

Degradation of Mechanical Properties of Advanced Composites Exposed to Aircraft Environment

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The degradation of the mechanical properties of Imperial Chemical Industries APC-2 "Victrex" Gr/PEEK laminates was measured after exposure to water, brine, ethylene glycol, hydraulic fluid, jet fuel, and paint stripper. Specimens of the subject material were simultaneously load conditioned in uniaxial tension and immersed in the chemical agents for at least 60 days. Other specimens were immersed in the chemicals with no load conditioning. Specimens from this group were weighed periodically to determine the chemical absorption time history. At the conclusion of the load/chemical exposure period, the specimens were tested to failure to determine the interlaminar shear strengths, the Young's modulus in tension and the ultimate strengths in compression and tension. The results of this investigation showed that after 60 days of immersion, the material absorbed significantly more of the paint stripper and the jet fuel than the other four chemicals. During the same period of time, the strength of the material exposed to the jet fuel and the paint stripper decreased by as much as 8 and 14%, respectively, whereas the other chemicals had less effect. The modulus did not change significantly as a result of chemical exposure.

Introduction

ADVANCED composite material technology has expanded during the past decade to the point that many aircraft manufacturers are committed to using a substantial amount of composite material in the future production of aircraft. The utilization of advanced composite materials in aircraft structures could reduce the aircraft structural weight by as much as 20–30% and thus reduce the fuel consumption and aircraft operating costs. Recently developed advanced composite materials show great promise in increasing the use of these materials in the future.

Composite material laminates inherently absorb fluids from the surrounding environment. The degradation of the mechanical properties of newly developed advanced composite materials, such as high-strain graphite-reinforced thermoplastic resins, exposed to the normal operational environment of aircraft, must be evaluated to assess the productive life of these materials. The normal aircraft operational environment might include moderate to severe long term exposure to ultraviolet light, ice, lightning, wind blown dust and debris, jet wash, and a variety of chemical agents. These chemicals (such as paint strippers, jet fuel, hydraulic fluids, deicing agents, and chemical agents associated with biological warfare) could seriously diminish the in-service mechanical properties of the material. Aircraft designers must be aware of the degree of material degradation to modify "undeteriorated" material property parameters and/or to design protective systems to shield the materials from adverse environmental conditions.

The purpose of this research program was to investigate the influence of several chemicals, found in the aircraft operational environment, on the mechanical properties of a Gr/PEEK composite material that has been proposed for extensive use on a large number of future aircraft. Laboratory tests were conducted in which samples of the material were im-

mersed separately in water, brine, ethylene glycol, hydraulic fluid, simulated jet fuel, and paint stripper for at least 60 days. Approximately half of these samples also were loaded in tension during the period of immersion in the chemicals. At the conclusion of the load/chemical exposure, the samples were tested to failure to determine the change in the Gr/PEEK mechanical properties due to the exposure.

Experimental Procedure

The major elements of this investigation are summarized as follows:

- 1) Initial tests were conducted on specimens of the Gr/PEEK material before exposure to loads and chemicals to determine the baseline mechanical properties of the material.
- 2) One group of specimens was simultaneously loaded to either 20 or 10% of the ultimate tensile strength and immersed in each of the chemical agents. A control group of specimens was loaded but was not exposed to the chemical agents.
- 3) Another group of specimens was immersed in the chemical agents with no accompanying load conditioning. These specimens were removed from the chemicals periodically and weighed to determine the degree of chemical absorption.
- 4) After specimens were exposed to the chemical agents for at least 60 days, mechanical property tests, identical to the initial baseline tests, were conducted on those specimens exposed to both load and chemical agents and those specimens exposed to the chemicals only. These tests were conducted to measure the degradation of the mechanical properties of the Gr/PEEK during this 60-day period of exposure.

This testing program, together with the facilities, equipment and materials necessary for these tests, will be discussed in some detail in the following section.

Design, Construction and Testing of the Environmental Fixture

Environmental test fixtures similar to the one shown in Fig. 1 were designed, constructed, and tested so that samples of the Gr/PEEK material could be uniaxially loaded up to 10–20% of their ultimate strength and exposed to a variety of chemical agents chosen for these tests. The basic design consisted of a wooden structure to transmit the uniaxial load and a tray of plastic gutter to contain the chemical agents. The specimens were linked to each other with connecting plates, and the load was applied in tension through two channel sections at either end of the fixture. One of the channel sections was strain gauged

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so that a specified load could be applied and monitored during the testing period. The fixtures were designed to accommodate a tensile load of up to 2300 lb through the specimens.

A prototype study of the environmental fixture indicated that it could adequately withstand the applied loads and that the loads could be applied and monitored with a reasonable effort. Figure 1 contains a photograph of an environmental fixture with a group of five specimens installed for the load/fluid conditioning.

Separate containers and support racks were designed and built for the specimens that were immersed in the test fluids for 60 days but not loaded. Short segments of plastic gutter capped at both ends contained the chemical, and a coil of steel wire held each specimen in a nearly vertical position and allowed exposure of its entire surface area to the fluid during immersion. With this design the specimens could be easily removed from the fluid (by removing the entire wire coil rack) for weighing.

