subject to combined states of stress over long periods of time. Consequently, it is necessary in most cases to determine the mechanical properties in tension, compression, and bending as a function of time. To minimize the amount of data required for reliable engineering design, superposition principles have been elucidated. Leaderman [1, 2] observed that creep recovery data obtained at different temperatures can be superposed by horizontal translation along the logarithmic time axis. Stress relaxation curves of modulus versus time at different temperatures can also be superposed by horizontal translation [3]. This is equivalent to the assertion that the effect of temperature on linear viscoelastic properties is to multiply (or divide) the time scale by a constant factor at each temperature. The shift factor A_T is chosen as unity at the reference temperature T_0 and is generally a function of temperature [4, 5]. By sliding curves of $E_T(t)$ along the log time axis to superpose with $E_{T_0}(t)$, a master curve covering a much wider range of log time can be constructed. This time-temperature superposition principle enables one to predict viscoelastic behavior in regions of time not experimentally accessible. Thus short-term tests, carried out at temperatures somewhat higher than those normally encountered by the material, are used to predict long-term properties. While no completely convincing theoretical analysis has yet been put forward to explain the validity of the time-temperature superposition principle, it has been verified by a number of investigators [6, 7].

Lohr [8] compared the time-temperature dependence of yield stress (YS) with that of stress relaxation (SR) for polymethylmethacrylate, polyethylene terephthalate, polystyrene, and polyvinylchloride. All of the yield stress data could be described by the equation $YS = K_1 + K_2 \ln (\dot{\varepsilon} A_T)$ where K_1 and K_2 are constants and $\dot{\varepsilon}$ is the strain rate. He found that in general the yield stress shift factors were smaller than the stress relaxation shift factors. His SR experiments were determined at 1.43% strain whereas YS experiments were at higher strains where nonlinearity and crack propagation occurs. This hints that A_T was a function of stress or strain as well as temperature.

Most previous investigations of the time-temperature superposition principle have been done under just one mode of loading such as tension. It is expected that in the region of linear viscoelastic behavior, one can predict the behavior in other modes from data on one. The behavior in the region of yielding, however, may not be predictable.

Ishai [9] carried out a series of loading tests on 1 : 1 weight ratio Epon 815-Versamid 140 samples at varying constant strain rates (CSR), under tension, compression, and flexure at room temperature. In all cases, the yield stress was linear with the log strain rate. The ratio of compressive to tensile yield stress ranged from 1.27

to 1.38. Formulae* relating the maximum yield moment and yield stress in flexure were utilized. These formulae enable one to use flexural yield data to predict compressive and tensile yield stress if one knows the ratio of compressive to tensile yield stress. A series of creep tests was also performed on this material at high stress levels under tension, compression, and flexure at room temperature [10]. In all cases, there was a linearity between creep stress and log secondary creep rate, which is almost coincident with the corresponding relationship between yield stress and log strain rate obtained in the CSR tests. The deformation during yielding which was not recoverable at room temperature was almost completely recoverable at high temperatures.

It was proposed that the linearity between yield stress and log strain rate was in accordance with Eyring's [11] theory of non-Newtonian viscous flow at high stress

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*The relation between maximum yield moment and that of compressive and tensile stresses in flexure is obtainable on the approximate assumption of a rectangular stress diagram for the plastic zone of the beam, whereby:

$$\sigma_{y_t} = [2(1 + \lambda)/\lambda]\ (M_y/bh^2)$$

$$\sigma_{y_c} = [2(1 + \lambda)]\ (M_y/bh^2)$$

where M_y = yield moment (at midsection), σ_{y_t} = yield stress at the extreme tensile fibers, σ_{y_c} = yield stress at the extreme compressive fibers $\sigma_{y_c}/\sigma_{y_t} = \lambda$ = compressive to tensile yield stress rates, and b, h = width and depth of beam cross-section, respectively.

levels and low temperatures. Whereas an apparent activation volume could be calculated from the above results at room temperature, calculation of the apparent activation energy required a study of the influence of temperature on yielding characteristics. Specimens with a 60/40 and 70/30 weight ratio of Epon 815-Versamid 140 were studied in tension at various temperatures and strain rates to determine transition regions, moduli, stress relaxation, yield stress, apparent activation volume and apparent activation energy* [12]. The paper presented here is an extension of this latter work, covering both a wider range of experimental conditions and compression and flexure modes of loading.

II. Experimental Procedure

A 60/40 weight ratio of Shell Epon Resin 815 and General Mills Versamid 140 Polyamide Resin was chosen because of the low viscosity of the resins and the ductility

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*The activation volume can be determined from the slope of the σ_y versus log strain rate curves

$$V_0 = \frac{4kT}{K_2 \log \epsilon}$$

where V_0 = activation volume, k = Boltzmann constant, T = absolute temperature and K_2 = slope of σ_y versus log strain rate. The activation energy can be determined from plots of σ_y versus temperature with strain rate as a constant parameter.

$$Q = - \frac{K_3 T_0 V_0 N}{4}$$

where Q = activation energy, T_0 = temperature obtained by extrapolating a linear σ_y versus T plot to zero stress, $N = 6.02 \times 10^{23}$, K_3 = slope of σ_y versus T.

of the hardened product. Epon resin 815 is a light-colored, low-viscosity, epichlorohydrin/bisphenol A -type epoxy resin containing a reactive diluent. Versamid resins are condensation products of polyamines and dibasic acids. Versamid 140 serves both as a curing agent and plasticizer. The reaction with amines involves opening the epoxide ring to give a β-hydroxyamino linkage. Since the versamid amine has a functionality greater than 2, the final product is cross-linked. The final product appears to be ductile because both the amine and the epoxy reactants have a high molecular weight. Consequently, the distance between cross-links is large, allowing large deformations to take place between the cross-link points. Also, an excess of versamid serves as a plasticizer or blended copolymer.

The Versamid 140 and Epon 815 were heated separately to 40°C and evacuated in a vacuum oven. Then the components were mixed under vacuum and visually checked to see that the air was removed. Flexural and tensile samples were cast between two sheets of glass while the compressive samples were cast in test tubes. The glass was treated with a 20 percent by volume dimethyl dichlorosilane in carbon tetrachloride mixture for a release agent. Standard ASTM D-638 tensile samples were machined. The compression cylinders were approximately 1/2 inch in diameter and 1 inch long, while the flexural bars were approximately 1/4 x 1/4 x 2-1/2 inches.

All tests were conducted on an Instron Testing Machine. An electrical extensometer was used to measure the strain of the tensile samples while the strain of the flexural and compression samples was determined from the recorder paper movement utilizing the paper to crosshead speed ratio and a calibrated correction of testing

machine deflection versus load. The tangent of the initial portion of the force versus deflection curve was used to determine the elastic modulus. The peak of the curve was used to calculate the yield stress and strain.

All compressive stress relaxation experiments were done by moving the crosshead at a speed to give a strain rate of 0.2 min^{-1}. The crosshead was stopped at a strain in the linear portion of the force-deformation curve and the force at this strain was recorded as a function of time and converted to modulus. The tensile and flexural SR experiments were performed like the compression tests except that the initial strain rate was 0.05 min^{-1} and 0.3 min^{-1}, respectively.

The temperature was varied with the use of an Instron environmental chamber. The temperature was measured by a thermometer suspended with the bulb near the sample.

III. Results

The stress-strain behavior is dependent on temperature and strain rate. Figure 1 shows the stress-strain behavior in compression at a constant strain rate of .2 min^{-1} and at different temperatures. At 61°C and below, the material behaves in a ductile manner with the stress-strain curves showing a yield maximum. At 74.8°C the material is ductile-rubbery with the stress-strain curve showing an inflection point but not a maximum. At 97°C and 107°C the material is rubbery with the stress-strain curve having constantly increasing slope showing no inflection or maximum. These behavior patterns define the present terminology of ductile, ductile-rubbery, and rubbery.

Figure 2 shows the stress-strain relationship in compression at 61°C at different constant strain rates. It demonstrates the transition from ductile to rubbery mode of behavior as affected by strain rate decrease. In tension at low temperatures and high crosshead rates, the material exhibits brittle behavior; i.e., it breaks in the decreasing slope region before the stress-strain curve reaches a maximum. Figure 3 shows the transition regions in terms of temperature and strain rate. The transition for tension is indicated by the dotted line. The transition from ductile to rubbery is about the same for both compression and tension. In flexure, the stress-strain curves almost always show a maximum except at low temperatures where brittle failure predominates.

Figure 4 plots yield stress in compression versus log strain rate at various temperatures in the ductile region. (In this paper only ductile behavior, where the stress-strain curve shows a definite yield maximum, is used to define yield stress.) Figure 5 shows the results in flexure. The strain rate and yield stress in flexure is calculated from the simple beam theory. Yield stress versus log strain rate is practically linear with the slope, decreasing slightly as the temperature decreases. The results in tension are similar.

The yield stress versus log strain rate data were shifted to obtain yield stress master curves in compression, tension, and flexure as shown in Figure 6. Figure 7 shows the shift factors used and indicates that shift factors for compression, tension, and flexure are the same. It is significant that the ratio of yield stress in compression to that in tension (λ) remains an almost constant value of 1.28 over eight decades of strain rate. Using this value of λ, one can calculate the yield in tension and compression from the flexural yield data [9]. It can be seen that flexural yield moment

data can be used to accurately predict the yield stress in tension and compression over the entire strain-rate and temperature (ductile) range if λ is known. It is also significant that yield stress versus log strain rate is practically linear over the entire ductile region. Thus the data can adequately be represented by the equation $YS = K_1 + K_2 \log (\dot{\epsilon} A_T)$.

