ADHESIVES IN THE LAP SHEAR MODE
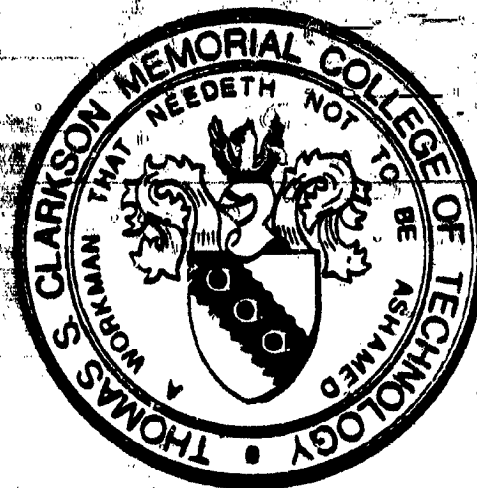
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ABSTRACT

A general method for characterizing structural adhesives in the bonded lap shear mode is proposed. Two approaches in the form of semi-empirical and theoretical approaches are used. The semi-empirical approach includes Ludwik's and Zhurkov's equations to describe respectively, the failure stresses in the constant strain rate and constant stress loading modes with the inclusion of the temperature effects. The theoretical approach is used to describe adhesive shear stress-strain behavior with the use of viscoelastic or nonlinear elastic constitutive equations. Two different model adhesives are used in the single lap shear mode with titanium adherends. These adhesives (one of which was developed at NASA Langley Research Center) are currently considered by NASA for possible aerospace applications. Use of different model adhesives helps in assessment of the generality of the method.

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ACKNOWLEDGMENTS

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NOMENCLATURE

A,B,C	material constants for Maxwell model
A',B',C'	material constants
A",B",C"	material constants for Kelvin model
F(T)	function of temperature
G	shear elastic modulus
H(t)	unit step function
K,n	material constants
R	universal gas constant
t_o	time constant
t_r	rupture time
T	absolute temperature
U_o	constant activation energy
$\gamma, \dot{\gamma}$	one dimensional shear strain and shear strain rate
$\epsilon_{ij}^E, \epsilon_{ij}^V$	elastic and viscoelastic strains
θ	elastic limit stress
μ	coefficient of viscosity in shear
$\tau, \dot{\tau}$	one dimensional shear stress and shear stress rate
τ_{ult}	ultimate shear stress
τ_o	level of constant stress
$\tau', \tau'', \dot{\gamma}'$	material constants
$\tau(t)$	time dependent shear stress
ϕ	elastic limit shear strain
χ	time dependent material property
γ_o	level of initial (instantaneous) strain in a creep test

INTRODUCTION

In the absence of catastrophic crack propagations, the failure of lap joints may occur in one of the following modes:

1. Rupture of the adhesive layer, when the ultimate stress is reached;

2. Creep rupture of the adhesive layer when a high level of constant load is used;

3. Failure of the adherends.

Failure in the third mode will be unlikely when metal adherends such as steel or titanium are used. It is necessary, however, to consider the first two modes of failure for the design of adhesively bonded joints.

For design purposes, one often needs only the elastic properties (namely Young's modulus and elastic limit stress) and failure stresses as a function of rate, time and temperature. If such is the case, then a satisfactory characterization can be obtained with the use of a semi-empirical approach to describe the failure stresses as a function of rate and temperature for constant strain rate loading and as a function of time and temperature for constant load conditions. This approach assumes that the viscoelastic effects are negligible in the initial portion of the stress-sti in curve, so that an initial elastic strain rate can be defined. Previous work on a variety of polymeric materials and adhesives proved this assumption to be a valid one [1, 2, 3, 4, 5].

If information on the magnitude of strains (passed the elastic limit) is also required, then one needs to use a theoretical approach with the application of a mechanical model to characterize the material. Information on failure stresses can be extracted from such an approach if perfectly plastic flow is present (i.e. if the constant strain rate stress-strain curve has an asymptote). However, one still needs to use semi-empirical methods associated

with the model, to obtain the creep rupture stresses. Furthermore, the use of a semi-empirical method is necessary to obtain rupture stresses for constant strain rate condition, when perfectly plastic flow is not present.

It should also be noted that for some linear thermoplastic adhesives t' effects of rate on the failure stresses may be negligible enough to permit the use of a nonlinear elastic relation to characterize the constant strain rate stress-strain behavior.

The semi-empirical relation proposed to describe rupture stresses at room and elevated temperatures are Ludwik's equation [6] for the constant strain rate condition and Zhurkov's [7] and Crochet's [8] equations for the creep condition. It should be noted that the original form of Ludwik's and Crochet's equations do not include thermal effects. This report proposes an empirical modification of Ludwik's equation and an empirical extension of Crochet's equation to describe the effects of temperature.

Data on the room temperature constant strain rate and room and elevated temperature creep behavior of two different structural adhesives in the bonded form is presented. The model adhesives are currently being considered by NASA for possible aerospace applications. They are:

1. Thermoplastic Polyimidesulfone, and

2. FM 73.

1. Thermoplastic Polyimidesulfone is a novel adhesive developed at NASA Langley Research Center. It has thermoplastic properties and solvent resistance. It is processable in the 482 – 662°F (250 – 350°C) range and has high temperature resistance [9].

2. FM 73 is a modified epoxy adhesive film with carrier cloth. It is manufactured by the American Cyanamid Company. Its product information brochure reports service temperature range of -67°F to 248°F (-55°C to 120°C) with high moisture resistance [10].

The applicability of proposed semi-empirical equations describing the rate dependent failure stresses in constant strain rate condition and time and temperature dependent rupture stresses in creep conditions are assessed with the use of experimental data. The constant strain rate stress-strain data for the FM 73 and Thermoplastic Polyimidesulfone adhesives are fitted with (linear viscoelastic) modified Bingham Model [1] and a nonlinear-elastic (power function) relation respectively.

The amount of data available at this stage of the investigation does not permit any conclusive assessment of the proposed characterization method. The authors hope, however, that as more adhesives are tested, a practical method will emerge to enable material characterization of structural adhesives in the bonded form.

ANALYTICAL CONSIDERATIONS

I. Semi-Empirical Approach

The semi-empirical approach considers failure behavior in constant strain rate and constant stress modes with the inclusion of thermal effects.

a) Rate Dependent Failure Stress:

In order to express the rate dependence of the rupture stresses in constant strain rate tests, a semi-empirical equation proposed by Ludwik (as reported by Thorkildsen [6]) will be used in the form

$$\tau_{ult} = \tau' + \tau'' \, \text{Log} \left(\frac{\overset{\circ}{\gamma}}{\overset{\circ}{\gamma}'} \right) , \tag{1}$$

where τ_{ult} is the ultimate shear stress, $\overset{\circ}{\gamma}$ is the initial elastic shear strain rate and τ', τ'' and $\overset{\circ}{\gamma}'$ are material constants. This equation was used successfully for metals and polymers in many previous investigations, some of which included structural adhesives in the bulk tensile, shear and bonded

lap shear modes [1, 2, 3, 5]. It should be noted that Equation (1) can also be used for describing the rate dependence of the elastic limit stresses [2, 3].

Any empirical modification of Equation (1) to account for thermal effects would require experimental justification. For example, examination of the tensile strength vs. strain rate data for copper at various temperatures (73°F ≤ T ≤ 1832°F, 23°C ≤ T ≤ 1000°C) [12] indicates a near parallel shift in the strain rate vs. strength relation particularly in the 392°F (200°C) to 1472°F (800°C) region. Such a parallel shift in Equation (1) could be expressed as

$$\tau_{ult} = \{F(T)\}[\tau' + \tau'' \ \mathrm{Log} \ (\frac{\overset{o}{\gamma}}{\gamma'})] \tag{2}$$

where F(T) would be any appropriate function of temperature.

b. Temperature Dependent Creep-Rupture Equation:

The semi-empirical equation

$$t_r = t_o \ \exp \ [\frac{U_o}{RT} - \gamma\sigma] \tag{3}$$

where

t_r = rupture time

t_o = a constant normally about 10^{-12} (stated to be the period of natural oscillation)

U_o = a constant activation energy (associated as an energy-barrier term)

γ = a constant

σ = applied uniaxial stress

R = universal gas constant

T = absolute temperature

represents the modified (by Slonimskii [12]) form of Zhurkov's creep rupture equation.

This equation was recently used by Brinson et al. to describe the creep rupture behavior of polymer-matrix composites [13].

c) Crochet's Delayed Failure Equation:

A decaying exponential relation of the form

$$\sigma(t) = A' + B' \exp(-C'\chi) \tag{4}$$

was proposed by Crochet to describe delayed failures. In Equation (4), $\sigma(t)$ is the time dependent maximum stress, A', B' and C' are material constants and χ is a time dependent material property given by

$$\chi = [(\epsilon_{ij}^V - \epsilon_{ij}^E)(\epsilon_{ij}^V - \epsilon_{ij}^E)]^{\frac{1}{2}} \tag{5}$$

where ϵ_{ij}^V and ϵ_{ij}^E refer to viscoelastic and elastic strains, respectively. Equation (4) can be interpreted for the pure shear condition. U viscoelastic model is necessary, however, in order to obtain the material property χ. For practical purposes, simple linear viscoelastic models, Maxwell fluid or Kelvin solid can be used to facilitate the solution to Equation (5). The use of Maxwell Model results in

$$\tau(t) = A + B \exp\left[-\frac{C\tau_o}{\mu} t\right], \tag{6}$$

and the use of Kelvin model results in

$$\tau(t) = A'' + B'' \exp\left[C''e^{-\frac{G}{\mu}t}\right] \tag{7}$$

where:

$\tau(t)$ = time dependent maximum shear (rupture) stress

τ_o = level of constant stress,

μ = coefficient of viscosity in shear

G = modulus of elasticity in shear

Apparently Equation (7) describes a much faster approach in time of the rupture stresses to their asymptotic values.

The asymptotic values A and (A" + B") of Equations (6) and (7) respectively are important design parameters, as they represent the maximum safe stress values below which creep failures are not expected to occur. One can express A and/or (A" + B") values as a function of the applied temperature to represent experimental data in an empirical fashion.

II. Theoretical Approach:

In order to predict the stress-strain behavior of the adhesive and to extract failure information for constant strain rate and constant stress modes, one needs to determine the viscoelastic model which will describe the material adequately. If the rate effects on the material are determined to be negligible, then the use of a nonlinear elastic relation may also be adequate. Brinson et al. [1] used the modified Bingham model (shown in Figure 2), which was modified from of the Schwedoff model [14], for characterizing the tensile behavior of Metlbond adhesive. The modified Bingham model was later used by Sancaktar [3] for characterizing the shear behavior of the same adhesive. For that work, the modified Bingham model was applied in the form,

$$\tau = G\gamma \qquad\qquad \tau \leq \theta$$
$$\overset{\circ}{\gamma} = \frac{\overset{\circ}{\tau}}{G} + \frac{\tau-\theta}{\mu} \qquad \theta < \tau \leq \tau_{ult} \tag{8}$$

where τ and γ are shear stress and strain respectively, and θ is the elastic limit shear stress.

To obtain the constant strain rate relation, the above constitutive equation is solved according to the condition

$$\overset{\circ}{\gamma} = R = constant \tag{9}$$

to result in

$$\tau = G \gamma \qquad \tau \leq \theta \tag{9}$$

$$\tau = \theta + \mu \overset{\circ}{\gamma} [1 - \exp ^{-\frac{(\gamma-\phi)G}{\mu \overset{\circ}{\gamma}}}] \qquad \theta < \tau \leq \tau_{ult} \tag{10}$$

where ϕ is the elastic limit strain.

The creep relation is obtained by using a step loading

$$\tau(t) = \tau_o H(t) \tag{11}$$

where $H(t)$ represents the unit step function and τ_o is the level of constant stress, with the initial condition

$$\gamma(t=o) = \frac{\tau_o}{G_o} \tag{12}$$

to obtain

$$\gamma(t) = \frac{\tau_o - \theta}{\mu} t + \frac{\tau_o}{G} \quad . \tag{13}$$

In order to extract creep-rupture information from the given model, Crochet's delayed failure equation (Equation 4) is used. Equation (4) and (5) can be applied to the shear condition by subtracting the elastic strain

$$\gamma_o = \tau_o/G_o \tag{14}$$

from the model's creep equation (13) (which describes viscoelastic strains), to obtain χ and substituting into Equation (4) to result in

$$\tau(t) = A + B \exp [-K(\tau(t)-\theta)t] \quad . \tag{15}$$

Equation (15) is the creep-rupture equation based on the modified Bingham model. It should be noted that the time-temperature superposition principle

can be used along with Equation (15) to make it useful at different temperature
levels. Naturally, it is not expected that the modified Bingham model will
characterize all available adhesives. One can, however, use other viscoelastic
models and follow the procedure described above to characterize most adhesives.
Some examples of such application would be the use of Chase-Goldsmith model
by Sancaktar [5] to characterize LARC-3 adhesive in the bonded form and the
use of Schapery's nonlinear constitutive equation [15] by Brinson et al. [16]
to characterize SMC-25 fiber reinforced polyester composite.

If the rate effects on adhesive behavior is determined to be weak, then
a nonlinear elastic relation can be used to fit the stress-strain data. The
power function relation expressed for the state of pure shear in the form:

$$\tau = K \gamma^n$$

(16)

where K and n are material constants, is proposed for this purpose.

EXPERIMENTAL PROCEDURES

The processing of the test specimens bonded with Thermoplastic Polyimidesulfone has been described in detail by St. Clair [9]. Two one-inch (2.54 cm) wide, 0.05 inch (0.127 cm) thick titanium alloy strips which are exact copies of each other are sand blasted with aluminum oxide of 120 mesh. These duplicate strips are then coated with poly (amide-acid sulfone) which is formed by mixing 0.0569 lbs (25.8 gms) of 3, 3', 4, 4' - Benzophenonetetracarboxylic dianhydride (BTDA) in a solution made up of 0.0439 lbs (19.9 gms) of 3, 3' - diaminodiphenylsulfone (3, 3' - DDS) in 0.5701 lbs (258.6 gms) of bis (2-methoxyethyl) ether. Fifteen minutes of thermal treatments at 212°F (100°C) and 392°F (200°C), respectively, are applied to convert the amide-acid to the imide by removing the solvent. The adhesive layers are then applied on to the adherend strips. A 0.004 inch (0.010 cm) thick piece of woven glass cloth is inserted between the strips to control the adhesive thickness. A pressure of 200 psi (1.38 MPa) is applied and the specimen is heated at a rate of 9°F/min (5°C/min) up to 572°F (300°C). The bonded specimen is then cooled and removed.

The adhesive, FM-73, is a modified epoxy adhesive film supplied with a polyester knit fabric that offers optimum physical properties. It is manufactured by the American Cyanamid Company. Its product information brochure reports a service range of -67°F to 248°F (-55°C to 120°C) with a high resistance to moisture.

