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CHARACTERIZATION OF ELASTOMERIC MATERIALS WITH APPLICATION TO DESIGN

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ABSTRACT

Redesign of the Space Shuttle Solid Rocket Booster has necessitated re-evaluation of the material used in the field joint O-ring seals. This research project was established to determine the viscoelastic characteristics of five candidate materials. The five materials are: two fluorocarbon compounds, two nitrile compounds, and a silicon compound. The materials were tested in a uniaxial compression test to determine the characteristic relaxation functions. These tests were performed at five different temperatures. A master material curve was developed for each material from the experimental data. The results of this study are compared to tensile relaxation tests. Application of these results to the design analysis is discussed in detail.

ACKNOWLEDGEMENTS

I wish to thank Dr. Mike Freeman, Mr. Frank Ledbetter, and Dr. Jerry Patterson for the opportunity to return to Marshall for this second summer in the NASA/ASEE Summer Faculty Fellowship Program. I have benefitted from the opportunity and I look forward to a long and fruitful relationship with each of you.

My special thanks goes to Mr. Frank Ledbetter for his assistance in the conduct of this research. He contributed many ideas which were essential to the success of this project. I greatly appreciate the time he took from his work to assist me throughout the summer. In this fateful summer, we survived a lot together. Thank you, Frank.

In addition, I want to thank Dr. Robert Landel of the Jet Propulsion Laboratory for his inspiration of this research project.

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INTRODUCTION

The accident involving the Space Shuttle Challenger on mission 51-L has generated a tremendous effort to evaluate the cause of the failure and to develop means for preventing a reoccurrence. The evaluation of the failure is essentially complete. The results of that evaluation [1] have indicated that the primary cause of the accident was the failure of Solid Rocket Booster (SRB) field joints to completely seal. This allowed exhaust gases to escape thru the side of the booster and led eventually to the rupture of the External Tank (ET). The "Report of the Presidential Commission on the Space Shuttle Challenger Accident" [1] goes into considerable detail in reviewing problems associated with the SRB field joint. These joints are now the primary focus of the SRB redesign effort.

The SRB field joint is sealed by use of a set of O-rings, putty, and grease. The set of O-rings are the primary seals, with the putty and grease acting to protect and support the O-rings. In general, all O-rings are made of elastomeric, or rubber like, materials. The O-rings in the field joint are no exception. They are made of a fluorocarbon based rubber. The behavior of elastomers is, in general, nonlinear, viscoelastic, and temperature dependent. The temperature dependent behavior of the elastomer used in the field joint O-rings has been cited as a contributing factor in the accident. An aspect of the SRB redesign effort is the evaluation and selection of a material for the O-ring. As part of this process studies are being performed on several candidate elastomeric materials. Some of these studies are directed toward assessing the temperature dependent behavior of the materials.

The goal of this study is to provide the redesign process additional information with which to select a material to be used in the field joint O-rings. This study encompasses testing which will produce information about the time and temperature behavior of the candidate materials.

OBJECTIVES

The objectives of this study are:

1. Characterize the viscoelastic behavior in compression of five elastomers which are candidate materials for the field joint O-rings.
    - 1.1 The five materials to be tested are: V747-75, a modified fluorocarbon rubber (This is the baseline material currently in use.); V835-75, a modified fluorocarbon rubber; N602-70, a nitrile compound; Arctic Nitrile, a nitrile compound; and S650-70, a silicon compound.
    - 1.2 Develop the methodology for determining the viscoelastic response of the elastomers in compression.
2. Review the methodology to be used in the design of systems which incorporate viscoelastic materials.
    - 2.1 Discuss the constitutive equations to be used.
    - 2.2 Discuss the methodology to be used in the evaluation of the constitutive equation under simple loading conditions.
    - 2.3 Discuss application of the results from the first objective to the design process.

METHODOLOGY

The majority of the test methods available for the characterization of the viscoelastic behavior of materials are based on either a uniaxial tension or shear deformation. The deformation state in an O-ring is very complicated. There are tensile stresses along the axis of the O-ring, shear stresses applied in several directions on the surface, and compressive stresses acting through the thickness. For the purposes of design analysis it may be sufficient to assume that the magnitude of the compressive stress dominates the other stresses. Therefore, the applied stresses can be modeled as uniaxial compression. This then necessitates knowing the behavior of the material when loaded in compression.

The Rheometrics Dynamic Spectrometer (RDS) has the capability of performing a large number of different tests for the characterization of viscoelastic materials. Some of these tests can be used to determine the dynamic, or complex, moduli of a viscoelastic solid or fluid. Other tests can be performed to determine the stress relaxation modulus of a viscoelastic solid or fluid. In this study, the primary objective is to determine the compressive relaxation behavior of the candidate materials at various temperatures. While the RDS has the capability of operating in compression, the system and data acquisition program do not have a dedicated uniaxial compression test fixture or procedure. For these tests the parallel plate test fixture was adopted for use as the uniaxial compression fixture. The uniaxial tension mode of the RDS was used for data acquisition and reduction.

The tests were performed over a range of temperatures. This was done for two reasons. First, for the design process it was desired to have the stiffness, or modulus, characterized over a range of temperatures from -20°F (-9.4°C) to 140°F (50.9°C). Second, an objective of the characterization process was to develop a so-called master stress relaxation curve. This requires knowledge of the relaxation behavior at various temperatures.

For each of the materials, five samples were tested at five different temperatures. The five temperatures used are -20, 0, 20, 40, and 60°C. For V747-75 the lowest temperature used was -10°C instead of -20°C. This change was made because at a temperature of -20°C the material was so stiff that a one percent strain caused the load transducer to overload.

In these tests the specimens where taken from O-ring stock. Lengths of the O-ring stock were cut, and then the lengths were then machined by turning on a lathe. This operation was performed to produce specimens which had flat ends and a regular, or square, geometry, such that the ends were perpendicular to the axis of the cylinder. The nominal diameter of the specimens is 7.14 mm. The lengths varied from 6.5 mm to 8.9 mm.

The geometry of the specimen was selected for several reasons. First, a sample with a square or rectangular cross-section develops stress concentrations at the corners when loaded by large strains. Samples with circular cross-sections do not have this problem. Second, O-ring stock with a circular cross-section was available. Third, the machining of a circular shape from flat stock is difficult at best, with relatively large dimensional flaws. Fourth, the length of the sample needs to be of the same order of magnitude as the diameter, or larger, to reduce end effects.

The procedure for the tests is as follows:

1. Soak the test specimens at the test temperature. The length of the the soak at each temperature was a minimum of one hour.

2. Measure the dimensions of the specimen prior to the test.

3. Place the specimen at the center of the lower parallel plate.

4. Lower the upper plate onto the specimen. The position of the upper plate was adjusted to the point where there was no load (zero dynes) registered by the load cell.

5. Apply a step strain in compression of magnitude one percent for a period of 1000 seconds. Set the four sampling zones of the RDS at 1, 9, 90, and 900 seconds.

6. Process the load - displacement information obtained from the test with the RDS "RECAP2" data acquisition program.

After the tests were completed the data was reviewed and an average stress relaxation function was computed from the five samples at each of the five temperatures. From these average stress relaxation curves a master stress relaxation curve was constructed. The procedure used in constructing the master curve is as follows:

1. Compute the logarithm of the modulus and the corresponding time for each of the temperatures, i.e. $\log(E(T_i,t_j))$ and $\log(t_j)$, where E is the compressive stress relaxation modulus of the material, T_i is the test temperature, and t_j is the time. The values of T_i are -20 (-10 for V747-75), 0, 20, 40, and 60°C for i of 1 to 5, respectively.