The apparent activation volume in compression was found to be 4090 $\mathring{A}^3$ and the activation energy was 50 KCal/mole. Values of 4800 $\mathring{A}^3$ and 58 KCal/mole were determined in tension. The difference in Q and V_0 for tension and compression is above the expected experimental scatter, but the cause of this difference is not clear at this time.

The deformation which occurred at yielding under any of the modes of loading was recoverable at high temperatures. Whereas from a rheological or macroscopic point of view, plastic non-recoverable yielding occurs, on a microscopic level some sort of retarded elasticity is taking place. Flow probably occurs in the material since there is excess of Versamid plasticizing the structure, but the loose cross-link network apparently is stretched and exerts an elastic restoring force. This force is strong enough to cause the sample to recover only when the temperature is high enough to activate segment mobility.

The total strain at the yield point in the ductile region seems to be relatively independent of temperature and strain rate. Over the range of variables studied, the compressive yield strain was (4.5 $\pm$ .8) percent, while the tensile yield strain was (5.0 $\pm$ 0.6) percent. Thus the yield strain in compression might be slightly less than

in tension. Since the compressive yield stress is 1.28 times the tensile yield stress, the secant modulus at yield for compression is about 1.4 times that in tension. When the materials approach the rubbery region, both the compressive and tensile yield strain increase rather rapidly. Flexural yield strain appears to be somewhat higher than yield strain in tension and compression, but this might only be due to the inadequacy of the simple beam theory for analyzing the strain behavior around the yield point.

Figure 8 shows the initial tangent modulus in compression versus log strain rate for various temperatures. The flexural and tensile moduli show identical behavior. Figure 9 shows the initial compression modulus versus temperature at constant strain rates of 0.02, .2, and 2 min^{-1}. Modulus increases with decrease in temperature and with increase in strain rate. The glass transition temperature of the material is seen to be about 70 ± 10°C and is strain rate dependent as expected. Figure 10 shows the initial modulus versus temperature for flexure, tension, and compression at a strain rate of 0.2 min^{-1}. The modulus determined by the three methods is for all practical purposes the same. One might expect at temperatures where modulus is affected by strain rate, that the modulus determined by flexure would be somewhat different from compression and tension because the strain rate in flexure varies from zero at the neutral axis to a maximum at the outer edge. It must be kept in mind, though, that the strain rate dependence of E is very small compared to its temperature dependence so that this effect can be neglected within the experimental error.

Figure 11 is a master curve of all the modulus data using 24°C as a reference temperature. The required shift factors, plotted in Figure 12, again indicate that the

shift factors for compression, flexure, and tension are roughly the same. Since flexure testing is by far the simplest to perform, it could provide the basic test for predicting the behavior under more complex testing modes and other combined states of stress.

Figure 13 is typical of the relaxation behavior at various temperatures. All of the stress relaxation data were shifted to form the stress relaxation master curve of Figure 14 with reference temperature of 24.0°C. The shift factors are plotted in Figure 15. It can be noted that the shift factors for stress relaxation are also identical for the three modes of loading. Furthermore, the shift factors for stress relaxation are identical to those for initial modulus, as might be expected for a linearly viscoelastic material. Also, as expected, the stress relaxation master curve (Figure 14) and the modulus master curve (Figure 11) are practically mirror images of one another. This is significant in that stress relaxation data can be used to predict modulus versus strain rate or vice-versa. It appears that this method of prediction would be accurate over as many orders of magnitude of time or strain rate as practical applications would require (e.g., 10^8 min is 191 years).

It is also clear that the shift factor for the yield stress is somewhat smaller (or the activation energy lower) than for the stress relaxation and initial modulus. This leads one to believe that there is also stress dependence in the shift factor as one would predict from simple rate theory.

IV. Conclusion

Comprehensive mechanical testing of this material has led to the following conclusions:

1. The transition behavior, elastic modulus, yield stress, and stress relaxation are temperature and strain rate dependent in the ductile region.
2. The transition from ductile to rubbery behavior occurs at about the same strain rates and temperatures in both compression and tensile testing.
3. Elastic moduli, stress relaxation, and yield stress data can be correlated by the time-temperature superposition principle.
4. The shift factors for stress relaxation and elastic moduli are the same but are slightly bigger than the yield stress shift factors. This leads to the probability that the shift factor is stress dependent.
5. The shift factors determined by flexure, tension, and compression testing are roughly the same for a given property.
6. Yield stress can adequately be represented by $YS = K_1 + K_2 \log (\dot{\epsilon} A_T)$ over greater than eight decades of shifted strain rate.
7. At a given temperature and strain rate, the yield stress determined in compression is 1.28 times as great as that in tension over the entire temperature and strain rate region tested.
8. Flexure yield stress quite accurately predicts the yield stress in tension and compression when one assumes a rectangular-shaped stress distribution at yielding.
9. Yield strain, unlike yield stress, is not simply related to temperature and strain rate.
10. The flexural test method is not a good method to determine the yield strain of a cross-linked material that is near Tg.
11. At a given temperature and strain rate, the elastic moduli and stress relaxation determined in tension, compression, and flexure are the same.

12. Elastic moduli versus (strain rate)$^{-1}$ can be used to predict stress relaxation and vice-versa.

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The work described in this paper is part of the research conducted by the Monsanto/Washington University Association sponsored by the Advanced Research Projects Agency, Department of Defense, under ONR contract N00014-67-C-0218. Calculations were performed using the Washington University computing facilities through NSF grant G-22296.

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Figure 1 - Compressive Stress-Strain Curves at Constant Strain Rate of 0.2 $(Min)^{-1}$

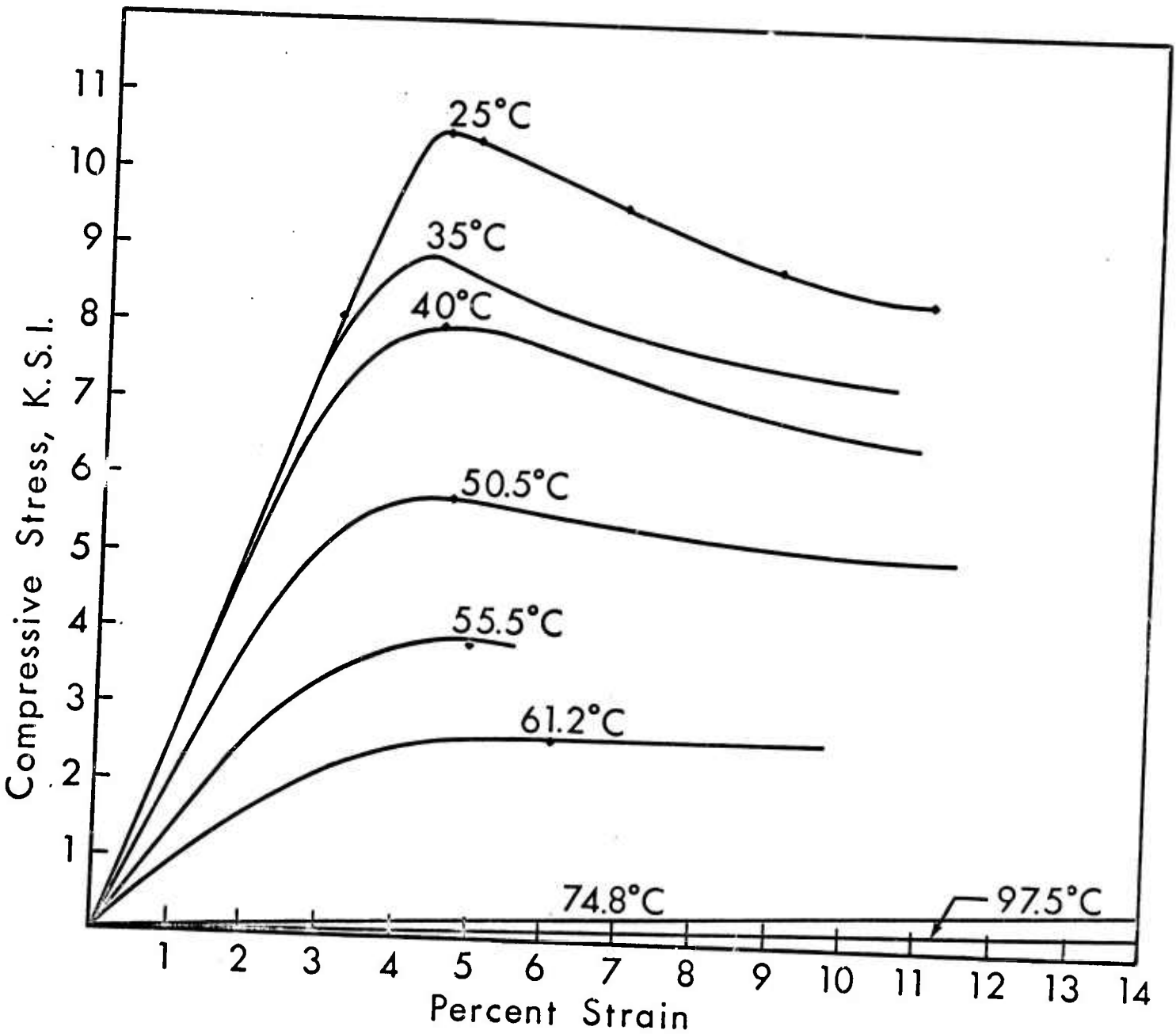


Figure 2 - Compressive Stress-Strain Curves at 61°C.

