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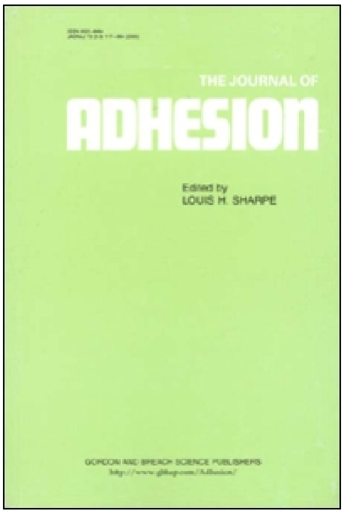

The Journal of Adhesion

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

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To cite this Article Mcabee, Elise , Tanner, William C. and Levi, David W.(1970) 'Prediction of Failure Times for Some Adhesive Bonded Joints', The Journal of Adhesion, 2: 2, 106 — 113

To link to this Article: DOI: 10.1080/0021846708544586

URL: http://dx.doi.org/10.1080/0021846708544586

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Prediction of Failure Times for Some Adhesive Bonded Joints

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Received February 25, 1970

ABSTRACT

Data are given for shear and tensile testing of adhesive bonds under conditions of a constant rate of loading. A rate equation is then used to predict lifetime from the mechanical data. The correlations appear to be satisfactory providing that the failure is cohesive within the adhesive.

INTRODUCTION

APPLICATION OF REACTION RATE THEORY to polymer mechanical behavior has been carried out in a number of laboratories[1-5]. An additional application of considerable interest is in the field of adhesive bonds. This paper describes the initial efforts at predicting the lifetimes of such bonds from limited mechanical property testing.

RESULTS AND DISCUSSION

Tobolsky and Eyring[1] first considered the lifetime of a material subjected to mechanical restraint to be a process proceeding according to a rate equation. For constant rate of loading Coleman and Knox[3] write the integrated rate equation in the form

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

$$A = \frac{\gamma_f h e^{\Delta F^{\neq}/RT}}{\lambda k T}$$

$$B = \delta / 2kT$$

where t_f is time to failure, S is stress, γ_f is mean displacement length at t_f, λ is jump distance, k is Boltzmann's constant, T is absolute temperature, $\Delta F^{\neq}$ is free energy of activation, R is the gas constant and δ is displacement volume.

Letting $\Delta F^{\neq} = \Delta H^{\neq} - T\Delta S^{\neq}$ and rearranging equation (1) we obtain

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

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

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

The apparent activation energy may then be evaluated by extrapolating several constant temperature lines to S/T = 0 and plotting 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 previously collected in these laboratories. The samples are designated I, II, III, and IV and may be briefly identified as follows:

I. Nylon Epoxy tape (Temperature Rise 10-12.5°/min. for cure). Reclaimed steel adherends.

II. NOL specimen Nylon Epoxy (Temperature Rise 10-12.5°/min.). Reclaimed steel adherends.

III. Nylon Epoxy Lap Shear (13.5°/min. cure). Reclaimed steel adherends.

IV. Modified Epoxy Lap Shear. Reclaimed steel adherends.

Since isothermal data were lacking for all of the samples I-IV, equation (3) could not be used in evaluation of parameters. Hence an alternative procedure was developed.

If we multiply equation (2) through by T and consider a data point t_{f_1} and S_1 at T_1, we obtain

$$T_1 \log\left(\frac{t_{f_1} T_1^2}{S_1}\right) = CT_1 + \frac{\Delta H^{\neq}}{2.3R} - b\,S_1 \tag{5}$$

and we may write a similar expression for t_{f_2}, S_2 and T_2. Assuming the constancy of $\Delta H^{\neq}$

$$\frac{\Delta H^{\neq}}{2.3R} = T_1 \log\left(\frac{t_{f_1} T_1^2}{S_1}\right) - CT_1 + bS_1 = T_2 \log\left(\frac{t_{f_2} T_2^2}{S_2}\right) - CT_2 + bS_2 \tag{6}$$

Rearranging and dividing through by $T_1 - T_2$, we obtain

$$\frac{T_1}{T_1 - T_2}\log\left(\frac{t_{f_1}T_1^2}{S_1}\right) - \frac{T_2}{T_1 - T_2}\log\left(\frac{t_{f_2}T_2^2}{S_2}\right) = C + b\left(\frac{S_2 - S_1}{T_1 - T_2}\right) \qquad (7)$$

For every possible pair of data points, the left hand side of equation (7) may be plotted against $(S_2 - S_1)/(T_1 - T_2)$. C and b are then evaluated as the intercept and slope respectively. After C and b are determined, we may go back to equation (2) in the form

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

The left hand side of equation (2a) is plotted against 1/T to evaluate $\Delta H^{\neq}$.