Material and Specimen Design

Three different types of specimens were fabricated for these tests from an APC-2 Victrex Gr/PEEK prepreg supplied by the Imperial Chemical Industries of the United Kingdom. APC-2 uses type Hercules AS-4 graphite fiber. The APC-2 prepreg had a carbon fiber volume fraction of 61% and corresponding weight fraction of 68%. The carbon fiber axial weight was 145 g/m², and the prepreg had a density of 1.60 g/cm³. Two test panels, one 16-ply quasi-isotropic and the other 18 ply with unidirectionally dominating fibers, were fabricated by the Boeing Military Airplane Company according to Boeing specifications for fabricating thermoplastic advanced composite materials. Test coupons were cut from these panels in the configurations shown in Fig. 2.

The test coupons labeled specimen T in Fig. 2a are no-hole tensile specimens that were prepared from the 16-ply quasi-isotropic panel. The specific layup of this panel, and thus the T specimens, was $(+45, 90, -45, 0, 0, -45, 90, +45)_s$. The principal purpose of the T specimens was to measure the stiffness

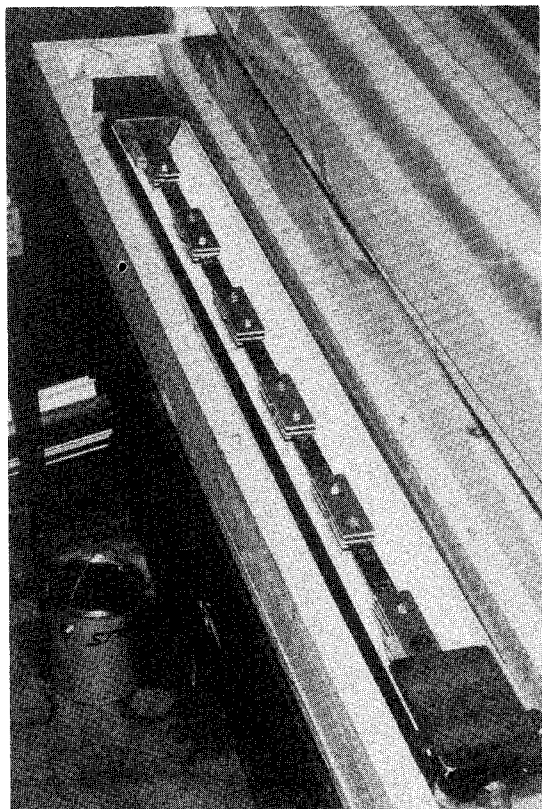


Fig. 1 Environmental test fixture with specimens installed for chemical/load exposure.

and strength of the material in tension. A total of 80 test coupons of this type were prepared.

The second form of test coupon in Fig. 2b is that of the open hole compression specimen, labeled specimen R/C. The specimens of this type also were cut from the 16-ply quasi-isotropic panel with the symmetric layup defined above. The specimens were used in two separate tests, one tension to failure and the other compression to failure, to determine the open-hole ultimate tensile strength and the open hole ultimate compressive strength of the Gr/PEEK material. A total of 120 test coupons were prepared for R/C specimen tests.

The third form of test coupon was that labeled specimen I in Fig. 2c. The specimens of this configuration were of 18 plies with a layup of $(+45, 90, -45, +45, -45, 0, 0, 0)_s$. They were tested in compression to measure the interlaminar shear strength of the material. A total of 80 test coupons were prepared for these tests.

Design and Construction of Compression Stabilizing Fixtures

The testing of specimens in compression required the design and construction of stabilizing fixtures in order to prevent the specimens from buckling. Stabilizing fixtures similar to those used by the Boeing Military Airplane Company for similar compression tests were designed.

The compression fixture shown in Fig. 3 was constructed for the testing with the open-hole compression specimens. The original Boeing design was modified slightly to facilitate the construction of the fixture. The results of the compression tests with this fixture and the R/C specimens appear to be reliable and consistent.

The compression tests for the interlaminar shear specimens also required a compression fixture. Therefore, a second fixture was designed that was similar in concept but smaller in dimensions to the fixture used for the open-hole compression tests.

Chemical Agents

Six chemical agents were selected as being representative of the most likely chemicals that an aircraft component will be exposed to during its service life: distilled water, brine (3% salt by weight), ethylene glycol (de-icing agent), hydraulic fluid, paint stripper (containing methylene chloride) plus methylethylketone and jet fuel. The jet fuel was represented by a simulated jet fuel, Monsanto Standard Test Fluid (MIL-S-3136 Type VII).

Initial Baseline Mechanical Properties

The mechanical properties of the Gr/PEEK material were determined before the load/chemical exposure of the study.

