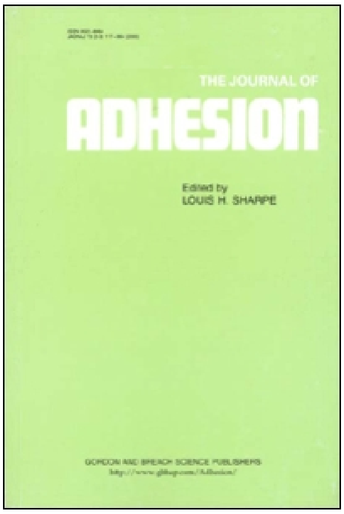

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Tensile Properties and Plane-Stress Fracture Toughness of Thin Film Aerospace Adhesives

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Tensile and Mode I plane-stress fracture toughness tests were performed at room temperature on three film-type aerospace adhesives, two epoxies and one polyimide. Specimens were cut from cured sheets. Tensile modulus, strength, failure strain, and fracture toughness values were obtained for supported versions of the epoxy films which contained a non-woven carrier ("scrim") cloth and for the polyimide which contained a woven carrier cloth. The same tensile and fracture properties were obtained for unsupported versions of the epoxies and for the supported polyimide subjected to thermal cycling or isothermal exposure. The polyimide adhesive displayed a higher tensile strength and fracture toughness than the epoxy adhesives. Isothermal exposure for 5,000 hours to a hot/wet (71°C [160°F], > 90% relative humidity [rh]) environment was the most detrimental condition for all of the adhesives. The presence of a scrim cloth reduced many of the tensile and fracture properties of the epoxy adhesives.

Keywords: Adhesives; epoxy; polyimide; tensile test; fracture toughness; environmental exposure; scrim (carrier) cloth; bonded joints

INTRODUCTION

In support of a larger project at Georgia Tech investigating the environmental durability of adhesively-bonded aircraft joints, tensile and Mode I plane-stress fracture toughness tests were performed on

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thin film specimens of three aerospace adhesives, AF-191, FM®73 and FM®x5. These film-type adhesives are in use or planned for use on aircraft ranging from subsonic planes to future supersonic transports. Because these aerospace vehicles operate under a variety of conditions, selected specimens were also subjected to various forms of environmental exposure. These adhesives are being carefully scrutinized for their mechanical properties and environmental durability because of the current emphasis on extending the lives of existing aircraft or producing new airframes with longer design lives.

Although manufacturers have performed traditional lap shear and thick adherend shear tests on the adhesives investigated in this report, the results of the tensile and planestress fracture toughness experiments presented here are believed to be unique. These results are expected to provide input to finite element analyses which often require tensile rather than shear properties. From a more general perspective, the current research will be valuable in understanding the mechanical behaviour and environmental durability of bonded aerospace joints and in developing analytical models to predict better the lifetime and performance of bonded structures.

Aerospace adhesives are normally used in a supported film form incorporating a scrim or carrier cloth to improve handling qualities and control bondline thickness. Thus, it was desired to conduct in-plane tensile and fracture toughness tests on as-received specimens containing a scrim cloth. Despite loading conditions on bonded structures which typically subject the bondline to out-of-plane peel and in-plane shear stresses, the in-plane tensile properties of the scrim-containing adhesives were obtained for two primary reasons: first, to highlight performance differences among the adhesives, and second, to generate tensile data required by some finite element programs. Furthermore, it was felt that this portion of the research may be valuable to bonded joint design, since previous research [1, 2] has indicated that bonded joint properties may depend upon the type of scrim cloth within the adhesive layer.

In addition to the properties of the scrim-containing adhesives, the environmental durability of the unsupported or "neat" polymer adhesives is also of interest. Therefore, the unsupported epoxies and the supported polyimide (unavailable in an unsupported form) were exposed to various conditions based upon typical environments encountered in their specific applications. Two types of environmental exposure were investigated: 1) thermal cycling between a high-altitude, subsonic cruise condition and the maximum use temperature of each adhesive, and 2) isothermal exposure to hot/dry or hot/wet conditions.

BACKGROUND

The use of adhesively-bonded structures in aerospace applications has increased dramatically during the last 15 years. Adhesively-bonded joints and repairs offer advantages over traditional, mechanically-fastened assemblies in terms of fatigue and corrosion resistance, aerodynamic properties and, arguably, manufacturing cost. During the last several decades, designs, fabrication procedures, and new adhesive and adherend materials have combined to improve bonded joints. Adhesives, perhaps because their formulations may be easily tailored, continuously evolve. Currently, toughened epoxy adhesives are being used to bond composites to composites, metals to metals, and composites to metals on primarily subsonic aircraft structures. However, changes in the performance envelope of future aircraft, such as the High Speed Civil Transport (HSCT), will require more advanced polymers capable of operating at higher temperatures.

The design and performance of adhesive bonds has previously been investigated from two different viewpoints. The first, a stress-based approach, was initiated by Goland and Reissner [3], and used extensively by Hart-Smith [4, 5]. A more recently developed approach based upon fracture mechanics principles was first proposed by Ripling. Mostovoy and Patrick [6], Shaw [7], Johnson and Mall [8, 9], Brussat *et al.* [10] and others have also employed fracture mechanics extensively.

Yet, regardless of the materials and methods of analysis used for bonded structures, bondline integrity depends heavily upon the adhesive properties. Some adhesive properties may often be obtained from manufacturer's data sheets, but the information available from these sources may be insufficient for detailed design or analysis. The desire for such designs and analyses necessitates the need for additional information on the behavior of adhesive materials, particularly on their resistance to environmental exposure.