The original data are shown in Table 1. All possible pairs of points for sample I are plotted according to equation (7) 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 was cohesive at the lower temperatures but that it became 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 failure. Hence the higher temperature data (344° and 366°K) where adhesive failure seemed to be pronounced, were omitted for samples I, II, and III. Figure 2 shows the appropriate plots. In these cases the linearity appears to be reasonably satisfactory considering the usual scatter in adhesive mechanical data.

Figure 3 shows the Arrhenius type plots according to equation (2a). The slopes of these plots permitted evaluation of apparent activation energy. After the parameters were evaluated as described above, they were put back in equation (2) to give the relation between failure time and stress and temperature in each case. The equations are:

Sample I

$$\log t_f = \log S - 2\log T + 4.28 - 195/T - 0.00086\,(S/T) \qquad (8)$$

Sample II

$$\log t_f = \log S - 2\log T + 4.29 - 213/T - 0.00022\,(S/T) \qquad (9)$$

Sample III

$$\log t_f = \log S - 2\log T + 4.20 - 155/T - 0.00467\,(S/T) \qquad (10)$$

Sample IV

$$\log t_f = \log S - 2\log T + 4.47 - 250/T - 0.0020\,(S/T) \qquad (11)$$

The values in equations (8) through (11) show that apparent $\Delta H^{\neq}$ values are unexpectedly low (of the order of 1 kcal/mole). However, use of equations (8) through (11) to calculate t_f from the experimental stresses and temperatures show that these equations fit the data quite well in every case.

Table 1. Mechanical Property Data for Samples I-IV

Temperature	Sample I		Sample II		Sample III		Sample IV	
(°K)	S(psi)	t_f(min)	S(psi)	t_f(min)	S(psi)	t_f(min)	S(psi)	t_f(min)
200	6380	4.91	7700	5.92	7600	5.84		
	6800	5.23	10100	8.53	8000	6.15		
	7200	5.54	11220	8.63	7680	5.91		
	7000	5.38	7090	5.44	7700	5.92		
	6800	5.23	10910	8.39	7840	6.02		
	8160	6.28	7340	5.65	Av. 7760	Av. 5.97		
	7270	5.59	10760	8.28				
	Av. 7090	Av. 5.45	Av. 9300	Av. 7.26				
219	6220	4.78	9970	7.67	7100	5.46	3420	2.63
	5800	4.46	8670	6.67	6880	5.29	3380	2.60
	7440	5.72	9490	7.29	7680	5.91	3230	2.48
	6250	4.81	7420	5.71	7320	5.63	3540	2.72
	7250	5.58	8110	6.23	7640	5.88	3640	2.80
	6540	5.03	7880	6.06	Av. 7320	Av. 5.63	Av. 3440	2.65
	6570	5.05	Av. 8590	Av. 6.61				
	Av. 6580	Av. 5.06						
250	7340	5.64	15910	12.24	6840	5.26		
	7400	5.69	15350	11.84	7200	5.54		
	7420	5.71	15300	11.77	7460	5.74		
	7380	5.68	Av. 15520	Av. 11.95	6640	5.11		
	Av. 7385	Av. 5.68			7420	5.71		
					Av. 7110	Av. 5.47		
283	5280	4.06	11530	8.86	5680	4.37		
	5720	4.40	12390	9.53	5780	4.44		
	5200	4.00	12950	9.96	5600	4.31		
	Av. 5400	Av. 4.15	Av. 12290	Av. 9.45	5710	4.39		
					6000	4.61		
					Av. 5750	Av. 4.42		
296	3200	2.46	6680	5.23	5200	4.00	4980	3.83
	3320	2.55	8110	6.24	4900	3.77	4080	3.14
	3240	2.49	8260	6.35	5240	4.03	4000	3.08
	3180	2.45	7550	5.81	5280	4.06	4920	3.78
	3380	2.60	6680	5.14	5080	3.91	4000	3.08
	3500	2.69	7850	6.04	Av. 5140	Av. 3.95	Av. 4400	Av. 3.38
	3780	2.91	Av. 7520	Av. 5.78				
	Av. 3370	Av. 2.59						
344	1640	1.26	3260	2.51	2760	2.12	4240	3.26
	1620	1.25	3825	2.94	2580	1.98	4540	3.49
	1580	1.22	3900	3.00	2760	2.12	4440	3.42
	1620	1.25	2960	2.28	2540	1.95	5300	4.08
	1580	1.22	4335	3.33	2800	2.15	5200	4.00
	Av. 1610*	Av. 1.24*	Av. 3660*	Av. 2.81*	Av. 2690*	Av. 2.06*	Av. 4740	3.65
355							3200	2.46
							3400	2.62
							4180	3.22
							4070	3.13
							3640	2.80
							Av. 3700	2.85
366	1250	0.96	2960	2.28	1500	1.15		
	1350	1.04	2750	2.12	1280	0.98		
			2680	2.06				
	1350	1.04	2960	2.28	1720	1.32		
	1280	0.98	2550	1.96	1400	1.08		
	1370	1.05	3190	2.45	1440	1.11		
	1380	1.06	Av. 2850*	Av. 2.19*	Av. 1470*	Av. 1.13*		
	1330	1.02						
	Av. 1330*	Av. 1.02						

* Adhesive Failure; not used in the correlation.

