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PREDICTION OF FAILURE TIME FOR SOME ADHESIVE-BONDED JOINTS

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JULY 1970

PICATINNY ARSENAL
DOVER, NEW JERSEY

Technical Report 4006

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by

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TABLE OF CONTENTS

		Page No.
Object		1
Summary		1
Introduction		1
Results and Discussion		1
Experimental Procedures		6
Acknowledgements		8
References		8
Distribution List		20
Tables		
1	Mechanical property data for Sample I	10
2	Mechanical property data for Sample II	12
3	Mechanical property data for Sample III	14
4	Mechanical property data for Sample IV	15
5	Calculated and experimental t_f values	16
Figures		
1	Data for Sample I, plotted in accordance with Equation 10	17
2	Coehesive failure data, in accordance with Equation 10	18
3	Arrhenius - type plots	19

OBJECT

The object of this investigation was to determine whether limited mechanical property data on adhesive bonds can be used for the estimation of failure times for such bonds.

SUMMARY

Data are given for shear and tensile testing of adhesive bonds under a constant rate of loading. A rate equation is then used to predict useful life from the mechanical data. The correlations are in general quite satisfactory, providing that the failure is cohesive[1] within the adhesive.

INTRODUCTION

Applications of reaction rate theory to polymer mechanical behavior have been carried out in a number of laboratories (Refs 1-4). We have successfully applied such methods to propellants (Ref 5), to various thermoplastics (Ref 6), and to glass-reinforced polymers (Ref 7). An additional application of considerable interest to the Army is in the field of adhesive bonds. This report describes the initial efforts at predicting the lifetimes of such bonds from limited mechanical property testing.

RESULTS AND DISCUSSION

The lifetime of a material subjected to mechanical restraint has been considered to be a process which proceeds according to a rate equation (Refs 1, 2). By integrating the rate equation and making certain reasonable assumptions it is possible to obtain an

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[1] In this work, cohesive failure always refers to failure within the adhesive layer

expression for constant rate of loading of the form (Ref 2):

$$\ln t_f - \ln S = \ln BA - BS \tag{1}$$

$$A = \frac{\gamma_b h}{\lambda kT} e^{\Delta F\neq/RT}$$

$$B = \sigma/2kT$$

where t_f is failure time, S is stress, γ_b is mean relative displacement, h is Planck's constant, λ is jump distance, $\Delta F\neq$ is free energy of activation, R is gas constant, T is the absolute temperature, σ is displacement volume, and k is Boltzmann's constant.

Putting A and B in (1), taking $\Delta F\neq = \Delta H\neq - T\Delta S\neq$ we may write

$$\log\left(\frac{t_f T^2}{S}\right) = C + \frac{\Delta H\neq}{2.3RT} - b(S/T) \tag{2}$$

where C and b are constants.

At constant temperature, the experimental data should give a straight line on plotting log (t_f/S) versus S/T according to

$$\log \frac{t_f}{S} = D - b(S/T). \tag{3}$$

The apparent activation energy may then be evaluated by extrapolating several constant temperature lines to the vertical intercept (S/T = O) and making an Arrhenius type plot in accordance with

$$\log\left(\frac{t_f T^2}{S}\right) = C + \frac{\Delta H\neq}{2.3RT}\ . \tag{4}$$

In a study of this sort, it appeared to be desirable to establish the feasibility of the approach by using available data. Then, if the method appears to be useful, an experimental program can be set up. The data selected for analysis in this initial study had been collected in these laboratories several years ago. The samples, designated

I, II, III, and IV, may be briefly identified as follows:

I. Nylon epoxy - Narmco 406 (temperature rise 10-12.5°/min). Reclaimed steel adherends.

II. NOL specimen (Ref 7a) Narmco 406 (temperature rise 10-12.5°/min). Reclaimed steel adherends.

III. Narmco 406 nylon epoxy lap shear (13.5°F/min cure). Reclaimed steel adherends.

IV. FM 97 lap shear. Reclaimed steel adherends.

Details of the experiments are given in the Experimental Procedure section of this report. The data are given in Tables 1-4.

