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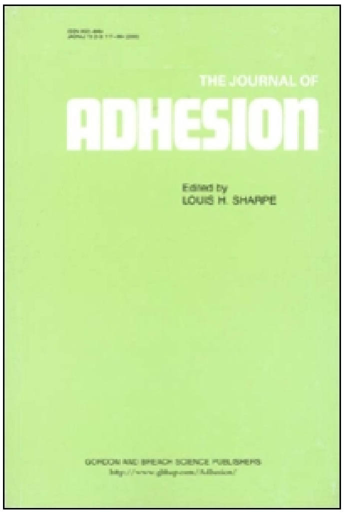

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Long Term Strength of Structural Adhesive Joints

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The long term static strength of adhesive joints is analyzed in terms of a modified Prot method and sustained load tests. Data from the failure times under different loading rates are used to predict the static stress that an adhesive joint will withstand for an infinite time, *i.e.*, the endurance limit. Despite theoretical shortcomings, the method is found to give reasonable estimates of the endurance limit as determined by standard sustained load tests. The ratio of the short term lap shear strength to the endurance limit is found to be independent of adhesive modulus, temperature, and sample geometry. For engineering calculations on lap shear structural adhesive joints under a static load (at 23°C., 50% R.H.), the endurance limit may be assumed to be equal to 0.25 of the short term strength.

INTRODUCTION

While adhesive joints may exhibit high strengths when tested by standard methods, such as the ASTM lap shear test, the strength value obtained in such a test is not easily related to the endurance of the adhesive joint in its use environment. In practice, an adhesive joint may be exposed to a combination of applied stress conditions such as static, dynamic or intermittent. The adhesive joint is usually subjected concurrently to variable external conditions such as temperature and humidity and may be exposed to a corrosive environment. While a great deal of literature exists on the strength and environmental behavior of adhesive joints, a phenomenological description of the absolute endurance of a structural adhesive joint is far from complete. This paper attempts to develop some basic understanding of the endurance strength of adhesive joints under the most lenient use condition,

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that is, adhesive joints under a static stress with constant humidity and temperature. The purpose of this work is to establish a relation between the short-term strength properties and the long-term strength of adhesive joints. To this end, the problem was first analyzed in the light of existing theories and approaches about the ultimate strength of materials.

Several schemes exist for predicting the long-term strength of materials. A widely used method is the stress—time—temperature approach presented by Larson and Miller[1] describing the rupture and creep behavior of metals. This method was later used by Goldfein[2] to describe the long-term rupture and impact stresses in reinforced plastics. In this approach, a time-temperature superposition parameter was devised by defining long-term strengths at ambient temperatures in terms of short-time static tests at elevated temperatures. Related to this, a superposition method for predicting the ultimate strength of polymers has also been proposed by Smith[3] and by Kaelble[4]. A review of the application of the time-temperature superposition principle to the long-term engineering properties of plastics has been published[5]. These approaches are based on the viscoelastic equations of state introduced by Williams, Landel and Ferry[6,7].

While these superposition methods have proven to be useful for empirically predicting the high-strain mechanical behavior of plastic materials, the interpretation is limited by its prediction of strength or stress endured through a finite time scale. Here there are difficulties in defining a meaningful stress endurance limit. On the other hand, since most practical structural adhesives are complicated blends of thermosetting polymers, the extrapolation of high-temperature strength data to represent data of a longer stress time at lower temperatures can lead to pitfalls since the adhesive material may undergo chemical and morphological changes during exposure to high temperature.

No satisfactory laboratory test has been designed which accelerates simulated service conditions. However, the direct sustained load test method for bulk polymers can be adapted to determine the stress levels which a polymeric material or adhesive joint can withstand for extended intervals of time. This method consists of loading several replicate test specimens of each sample at different stress levels, and recording the time to failure. Although the endurance limit is defined as the stress level which a sample can withstand indefinitely, practical limitations require that the stress level endured without failure be determined for a finite period. This has been arbitrarily taken in practice as 10,000 hours. This method, although giving direct results, is time consuming. From the standpoint of endurance under a stress environment, it would be convenient if an accelerated test were devised. In 1948, E. M. Prot[8] developed an accelerated test for estimating the cyclic fatigue life of metals.