2. Compare the values of $\log(E(T_i,t_j))$ with those of $\log(E(T_k,t_l))$ to determine the time t_l at which

   $$\log(E(T_i,t_j)) = \log(E(T_k,t_l)),$$

   and

   $$\frac{\partial \log(E(T_i,t_j)}{\partial t_j} = \frac{\partial \log(E(T_k,t_l)}{\partial t_l}.$$

3. Compute the time shift factor, a_T, between temperature T_i and T_k from the expression

   $$a_T = \log(t_l) - \log(t_j),$$

   where $T_i \le T_k$. This is described in [2] and [3].

4. Compute the time shift factors, a_{T1}, a_{T2}, a_{T4}, and a_{T5}, relative to the reference temperature T_3. Therefore, a_{T3} is 0.

5. Shift the time scales at each of the temperatures such that

   $$\bar{t}_j = 10^{(\log(t_j) - a_{Ti})},$$

   where $\bar{t}_j$ is the time of the relaxation value as at temperature T_i as shifted to the reference temperature, T_3.

References [2] and [3] suggest that the master stress relaxation curve should be referenced to the glass transition temperature. This information was not available at the time of the study. Thus, an arbitrary reference temperature (near room or standard temperature) was selected. When the glass transition temperature values become available the above procedure can be used to develop the master curves at that temperature.

RESULTS

The results from the material characterization tests are presented in Figures VIII-1 through VIII-10 and Tables VIII-1 and VIII-2. These results are discussed in this section.

In all figures the stress relaxation modulus, E(t), is presented in pounds per square inch, psi, and time is presented in seconds. The horizontal axis in all figures is the time axis and has a logarithmic scale. The vertical axis in all figures is the stress relaxation value. In the presentation of the stress relaxation data, Figures VIII-1, VIII-3, VIII-5, VIII-7, and VIII-9, the scale for the stress relaxation data is linear. In the figures which show the master stress relaxation curves, VIII-2, VIII-4, VIII-6, VIII-8, and VIII-10, the stress relaxation modulus is displayed on a logarithmic scale.

Figure VIII-1 shows a plot of the stress relaxation data for the baseline material, V747-75, as taken at temperatures of -10, 0, 20 and 60°C. The curves indicate the relatively strong temperature dependence of the material at temperatures at and below 0°C, while it is relatively insensitive to temperature effects above 0°C. Figure VIII-2 shows a plot of the master stress relaxation data for V747-75 at a reference temperature of 20°C, as assembled from the four relaxation curves shown in Figure VIII-1. The assembled master curve at this temperature extends from times of approximately 10^{-5} seconds to over 10^{6} seconds. Although the slope of the master curve is not zero at the maximum time, the rate of decay is less than 10 psi per 10^{3} seconds.

Comparison of these results to results obtained by Morton Thiokol [4] for tensile stress relaxation of the V747-75 material indicates significant differences. With a two percent tensile strain and a temperature of 75°F their results find a relaxation modulus of approximately 4000 psi at a time of 10^{-6} seconds, 1780 psi at 10^{-3} seconds, 1420 psi at 1.0 seconds, and 1070 psi at 10^{3} seconds. The results from these tests (one percent compressive strain) at 68°F are: a modulus of approximately 6000 psi at 10^{-5} seconds, 900 psi at 10^{-3} seconds, 200 psi at 1.0 seconds, and 170 psi at 10^{3} seconds. These differences indicate that these tests found the behavior of V747-75 to be more strongly viscoelastic in nature. The differences in magnitude may be due to a nonlinear strain dependence of the material. However, one would expect that the material would have nearly linear behavior on the region of small strains, particularly between +2 and -2 percent strain.

A plot of the stress relaxation data obtained for the modified fluorocarbon V835-75, as taken at temperatures of 20, 40 and 60°C, are shown in Figure VIII-3. Figure VIII-4 presents a plot of the master stress relaxation data for V835-75 at a reference temperature of 20°C. As indicated above, this curve is assembled from the four relaxation curves shown in Figure VIII-3.

The stress relaxation data for N602-70 is plotted in Figure VIII-5. The data is shown for temperatures of -20, 20, 40 and 60°C. The master stress relaxation curve for N602-70 at a reference temperature of 20°C is presented in Figure VIII-6. This figure shows that this nitrile compound has little change in behavior with temperature. As a result of the relative insensitivity of the material to temperature effects the maximum time obtained on the master stress relaxation curve is less than 10^6 seconds. However, it should be noted that the rate of decay at the maximum time obtained is less than 5 psi per 10^3 seconds.

Figure VIII-7 shows a plot of the stress relaxation data obtained for Arctic Nitrile at temperatures of 20, 40, and 60°C. Figure VIII-8 presents a plot of the master stress relaxation curve for Arctic Nitrile at a reference temperature of 20°C. This curve indicates that this material is relatively temperature sensitive. Further, the curve shows that the material has not reached an equilibrium modulus at 10^9 seconds. However, the rate of decay is very small (less than 10 psi per 10^8 seconds).

The stress relaxation data obtained for S650-70 at temperatures of -20, 20, 40, and 60°C are presented in Figure VIII-9. Observe that the curve shown for a temperature of 20°C has some anomalous data at approximately 0.8 seconds. No explanation is available. However, it is certain this anomaly is not indicative of the true material behavior. The data shown in Figure VIII-9 is assembled into the master stress relaxation curve for S650-70 at a reference temperature of 20°C. This figure shows that the material is relatively temperature sensitive. Note the need for test data at a temperature of 50°C to fill in the gap between the segments at 40 and 60°C. This figure also shows that the material is only slightly viscoelastic having a very small rate of decay (less than 10 psi per 10^{12} seconds).

The time shift factors obtained in the process of assembling the master stress relaxation curves by the method described in the previous section are presented in Table VIII-1. Values are listed for the five materials tested. This data is as to be expected from tests of this type with a_T monotonically increasing with increasing temperature. Insufficient results, in terms of the number of temperatures tested, are available to draw specific conclusions about the behavior of the time shift factor with temperature. For this reason, no function for a_T is suggested for any of the materials and only general comments about the temperature sensitivity are made.

Table VIII-2 lists the values of the maximum times obtained for the master stress relaxation curves of each of the materials. These values give an additional indicator of the temperature sensitivity of the material. Materials with a lower value of the maximum time tend to be less temperature sensitive. Similarly, large values of the maximum time tend to indicate a greater temperature sensitivity.

In general, the test methods developed for determining the compressive relaxation behavior of elastomers using the Rheometrics Dynamic Spectrometer worked well. The results obtained were within the realm of expected material behavior. Both fluorocarbon compounds and both nitrile compounds exhibited the same general time and temperature dependent behaviors. The methods are not without problems, however. The machining of the test samples needs improvement. Some of the samples were not square (i.e. the end surfaces were not perpendicular to the axis of the cylinder). This led to erroneous results from those samples. Some samples had a small nub, or knob, at the center of the end surface where the sample had been faced-off on the lathe. This also tended to cause erroneous results. These factors led to a very high standard deviation in the test results for some of the materials. Additional problems were encountered in the setting of the zero load state prior to the test. This procedure needs to be systematized in order to yield consistent results. These errors and equipment problems with the RDS caused the results from tests taken at some temperatures to be in error to such an extent that they were clearly not indicative of the true material behavior. Such problems were encountered for at least one temperature for each of the materials. Because of these problems, results from those temperatures are not presented. Those test results not reported are: V747-75 at 40°C, V835-75 at -20, and 0°C, N602-70 at 0°C, Arctic Nitrile at -20, and 40°C, and S650-70 at 0°C.