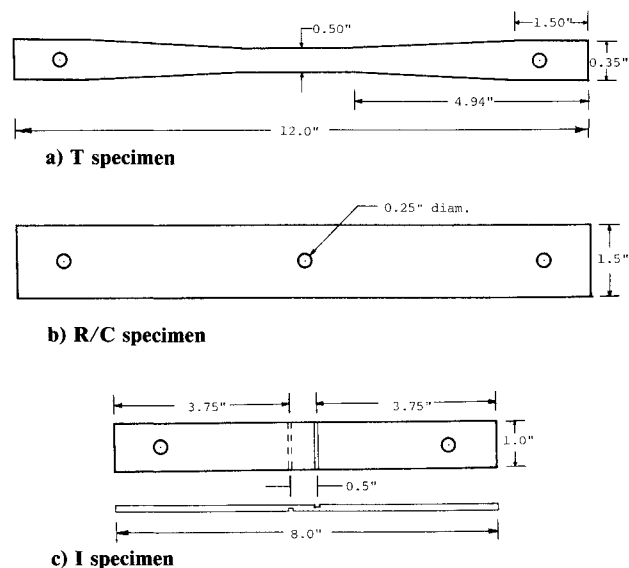


Fig. 2 Test specimen configurations used in the investigation.

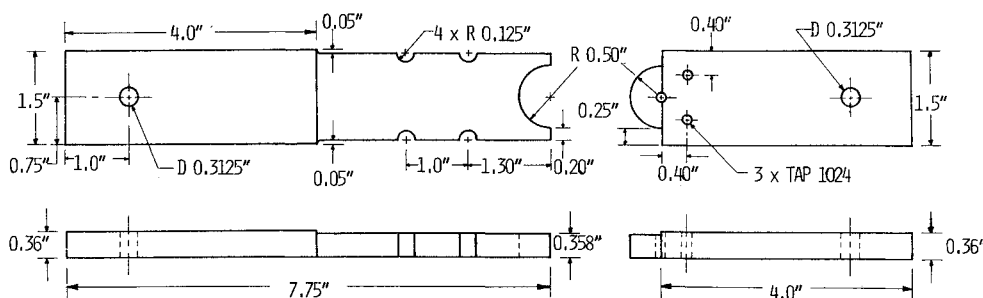


Fig. 3 Compression fixture for the open-hole compression specimen C.

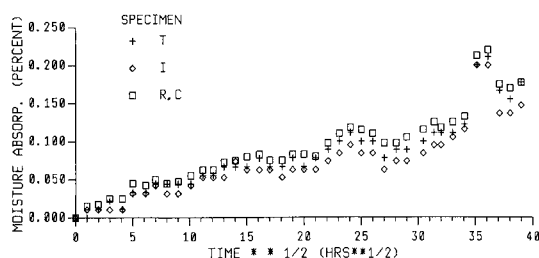


Fig. 4 Water absorption in Gr/PEEK composites.

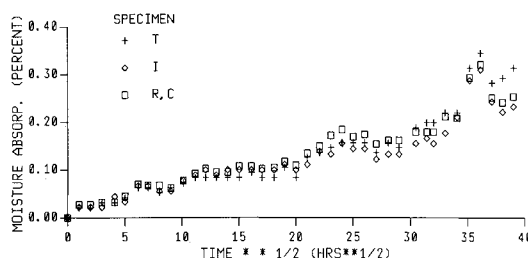


Fig. 5 Saltwater absorption in Gr/PEEK composites.

Five specimens of each combination of specimen type and load were tested to failure in an MTS Universal Testing Machine. The tests conducted and mechanical properties measured are as follows:

- 1) Specimen T: Tension to failure to determine Young's modulus E_T and the ultimate strength in tension, f_T .
- 2) Specimen C: Compression test to failure to determine the open-hole ultimate compressive strength, f_{OC} .
- 3) Specimen R: Tension test to failure to determine the open-hole ultimate tensile strength f_{OT} .
- 4) Specimen I: Compression load to failure to determine the interlaminar shear strength f_S .

The following procedure was employed to determine the mechanical properties of interest:

- 1) Each of the specimens was weighed, placed in an oven at 300°F for 3 h, and reweighed.
- 2) The width and thickness of each specimen was measured at five points within the gauge region.
- 3) The length of each specimen was measured and recorded.
- 4) For compression tests, the specimens were placed in the appropriate compression test fixture
- 5) The specimen (and compression fixture if appropriate) was mounted in the hydraulic grips of the testing machine, with care taken to align the specimen within the machine.
- 6) An extensometer was attached.
- 7) The stroke rate was set to produce a strain rate of 0.02 in./in./min.
- 8) The load-strain history of each test was recorded graphically using the automatic plotting feature of the testing equipment.
- 9) The machine was set to pause at approximately 60% of the ultimate load of the specimen, and the extensometer was removed.
- 10) The load and stroke of the machine were then plotted using the automatic plotting option and the specimen was loaded to failure.

Exposure of Specimens to Chemical Agents and Conditioning Loads

The test specimens were exposed to the six chemical agents of the study for a period of 60 days in two ways. One group of specimens was simultaneously immersed in the chemical agents and uniaxially loaded at either 10 or 20% of their ultimate strength. The other group was immersed in the chemical agents but was not loaded. This was done to simulate the loaded condition of the aircraft while it is exposed to its environment

and to determine the influence of this loaded condition on the absorption of the chemicals and subsequent degradation of the mechanical properties of the material.