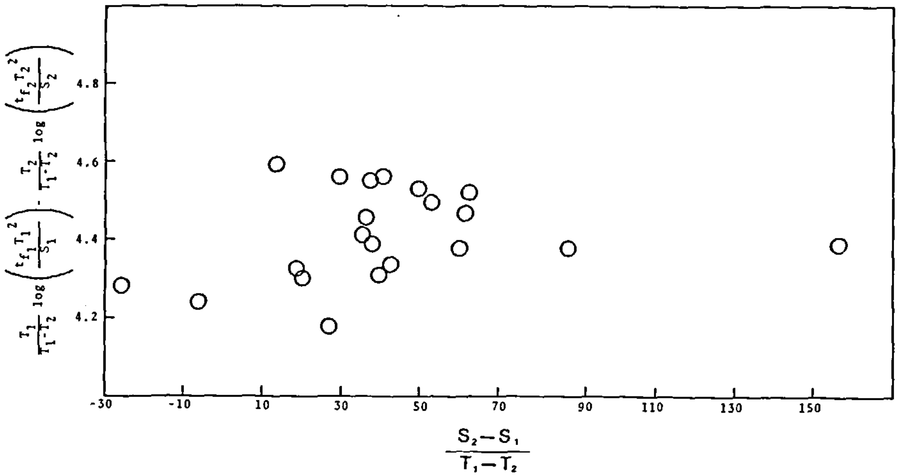

Figure 1. $\frac{T_1}{T_1-T_2}\log\left(\frac{t_{f_1}T_1^2}{S_1}\right)-\frac{T_2}{T_1-T_2}\log\left(\frac{t_{f_2}T_2^2}{S_2}\right)$ vs. $\frac{S_2-S_1}{T_1-T_2}$ for all the data of Sample I.

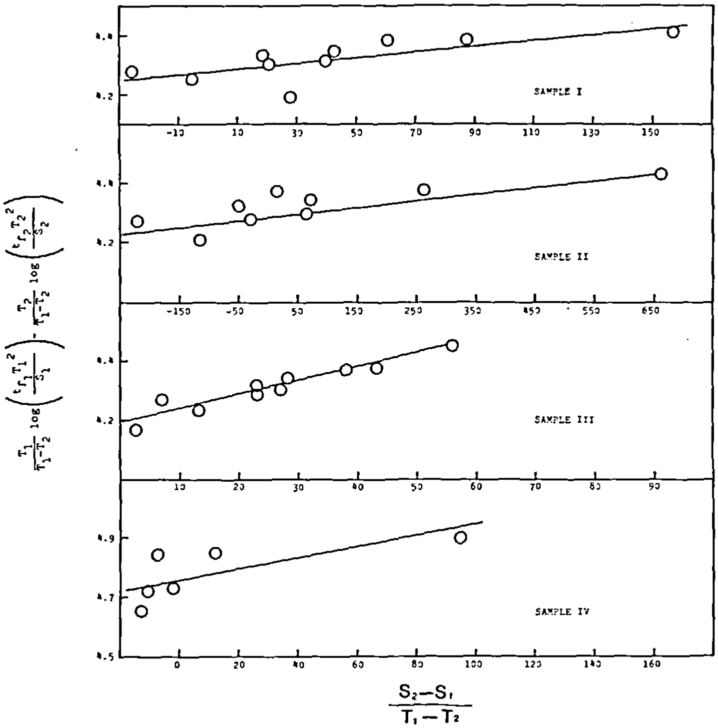

Figure 2. $\frac{T_1}{T_1-T_2}\log\left(\frac{t_{f_1}T_1^2}{S_1}\right)-\frac{T_1}{T_1-T_2}\log\left(\frac{t_{f_2}T_2^2}{S_2}\right)$ vs. $\frac{S_2-S_1}{T_1-T_2}$ for Cohesive Failure Data.