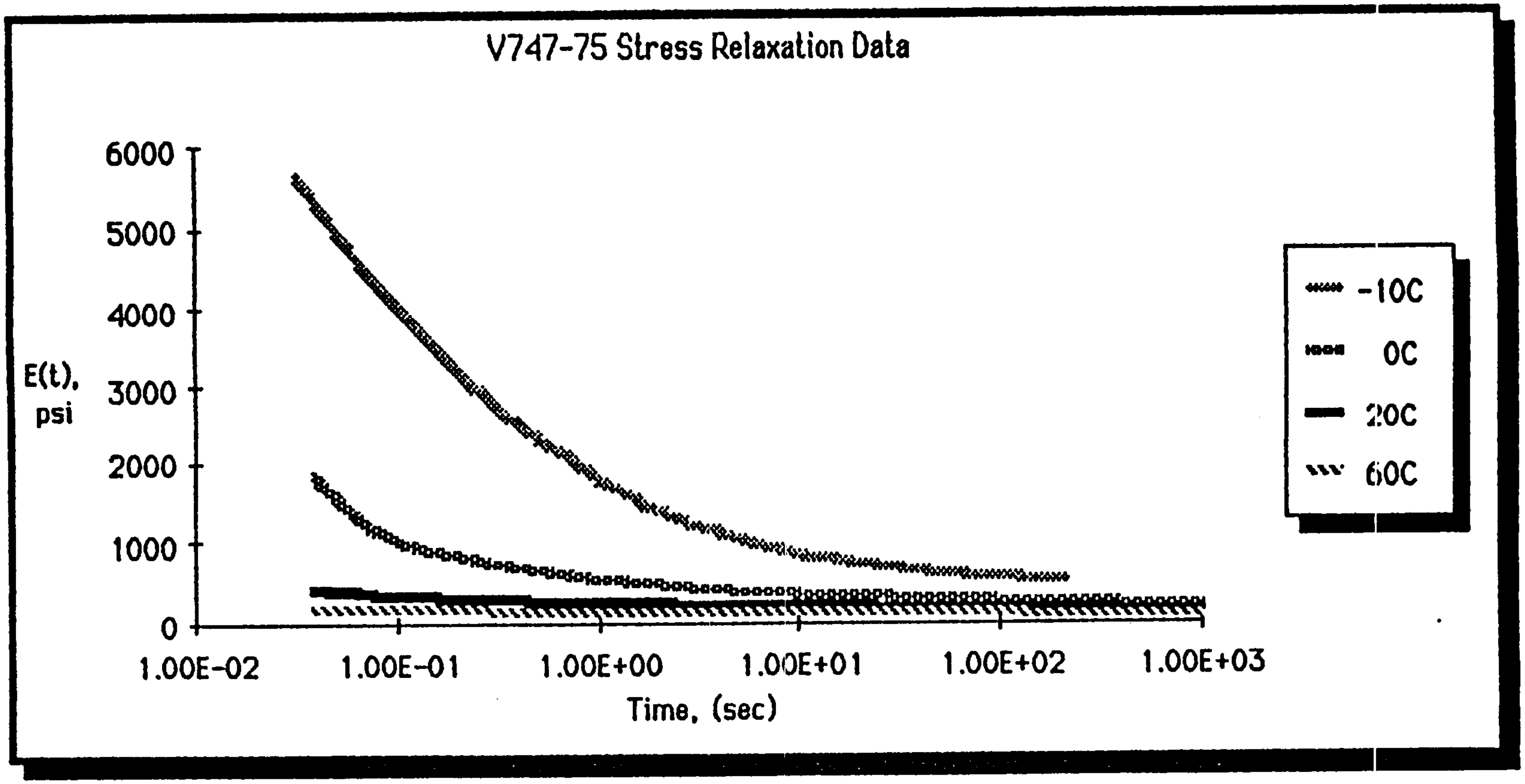

Figure VIII-1.
A Plot of the Stress Relaxation Data for V747-75
Taken at Temperatures of -10, 0, 20, and 60°C.

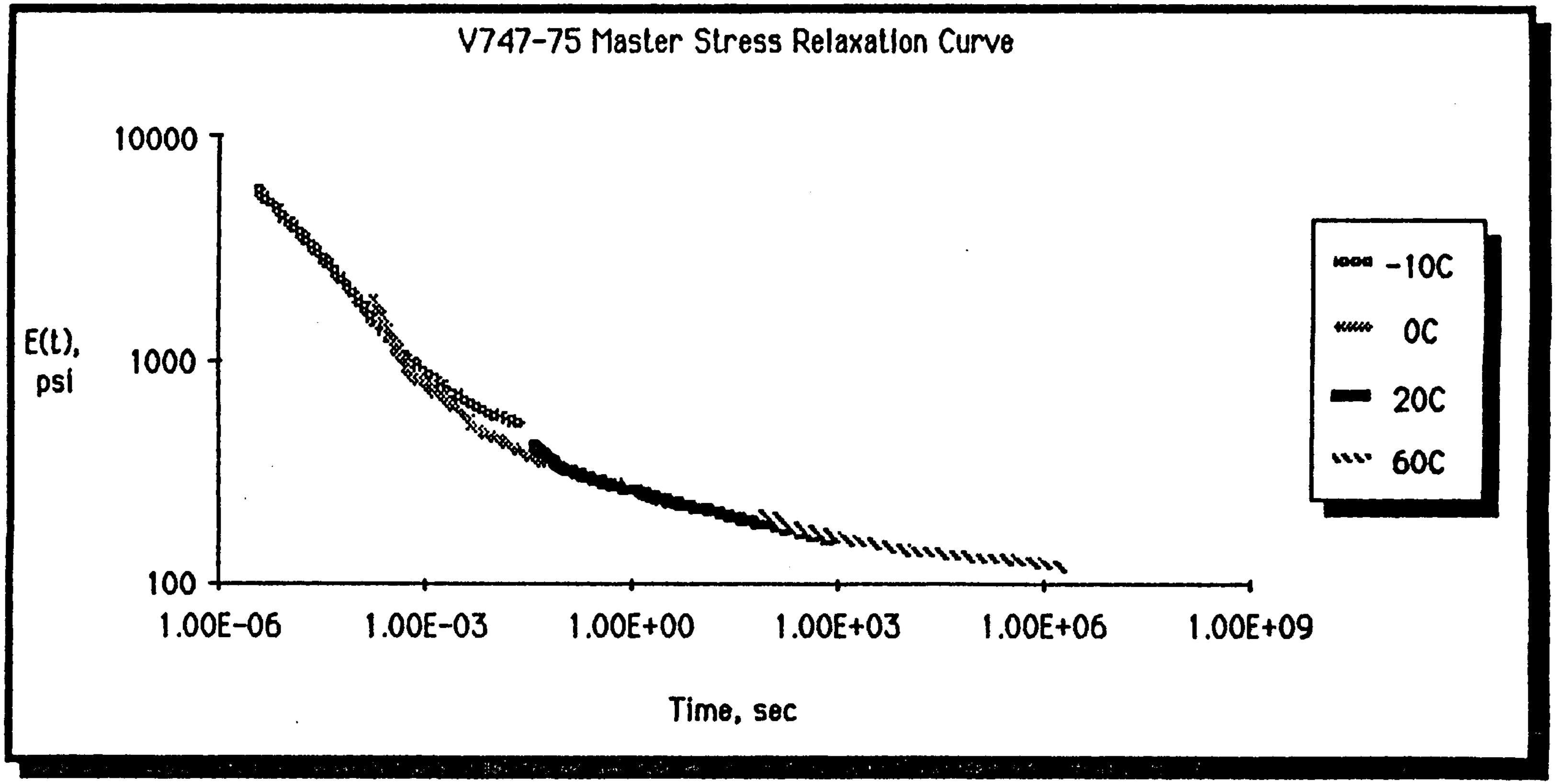

Figure VIII-2.
A Plot of the Master Stress Relaxation Curve for V747-75
at a Reference Temperature of 20°C.