Research conducted by Hinckley and Mings [11], Tsou *et al.* [12], and Klemann and DeVilbiss [13] has resulted in procedures for conducting tensile and fracture toughness tests of thin polymeric films. Their techniques were applied in the present research to investigate film-type adhesives and to determine the tensile and fracture properties critical for stress-based and fracture mechanics approaches to bonded joint design.

MATERIALS

Two epoxy-based adhesives, AF-191 and FM®73, and one polyimide-based adhesive, FM®x5, were examined for this project. AF-191 is manufactured by 3M Corporation (St. Paul, MN, USA). FM®73 and FM®x5 are manufactured by CYTEC Engineered Materials, Inc. (Havre de Grace, MD, USA).

AF-191 is a modified epoxy adhesive with an advertised use temperature of 177°C (350°F) [14]. Plans call for the use of this adhesive on F-22 fighter aircraft at temperatures as high as 104°C (220°F). Two varieties of AF-191 were tested: AF-191M containing a non-woven nylon scrim cloth (Fig. 1a); and AF-191U, an unsupported "neat" resin. The volume fraction of the scrim cloth was approximately 2%. Cured films of both varieties had a nominal weight of 260 g/m^2 (0.05 lb/ft^2) and an approximate thickness 0.25 mm (0.010 in). The cured AF-191 film had a pale yellow color and, as a single layer, was translucent.

FM®73 is also a modified epoxy adhesive with an advertised use temperature of 82°C (180°F) [15]. This adhesive was used in the U.S. Air Force's successful Primary Adhesively Bonded Structures Technology (PABST) program [16] in the 1970s. It is currently being used to bond composite patches to cracked metallic aerospace structures on military and commercial aircraft where conditions may approach 71°C (160°F) and high (> 90%) relative humidity. Two varieties of FM®73 were tested: FM®73M containing a non-woven polyester scrim cloth (Fig. 1b); and FM®73U, an unsupported "neat" resin. The volume

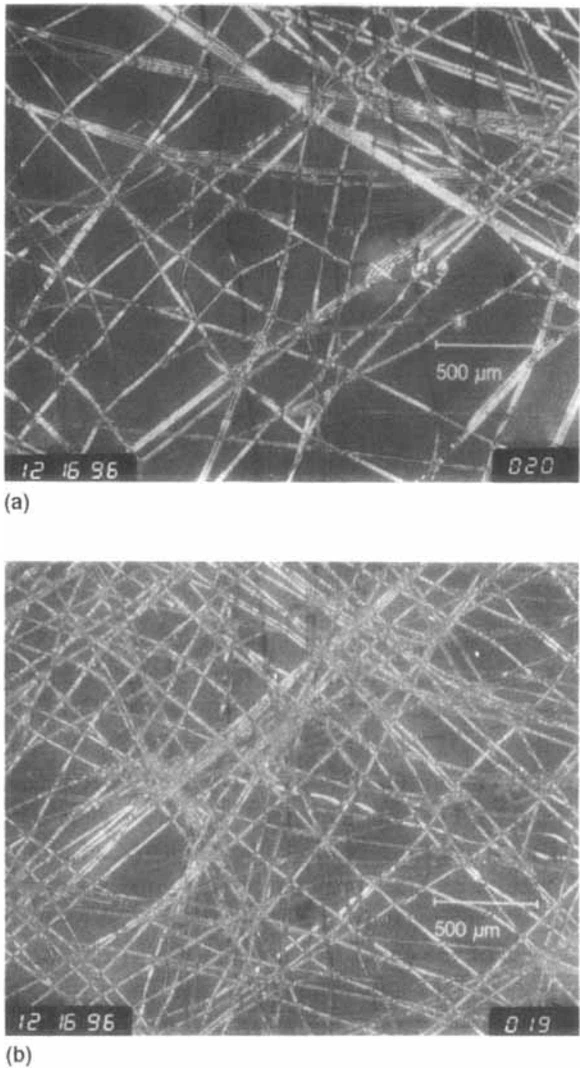
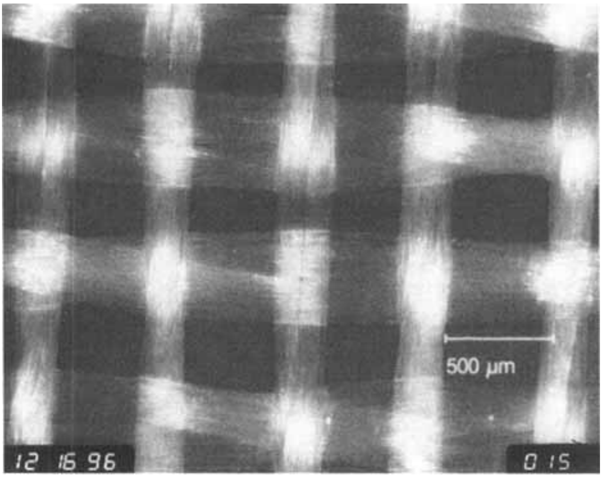


FIGURE 1 Scrim cloths contained in a) FM® 73M, b) AF-191M, and c) FM® x5 .

fraction of the scrim cloth was approximately 4%. Cured films of both varieties had a nominal weight of 290 g/m^2 (0.06 lb/ft^2) and an approximate thickness of 0.25 mm (10 mils). The cured FM® 73 film had a yellow-orange color and, as a single layer, was translucent.



(c)

FIGURE 1 (Continued).

