ULTRA-HIGH MODULUS ORGANIC FIBER HYBRID COMPOSITES

by

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ULTRA-HIGH MODULUS ORGANIC FIBER HYBRID COMPOSITES

SUMMARY

An experimental high modulus organic fiber, designated for the purposes of this contract as Fiber D, has been characterized, and its performance as a reinforcement for composites has been investigated. The fiber has a modulus of 172 GPa (24.9 x 10 psi), tensile strength of 3.14 GPa (456 ksi) and density of 1.463 gm/cm. Thermogravimetric and differential thermal analyses show that Fiber D is stable in air to 500°C. The fiber was also found to have a relatively low moisture gain of 1.3% after exposure to 75% relative humidity at 21°C.

Fiber D/epoxy laminates containing 60% fiber by volume were evaluated in flexure, shear, and compression at room temperature and 121°C in both the as-fabricated condition and after humidity aging at 95% RH and 82°C for 14 days. Moduli of 94.1 GPa (13.7 x 10° psi), flexure strengths of 700 MPa (102 ksi), shear strength of 54 GPa (7.8 ksi) and compressive strengths of 232 MPa (34 ksi) were obtained at room temperature. As-fabricated composites at elevated temperature and humidity-aged material at room temperature had properties 1-20% below these values. Combined humidity aging plus elevated temperature testing resulted in decreases of 50% or more in mechanical properties due to degradation of the epoxy matrix.

Hybrid composite laminates of Fiber D with 20 and 40 volume percent FP alumina fiber or Thornel® 300 graphite were also evaluated. For Fiber D/Fiber FP hybrids, modulus increases of 60% and compressive strength increases of up to 4X were obtained over the all-Fiber D composite. Flexure strength of the Fiber D/Fiber FP system was observed to go through a maximum at 20-30% Fiber FP because of the large differences in moduli and elongation of the two fibers. For Fiber D/graphite hybrids, increases in modulus, flexure strength, and compressive strength of 20%, 60% and 150%, respectively, were observed.

Impact behavior, as measured by the instrumented Charpy test, was also evaluated for Fiber D/epoxy composites as well as for hybrids of Fiber D with Fiber FP or Thornel® 300 graphite. A Charpy impact energy of 0.15 J/mm² (130 ft-lb/in²) was observed for Fiber D/epoxy laminates containing 60 volume per cent fiber. This impact energy is \$2X\$ higher than Thornel® 300/epoxy and \$25X\$ higher than Fiber FP/epoxy laminates and is comparable to Kevlar® 49/epoxy laminates.

I INTRODUCTION

This is the final report for NASA Contract No. NAS3-21837 entitled "Ultra-High Modulus Organic Fiber Hybrid Composites". This program originated out of NASA's interest in a high modulus, high strength organic fiber as a potential replacement for graphite fibers. This interest was stimulated by concerns that electrically conductive graphite fibers could potentially short out electrical equipment if they were accidentally released to the atmosphere and became airborne.

The Du Pont Company has been involved in research on high modulus organic fibers for over 15 years, and during this time many new compositions have been identified. Kevlar® aramid, commercialized by Du Pont, is one example. In addition, several fiber compositions having tensile strength ≥ 2.8 GPa ($\geq 400,000$ psi) and moduli ≥ 172 GPa ($\geq 25 \times 10^{\circ}$ psi) have been demonstrated on a laboratory scale. Densities of these organic fibers are low (<1.5 gm/cm³), and therefore specific properties are attractive for reinforcing advanced composities.

For this contract Du Pont supplied a research quantity of an experimental high modulus fiber, designated for the purposes of this program as Fiber D. A 12 month, 4 task effort was then carried out to characterize the fiber, fabricate and characterize the mechanical properties of Fiber D/epoxy laminates, fabricate and evaluate hybrid composites of Fiber D with graphite and Fiber FP alumina fiber, and evaluate the impact behavior of these materials. In the following sections the results of these tasks are presented and discussed.

II. FIBER CHARACTERIZATION

Approximately 1.4 kg (3 lbs) of Fiber D were obtained to carry out the tasks of this contract. The fiber supply consisted of 23 bobbins of continuous yarn containing 160 filaments. Before any composite laminates were fabricated, the fiber was characterized in the following ways: tensile strength and modulus, density, specific properties, optical and scanning electron microscopy, thermogravimetric analysis, differential thermal analysis, specific heat, and moisture regain.

a. Fiber Tensile Strength and Modulus -Resin Impregnated Strands

Fiber tensile strength and modulus were determined for each of the 23 yarn bobbins. The method used was the ASTM D2343 resin impregnated strand test. Strand tests were performed on each bobbin at a gage length of 24.5 cm (10 in). Strands were impregnated with an epoxy formulation consisting of 100 parts "Epon" 826 epoxy, 25 parts "Araldite" RD-2 epoxy, and 30 parts "Tonox" 60-40 curing agent. A typical stress-strain curve from a resin impregnated strand test is shown in Fig. 1. The ultimate tensile strength of this strand was approximately 3.4 GPa (500 ksi). The stress-strain curve shows a slight upward curvature indicating that the fiber stiffness increases slightly with increased strain. The strand modulus was obtained from a least squares fit to the initial portion of the curve.

Five strand tests were performed for each yarn bobbin. The strand data were then corrected to subtract out the strength and stiffness contribution of the epoxy resin in order to obtain fiber strength and modulus. Results are summarized in Table 1. The values given for each bobbin are the average and standard deviation of 5 strand tests. The average values for all 115 tests were:

Ultimate Tensile Strength: 3.14 ± 0.23 GPa (456 ± 33 ksi) Tensile Modulus: 172 ± 4 GPa (24.9 x 10⁶ psi)

b. Fiber Density

The density of Fiber D was determined by the Density Gradient Column Method - ASTM D15056-68. A density gradient column consists of a glass tube containing a mixture of liquids with densities increasing smoothly and approximately linearly from top to bottom. A small sample put into the column comes to equilibrium at the level corresponding to its own density. The column is calibrated with floats of known density. The density of the sample is determined by comparing its position in the column relative to the position of the calibration standards.

The 23 bobbins of yarn represent six separate spins. One bobbin was chosen from each spin and four density determinations were made on each bobbin. Results are shown in Table 2. The values given in the table are the average and standard deviation of four determinations. The average of all 24 determinations gave a density for Fiber D of $\rho = 1.463 + .002 \text{ gm/cm}^2$.

c. Specific Strength and Modulus

The specific tensile strength σ/ρ and specific tensile modulus E/ρ were calculated from the average tensile strength and modulus shown in Table 1 and the average density given in Table 2. The results are:

Specific tensile strength: $\sigma/\rho = 21.8 \times 10^6$ cm (8.63 x 10^6 in) Specific tensile modulus: $E/\rho = 12.0 \times 10^8$ cm (4.71 x 10^8 in)

The specific strength and modulus of Fiber D are plotted in Fig. 2 along with values for several common fibers and materials. It can be seen that the specific tensile properties of Fiber D compare favorably with HT graphite. The specific modulus of Fiber D is approximately 35% greater than Kevlar® 49. (Data for Kevlar® 29 and 49 are from Du Pont. Data for other fibers in Fig. 2 are from manufacturer's literature.)

d. Modulus of Single Filaments

In addition to measuring the modulus of Fiber D on 25.4 cm resin impregnated strands the modulus of 25.4 cm single filaments was determined for comparison. Five determinations of single filament modulus were made for each of the 23 yarn bobbins. Tests were performed on a Model TM Instron tensile test machine using rubber-faced pneumatic grips. Results are shown in Table 3. The average single filament tensile modulus of 169 ± 12 GPa (24.5 $\pm 1.7 \times 10^{\circ}$ psi) is statistically indistinguishable from the value of 172 ± 4 GPa (24.9 $\pm 0.6 \times 10^{\circ}$ psi) obtained from the strand tests. The scatter in the single filament data is somewhat larger than in the impregnated strand test data as one would expect.

e. <u>Tensile Strength of Single Filaments as a</u> Function of Gage Length

Tensile strengths of single filaments were measured for each of the 23 fiber bobbins at 0.254, 2.54 and 25.4 cm (0.1, 1 and 10 in.) gage lengths. Five breaks were made at each length. Results are given in Table 4 and are plotted in Fig. 3. At 0.254 cm gage length an average value of 3.44 ± 0.61 GPa (499 \pm 89 ksi) was obtained. This value is significantly higher than the values of 2.35 ± 0.59 GPa (341 \pm 86 ksi) and 1.93 ± 0.69 GPa (280 \pm 100 ksi) obtained at 2.54 and 25.4 cm gage lengths, respectively, and is comparable to the value of 3.14 ± 0.23 GPa (456 \pm 33 ksi) for 25.4 cm resin impregnated strands. The strength determined from resin impregnated strands should be more representative of the fiber strength contribution in composite laminates.

The single filament strengths show considerable scatter as indicated by the large standard deviations. The lower strength at longer gage length is consistent with a flaw-dominated failure mode (i.e., the probability of finding a severe flaw is higher for longer gage lengths). The tensile strength of Kevlar[®] 49 vs gage length is shown in Fig. 3 for comparison (Ref. 1). Results show that Fiber D has a stronger dependence of strength on gage length. However, it must be remembered that Fiber D is an experimental fiber for which processing conditions have not been optimized.

f. Optical and Scanning Electron Microscopy

An optical micrograph of a resin impregnated strand of Fiber D is shown in Fig. 4. The impregnated strand was encapsulated in an epoxy mount for stability, and a cross-section was cut with a diamond knife microtome and photographed on a Reichert Model ME F2 metallograph. (Lines in the micrograph are caused by the microtome.) The micrograph shows that the filaments are round in cross-section and the the yarn contains 160 filaments.

Diameters of individual filaments were measured at 675X magnification using a Bausch & Lomb microscope with a calibrated eyepiece. The 23 bobbins of yarn prepared for this program represent six separate spins. One bobbin was chosen from each spin,

and 5 measurements of filament diameter were made on each bobbin. Results are shown in Table 5. An average filament diameter of 11.7 <u>+</u> 0.6 microns was obtained.

Samples of Fiber D were also examined by scanning electron microscopy (SEM). Figs. 5a and b show the fiber surface at 500X and 2000X, respectively. (A 10 micron marker is shown in each figure.) The virgin fibers have essentially smooth surfaces, but some fibrils are seen along the fiber length. Figs. 5c and d show a yarn end which was cut with scissors. In these micrographs the fibrilar nature of Fiber D is clearly seen. This type of internal microstructure is also seen in other high modulus organic fibers such as Kevlar[®].

g. Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA)

Thermogravimetric analysis of Fiber D was run on a Du Pont Analyzer Model 950. A 3.4 mg sample was heated at 20°C/min from room temperature to 750°C in nitrogen. TGA trace is shown in Fig. 6. The figure shows that there is almost no weight change to approximately 550°C. Between 550° and 600°C a weight loss of $^{25\%}$ is observed. A more gradual weight loss is observed between 600° and 750°C. This result shows that Fiber D has excellent stability in nitrogen.