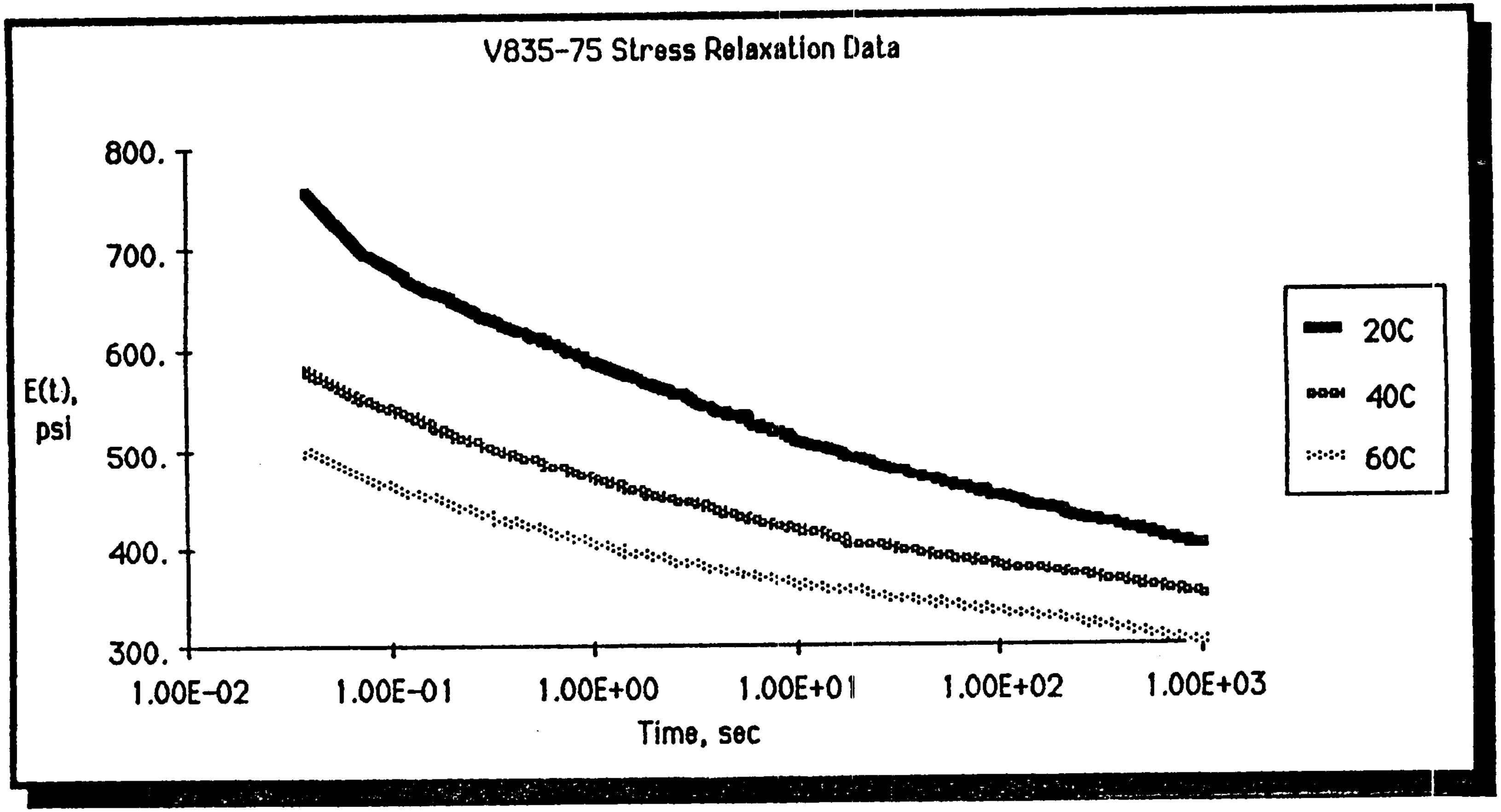

Figure VIII-3.
A Plot of the Stress Relaxation Data for V835-75
Taken at Temperatures of 20, 40, and 60°C.

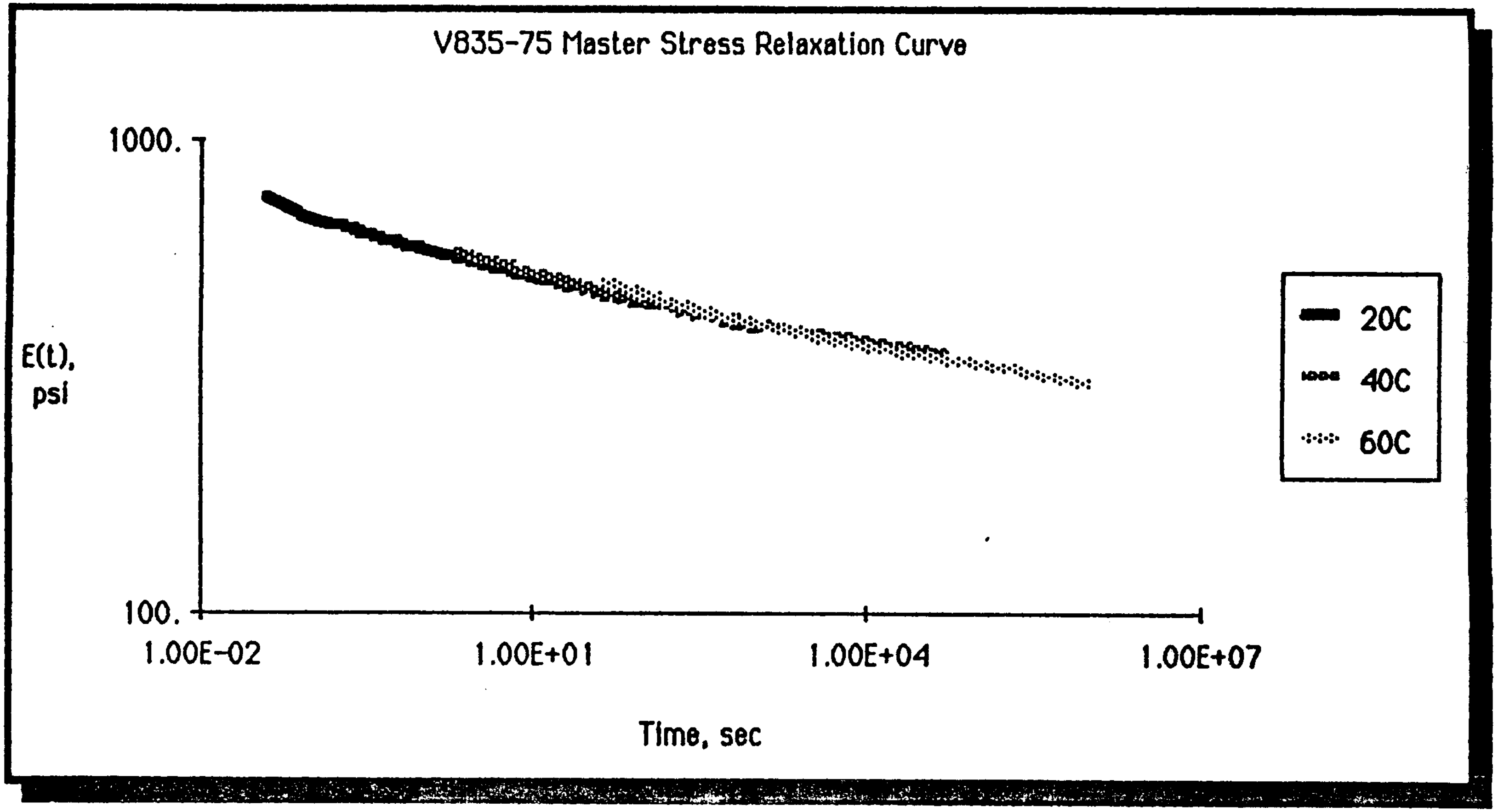

Figure VIII-4.
A Plot of the Master Stress Relaxation Curve for V835-75
at a Reference Temperature of 20°C.