Since isothermal data were lacking for all of the samples (I-IV), Equation 3 could not be used in evaluation of parameters. In a similar case, propellant data were correlated by making a plot of log $t_fT + b(S/T)$ versus $1/T$ and using trial values of b to find the case where the plot is linear (Refs 5, 8). Since such a method is quite tedious and laborious, it seemed desirable to develop an alternative procedure.

If we multiply Equation 2 through by T, we get

$$T \log\left(\frac{t_f T^2}{S}\right) = CT + \frac{\Delta H^{\neq}}{2.3R} - bS. \qquad (5)$$

At a data point T_1, t_{f_1} and S_1

$$T_1 \log\left(\frac{t_{f_1} T_1^2}{S_1}\right) = CT_1 + \frac{\Delta H^{\neq}}{2.3R} - bS_1 \qquad (6)$$

and similarly at data point T_2, t_{f_2} and S_2

$$T_2 \log \frac{t_{f_2} T_2^2}{S_2} = CT_2 + \frac{\Delta H^{\neq}}{2.3R} - bS_2. \qquad (7)$$

Assuming the constancy of $\Delta H^{\neq}$,

$$\frac{\Delta H^{\neq}}{2.3R} = T_1 \log\left(\frac{t_{f_1} T_1^2}{S_1}\right) - CT_1 + bS_1 = T_2 \log\left(\frac{t_{f_2} T_2^2}{S_2}\right) - CT_2 + bS_2. \qquad (8)$$

Hence,

$$T_1 \log\left(\frac{t_{f_1} T_1^2}{S_1}\right) - T_2 \log\left(\frac{t_{f_2} T_2^2}{S_2}\right) - C(T_1 - T_2) - b(S_2 - S_1) = 0 \qquad (9)$$

and, on dividing through by $T_1 - T_2$,

$$\frac{T_1}{T_1 - T_2} \log\left(\frac{t_{f_1} T_1^2}{S_1}\right) - \frac{T_2}{T_1 - T_2} \log\left(\frac{t_{f_2} T_2^2}{S_2}\right) = C + b\left(\frac{S_2 - S_1}{T_1 - T_2}\right). \qquad (10)$$

For every possible plan of data points, the left hand side of Equation 10 may be plotted against $(S_2 - S_1)/(T_1 - T_2)$. C and b are then evaluated from the intercept and slope, respectively. After C and b are determined, we may go back to Equation 2 in the form,

$$\log\left(\frac{t_f T}{S}\right) - C + b(S/T) = \frac{\Delta H^{\neq}}{2.3RT}. \qquad (2a)$$

The left-hand side of Equation 1a is plotted against $1/T$ to evaluate $\Delta H^{\neq}$.

All possible pairs of points were taken for Sample I and are plotted according to Equation 10 in Figure 1. Obviously, the plot is not satisfactorily linear. The same sort of behavior was observed for Samples II and III. Examination of the data indicated that the mode of failure is cohesive at the lower temperatures but becomes largely adhesive at the highest temperatures. It seemed reasonable to postulate that the one set of parameters could not describe both the cohesive and adhesive failures. Hence, the data at $344^{\circ}K$ and $366^{\circ}K$ were omitted for Samples I, II, and III. The appropriate plots for cohesive failure are shown in Figure 2. Considering the usual adhesive data scatter, these plots appear to be satisfactorily linear.

Figure 3 shows the Arrhenius type plots drawn according to Equation 2a. From the slope of the plots, $\Delta H^{\neq}$ was evaluated in each case. These values were surprisingly low, falling in the range of 1 kcal/mole (see Equations 11 through 14 below).

After the appropriate parameters were evaluated as discussed above, the values obtained were put back into Equation 2 to give the relationships between failure time, stress, and temperature in each case. The equations are:

Sample I

$$\log t_f = \log S - 2 \log T + 4.28 - 195/T - 0.00086 (S/T) \tag{11}$$

Sample II

$$\log t_f = \log S - 2 \log T + 4.29 - 213/T - 0.00022 (S/T) \tag{12}$$

Sample III

$$\log t_f = \log S - 2 \log T + 4.20 - 155/T - 0.00467 S/T \tag{13}$$

Sample IV

$$\log t_f = \log S - 2 \log T + 4.51 - 241/T - 0.00422 S/T \tag{14}$$

As a further check on the validity of the treatment, Equations 11 through 14 were used to calculate log t_f at the temperatures and stresses at which the experimental values had been recorded. Table 5 shows the results. It is noteworthy that in every case the agreement is good.