The bonding procedure with FM 73 adhesive is described in detail by its product information brochure [10]. The adherends are one-inch (2.54 cm) wide, 0.05 inch (0.127 cm) thick titanium alloy strips. Patterns of FM 73 adhesive film are cut and the adhesive's protective covering is peeled off at room temperature. The adhesive film is applied smoothly

on the duplicate titanium strips at approximately 110°F (43°C) to provide additional tacking. Curing of the joint is done by first heating it up to 250°F (120°C) at a rate of approximately 6°F/min (3.3°C/min). At the temperature of 250°F (120°C) a pressure of 40 psi (276 kPa) is applied for 60 minutes. The bonded specimen is then cooled and removed.

Specimen dimensions (width, length of overlap, and adherend thickness) are in accordance with ASTM D1002 specifications. The shear stress in the lap joints is assumed to be uniform and is calculated by dividing the load applied to the adherends by the overlap area. The mechanical testing of the FM 73 and Polyimidesulfone-titanium specimens was performed at Clarkson College of Technology by the methods described in the following paragraphs. All test specimens were prepared at NASA-Langley Research Center.

Geometrical Measurements

Precision measurement of bondline thicknesses and bondline lengths were done with the aid of a microscope under a magnification of 40 X. Twenty-five or twenty-six measurements of the bondline thickness and one measurement of the bondline length were taken along both sides of the overlap area. The average bondline thicknesses were used in calculating shear strains which were defined as bondline deformation divided by the average bondline thickness. Adherend width and thicknesses were measured with the aid of a Kanon Dial Caliper.

A high precision, air cooled clip-on gage was used for shear strain measurements. Two notches were milled 0.99 inches (2.51 cm) apart on the overlap side of the single lap specimens so that the clip-on gage could be attached in them (Fig. 1). The notches were 0.1 inch (0.254 cm) deep and 0.01 inch (.0254 cm) wide. Stress concentrations due to the notches

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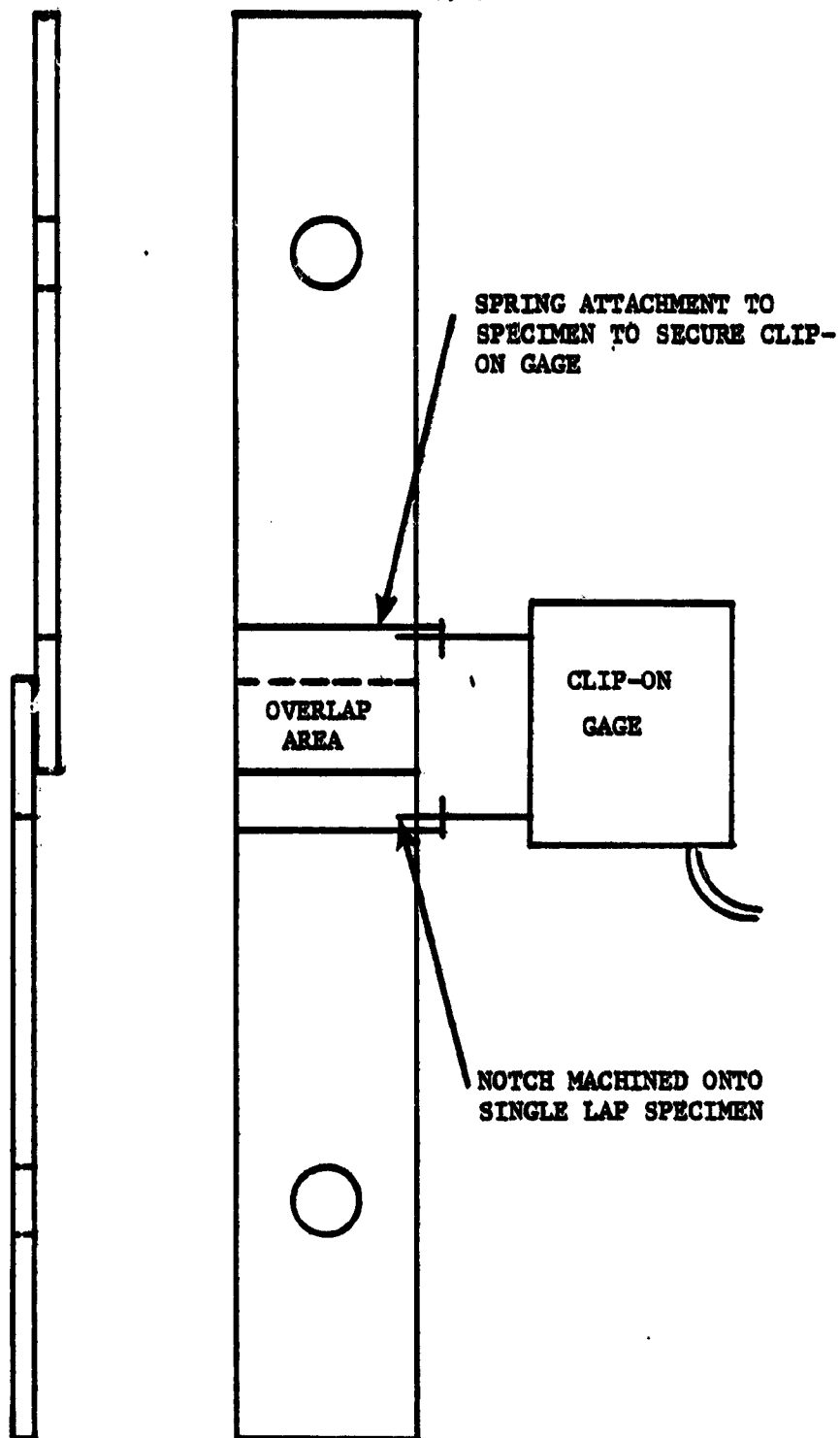OF POOR QUALITY

SPRING ATTACHMENT TO
SPECIMEN TO SECURE CLIP-
ON GAGE

CLIP-ON
GAGE

OVERLAP
AREA

NOTCH MACHINED ONTO
SINGLE LAP SPECIMEN

Figure 1.   Clip-on Gage Attachment to the Single Lap
            Specimen for Adhesive Deformation Measurement.

in the adherends were estimated to be negligible. Any adherend deformation that occurred within the measuring range of the clip-on gage was calculated and subtracted from the experimental data to obtain adhesive strains.

The clip-on gage was calibrated with a high magnification extensometer calibrator. Load and strain charts were obtained from a three channel strip chart recorder. The output signal from the clip-on gage was amplified through the testing machine's internal amplifier system. All test specimens were stored at approximately 70°F (21°C) temperature and 65% relative humidity.

Testing Methods

For each adhesive, twenty four specimens were provided by the NASA - Langley Research Center. Room temperature constant strain rate tests were performed on a Tinius-Olsen Universal testing machine at crosshead rates of ≥ 0.0, 0.1, 1, 5 and 10 in/min (≥ 0.0, 0.254, 2.54, 12.7 and 25.4 cm/min).

In the creep to rupture tests, each adhesive was tested in creep at least at four different stress levels above the elastic limit stress (found from the room temperature constant strain rate tests) and at four different temperature levels. The FM 73 adhesive creep tests were performed at temperature levels of 70°F (21°C), 130°F (54°C) and 180°F (82°C). The Polyimidesulfone adhesive creep tests were performed at temperatures of 70°F (21°C), 250°F (121°C), 350°F (177°C) and 450°F (232°C).

The room temperature creep tests were performed on a Tinius-Olsen Universal testing machine using its "load hold" system with an initial crosshead rate of 0.3 in/min (0.76 cm/min). The high temperature creep tests and constant strain rate tests were performed with the use of an environmental oven which was attached to a Model 1331 Instron Servohydraulic testing machine. Deformation and load signal outputs from the testing machine's amplifier were channelled to a PDP-11 computer for processing of the data.

RESULTS AND DISCUSSION

Constant strain rate, creep and creep rupture data for adhesives FM 73 and Polyimidesulfone will be presented in this chapter. Data on FM 73 will be discussed first.

Figure 2 shows the constant strain rate shear stress-strain behavior of FM 73 adhesive. Apparently, the 500 times increase in the shear strain rate, did not result in a drastic increase in the ultimate shear stress. The presence of perfectly plastic flow at the low strain rate, however, suggests that the modified Bingham model can be used to describe the constant strain rate stress-strain behavior of FM 73 adhesive. Comparison of theory (modified Bingham model) and experiment shows good agreement (Fig. 2). The constants (namely θ, μ, $\overset{\circ}{\gamma}$ and G) used in fitting the modified Bingham model to the experimental data are also shown in Figure 2.

The variation of ultimate shear stress with initial elastic shear strain rate for FM 73 adhesive is shown in Figure 3. Apparently a 500 times increase in the strain rate (from 7.33×10^{-1} to 3.63×10^2 %/sec) results in ~12% increase in the ultimate shear stress. Figure 3 also reveals that Ludwik's equation (Equation 1) provides a good fit to the experimental data. Figure 4 shows the variation of maximum shear strain with initial elastic shear strain rate. As expected from viscoelastic materials, the maximum strain decreases with an increase in strain rate. The data indicates a ~0.1 in/in (0.1 cm/cm) decrease in maximum shear strain values over a 500 times increase in strain rate. An empirical equation used to represent the data is also shown in Figure 4.

The effects of temperature on the constant strain rate stress-strain behavior of FM 73 is shown in Figure 5. The ultimate shear stress is ~53% lower at 180°F (82°C) condition; and it is ~87% lower at 250°F (121°C) condition. The maximum shear strain is increased by about twofold (from 55%) at elevated temperatures.
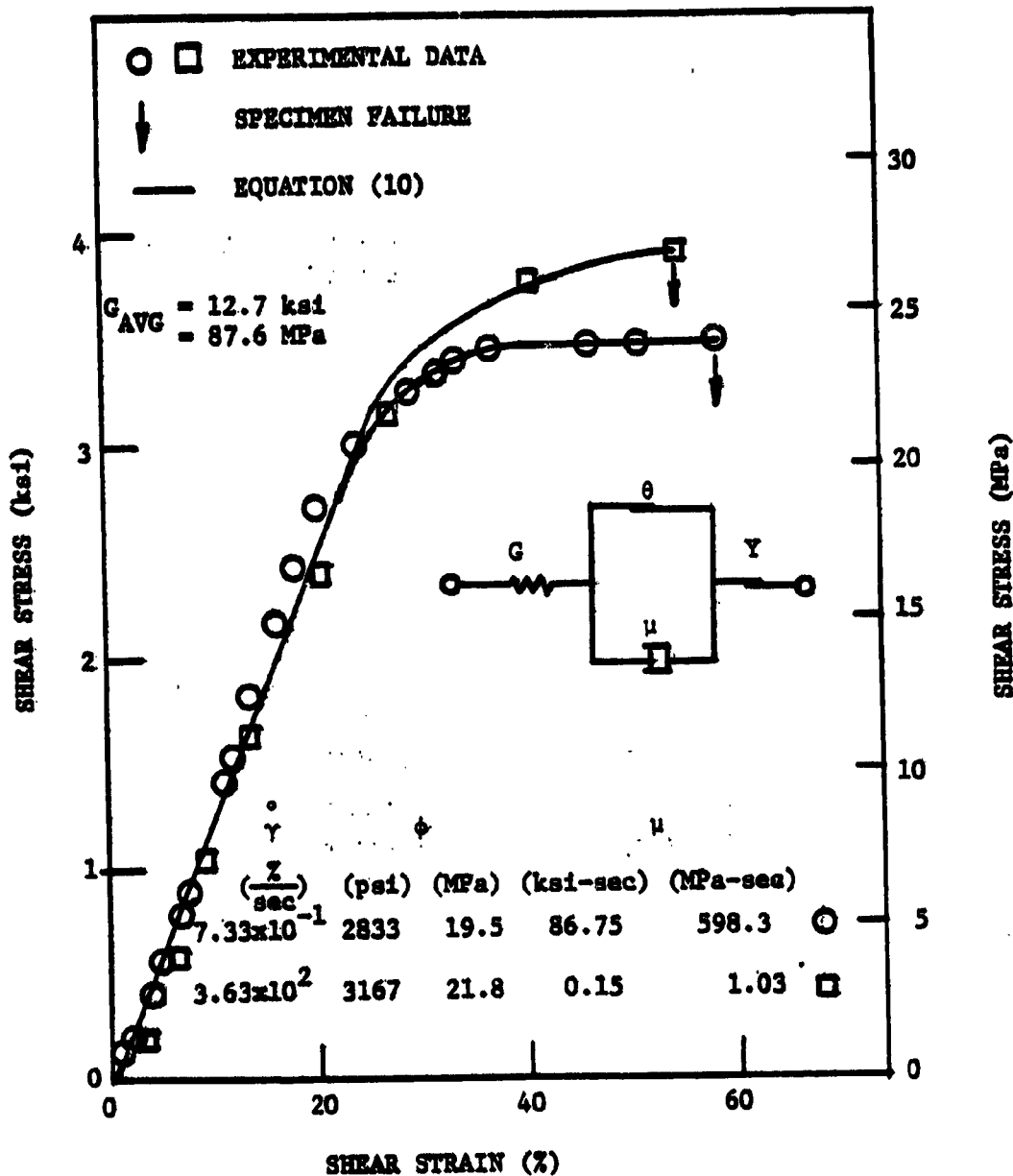
O ☐ EXPERIMENTAL DATA

↓ SPECIMEN FAILURE

—— EQUATION (10)

30

G_{AVG} = 12.7 ksi
= 87.6 MPa

4

25

3

20

SHEAR STRESS (ksi)

SHEAR STRESS (MPa)

θ

G   Y

μ

2

15

10

$\overset{\circ}{\gamma}$ φ   μ

1

5

$(\frac{\%}{sec})$ (psi) (MPa) (ksi-sec) (MPa-sec)

7.33x10^{-1} 2833   19.5   86.75   598.3 O

3.63x10^{2} 3167   21.8   0.15   1.03 ☐

0

0

0   20   40   60

SHEAR STRAIN (%)