FM®x5 is an amorphous polyimide blend of PETI-5 and other thermoplastic resins. This adhesive is being considered for wing and fuselage structures on the High Speed Civil Transport where temperatures may approach 177°C (350°F), the adhesive's advertised maximum use temperature [17, 18]. Sheets of cured FM®x5 were provided by CYTEC and contained a woven glass scrim cloth (Fig. 1c). This scrim cloth has a volume fraction of approximately 40% and imparts physical integrity to the cured adhesive; without scrim cloth, the cured resin is extremely fragile and friable [19]. The nominal weight of the cured film was 515 g/m^2 (0.10 lb/ft^2) and its approximate thickness was 0.34 mm (0.013 in). The FM®x5 film had a dark brown color and, as a single layer, was nearly opaque.

EXPERIMENTAL PROCEDURES

Adhesive Curing

Essentially void-free sheets (approximately 250 mm × 250 mm [10 in × 10 in]) of the AF-191 and FM®73 adhesives were cured on a were cured on a porous Teflon™ cloth-covered aluminium plate in a circulating air oven. Curing profiles were tailored to duplicate manufacturers' recommended procedures used for bonded joint specimens under investigation at Georgia Tech. However, the AF-191 and FM®73 used in the present study were cured without pressure using a single layer of the adhesive film. Use of a vacuum bag or autoclave and attempts at curing multiple film layers resulted in cured sheets with unacceptably high levels of voids. The following curing procedure was used for the AF-191 and FM®73 adhesives:

1. ramp to 177°C (350°F) [AF-191] or 115°C (240°F) [FM®73] at 4–6°C (8–10°F) per minute
2. hold at temperature for 60 min [AF-191] or 150 min [FM®73]
3. remove from oven, air cool.

FM®x5 sheets provided by CYTEC were cured using the following procedure:

1. apply full vacuum
2. ramp to 250°C (482°F) at 3–4°C (5–7°F) per minute
3. hold at 250°C (482°F) for 60 minutes
4. add 0.34 MPa (50 psi) and vent the vacuum
5. ramp to 350°C (662°F) at 2–3°C (3–4°F) per minute
6. hold at 350°C (662°F) for 60 minutes
7. cool to 38°C (100°F) at 3–4°C (5–7°F) per minute.

Specimen Fabrication

Testing utilized two specimen geometries (Fig. 2). Tensile tests employed a "dogbone" shape conforming to ASTM D 638M [20] Type M-III. Fracture toughness tests employed a single-edge notched geometry with the same overall dimensions as the "dogbone".

Test specimens were cut using steel rule dies. To cut the specimens, a die was placed on a flat surface with the cutting edge facing up, a piece of cured adhesive film was placed on top of the die, and a small, rigid sheet of polyethylene was placed on top of the adhesive film. The top surface of the polyethylene sheet was struck firmly with a rubber or dead weight mallet. This produced specimens with clean edges and consistent dimensions.

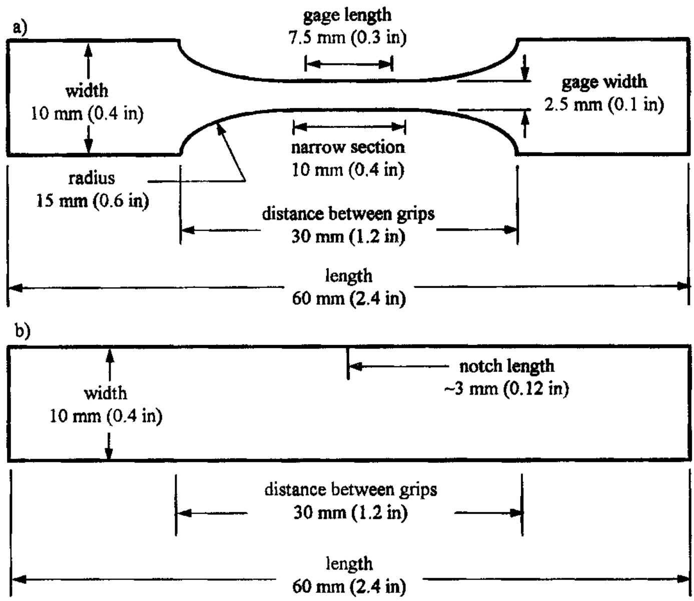


FIGURE 2 Specimen geometry for (a) the ASTM D 638M Type M-III "dogbone" specimen used for tensile testing and (b) the "straight-sided" specimen used fracture toughness testing.

Fracture toughness specimens were notched using a razor blade carefully to "saw" a starter notch. During this procedure, specimens were supported in a simple jig fabricated from two pieces of polyethylene cut to the dimensions of the single-edge notched specimen and containing slots to guide the razor blade. The jig prevented out-of-plane buckling and aided in locating and forming the starter notches.

Multiple specimens were used for tensile and fracture toughness testing for each adhesive subjected to each condition. However, availability of material limited the number of duplicate tests performed. The number of specimens used for each test may be found in the Results section.

Environmental Conditioning

Specimens were tested either in the "as-received" state (*i.e.*, no pre-test environmental exposure), following thermal cycling, or following

5,000 hours of isothermal exposure. To isolate better the effects of long-term exposure and thermal cycling on the adhesive resin, environmental conditioning was performed on the unreinforced AF-191U and FM®73U. The FM®x5 material was also subjected to environmental conditioning. Versions of the epoxies which contained the scrim cloth (AF-191M and FM®73M) were tested only in the as-received condition.

Groups of specimens were subjected to thermal cycling conditions indicative of the service environments for the particular applications in which the adhesives are used. (Tab. I) The low temperature extreme (−54°C [−65°F]) corresponds to high-altitude, sub-sonic cruise conditions and the high temperature extreme corresponds to the maximum use temperature. Thermal cycling was conducted in a chamber consisting of two compartments maintained at the desired temperature extremes and a pneumatically-powered cage which shuttled specimens between the compartments. Specimens remained in each compartment for 20 minutes (FM®73U) or 30 minutes (AF-191U and FM®x5) in order to achieve the desired time-temperature profile. No humidity control was possible with this apparatus. The better to simulate operational conditions, AF-191U and FM®73U specimens were exposed to "hot/wet" (71°C [160°F], >90% rh) conditions for approximately 300 hours prior to thermal cycling.