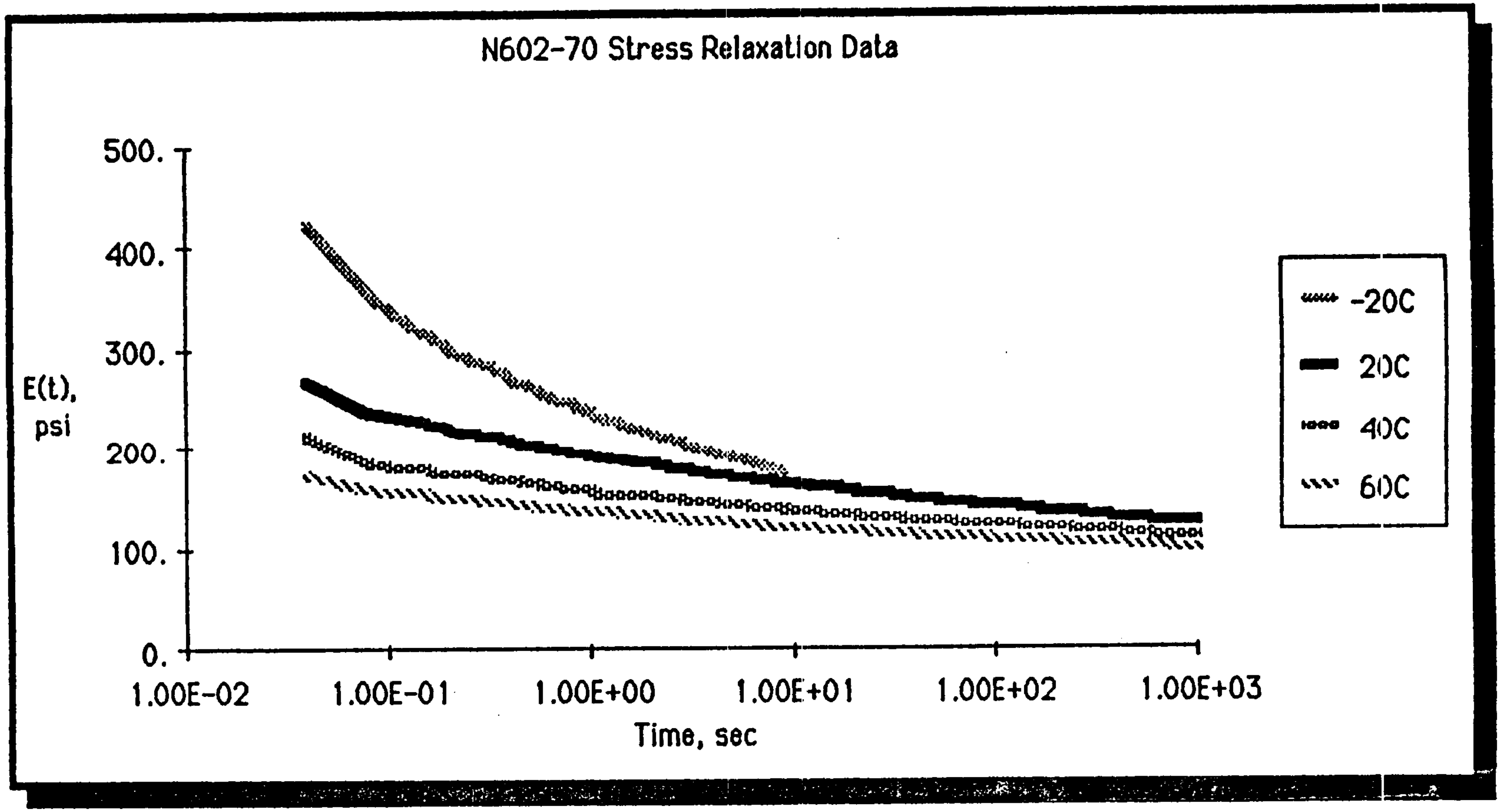

Figure VIII-5.
A Plot of the Stress Relaxation Data for N602-70
Taken at Temperatures of -20, 20, 40, and 60°C.

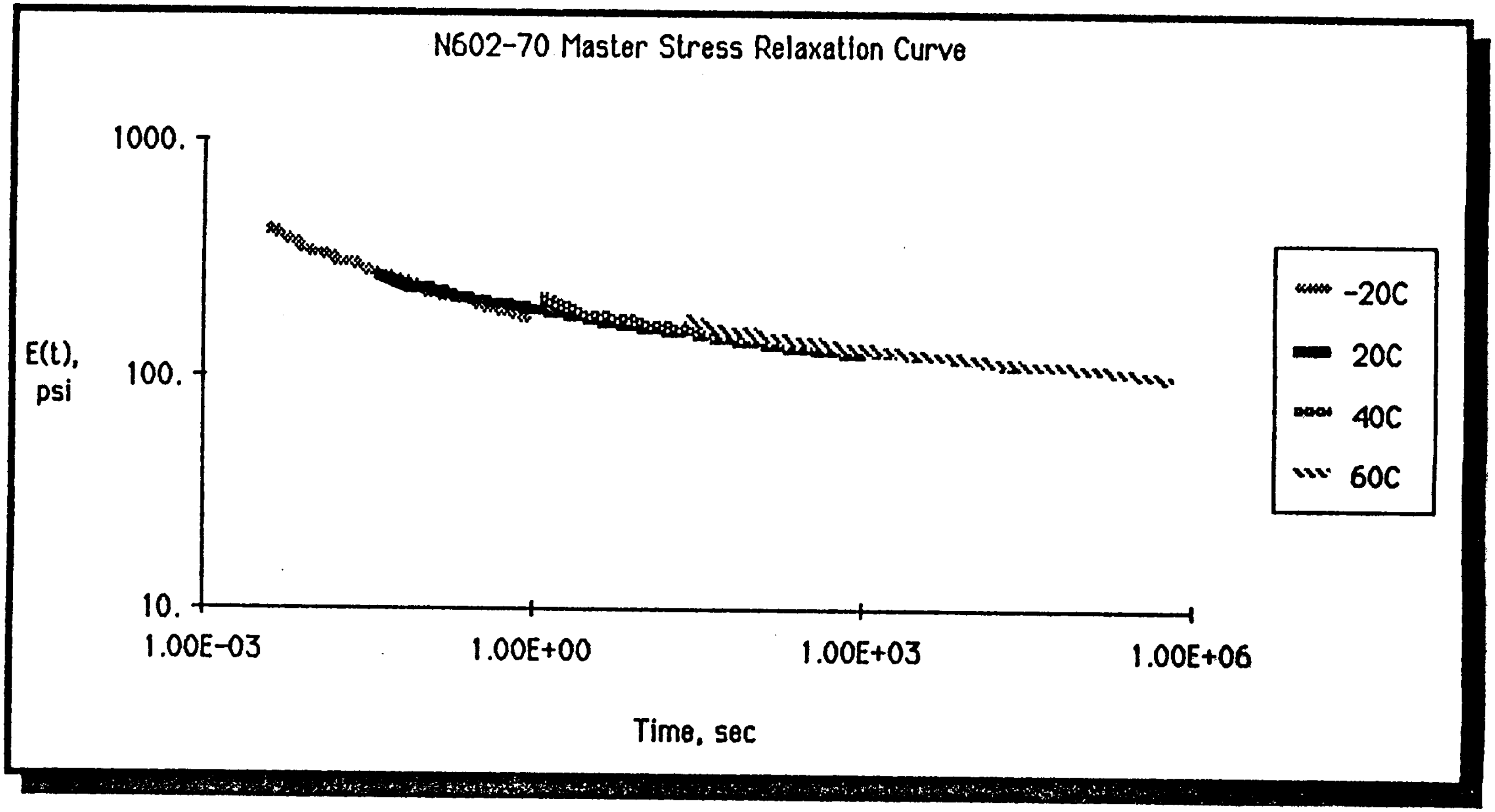

Figure VIII-6.
A Plot of the Master Stress Relaxation Curve for N602-70
at a Reference Temperature of 20°C.