Two load levels were used during the load conditioning of the specimens. The interlaminar shear specimens (I-specimens) were loaded to a level corresponding to a shearing stress within the gauge region of approximately 10% of the interlaminar shear strength of the specimen determined during the initial baseline tests. The other two types of specimens (the T specimens and the R/C specimens) were load conditioned to a level of 20% of their ultimate strengths as determined from the initial baseline tests.

With one exception, the specimens were completely immersed in the chemical agents in both the loaded and the unloaded exposure. The one exception was the case of the loaded specimens exposed to the paint stripper. For those specimens, an effort was made to simulate the actual cycle of exposure of an aircraft to paint stripper during its service life. Therefore, the specimens were continuously loaded during the 60 day period but were exposed to the paint stripper according to the following cycle: paint stripper was "painted" onto one flat surface of the specimens and left for 1 day, the paint stripper was thoroughly removed, and the surface cleaned with methylethylketone and remained clean for 6 days. This cycle was repeated every 7 days during the 60 days of exposure.

Chemical Absorption Measurements

The following procedure was implemented to measure the absorption of the chemical agents by the Gr/PEEK material during the 60 days of exposure. Before exposure to the chemical agents, each of the specimens was dried by placing it in an oven at 300°F for 3 h. This was an adequate temperature and duration to dry the material but was low enough to prevent polymer deterioration. The specimens were removed from the oven, cooled, and weighed to determine the initial weight of each specimen.

The specimens then were placed in the appropriate fluid. Each specimen was removed from the chemical according to a schedule such that the time for the i th weighing, t_i , was determined by

$$t_i = i_2 \text{ (hr)}, \quad i = 1, 2, 3, \dots$$

The specimens were weighed individually and in groups of five with a digital scientific scale with an accuracy of ± 0.0005 g.

Special care was taken to remove the excess chemical from the surface of the specimens. Each time the specimens were removed from the fluid to be weighed they were allowed to drain in their supporting rack for a few minutes and then were dried thoroughly with clean dry paper towels. Therefore, each time a specimen was weighed it had a surface that was dry to the touch.

Postexposure Mechanical Property Tests

The mechanical properties of the specimens were determined after exposure to the array of chemical agents and loads of this test program. These tests were initiated after the specimens had been immersed for at least 60 days. The same procedure followed during the initial testing to determine the baseline mechanical properties was followed during these tests, to determine the postexposure properties of stiffness and strength in both tension and compression.

Results and Discussion

The results of the fluid absorption tests are presented in graphical form in Figs. 4–10. The absorption of the fluids as functions of time are presented in Figs. 4–9 for each individual fluid tested, and a summary absorption plot is presented in Fig. 10 for the T specimens.

In each of these figures, the weight gained as a result of fluid absorption was computed in terms of a percent fraction of the total weight of the specimens and plotted as a function of the square root of time. With the exception of the absorption time histories for the paint stripper, each of the plotted points on the graphs represents the average of five specimens. Each of the data points for the paint stripper represent the results for one specimen. Individual graphs are presented for each of the three types of specimens investigated to determine the influence of stacking order, thickness, or other aspects of the geometry on the degree of absorption.

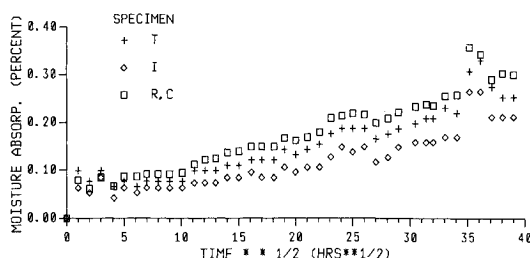


Fig. 6 Hydraulic fluid absorption in Gr/PEEK composites.

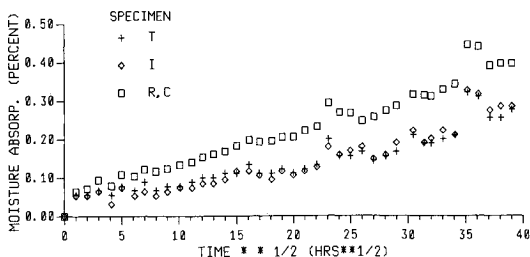


Fig. 7 Ethylene glycol absorption in Gr/PEEK composites.

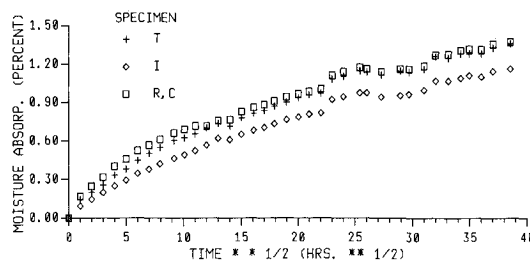


Fig. 8 Jet fuel absorption in Gr/PEEK composites.

The summary plot of Fig. 10 contains the absorption time histories in each of the fluids investigated. Again, each data point represents the summary of measurements of the weight gain of five specimens. These results are for room temperature conditions only and for those specimens that were not subjected to load conditioning during immersion in the chemical agent.