Other groups of specimens were subjected to isothermal exposure for 5,000 hours to high temperature and humidity conditions simulating extreme service environments (Tab. II).

Test Apparatus

Testing was performed in laboratory conditions on a benchtop, screw-driven mechanical test frame (Fig. 3). Specimens were gripped using

TABLE I Summary of thermal cycling parameters for adhesive specimens (low temperature extreme for all cycles −54°C [−65°F])

Adhesive	*Pre-conditioning 71°C [160°F] > 90%rh*	*Sealed during cycling?*	*High Temperature Extreme*	*Number of Cycles*
AF-191U	yes	yes, in foil bag	+104°C (+220°F)	100
FM®73U	yes	no	+71°C (+160°F)	100
FM®x5	none	no	+163°C (+325°F)	500

TABLE II Summary of 5,000 hour isothermal exposure conditions

Adhesive	*"RT/Wet"*	*"Hot/Dry"*	*"Hot/Wet"*
AF-191U	none	104°C [220°F], 0% rh	71°C [160°F], > 90% rh
FM®73U	71°C [160°F], >90% rh	71°C [160°F], 0% rh	71°C [160°F], > 90% rh
FM®x5	none	177°C [350°F], 0% rh	71°C [160°F], > 90% rh

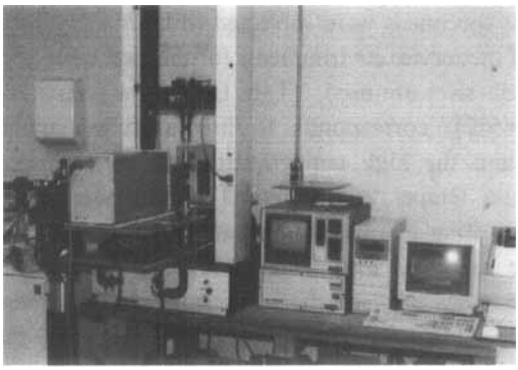


FIGURE 3 Test apparatus showing (a) Quester Microscope (partially hidden behind laser unit), (b) laser extensometer, (c) mechanical test frame, (d) pneumatic grips, and (e) PC for test control and data acquisition.

flat-faced, pneumatic grips pressurized to 0.62 MPa (90 psi). To prevent slipping, abrasive cloth tabs were used in addition to maximum grip pressure with the high-strength FM®x5 specimens.

A non-contact laser extensometer provided gage section extension measurements accurate to ± 0.1 mm (4 mils) during tensile testing. The laser beam scanned through an aperture created by paper flags affixed to each specimen at the boundaries of the gage section.

A PC-based software program provided test control and continuously acquired load, crosshead displacement, and gage section extension for each test.

Tensile Test Procedures

Tensile testing was performed in ambient conditions (21–23°C [70–74°F], 45–55% rh) under displacement control with a crosshead speed of 1 mm (0.04 in) per minute.

For each tensile test specimen, an engineering stress-strain curve was produced based on force and displacement values and specimen dimensions. From this curve, the elastic modulus (E), failure strain (ε_f), yield strengths ($\sigma_{ys(0.2)}$, σ_{iys}), and ultimate strength (σ_{uts}) were calculated.

The elastic modulus was determined from a least-squares fit to a range of points from the initial linear portion of the stress *vs.* strain curve. The particular range of points used was determined visually and by comparing the R^2 values for different ranges. For the epoxies (AF-191 and FM®73) the data between 5 and 25 MPa were used ($0.88 < R^2 < 0.95$). For the FM®x5, data between 20 and 100 MPa were used ($0.97 < R^2 < 0.99$).

Two approaches were used to determine the yield strength of the adhesives. The first was a traditional 0.2% offset method. The second method involved constructing a line parallel to the elastic modulus (Fig. 4, line a) and a line indicating a secondary modulus corresponding to a linear strain hardening rate (line b). If the stress-strain curve did not contain a secondary modulus or if it peaked before relaxing and leveling-off (Fig. 4), a horizontal line was drawn through the point of ultimate stress. A vertical line (c) was drawn at the intersection of lines (a) and (b). The point where this line (c) intercepted the stress *vs.* strain curve was identified as the intercept yield strength (σ_{iys}) of the material.

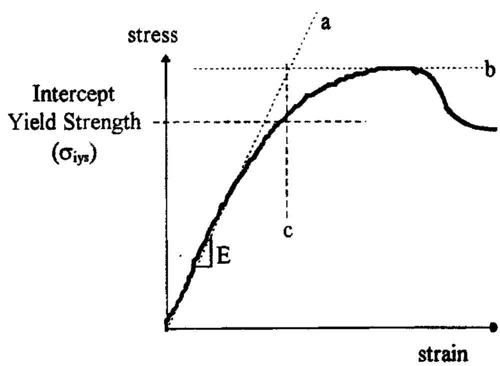


FIGURE 4 Obtaining the intercept yield strength.

Fracture Toughness Test Procedures

Fracture toughness testing was also performed in ambient conditions (21 – 23°C [70 – 74°F], 45 – 55% rh) under displacement control with a crosshead speed of 1 mm (0.04 in) per minute. Toughness testing employed a single-edge notched, straight-sided specimen geometry (Fig. 2). Due to the thin nature of the adhesive films being tested, plane-strain fracture toughness was unobtainable. However, testing to obtain a valid plane-stress fracture toughness was carried out in accordance with procedures developed in previous research [11 – 13]. Fracture toughness tests were monitored using a Questar long focal length microscope (200X magnification).