Several years later, Boller[9] made some predictions about the long-term strength of reinforced plastics by the Prot technique. In the Prot method, the load is increased at a constant rate until the test specimen fails. Prot assumed that sustained load test results were representative of a hyperbolic law expressed as:

$$t_b = K'/(S_b - EL)$$

where t_b = time to break (1)
S_b = stress at break
EL = endurance limit
K' = material constant

He considered a variably applied stress starting at an arbitrary value ("preload") and increasing linearly with time. Thus, the stress, S, at any given time, t, may be expressed in terms of the initial stress, S_0, and the rate of increase of stress, α, in the equation,

$$S = S_0 + \alpha t. \tag{2}$$

If S_0 is set equal to EL, the theory predicts that the stress at break, S_b, depends linearly on the half-power of the rate of increase of stress, such that,

$$S_b = EL + K\alpha^{\frac{1}{2}} \tag{3}$$

A general theory, related to the Prot method, has been proposed by Loveless, Deeley and Swanson[10]. In their work it was assumed that under stress level conditions which are higher than the endurance limit, a specimen will suffer damage. This damage was considered to be proportional to the amount by which the stress exceeds the endurance limit and also to the time for which the stress acts. They derived the following equation for the endurance limit,

$$\frac{\alpha t_b^2}{2} = K - (S_0 - EL)t_b \tag{4}$$

where t_b is the time to break a test specimen that is stressed to failure at a rate α, K is a material constant and S_0 is a so-called preload term or the stress at $t = 0$. The endurance limit can then be determined by plotting αt_b^2 vs. t_b, and determining the slope of the straight line obtained which is equal to $2(EL - S_0)$. The preload term S_0 is an experimentally adjustable parameter. This analysis was found by Loveless[10,11] and others[12] to present a useful interpretation of the endurance limit concept.

In the derivation of the modified Prot equation (see Reference 10), the definitions of damage and damage criteria are vague. A detailed analysis of the derivation of the modified Prot approach, shows that in order to arrive at the form of Eq. 4, one must propose that during the loading period between $S = 0$ and $S = EL$, some type of stress annealing ("negative damage") occurs. If this assumption is not made, an equation resulting in unreasonable values of EL is obtained. This circumstance is of interest mechanistically since the values of EL determined by the form of Eq. 4 were checked experimentally[10,11] using the direct sustained load method and values of EL were found to be reasonable. In the present work, the long-term static strength of adhesive joints is analyzed in terms of this modified Prot method. Factors such as adhesive modulus, specimen geometry, test temperature and adhesive type are examined.

II EXPERIMENTAL METHODS AND MATERIALS

A modified creep testing apparatus, described in detail by Loveless *et al.*[10], was used for Prot-type tests. Progressive loading was obtained by dropping lead shot at a controlled rate into a container supported by the lever of the creep machine. Time-to-break was measured with a running time meter powered through a micro-switch actuated through the lever arm. When the specimen failed, the timer and the flow of lead shot were shut off simultaneously. The apparatus was operated in a constant temperature laboratory maintained at 23°C and 50% R.H. No preloads ($S_0 = 0$) were used in these experiments.

The adhesives selected for this study are commercially available types and vary in their rheological properties from a low modulus, elastomeric adhesive to a high modulus, glassy material. Single lap joint specimens were prepared using chromic acid cleaned 0.063″ 2024-T3 Alclad aluminum as the substrate. The manufacturer's instructions were followed in preparation, application and cure of the adhesives. Four specimen widths, $\frac{1}{4}''$, $\frac{1}{2}''$, $\frac{3}{4}''$ and 1″, and three overlap lengths were examined in this study. The short-term lap shear strength, LSS, of the adhesive joints were measured according to ASTM D 1002 specifications. The sustained load tests were performed using creep testing devices.

III STRENGTH OF ADHESIVE JOINTS AS INTERPRETED BY THE MODIFIED PROT METHOD

In adhesive joint strength tests, considerable scatter is commonly observed. Thus, in analyzing the various parameters that might affect the endurance limit of adhesive joints, the statistical variability of the test procedure must first be established. Table I presents strength data on four representative adhesive types. Standard deviations are given as a measure of the scatter in the determination of short-term strength and endurance limit. It is generally impractical to conduct sufficient replicate samples for each of the variables affecting adhesive joint strength. Therefore, in the discussion that follows, results for a limited number of replicates will be considered in terms of the statistical variability as shown in Table I.