Figure 2. Constant Strain Rate Stress-Strain Behavior of
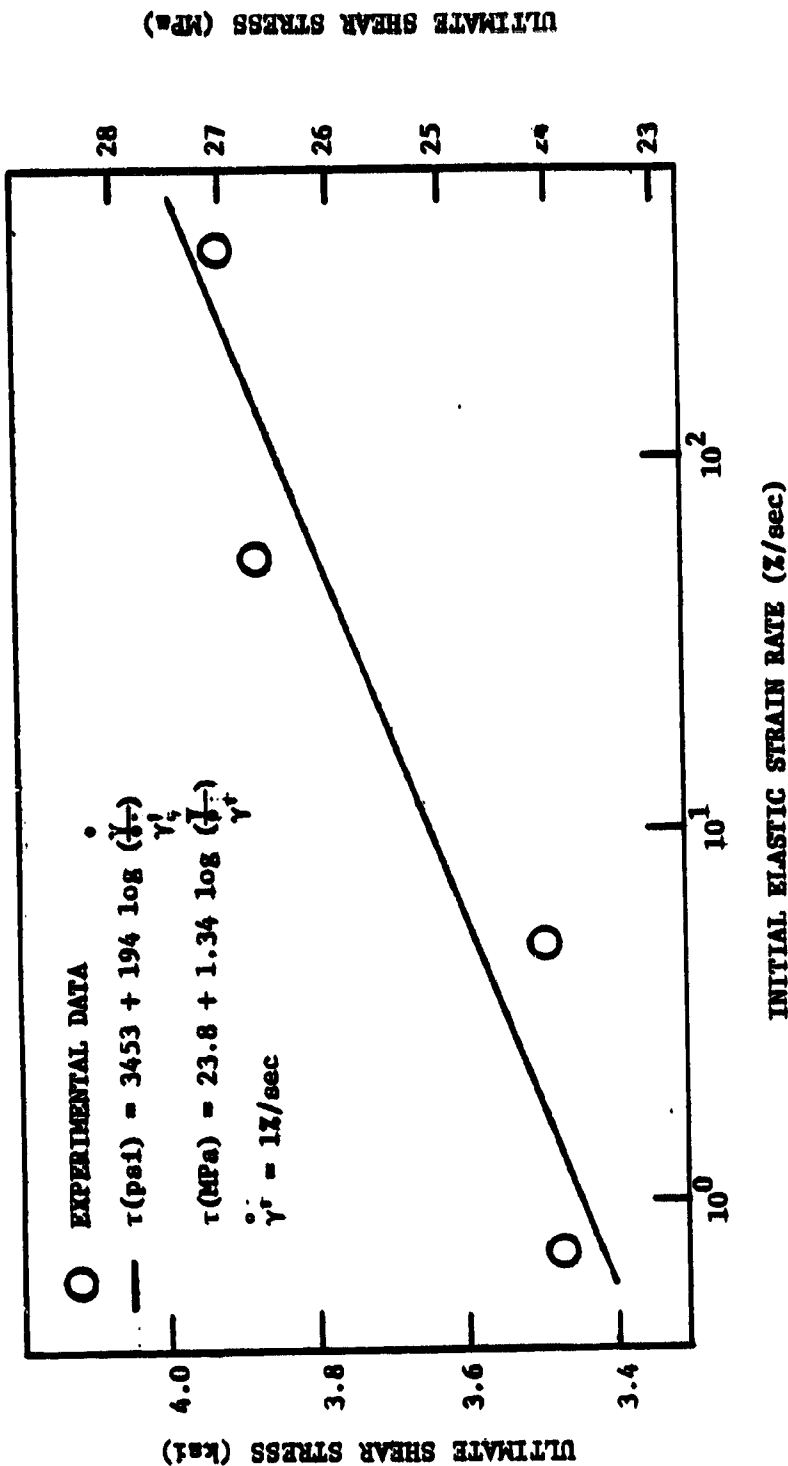FM 73 and Comparison with Theory.

Figure 3. Variation of Ultimate Shear Stress with Initial Elastic Strain Rate
and Comparison with Ludwik's Equation for FM 73 Adhesive.
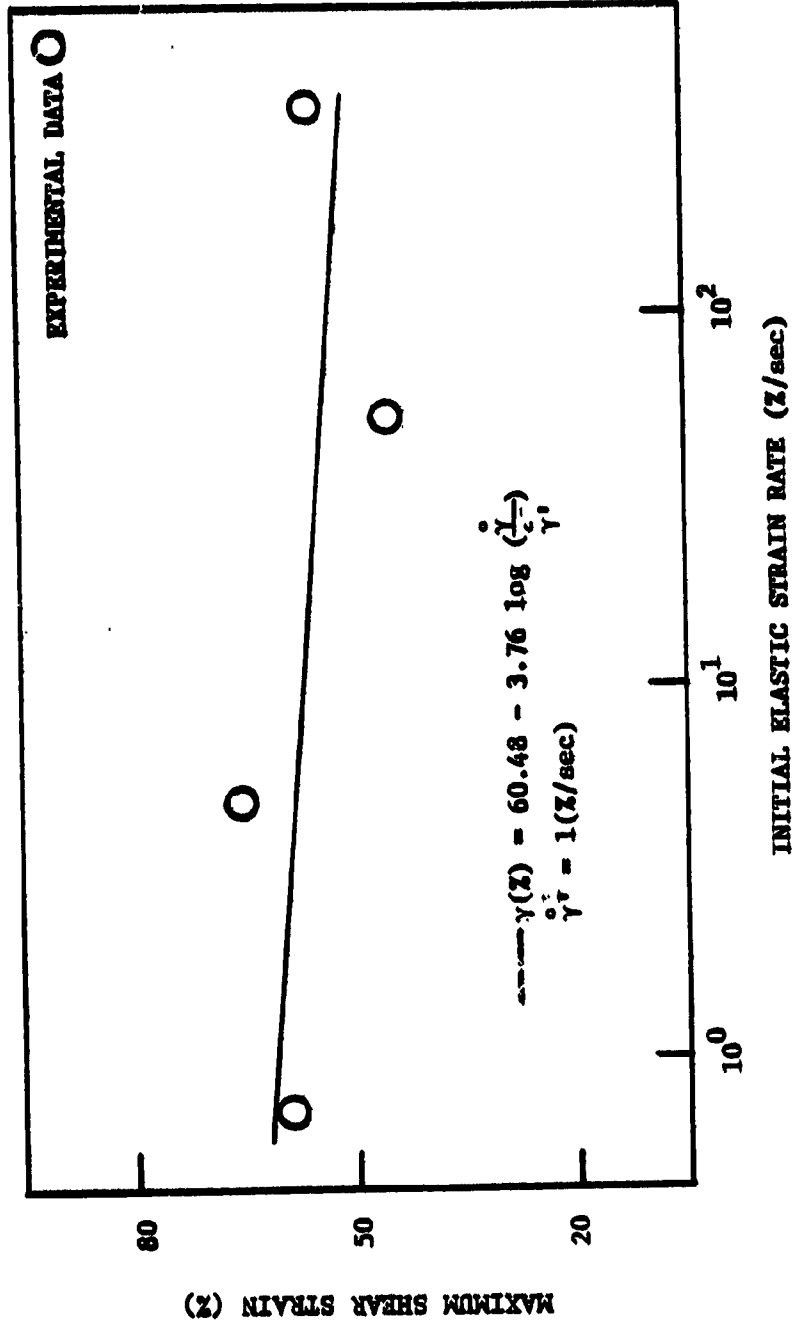
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Figure 4. Variation of Maximum Shear Strain with Initial Elastic Strain Rate for FM 73 Adhesive.

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SHEAR STRESS (MPa)

SHEAR STRAIN (%)

SHEAR STRESS (ksi)

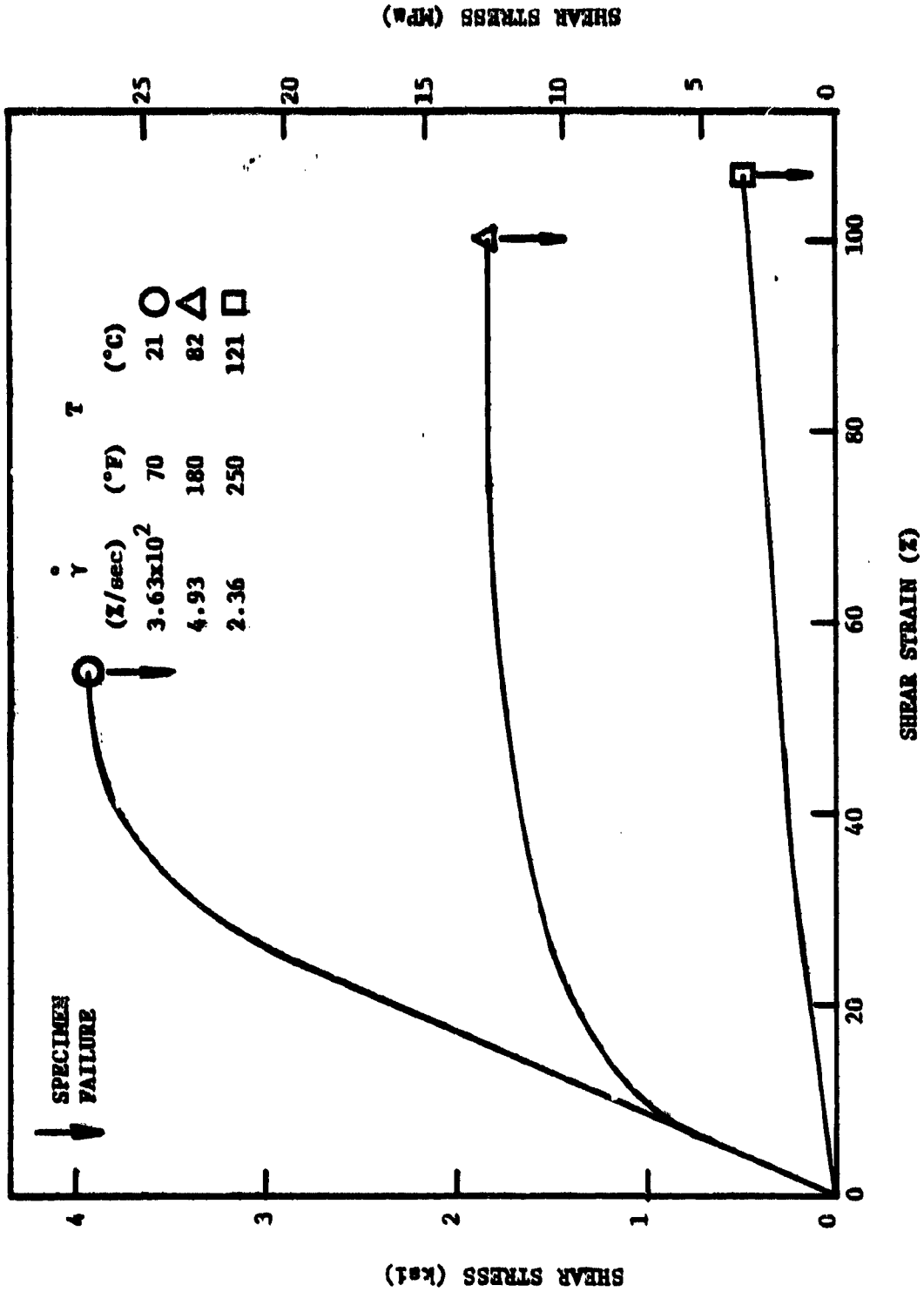| $\dot{\gamma}$	τ	
(%/sec)	(°F)	(°C)
3.63×10^2	70	21 ○
4.93	180	82 △
2.36	250	121 □

SPECIMEN
FAILURE

Figure 5. The Effects of Temperature on the Constant Strain Rate Stress-Strain
Behavior of FM 73 Adhesive.

Figure 6 shows the room temperature creep behavior of FM 73 adhesive.
A sudden plastic flow to failure is evident in the tertiary regions at
higher levels of stress. Creep response of FM 73 at 130°F (54°C) and 180°F
(82°C) conditions are shown in Figures 7 and 8 respectively. Apparently
higher levels of shear strains are reached at comparatively lower levels
of shear stresses, as the environmental temperature is increased. The
sudden plastic flow to failure observed with room temperature creep (Fig. 6)
is not evident at elevated temperatures.

Figure 9 shows comparison of experimental data with Zhurkov's creep
rupture relation (Equation 3). Data and theory show good agreement at
elevated temperatures. The room temperature data, however, does not show
agreement with the theory. The constants u_o and γ used to fit Zhurkov's
equation to the elevated temperature creep rupture data is also shown
in Figure 9.

Comparison of the creep rupture data with Crochet's equation (Equation
6) is shown in Figure 10 along with the appropriate constants used to fit
the data. Evidently theory provides a good fit to the experimental data.
Figure 11 shows variation of the maximum - or safe - level of creep stress
values (below which creep failures are not expected to occur) with environmental
temperature. The maximum creep stress values represent the asymptotic stress
levels (i.e. material constants A in Figure 10) obtained with the application
of Crochet's equation. Apparently the relation between the asymptotic stress
level and temperature is a linear one, for the range shown.

Experimental results revealed that Thermoplastic Polyimidesulfone
exhibits weak rate and time dependence. Because of this reason, the observed
mechanical behavior of Polyimidesulfone can be considered to be closely
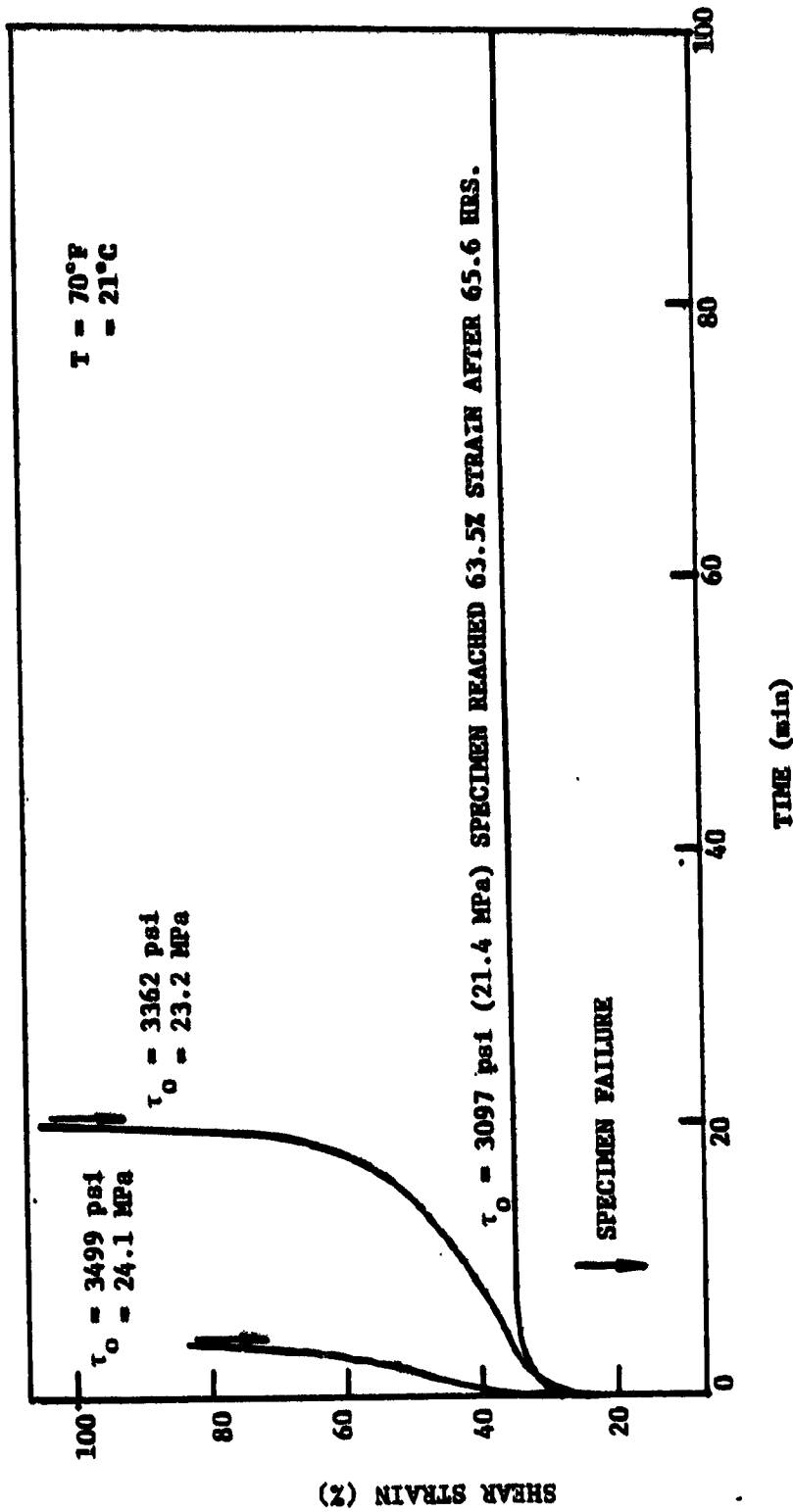(nonlinear) elastic. The relatively high levels of maximum shear strain

Figure 6. Room Temperature Creep Response of FM 73 Adhesive.