To satisfy the requirement of plane stress, the samples met the following criteria:

$$\sigma < \frac{2}{3}\sigma_{ys} \quad (1)$$

$$\frac{a}{W} < \frac{1}{3} \quad (2)$$

where:
σ = applied far-field stress in the specimen
σ_{ys} = yield strength of the material
a = crack length
W = specimen width.

Two measures of fracture toughness were obtained from each test. The effective fracture toughness (K_{Ie}, Equation (3)) relates the initial crack length to the stress at fracture instability. This version of toughness is generally more useful since the initial crack size is often known or is simple to measure. The true fracture toughness (K_{It}, Equation (4)) relates the stress at fracture instability and the crack length at instability. The crack length at instability was visually determined by observing how far each crack extended prior to final, catastrophic fracture.

$$\text{effective fracture toughness } K_{Ic} = f(a/W)\sigma_f\sqrt{a_0} \quad (3)$$

$$\text{true fracture toughness } K_{It} = f(a/W)\sigma_f\sqrt{a_f} \quad (4)$$

where:
σ_f = applied far-field stress at fracture instability
a_0 = initial crack length
a_f = crack length at fracture instability
$f(a/W)$ = geometric correction factor for single-edge notched specimens = $1.12\sqrt{\pi}$.

RESULTS AND DISCUSSION

Properties of Adhesive Films Containing a Scrim Cloth

To compare the behavior of the three adhesives evaluated in this program, in-plane tensile and fracture toughness tests were conducted on versions of the adhesives which contained a scrim cloth. All tests conducted for this portion of the program were performed on specimens in the as-received condition. It should also be reiterated that these tests were conducted with the loading direction in the plane of the scrim cloth although peel and shear loading of bonded structures subjects the adhesive films to out-of-plane tensile and shear loads.

Figures 5 and 6 show tensile curves for the unsupported and scrim-containing versions of the AF-191 and FM® 73 epoxy adhesives. These stress-strain curves are from individual specimens but are typical of the behavior exhibited by multiple specimens. Although the non-woven scrim cloth in these two adhesives is described as a "random mat", specimens containing the scrim cloth were tested in two orientations based upon the rolling direction in which the uncured adhesive film was received: longitudinal (with the loading axis parallel to the rolled direction) and transverse (with the loading axis perpendicular to the rolled direction).

Figure 7 illustrates the behavior of the FM®x5 adhesive in the as-received condition. This curve is typical of the behavior exhibited by multiple specimens. Note that this adhesive has a much higher strength and fracture toughness and a lower failure strain than either of the epoxies. These differences are likely due to the presence of the high volume fraction woven glass carrier cloth which reinforces the brittle adhesive resin.

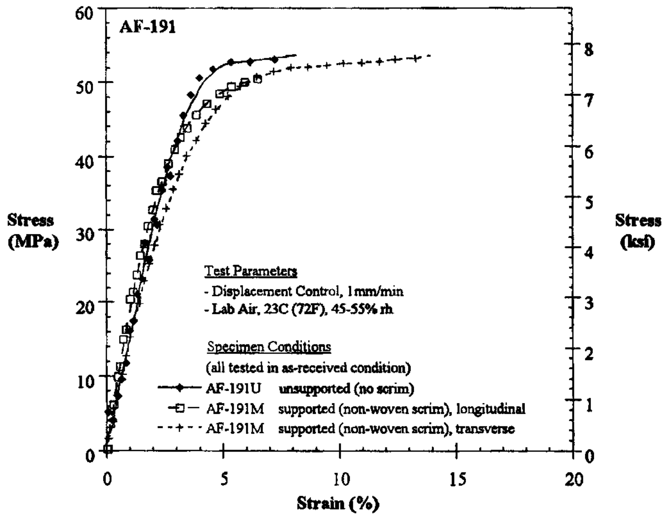


FIGURE 5 Characteristic stress-strain behavior of AF-191U and AF-191M adhesive films.

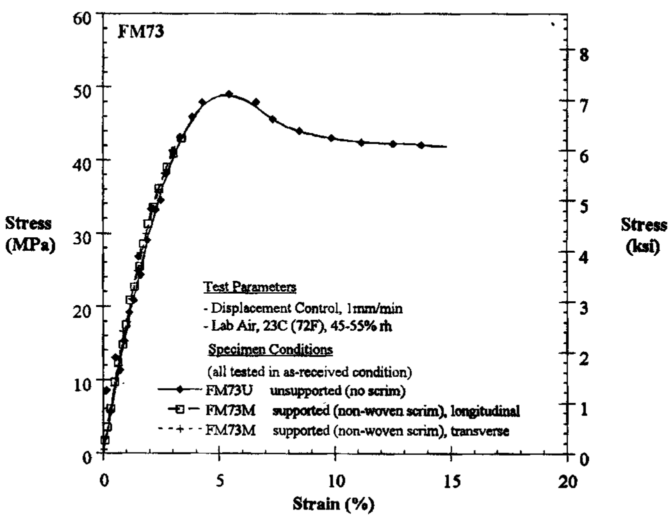


FIGURE 6 Characteristic stress-strain behavior of FM®73 U and FM®73M adhesive films.