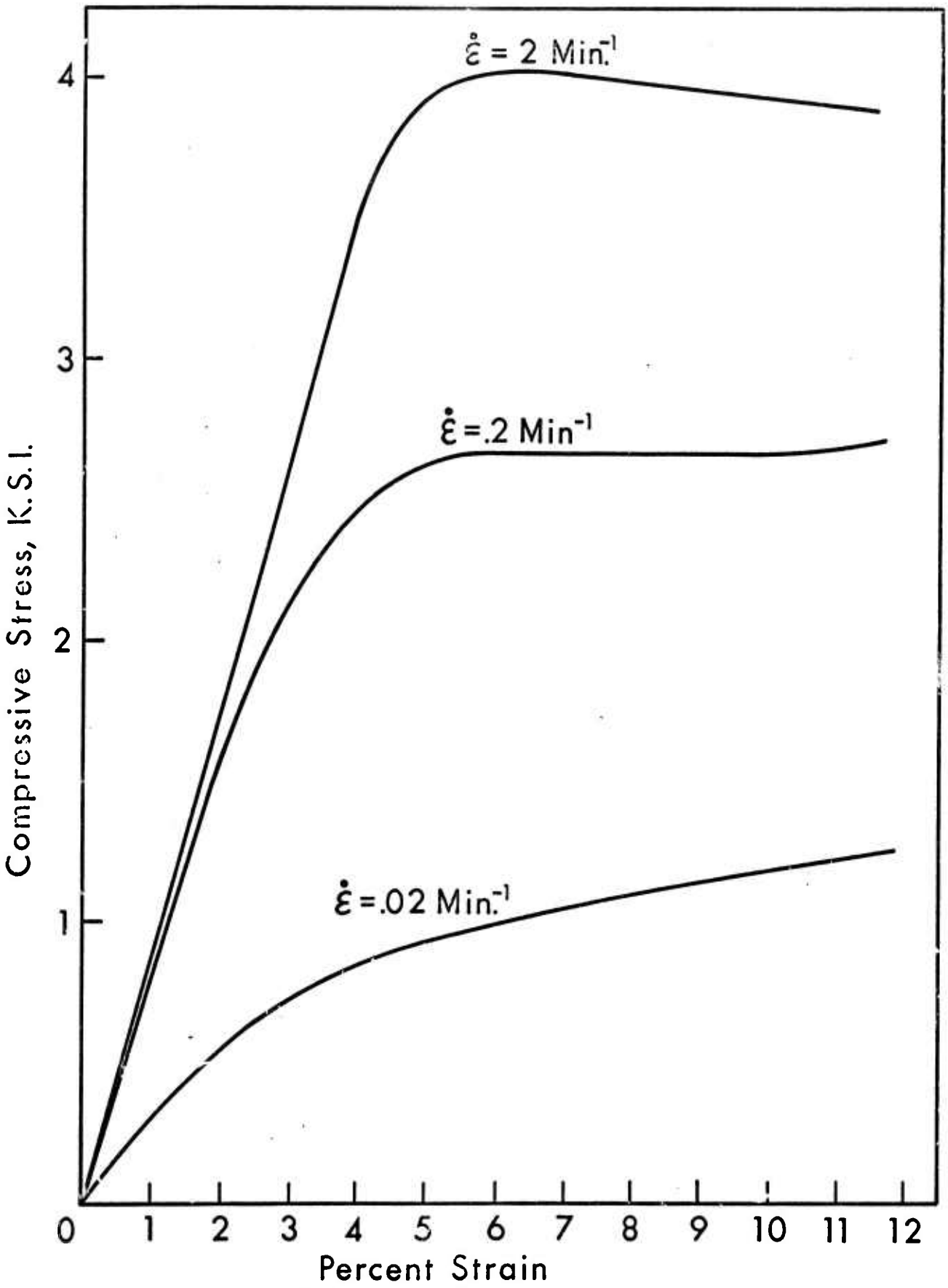


Figure 3 - Transitions in Failure Modes Based on Stress-Strain Curves

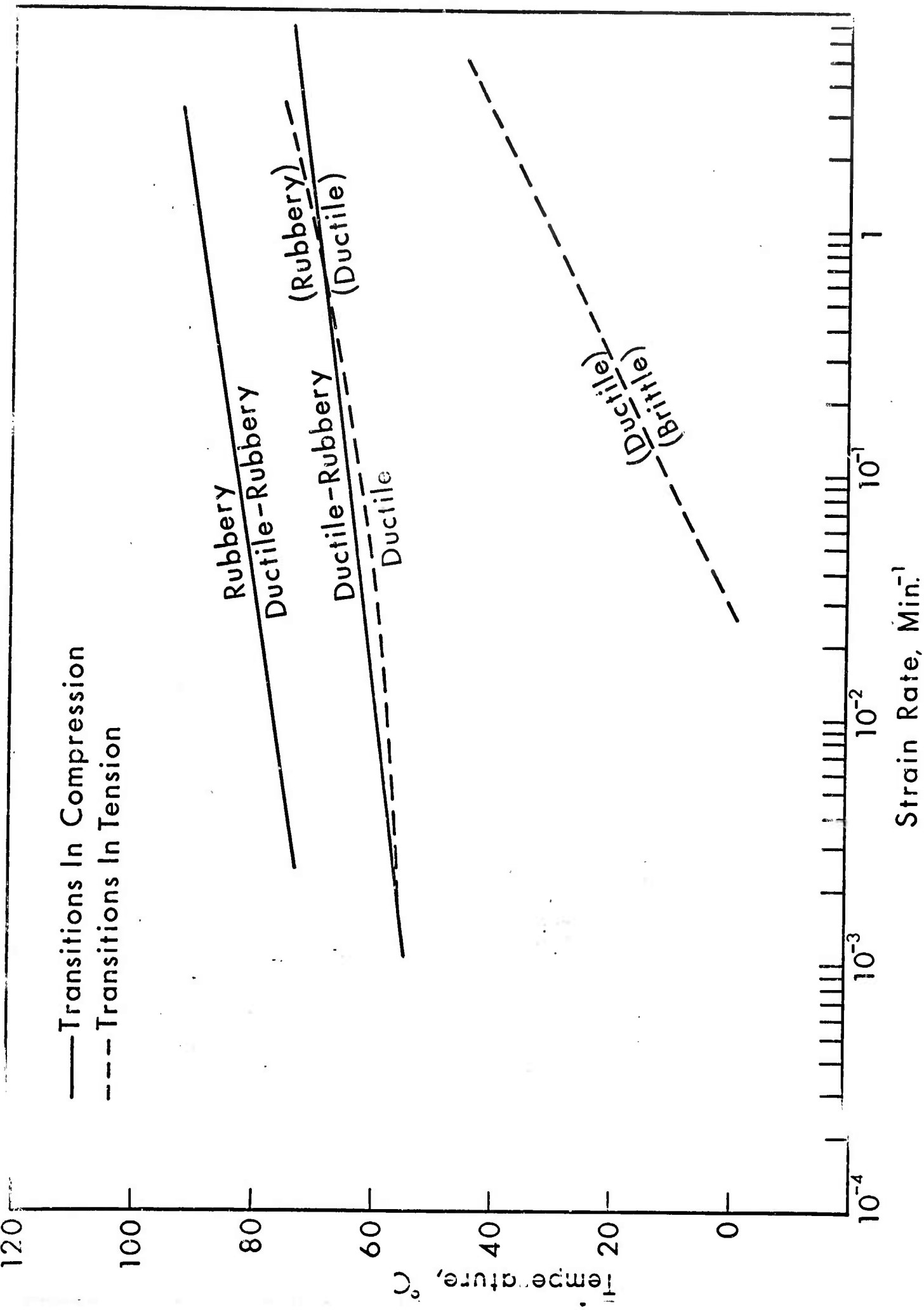


Figure 4 - Compression Yield Stress Vs. Strain Rate