TABLE I

Statistical variability of measured lap shear and estimated long-term (endurance limit) strength of adhesive joints [abc]

Adhesive type	LSS (psi)	EL (psi)	EL/LSS
Nylon-epoxy	6258 ± 953	2826 ± 497	0.45 ± 0.16
Nitrile-phenolic	4165 ± 393	1728 ± 254	0.42 ± 0.10
Epoxy-novolac	5052 ± 411	2181 ± 870	0.43 ± 0.18
Nitrile-epoxy	5482 ± 225	2455 ± 280	0.45 ± 0.05

[a] Test specimens (aluminum) according to ASTM D 1002 procedure

[b] The reported values represent adhesive joint strength measurements on at least four different test panels

[c] The deviations are reported as standard deviations

Using Equation 4, endurance limit values were determined for a diversity of adhesive systems. The data in Table II compare the short-term lap shear strength, LSS, with the endurance limit, *EL*, for 1″ wide, ½″ overlap test specimens. It is shown that within experimental uncertainty, the *EL*/*LSS* ratio appears to be independent of the modulus of the bulk adhesive. Furthermore, the ratio *EL*/*LSS* is independent of the value of *LSS*.

The effects of specimen geometry and test temperature were examined. Table III presents data that show that although specimen geometry may cause considerable changes in the pounds per square inch *LSS* value as one changes overlap length from ¼″ to ¾″ or width from ¼″ to 1″, the *EL*/*LSS* ratio of a given adhesive system is relatively insensitive to these changes. Table IV shows that the *EL*/*LSS* ratios of the adhesive joints are unaffected by temperature.

TABLE II

Endurance limits of various structural adhesives[c]

Adhesive type	Modulus[d] (PSI)	LSS[a] (PSI)	EL[b] (PSI)	EL/LSS
Epoxy-novolac DET cured	740,000	5100	2200	0.43
Epoxy resin[e]	440,000	2100	760	0.36
Nylon-epoxy	80,000	6300	2800	0.44
Nitrile-phenolic	75,000	4200	1700	0.41
Polyurethane	2,200	3300	1100	0.33

[a] Tested according to ASTM D 1002 procedure

[b] Determined according to equation 4

[c] All specimens exhibited cohesive failure

[d] Flexural modulus of bulk adhesive system

[e] Diethylenetriamine cured epoxy resin

TABLE III

Effect of specimen geometry on endurance[a]

				Limit values		
Adhesive	Width	Overlap	W/O	LSS (PSI)	EL (PSI)	EL/LSS
Nylon-epoxy	½″	¼″	2.0	8430	3700	0.44
	½″	½″	1.0	7030	3200	0.46
	¼″	½″	0.5	4760	2800	0.59
	¾″	½″	1.5	6890	3100	0.45
	1″	½″	2.0	6850	3100	0.45
Nitrile-phenolic	½″	¼″	2.0	5080	2200	0.42
	½″	½″	1.0	4240	1800	0.42
	½″	¾″	0.7	4190	1700	0.41
	¼″	½″	0.5	3805	1600	0.42
	¾″	½″	1.5	4365	1700	0.39
	1″	½″	2.0	4350	1900	0.45
Epoxy-novolac	½″	¼″	2.0	8170	4100	0.50
	½″	½″	1.0	5070	2500	0.49
	½″	¾″	0.7	4300	2000	0.46
	¼″	½″	0.5	3820	2100	0.55
	¾″	½″	1.5	4605	2500	0.52
	1″	½″	2.0	5360	3000	0.56

[a] Strength values are for single independent test specimens

TABLE IV

Effect of temperature on endurance limit values[a]

Adhesive Type	Test Temp. (°C)	LSS (PSI)	EL (PSI)	EL/LSS
Nylon-epoxy	RT	6258	2826	0.47
	82	4210	1911	0.45
Nitrile-phelolic	RT	4165	1728	0.42
	82	2760	1014	0.37
Epoxy-novolac	RT	5052	2181	0.43
	149	2090	1224	0.59
Nitrile-epoxy	RT	5482	2455	0.45
	82	3020	1531	0.50

[a] Strength values are for single independent test specimens

An indication of the reliability of the modified Prot method of predicting endurance limits of adhesive joints can only be obtained through long-term sustained load testing. Such tests were conducted on several representative adhesive samples. Lap shear specimens were loaded at stress levels equivalent to and greater than the predicted *EL* values. The times-to-break were then observed. The results of these experiments are presented in Table V.