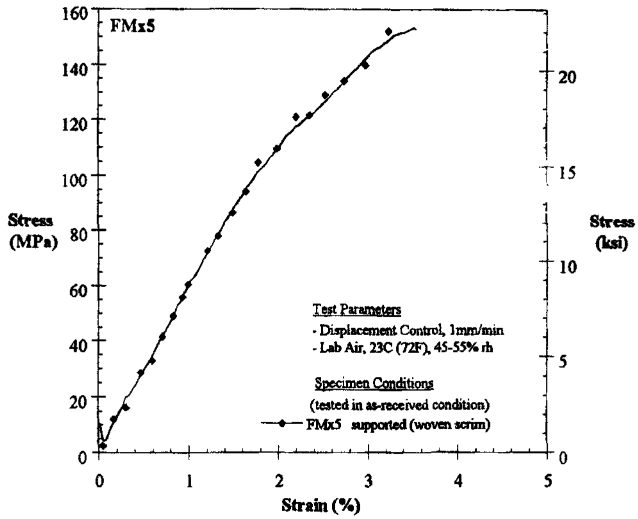


FIGURE 7 Characteristic stress-strain behavior of FM®x5 adhesive film.

Table III shows the mean values of key tensile and fracture properties for the AF-191U, AF-191M, FM®73U, FM®73M and FM®x5 adhesives tested in the as-received condition. A 95% confidence interval was calculated for each adhesive property using the mean, standard deviations, and number of specimens tested. Significant differences between values of the unsupported and supported epoxies occurred when the mean values differed **and** when there was no overlap of the confidence intervals. Such significant differences are indicated by highlighted portions of the Table III. Because confidence interval size depends upon the number of tests conducted, further testing may reduce the size of the current intervals and result in the classification of more of the differences in the mean values of various properties as "significant".

The presence of the scrim cloth reduced the failure strains, strengths, and fracture toughnesses of these two adhesives. No significant differences were noted between the longitudinal and transverse specimen orientations. Therefore, in contrast to the role of the woven glass scrim cloth in the FM®x5, the non-woven scrim cloth did not appear to impart reinforcement and appears to serve only to improve the "handle-ability" of the epoxy adhesive films.

TABLE III In-pane properties of AF-191U (unsupported), AF-191M (random mat scrim cloth), FM®73U (unsupported), FM®73M (random mat scrim cloth), and FM®x5 (woven scrim cloth)

Adhesive, Scrim Orientation [#tested][1]	E (*MPa*)	ε_f (%)	σ_{uts} (*MPa*)	$\sigma_{ys(0.2)}$ (*MPa*)	σ_{iys} (*MPa*)	K_{Ie} ($MPa\sqrt{m}$)	K_{It} ($MPa\sqrt{m}$)
AF-191U							
no scrim [6]	1396±51	8.7±2.3	51±2	40±2	44±1	1.23±0.06	1.31±0.05
AF-191M							
longitudinal [6]	1611±257	7.1±3.8	46±5	**30±2**	**37±3**	**1.03±0.12**	**1.07±0.13**
AF-191M							
tranverse [5]	1457±263	16.1±7.0	51±2	**29±2**	**37±2**	1.09±0.11[2]	1.14±0.13[2]
FM®73U							
no scrim [6]	1432±131	12.2±1.0	47±2	38±2	41±3	2.13±0.14	2.49±0.16
FM®73M							
longitudinal [4]	**1778±138**	**3.6±0.5**[2]	44±1[2]	**30±1**	**35±0**	**1.42±0.15**	**1.61±0.13**
FM®73M							
transverse [5]	1597±133	**3.3±0.4**	**41±2**	**32±1**	**35±1**	**1.54±0.17**	**1.67±0.13**
FM®x5							
woven[6]	5296±763	3.6±1.0	160±19	133±17	140±18	4.63±1.52	4.63±1.52

• significant differences from unsupported values are **boldfaced**.
• 95% confidence intervals shown following ± sign.
[1] number of specimens tested is indicated in brackets [ ] unless note appears within table.
[2] 6 specimens tested instead of number listed in brackets [ ].

Effects of Environmental Exposure

Figures 8 – 10 show characteristic tensile curves for each combination of adhesive and environmental condition tested for this study. These stress-strain curves are typical of the behavior exhibited by multiple specimens.

The unsupported epoxies (AF-191U and FM®73U) exhibited relatively high failure strains as compared with the polyimide (FM®x5) with the failure strains of the FM®73U being highest. These higher strain levels were mainfested by noticeable necking in the gage section of the FM®73U. In general, the AF-191U material showed a smooth transition from elastic to plastic behavior while the FM®73U displayed some stress relaxation. The FM®x5 was almost entirely linearly elastic and exhibited a much higher strength than the epoxies. Though it was expected that the strength of the polyimide would exceed that of the epoxies, the difference observed was magnified by the high volume fraction of woven glass scrim cloth in the FM®x5.

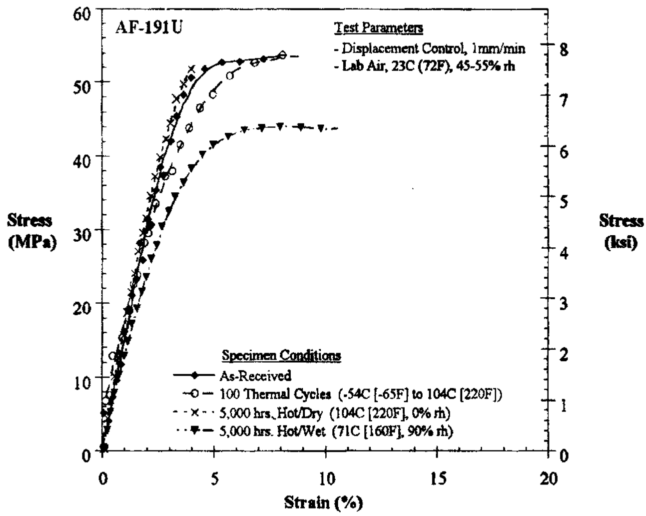


FIGURE 8 Characteristic stress-strain behavior of AF-191U adhesive film.