747

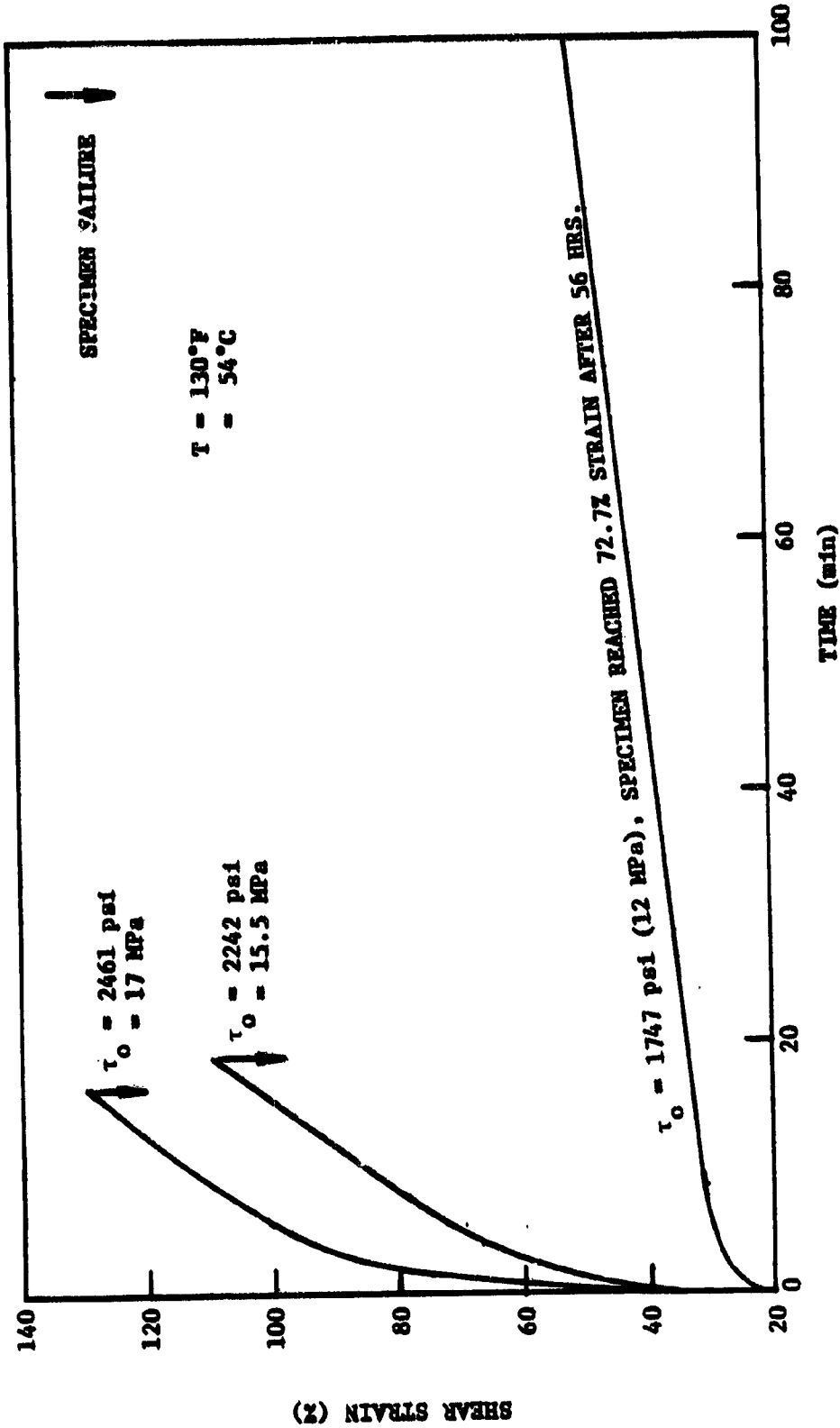

Figure 7. Creep Behavior of FM 73 Adhesive at 130°F (54°C) Condition.

SHEAR STRAIN (%)

TIME (min)

$\tau_o = 2461$ psi = 17 MPa

$\tau_o = 2242$ psi = 15.5 MPa

$\tau_o = 1747$ psi (12 MPa), SPECIMEN REACHED 72.7% STRAIN AFTER 56 HRS.

SPECIMEN FAILURE

T = 130°F = 54°C

20

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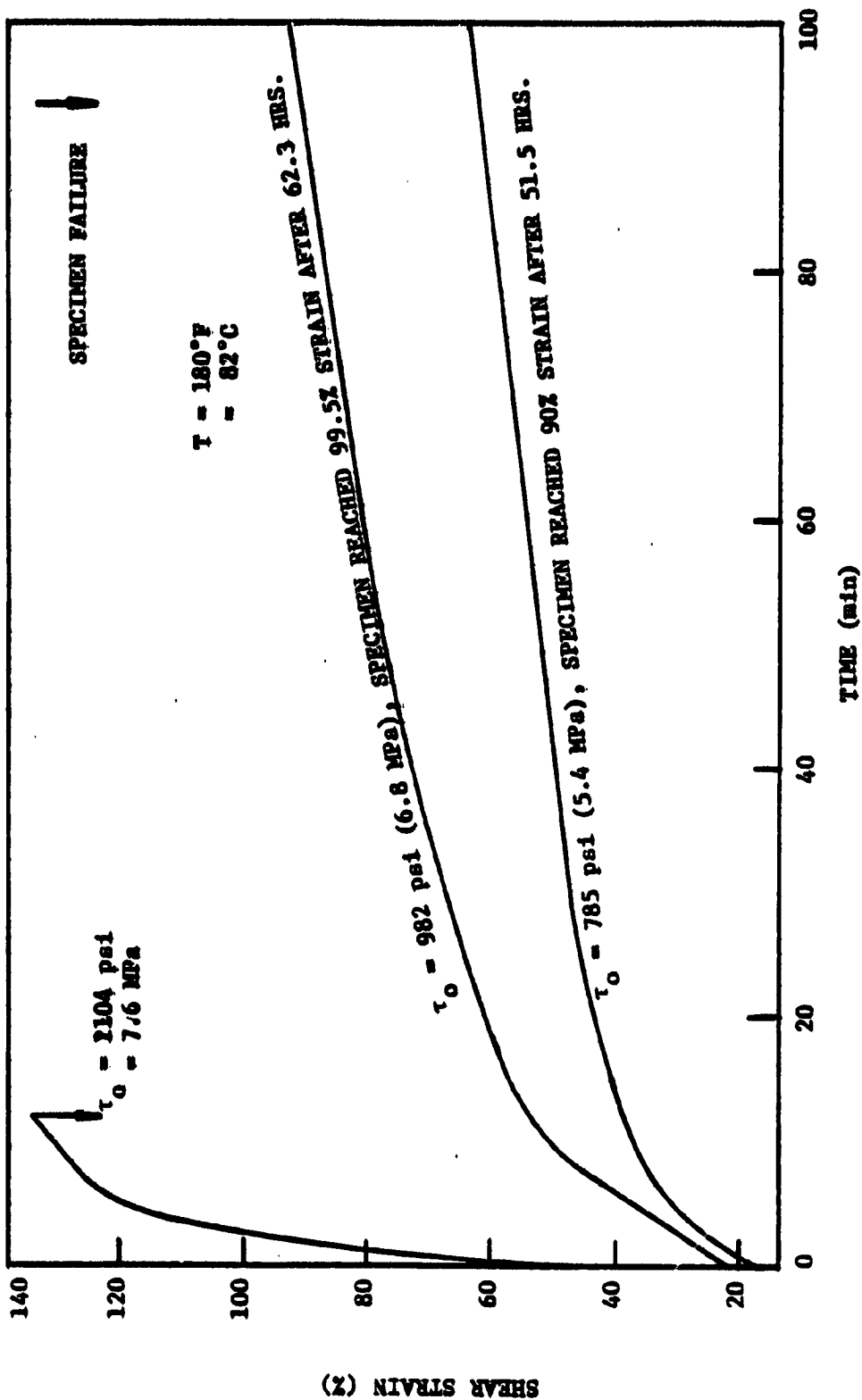
Figure 8. Creep Behavior of FM 73 Adhesive at 180°F (82°C) Condition.

SHEAR STRAIN (%)

TIME (min)

SPECIMEN FAILURE

T = 180°F
  = 82°C

τ_o = 982 psi (6.8 MPa), SPECIMEN REACHED 99.5% STRAIN AFTER 62.3 HRS.

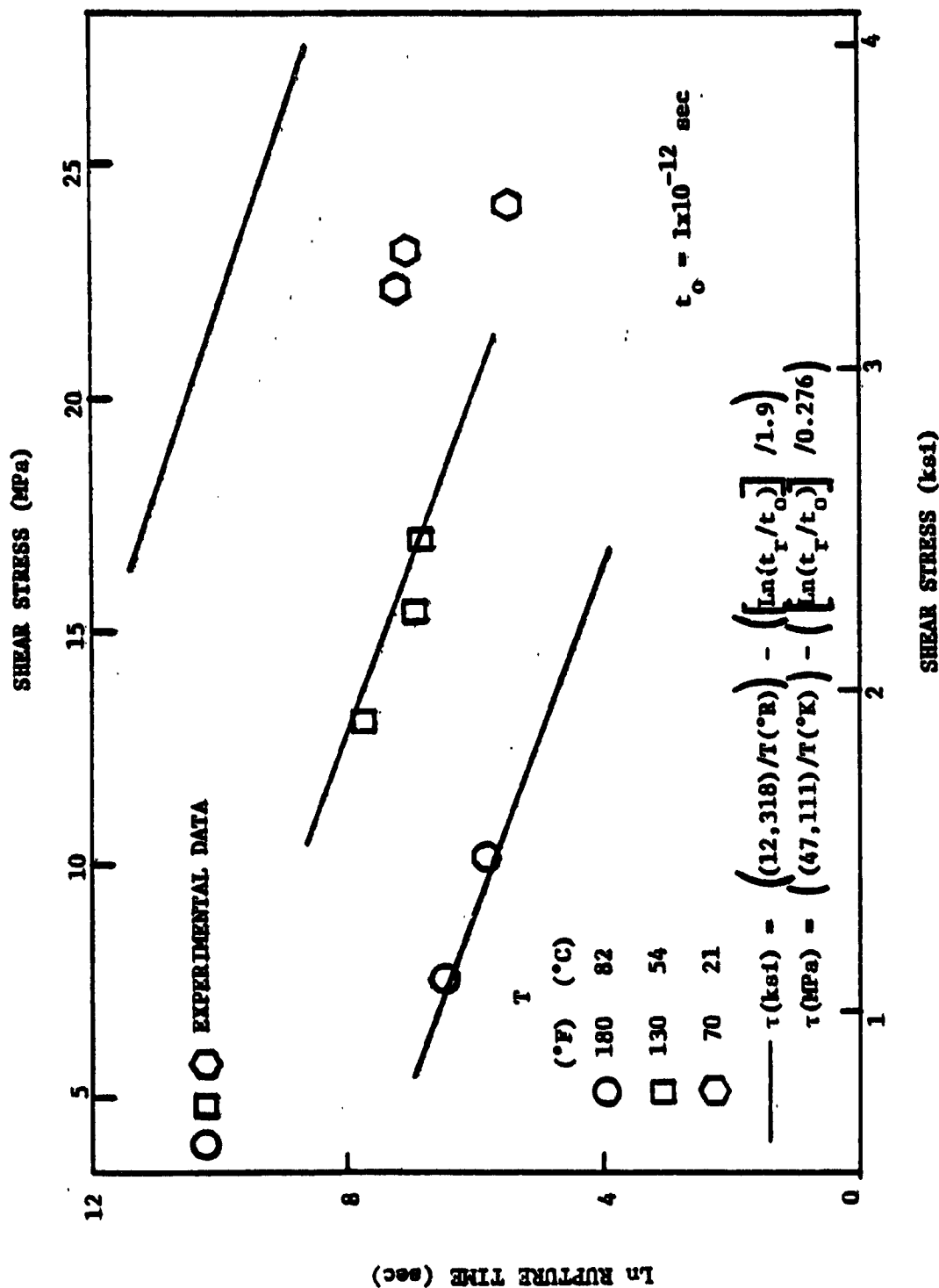τ_o = 785 psi (5.4 MPa), SPECIMEN REACHED 90% STRAIN AFTER 51.5 HRS.

τ_o = 2104 psi
   = 7.6 MPa

Figure 9. Creep Rupture Data and Comparison with Zhurkov's Equation for FM 73 Adhesive.