The experiment was also carried out in air, and results are shown in Fig. 7. A 5.5 mg sample of Fiber D was heated in air at 20°C/minute from room temperature to 625°C. As shown in Fig. 7 only a small weight loss (5%) was observed between room temperature and 500°C. This indicates excellent oxidative stability of Fiber D. Between 500° and 580°C rapid weight loss is observed. At 600°C a residue of approximately 0.1 mg (σ 2 wt %) remained.

Differential thermal analysis was also performed on Fiber D. A DTA trace is shown in Fig. 8. A sample of fiber was heated in nitrogen from room temperature to 700°C at 20°C/minute in a Du Pont Model 900 Thermal Analyzer. In the region between 500° and 600°C there is a small endotherm at 510°C and a larger one at 580°C. TGA shows that the fiber loses weight in this temperature region. These two results are consistent with fiber decomposition.

h. Specific Heat

The specific heat of Fiber D was determined at 45°C and 250°C using a Du Pont Differential Scanning Calorimeter Model 990. A sapphire standard was used to calibrate the instrument. The following values were obtained:

Temperature	Specific Heat
(°C)	<u>(joules/gm°C)</u>
45	1.44
250	1.58

i. Moisture Regain

Moisture regain experiments were carried out to determine if Fiber D absorbs a significant amount of moisture. 4 gm skeins of yarn were dried in an oven at 105-110°C overnight and were then exposed to two controlled humidity environments: 65% and 75% RH at 21°C (70°F). Weight gain as a function of time was measured, and results are shown in Fig. 9. At 65% RH the yarn gained 0.95% moisture by weight in 48 hrs, and at 75% RH a 1.3% weight gain was observed. Heating the samples to 105-110°C reversed the weight gain. Because of this tendency of Fiber D to absorb a small amount of moisture, all yarns were carefully dried before fabrication of composite laminates.

III. FIBER D/EPOXY LAMINATE FABRICATION AND EVALUATION

a. Preliminary Evaluation and Resin Selection

After Fiber D was characterized as discussed in Section II, its performance as a reinforcement in resin matrix composites was evaluated. Preliminary experiments were carried out to investigate fabrication procedures and to select the resin matrix that would be used throughout the remainder of this program.

Unidirectional laminates 12.7 x 20.3 x 0.3 cm (5 x 8 x 0.12 inches) having nominal 60 volume percent Fiber D were fabricated for preliminary evaluation. Four epoxy resins were selected; these are shown in Table 6 along with the cure cycles used. 12.7 cm (5 in.) wide prepreg strips of Fiber D were made with each resin system. The strips were then stacked into a 12.7 x 20.3 cm die and vacuum bag/compression molded to give the desired laminates.

Density, fiber volume fraction and void content of the four laminates were determined, and results are shown in Table 7. Densities were determined by the Density Gradient Column method described in Section II above. Obtaining an accurate value of the void content of composite laminates is difficult. Digestion of the epoxy matrix with nitric acid to obtain resin and fiber content is a frequently used method. However, it was found that nitric acid also reacts to some extent with Fiber D and gives inaccurate determinations of fiber volume fraction. The method used here was to keep accurate account of the weights of fiber and resin used in each laminate and calculate what the density would be if there were no voids. This value was then compared to the measured density, and any difference was taken as an estimate of porosity in the laminates. The four laminates had void contents of 1-3%. Values of 3% are higher than desired, and it was determined that this resulted from an insufficient amount of resin being applied during laminate fabrication. For fabrication of future laminates, a slight excess of resin was applied and allowed to bleed off during the cure cycle so that void contents of less than 1% could be obtained.

Test specimens were obtained from the four Fiber D/epoxy laminates, and flexural strength and modulus, short beam shear strength, and compression strength were determined. Flexure specimens were 20.3 x 1.3 x 0.3 cm (8 x 0.5 x 0.12 in.) and compression specimens were 11.4 x 0.64 x 0.3 cm (4.5 x 0.25 x 0.12 in.). Flexural moduli were determined in 3-point bending using a span/thickness ratio of 60/1. Samples were not loaded to failure for modulus determinations. Flexural strengths were then determined on the same specimens using a span/thickness ratio of 17/1. Short beam shear strengths were determined on the ends of the flex bars using a span/thickness ratio of 4/1. Compression strengths were determined by using a Celanese test fixture. The test specimens were fitted with doubler tabs so that a 1.27 cm (1/2)in.) gage section remained.

Test results are shown in Table 8. Four replicate tests were made for each mechanical property. Flexural moduli vary from 92.4 to 115.1 GPa (13.4 to 16.7 x 10° psi). These variations are believed to be due to variations in V_f and void content.

Both flexural yield strengths and ultimate flexural strengths are reported in Table 8. A typical load vs. deflection curve from a 3-point bend test is shown in Fig. 10. Yield srength values are calculated at 0.02% offset. Flexural yield strengths varied from 254 MPa (36.8 ksi) for the "Epon"-RD2/"Tonox" system to 310 MPa (44.9 ksi) for "Epon"/NMA/BDMA. Ultimate flexural strengths ranged from 564 MPa (81.8 ksi) for 3501-6 resin to 745 MPa (108 ksi) for the BP-907 and "Epon"/NMA/BDMA systems. Failures appear to initiate on the compressive side of the flexure specimens. This is consistent with the fact that flexure strengths are only about 1/2 what one would expect for tensile failures. This behavior has been observed in other high modulus organic fibers. For example, flexural yield and ultimate strengths for Kevlar[®] 49/epoxy composites are typically 345 MPa (50 ksi) and 620 MPa (90 ksi), respectively, at 60 volume "% fiber (Ref. 1).

Compressive yield strengths were 180 to 223 MPa (26.1 to 32.3 ksi) and ultimate compressive strengths were 218 to 241 MPa (31.6 to 35.0 ksi) for all four resin systems. These values are also similar to those for Kevlar® 49/epoxy composites (221 MPa (32 ksi) and 276 MPa (40 ksi), respectively).

The largest variations occurred in the shear strengths of the four systems. Short beam shear strengths varied from 28.3 MPa (4.1 ksi) for the Hercules 3501-6 resin to 51.7 MPa (7.5 ksi) for BP-907. In an attempt to obtain higher shear strengths, two experiments were performed in which Fiber D was precoated with a dilute resin system and cured before fabricating composites. The two precoating formulations were: (1) 100 parts "Epon" 828/13 parts MPDA and (2) 100 parts "Epon" 828/40 parts "Anchamide"-400. In both cases a 1% solution of the precoating system was prepared Fiber D was passed through the in methylene chloride solvent. solution and then through a hot air oven at 200°C. These precoating systems are sometimes observed to give improved composite shear strengths. Unidirectional composite bars 15.2 x 1.27 x 0.32 cm (6 x 0.5 x 0.125 in.) were wound using the precoated Fiber D and the "Epon" 828-RD2/"Tonox" resin formulation of Table 6 as the Fiber volume fraction was 0.60-0.65. The composite bars matrix. were tested for flex modulus, flex strength, and short beam shear strength. Results are shown in Table 9. Values for flex strength and modulus are the average of two measurements and shear values are the average of four determinations. Precoating Fiber D gave a small improvement in shear strength (σ 15%) as shown in the table. However, the increase was not large enough to justify the time consuming effort that would be required to precoat the entire supply of low denier Fiber D for use in the remainder of the pro-Therefore, no additional precoating experiments were gram. carried out.

Based on the experimental results shown in Tables 8 and 9 and the experience gained in fabricating these initial composites, the BP-907 epoxy system was recommended for use in the remainder of the program. BP-907 is a 177°C (350°F) modified epoxy system designed for high toughness and shear properties. In the preliminary evaluations shown in Table 8, BP-907 gave the highest flexure and shear strengths with Fiber D. The recommendation of this resin system was approved by the NASA Project Manager.

b. Final Evaluation

For final evaluation of the performance of Fiber D as a reinforcement for composites, two 22.9 x 22.9 x 0.3 cm (9 x 9 x 0.12 in.) Fiber D/BP-907 epoxy laminates were fabricated. 22.9 cm (9 in.) wide prepred strips were prepared by winding Fiber D on a 102 cm (40 in.) diameter McClean-Anderson drum winder and then impregnating the fiber with a 50% solution of BP-907 in methylene chloride. After allowing the solvent to evaporate, the prepred strip was removed from the drum and cut into 22.9 cm (9 in.) lengths. These were then stacked in a die and vacuum bag/compression molded. The prepred contained a slight excess of resin which was allowed to bleed off during the cure cycle in an attempt to obtain void contents of less than 1%. The resulting laminates are shown in Fig. 11.

Porosity of these two laminates was significantly lower than the porosity of the initial laminates discussed in Section IIIa. Void content, density and fiber volume fraction are shown below:

Laminate No.	Void	Fiber Volume	Density
	<u>Content</u>	Fraction, V _f	(gm/cm ³)
8266-3	0.8%	0.62	1.360
8266-4	0.4%	0.61	1.361

Before cutting test specimens from the laminates, ultrasonic C-scans were made in both the pulse echo (PE) and loss of back reflection (LOB) modes at NDT International Corp., West Chester, Pa. The scans are shown in Figs. 12 and 13. The PE scans (Figs. 12a and 13a) show no indications except near the extreme edges of the laminates. The LOB scans (Figs. 12b and 13b) show some indications along the right hand edges of both laminates, however. In order to determine if such indications affect mechanical properties, test specimens were taken from the right hand edge of one laminate and compared to specimens from clear areas. Results are discussed below. (For the LOB scans, a small strip of masking tape was placed on each laminate and the sensitivity of the ultrasonic equipment was adjusted in such a way that the small indication caused by the tape could be resolved. These tape indications can also be seen in Figs. 12b and 13b).

Fiber D/BP-907 epoxy laminates were evaluated at room temperature (21°C) and 121°C in both the as-fabricated condition and after humidity aging. The humidity aging conditions consisted of exposing test specimens to 95% relative humidty at 82°C for 14 Flexure modulus and strength, short beam shear strength and days. compressive strength were determined. Flexure moduli were obtained from 22.9 cm (9 in.) long x 1.27 cm (0.5 in.) wide x 0.30 cm (0.12 in.) thick unidirectional [0°] specimens using a span/thickness ratio of 60/1. Samples were subjected to maximum strains of 0.3% or less during modulus determinations to insure that only elastic strains were produced. Flexure strengths were then determined on the same specimens at a span/thickness ratio of 16/1-18/1. Short beam shear strengths were determined at a span/thickness ratio of 4/1. Compressive strengths were determined using a standard Celanese Test Fixture with 11.4 x 0.64 x 0.30 cm (4.5 x 0.25 x 0.12 in.) specimens as described in Section Typical flexure and compression specimens are shown in Fig. IIIa. 14.

1. As-Fabricated Fiber D/BP-907 Epoxy Specimens

Results for the as-fabricated specimens (before humidity aging) are shown in Table 10. All results in this table are for specimens taken from areas of the laminates that were nearly free of ultrasonic indications.