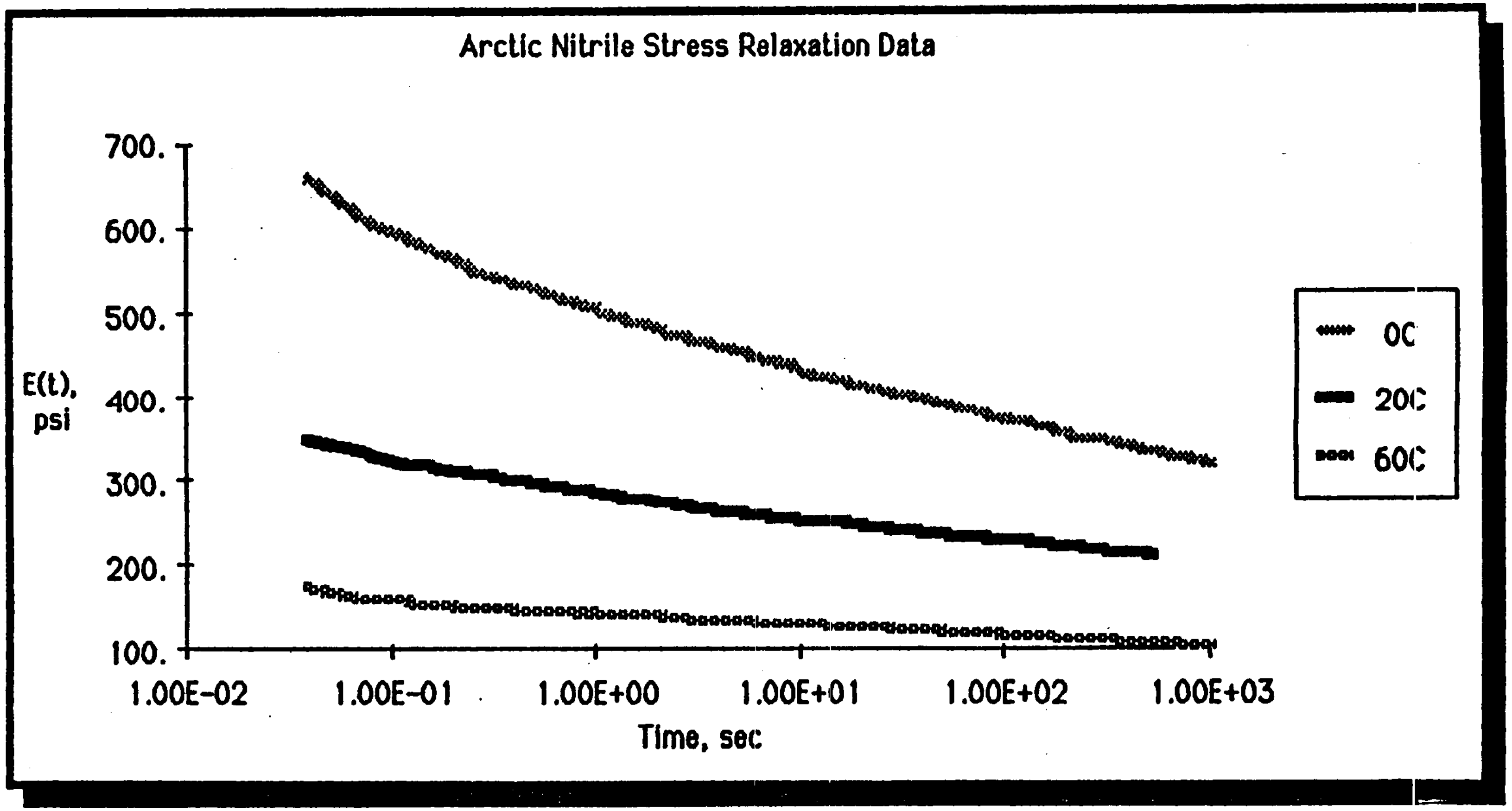

Figure VIII-7.
A Plot of the Stress Relaxation Data for Arctic Nitrile
Taken at Temperatures of 20, 40, and 60°C.

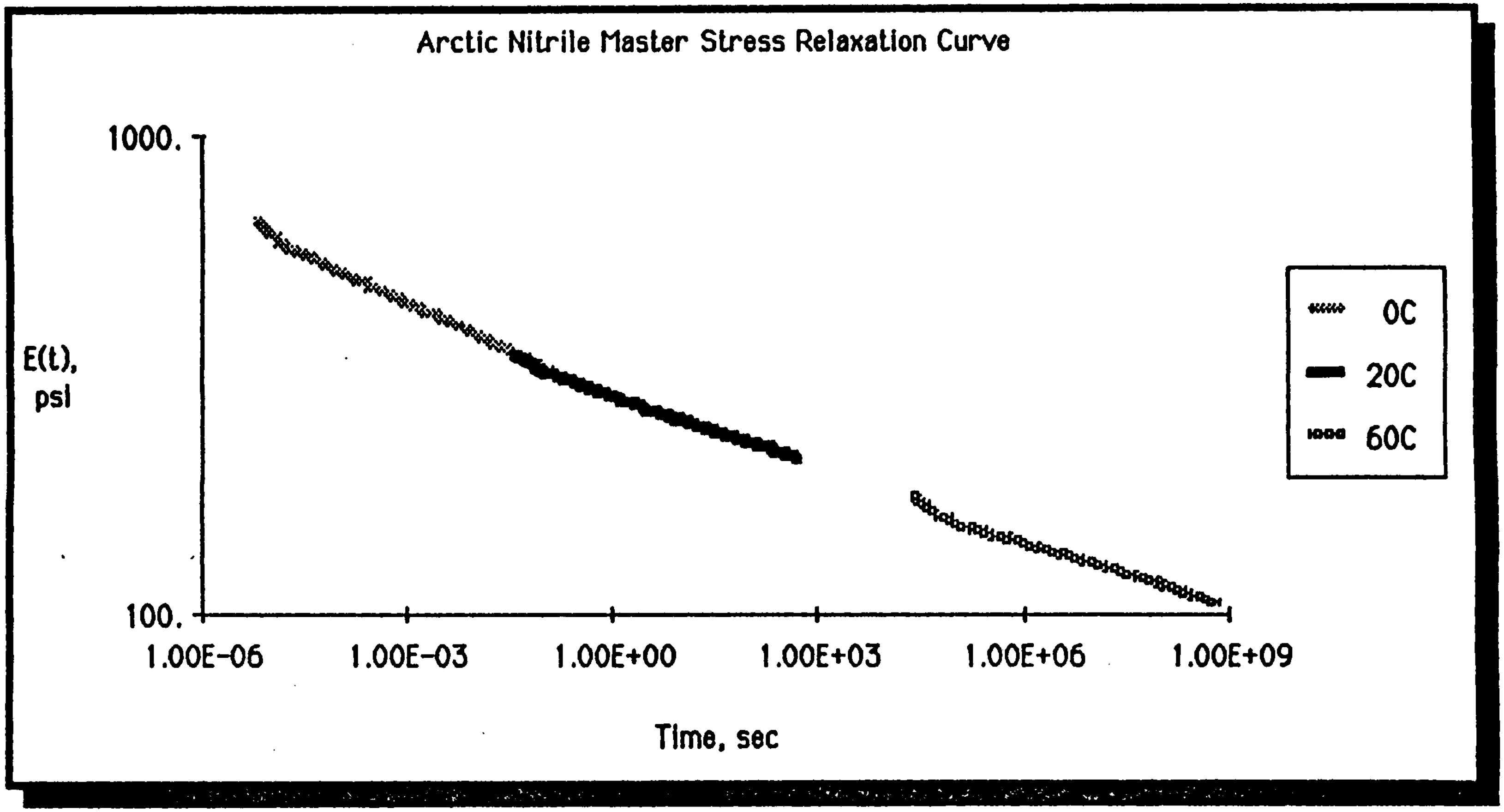

Figure VIII-8.
A Plot of the Master Stress Relaxation Curve for Arctic Nitrile
at a Reference Temperature of 20°C.