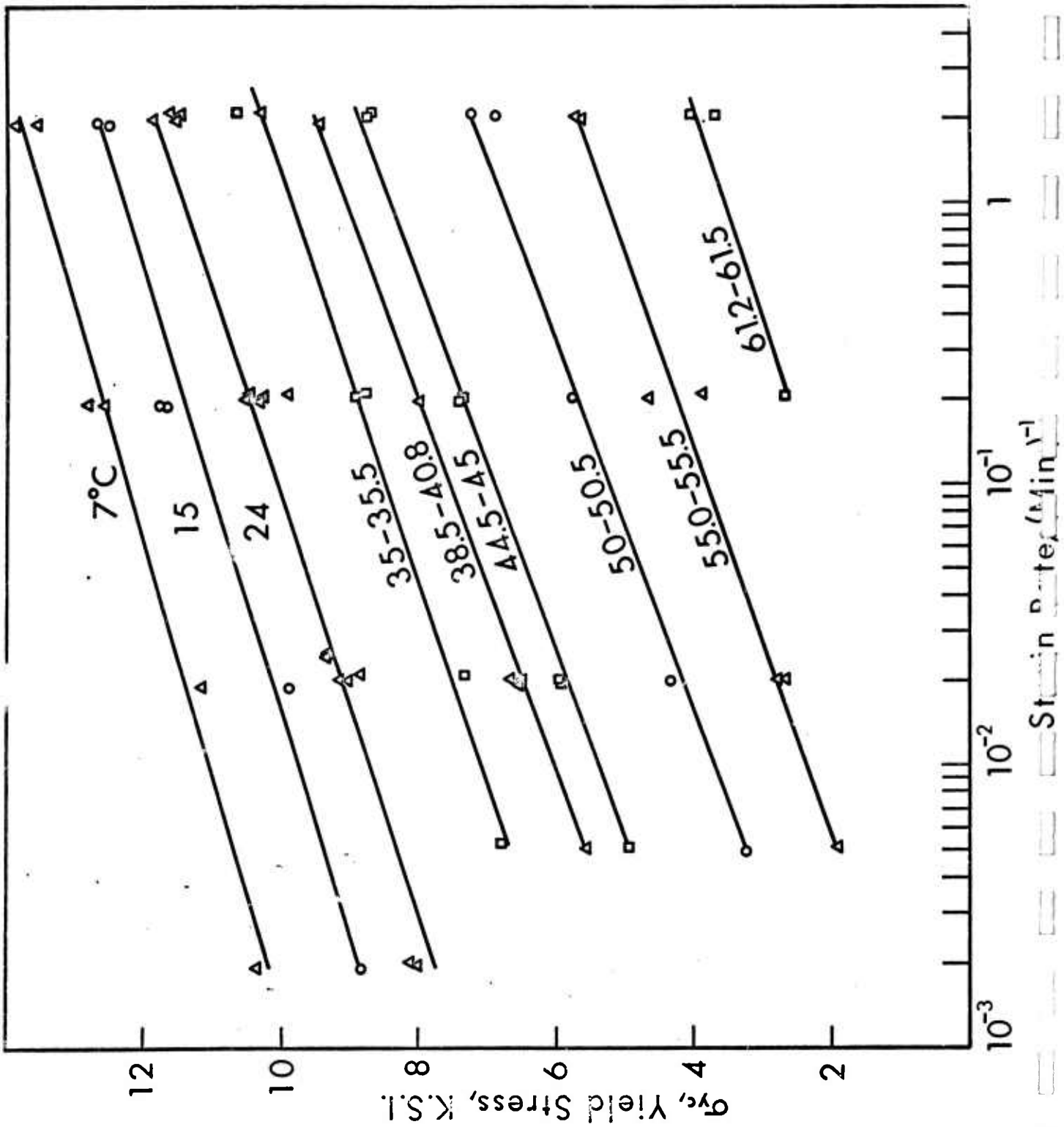


Figure 5 – Flexural Yield Stress (Simple Beam Theory) Vs. Strain Rate

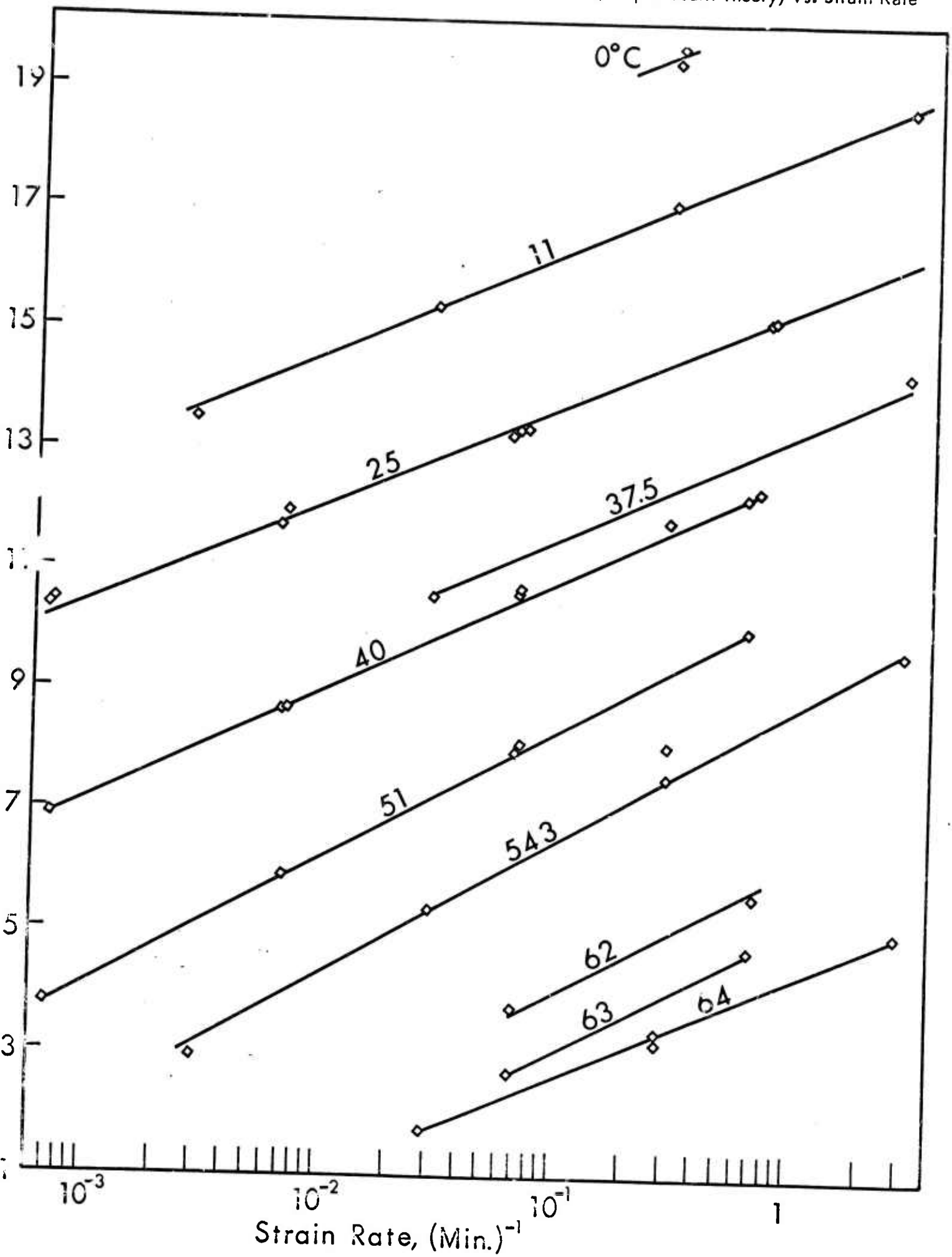
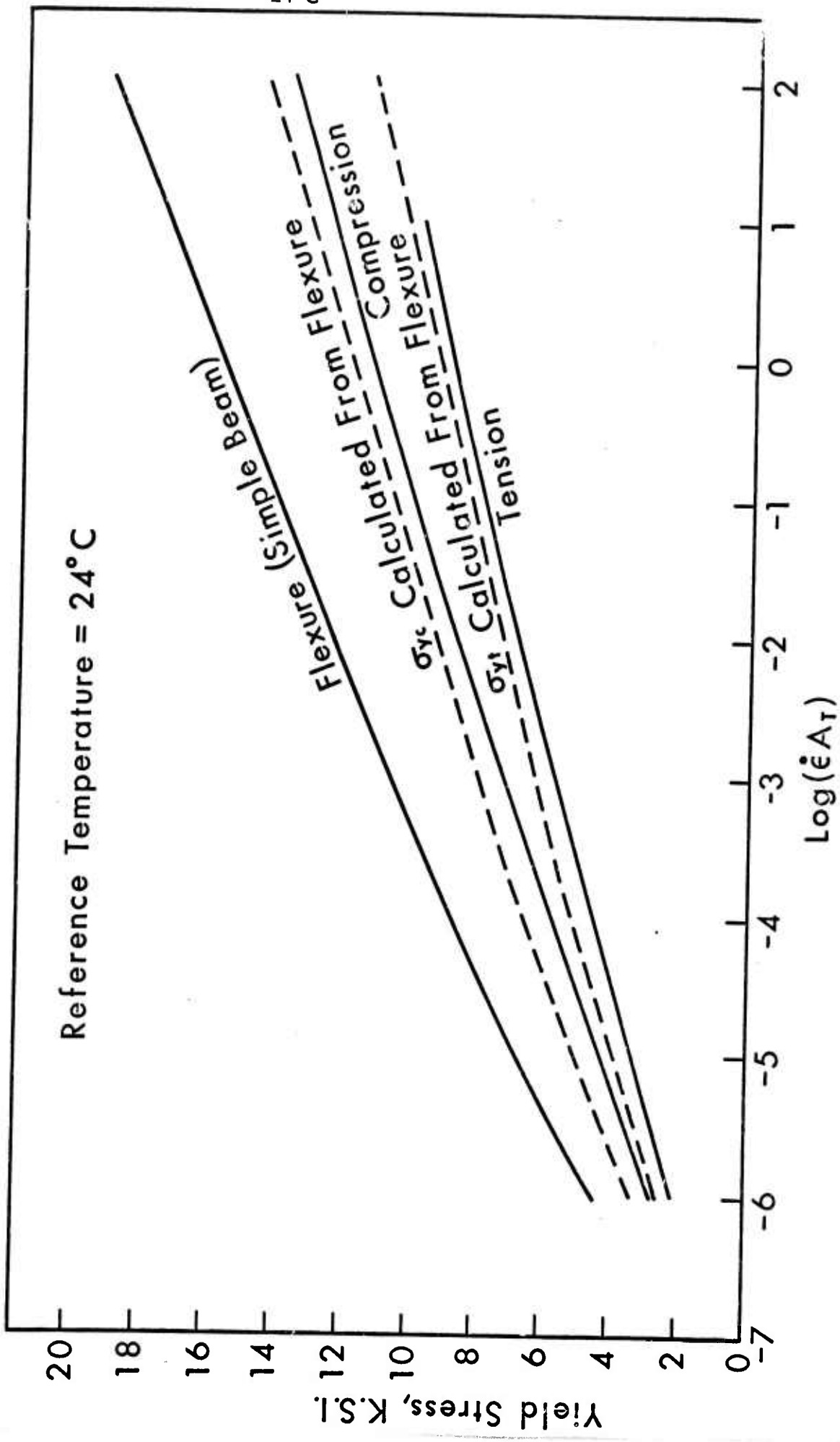