At room temperature (21°C), an average flexural modulus of 94.1 GPa (13.7 x 10° psi) was obtained. This value is lower than the rule-of-mixtures (ROM) prediction of 104.8 GPa (15.3 x 10° psi). It is sometimes observed that flexure moduli can be lower than tensile moduli even at large span/depth ratios (Ref. 2) because the shear defelection is not negligible for resin matrix composites. The lower flexure moduli may also reflect a nonlinear compression behavior for Fiber D. Therefore, two measurements of tensile modulus were made, and values of 100.7 and 102 GPa (14.6 and 14.8 x 10° psi) were obtained. These modulus values are in better agreement with ROM prediction. Flexure strength of 700 MPa (101.5 ksi), short beam shear strength of 53.6 MPa (7.8 ksi), and compressive strength of 232 MPa (33.6 ksi) were obtained at room temperature.

Two test specimens were taken from the right hand side of laminate 8266-3 where ultrasonic tests showed indications. Mechanical properties of these two specimens are nearly identical to those of Table 10:

Specimen No.	Flex Modulus GPa (10° psi)	Flex Strength MPa (ksi)	Shear Strength MPa (ksi)
8266-3-A	92.4 (13.4)	695 (100.8)	53.1(7.7)
Averages from	91.0 (13.2)		$= 2 \cdot 0 (7 \cdot 2)$
Table 10	94.1 (13./)	/00 (101.5)	53.0 (7.0)

These results show that the indications seen in the ultrasonic C-scans are very low level (i.e. the sensitivity of the electronic instrumentation was very high) and that these indications do not arise from serious defects that adversely affect the mechanical properties measured here. Optical microscopy of specimens containing indications was unable to detect any defects.

At 121°C, a flexure modulus of 70 GPa (10.2 x 10° psi) was obtained. This value is 26% below the room temperature value. The flexure strength of 530 MPa (76.8 ksi) is also 24% below the room temperature value. The short beam shear strength was reduced 11.5% to 47.7 MPa (6.9 ksi). A compression strength of 132 MPa (19.1 ksi) was obtained at 121°C. This value is 43% below the value of 232 MPa (33.6 ksi) obtained at room temperature. These decreases in mechanical properties suggest that the glass transition temperature of the resin is below the 121°C test temperature. For these elevated temperature flex and shear tests, specimens were heated in a laboratory oven to 121°C and then transferred to a high temperature flex test fixture which was also maintained at 121°C. Temperature measurements on a representative Fiber D/epoxy specimen showed that a holding time of 5 minutes in the fixture before test was sufficient to insure that samples were at the desired test temperature. For the elevated temperature compression tests, specimens were mounted in the Celanese text fixture at room temperature and the fixture was then placed in the temperature chamber on the Instron test machine and allowed to reach 121°C.

2. Humidity Aged Fiber D/BP-907 Epoxy Specimens

In order to determine the effects of moisture on the Fiber D/BP-907 system, individual specimens were exposed for 14 days to 95% relative humidity at 82°C (180°F). Moisture gains of

approximately 2.4% by weight were observed as a result of the humidity exposure. No measureable dimensional changes were observed for the test specimens.

Mechanical properties of the humidity-aged specimens are shown in Table 11. Samples were tested at room temperature and 121°C. Results show that the humidity aging exposure had little effect on the room temperature properties. Flexure modulus of 91.9 GPa (13.3 x 10° psi) and short beam shear strength of 53.6 MPa (7.8 ksi) are essentially identical to the values for the asfabricated material. Flexure strength of 654 MPa (95.3 ksi) is 6% lower and compressive strength of 224 MPa (32.5 ksi) is 3% below the results for the as-fabricated material.

At a test temperature of 121°C, however, a significant degradation in properties was observed as shown in Table 11. Flexure modulus, flexure strength and short beam shear strength were reduced 56%, 84%, and 53%, respectively, below their room temperature values, and compressive strength was reduced 88%. These decreases in mechanical properties are 2-4X greater than the decreases observed for the as-fabricated material between 21°C and 121°C.

For elevated temperature flexure and short beam shear tests on the humidity aged material, the specimens were not preheated in an oven as was done for the as-fabricated material. In order to minimize any losses in absorbed moisture prior to test, the specimens were placed directly into the elevated temperature flex fixture which was held at 127°C. Temperature measurements on a Fiber D/epoxy sample with a thermocouple imbedded at the centerline showed that the sample reached 121°C in 10 minutes. Therefore all samples were held for 10 minutes prior to test.

Load vs deflection curves for flexure and short beam shear tests changed significantly between room temperature and 121°C. Typical flex curves are shown in Fig. 15 for a span/thickness ratio of 16/1. The room temperature curve has a linear region followed by a region of curvature as shown. Samples failed in compression directly under the loading point as expected. At 121°C, the curve shows that strength was greatly reduced. Samples failed by buckling on the top (compression) surface but did not always fail under the loading point. Similar behavior was also observed for the short beam shear tests. At room temperature the load-deflection curves were linear up to the point of failure initiation, while at 121°C the curves were very non-linear as shown in Fig. 16. Load increased gradually until the deflection became large enough that the specimen became jammed in the three-point bend fixture, and then the load increased rapidly. The portion of the curve beyond 0.1 in. deflection is therefore not considered meaningful. With such a non-linear behavior it is not possible to obtain an accurate shear strength value; a value of 7 MPa (1 ksi) or less was estimated.

The large decreases in mechanical properties for the humidity-aged material are believed to be the result of moisture plasticizing the epoxy matrix and lowering its glass transition temperature significantly. This would result in a large decrease in shear strength of the matrix and would reduce the flex, compressive, and shear properties of the composite dramatically.

IV. FIBER D/FIBER FP/BP-907 HYBRID COMPOSITES

In addition to the Fiber D/BP-907 composites discussed in Section III, hybrid composites of Fiber D with Fiber FP were evaluated. Fiber FP is Du Pont's designation for an experimental polycrystalline aluminum oxide ceramic fiber. Fiber FP is prepared in the form of continuous yarn containing 210 filaments and is >99% α -alumina. Typical properties of Fiber FP are shown in Table 12. Fiber FP has excellent compressive strength in composites; values over 2070 MPa (300 ksi) are typically obtained in resin matrices and values approaching 3450 MPa (500 ksi) have been observed in metal matrix composites for fiber Volume fractions of V_f = 0.6. Therefore, combining Fiber FP with Fiber D is expected to increase the compressive and flexure strengths of Fiber D composites.

Two unidirectional Fiber D/Fiber FP/BP-907 hybrids were prepared: 40% Fiber D/20% Fiber FP and 20% Fiber D/40% Fiber FP by volume. Hybrid prepreg was first prepared by winding the two fibers on a drum winder in the desired ratio and impregnating with a 50% resin solution of BP-907 epoxy in methylene chloride. After the solvent was allowed to evaporate, the prepreg strip was cut into 22.9 x 22.9 cm (9 x 9 in.) squares and stacked in a mold. Laminates were prepared by standard vacuum bag/compression molding techniques as described in Section III. Micrographs the of hybrid composites showing fiber distribution are given in Fig. 17.

Void content, fiber volume fraction and density of the two hybrid laminates were determined and are shown below:

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Laminate No.	Void	Fiber Volume Fraction	Density
	Content	(D/FP)	(gm/cm ³)
8266-5	1.3%	0.41/0.20	1.84
8266-6	0.9%	0.21/0.41	2.35

Ultrasonic inspection of the laminates was carried out before test specimens were obtained. C-scans of the two hybrids are shown in Figs. 18 and 19. As discussed in Section II for the Fiber D/BP-907 laminates, ultrasonic indications could be observed in the hybrid composites if sufficiently high gains were used. However, test specimens taken from areas with and without indications showed no significant difference in mechanical properties indicating that the ultrasonic indications did not represent serious defects. For each hybrid composition one series of specimens was evaluated in the as-fabricated condition at room temperature (21°C) and 121°C. A second series was subjected to the humidity aging treatment at 95% relative humidity and 82°C for 14 days and was then tested at 21°C and 121°C. Test specimen sizes and test procedures were identical to those described for the Fiber D/BP-907 composites in Section III.

Results for the hybrid composites are summarized in Tables 13-16. Flexural modulus and strength data are plotted in Figures 20 and 21, respectively. Typical load-vs-deflection curves are shown in Figs. 22-25. The short beam shear strengths and compressive strengths of the hybrids are plottted as a function of composition in Figures 26 and 27, respectively.

a. Flexural Modulus

Fig. 20 shows that the composite flexural modulus of the as-fabricateed material at 21°C increases linearly with increasing Fiber FP content. The value increases from approximately 95.5 GPa (14 x 10° psi) for 60% Fiber D/0% Fiber FP to 152 GPa (22 x 10° psi) for 20% Fiber D/40% Fiber FP. This 57% increase in stiffness shows one of the advantages that can be obtained with hybridizing. For the as-fabricated material at 121°C, the composite with 60% Fiber D showed a decrease in modulus from 96.5 to 69.0 GPa (14 to 10 x 10° psi) while the 40/20 and 20/40 hybrids showed essentially no change from the room temperature values. The combined effects of humidity aging plus testing at 121°C showed a significant decrease in modulus for all three compositions, however, and indicates that this treatment seriously degrades the fiber/matrix interfaces and/or the matrix shear strength. This was confirmed by measurements of interlaminar shear strength discussed below.

b. Flexure Strength

Fig. 21 shows the flexure strength as a function of laminate composition. For the as-fabricated composites at 21°C the flexure strength is approximately 690 MPa (100 ksi) for 60% Fiber D. Strength increases to 1117 MPa (162 ksi) for the hybrid with 20% Fiber FP and decreases to 889 MPa (129 ksi) for the hybrid with 40% Fiber FP. For comparison, the flexure strengths of Kevlar[®] 49/Fiber FP hybrids are shown by the solid circles. The behavior of the two systems is quite similar and indicates that there is an optimum hybrid combination for obtaining maximum flexure strength. This maximum appears to occur at $\sqrt{20-30\%}$ volume % FP/40-30 volume % Fiber D (or Kevlar® 49). Visual inspection of the test specimens indicates that the 60% Fiber D composites fail in compression on the upper surface under the loading point while failure of the Fiber D/Fiber FP hybrids is controlled by tensile failure at the lower surface of the three point bend specimens.

For as-fabricated composites tested at 121°C the flexure strength is decreased approximately 15-25% in all cases, but the behavior otherwise parallels the room temperature data. Samples which were humidity aged at 95%RH/82°C for 14 days and then tested at room temperature showed only a 7% decrease in flexure strength for 60% Fiber D but a significantly larger decrease for the hybrid laminates. The combined effects of humidity aging plus testing at 121°C again gave the most dramatic decreases in properties as shown in Fig. 21. The decreases again indicate a significant decrease in fiber/matrix interface and/or matrix shear strength.

Typical load-vs-deflection curves for the hybrids are shown in Figs. 22-25. Fig. 22 shows the behavior of 40% Fiber D/20% Fiber FP laminates in the as-fabricated condition. At 21°C the curve shows an initial sharp rise to maximum load followed by a drop of \$50%. This drop is followed by a partial recovery before final failure of the flexure specimen. The presence of the Fiber FP prevents compressive failure at the upper surface of the specimen and allows failure to occur at the lower (tension) sur-The Fiber FP fails first because of its lower strain to face. The load is then carried by the Fiber D until it fails. failure. At 121°C, failure occurs at a lower load than at 21°C. The curve shows a sharp rise followed by a rapid decrease. The humidity aged laminates at room temperature (Fig. 23) show a similar behavior to the as-fabricated material, but have a lower maximum stress. The samples also show a partial recovery before final failure. At 121°C the humidity aged samples show a non-linear initial rise followed by several broad steps before failure.