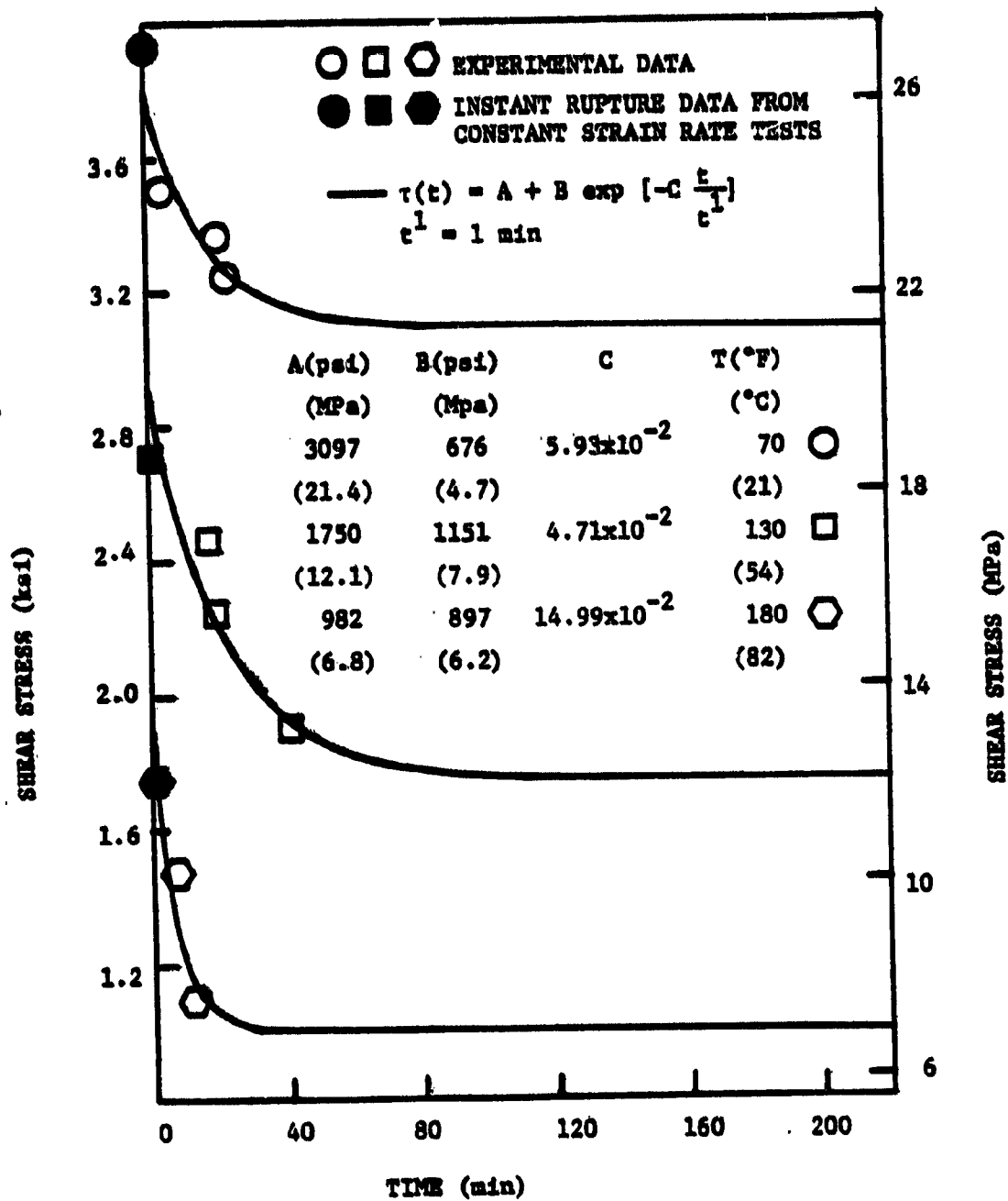**Figure 10. Creep Rupture Data and Comparison with Crochet's Equation for FM 73 Adhesive.**
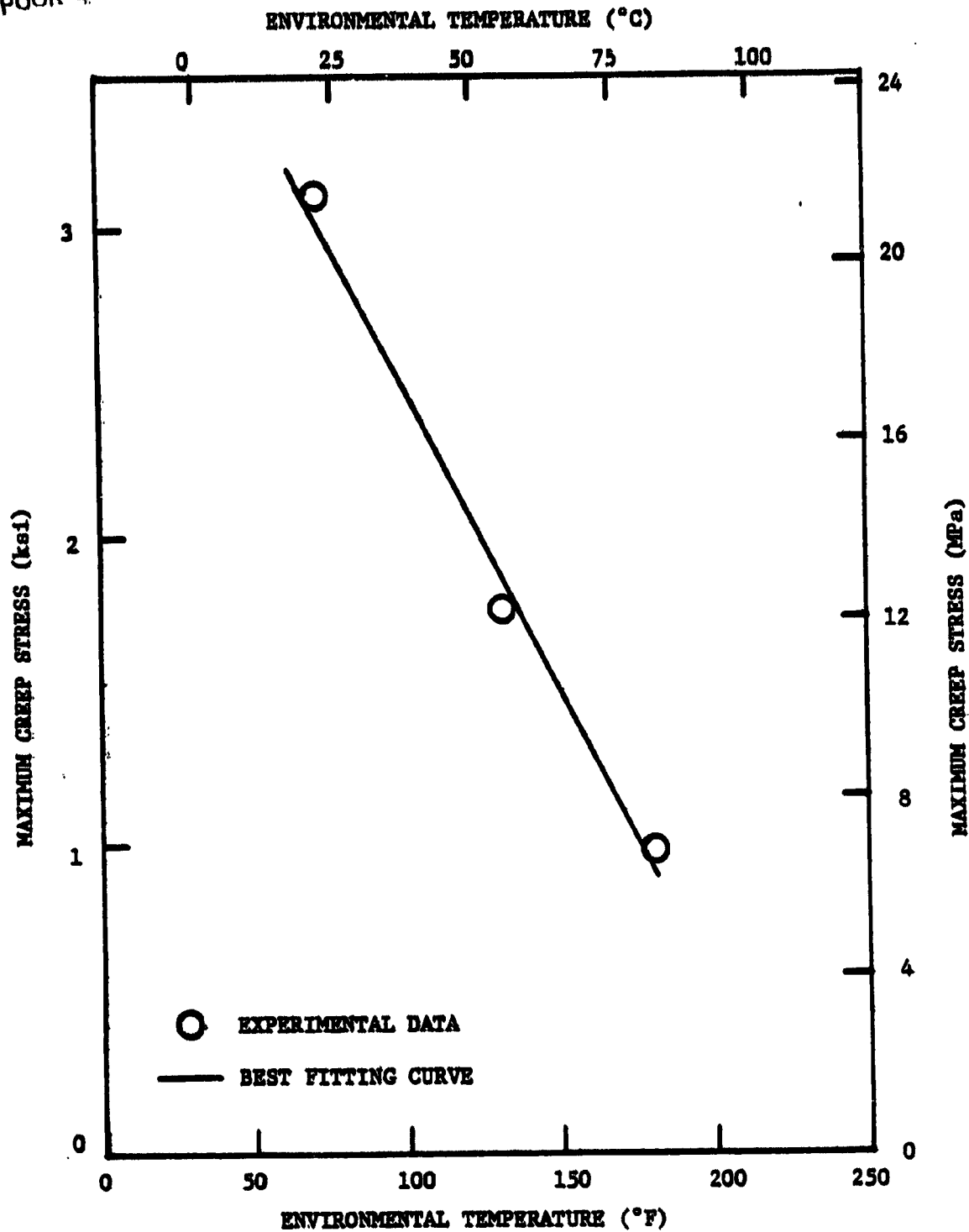
Figure 11. Variation of Maximum (safe) Creep Stress with
Environmental Temperature for FM 73 Adhesive.

and the adhesive's resistance to creep are characteristic of linear
thermoplastics. Figure 12 shows the typical shear stress-strain diagram
for the material in the lap shear mode. In this case the stress-strain
diagrams obtained with initial elastic strain rates of 24.6%/sec and 0.26%/sec
are almost coincidental. As can be seen in Figure 12, the power function
relation (Equation 16) provides a good fit to the experimental data with
the use of material constants shown. Rate dependence of maximum shear stresses
is shown in Figure 13. An increase of only ~8% in strength is observed when
the strain rate is increased ~100 times from 0.26%/sec to 24.6%/sec. It is
evident in the same figure that such variation is well within the scatter
band which would occur due to inherent flaws and/or variations in the
cure process. Ludwik's equation fitted to the experimental data is also
shown in Figure 13. The variation of maximum lap shear strains with respect
to initial elastic shear strain rates is shown in Figure 14. Apparently
scatter is more likely with the maximum strain values in comparison to the
behavior of maximum stresses. The overall average of the data, however,
suggests a relatively constant value of maximum shear strain over a wide
range of strain rates. An empirical equation fitted to describe the
experimental data is also shown in Figure 14.

The effects of temperature on the constant strain rate stress-strain
behavior of Polyimidesulfone is shown in Figure 15. The ultimate shear
stresses at 250°F (121°C) and 350°F (177°C) conditions are ~22% less than
the ultimate stress at 70°F (21°C) condition. Even though the magnitudes
of ultimate shear stresses are approximately the same, the 350°F (177°C)
condition results in a maximum shear strain level which is 0.147 in/in
higher i. magnitude than that obtained at 250°F (121°C) condition. At
450°F (232°C), we observe a ~44% drop in the ultimate stress and 0.147
in/in reduction in the maximum shear strain. We should note that, excluding
the 350°F (177°C) condition, a decrease in the levels of maximum shear
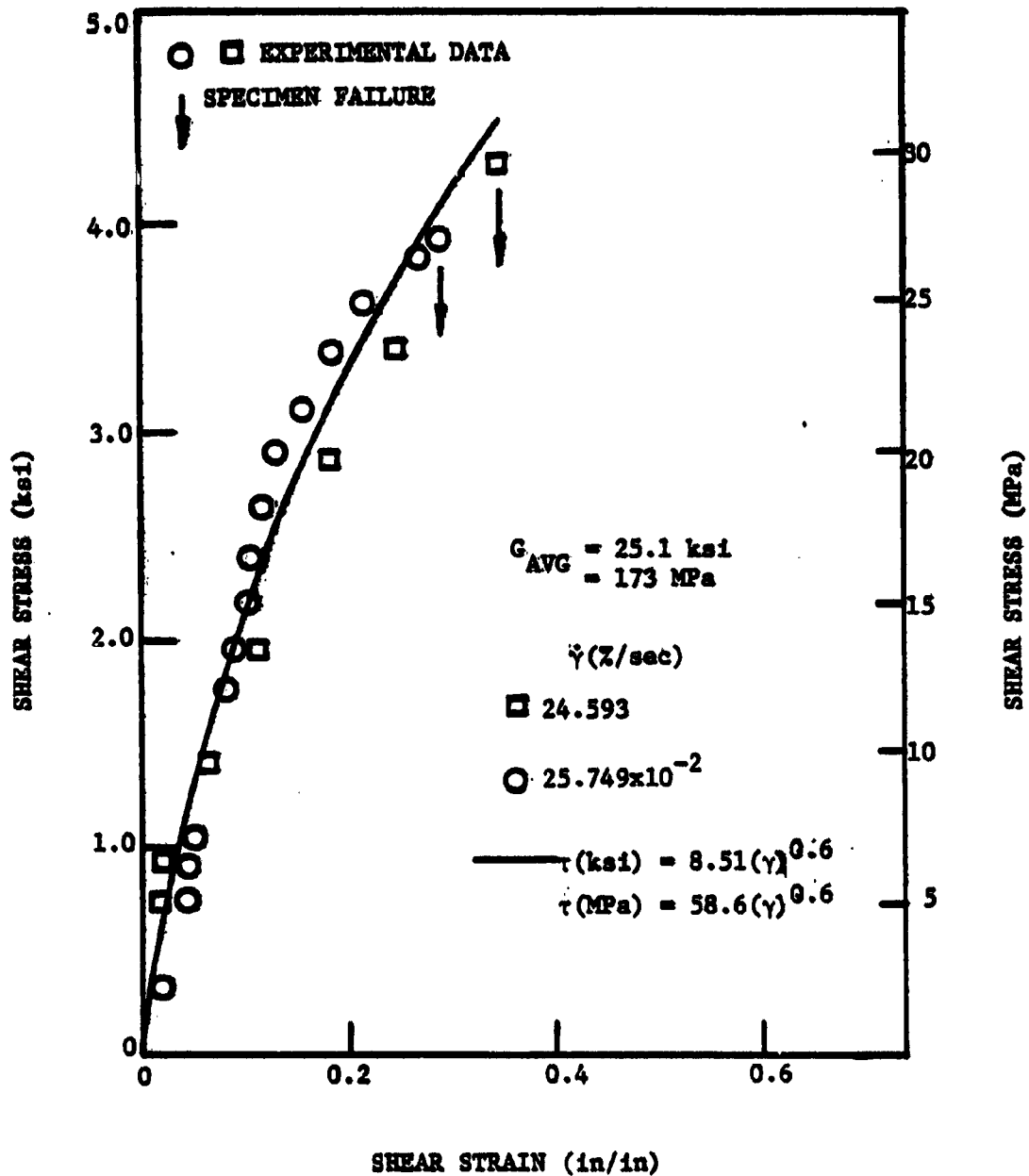
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Figure 12. Constant Strain Rate Stress-Strain
Behavior of Thermoplastic Polyimide-
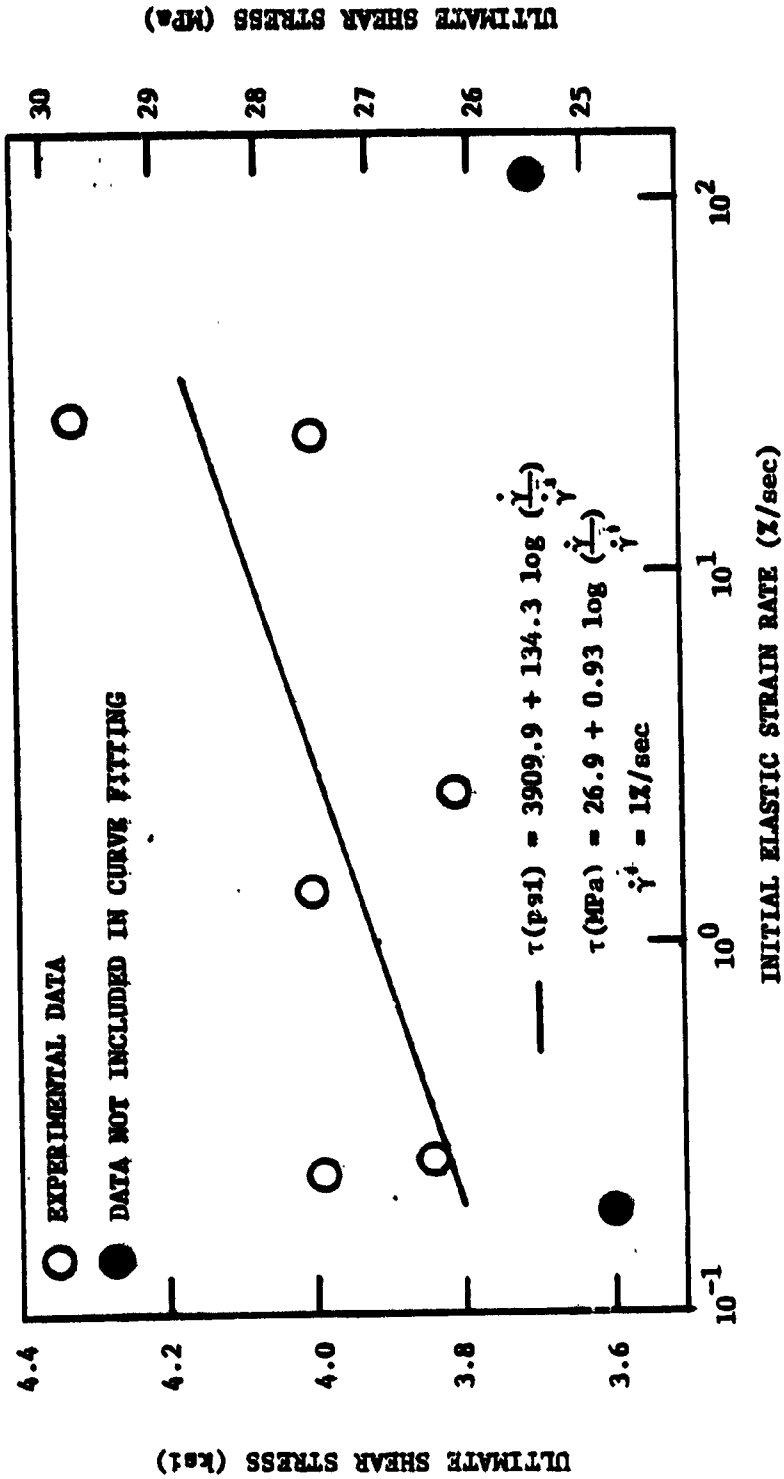sulfone and Comparison with Theory.