Figure 6 - Master Plot of Yield Stress Vs. Shifted Strain Rate, Reference Temperature = 24°C



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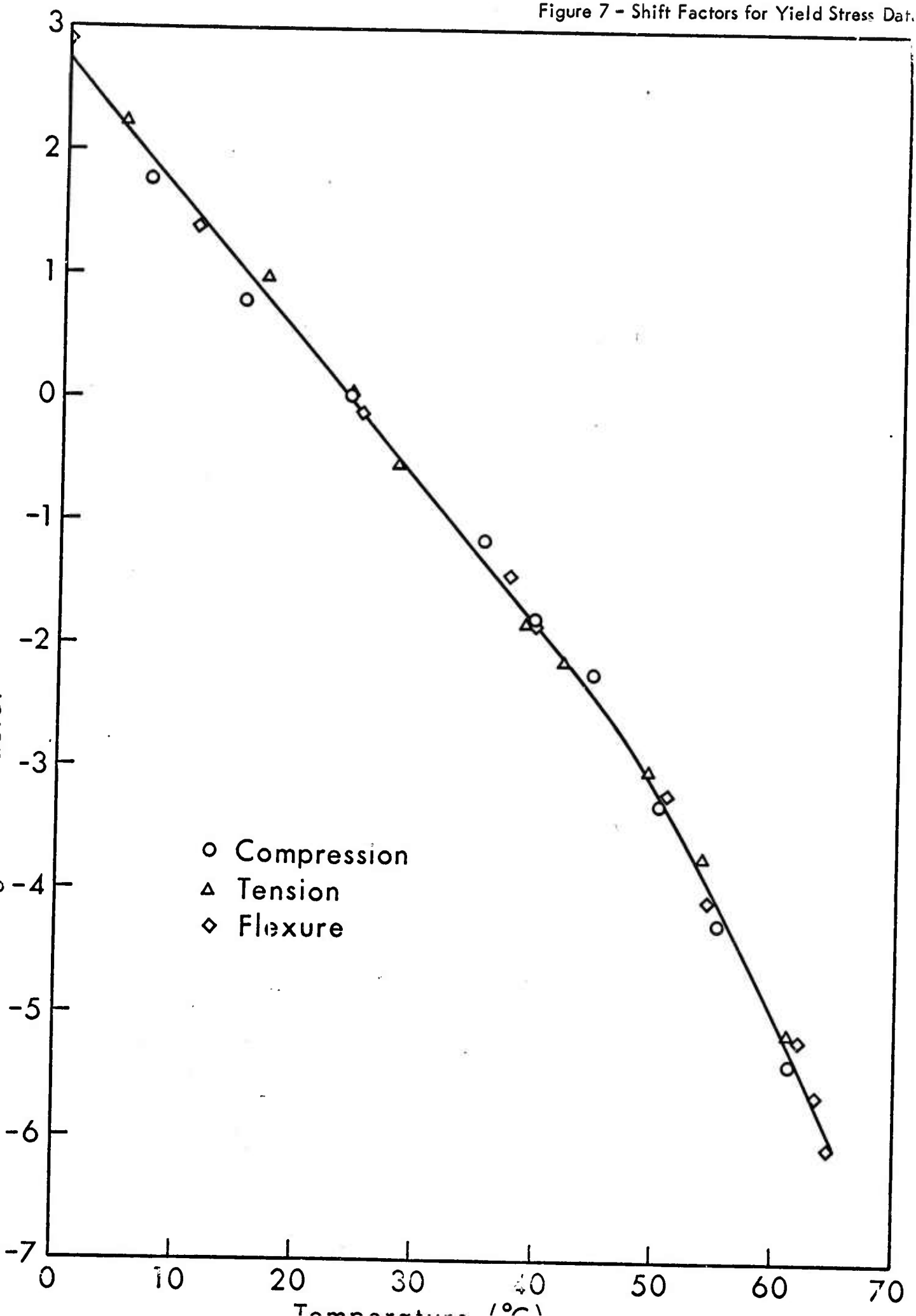

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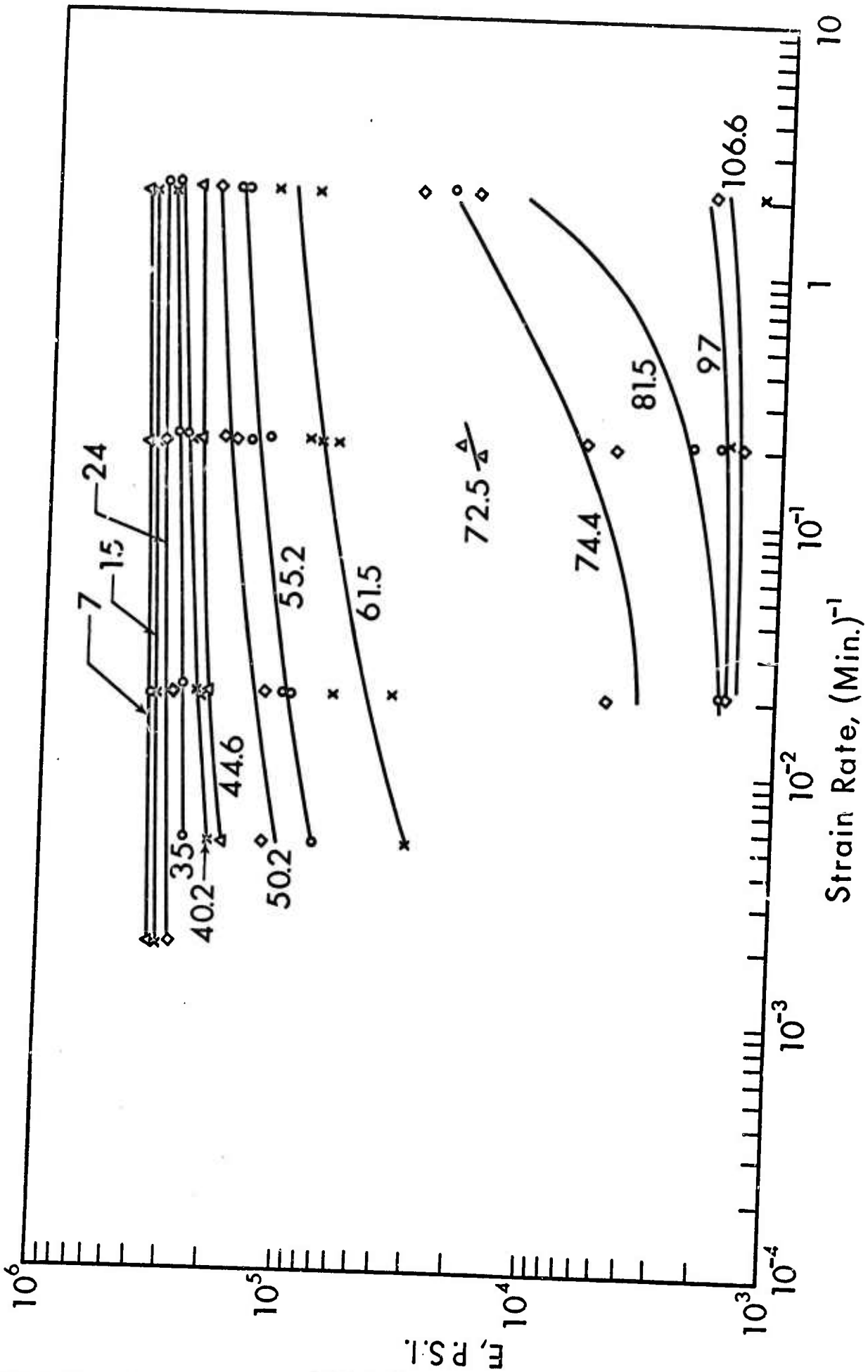