These data demonstrate that within the uncertainty of strength measurement, the endurance limits predicted by the modified Prot approach are reasonable estimates of the long-term load bearing capability of lap shear joints.

TABLE V

Long-term (sustained load) lap shear strength of various adhesives
Nylon—epoxy—lap shear specimen 1″ wide, ½″ overlap

LSS = 6300 psi EL = 2800 psi

S (psi)	S/LSS	t_b (hrs.)
5000	0.730	0.5
4920	0.727	0.2
4900	0.715	0.1
4880	0.714	58
4700	0.687	166
4600	0.672	91
4400	0.642	545
4000	0.583	574
3000	0.438	>28,100

Nitrile-phenolic—lap shear specimen 1″ wide, ½″ overlap
LSS = 4200 psi EL = 1700 psi

S (psi)	S/LSS	t_b (hrs.)
3000	0.707	0.3
2350	0.648	0.8
2600	0.612	109
2567	0.604	465
2100	0.494	5,639
1900	0.448	7,632
1720	0.405	>11,430

Epoxy-novolac I—lap shear specimen 1″ wide, ½″ overlap
LSS = 5000 psi EL = 2200 psi

S (psi)	S/LSS	t_b (hrs.)
4200	0.783	0.1
4140	0.773	0.1
4100	0.766	1,448
4040	0.755	>15,430
3900	0.728	>29,100
3500	0.654	>29,200

TABLE V (contd.)

Epoxy-novolac II—lap shear specimen ½″ wide, ¾″ overlap
LSS = 4300 psi EL = 2000 psi

S (psi)	S/LSS	t_b (hrs.)
3322	0.722	0.1
2900	0.629	0.4
2845	0.618	0.7
2829	0.615	0.3
2812	0.611	29
2750	0.597	74
2600	0.565	111
2550	0.553	213
2499	0.542	13,135
2400	0.521	> 36,400

Polyurethane—lap shear specimen 1″ wide, ½″ overlap
LSS = 3300 psi EL = 1100 psi

S (psi)	S/LSS	t_b (hrs.)
1300	0.394	> 36,300
1100	0.333	> 36,300

DISCUSSION AND CONCLUSIONS

As noted in Tables II, III, and IV, the ratio of endurance limit to short-term strength appears to be independent of specimen geometry, adhesive modulus and temperature. Even though the short-term strength is strongly dependent on these variables, considering the uncertainty in the determination of *EL* and *LSS* as shown in Table I, we can assume a lower limit of *EL*/*LSS* to be about 0.25. For engineering calculations, it would appear that for lap shear structural adhesive joints under a static load the endurance limit may be assumed to be equal to 0.25 of the short-term strength. While this is an important generalization, under the present state of this analysis, it would not be justifiable to refine this conclusion any further.

While the modified Prot approach described here has been found to give a reasonable estimate of the long-term endurance strength of an adhesive joint, it must at this point be considered as an empirical conclusion, because of certain assumptions in the theory. The most obvious deficiency in the theory is the need to assume that some form of annealing occurs in the material at low stress level. While some forms of stress relaxation might be expected, it does not seem reasonable to expect an annealing effect of a magnitude required in the modified Prot theory.

Another difficulty is the hyperbolic law, which is the basic assumption of the Prot method. This hyperbolic relationship predicts that the time-to-break under a constant load is inversely proportional to the difference between the applied stress and the endurance limit. The data presented in Table V does not appear to follow this relationship. For these adhesive joint strength data, some type of a log function would seem to be a better fit to this data.

It is recommended that future work should be devoted to resolving these uncertainties by considering in more detail the mechanism of damage in adhesive joint systems. However, the observation that the Prot technique gives reasonable estimates of the endurance limit suggests that the method is worthy of further analysis and refinements.

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