Figure 13. Variation of Ultimate Shear Stress with Initial Elastic Strain Rate and Comparison with Ludwik's Equation for Thermoplastic Polyimidesulfone Adhesive.
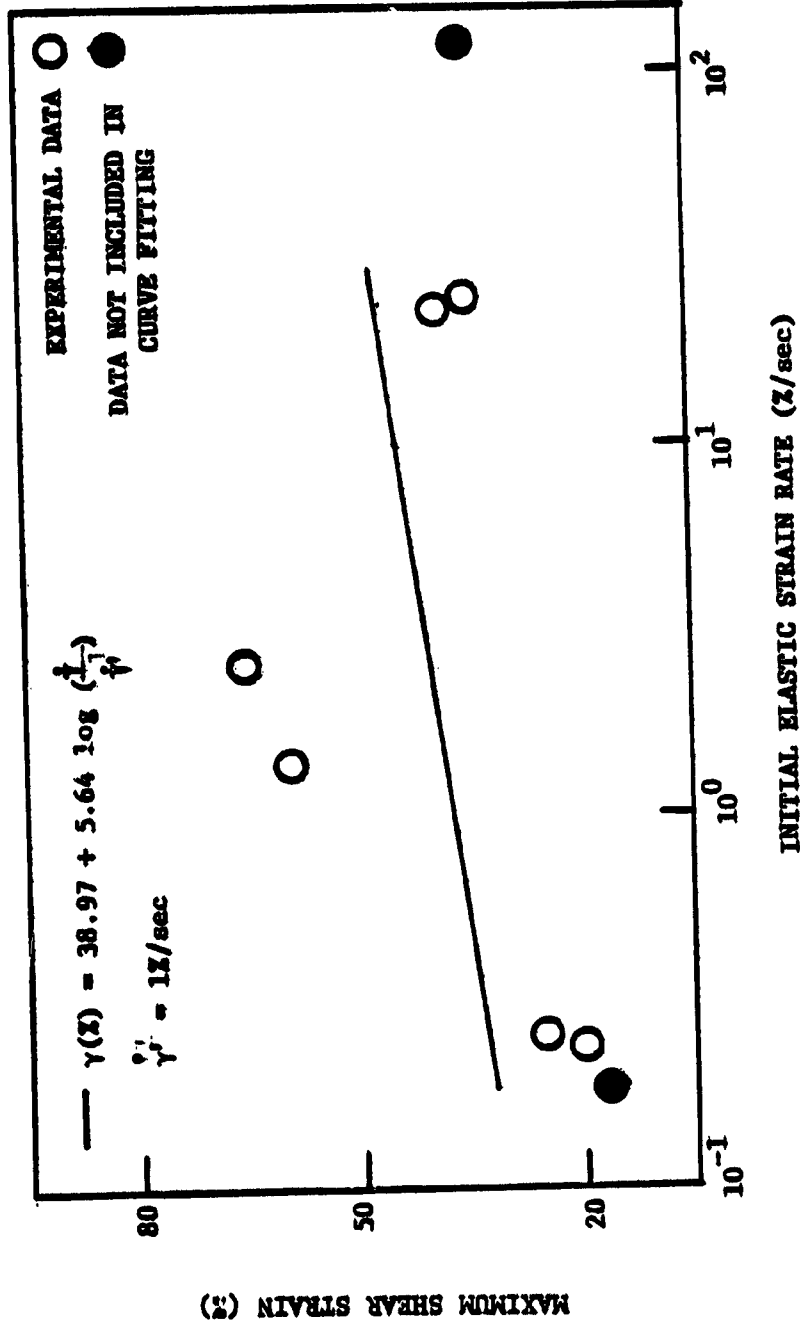
Figure 14. Variation of Maximum Shear Strain with Initial Elastic Strain
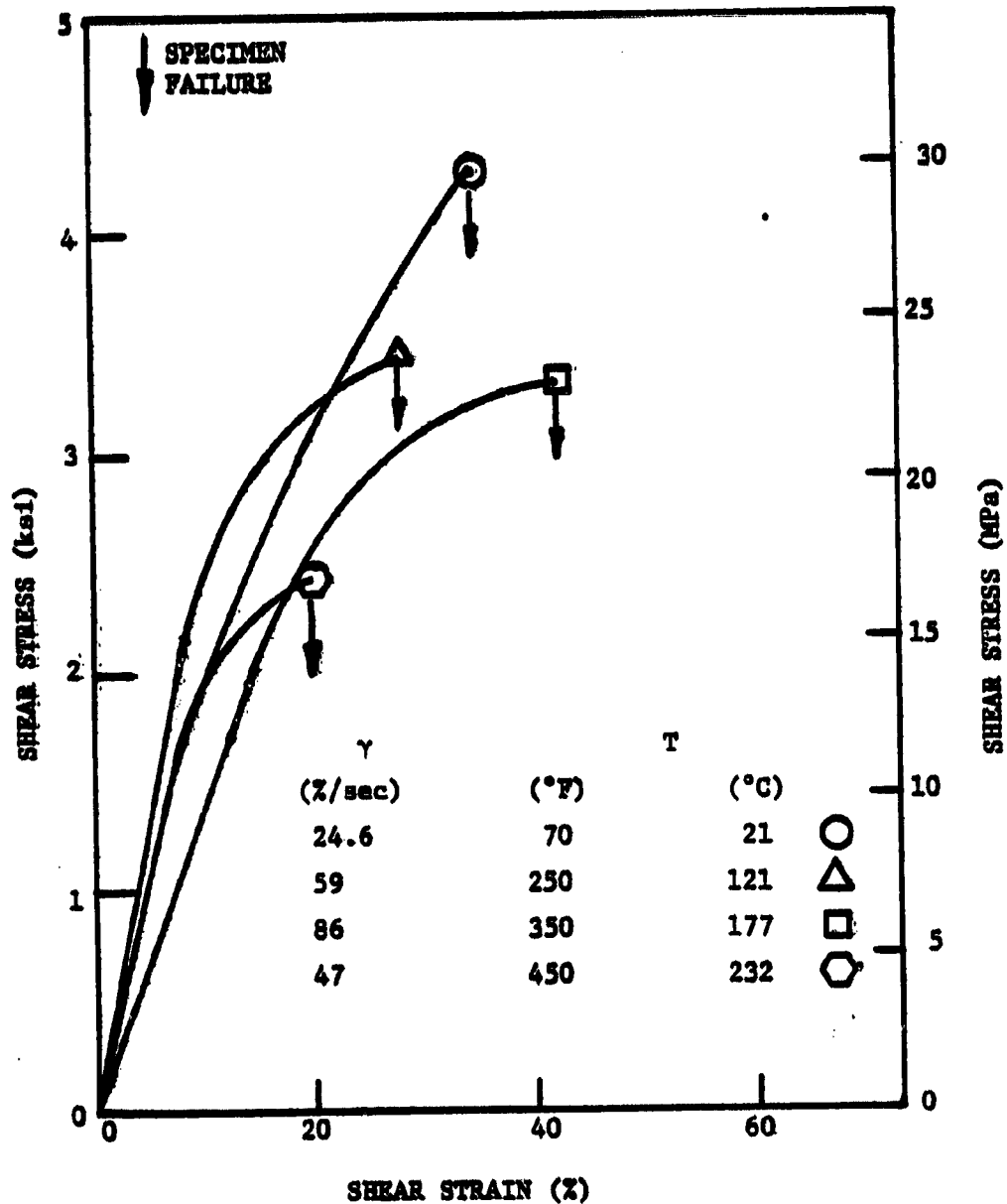Rate for Thermoplastic Polyimidesulfone Adhesive.

Figure 15. The Effects of Temperature on the
Constant Strain Rate Stress-Strain
Behavior of Thermoplastic Polyimide-
sulfone Adhesive.

strains is observed as the environmental temperature is increased. The authors attribute such reduction in the level of maximum shear strain to a change in the failure mechanism (possibly from adhesive matrix to adhesive - fiber and/or adhesive - adherend interfacial).

Figure 16 shows room temperature creep response of Polyimidesulfone. Apparently, creep occurs at relatively high levels of stress. The amount of creep strain, however, is quite small for a polymeric material, especially in the primary and secondary creep regions. Secondary creep rates are much lower than those for adhesives with comparable strength values. The presence of a tertiary creep region is evident. The scatter in the strain values which was observed in Figure 14 is also present in Figure 16. Evidently the level of initial strain (γ_o) reached for a given level of stress is what controls the oncoming creep process (compare creep behavior at $\tau_o = 3273$ psi - 22.6 MPa and $\tau_o = 3351$ psi - 23 MPa). Such scatter in strain levels suggests the presence of different failure mechanisms and is likely to occur with the presence of carrier cloth.

Figures 17, 18 and 19 show creep behavior of Polyimidesulfone at elevated temperatures. The common behavior observed is a general decrease in the levels of shear strains at failure when the environmental temperature is increased. Particularly, at 450°F (232°C) condition an almost constant level of creep strain to failure is evident. The reasons for decrease in the levels of maximum strain with increasing temperature can be deduced when one examines the post-failure surfaces of the specimens (Fig. 20). The extent of interfacial separation, especially adhesive-adherend (and also adhesive-fiber) separation increases with an increase in the environmental temperature. The gradual darkening of the post-failure overlap areas with increasing temperatures (in Figure 20) is an
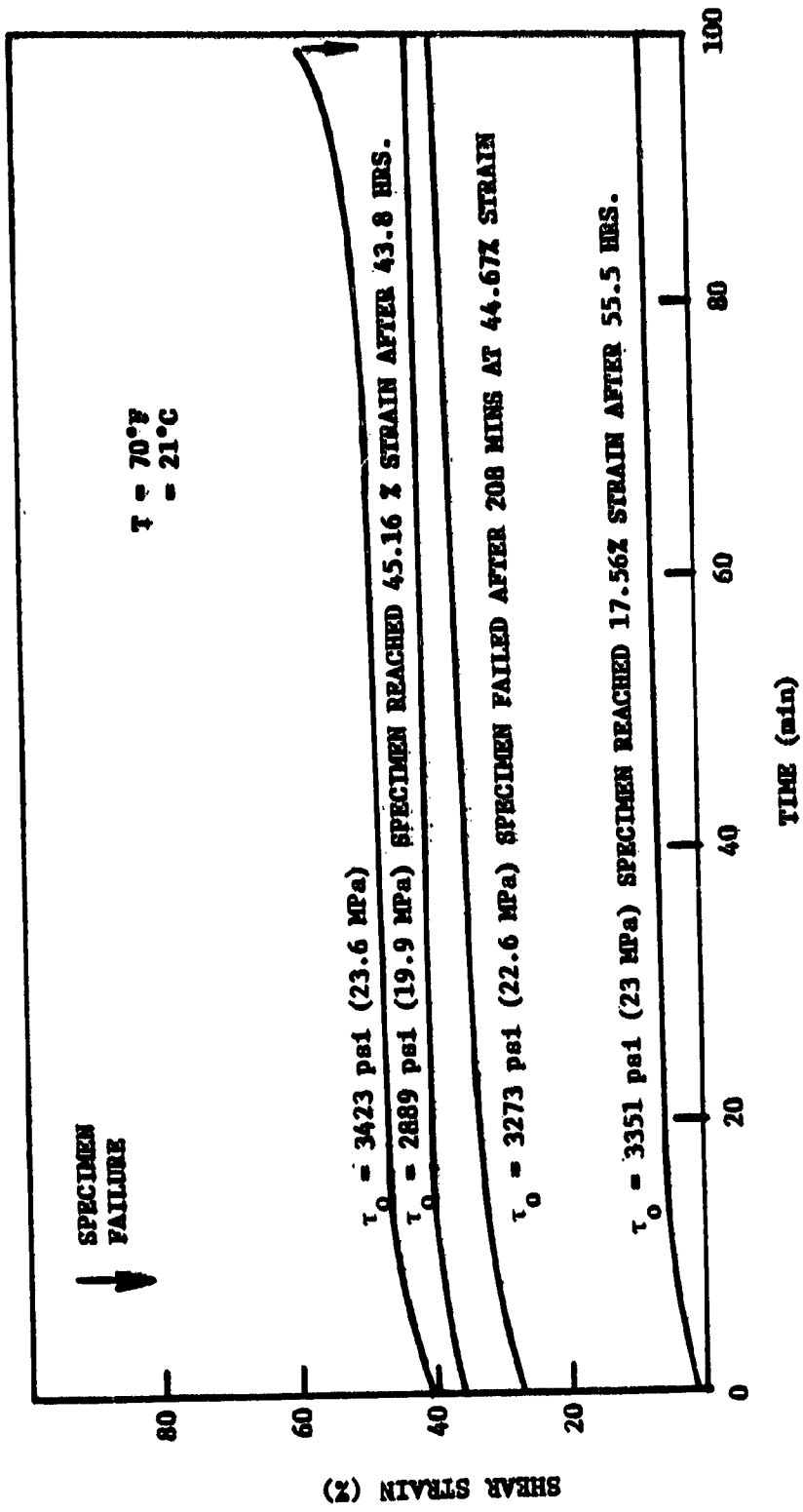
Figure 16. Room Temperature Creep Response of Thermoplastic Polyimidesulfone
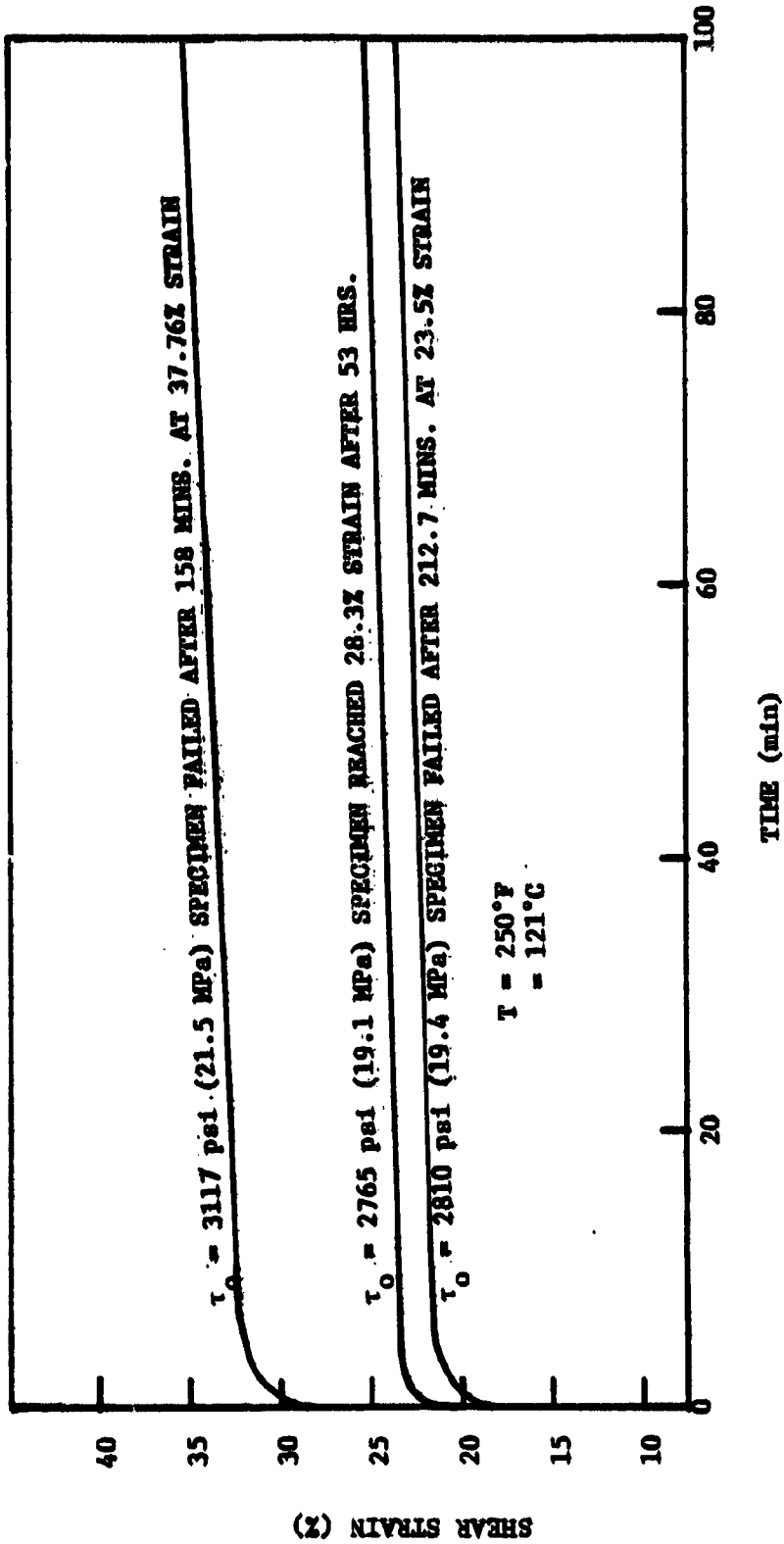Adhesive.