Fig. 24 shows the load-vs-deflection curves for specimens with 20% Fiber D/40% Fiber FP in the as-fabricated condition. The initial sharp rise to maximum load is followed by rapid failure without any indication of partial recovery. Failure again occurs on the tensile side of the flexure specimens. However, with only 20% Fiber D, failure of the FP fiber results in complete failure of the specimen; this explains the absence of the partial recovery that was observed in the 40% Fiber D/20% Fiber FP laminates.

Load-vs-deflection curves for the humidity aged 20% Fiber D/40% Fiber FP laminates are shown in Fig. 25. Maximum load is much lower and the curves are much broader than the as-fabricated specimens. At l21°C the samples showed multiple delaminations in addition to the final tensile failure at the bottom surface. Delaminations are consistent with the decreased shear strength of the humidity aged material as discussed below.

c. Shear Strength

The short beam shear strength data are summarized in Fig. 26. For the as-fabricated material at room temperature the shear strength increases from 53.8 MPa (7.8 ksi) for 60% Fiber D to 63.4 MPa (9.2 ksi) for 20% FP/40% Fiber D and then decreases to 56.5 MPa (8.2 ksi) for 40% FP/20% Fiber D. Data for Kevlar[®] 49/Fiber FP/BP-907 hybrids are shown by solid circles and fall on the same curve. The data indicates an optimum fiber combination for maximum short beam shear strength; this maximum occurs at 20-30% Fiber FP as was observed for maximum flexure strength (see Fig. 21). The elevated temperature and humidity aged specimens show a trend in shear strength which is similar to the flexure strength data. The shear strength of the as-fabricated materials at 121°C is below the room temperature values; humidity aged specimens at 21°C have still lower strength; and the greatest decrease occurs for humidity aging plus 121°C testing temperature.

d. <u>Compressive Strength</u>

Compressive strength as a function of laminate composition is summarized in Fig. 27. Data for the as-fabricated material at room temperature show the large improvements in compressive strength that can be achieved by the hybrid composite approach. Specimens with 60 volume % Fiber D have a compressive strength of only 232 MPa (33.6 ksi). For 40% Fiber D/20% Fiber FP compressive strength increases to 664 MPa (96.3 ksi) - a 3X improvement. Increasing the Fiber FP content to 40% results in a compressive strength of 945 MPa (137 ksi) - a 4X improvement over the all-Fiber D material. The humidity aging treatment caused a significant decrease in the room temperature compressive strength of the hybrids. The percentage decreases in room temperature compressive strengths are very similar to the percentage decreases in short beam shear strengths.

v.

FIBER D/GRAPHITE/BP-907 HYBRID COMPOSITES

In addition the the Fiber D/Fiber FP hybrids discussed in the previous section, hybrids of Fiber D with Thornel[®] 300 (T-300) graphite fiber were also made and evaluated. The modulus, tensile strength and elongation of Fiber D are closer to T-300 than to Fiber FP as shown in Table 17, and therefore the Fiber D/T-300 system was expected to show different mechanical behavior from the Fiber D/Fiber FP hybrids.

Two Fiber D/graphite hybrid compositions were evaluated: 40% Fiber D/20% T-300 and 20% Fiber D/40% T-300 (nominal) by volume. An all-graphite control containing 60% T-300 in BP-907 epoxy was also evaluated. The hybrid composites were prepared by winding Fiber D and T-300 together on a drum winder in the desired ratios and impregnating with resin, as discussed in the previous section, to obtain prepreg tape. This tape was then cut, stacked, and vacuum bag/compression molded to produce laminates 22.9 x 22.9 x 0.31 cm (9 x 9 x 0.12 in.). The all-graphite laminate was prepared in the same manner as the Fiber D laminates discussed in Section III. Micrographs of the two Fiber D/T-300/BP-907 hybrids are shown in Fig. 28. Although the Fiber D/T-300 and Fiber D/Fiber FP hybrids were prepared by the same technique, the Fiber D/T-300 laminates have a layered structure while the Fiber D/Fiber FP laminates show considerable interpenetration of the yarn bundles. This difference may result from the fact that Fiber D contains 160 filaments/yarn and Fiber FP contains 210 filaments/yarn while the graphite yarn consists of approximately 1000 filaments.

Void content, fiber volume fraction, and density were determined for each laminate.

Laminate No.	Void Content	Fiber Volume Fraction (D/T-300)	Density (gm/cm ³)
8266-2	1.7%	0/0.61	1.51
8266-41	0.8%	0.18/0.37	1.44
8266-42	0.4%	0.41/0.20	1.42

Void contents were below 1% for the Fiber D/T-300 hybrids and less than 2% for the all-graphite control.

Ultrasonic C-scans were made on all three laminates as shown in Figs. 29-31. Indications were obtained only at very high levels of sensitivity, and these indications were distributed reasonably uniformly throughout the laminates. These results plus the low levels of porosity indicate that the laminates are of good quality, and this is supported by the mechanical properties discussed below.

Two sets of test specimens were evaluated. One series was evaluated in the as-fabricated condition at room temperature (21°C) and at 121°C. A second series was subjected to the humidity aging treatment at 95% relative humidity and 82°C for 14 days and was then tested at 21°C and 121°C. Results are given in Tables 18-23. The data are also shown in Figs. 32-35 along with data for 60% Fiber D/BP-907 epoxy and 60% T-300/BP-907 laminates.

a. Flexural Modulus

Fig. 32 shows flexural modulus as a function of graphite The modulus of the as-fabricated laminates at 21°C incontent. creases linearly with increasing graphite contentas shown by the The nominal 20% Fiber D/40% T-300 composition was solid line. found to be 18% Fiber D/37% T-300 (i.e. total fiber content 55% by volume) and accounts for the lower values for this laminate at room temperature. The flexural moduli fall below the rule of mixtures prediction shown by the dashed line. Measurements of tensile moduli are shown for comparison and are closer to the rule of mixtures behavior. Although a span to thickness ratio of 60/1 was used for the flexural modulus determinations, this was not sufficient to bring flexural and tensile moduli into agreement. At 121°F, moduli of the as-fabricated material are 7 to 20% below the room temperature values as shown. The room temperature moduli of the humidity-aged samples were essentially identical to the asfabricated material. When the humidity-aged specimens were tested at 121°C, however, moduli were 21% to 64% below the room temperature values. This large decrease in flexural modulus is believed to result from a decrease in fiber/matrix bond strength and/or matrix shear strength as discussed in previous sections.

b. <u>Flexure Strength</u>

Flexure strength as a function of laminate composition is shown in Fig. 33. The open circles show the data for the asfabricated laminates at room temperature. The upper line in the figure is a least squares fit to these data. Flexure strength increases reasonably linearly with increasing graphite content. The solid circles show the behavior of Kevlar® 49/T-300 hybrid composites (Ref. 3); a linear increase of flexure strength with graphite content is also observed as shown by the second line. The behavior of the Fiber D/T-300 and Kevlar® 49/T-300 systems should be compared to the Fiber D/Fiber FP and Kevlar[®] 49 Fiber FP hybrids shown in Fig. 21. These latter two systems showed a maximum in flexure strength at 20-30 volume % Fibere FP followed by a decrease in flexure strength higher FP contents. No such maximum is observed in the graphite hybrids. The difference in behavior of the two systems results from the fact that the strength, modulus, and elongation of T-300 graphite match the values of Fiber D more closely than the values of Fiber FP as shown in Table 17. In the Fiber D/Fiber FP system, Fiber FP picks up load much more rapidly than Fiber D because of its 2X higher modulus, and for FP contents above 20 -30% failure of the hybrid is controlled by failure of the lower elongation Fiber FP. In the Fiber D/T-300 system, however, the strengths and moduli of the two fibers are more nearly the same and a linear relation between composition and strength is observed.

The as-fabricated laminates at 121°C and the humidityaged laminates at room temperature show a similar trend for flexural strength vs. composition, but strength levels are 10 -20% lower than the as-fabricated/room temperature values. The combination of humidity aging plus testing at 121°C again gives the largest decrease in properties; the curve of strength vs. graphite content is very similar to modulus vs. graphite content as shown in Fig. 32. This large decrease again is consistent with a large decrease in fiber/matrix bond strength and/or matrix shear strength.

c. Compressive Strength

Compressive strengths of Fiber D/T-300/BP-907 hybrids laminates are shown in Fig. 34. Data for Kevlar® 49/T-300/BP-907 hybrids (Ref. 3) are shown for comparison. The upper curve is a least squares fit through the Kevlar® 49/T-300 data. Within experimental scatter, the Fiber D/T-300 data follow the same behavior. These data show the significant increase in compressive strength (>2X) that can be obtained by hybridizing when one is working with fibers such as Fiber D and Kevlar® that have low compressive properties. The compressive strengths of 552-827 MPa (80 - 120 ksi) observed for the all-graphite composites in this work and Ref. 3 are lower than expected and may indicate that the Celanese test fixture is causing premature failure of the specimens. Some sample damage at the grips has been observed in this study. The humidity-aged laminates tested at room temperature show a similar linear increase in compressive strength with increasing graphite content. A compressive strength increase of v2X is again observed for a hybrid containing v40% T-300. The lower line in Fig. 34 is a least squares fit to the data for the humidity-aged samples.

d. Shear Strength

Short beam shear strengths as a function of laminate composition are shown in Fig. 35. The open circles show the data for as-fabricated Fiber D/T-300/BP-907 laminates tested at room temperature. The solid circles show data for Kevlar® 49/T-300/BP-907 laminates (Ref. 3). Both systems show a similar trend. Shear strength increases slightly from 55.2 MPa (8 ksi) for the all-Fiber D and all-Kevlar® materials to ∿62-69 MPa (9-10 ksi) for hybrids containing \$40% T-300. Above 40% T-300 a more rapid increase in shear strength is observed, and values of 83-97 MPa (12-14 ksi) were obtained for 60 v/o T-300 lamainates. (The data point at 74.5 MPa for 40% Fiber D/20% T-300 appears to be higher than one would expect based on comparison with the remainder of The data suggest that BP-907 epoxy gives better adthe data). hesion to Thornel®-300 graphite than to either Fiber D or Kevlar® 49.

The shear behavior of the as-fabricated laminates at 121°C and the humidity-aged laminates at room temperature is very similar to the as-fabricated/room temperature data as seen in Fig. 35. Short beam shear strengths incressed slightly from 48-55 MPa (7-8 ksi) for 60% Fiber D laminates to 55-69 MPa (8-10 ksi) for 20% Fiber D/40% T-300 and then increased to ∞83 MPa (12 ksi) for 60% T-300. The only exception is the as-fabricated T-300/epoxy laminate at 121°C which had a shear strength of only 58 MPa (8.4 ksi).

