Shear Properties of Promising Adhesives for Bonded Joints in Composite Material Structures

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The results of an experimental program are given which provide quantitative and reproducible shear property data for many of the present and promising adhesives for use in bonded joints in composite material structures. The experiments employ a composite material test specimen in which the adhesive material is in a film configuration, typical of bonded joints, rather than measuring bulk adhesive properties. Because both the flexural and in-plane stiffnesses of the glass-epoxy adherends of the test piece are extremely large ($>10^6$) compared to a parameter proportional to the shear and extensional modulus of the adhesive film, the adhesive film is virtually in a state of constant shear stress with negligible normal stresses. The details of the shear test piece have been published previously. The adhesives tested were those recommended by the major adhesive manufacturers, those selected by NASA Langley in the civil aviation program, and those utilized in the USAF PABST program. The data reported includes the ultimate shear stress, initial shear modulus, and elongation to failure, both mean values and standard deviations, for specimens made by the adhesive manufacturer and the University of Delaware.

I. Introduction

FOR several years, research has been conducted toward the goals of being able to determine accurately the stresses and deformations in both the adhesive and the adherends of bonded composite material structures, subjected to static, dynamic, and thermal loads, including the very important effects of combined high temperature and high relative humidity termed "hygrothermal."[1,2] Accurate methods of analysis were developed which analyze the stresses and deformations in both the adhesive and adherends in panels bonded together in a single lap joint configuration, subjected to a uniaxial in-plane panel loading.[3-5] These methods of analysis were found to be quite accurate.[6] Subsequently, these methods were used to conduct parametric studies to determine the relative effects of altering all geometric and material property variables.[7,8] More recently, these methods have been improved and extended to include hygrothermal effects, in a form that can be utilized for many joint configurations.[2] In order to determine the stresses in both adhesive and adherend, it is necessary to determine the mechanical properties of the adhesive materials utilized. In particular, the stress-strain curve of the adhesive materials must be known in order to know the effective moduli, yield strengths, ultimate strengths, and elongations to failure of the adhesive materials in shear, tension, and compression.[3] Not only should these properties be determined for each temperature and humidity combination, but they should be obtained through test pieces which employ the adhesive in a film or bond configuration, i.e., a test piece that has nearly the same surface-area-to-volume ratio as the adhesive in a typical joint configuration, rather than obtaining bulk form adhesive mechanical properties. Proper shear test and tension test pieces have been developed in this program.[3] The shear test specimen will be discussed herein. It differs from existing ASTM standards[9] because they utilize thin, metal adherends in a simple lap configuration, and a shorter joint length.

The methods of analyses derived through this program are elastic only. The hypothesis that in a fatigue environment, the maximum stress anywhere in the adhesive at the mean load level should be at or below the proportional limit of the adhesive material to successfully withstand a "runout" of 4×10^6 cycles has been confirmed experimentally through more than a hundred tension-tension fatigue tests employing glass adherends and Hysol EA951 and EA9628 adhesives, at room temperature and low humidity, and mostly with the stress ratio $R=+0.1$.[10-12]

Recently conducted research provides a comparative study of the mechanical properties at room temperature and low relative humidity of various adhesive systems employing the standard shear test specimen developed during this program.[13] Shear tests at 212°F were also conducted. That temperature corresponds to the utilization of the adhesive in a Mach 2 vehicle.[14]

Inclusive descriptions of research of others in adhesive bonded joints are readily available.[3,4,15,18]

II. Experimental Program

The shear test specimen utilized is shown in Fig. 1. It is the same test piece designed and utilized earlier in the research program. The design results in both the flexural and in-plane stiffness of the adherend being millions of times larger than a parameter proportional to the shear stiffness and extensional stiffness of the adhesive film, namely,[3-5,19]

$$D_{11}/E_{\text{eff}}\eta^3 > 10^9 \tag{1}$$

$$A_{11}/G_{\text{eff}}\eta > 10^6 \tag{2}$$

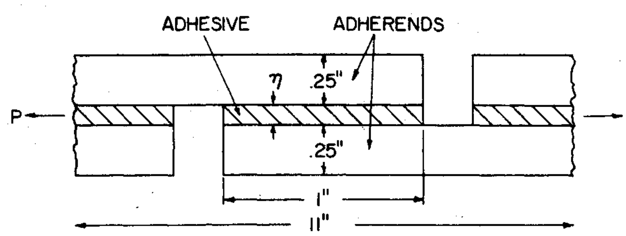

Fig. 1 Shear test specimen.