Figure 17. Creep Behavior of Thermoplastic Polyimidesulfone Adhesive at 250°F (121°C) Condition.
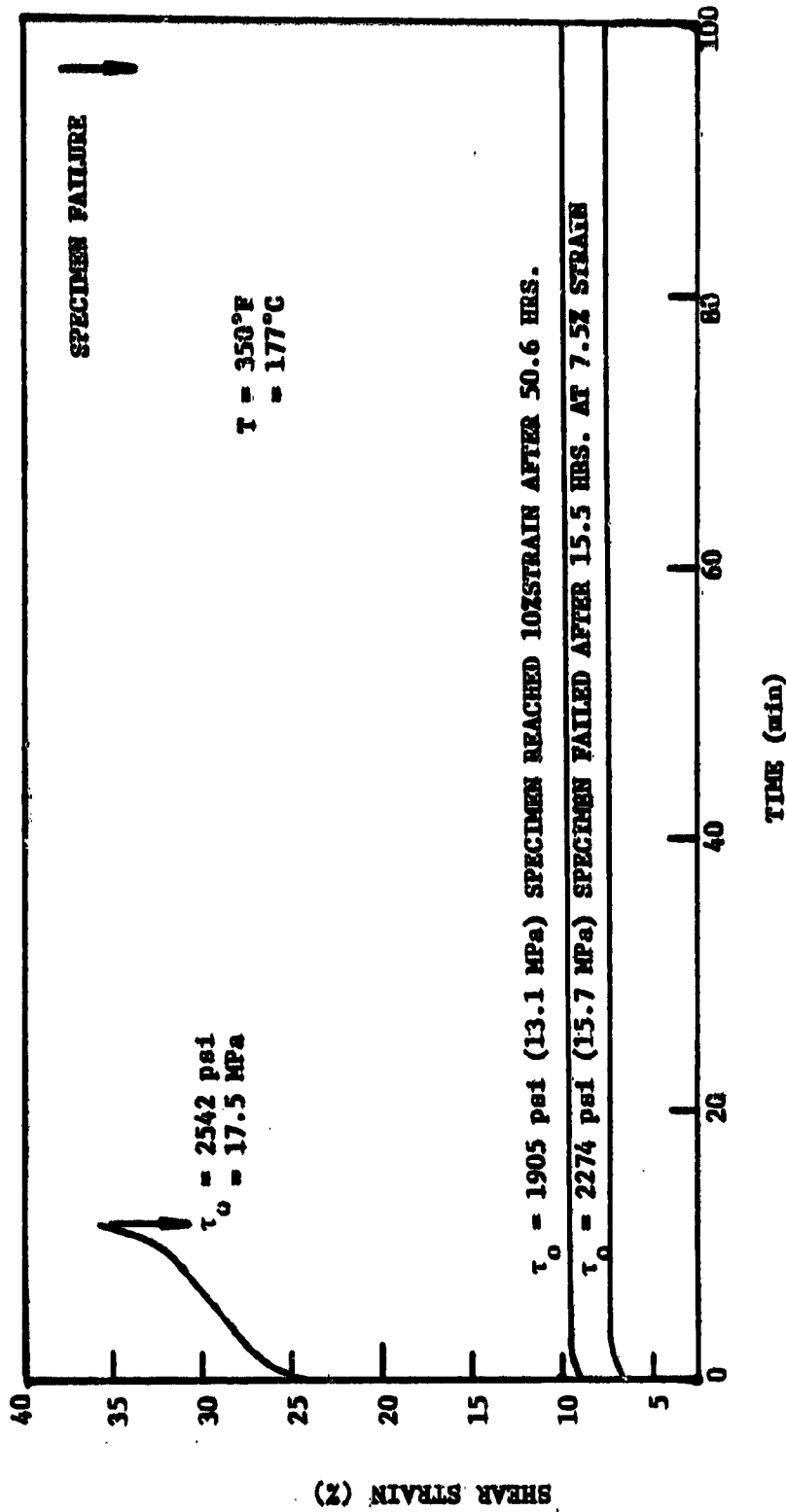
Figure 18. Creep Behavior of Thermoplastic Polyimidesulfone Adhesive at 350°F (177°C) Condition.
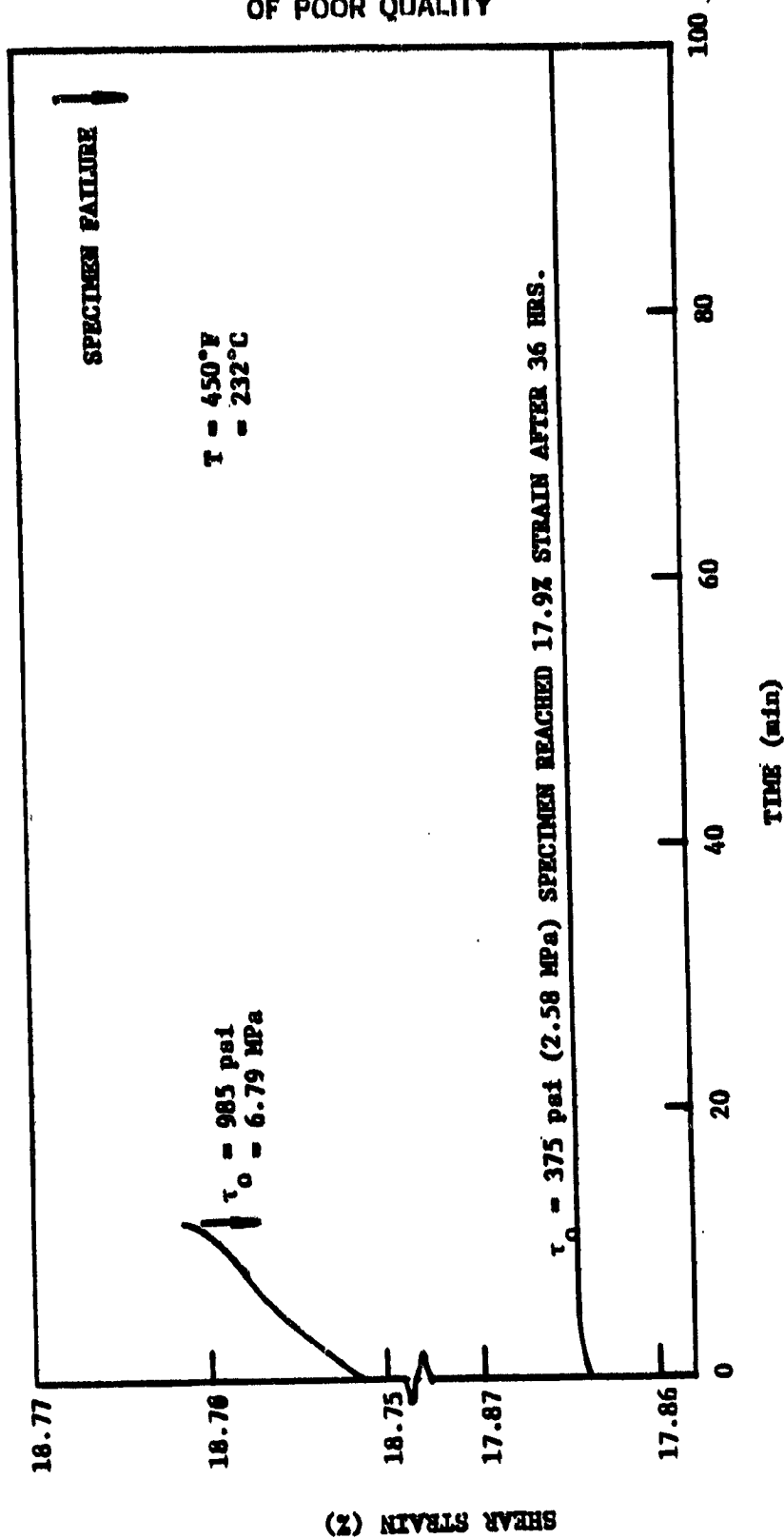
Figure 19. Creep Behavior of Thermoplastic Polyimidesulfone Adhesive at 450°F (232°C)
Condition.
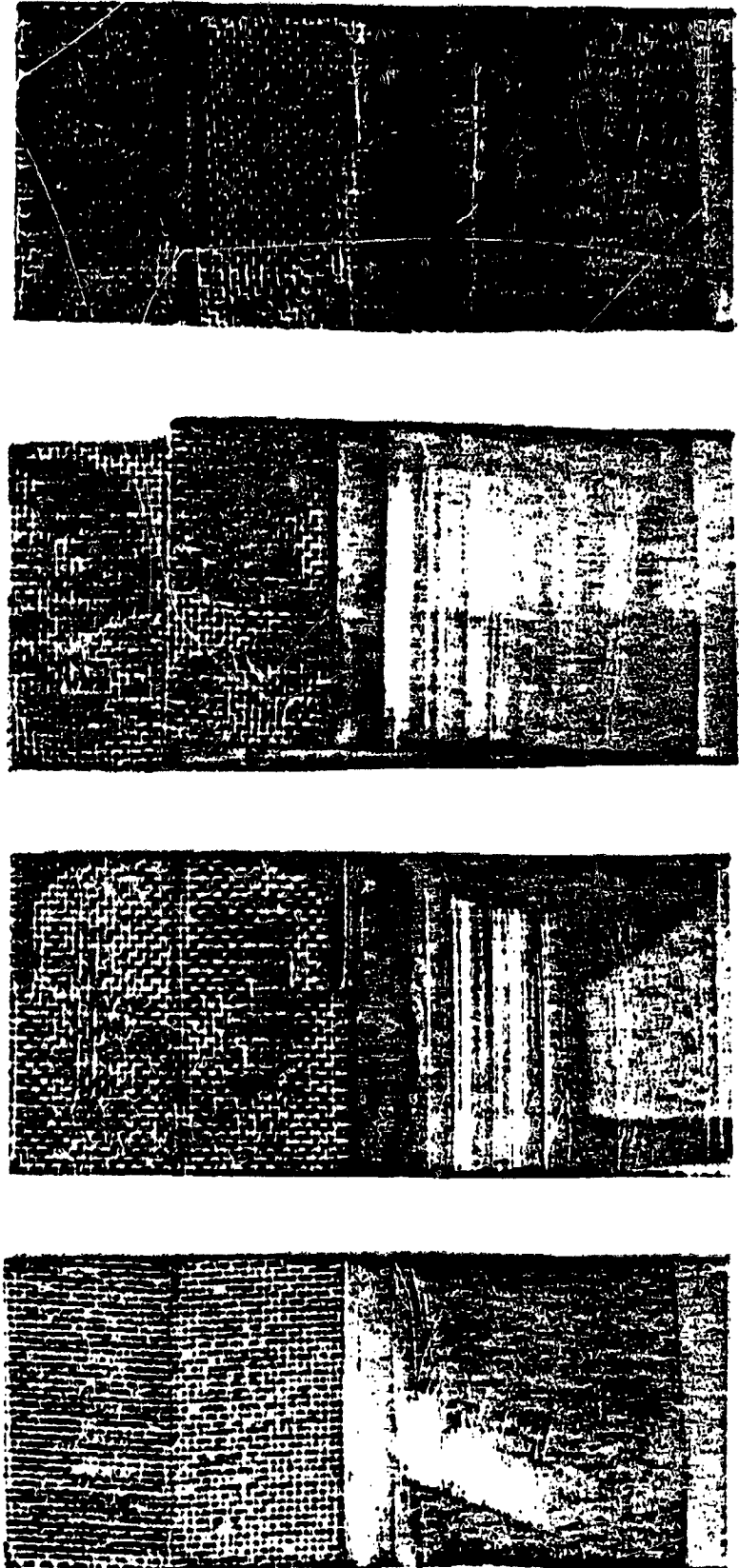
T = 450°F
= (232°F)

T = 350°F
= (177°C)

T = 250°F
= (121°C)

T = 70°F
= (21°C)

Figure 20. Typical Fracture Surfaces of Titanium-Polyimidesulfone Specimens Exhibiting
the Effects of Increasing Temperature.

indication of adhesive matrix layers which have separated from the adherend and/or the carrier cloth.