The humidity-aged Fiber D/T-300/BP-907 laminates showed large decreases in shear strength when tested at 121°C. Short beam shear behavior was such that it was not possible to obtain an accurate shear strength value. Load-vs-deflection curves for short beam shear tests were essentially identical to the curve shown in Fig. 16 for the Fiber D/BP-907 specimens. Shear strengths of less than \circ 7 MPa (l ksi) were again estimated. For the humidity-aged graphite laminates (60% T-300/BP-907) the decrease in shear strength with increased temperature was not as severe. Values decreased from 85.5 MPa (l2.4 ksi) at room temperature to 50 MPa (7.3 ksi) at 121°C as shown in Fig. 35.

VI. IMPACT BEHAVIOR

In the previous sections, flexure, compression, and shear properties of Fiber D/epoxy and hybrid composites of Fiber D with Thornel® 300 graphite and Fiber FP were presented and discussed. It was shown that the hybrid composites can have significantly improved flexure and compressive strengths compared to the all-Fiber D composites. The impact or energy absorbing capabilities of these composite systems are also of interest because the ability to withstand impacts by foreign objects is a necessary characteristic for structural materials.

a. <u>Test Procedure</u>

The impact behavior of unidirectional composite specimens was investigated using the Instrumented Charpy Technique. The conventional Charpy impact tester consists of a heavy pendulum which strikes the sample at its midpoint and fractures it in 3point bending. Total energy absorbed during impact is read out on a dial. With the instrumented Charpy test, a strain gage is attached to the striker, or tup, and load-vs-time and energy-vstime histories are obtained during the impact event. These load and energy histories are recorded on an oscilloscope and provide additional information about the fracture behavior of the material.

Impact behavior of unnotched Fiber D/BP-907 and T-300/BP-907 composites and Fiber D/T-300/BP-907 and Fiber D/Fiber FP/BP-907 hybrids was investigated. A sample size of 57.2 x 17.2 x 3.0-3.8 mm (2.25 x 0.5 x 0.12-0.15 in.) was used. All samples contained a total fiber content of 60 volume per cent with fibers parallel to the long dimensions of the sample. The anvil of the Charpy impact tester supports the test specimen with a span of 40 mm (1.574 in.) as shown schematically in Fig. 36. Thus, with the chosen sample thickness of 3.0-3.8 mm (0.12-0.15 in.), the Charpy test is essentially a dynamic 3-point flexure test with span/depth ratio of $\circ 10/1-13/1$. Three impact tests were performed on each laminate composition. Both as-fabricated and humidity-aged specimens were evaluated.

b. Results and Discussion

1. Fiber D/Thornel® 300/BP-907 Composites

Typical oscilloscope records for the Fiber D/T-300 system are shown in Figs. 37 and 38. Load and energy traces are indicated in each photograph, and the vertical scale for each trace is shown. The time base is 1 millisecond/division in all cases. Table 24 shows data which were obtained from such oscilloscope records.

Fig. 37 shows the impact behavior of the Fiber D/T-300/BP-907 system in the as-fabricated condition. For the all-Fiber D composite (60% Fiber D) the load is seen to increase, with some non-linearity, to a maximum value of 145 kg (320 lbs) and then decreases sharply as failure initiates. The sample does not fail completely however, but shows partial recovery of load followed by a more gradual decay. Total energy absorbed was 10.8 joules (8.0 ft-lbs). For a hybrid containing 40% Fiber D/20% T-300 graphite, the load increases linearly to a much higher value

(268 kg (590 lb)). This is followed by a sharp drop and then a partial recovery as observed for the all-Fiber D composite, but total energy absorbed is still only about 10.8 joules (8 ft-1b). Increasing the graphite content to 40% results in a further increase in maximum load to 277 kg (610 lbs). However the total area under the load-vs-time curve is decreased, and total absorbed energy is only 8.9 joules (6.6 ft-lbs). The all-graphite laminate (60% T-300) shows behavior typical of a brittle system. The load increases linearly to a high value (322 kg (710 lbs)) and then drops catastrophically to zero as the sample fails. There is no evidence of partial recovery, and no additional energy is absorbed once maximum load is reached. This all-graphite specimen had a total absorbed energy of only 4.6 joules (3.4 ft-1bs) compared to 10.8 joules (8.0 ft-lbs) for the all-Fiber D specimen of the same dimensions. The instrumented Charpy test is thus seen to give considerably more information about impact behavior than can be obtained from a standard Charpy experiment which measures only the total energy absorbed.

Fig. 38 shows the corresponding series of experiments for the Fiber D/T-300 system after humidity aging at 82°C and 95% RH for 14 days. The behavior trend is nearly identical to the asfabricated series in Fig. 37 except that the maximum load and total absorbed energy are 5-30% lower in each case than the corresponding as-fabricated composites.

Fig. 39 shows typical as-fabricated and humidity-aged samples after impact. In all cases, the appearances of the asfabricated and humidity-aged samples are very similar. For 60% Fiber D, the specimens did not fracture into two distinct pieces but showed one or more shear delaminations parallel to the speci-The initial drop in load seen in Fig. 37 is believed to men axis. result from the onset of delamination. For 40% Fiber D/20% T-300, the impact damage appears more brittle with evidence of tensile failure on the bottom surface. Both the as-fabricated and humidity-aged specimens show multiple delamination. For 20% Fiber D/40% T-300, completely brittle failure is observed; samples fracture catastrophically into two pieces. The load-vs-time trace for the 60% T-300 composite (Fig. 37) shows this brittle behavior with catastrophic failure very clearly.

In Table 24 the total absorbed energy obtained from the oscilloscope trace is compared to the value obtained from the dial reading on the Charpy impact tester. For all samples, the energy was divided by the cross-sectional area to account for small differences in specimen dimensions. The oscilloscope and dial values are seen to be in reasonable agreement and show that the electronics were properly calibrated. Fig. 40 shows the Charpy impact energy as a function of graphite fiber content. Within experimental variations, impact energy decreases linearly with increasing graphite content for both the as-fabricated and humidity-aged conditions as shown by the upper and lower lease squares lines, respectively. This decrease is resonable in view of the fact that while Thornel® 300 has similar tensile strength to Fiber D, it has a higher modulus and lower strain-to-failure (i.e. is more brittle) than Fiber D. Data for the Kevlar® 49/T-300/BP-907 system are shown for comparison (Ref. 3). The behavior of the Kevlar® 49/T-300 system is seen to be very similar, within experimental error, to the Fiber D/T-300 system.

If one assumes that during the impact experiment the specimen behaves initially as a simple beam, then it is possible to use the maximum load from the oscilloscope trace and the flexural formula

 $\max^{\sigma} = \frac{3}{2} \frac{PL}{WT^2}$

to calculate an "apparent flexure strength" for the impacted material. (σ max = flexure strength, P = maximum load, L = span, W and T = specimen width and thickness.) Values of apparent flexure strength are given in Table 24 and are shown in Fig. 41 together with the flexure strengths measured in the quasi-static 3-point bend tests described earlier (Fig. 33). Agreement between the two flexure strengths is reasonably good and shows that the instrumented Charpy test can be used to obtain an estimate of flexure behavior of the Fiber D/T-300 system. Within experimental variation, apparent flexure strength increases linearly with increasing graphite content. Thus, when one compares Figs. 40 and 41 a trade-off is apparent: while the relatively low flexure strength of Fiber D/epoxy laminates can be increased significantly by adding graphite fiber, this strength increase is accompanied by a decrease in impact performance. Such trade-offs are common in composite materials, and designers must tailor-make laminates for specific applications. Fiber D/T-300 hybrids with 20 to 40% Fiber D have significantly better impact performance than an all-graphite laminate and have higher flexure strengths than all-Fiber D composites.

2. Fiber D/Fiber FP/BP-907 Composites

Typical oscilloscope records for impact tests of the Fiber D/Fiber FP/BP-907 system are shown in Figs. 42 and 43 for as-fabricated and humidity-aged laminates, respectively. Data obtained from such records are given in Table 25. Qualitatively, the behavior of this system is similar to the Fiber D/T-300/BP-907 system with respect to energy absorbed during impact. For the all-Fiber D laminate, the load-vs-time trace shows a non-linear increase up to the point of failure initiation, then a sharp decrease followed by partial recovery and gradual decay as discussed in the above section. With the addition of Fiber FP, impact behavior becomes more brittle. At 20 volume % Fiber FP the initial portion of the load-vs-time curve becomes linear to the point of fracture initiation, and while there is some partial recovery of load carrying capability, the total area under the curve is significantly reduced. Impact energy is 6.8 joules (5 ft-lbs) compared to 10.7 joules (7.9 ft-lbs) for the all-Fiber D laminate.

At 40% Fiber FP there is almost no partial recovery after fracture initiation, and total impact energy is only 3.8 joules (2.8 ft-lbs). The behavior of the humidity-aged laminates is nearly identical to the as-fabricated material.

The Fiber D/Fiber FP/BP-907 impact samples are shown in Fig. 39. The addition of Fiber FP makes the impact behavior increasingly more brittle as was observed for the Fiber D/T-300 hybrids. For 40% Fiber D/20% Fiber FP evidence of tensile failure is seen, as well as one or more shear delaminations. For 20% Fiber D/40% Fiber FP the specimen is almost completely fractured into two parts, and shear delaminations are also observed. The brittle behavior also is seen clearly in the load-vs-time trace (Fig. 42).

Fig. 44 shows Charpy impact energy as a function of Fiber FP content. Energy decreases linearly with increasing volume fraction of Fiber FP. The decrease is larger than observed for the Fiber D/T-300 system as expected. Fiber FP has a modulus of371 GPa (55 X 10⁶ psi), strength of 1550 MPa (225 ksi), and elongation of 0.35-0.4% compared to 228 GPa (33 X 10⁶ psi), 3290 MPa (477 ksi) and 1.45% for Thornel[®] 300 and therefore is more brittle than the graphite fiber.

Fig. 44 shows "apparent flexure strengths" calculated from the maximum loads on the oscilloscope records. Flexure strengths obtained from quasi-static 3-point blend tests (Fig. 21) are shown for comparison. While agreement between the two flexure strengths is not as good as was observed for the Fiber D/T-300system, both results show a maximum in flexure strength at approximately 20 v/o Fiber FP.

Examination of Figs. 44 and 45 shows that for the Fiber D/Fiber FP system, the best combination of flexure and impact properties is obtained for a hybrid with 20% Fiber FP/40% Fiber D. At this composition, impact energy and flexure strength show approximately a 20X and 2X improvement, respectively, over an allfiber FP laminate.

VII. CONCLUSIONS

Fiber D, an experimental organic fiber, has a tensile strength of 3.14 GPa (456 ksi), modulus of 172 GPa (24.9 x 10 psi) and a density of 1.46 gm/cm³. Its modulus is approximately 40% higher than Kevlar[®] 49 aramid.

The high tensile modulus of Fiber D translates reasonably well into composite modulus. Room temperature tensile moduli of the order of 101 GPa (14.7 x 10 6 psi) were obtained from unidirectional Fiber D/epoxy laminates containing 60% volume % fiber.

Flexure strengths of Fiber D/epoxy laminates were approximately 700 MPa (101.5 ksi) at room temperature. Based on fiber tensile strength this value is lower than expected the lower composite flexural strengths result from low compressive strength of Fiber D. Unidirectional laminates containing 60 volume % Fiber D had compressive strengths of 230 MPa (34 ksi). This value is similar to those obtained from Kevlar® 49 aramid/epoxy laminates.