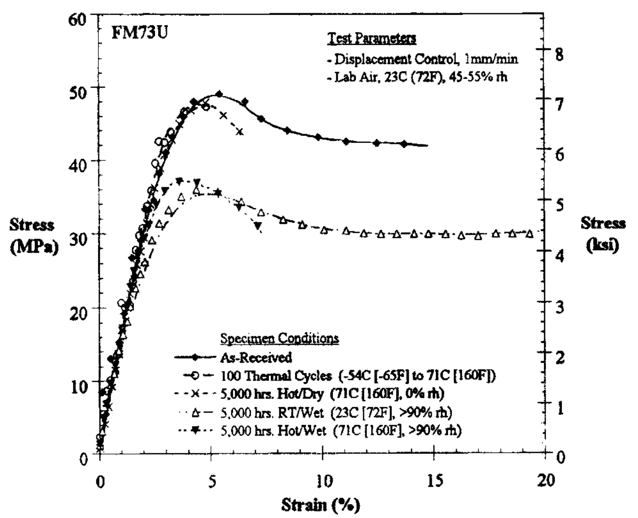


FIGURE 9 Characteristic stress-strain behavior of FM®73U adhesive film.

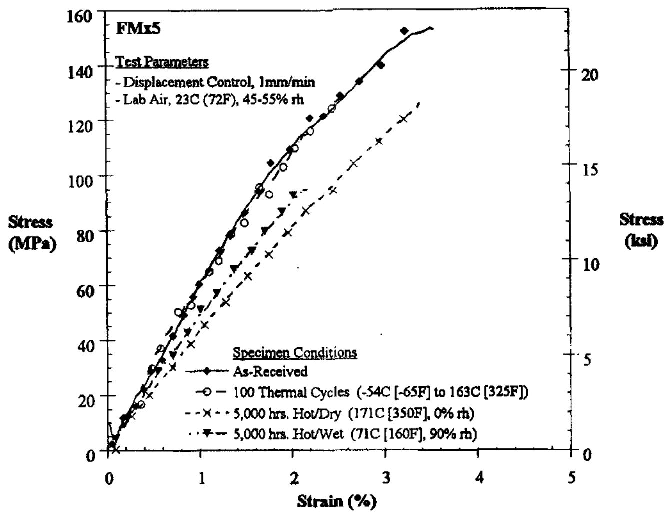


FIGURE 10 Characteristic stress-strain behavior of FM®x5 adhesive film.

Table IV shows mean tensile and fracture properties for the three adhesive film types subjected to various environmental conditions. Significant differences between values of the as-received and exposed specimens are indicated by highlighted portions of the following table.

The confidence intervals for the FM®x5 tests are particularly large. It is likely that slightly different numbers and orientations of scrim fibers present within the gage sections of the FM®x5 specimens increased the observed scatter bands.

Although the moduli of the epoxies were similar, a slight increase in the modulus of the AF-191U was observed in specimens exposed to a hot/dry environment. One possible explanation for this is additional crosslinking caused by the extended time at an elevated temperature. The FM®x5 adhesive had a much higher tensile modulus than the epoxies, but exposure to a hot/dry environment resulted in a slight decrease in the modulus.

The failure strains of the AF-191U and FM®73U films were reduced by exposure to a hot/dry environment, possibly due to increased crosslinking. Exposure to the hot/wet environment resulted

TABLE IV Properties of AF-191U, FM®73U, and FM®73Mx5 adhesive films following exposure to various environments

Adhesive, Condition [#tested][1]	*E (MPa)*	ε_f (%)	σ_{uts} *(MPa)*	$\sigma_{ys(0.2)}$ *(MPa)*	σ_{iys} *(MPa)*	K_{Ie} $MPa\sqrt{m}$	K_{It} $MPa\sqrt{m}$
AF-191U							
as-rec'd [6]	1396±51	8.7±2.3	51±2	40±2	44±1	1.23±0.06	1.31±0.05
cycled [4]	1386±235	11.1±2.7	55±2	37±3	45±2	1.14±0.09	1.22±0.12
hot/dry [3]	**1666±92**	**3.8±0.6**	52±2	41±6	46±1	1.18±0.09	1.18±0.09
hot/wet [3]	1210±159	9.7±3.9	**43±1**	**28±2**	**35±1**	**1.06±0.09**	1.15±0.12
FM®73U							
as-rec'd [6]	1432±131	12.2±1.0	47±2	38±2	41±3	2.13±0.14	2.49±0.16
cycled [4]	1340±43	**5.6±1.2**	49±1	38±3	43±1	2.04±0.10	2.34±0.08
hot/dry [3]	1565±78	**5.9±1.7**	46±3[2]	37±5[2]	40±4[2]	1.89±0.11	**2.20±0.04**
Rt/wet [3]	1386±188	**21.9±3.1**	**36±0**	**29±2**	**31±0**	**1.70±0.08**	2.54±0.54
hott/wet [3]	1543±217	9.6±3.9	**37±2**	**28±1**	**31±1**	**1.79±0.18**	2.36±0.44
FM®x5							
as-rec'd [4]	5296±763	3.6±1.0	160±19	133±17	140±18	4.63±1.52	4.63±1.52
cycled [3]	4827±886	3.6±1.1	151±22	126±19	136±21	4.29±0.91	4.29±0.91
hot/dry [2]	**4177±108**	3.3±0.3	**130±4**	**96±1**	**114±3**	3.42±0.79[3]	3.46±0.84[3]
hott/wet [3]	4778±621	2.6±0.3	**99±9**	**80±32**	**84±19**	3.91±0.59	3.96±0.49