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Table 1 Room temperature adhesive properties

	Manufacturer	No. of specimens	τ	G_{eff}	ϵ
1	Conap DPAD-5633	5	1460/84	1065/213	1.748/.396
2	Conap AD-3	6	2065/98	3130/1015	1.883/.300
3	3M AF-30	12	2256/260	2004/530	2.644/.387
4	3M XB-163K	12	2458/261	2189/744	2.136/.809
5	3M AF-143	11	2173/192	3092/1325	1.543/.661
6	3M AF-147	11	2476/118	2954/983	1.714/.673
7	Mobay Mondur CB-75	11	1791/187	761/240	6.052/2.457
8	Mobay Mondur MR	17	2341/202	2180/1584	4.161/3.368
9	Mobay Mondur PF	12	2329/87	1867/1179	>4.639
10*	Atlas Amphesive 801	3	1229/102	6089/382	.328/.051
11*	Atlas Epoxy Bond Paste	3	711/27	4288/1034	.380/.052
12*	Hysol EA9628	6	3011/198	2138/380	>1.931
13*	Hysol EA9320	6	2746/95	5577/947	.626/.181
14*	Hysol ADX-663	6	3003/251	1945/667	>2.398
15*	Adhesive Engineering Aerobond 3000	4	928/183	2972/932	.447/.129
16*	Adhesive Engineering Aerobond 2185	6	2195/249	1536/284	....
17*	Adhesive Engineering Aerobond 2143	5	1800/55	11,084/766	>.281
18*	Dupont Cavalon 3200S	3	1091/265	1699/294	.959/.153
19	Dupont Cavalon 3000	6	1576/148	574/62	3.238/.583

where D_{11} is adherend flexural stiffness in the longitudinal direction, A_{11} is the adherend in-plane stiffness in the longitudinal direction, E_{eff} is the adhesive tensile modulus, G_{eff} is the adhesive shear modulus, and η is the adhesive film thickness.

Because of this, the shear stress over the length of the bond is virtually constant, except for it going to zero over a very small but measurable region at each end of the adhesive bond line. Obviously, the shear stress in the adhesive must be exactly zero at each end because it is a free surface. That fact was not accounted for in the earlier, more simplified analyses of Goland and Reissner.[20]

Also because of this design, the normal stresses in the adhesive (those normal to the load, or the peel stresses) are virtually zero due to negligible specimen rotation during loading because of the very stiff adherends.

Thus, through this design the shear stress in the adhesive can accurately be calculated as the applied load divided by the planform area of the adhesive film.

The tests are conducted by aligning the shear test specimen (Fig. 1) in the jaws of a static tensile test machine. Then, a zero length extensometer, in this case a Tinius–Olsen Model S-1000-2 LVDT, is attached such that the knife edges are aligned exactly normal to the load at the midpoint of the adhesive bond line. Because of the specimen design, in which the extensional stiffness of the adherend is millions of times larger than the adhesive shear modulus parameter, any relative axial displacement measured by the extensometer is due to shear deformation in the adhesive film.

Because the shear stresses are virtually constant over the length of the bond line, hence, so are the shear strains ϵ, defined here as the relative axial displacement of the extensometer δ divided by the adhesive bond line thickness η.

In the test program, the bond line thickness is measured at the midpoint on each side of the 1 in. wide specimen and then averaged to give the adhesive thickness η.

The following approach was used to identify the promising adhesive systems. Each of the major adhesive manufacturers was contacted for their recommendations as to the best structural adhesives they could suggest. In all cases recomendations were offered, bonding procedures given, and in most cases, samples of the adhesive material were provided gratuitously. In addition, NASA-Langley provided valuable recommendations concerning the adhesives being used in their commercial aircraft program. Likewise, we were aware of the adhesive systems being utilized in the Air Force PABST program.[17,18] Most of the adhesives utilized in the NASA and PABST programs were included in this test program. Hopefully, therefore, all of the promising adhesive systems have been characterized herein.

Manufacturing facilities for making the shear test specimens are available at the University of Delaware, and those specimens fabricated strictly adhered to the procedures and practices suggested or specified by the adhesive manufacturer. As a check, each adhesive manufacturer was requested to fabricate a group of six specimens in order that we could determine whether the University of Delaware manufactured specimens were statistically identical to the specimens manufactured by the adhesive manufacturer.