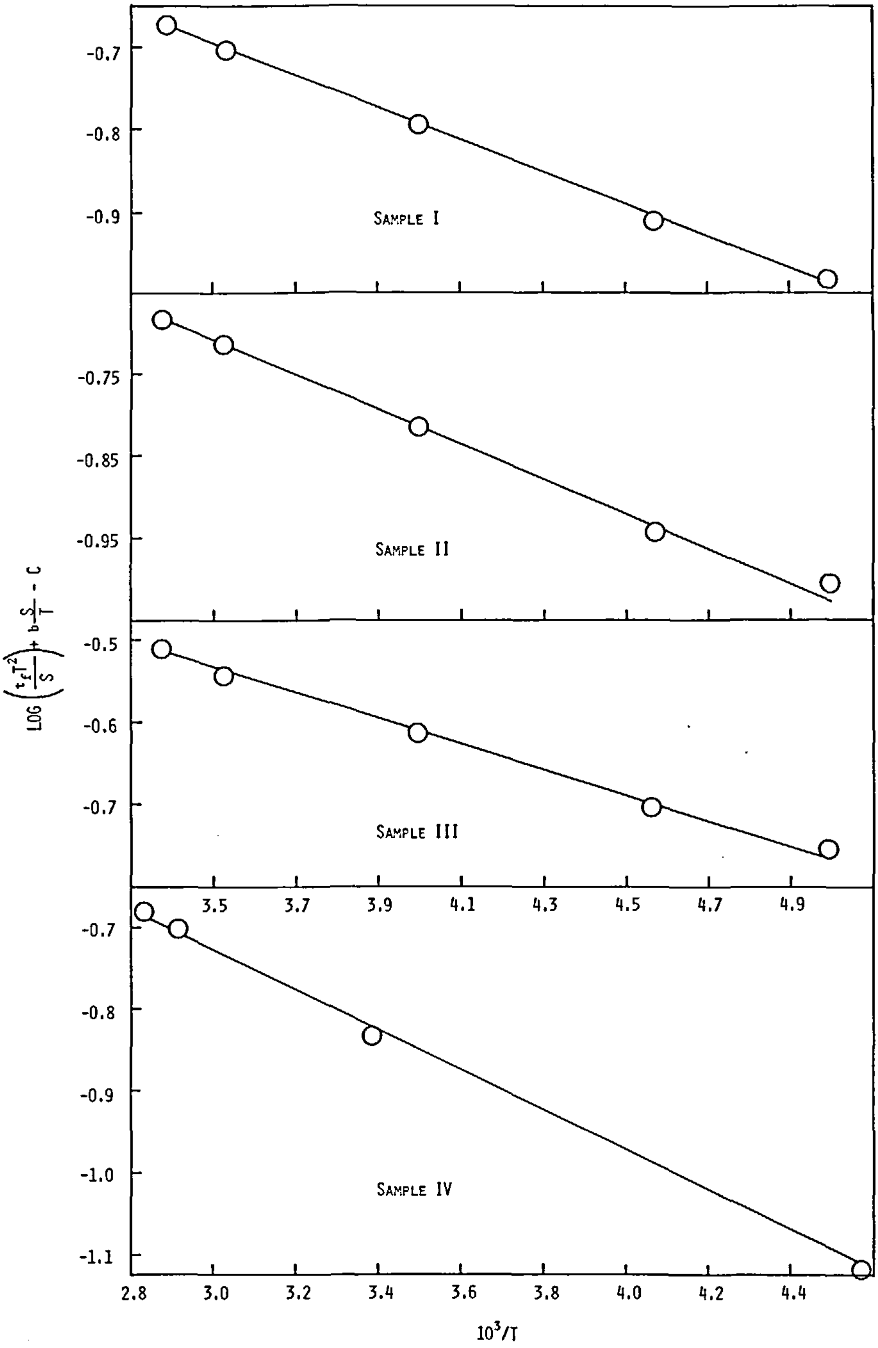

Figure 3. Arrhenius Type Plots.

EXPERIMENTAL

Shear Specimens (Samples I, III, IV)

Materials

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

An unsupported tape, nylon-epoxy adhesive was used to bond one group of specimens (Samples I and III).

A modified epoxy adhesive supported on a light glass fabric, was used to bond a group of specimens (Sample IV).

Adherend Preparation

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

Specimen Preparation

Individual coupons were marked with a scribe to establish the 1/2″ overlap. An aluminum jig was used to maintain alignment during the cure of the adhesive. The jig was made of 1/2″ aluminum plate with 1/8″ brass pins press-fit 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 particular adhesive was used in the joint. The assembly was placed in a hydraulic press and sufficient pressure was applied to assure 25 psi on 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°F was held for one hour after the bond line reached this temperature. The temperature was followed 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 (Samples II)

Materials

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

The nylon epoxy tape described above was the adhesive used.

Adherend Preparation

The steel rod adherends were prepared similarly to the shear coupons as 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 rate was 1,300 pounds/in^2/min. for shear tests.

ACKNOWLEDGEMENTS

The authors are grateful to Mr. M. J. Bodnar for helpful discussions. Thanks are also due Mrs. Dorothy Teetsel for extensive assistance with the manuscript.

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