The general nature of the absorption time histories appears to be very similar for each of the chemical agents investigated. That is, the absorption increased steadily with time with a rate that appears to diminish with increased time. There is no evidence that an equilibrium state was achieved; thus, none of the specimens was saturated after 60 days of immersion. This is apparent from the plotted results, since none of the curves had peaked, nor had they reached a constant level of absorption. With very few exceptions the larger R/C specimens, with the greatest exposed surface area per weight and a quasi-isotropic layup, had the greatest rate of absorption, whereas the interlaminar shear specimens (I specimens), with a unidirectional fiber dominated layup, had the least rate of absorption.

The comparison of the absorption histories of the individual types of specimens in each of the chemical agents (Fig. 10) points out the obvious differences in the diffusivity of the Gr/PEEK test material when exposed to these six chemical agents. That is, regardless of the geometry and layup of the specimen, the absorption of water (0.18%), brine (0.25%), ethylene glycol (0.36%), and hydraulic fluid (0.25%) is very small when compared to the absorption of the jet fuel (1.4%) or the paint stripper (as much as 2.4%).

With this difference in the levels of absorption between the various chemical agents, one would anticipate a similar trend in the degradation of the mechanical properties of the Gr/PEEK material. That is, the mechanical properties of the material should be influenced to a greater degree by exposure to the jet fuel and the paint stripper than similar exposure to the other four chemical agents. The results of the mechanical property testing only partially supported this initial hypothesis.

Table 1 contains a summary of the variation of the mechanical properties of the Gr/PEEK material after exposure to the chemical agents only, and Table 2 contains a similar summary of mechanical properties for those specimens that were exposed to both the chemical agents as well as a uniaxial tensile load. A common format of presentation has been adopted for

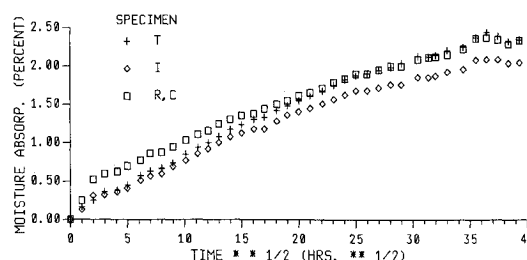


Fig. 9 Paint stripper absorption in Gr/PEEK composites.

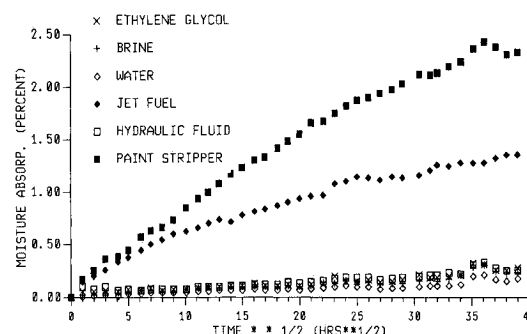


Fig. 10 Comparison of the fluid absorption of the six fluids tested (results from T-specimen data).

Table 1. Variation of the mechanical properties of Gr/PEEK after 60 days of immersion in chemical agents with no load conditioning

Chemical agent	Mechanical properties				
	f_T ksi	f_{OT} ksi	f_{OC} ksi	E_T 10 ³ ksi	f_s ksi
Baseline	125.04 (9.46) ²	72.17 (1.86)	55.71 (2.35)	6.80 (0.53)	5.64 (0.33)
Paint stripper	107.34 (6.58) ⁴	64.79 (1.23)	47.70 (0.76)	6.59 (0.64)	6.12 (0.18) ³
Brine	119.28 (7.84) ⁴	71.54 (3.01)	57.49 (0.35) ³	6.65 (0.44)	6.34 (0.32)
Water	119.92 (7.25) ³	72.71 (4.26)	53.02 (1.85)	6.55 (0.34)	6.33 (0.43)
Ethylene glycol	121.06 (0.91) ²	69.79 (2.23)	54.48 (2.49)	6.77 (0.22)	6.10 (0.53)
Hydraulic fluid	123.60 (7.99) ⁴	73.75 (4.36)	54.22 (2.13)	6.98 (0.46)	5.97 (0.35)
Jet fuel	126.53 (3.03) ²	68.18 (0.51)	55.88 (1.35)	7.03 (0.22)	6.06 (0.34) ⁴
None	120.48 (12.95) ²	71.42 (2.75)	54.44 (2.50)	6.98 (0.19)	5.64 (0.33)

the information contained in these tables. Each column of the tables contain the mean of the measured values of a mechanical property for five specimens (except where noted) exposed to each chemical agent for approximately 60 days. The numbers in parentheses in Tables 1 and 2 are the standard deviations of the mechanical property and the superscript on some of these parentheses indicate the sample size if fewer than 5.

Ultimate Strength in Tension

The influence of the chemical agents on the ultimate strength of the Gr/PEEK material was determined by the examination of the results from the uniaxial load tests in tension on both the open hole specimens (R specimens) and the tension specimens (T specimens). These results are summarized in columns 1 and 2 of Tables 1 and 2.