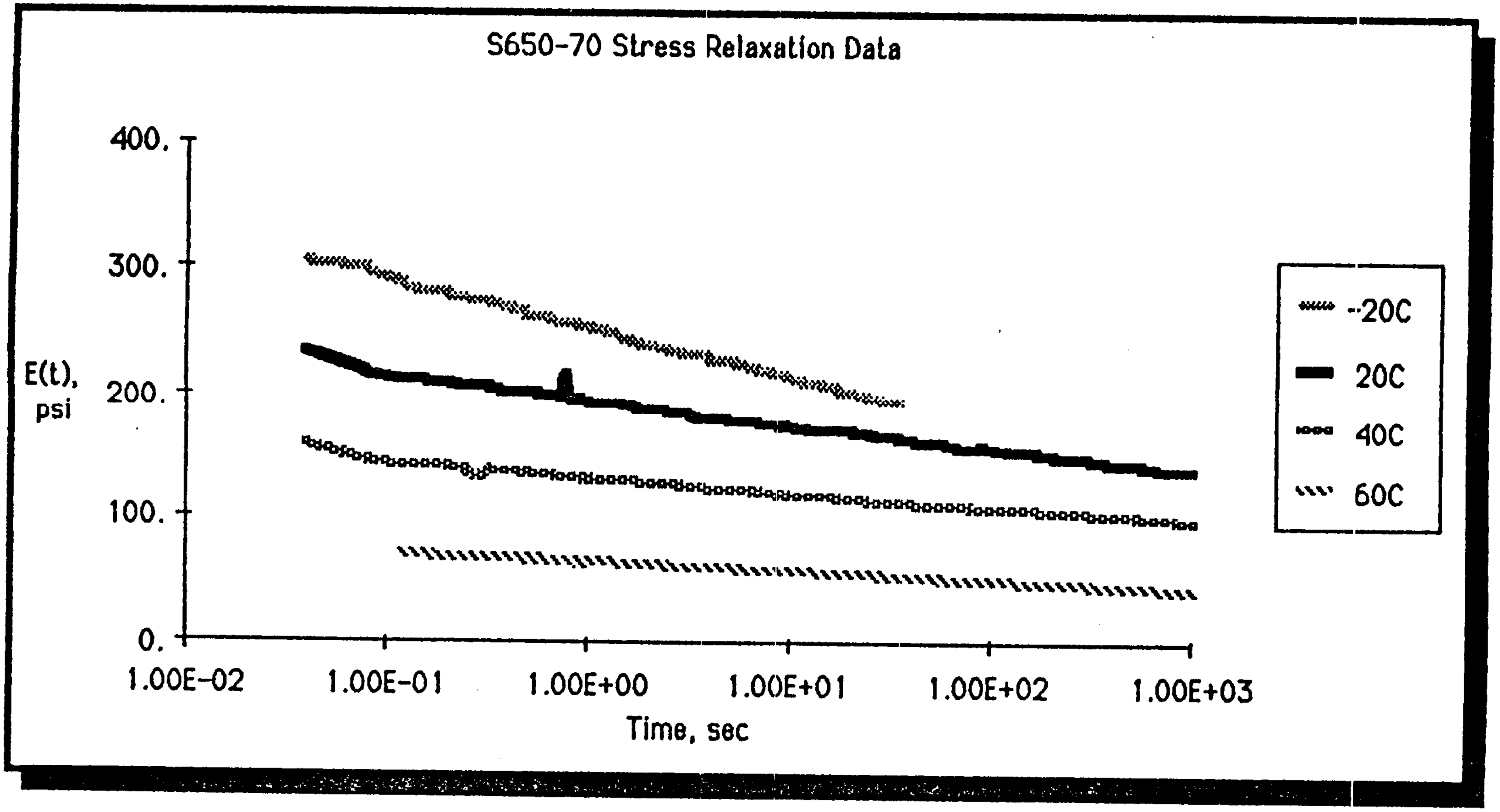

Figure VIII-9.
A Plot of the Stress Relaxation Data for S650-70
Taken at Temperatures of -20, 20, 40, and 60°C.

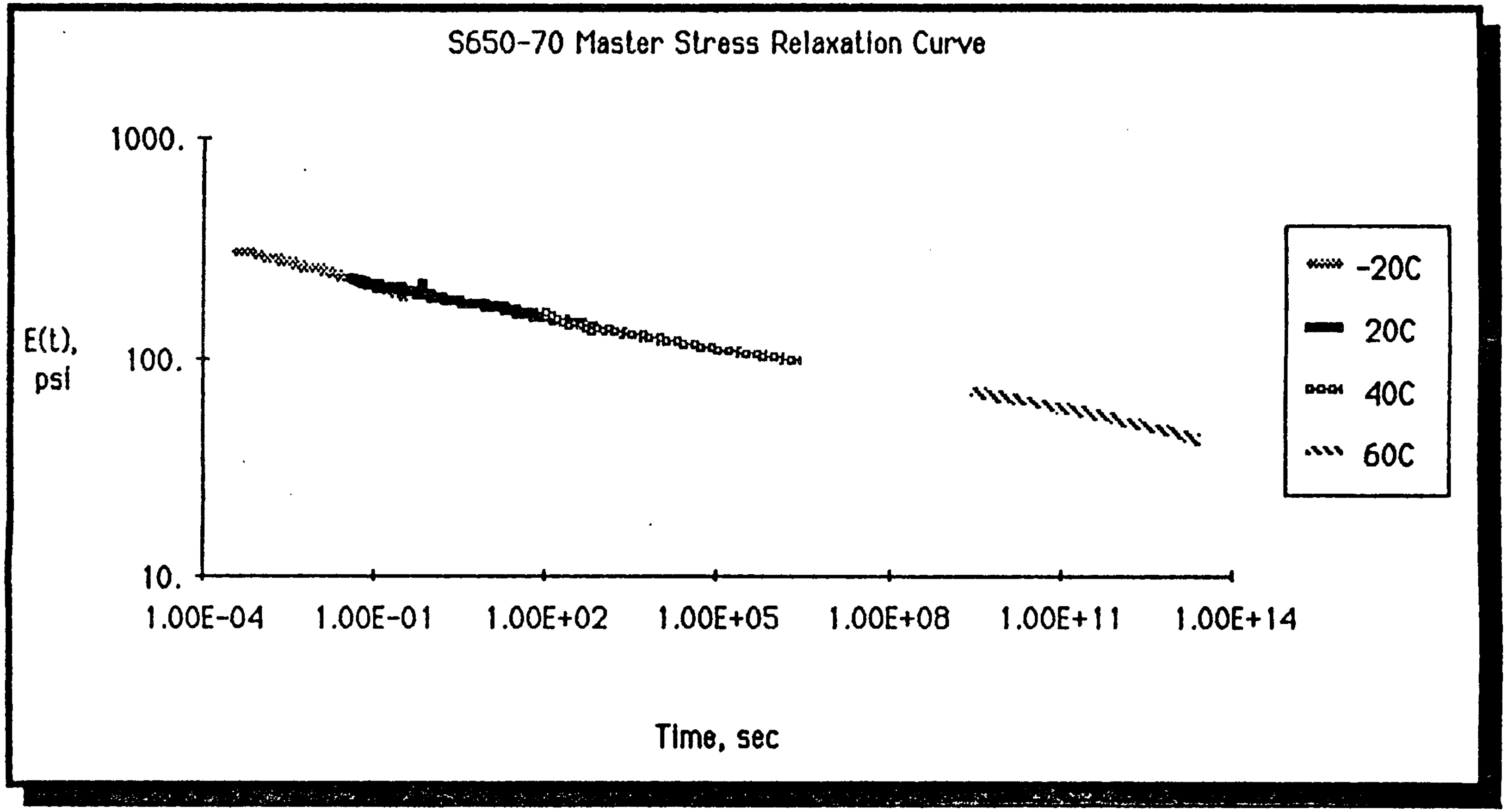

Figure VIII-10.
A Plot of the Master Stress Relaxation Curve for S650-70 at a Reference Temperature of 20°C.