EXPERIMENTAL PROCEDURES

Shear Specimens (Samples I, III, IV)

Materials

Reclaimed 1020 steel coupons, 1 inch wide by 4 inches long by 1/8 inch thick, were used to prepare specimens with 1/2 inch overlap.

Metlbond 406, an unsupported nylon-epoxy adhesive tape, was used to bond the Sample I and III specimens.

FM 97, a modified epoxy adhesive supported on a light glass fabric, was used to bond the Sample IV group.

Adherend Preparation

The steel coupons were immersed in toluene and washed with a cloth to remove preservative oils. They were then washed in acetone to remove any remaining contamination; vapor degreased in the hot vapors of stabilized perchloroethylene; cooled to room temperature in a dessicator charged with silica gel; and stored in the dessicator until used.

Specimen Preparation

Individual coupons were marked with a scribe to establish the 1/2 inch overlap. An aluminum jig was used to maintain alignment during the cure of the adhesive. The jig was made of 1/2 inch aluminum plate with 1/8 inch brass pins press-fitted into holes drilled and geometrically arranged to provide for accurate alignment of the specimens. Four pins on each side and one pin at each end restricted the coupons during the curing operation. Unused coupons were placed under the overlapped portion of the specimen to provide support and alignment. A single layer of the adhesive being tested was used in the joint. The assembly was placed in a hydraulic press and sufficient pressure was applied to assure 25 psi in each of the adhesive joints. The platens of the press were electrically heated. Two different heating rates were achieved by either using or omitting pressure distribution pads between the platens and the specimen assembly. Resilient long-fiber asbestos pads were used to achieve the 10° - 12.5°/min heating rate. The faster heating rate of 13.5°/min was obtained without asbestos pads. In each case, cure was started with cold platens; and the temperature of $350^{\circ}F$ was held for one hour after the bond line reached this temperature. The temperature was monitored with thermocouples in the joint area of the assembly. Calibration of the press and assembly for temperature rise and control preceded their use for this investigation.

Tensile Specimens (Sample II)

Materials

Reclaimed 1020 steel tensile pieces, 1/2 inch in diameter by 2 inches long were bonded end-to-end to form the NOL tensile specimens.

The Metlbond 406 described above was the adhesive used.

Adherend Preparation

The steel rod adherends were prepared similarly to the shear coupons described above.

Specimen Preparation

The NOL tensile specimens were prepared in a jig wherein the individual rods were butted end-to-end and rigidly constrained. A single wafer of the adhesive film was placed between the surfaces, and a five-pound lead weight was suspended on the top rod section to provide the required 25 psi cure pressure. The entire assembly was heated to cure temperature in an air-circulating oven.

Testing

The static testing was conducted using a 60,000-pound Baldwin test machine. The load was 1,300 pounds/in^2./min for both tensile and shear tests.

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TABLE 1

Mechanical property data for Sample I

Test Temperature ^{o}K	Shear Strength, psi Individual Values	Average	Failure Time (t_f), min Individual Values	Average
200	6380		4.91	
	6800		5.23	
	7200		5.54	
	7000	7090	5.38	5.45
	6800		5.23	
	8160		6.28	
	7270		5.59	
219	6220		4.78	
	5800		4.46	
	7440		5.72	
	6250	6580	4.81	5.06
	7250		5.58	
	6540		5.03	
	6570		5.05	
250	7340		5.64	
	7400	7385	5.69	5.68
	7420		5.71	
	7380		5.68	
283	5280		4.06	
	5720	5400	4.40	4.15
	5200		4.00	
296	3200		2.46	
	3320		2.55	
	3240		2.49	
	3180	3370	2.45	2.59
	3380		2.60	
	3500		2.69	
	3780		2.91	
344	1640		1.26	
	1620		1.25	
	1580	1610	1.22	1.24
	1620		1.25	
	1580		1.22	
366	1250		1.96	