The results obtained for the tensile tests with the R specimens were more consistent and reliable than the T specimen results. This was due to the tendency for the T specimens to fail at cross sections out of the gauge region of the specimen. Those measured data associated with that mode of failure were not included in the reduced results, thus reducing the statistical accuracy of these results. Furthermore, many of the measured data points retained in the results represent specimens that failed at the point of intersection of the gauge region and the shoulder of the specimen (see Fig. 2a). This, then, could be representative of a severe stress concentration at this inside corner rather than a true failure in tension as a result of exceeding the ultimate tensile strength uniformly across the cross section of the specimen.

Contrary to the ultimate strength results of the T specimens, those results for the R specimens were generally considered to be excellent, that is, consistent and reliable. In fact, all of the specimens failed in the immediate vicinity of the hole at the midpoint of the specimen. The baseline results were consistent, which offered a great deal of confidence about their validity. It also was observed that the ultimate strength of specimens that were aged with no loading or fluid exposure (last entry of column 2 of Table 1) did not vary significantly from the initial baseline test results.

The results of the series of tests on both the T specimens and the R specimens indicate that the ultimate strength of the Gr/PEEK material is altered when immersed in the various chemicals. Somewhat surprising is the observation that the strength did not always decrease when the specimens were exposed to the various chemicals (i.e., the unloaded R speci-

Table 2. Variation of the mechanical properties of Gr/PEEK after 60 days of both immersion in chemical agents and uniaxial load

Chemical agent	Mechanical properties				
	f_T^a ksi	f_{OT}^b ksi	f_{OC}^c ksi	E_T^d 10 ³ ksi	f_s^e ksi
Baseline	125.04 (9.46) ²	72.17 (1.86)	55.71 (2.35)	6.80 (0.53)	5.64 (0.33)
Paint stripper	121.83 (2.88) ²	71.29 (1.89)	54.88 (2.36) ⁴	6.73 (0.51)	5.56 (0.36)
Brine	117.62 (5.16) ⁴	70.80 (0.99)	51.94 (2.84)	6.83 (0.35)	5.75 (0.27)
Water	117.49 (1.83) ³	71.33 (2.81)	54.39 (2.36)	6.76 (0.39)	5.96 (0.27)
Ethylene glycol	129.69 (0.00) ¹	69.05 (3.83)	53.91 (2.90)	7.07 (0.42)	5.69 (0.28)
Hydraulic fluid	124.53 (5.66) ²	69.77 (3.14)	53.82 (1.15)	7.22 (0.18)	6.10 (0.37)
Jet fuel	114.09 (0.00) ¹	67.39 (2.62)	51.11 (1.45)	7.13 (0.23)	5.57 (0.63) ⁴
None	124.18 (7.17)	69.78 (3.47)	55.23 (3.81)	7.05 (0.33)	6.26 (0.37)

f_T^a Ultimate strength in tension for T specimens.

f_{OT}^b Open-hole ultimate strength in tension for R specimens.

f_{OC}^c Open-hole ultimate strength in compression for C specimens.

E_T^d Young's modulus in tension for T specimens.

f_s^e Interlaminar shear strength for I specimens.

mens in either water or the hydraulic fluid). The R specimen results showed that the ultimate strengths of those specimens subjected to an axial stress of approximately 20% of ultimate stress, as well as being immersed in the chemical agents, were always reduced and always decreased as much as or usually more than the unloaded specimens. The paint stripper data would appear to dispute this observation, but it should be noted that the conditions differed drastically between the loaded and unloaded specimens, specifically, the loaded specimens were not immersed in the paint stripper for the full period of testing.

The T-specimen results did not completely support these general findings from the R specimens. That is, the decrease in ultimate strength of the loaded specimens did not always exceed the loss of strength in the unloaded specimens, as shown by the results for the ethylene glycol and no fluid in column 1 in Tables 1 and 2. However, the raw data for these two cases indicate that the unloaded specimens with no fluid exposure were represented by just two valid measured values of ultimate strength that differed by approximately 15%, and the loaded specimens immersed in ethylene glycol had but one valid measured strength value. Thus, it would be difficult to dispute the general trend between the difference in the degradation of loaded specimens and unloaded specimens established by the R-specimen results on the basis of the limited valid data of the T specimens.

Because of this general concern about the overall validity of the T-specimen ultimate strength data, the evaluation of the effect of exposure to the chemical agents and load on the ultimate strength of Gr/PEEK has been based on the R-specimen results. Column 2 of Tables 1 and 2 clearly indicates that the immersion in the paint stripper, the jet fuel, and the ethylene glycol had a much more significant effect on the degradation of the ultimate strength of the material than the other chemicals. The material lost an average of 4% of its initial ultimate strength when immersed in the ethylene glycol and 6% when immersed in the jet fuel in the loaded condition, and it lost about 3% when immersed in ethylene glycol in the unloaded condition. The loss of ultimate strength was even more pronounced when the specimens were immersed in the paint stripper where the material lost an average of 10% of its initial ultimate strength. Note again that the material was not

immersed in the paint stripper in the loaded condition. Based on the results of these tests (R-specimen tests), one might speculate that the loss of strength would be greater for the combination of immersion in the paint stripper in the loaded condition.