Figure 9 - Compression Modulus Vs. Temperature

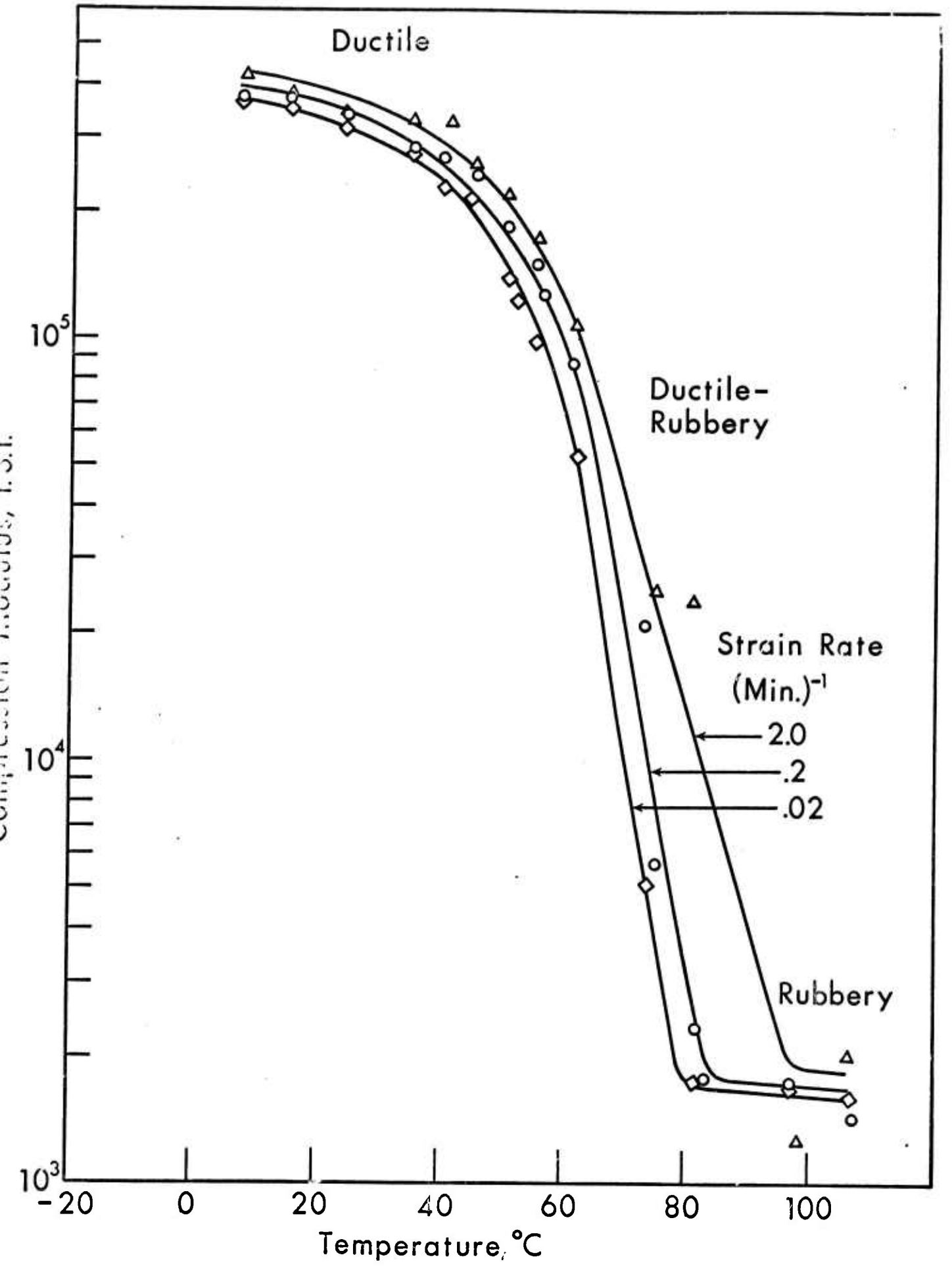


Figure 10 - Initial Modulus Vs. Temperatu

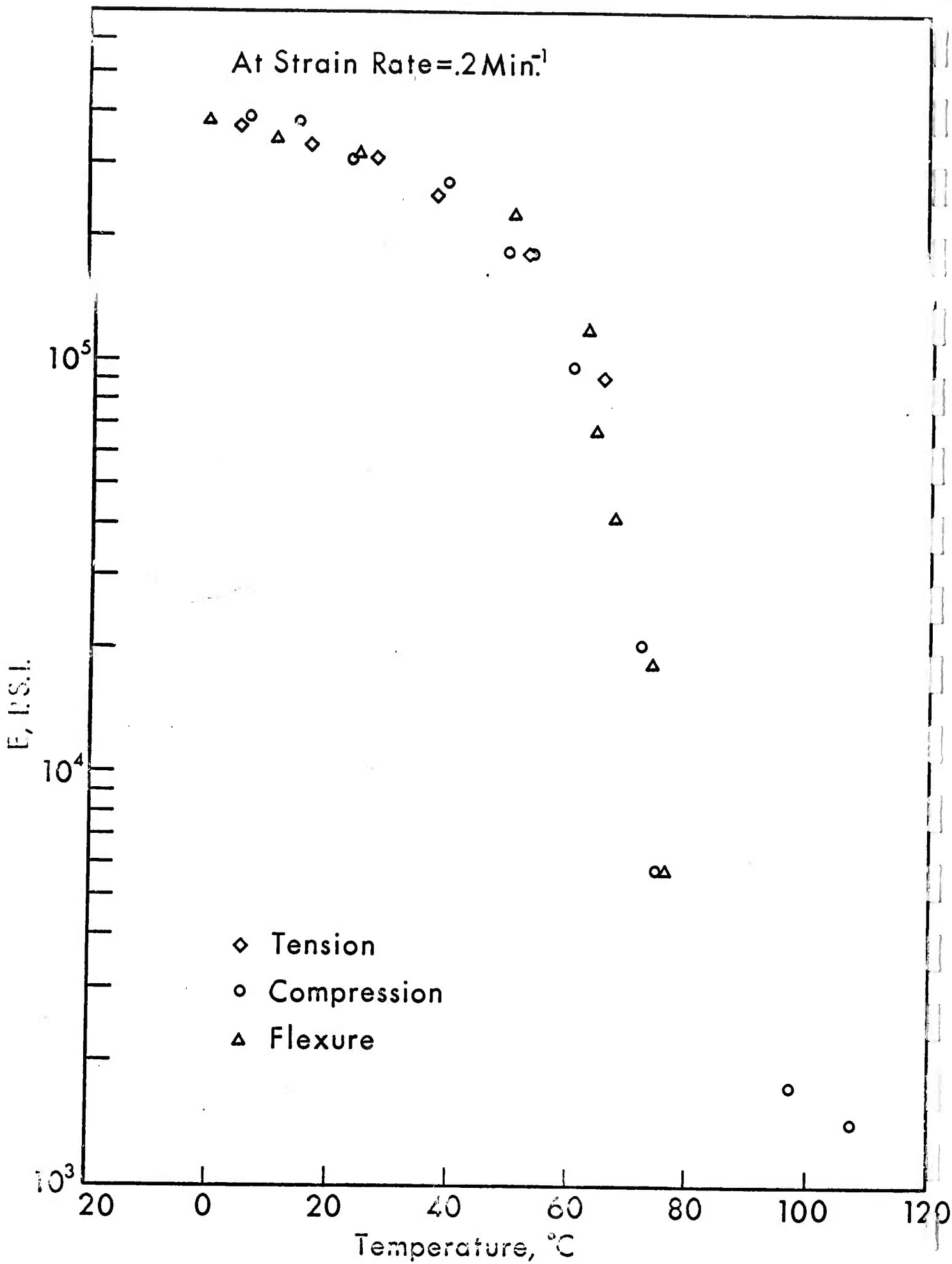


Figure 11 - Initial Tangent Modulus Vs. Shifted Strain Rate Master Curve, Reference Temperature = 24°C