In either case, specimens were manufactured from bonding two 11 in. × 7 in. plates of ¼-in.-thick 3M glass-epoxy spring stock together. After proper bonding, a ¼-in.-wide machined groove was cut in the 7-in. direction (see Fig. 1), down through one of the ¼-in. adherends and through the adhesive bond lines. Then the plates were inverted and a corresponding milling procedure was used at a distance 1 in. away from the previous operation to provide an adhesive test length of 1 in. The plates were then cut parallel to the 11-in. direction, such that from the 7-in.-wide plates, six 1-in.-wide test specimens result. Finally, measurements of the bond line thickness are made photographically with a Bausch & Lomb Balphot Metallograph. As discussed earlier, the length of the bond line is measured, as well as the width of the adherends.

It is noted that the bond line thickness depends solely on the adhesive material used and the bonding procedures prescribed by the manufacturer. Shims or other artificial means of maintaining bond line thickness prevent the application of prescribed pressure, and thus are unacceptable. In spite of the variations in bond line thickness, the reproducibility of test results is excellent.

III. Test Results

All the adhesives tested are listed in Table 1, not in any specified order.

It should be noted that XB-163K is the 3M replacement for their AF55. One material utilized in the NASA Langley program is the Cavalon, and the materials used in the PABST program include AF55 (now replaced by XB-163K), and the EA9628.

In Table 1, mean values of the ultimate shear strength τ, the effective shear modulus G_{eff}, and the ultimate shear strain $\epsilon = \delta/\eta$, as defined earlier, are given. Also included are the

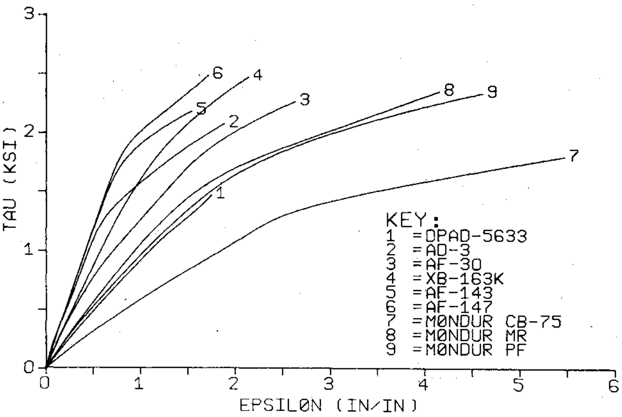

Fig. 2 Shear stress vs strain for adhesives 1-9 of Table 1.

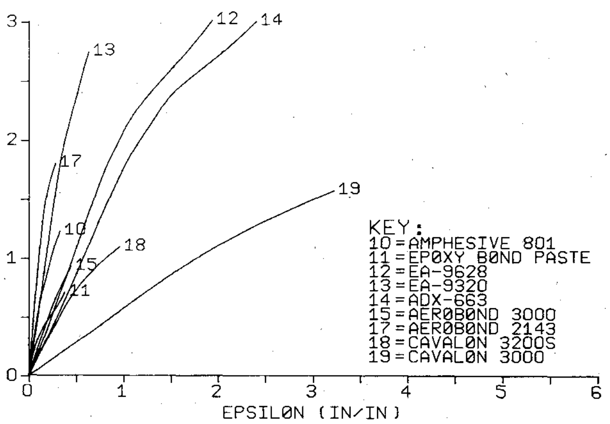

Fig. 3 Shear stress vs strain for adhesives 10-19 of Table 1.

standard deviations associated with each mean value and the number of tests performed. In each case, the specimens tested include those fabricated by the manufacturer and also by the University of Delaware, except for those which are asterisked, signifying that only the University of Delaware fabricated specimens were tested. In each case the ultimate strengths of both the manufacturer and University of Delaware specimens indicate that they come from the same statistical population, based upon the mean values and standard deviations calculated.

It is seen that, for many of the adhesives, the measured ultimate strains are greater than one. When the strains are large, the shear stress value τ should be regarded only as the applied load per unit adhesive film area, because the adhesive in that case is not in a state of pure shear.

The effective shear modulus is the initial slope measured below the proportional limit.

From Table 1, it is seen that the strongest adhesives in shear are the Hysol EA9628 and the ADX-663, while the lowest value is that of the Atlas Epoxy Bond Paste.

The stiffest adhesive by far is the Adhesive Engineering Aerobond 2143 adhesive, while the least stiff is the Cavalon 3000. The most ductile adhesive is the Mobay Mondur CB-75, and the most brittle is the Atlas Amphesive 801.

It is noted that most of the ultimate strength values have standard deviations considerably less than 10% of the mean values, and only a few exceed that percentage. Looking at the failure surfaces of specimens fabricated either by the supplier or by the University of Delaware, it is seen that some failures are cohesive, some are adhesive failures, while none are adherend failures. The low values of standard deviation indicate little influence of cohesive or adhesive type failure. As would be expected the standard deviations are relatively larger for the effective modulus values and the ultimate strains.