Relative to the loss of strengths in paint stripper, jet fuel, and ethylene glycol, the losses of strength in the other chemical agents were very small, on the order of 1–3%. In fact, the ultimate strengths of the specimens immersed in the distilled water and the hydraulic fluid with no applied axial force actually show an increase in ultimate strength of 1–2%. Finally, note also that the specimens that were loaded in tension but not immersed in a fluid (last entry of column 2 of Table 2) experienced a loss of 3% ultimate strength.

Ultimate Strength in Compression

The general consistency of the results of the compression tests on open hole specimens (C specimens) was nearly as good as that of the R specimens. As expected, the influence of the chemical agents on the ultimate strength in compression was found to be very similar to that of the ultimate strength in tension. That is, all of the loaded specimens experienced a loss in strength, and the loss was always equal to or greater than the loss for the specimens that were immersed in the fluids but not loaded in tension. The only exceptions to this general behavior were the specimens in the paint stripper (for the obvious reasons discussed in the previous section) and those in the distilled water. The difference in the average strengths of the loaded and unloaded specimens in the distilled water was about 2%. The only average strength that increased significantly (3%) during the test period was that of the unloaded specimens immersed in the brine solution for 60 days. The measured data for this particular test indicate that there were only three valid data points for the unloaded test.

Again, similar to the ultimate strength in tension results, the chemicals that had the most influence on the ultimate compressive strength were the paint stripper and the jet fuel. Those specimens immersed in the paint stripper lost approximately 14% of their compressive strength, whereas the loaded specimens in the jet fuel lost approximately 8% of their compressive strength. Unlike the tension tests, the ethylene glycol did not seem to have a particularly strong influence on the compressive ultimate strength, but the loaded specimens in the brine had a 7% loss of strength. Furthermore, the loaded and the unloaded specimens that were not immersed in a chemical agent lost about 1–2% of their initial compressive strengths.

Axial Modulus of Elasticity

The modulus data measured during the testing of the T specimens are contained in column 4 of Tables 1 and 2. Even though the ultimate strength data associated with these same T specimen tests were considered to be seriously deficient, the modulus data appear to be consistent and complete. These results are in distinct contrast to the strength results discussed earlier.

For example, the chemical agents appeared to have much less effect on the modulus of the material than on the strength. The greatest deviation from the initial baseline modulus value was only 6% and occurred in the case of loaded specimens in hydraulic fluid. Nine of the fourteen groups of five specimens either increased in stiffness or changed by less than 4% from the initial value. Even though the Gr/PEEK material absorbed significantly more paint stripper and jet fuel than the other chemical agents, and the strengths of those specimens immersed in paint stripper and jet fuel were reduced more than those in the other fluids, the modulus was not significantly reduced by either agent. In fact, the modulus of the specimens immersed in the paint stripper was reduced by approximately 3%, whereas those immersed in the jet fuel actually experienced a 3–4% increment in modulus. Finally, in most cases the modulus of the unloaded specimens always decreased more

than that of the loaded specimens. This finding is also the opposite of the trend observed in the strength property.

Interlaminar Shear Strength

The interlaminar shear results are the most difficult to explain. The primary difficulty arises from the fact that the baseline data is not well established. Early difficulties with the original compression support fixture led to the initial baseline tests being dominated by a buckling mode of failure. The initial compression fixture was abandoned, and a new fixture was designed and constructed. The new fixture appeared to work very well, since the compression failure loads were fairly consistent within each group of five specimens as well as between groups. The only exception was the no-load, no-fluid group of specimens that was to be used as the baseline results. That group of specimens had the smallest interlaminar shear strength except for those loaded specimens in the paint stripper and those in the jet fuel. Furthermore, the shear strength of that no-load, no-fluid group is significantly (11%) less than the shear strength of the loaded, no-fluid group. This is also inconsistent with the remainder of the I specimen test results. Additional no-load, no-fluid tests were conducted on the specimen to better determine an appropriate value for the baseline. The results of these two additional tests supported the earlier tests for the no-load, no-fluid specimens; thus, the interlaminar shear strength results of these no-load, no-fluid results were accepted as the baseline of comparison of the effects of the environmental conditions on the interlaminar shear strength.

Based on the results of these tests, the interlaminar shear strength was increased by immersion in all of the chemical agents. It increased by a smaller amount if the specimens were also subjected to an axial stress in tension of approximately 10% of the initial ultimate stress.

Statistical Analysis

A statistical evaluation of the experimental data was conducted to provide a measure of the statistical evidence that the fluid exposure significantly altered the mechanical properties of the material. A Student's t test was employed to test the hypothesis that the mean value of the mechanical property was different from the baseline mean value of the mechanical property.⁶ The standard deviations of the populations of the baseline mechanical property and the mechanical property after exposure were both unknown but were assumed to be equal. The basic procedure involved computing a test value of t , given by

$$t = \frac{\bar{x} - \bar{y}}{s[1/n + 1/m]^{1/2}}$$

where \bar{x} is the mean value of the sample of the baseline property, \bar{y} the mean value of the sample of the property after exposure, n the baseline sample size, and m the postexposure sample size. The parameter S is given by the expression

$$s^2 = \frac{(n-1)v^2 + (m-1)w^2}{n+m-2}$$

where v^2 is the variance of the baseline sample and w^2 the variance of the afterexposure sample.