Table VIII-1.
Table of Time Shift Factors, a_T, Referenced to 20°C and the Associated Temperatures for the Five Materials Tested.

Material	Shift Factor, a_T, at Temperature of -20°C	-10°C	0°C	20°C	40°C	60°C
V747-75	---	-3.9	-2.3	0.0	---	3.3
V835-75	---	---	---	0.0	1.7	3.0
N602-70	-1.0	---	---	0.0	1.5	2.8
Arctic Nitrile	---	---	-3.8	0.0	---	5.8
S650-70	-2.0	---	---	0.0	3.4	10.4

Table VIII-2.
Table of Maximum Time Values of Test Data as Shifted with a Reference Temperature of 20°C for the Five Materials Tested.

Material	Maximum Time (seconds)
V747-75	1.99×10^{6}
V835-75	1.02×10^{6}
N602-70	6.30×10^{5}
Arctic Nitrile	6.80×10^{8}
S650-70	2.51×10^{13}

APPLICATIONS TO DESIGN

The linear theory of the viscoelastic behavior of materials is well established. The development of this theory is a logical extension of the linear theory of elasticity. In linear elasticity (LE) the analysis of all problems is based on three fundamental steps. They are:

1. The analysis of forces. In this step the applied forces are identified and the force and moment balance equations (equations of motion or equilibrium equations) are written. Where possible the unknown forces are computed from these equations.

2. The analysis of deformations. Here the constitutive equations are written in terms of the applied force. When the forces are known from the first step the deformation can be computed directly. Conversely, when the deformation is known and the force unknown, the constitutive equation can be inverted to compute the applied force.

3. The analysis of the deformed geometry. When all of the unknown displacements or deformations and forces can be computed from information in the first two steps, this step is not required. However, in situations which are termed indeterminate (i.e. those where the first two steps are not sufficient to determine the unknowns) additional information is required to compute the unknowns. This information is obtained from analysis of the deformed geometry.

The primary difference between linear elasticity and linear viscoelasticity (LVE) is the constitutive equation used in step two and three listed above.

There are two forms of the constitutive equation used in linear viscoelasticity. The two models are a differential model and a single integral model. The differential model requires initial conditions on the derivative of the stress with respect to time. This information is not typically not available. Therefore, this model is used infrequently. The single integral model used is a heredity integral involving the stress relaxation function and the rate of strain. It is:

$$\sigma(t) = \int_{-\infty}^{t} E(t-\tau)\dot{\epsilon}(\tau)d\tau \ , \qquad \text{(a)}$$

where $\dot{(\ )}$ indicates derivative with respect to the argument, $\sigma(t)$ is the stress function, $\epsilon(t)$ is the strain function, t is the current time, and τ is some time on the interval from $-\infty$ to t [2][3]. The inverse of this relation is:

$$\epsilon(t) = \int_{-\infty}^{t} E^{-1}(t-\tau)\dot{\sigma}(\tau)d\tau \ , \qquad \text{(b)}$$

where $E^{-1}(t)$ is the creep compliance function. Note that:

$$E(t) \neq \frac{1}{E^{-1}(t)} \ .$$

The relationship between the stress relaxation function and the creep compliance function is:

$$1 = \int_{-\infty}^{t} E(\tau)E^{-1}(t-\tau)d\tau \ . \qquad \text{(c)}$$

When one of the material property functions is unknown and necessary for the solution of a problem it is possible to compute the needed function from the above equation. However, this is often a difficult problem if an analytical expression is not available for the known material function.

One of the most common analytical expressions available for viscoelastic solid is the so-called three parameter solid. The three parameter solid stress relaxation function is:

$$E(t) = E_{\infty} + (E_0 - E_{\infty})\exp(-t/\tau_R) \ , \qquad \text{(d)}$$

where E_0 is the initial (t=0) value of the stress relaxation function, E_{∞} is the asymptotic value of the stress relaxation function, and τ_R is the relaxation time for the material. The asymptotic value of the stress relaxation function, E_∞, is sometimes called the equilibrium modulus. The name for the three parameter solid is derived from viewing the material as a mechanical analog. In this analog a spring is combined in series with an element which is made of a spring connected in parallel to a dash pot, or damper (a Voigt element). Alternate expressions commonly used are prony series (a series of exponentials) expressions. While these expressions may more accurately represent the material behavior (i.e. the stress relaxation function) it is very difficult to compute the various coefficients and time constants. In general, for these expressions the time constants are selected arbitrarily and the coefficients computed based on these values. This procedure may not yield an expression which correctly describes the material behavior.

A simple example of a viscoelastic analysis is presented in the following section. Consider a viscoelastic solid which is loaded by an applied strain (as may be the case of an installed O-ring). The applied strain is known to be a function which is a ramp to a constant value at time T^*. This is:

$$\epsilon(t) = \begin{cases} 0 & -\infty < t < 0 \\ \alpha t & 0 \le t < T^* , \\ \alpha T^* & T^* \le t < \infty \end{cases} \qquad \text{(e)}$$

where α is a known constant. The stress in the solid can then be computed from equations (a) and (e). This may be done analytically when an expression such as equation (d) is available, or numerically by using a simple spline interpolation to determine the value of the stress relaxation function at the necessary times. For a three parameter solid the stress is:

$$\sigma(t) = E_\infty \alpha T^* + \alpha \tau_R (E_0 - E_\infty)\{\exp([T^*-t]/\tau_R) - 1\} .$$

The limiting, or asymptotic value of the stress in the solid is $E_\infty \alpha T^*$. From this value a critical value of time, T_p, can be established such that at time T_p the stress has relaxed to some percentage of the limiting stress. For the purposes of this example let p represent this value. Note that p must be greater than one since the stress is decaying to a value. For the above expression the result is:

$$T_p = T^* - \ln\left\{\frac{(p-1)}{(E_0/E_\infty - 1)}\,\frac{T^*}{\tau_R} - 1\right\} .$$

Observe that the ratio of the process time to the characteristic material time (T^*/τ_R) appears in the expression for T_p. This ratio is known as the Deborah number and is significant in the analysis of viscoelastic response of a system.

CONCLUSIONS AND RECOMMENDATIONS

The conclusions of this study are:

1. The compressive stress relaxation function for elastomers can be experimentally obtained using the Rheometrics Dynamic Spectrometer available in the MSFC Materials and Processes Laboratory.

2. The methodology developed in this study is effective for determining the compressive relaxation modulus of elastomers.
3. The elastomers under consideration for use as the O-ring material display a both time and temperature dependence. The degree of dependence differs from one material to another.

4. The time - temperature shift of the relaxation function developed for polymers applies to the candidate elastomers and the master stress relaxation curves are presented.

The recommendations of this study are:

1. Tests at the temperatures at which problems were encountered should be rerun and the results incorporated into the master stress relaxation curves presented here.

2. The method of machining the test samples should be modified to produce higher quality samples. One possible technique to use is to soak the stock to be machined in liquid nitrogen and then spray the stock with liquid nitrogen while machining. This should eliminate machining problems encountered due to the softness of the material without significantly affecting the material properties.

3. Similar tests should be performed on the candidate materials after they have been subjected to a liner - propellant cure cycle (If the factory joint O-ring materials are to be changed in the new SRB design.). The objective of this test series is to determine if the material properties of the O-ring would be changed by the cure process.

4. The results from this study should be used to predict the compression set of the O-ring. The first step in this analysis is to compute the value of the compression set (as defined in the ASTM test specification) for V747-75 and compare the results to the experimental data available. If these results indicate good correlation, the compression set of the other materials should be computed.

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