Table 2 Adhesive properties at 212°F (100°C)

Manufacturer	No. of specimens	τ	G_{eff}	ϵ
1* 3M AF-147	6	1548/49	491/25	4.292/.711
2* XB-163K	6	2041/161	648/144	3.770/.469
3* M AF-143	6	1927/51	984/188	2.282/.278
4* Hysol ADX-663	6	2379/105	587/116	4.263/.644
5* Hysol EA9628	6	2202/71	861/118	2.578/.365
6* Adhesive Engineering Aerobond 3000	6	1246/147	2167/628	.769/.277

Table 3 Change in adhesive properties at 212°F (100°C) from room temperature properties

Manufacturer	τ, %	G_{eff}, %	ϵ, %
3M AF-147	−37.5	−83.4	150.4
3M XB-163K	−17.0	−70.4	76.5
3M AF-143	−11.3	−68.2	47.9
Hysol ADX-663	−20.8	−69.8	<77.8
Hysol EA 9628	−26.9	−59.8	<33.5
Adhesive Engineering Aerobond 3000	34.3	−27.1	72

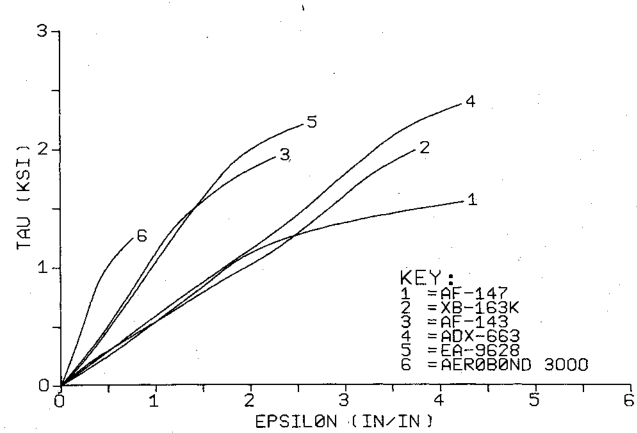

Fig. 4 Shear stress vs strain for adhesives 1-6 of Table 2.

In Figs. 2 and 3 are plotted the averaged values of shear stress vs. strain curves for 18 of the adhesives tested. The ordinate is τ, which in the linear elastic range can be aptly called the shear stress, and in the nonlinear range should be considered only as the axial load divided by the planform area of the adhesive film, as discussed earlier. Likewise, the absissa ϵ is the axial displacement of the extensometer δ divided by the adhesive thickness η. The method used to plot the data in Figs. 2-4 involves the use of the cubic spline,[21] an innovative procedure which insures the curve goes through each data point measured. In the case here the curve goes through the average of the samples at each load level the displacements were measured.

Table 2 provides data analogous to Table 1 for selected adhesives at 212°F, after the specimens had come to constant temperature, and at low relative humidity. It is seen that the strongest adhesives are Hysol ADX-663 and EA9628, which were the strongest at room temperature.

Again, the stiffest by far is the Adhesive Engineering Aeroband 3000, and it was by far the most brittle. The 3M AF-147 was the least stiff and the most ductile of those tested.

Again, all standard deviations on strength values are low, being less than 8% for the ductile adhesives and less than 12% for the brittle Adhesive Engineering Aerobond 3000. Once more the standard deviations are higher for both the shear modulus and the strain to failure.

Figure 4 is a plot of the average values of shear stress as a function of strain for the dry adhesives at 212°F, in which it is seen that the total strain energy to failure is considerably more for Hysol ADX-663 than for the other adhesives tested.

Table 3 shows a comparison of each mean value of shear stress at failure, shear modulus, and strain at failure between the value at 212°F and the value at 70°F. It is seen that while the percent reduction in strength is in the order of 10-40%, the reduction in stiffness is considerably greater, as is the increase in strain to failure.

IV. Conclusions

First of all, the overall reproducibility of the ultimate strength results (i.e., the low standard deviations) lends confidence to the test, the testing method, and to the usability of the adhesives systems tested in routine manufacturing environments.

The Hysol EA9628 and ADX-663 are the strongest of the 19 adhesives tested, while the Adhesive Engineering Aerobond 2143 is the stiffest adhesive in shear, and the Mobay Mondur CB-75 is the most ductile material.

At 212°F the two Hysol adhesives, ADX-663 and EA9628, are the strongest tested, the Adhesive Engineering Aerobond 3000 is the stiffest tested, and the adhesive with the largest strain energy to failure is the Hysol ADX-663.

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