This value of t was then compared to $t_{n+m-2, \alpha}$ of a standard Student's t table, where α is the selected level of significance. Thus, if

$$t > +t_{n+m-2, \alpha}$$

the mean value of the mechanical property was said to have decreased significantly at the $100\alpha\%$ level of significance. Similarly, the mean value was judged to have increased significantly if

$$t < -t_{n+m-2, \alpha}$$

The results of this statistical evaluation indicated that the ultimate strength in compression was reduced significantly at the 5% level of significance when the material was immersed in paint stripper and water in an unloaded condition and when immersed in jet fuel accompanied by axial load. Likewise, the ultimate strength in tension was seen to decrease after immersion in paint stripper and jet fuel with no accompanying load and after immersion in ethylene glycol and jet fuel with axial load during exposure. On the other hand, the increase in interlaminar shear strength was found to be statistically significant at the 5% level of significance when the material was exposed to paint stripper, brine, water, and jet fuel with no load and after exposure to hydraulic fuel and aging (with no chemical exposure) while stressed at 10% of ultimate stress for 60 days. The same statistical test applied to the modulus data indicated that the modulus was unaltered by the exposure to any of the chemical agents with the same 5% level of significance.

Conclusions

The following general conclusions are submitted regarding the chemical absorption characteristics of the Gr/PEEK (APC-2) composite material and the degradation of the mechanical properties of this material as a result of exposure to the chemical agents.

Absorption Characteristics

1) None of the specimens reached an absorption equilibrium condition (saturation) in any of the six fluids after 60 days of immersion.

2) The material absorbed a great deal more of both the simulated jet fuel and the paint stripper than any of the other four chemicals.

3) The 16-ply, quasi-isotropic, open-hole compression specimens consistently absorbed the fluids at the greatest rate. The 16-ply, quasi-isotropic tensile specimens had the next greatest rate of absorption, and the 18-ply interlaminar shear specimens had the smallest rate.

Mechanical Property Degradation

1) The T-specimen configuration did not prove to be well suited for the ultimate strength measurements. The failure surface was frequently not in the gauge region of the specimen.

2) Very consistent open-hole ultimate tensile strength data were obtained with the open-hole compression specimens. The failure surface was always at the center hole, and the variation of the measured strength was very small from one specimen to another.

3) The ultimate strengths in tension and compression of the specimens immersed in the chemicals and loaded to 20% of ultimate strength always decreased and decreased as much as, and generally more than, those exposed to the chemicals in the unloaded state.

4) The ultimate tensile strength usually decreased after exposure to the chemicals; however, the average strength of

those unloaded specimens exposed to water and hydraulic fluid did increase.

5) The greatest reduction in the tensile ultimate strength occurred in the specimens immersed in the paint stripper, ethylene glycol, and simulated jet fuel. Typically, the change in the ultimate tensile strength due to other chemicals was significantly small.

6) The greatest reduction in compressive ultimate strength occurred in the specimens immersed in the paint stripper, the loaded specimens in the simulated jet fuel, and the loaded specimens in the brine solution.

7) The change in the compression ultimate strength in the water, ethylene glycol, and hydraulic fluid was significantly, small.

8) The only increase in compressive ultimate strength was for the unloaded specimens in the brine solution.

9) The chemicals effected the tensile modulus of the material much less than the ultimate strength. The greatest deviation occurred for the loaded specimens in the hydraulic fluid.

10) The modulus of the specimens immersed in the paint stripper and the simulated jet fuel was not altered significantly.

11) In most cases the modulus of the unloaded specimens was less than that of the loaded specimen after being immersed in the same fluid for 60 days.

12) The interlaminar shear strength was increased by immersion in each of the fluids.

13) The increase in interlaminar shear strength was less for those specimens that also were loaded to 10% of the ultimate load while immersed in the chemical than for the unloaded specimens.

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References

- ¹Shen, C. H. and Springer, G. S., "Moisture Absorption and Desorption of Composite Materials," *Journal of Composite Materials*, Vol. 10, Jan. 1976, pp. 2-20.
- ²Loos, A. C. and Springer, G. S., "Moisture Absorption for Graphite-Epoxy Composites Immersed in Liquids and in Humid Air," *Journal of Composite Materials*, Vol. 13, April 1979, pp. 131-147.
- ³Shirrell, C. D. and Halpin, J., "Moisture Absorption and Desorption in Epoxy Composite Laminates," *Composite Materials: Testing and Design*, American Society for Testing and Materials, ASTM STP 617, 1977, pp. 514-528.
- ⁴Hancox, N. L., "The Environmental Response of Hybrid Composites," *Journal of Materials Sciences*, Vol. 19, April-June 1984, pp. 1969-1979.
- ⁵Neumann, S. and Marom, G., "Free-Volume Dependent Moisture Diffusion Under Stress in Composite Materials," *Journal of Materials Science*, Vol. 21, Jan. 1986, pp. 26-30.
- ⁶Bowker, A. H. and Lieberman, G. J., *Engineering Statistics*, 2nd ed., Prentice-Hall, Englewood Cliffs, NJ, 1972, pp. 235-240.