Hybrid composites of Fiber D with 20 and 40 volume percent Thornel® 300 graphite and Fiber FP alumina fiber showed significant increases (20% to 400%) in flexure modulus, flexure strength and compressive strength over all-Fiber D laminates.

As-fabricated Fiber D and hybrid laminates at elevated temperature (l21°C), and humidity-aged laminates at room temperature, had flexure strengths and moduli, compressive strengths, and shear strengths which were l-20% below the values determined for as-fabricated materials at room temperature. (Humidity aging consisted of l4 days at 95% RH and 82°C.)

Combined effects of humidity-aging plus testing at elevated temperature resulted in decreases of greater than 50% in mechanical properties of the laminates. The decrease is believed to result from moisture plasticizing the BP-907 epoxy matrix thereby lowering its glass transition temperature. Any future work on Fiber D should be carried out with a more moisture resistant matrix system.

Charpy impact behavior of Fiber D/epoxy laminates is comparable to Kevlar[®] 49 aramid/epoxy. Charpy impact energies of 0.15 J/mm² (130 ft-lbs/in²) were observed. Hybridizing Fiber D with relatively brittle graphite or Fiber FP results in significant improvements in impact performance over all-graphite or all-FP laminates because of the fact that the impact energy of Fiber D/epoxy laminates is \$2X Thornel[®] 300/epoxy and \$25X Fiber FP/epoxy.

VIII. RECOMMENDATIONS

The modulus of Fiber D of 172 GPa represents a significant increase over commercially available organic fibers (approximately 40% greater than Kevlar® 49 for example). Its tensile strength of 3.14 GPa makes it attractive as a reinforcement and its density is about 20% lower than graphite. In addition, the fact that it is an electrical non-conductor offers an advantage over graphite fibers in applications where conductivity and corrosion are important. However, a significant improvement in compressive strength is needed before Fiber D could replace graphite fibers in primary structure applications. Fiber D may still be attractive for secondary structures or non-structural composite applications and additional evaluation should be carried out in these areas. Determination of composite tensile behavior, elastic constants, fatigue performance, off-axis properties, and damage tolerance, further studies of Fiber D in hybrids, and evaluations in high temperature matrices such as polyimides are recommended.

REFERENCES

- Kevlar[®] 49 Data Manual, E. I. du Pont de Nemours & Co., (Inc.), Textile Fibers Department, Kevlar[®] Special Products, Wilmington, Delaware 19898.
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- 3. P. G. Riewald and C. Zweben, "Kevlar® 49 Hybrid Composites for Commercial and Aerospace Applications". 30th Annual Conference of the SPI Reinforced Plastics/Composites Institute, (Feb. 1975).

TABLE 1

Bobbin No.	Tensile <u>G</u> Pa	Strength (ksi)*	Tensi] GPa	.e Modulus (106 psi)*
1	3.11+.13	(451+19)	185 <u>+</u> 2	(26.9 <u>+</u> 0.3)
2	3.45+.10	(500+15)	170+2	(24.6+0.3)
3	2.79+.13	(404+19)	171+2	(24.8 <u>+</u> 0.3)
4	2.75+.32	(399 <u>+</u> 47)	168 <u>+</u> 3	(24.5+0.4)
5	2.81 <u>+</u> .37	(408+54)	168+1	(24.4+0.2)
6	3.34 <u>+</u> .12	(485+17)	172+1	(25.0+0.2)
7	3.03+.14	(440+21)	174+1	(25.2 <u>+</u> 0.2)
8	3.12 <u>+</u> .32	(453+47)	172 <u>+</u> 1	(25.0 <u>+</u> 0.2)
,9	3.06+.13	(444+19)	169 <u>+</u> 3	(24.5+0.5)
10	2.99 <u>+</u> .09	(433+13)	173 <u>+</u> 1	(25.1 <u>+</u> 0.1)
11	2.96 <u>+</u> .14	(429 <u>+</u> 21)	171 <u>+</u> 1	(24.8 <u>+</u> 0.2)
12	3.27 <u>+</u> .07	(474 <u>+</u> 10)	163 <u>+</u> 1	(23.7 <u>+</u> 0.2)
13	3.37 <u>+</u> .19	(489+28)	172 <u>+</u> 2	(24.9 <u>+</u> 0.3)
14	2.99+.40	(434+58)	168 <u>+</u> 1	(24.3 <u>+</u> 0.2)
15	3.54 <u>+</u> .18	(513+26)	172 <u>+</u> 2	(25.0 <u>+</u> 0.3)
16	2.90 <u>+</u> .32	(421+47)	174+2	(25.2+0.3)
17	3.32 <u>+</u> .39	(481 <u>+</u> 56)	175 <u>+</u> 3	(25.4 <u>+</u> 0.4)
18	2.99 <u>+</u> .27	(433 <u>+</u> 39)	173 <u>+</u> 4	(25.1 <u>+</u> 0.6)
19	3.30 <u>+</u> .17	(479 <u>+</u> 24)	174 <u>+</u> 1	(25.2 <u>+</u> 0.2)
20	3.37 <u>+</u> .13	(489 <u>+</u> 19)	174+2	(25.3 <u>+</u> 0.3)
21	3.33+.25	(483 <u>+</u> 3Ĝ)	174 <u>+</u> 1	(25.2 <u>+</u> 0.2)
22	3.41 <u>+</u> .06	(494+9)	177 <u>+</u> 1	(25.6 <u>+</u> 0.1)
23	3.15+.18	(457 <u>+</u> 26)	173+1	(25.1 <u>+</u> 0.2)

TENSILE STRENGTH AND MODULUS OF FIBER D (Resin Impregnated Strands)

*All values are the average of 5 measurements

Average of All Measurements: Tensile Strength = 3.14 ± 0.23 GPa (456 ±33 ksi) Modulus = 172 ± 4 GPa (24. 9 ± 0.6 Msi)

TABLE 2

DENSITY	OF	FIB	ER	D	AS	DETERMINED
BY I	DENSI	ΤY	GRA	DI	ENT	COLUMN

Bobbin No.	Density $(gm/cm^3)*$
1	1.4621 ± .0004
2	1.4611 ± .0002
3	1.4640 ± .0002
10	1.4658 ± .0005
15	1.4615 ± .0003
19	1.4606 ± .0001

*Each value is the average of 4 determinations

Average of all 24 determinations: $1.4625 \pm .0019 \text{ gm/cm}^3$

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TABLE 3

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FIBER	D	S	INGI	E	FII	LAMEN	T I	MODULUS
	(]	LO	in,	Ga	.ge	Leng	th)

		Tensile	Modulus*
Bobbin N	0.	GPa	(106 psi)
1		172 <u>+</u> 12	(25.0 ± 1.7)
2		165 <u>+</u> 21	(24.0 ± 3.1)
3		168 <u>+</u> 10	(24.3 <u>+</u> 1.5)
4		164 <u>+</u> 14	(23.8 + 2.0)
5		167 <u>+</u> 7	(24.2 ± 1.0)
6		167 <u>+</u> 10	(24.2 ± 1.5)
7		171 <u>+</u> 7	(24.8 <u>+</u> 1.0)
8		161 <u>+</u> 7	(23.3 <u>+</u> 1.0)
9		212 <u>+</u> 64	(30.8 <u>+</u> 9.3)
10		154 + 22	(22.3 ± 3.2)
11		152 <u>+</u> 10	(22.1 ± 1.4)
12		155 <u>+</u> 7	(22.5 ± 1.0)
13		172 <u>+</u> 9	(25.0 ± 1.3)
14		169 <u>+</u> 8	(24.5 ± 1.2)
15		164 <u>+</u> 11	(23.8 ± 1.6)
16		177 <u>+</u> 10	(25.6 ± 1.5)
17		177 <u>+</u> 5	(25.6 ± 0.7)
18		162 <u>+</u> 15	(23.5 ± 2.2)
19		165 <u>+</u> 8	(24.0 ± 1.2)
20		172 <u>+</u> 7	(25.0 ± 1.0)
21		169 <u>+</u> 9	(24.5 <u>+</u> 1.3)
22		171 <u>+</u> 10	(24.8 ± 1.5)
23		176 <u>+</u> 13	(25.5 ± 1.9)
	Average of all tests	169 <u>+</u> 12	(24.5 <u>+</u> 1.7)

*All values are the average of 5 tests
SINGLE FILAMENT STRENGTH OF FIBER D VS GAGE

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* All values are the average of 5 measurements

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TABLE

FIBER D

FILAMENT DIAMETER

Bobbin	No.			Filament Diameter (microns)*
1				11.6 <u>+</u> 0.7
2				12.3 <u>+</u> 0.5
8				11.7 <u>+</u> 0.5
12				11.7 <u>+</u> 0.5
15				11.6 <u>+</u> 0.7
21				11.3 <u>+</u> 0.3
Average	of	all	determinations:	: 11.7 + 0.6

*Each value is the average of 5 measurements

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TABLE 6

RESIN SYSTEMS EVALUATED

	Resi	Formulation	Cure Cycle
1.	"Epon" 826 ⁽¹⁾ _{NMA} ⁽²⁾	100 parts 84 parts	2 hrs @ 90°C 4 hrs @ 165°C
	BDMA ⁽³⁾	1.5 parts	16 hrs @ 180°C
2.	"Epon" 826/RD2 ⁽⁴⁾	100 parts/25 parts	1.5 hrs @ 75°C
	"Tonox" 60-40 ⁽⁵⁾	24 parts/100 parts resin	1.0 hr @ 150°C
3.	Hercules 3501-6 ⁽⁶	5)	Raise temp to ll6°C in 60 min. Hold 1 hr Raise temp to 176°C in 40 min. Hold 2 hrs Cool
4.	BP-907 ⁽⁷⁾		Raise temp to 93°C in 30 min. Hold for 1 hr Raise temp to 177°C in 30 min Hold for 1 hr Cool

(1) Shell Chemical Co., epoxy resin

(2) NADIC Methyl Anhydride

(3) Benzyldimethylamine

- (4) Ciba Geigy Co., epoxy resin
- (5) Uniroyal Chemical Co.
- (6) Hercules Chemical Co., epoxy resin
- (7) American Cyanamid Co., epoxy resin

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TABLE 7

DENSITY, FIBER VOLUME FRACTION AND VOID CONTENT OF INITIAL FIBER D/EPOXY LAMINATES

Resin System	Density (gm/cm ³)	V _f	Void Content
"Epon" 826/NMA/BDMA	1.374	0.67	<1%
BP-907	1.344	0.65	2-3%
"Epon" 826-RD2/"Tonox"	1.345	0.63	2-3%
Hercules 3501-6	1.342	0.64	2-3%

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PRELIMINARY PROPERTIES OF FIBER D/EPOXY COMPOSITES

Resin System			Flex	ıre				Compr	ession	c		
	I GPa	Modulus (10 ⁶ psi)	0.02% Yield MPa	Offset Stress (ksi)	Ultj Str MPa	imate ress (ksi)	yj St MPa	ield tress (ksi)	Ult St MPa	timate tress (ksi)	Shor Shear ^{MPa}	t Beam Stress (ksi)
"Epon" 826/NMA/BDMA	115	(16.7)	310	(44.9)	741	(107.5)	223	(32.3)	241	(35.0)	48	(.0 ° (.)
BP-907	103	(14.9)	294	(42.7)	745	(108.0)	206	(29.9)	233	(33.8)	52	(7.5)
"Epon" 826-RD2/ "Tonox"	92	(13.4)	254	(36.8)	687	(9°66)	180	(26.1)	218	(31.6)	46	(6.6)
Hercules 3501-6	101	(14.6)	285	(41.4)	564	(81.8)	205	(29.8)	228	(33°0)	28	(4.1)

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FLEX AND SHEAR PROPERTIES OF COMPOSITES REINFORCED WITH PRECOATED FIBER D

ort ihear ength (ksi)	(6.6)	(7.1)	(7.8)	
Shc Beam S Stre MPa	46	49	54	
: Modulus (106 psi)	(14.0)	(13.0)	(12.8)	
Flex GPa	67	06	88	
'lex cength (ksi)	(0.66)	(95.5)	(94.1)	
E Str MPa	683	658	649	
Precoat	None	"Epon" 828 ⁽¹⁾ /MPDA ⁽²⁾	"Epon" 828/"Anchamide" 400 ⁽³⁾	
Matrix	"Epon" 826-RD2/"Tonox"	Ξ	Ξ	

- (1) Shell Chemical Co., epoxy resin
- (2) Metaphenylenediamine
- (3) Pacific Anchor Chemical Corp.