TABLE 1 (cont'd)

Test Temperature °K	Shear Strength, psi Individual Values	Average	Failure Time (t_f), min Individual Values	Average
	1350		1.04	
	1350		1.04	
	1280	1330	0.98	1.02
	1370		1.05	
	1380		1.06	
	1330		1.02	

TABLE 2

Mechanical property data for Sample II

Test Temperature °K	Shear Strength, psi Individual Values	Average	Failure Time (t_f), min Individual Values	Average
200	7700		5.92	
	10,100		8.53	
	11,220		8.63	
	7090	9300	5.44	7.26
	10,910		8.39	
	7340		5.65	
	10,760		8.28	
219	9970		7.67	
	8670		6.67	
	9490	8590	7.29	6.61
	7420		5.71	
	8110		6.23	
	7880		6.06	
250	15,910		12.24	
	15,350	15,520	11.84	11.95
	15,300		11.77	
283	11,530		8.86	
	12,390	12,290	9.53	9.45
	12,950		9.96	
296	6680		5.23	
	8110		6.24	
	8260		6.35	
	7550	7520	5.81	5.78
	6680		5.14	
	7850		6.04	
344	3260		2.51	
	3825		2.94	
	3900	3660	3.00	2.81
	2960		2.28	
	4335		3.33	
366	2960		2.28	
	2750		2.12	
	2680		2.06	

TABLE 2 (cont'd)

Test Temperature ^{o}K	Shear Strength, psi Individual Values	Average	Failure Time (t_f), min Individual Values	Average
	2960	2850	2.28	2.19
	2550		1.96	
	3190		2.45	

TABLE 3

Mechanical property data for Sample III

Test Temperature °K	Shear Strength, psi Individual Values	Shear Strength, psi Average	Failure time(t_f), min Individual Values	Failure time(t_f), min Average
200	7600		5.84	
	8000		6.15	
	7680	7760	5.91	5.97
	7700		5.92	
	7840		6.02	
219	7100		5.46	
	6880		5.29	
	7680	7320	5.91	5.63
	7320		5.63	
	7640		5.88	
250	6840		5.26	
	7200		5.54	
	7460	7110	5.74	5.47
	6640		5.11	
	7420		5.71	
283	5680		4.37	
	5780		4.44	
	5600	5750	4.31	4.42
	5710		4.39	
	6000		4.61	
296	5200		4.00	
	4900		3.77	
	5240	5140	4.03	3.95
	5280		4.06	
	5080		3.91	
344	2760		2.12	
	2580		1.98	
	2760	2690	2.12	2.06
	2540		1.95	
	2800		2.15	
366	1500		1.15	
	1280		0.98	
	1720	1470	1.32	1.13
	1400		1.08	
	1440		1.11	

TABLE 4

Mechanical property data for Sample IV

Test Temperature °K	Average[a] Shear Strength, psi	Average[a] Failure Time (t_f), min
219	3440	2.65
296	4400	3.31
344	4740	3.65
355	3650	2.86

[a] Average of 5 samples in each case

TABLE 5

Calculated and experimental t_f values

T, ^oK	S, psi	Log t_f, sec Calculated	Experimental
	Sample I		
200	7090	2.52	2.52
219	6580	2.50	2.48
250	7385	2.54	2.53
283	5400	2.40	2.40
296	3370	2.20	2.19
	Sample II		
200	9300	2.58	2.64
219	8590	2.56	2.60
250	15,520	2.82	2.86
283	12,290	2.72	2.75
296	7520	2.50	2.54
	Sample III		
200	7760	2.55	2.55
219	7320	2.52	2.53
250	7110	2.50	2.52
283	5750	2.41	2.42
296	5140	2.37	2.38
	Sample IV		
219	3440	2.16	2.20
296	4400	2.30	2.30
344	4740	2.32	2.34
355	3650	2.22	2.24