Figure 21 shows comparison of experimental data with Zhurkov's creep rupture relation (Equation 3). Apparently, Zhurkov's equation does not provide a good fit to the experimental data. The discrepancy between the theory and data becomes greater at room temperature and also at 450°F (232°C) conditions. Comparison of the creep rupture data with Crochet's equation (Equation 6) is shown in Figure 22 along with the appropriate constants used to fit the data. The authors believe that Crochet's equation provides a better fit to the experimental data in comparison to Zhurkov's equation. Figure 23 shows variation of the maximum - or safe - level of creep stress values (i.e. material constants A in Figure 22) with environmental temperature. Experimental results indicate a sharp decrease in the level of maximum creep stress for temperatures above 350°F (177°C).

SHEAR STRESS (MPa)

EXPERIMENTAL DATA ⬡ △ □ ○

	τ	
(°F)	(°C)	
450	232	○
350	177	□
250	121	△
70	21	⬡

$$\tau(ksi) = \frac{[4397)/T(°R)]}{[\ln(t_r/t_o)]}/10.13$$

$$\tau(MPa) = \frac{[(\ ,197)/T(°K)]}{[\ln(t_r/t_o)]}/0.68$$

$t_o = 1 \times 10^{-12}$ sec

SHEAR STRESS (ksi)
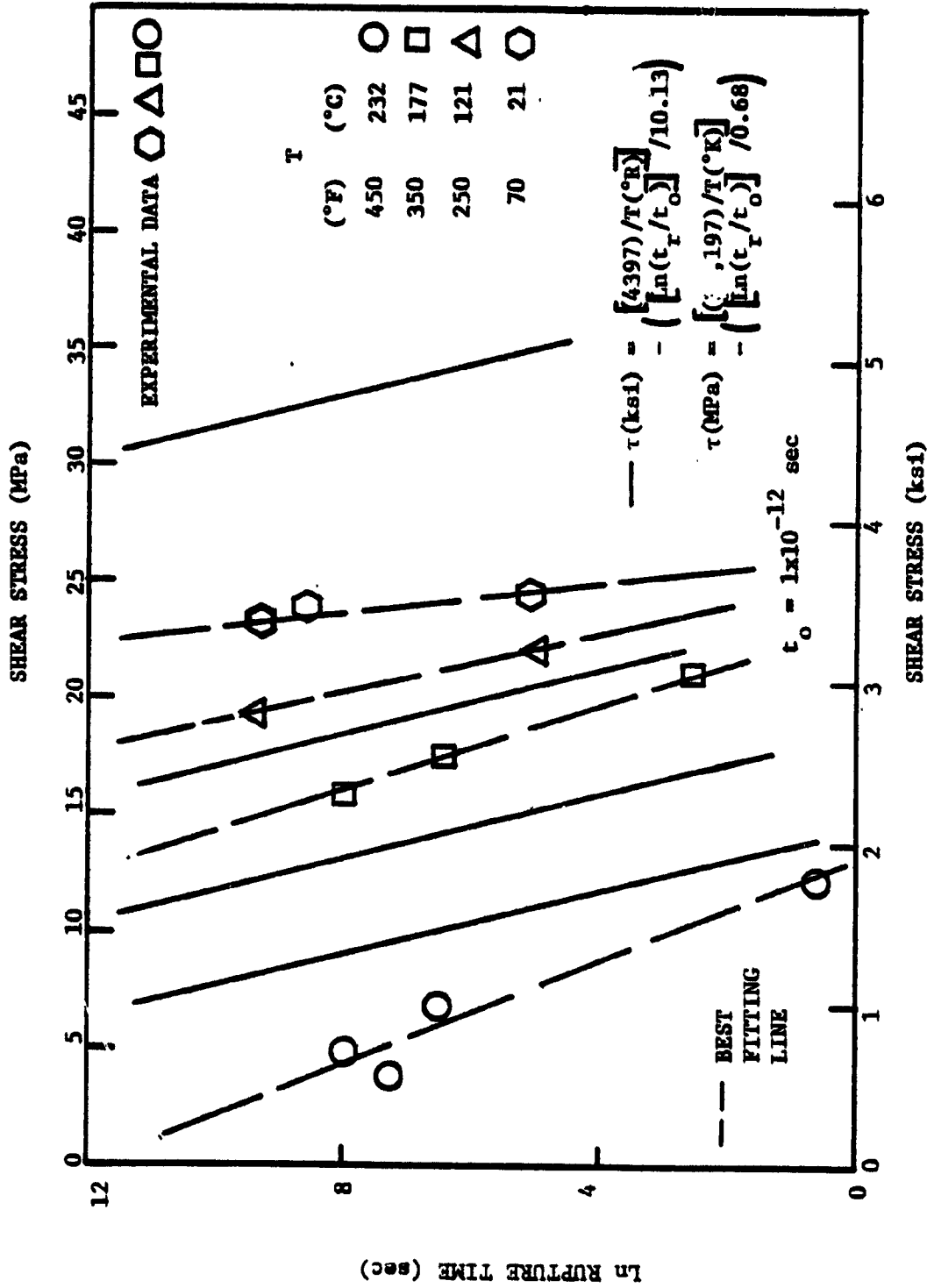
—— BEST
- - - FITTING
LINE

Ln RUPTURE TIME (sec)

Figure 21. Creep Rupture Data and Comparison with Zhurkov's Equation for Thermo-plastic Polyimidesulfone Adhesive.
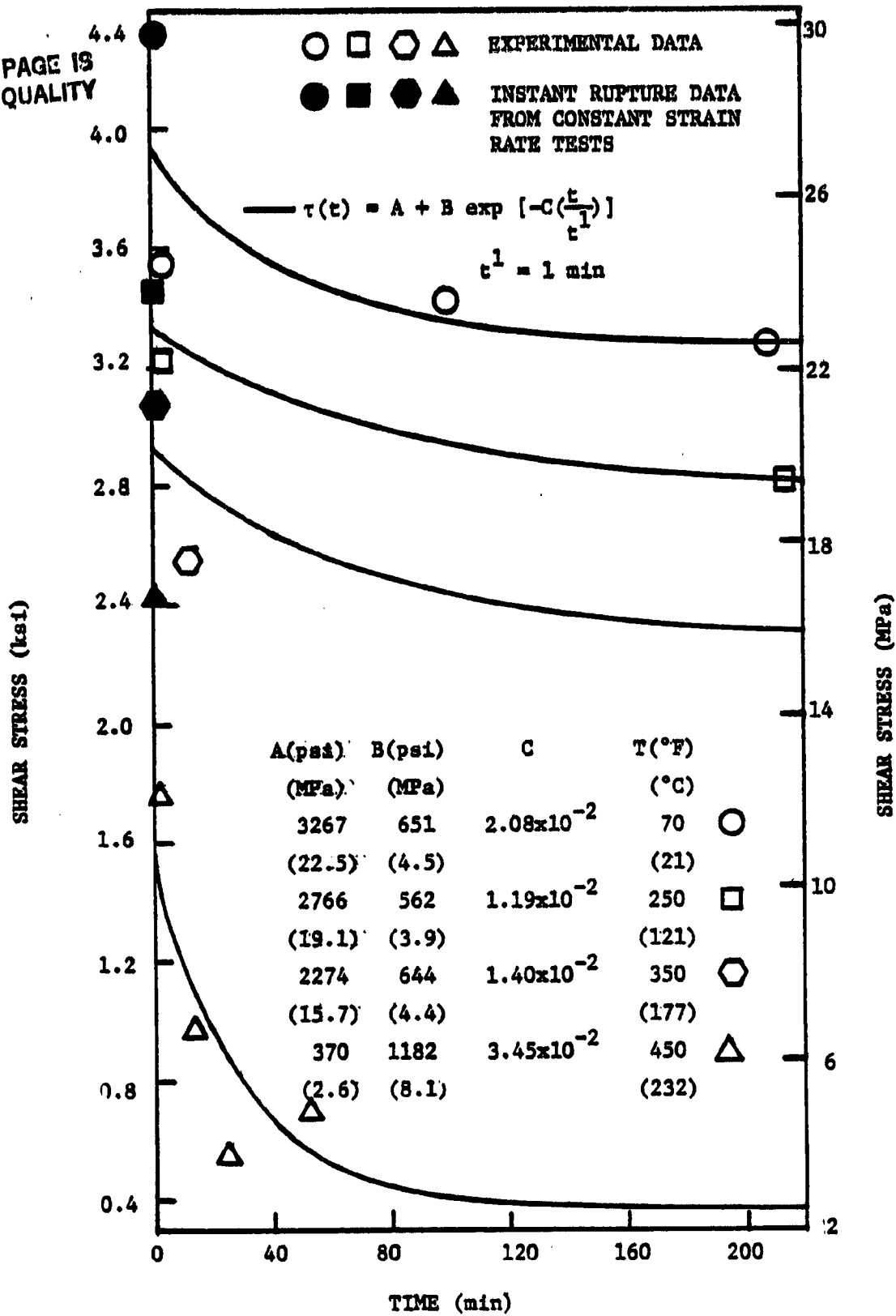
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| ○ □ ⬡ △ | EXPERIMENTAL DATA |
| ● ■ ⬢ ▲ | INSTANT RUPTURE DATA FROM CONSTANT STRAIN RATE TESTS |

$$\tau(t) = A + B \exp\left[-C\left(\frac{t}{t^1}\right)\right]$$

$$t^1 = 1 \text{ min}$$

A(psi) (MPa)	B(psi) (MPa)	C	T(°F) (°C)	
3267 (22.5)	651 (4.5)	2.08×10^{-2}	70 (21)	○
2766 (19.1)	562 (3.9)	1.19×10^{-2}	250 (121)	□
2274 (15.7)	644 (4.4)	1.40×10^{-2}	350 (177)	⬡
370 (2.6)	1182 (8.1)	3.45×10^{-2}	450 (232)	△

SHEAR STRESS (ksi)

SHEAR STRESS (MPa)

TIME (min)

Figure 22. Creep Rupture Data and Comparison with
Crochet's Equation for Thermoplastic
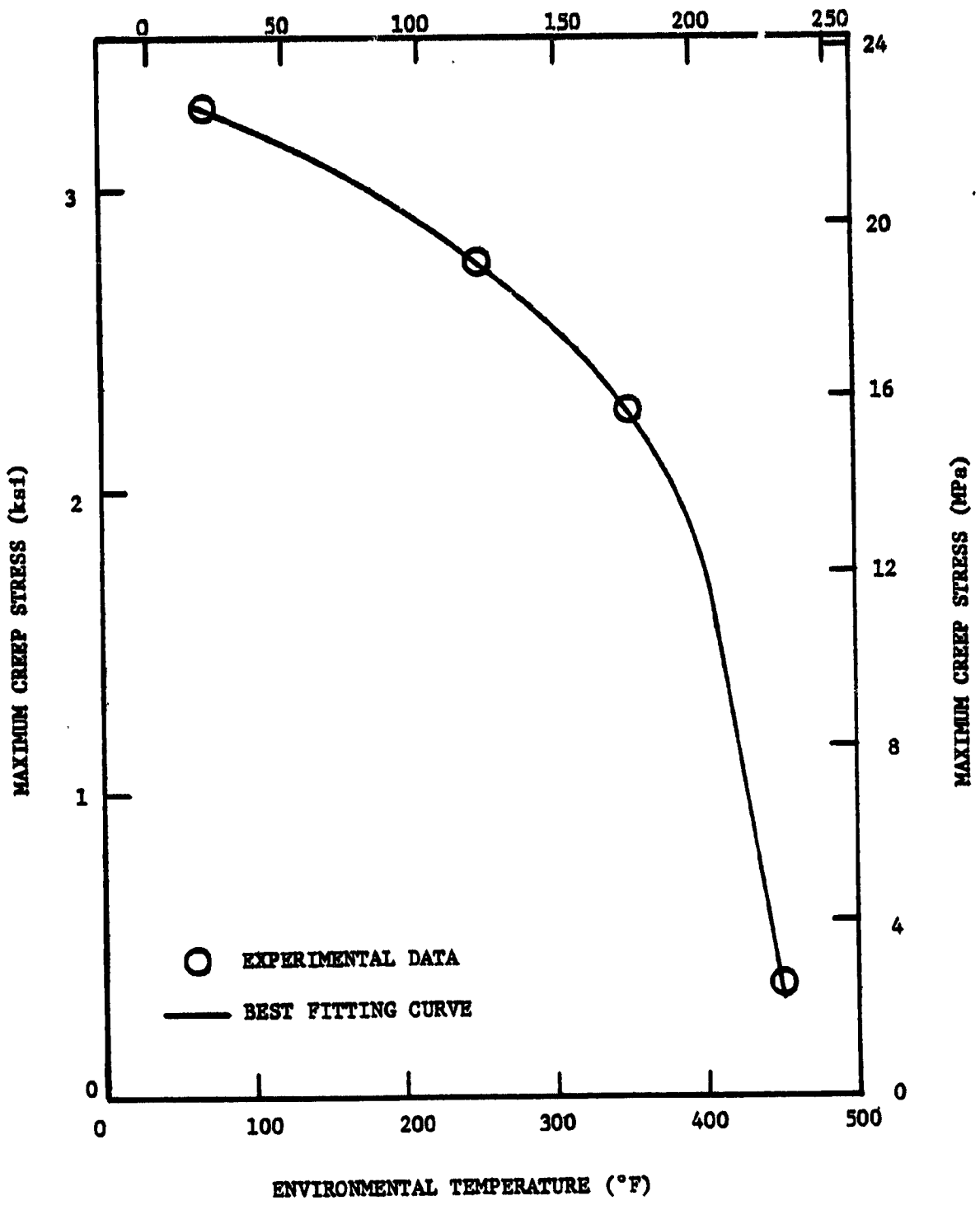Polyimidesulfone Adhesive.

Figure 23. Variation of Maximum (safe) Creep Stress with Environmental Temperature for Thermoplastic Polyimidesulfone Adhesive.

CONCLUSIONS

The amount of data available at this stage of the investigation does not permit any conclusive assessment of the proposed characterization method. The authors hope, however, that as more adhesives are tested, a practical method will emerge to enable material characterization of structural adhesives in the bonded form.

Based on the data from the two adhesives tested, the following (preliminary) conclusions can be drawn:

.It is possible to describe the constant strain rate shear stress-strain behavior of structural adhesives in the bonded form by using viscoelastic or nonlinear elastic relations.

.Ludwik's equation provides an adequate description of the rate dependent adhesive ultimate shear stresses.

.Crochet's equation provides a better description of the temperature dependent creep rupture data in comparison to Zhurkov's equation.

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