FIBER D/BP-907 EPOXY COMPOSITES; AS FABRICATED

TEST TEMPERATURE : 20°C (70°F)

No. Vf	Flex GPa	Modulus (106 psi)	Flex 9 MPa	Strength (ksi)	Short Shear S MPa	: Beam Strength (ksi)	Compr Str MPa	essive ength (ksi)
	92.2 97.2 91.7	(13.8) (13.4) (14.1) (13.3)	692 693 692	(100.4) (100.5) (104.8) (100.3)	51, 2 55, 8 55, 8 53, 8 53, 8 53, 8 53, 8 53, 8 54 54 54 54 54 54 54 54 54 54 54 54 54	(7.5) (7.8) (8.0) (7.8)	,	
0.61							224 228 233 232	(32.5) (33.0) (35.3) (33.6)
u	94.1 +2.5	(13, 7) +0.4	700	(101.5) +2.2	53.6 +1.4	(7.8) +0.2	232+8	(33.6) +1.2
		TEST	L TEMPERS	ATURE : 12	1°C (250°	E)	l	1
0.62	73.8	(10.7)	í 1	t	50.3	(2.3)		
0.61	63.4	(TO°9) (9°2)	541 519	(78.4)	46°2 46'9	(6°7) (6°8)		
0.61	67.6	(8°6)	529	(76.7)	47.6	(6.9)		
0.62							120 120	(23°3) (17 A)
0.61							122	(T 2°7)
10.0							123	(J.2))
	70.0	(10.2)	530	(76.8)	47.7	(6.9)	132	(1.61)
n	+5.5	+0°8	+11	+1.6	+1。8	+0.3	+19	+7°.8

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TABLE 11

FIBER D/BP-907 EPOXY COMPOSITES: HUMIDITY AGED 14 DAYS @ 95% RH, 82°C

TEST TEMPERATURE : 21°C (70°F)

Sample No.	Vf	Flex GPa	Modulus (10 ⁶ psi)	Flex MPa	Strength (ksi)	Shor Shear MPa	t Beam Strength (ksi)	Compr Str MPa	essive ength (ksi)	Moisture Gain (%)
8266-3-1 8266-3-5 8266-4-5	0.62 0.62 0.61	91.7 93.8 91.7	(13.3) (13.6) (13.3)	653 683 641	(96.5) (99.1) (92.9)	53.8 55.8 52.4	(7.8) (8.1) (7.6)			1.93 1.84 2.73
8266-4-8 8266-3-9 8266-3-16 8266-4-9 8266-4-16	0.61 0.62 0.61 0.61	90.3	(13.1)	638	(92.6)	52.4	(7.6)	229 234 234	(33.2) (32.9) (23.9) (29.8)	875 875 875 875 875 875 875 875 875 875
Average Std. Deviati	uo	91.9 +1.4	(13.3) +0.2	657 +21	(95.3) +3.1	53.6 +1.6	(7.8) +0.2	224 +13	(32,5) +1,8	2.41+0.43
			ריו	FEST TEM	PERATURE	: 121°C	(250°F)			
8266-3-3 8266-3-7 8266-4-2 8266-4-2	0.62 0.62 0.61	47.6 36.5	(6.9) (5.3)	134 133 78	(19.4) (19.3) (11.3)		(1) ((1) ((1))			1.87 1.91 2.57
8266-3-11 8266-3-11 8266-4-11 8266-4-11 8266-4-14	0.62 0.62 0.61 0.61	n 2 1		0	(0.11)	~	(7)	34.5 26.9 22.1	(5.0) (3.9) (3.5) (3.2)	2.62 2.18 2.94 89
Average Std. Deviati	uo	40.2 +6.4	(5.8) +0.9	106 +31	(15.4)	<7	(<1)	26,9 +5.4	(3,9) <u>+</u> 0.8	2.38 +0.43

TYPICAL PROPERTIES OF FIBER FP

Tensile Strength	1550 MPa (225 ksi)
Tensile Modulus	380 GPa (55 x 10 ⁶ psi)
Density	3.9 gm/cm ³ (0.14 lb/in ³)
Filament Diameter	20 µm (0.0008 in.)
Cross Section	Round
Filaments per yarn	210

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TABLE 13

FIBER D/FIBER FP/BP-907 HYBRID COMPOSITES: AS FABRICATED

TEST TEMPERATURE : 21°C (70°F)

Compressive Strength MPa (ksi)	695 (100.8) 633 (91.8)	$\begin{array}{c} 664 \\ +31 \\ +4.5 \\ \end{array}$			
t Beam Strength (ksi)	(9.3) (8.9) (9.5)	(9.2) +0.3		(9.1) (8.8) (8.6)	(8.9)
Shor Shear MPa	64.1 61.4 65.5	63,4 +2.1	(250°F)	62.7 60.7 59.3	61.4 +2.1
Strength (ksi)	(152.8) (167.7) (166.2)	(162.3) +8.2	RE : 121°C	(138.8) (143.3) (145.1)	(142.4)
Flex MPa	1054 1156 1146	1119 +57	MPERATUI	957 988 1000	982 +22
t Modulus (106 psi)	(18.6) (18.9) (18.8)	(18.7)	TEST TE	(18.3) (18.5) (18.4)	(18.4) +0.1
Flex GPa	128 130 129	129 +1		126 128 127	127 + 0.7
v_{f} D/FP	0.41/0.20	tion		0.41/0.20	cion
Sample No.	8266-5-2 8266-5-6 8266-5-6 8266-5-14 8266-5-14 8266-5-18	Average Std. Devia		8266-5-4 8266-5-8 8266-5-12	Average Std. Deviat

FIBER D/FIBER FP/BP-907 HYBRID COMPOSITES: HUMIDITY AGED 14 DAYS @ 95% RH, 82°C

TEST TEMPERATURE : 21°C (70°F)

Sample No.	V_{f}	Flex GPa (Modulus 10 ⁶ psi)	Flex GPa	Strength (ksi)	Shear S MPa	strength (ksi)	Compr Str MPa	essive ength (ksi)	Moisture Gain (%)
8266-5-1 8266-5-1 8266-5-5 8266-5-9 8266-5-13 8266-5-13	0.40/0.21	125 128 128	(18.2) (18.7) (18.6)	712 752 760	(103.3) (109.0) (110.2)	31。0 29。6 42。7	(4.5) (4.3) (6.2)	447 377	(64.9) (54.7)	1,61 1,58 1,81 1,93
/T-C-9978		Second Second Sec						410	(59°5)	J.8
Average Std. Deviat	rion	128 +2	(18.5) +0.3	741 +26	(107,5) <u>+</u> 3,7	34,5 +6,9	(5.0) +1.0	412 +35	(59.7) +5.1	1.76 +0.14
			TEST	TEMPER	ATURE : 12	1°C (250	(<u></u>			
8266-5-3 8266-5-7 8266-5-7	0.41/0.20	92.4 98.6	(13.4) (14.3) (15.7)	332 412 655	(48.1) (59.7) (67.5)	26.2 18.6 21.4	(3.8) (2.7)			1°61 1°61
		n n n n n n n n			(/	₽•T7	(T.C)			1.4.1
Average Std. Deviat	rion	99.7 +8.0	(14.5) <u>+</u> 1.2	403 +67	(58.4) <u>+</u> 9.7	22 °1 4 ° 1	(3.2) 0.6			1。54 +0。12

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FIBER D/FIBER FP/BP-907 HYBRID COMPOSITES: AS-FABRICATED

TEST TEMPERATURE : 21°C (70°F)

						Ter of d		Ċ	-	
Sample No.	v_{f}	Flex GPa	<pre>K Modulus (106 psi)</pre>	Flex MPa	Strength (ksi)	short Shear S MPa	trength (ksi)	Compi Sti MPa	ressive rength (ksi)	
8266-6-2 8266-6-6 8266-6-10 8266-6-14 8266-6-14 8266-6-14 8266-6-18	0.21/0.41	153 150 150	(22.2) (21.8) (21.8)	867 907 890	(125.8) (131.5) (129.1)	61.4 64.8 42.7	(8.9) (9.4) (6.2)	945 848 -	(137.1) (123.0)	
Average Std. Deviat	ion	151 +2	(21.9) +0.3	888 +19	(128.8) +2.8	56.5 <u>+</u> 11.7	(8.2) +1.7	897 +69	(130.1) +10.0	
			TEST TEMP	ERATURE	: 121°C	(250°F)				
8266-6-4 8266-6-8 8266-6-12	0.21/0.41	148 146 147	(21.4) (21.2) (21.3)	790 801 674	(114.6) (116.2) (97.8)	39.3 35.9 34.5	(5.7) (5.2) (5.0)			
Average Std. Deviat	ion	147 +0.7	(21.3) +0.1	756	(109.6) +10.2	36.5 +2.1	(5.3) +0.3			

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TABLE 15

FIBER D/FIBER FP/BP-907 HYBRID COMPOSITES: HUMIDITY AGED 14 DAYS @ 95% RH, 82°C

TEST TEMPERATURE : 21°C (70°F)

Sample No.	V _E D/FP	Flex GPa	(106 psi)	Flex MPa	Strength (ksi)	Short Shear S MPa	Beam trength (ksi)	Compr Str MPa	essive ength (ksi)	Moisture Gain (%)
8266-6-1 8266-6-5 8266-6-5 8266-6-13 8266-6-13 8266-6-13	0.21/0.41	148 154 153	(21.4) (22.4) (22.2)	356 405 387	(51.7) (58.7) (56.2)	17.9 19.3 19.3	(2.6) (2.8) (2.8)	319 - 414	(46。3) - (60。0)	1.49 1.56 1.73 1.15 1.22
Average		152	(22.0)	383	(52.5)	18,6	(2.7)	367	(53,2)	1.43
Std. Devia	tion	€ +1	+0,2	+24	+3°5	+0°8	+0.1	+67	+9°7	+0.24
			TEST TI	EMPERAT	URE: 121°C	(250°F)				
8266-6-3 8266-6-7	0.21/0.41	111 103	(16.1) (15.0)	206 223	(29.9) (32.3)	12。4 7,6	(1.8)			1,39 7,39
8266-6-11		100	(14.5)	206	(29.9)	7.6	(1.1)			1.47
Average Std. Deviat	tion	105 +6	(15.2) +0.8	212 +10	(30°7) +1.4	0°0 +	(1.3) +0.4			1.50 10.13
		I	-) -	• • •]) 1 -				0 1 2

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FIBER PROPERTY COMPARISONS

	M GPa	odulus (10 ⁶ psi)	Tensile _MPa	Strength (Ksi)	Elongation (%)
Fiber D	172	(24.9)	3144	(456)	1.8
Thornel® 300* (T-300)	225	(32.7)	3289	(477)	1.5
Fiber FP	379	(55)	1551	(225)	0.4

*Trade Mark - Union Carbide Corp.