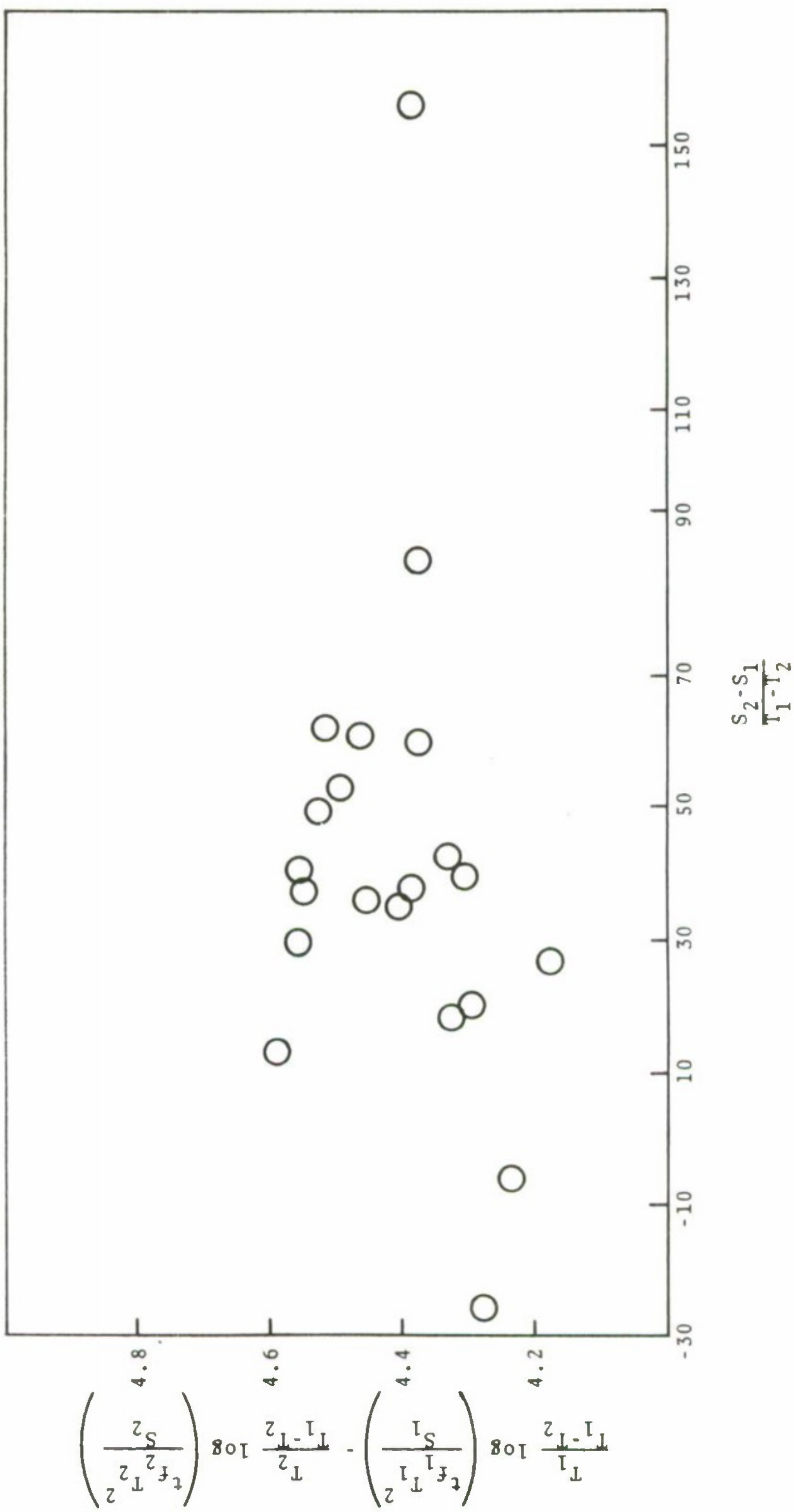

Fig 1 Data for Sample I, plotted in accordance with Equation 10

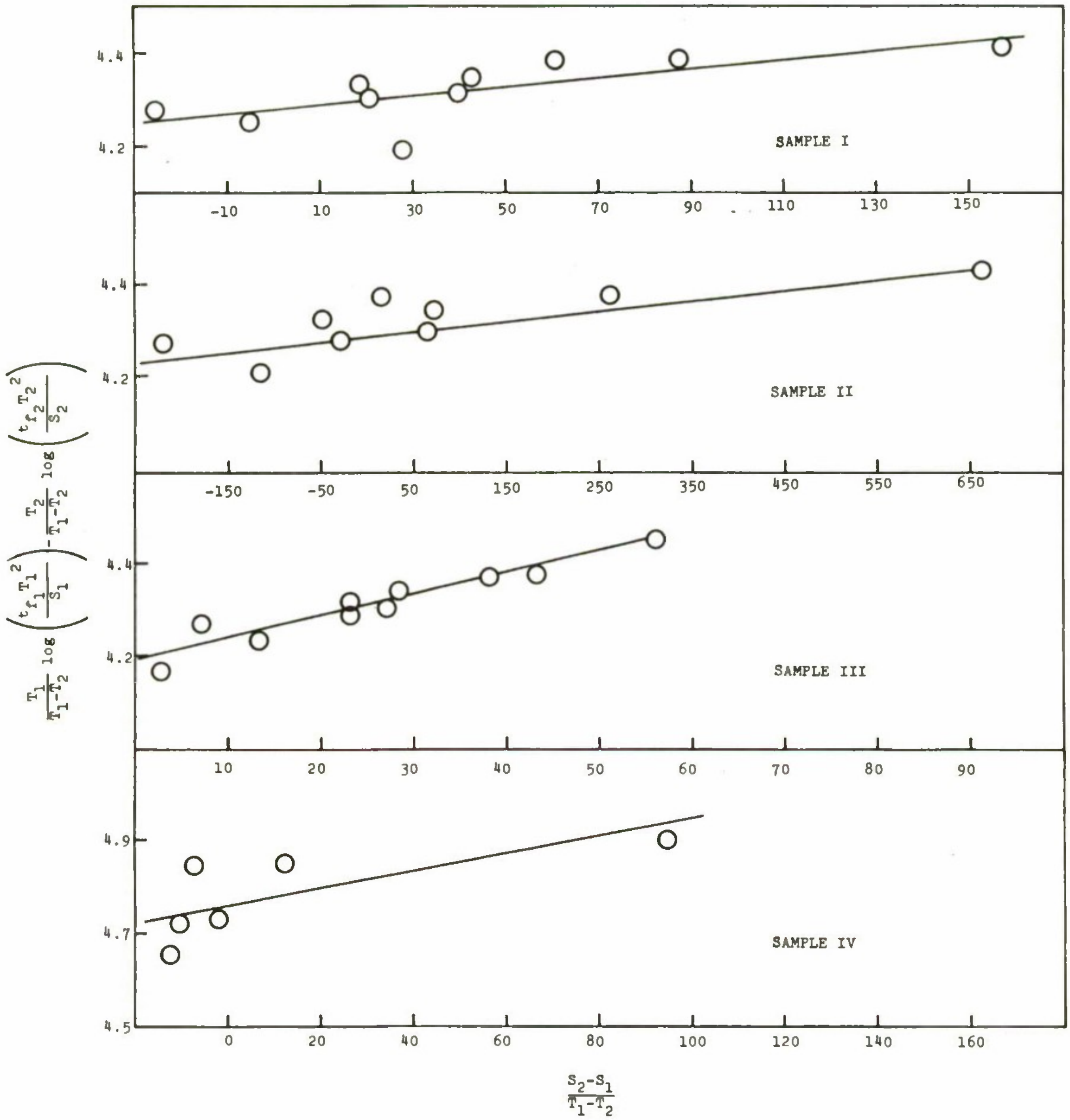

Fig 2 Cohesive failure data, in accordance with Equation 10

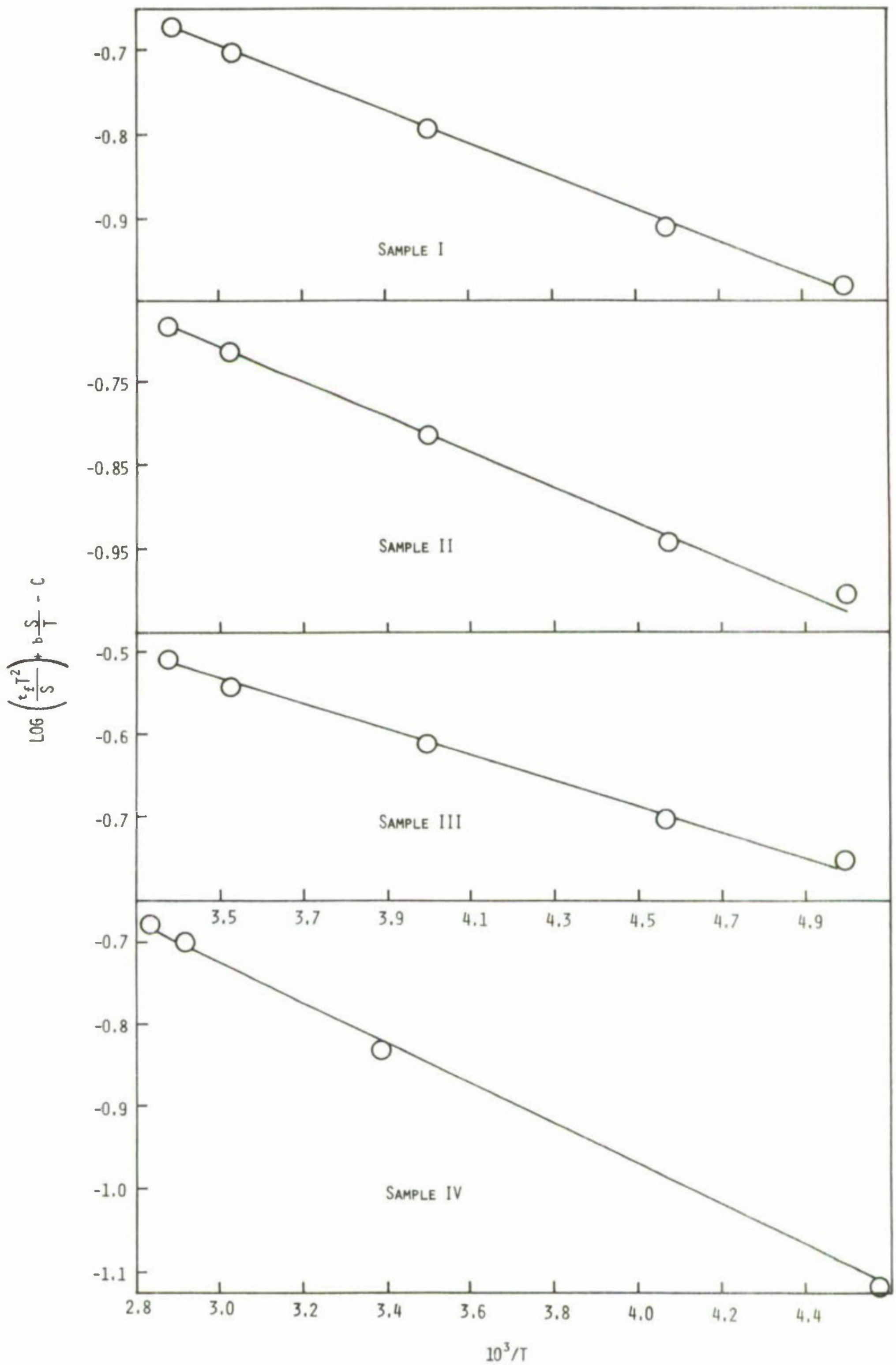

Fig 3 Arrhenius - type plots

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13. ABSTRACT

Data are given for shear and tensile testing of adhesive bonds under a constant rate of loading. A rate equation is then used to predict useful life from the mechanical data. The correlations are in general quite satisfactory, providing that the failure is cohesive[1] within the adhesive.

[1] In this work, cohesive failure always refers to failure within the adhesive layer

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