• significant differences from unsupported values are **boldfaced**.
• 95% confidence intervals shown following ± sign.
[1] number of specimens tested is indicated in brackets [ ] unless note appears within table.
[2] 2 specimens tested instead of number listed in brackets [ ].
[3] 3 specimens tested instead of number listed in brackets [ ].

in an increase in the failure strain of the FM®73U. This effect may be due to increased plasticization of the epoxy caused by moisture absorption. The failure strain of the FM®x5 appeared to be unaffected by environmental exposure. It should be noted that large scatter bands (*i.e.*, large confidence intervals) were present in the failure strain data perhaps due to sensitivity of the films to internal flaws, or compositional variability within the specimens.

Exposure to the hot/wet environment resulted in a significant reduction in the ultimate tensile strengths, 0.2% offset yield strengths, and intercept yield strengths for all three adhesives. In addition, the strength of the FM®73U specimens was also reduced during exposure to the RT/wet environment. These trends suggest that high humidity, which resulted in moisture absorption and possible plasticization, played the dominant role in strength degradation under these conditions.

Although the tensile properties of the two epoxies were quite similar, the fracture toughness of the FM®73U was nearly twice that of the AF-191U. This is consistent with specimen behavior observed during preparation. Notching of the FM®73U specimens was performed relatively easily with the sawing motion of a razor blade. During notching of the AF-191U specimens, however, a lead crack immediately formed ahead of the tip of the razor blade.

The behavior of the effective fracture toughness for the epoxies paralleled that of the strength values. K_{Ie} for the AF-191U decreased with exposure to the hot/wet environment. K_{Ie} for the FM®73U decreased with exposure to the hot/wet and RT/wet environments. This trend again suggests that high humidity is the prime detrimental agent. The effective fracture toughness of the FM®x5 was not significantly affected by any of the environmental exposure conditions.

The true fracture toughness did not follow a trend similar to the effective fracture toughness, although it was expected to do so. The difference in the behavior of these two forms of fracture toughness is probably not attributable to material behavior but to the difficulty in accurately determining the crack length at instability. This difficulty was often caused by an inability to traverse the Questar microscope quickly enough to keep the crack tip in the field of view and may have resulted in low values of a_f.

Crack growth in the unsupported epoxies was preceded by a slight blunting of the crack tip and the formation of a whitened, Dugdale-like process zone. The crack then propagated evenly for up to approximately 1 mm in the FM®73U and 0.3 mm in the AF-191U before the growth become unstable.

In contrast to the epoxy specimens, the FM®x5 specimens exhibited much higher fracture toughnesses, larger crack mouth openings, and very limited stable crack growth. Therefore, the effective and true fracture toughnesses were nearly the same for the FM®x5. The adhesive appeared to be significantly reinforced by the scrim cloth since individual fibers bridged the crack. In addition, both forms of fracture toughness of the FM®x5 appeared to be unaffected by environmental exposure. Once again, different numbers and orientations of scrim fibers within the specimen gage sections suggest a reason for the larger confidence intervals for the FM®x5.

Fracture toughness values for all three of these adhesives are of the same order of magnitude as those obtained for other polymers such as cellulose acetate [11], LARC-TPI [12], polyimide-imide [12], Kapton® polyimide [12, 13], and polystyrene [13].

CONCLUSIONS

This study investigated the room temperature tensile and fracture behavior of three adhesive films, AF-191, FM®73, and FM®x5. In a general comparison of all of the adhesives, significant differences existed among the materials. The FM®73 and AF-191 epoxies displayed lower moduli, strengths, and toughnesses but higher failure strains than the FM®x5 polyimide. Between the epoxies, the FM®73 adhesive exhibited the higher failure strain and fracture toughness.

Because structural bonding often employs film adhesives containing a scrim cloth, scrim-containing versions of all three adhesives were tested. The presence of the low volume fraction, non-woven, random mat scrim reduced the strength and fracture toughness of the epoxies (AF-191 and FM®73). However, the strength and fracture toughness of the FM®x5 polyimide benefited from the presence of a high volume fraction woven glass scrim. It is important to realize that this testing was conducted with loads applied in the plane of the scrim cloth in contrast to the out-of-plane peel or shear loads applied in most structural joints.

The understand better the effect of aging and environmental exposure on the adhesives, selected groups of unsupported AF-191 and FM®73, and supported FM®x5 specimens were subjected to thermal cycling or isothermal exposure. Exposure to various environmental conditions affected some of the tensile and fracture properties of the adhesive materials. Thermal cycling between −54°C (−65°F) and the maximum use temperature of the adhesive significantly affected only a single property of one adhesive, reducing the failure strain of the FM®73U. Five thousand hours of exposure to maximum use temperature, low-humidity conditions reduced the failure strains of the AF-191U and FM®73U epoxies and the ultimate and yield strengths of the FM®x5 polyimide. Five thousands hours of exposure to 71°C [160°F], > 90% rh conditions appeared to be the most detrimental. Exposure to this hot/wet condition resulted in significant losses in the ultimate and yield strengths of all three adhesives and a reduction in the effective fracture toughness of the epoxy adhesives.

The results contained in this report suggest a general trend concerning the effects of various service environments on the mechanical behavior of popular aerospace adhesives. These effects, manifested by degraded failure strain, strength, and fracture toughness, can result in the reduction of load-carrying capability and fatigue resistance of adhesively-bonded joints. These reductions in performance should highlight the importance of considering environmental conditions when designing bonded structures.

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