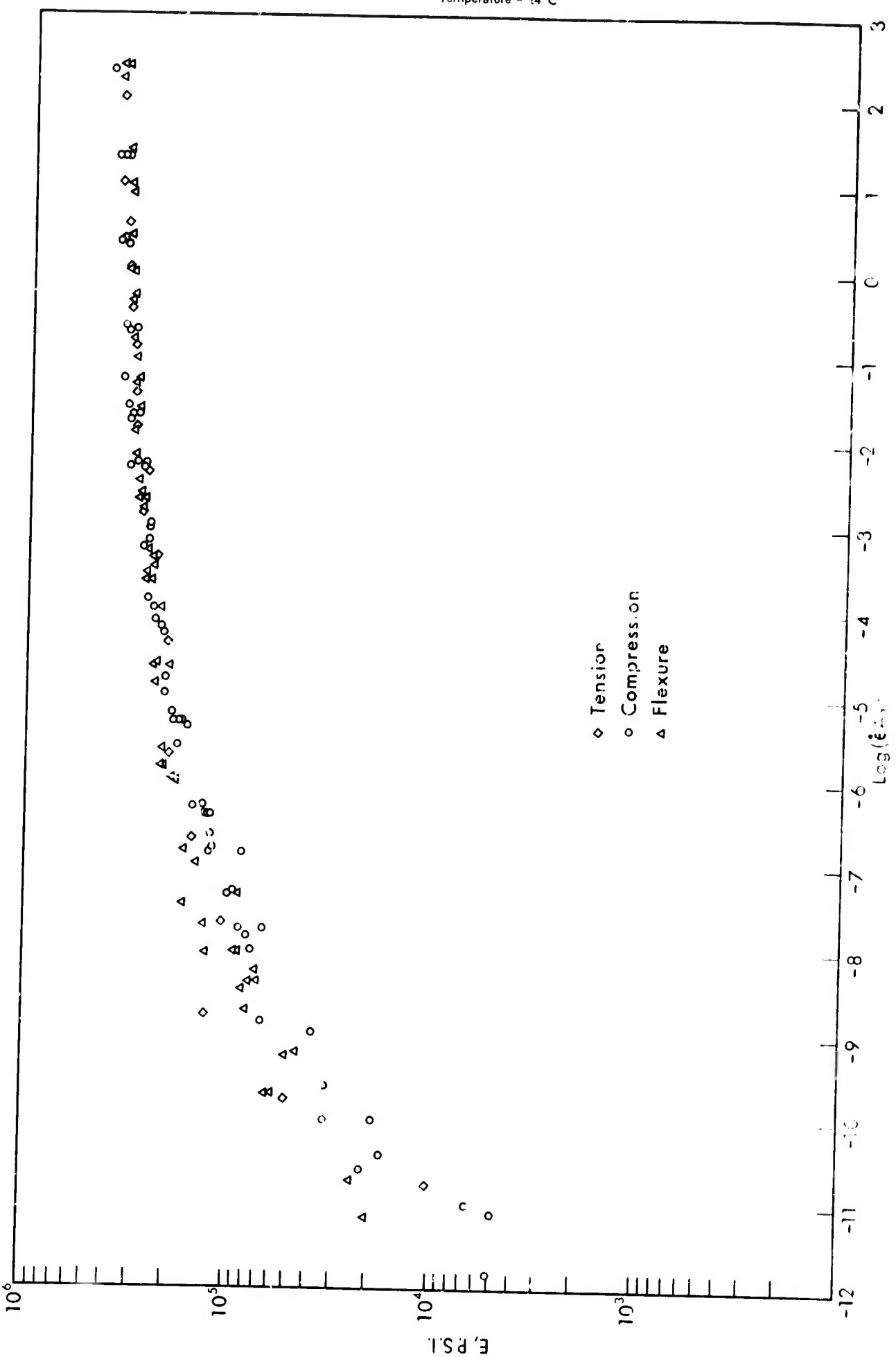


Figure 12 - Shift Factors for Initial Modulus Data

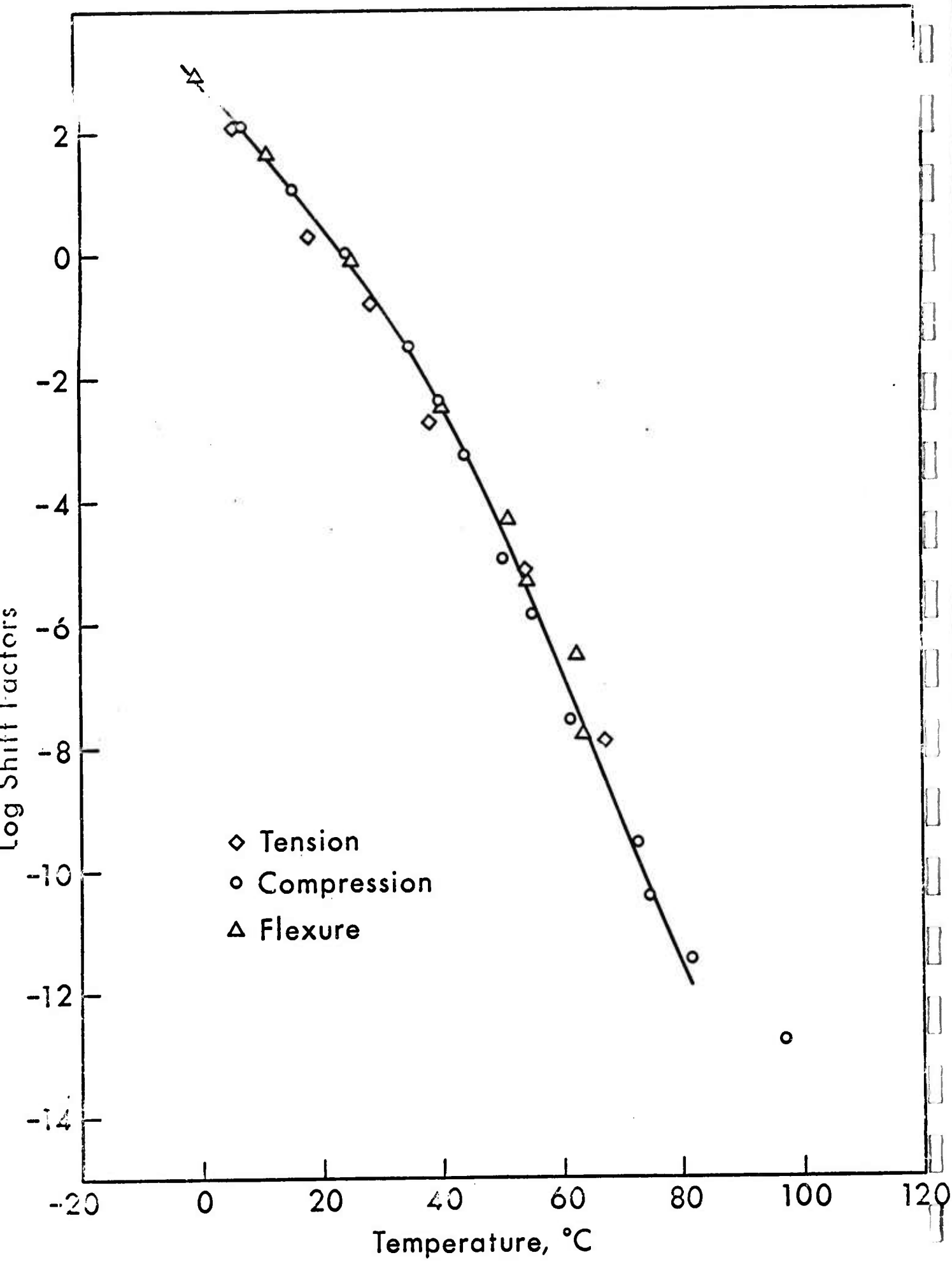


Figure 13 - Compression Stress Relaxation

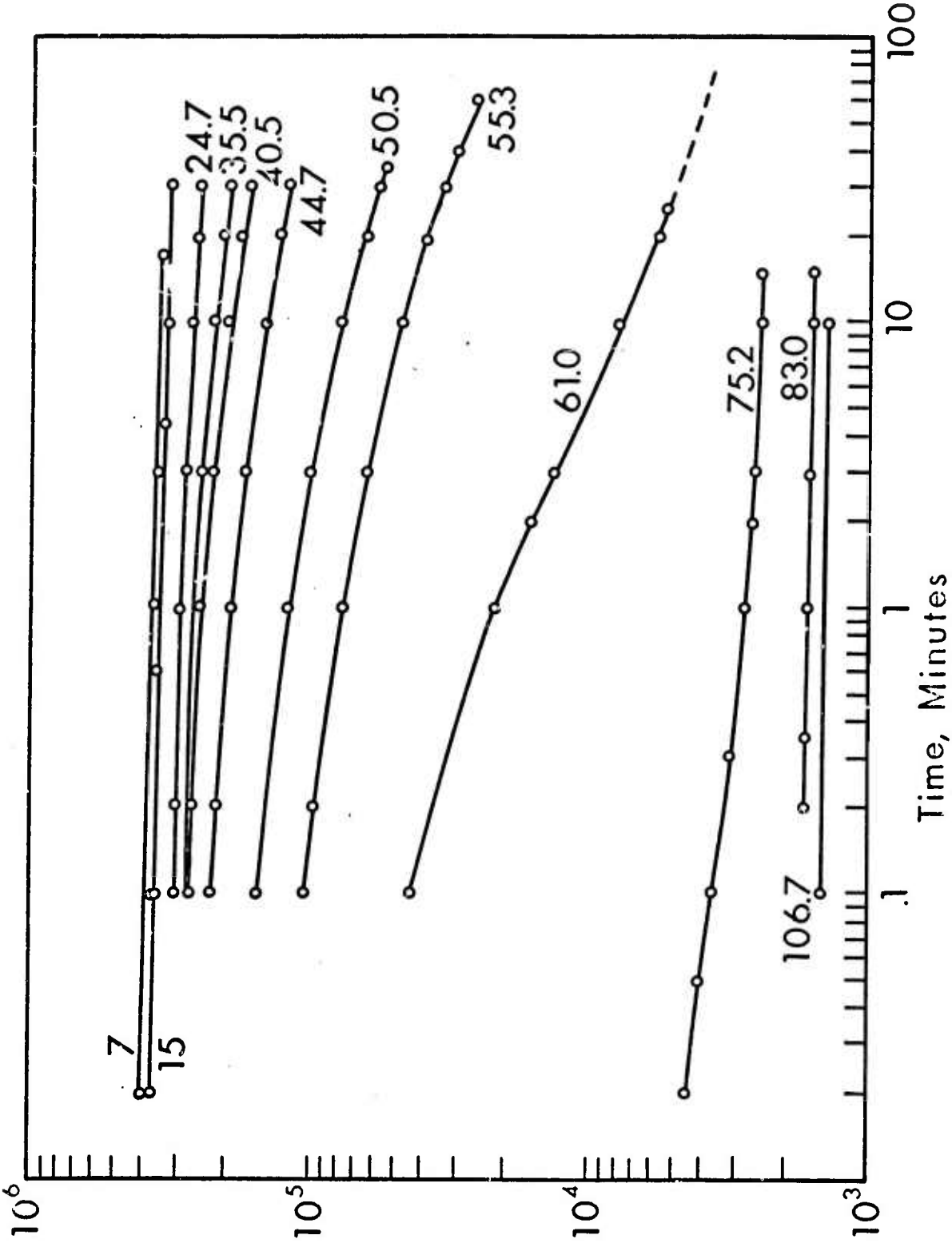


Figure 14 - Stress Relaxation Master Curve, Reference Temperature = 24°C

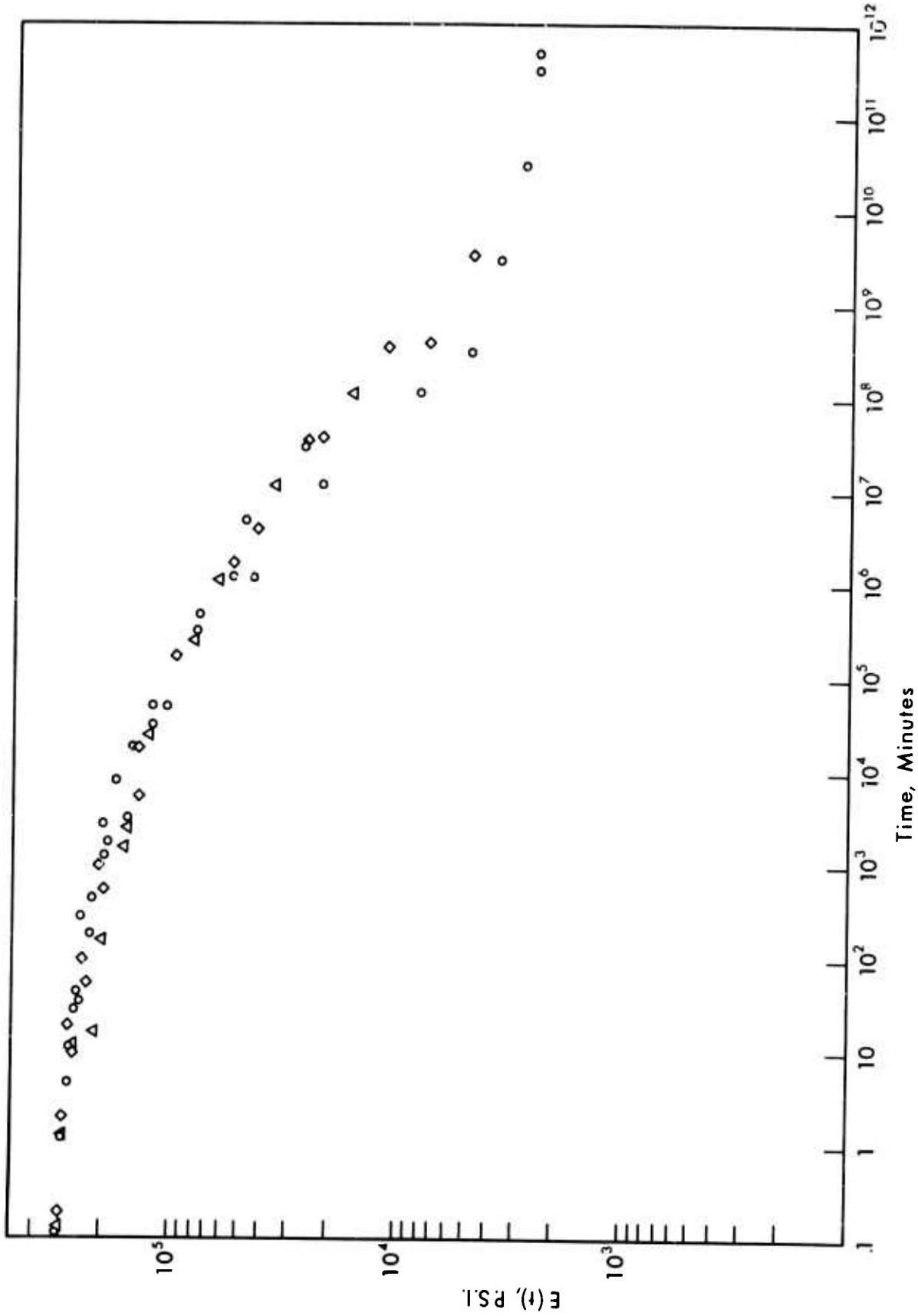
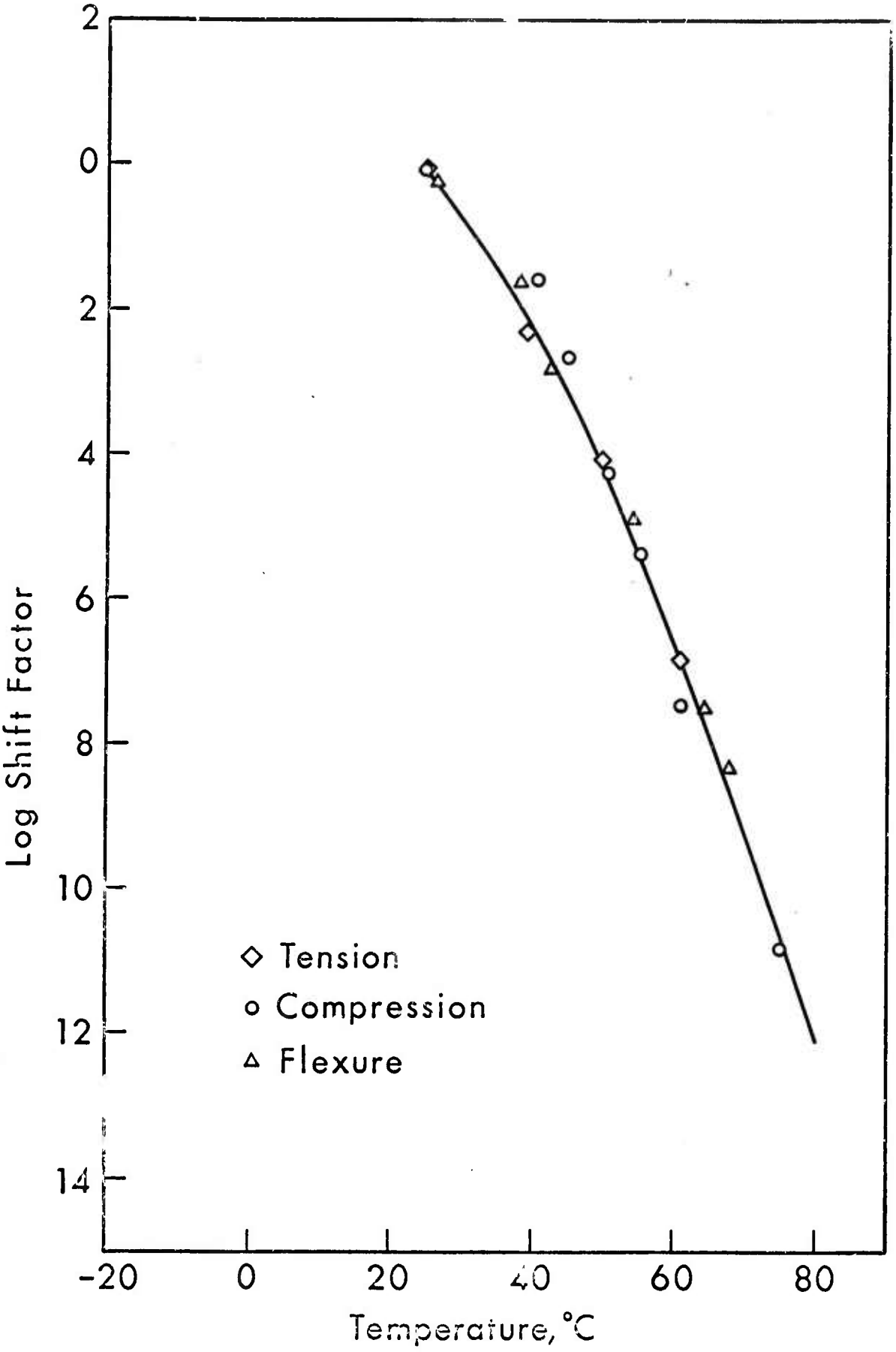


Figure 15 - Shift Factors for Stress Relaxation



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11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

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13. ABSTRACT

Epoxy-Versamid specimens were loaded in tension, compression, and flexure at different strain rates and temperatures to determine mode of failure, yield stress and strain, and tangent and relaxation moduli. Stress-strain curves were used to define brittle, ductile, ductile-rubbery, and rubbery modes of behavior which prevailed in different temperature-strain rate regions. The time-temperature superposition principle was applied to yield stress, initial tangent moduli, and relaxation moduli data for all three types of loading.

The transition regions, tangent and relaxation moduli, and shift factors were the same in tension, compression, and flexure. Thus the most convenient mode of loading can be used to determine the general time-temperature dependence. The ratio of compressive to tensile yield stress was almost constant over the entire ductile region. Flexural yielding data were used to predict yield stress in tension and compression, and stress relaxation master curves were shown to be related to elastic modulus versus strain rate curves. The yielding phenomenon was interpreted using Eyring's theory of non-Newtonian viscoplastic flow. The apparent activation energy and activation volume were larger for tension than compression. A theory is offered to explain why yielding can occur in a cross-linked system.

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Security Classification

14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
mechanical properties						
moduli						
yield strength						
stress strain behavior						
stress relation						
time-temperature superposition						
epoxy resins						
shift factors						

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