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FIBER D/T-300/BP-907 HYBRID COMPOSITES: AS FABRICATED

TEST TEMPERATURE : 21°C (70°F)

Sample No.	V _f D/T-300	Flex GPa	Modulus (106 psi)	Flex S MPa	trength (ksi)	Short Shear S MPa	Beam trength (ksi)	Compr Str MPa	essive ength (ksi)
8266-42-2 8266-42-2 8266-42-6 8266-42-10 8266-42-14 8266-42-16 8266-42-18	0.41/0.20	104 104 105	(15.1) (15.1) (15.2)	1101 1088 1089	(159.7) (157.8) (158.0)	79.3 73.8 71.7	(11.5) (10.7) (10.4)	557 558 7	(74.6) (80.9) (75.0)
			Statement of the statem					•	
Average		104	(12,1)	1093	(158.5)	74.9	(10.9)	530	(76.8)
Std. Deviation	u	+0.6	+0.1	+7	+1.0	+3°9	+0.6	+25	+3°5
		TEST	TEMPERATURE	: 1210	C (250°F)				
8266-42-4	0.41/0.20	ł	ſ	868	(125.9)	54.5	(6°2)		
8266-42-8		97.2	(14,1)	872	(126.5)	59,9	(8,7)		
8266-42-12		96.5	(14.0)	861	(124.8)	58.6	(8.5)		
Average		96.9	(14.1)	867	(125.7)	57.7	(8,4)		
Std. Deviatio	u	+0.5	+0.07	9+1	+0°9+	+2.8	+0.4		

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TABLE	

FIBER D/T-300/BP-907 HYBRID COMPOSITES: HUMIDITY AGED 14 DAYS @ 95% RH, 82°C

TEST TEMPERATURE : 21°C (70°F)

ve Moistur 1 Gain <u>i) (%)</u>	2.62 2.61 2.61 2.64 2.63 2.71 2.63			2.52 2.63 2,47	2.63 +0.11
pressiv trengtl (ksj	(63, (55,	(58.			
Com S MPa	4381 381 390	403+31			
rt Beam Strength (ksi)	(8.7) (8.3) (8.7)	(8.6) +0.2		(1>) (1>) (1>)	(<1)
Sho Shear <u>MPa</u>	60.0 57.2 60.0	59.1 +1.6	(250°F)	<pre><7 <7 <7 <7 </pre>	L>
Strength (ksi)	(129.5) (132.4) (125.5)	(129.1) +3.5	E : 121°C	(19.1) (20.6) (20.6)	(20.1) +0.9
Flex MPa	893 913 865	890 +2.4	MPERATUR	132 142 142	139
x Modulus (10 ⁶ psi)	(14.7) (15.1) (14.9)	(14.9) +0.2	TEST TE	(6.7) (6.6) (6.8)	(6.7) +0.1
Fle GPa	101 104 103	103		46.2 45.5 46.9	46.2
V _E <u>D/T-300</u>	0.41/0.20	ion		0.41/0.20	ion
Sample No.	8266-42-1 8266-42-5 8266-42-9 8266-42-13 8266-42-13 8266-42-15 8266-42-17	Average Std. Deviat		8266-42-3 8266-42-7 8266-42-11	Average Std. Deviati

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TABLE 20

FIBER D/T-300/BP-907 HYBRID COMPOSITES: AS FABRICATED

TEST TEMPERATURE : 21°C (70°F)

V _f e No. <u>D/T-300</u>	Fley GPa	x Modulus (10 ⁰ psi)	Flex 9 MPa	Strength (ksi)	Shor Shear MPa	t Beam Strength (ksi)	Compress Strenc MPa (k	rive rth si)
0.18/0.5	37 109 101 109	(15.8) (14.7) (15.8)	1118 1105 1160	(162.2) (160.2) (168.2)	66.2 68.9 68.3	(9.6) (10.0) (9.9)	5 4 5 5 6 0 6 0 3 7 6 0	9.0) 11.2) 7.4)
ation	106	(15.4) +0.6	1127 +29	(163.5 +4.2	67.8 +1.4	(9 ° 8) +0 ° 2	569 (8 +30 +	2.5)
	TES	ST TEMPERAT	JRE : 121	l°C (250°F)				
0.18/0.3	37 - 102 103	- (14.8) (15.0)	899 937 931	(130.4) (135.9) (135.0)	57.9 64.8 62.7	(8.4) (9.4) (1.6)		
ation	103 +1.0	(14.9) +0.1	922 +20	(133.8) +3.0	61.8 +3.5	(9.0) +0.5		

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Sample No.	V _f D/T-300	Fley GPa	x Modulus (106 psi)	Flex MPa	Strength (ksi)	Shor Shear MPa	rt Beam Strength (ksi)	Compr Str MPa	ression rength (ksi)	Moisture Gain (%)
8266-41-1 8266-41-5 8266-41-9 8266-41-13 8266-41-13 8266-41-15 8266-41-17	0.18/0.37	100 105 106	(14.5) (15.2) (15.3)	896 896 910	(129.9) (130.0) (132.0)	57.9 56.5 55.2	(8.4) (8.2) (8.0)	405 479 530	(58.7) (69.4) (76.8)	2.92 2.92 2.93 2.93 2.93 2.93 2.69
Åverage		1 N 4	(15 0)	106	19 08 17	צע	10 01		10 07/	
Std. Deviati	on	- () +	+0.4	+ 80 > +	+1.2		+0.2	+ / + + (3	0.6+	
			TEST TEMP	ERATURE	: 121°C (:	250°F)				
8266-41-3 8266-41-7	0.18/0.37	35.9 39.3	(5.2) (5.7)	158 177	(22.9) (25.7)	1> 1>	(<1) (<1)			2.77 2.90
8266-41-11		40.0	(5.8)	174	(25.2)		(<1)			2.86
Averge Std. Deviati	on	38.4 +2.2	(5.6) <u>+</u> 0.3	170 <u>+</u> 10	(24.6) <u>+</u> 1.5	<7	(1>)			2.83 +0.11

FIBER D/T-300/BP-907 HYBRID COMPOSITES: HUMIDITY AGED 14 DAYS @ 95% RH, 82°C

TEST TEMPERATURE : 21°C (70°F)

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THORNEL® 300/BP-907 EPOXY COMPOSITES: AS FABRICATED

TEST TEMPERATURE : 21°C (70°F)

Sample No.	٧f	Flex GPa	: Modulus (10 ⁶ psi)	Flex MPa	Strength (ksi)	Short Shear S MPa	: Beam Strength (ksi)	Compi Sti MPa	cessive cength (ksi)
8266-2-2 8266-2-6 8266-2-6	0.61	127	(18.4) (18.4)	1402 1423	(203.4) (206.4)	95.8 99.3	(13.9) (14.4)	5 ***	
6200-2-10 8266-2-14 8266-2-16 8266-2-18 8266-2-18	0.61 0.61 0.61	87T	(c.8T)	1487	(215.6)	95 ° 2	(13.8)	561 769 509	(81.4) (111.5) (73.8)
Average Std. Deviati	ion	127 +0.4	(18.4) +0.6	1437 +44	(208.5) +6.4	96.8 +2.2	(14.0) +0.3	613	(88.9) +19.9
			TEST TEMPEF	ATURE :	121°C (25	(J°F)			
8266-2-4 8266-2-8 8266-2-12	0.61 0.61 0.61	91.0 102.7 106.2	(13.2) (14.9) (15.4)	988 952 967	(143.3) (138.1) (140.3)	58.6 57.2 57.9	(8.5) (8.3) (8.4)		
Average Std. Deviati	ion	100.0 +8.0	(14.5) +1.2	969 +18	(140.5) +2.6	57.9	(8.4) +0.1		

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Sample No.	Vf	Flex GPa	Modulus (10 ⁶ psi)	Flex MPa	Strength (ksi)	Shor Shear MPa	t Beam Strength (ksi)	Compressive Strength MPa (ksi)	Moisture Gain (%)
8266-2-1 8266-2-5 8266-2-9	0.61 0.61 0.61	123 125 126	(17.8) (18.2) (18.3)	1257 1280 1343	(182.3) (185.7) (194.8)	- 85.5 84 в	- (12.4)		1.31 1.31
8266-2-13 8266-2-15 8266-2-17	0.61 0.61 0.61					0 • F	(C•71)	732 (106.1) 449 (65.1) 781 (113.2)	1. 30 1. 43 1. 52 1. 29
Average Std. Deviatíc	n	125 +2	(18.1) +0.3	1293 +45	(187.6) +6.5	85.2 +0.5	(12.4) +0.1	$\begin{array}{c} 654 & (94.8) \\ +179 & +26 \end{array}$	
			TEST TE	MPERATI	JRE : 121°(с (250°F			
8266-2-3 8266-2-7	0.61 0.61	95.8 97.2	(13.9) (14.1)	805 805	(116.8) (116.7)	54.5 49.0	(7.9) (7.1)		1.28 1.32
8266-2-11	0.61	100.7	(14.6)	845	(122.6)	48.3	(1.0)		1.28
Average Std. Deviatic	u	97.9 +2.5	(14.2) +0.4	818 +23	(118.7)	50.6 +3.4	(7.3)		1.34 +0.08

THORNEL® 300/BP-907 EPOXY COMPOSITES: HUMIDITY AGED 14 DAYS @ 95% RH, 82°C

TEST TEMPERATURE : 21°C (70°F)

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TABLE 24

	Impact Energy	/ Per Unit Area	Flexure	Strength
	ft-lb/ir	n ² (J/mm ²)	ksi	(MPa)
Composite	Dial	Oscilloscope	Charpy	Quasi-Static
As-Fabricated				
60% Fiber D	120.4(0.138)	130.5(0.149)	94.3(650)	101.9(703)
	<u>+</u> 7.5(<u>+</u> .009)	<u>+</u> 9.9(<u>+</u> .011)	+4.5(+31)	+2.5(+17)
40% Fiber D/	113.6(0.130)	126.2(0.144)	152.7(1053)	158.5(1093)
20% T-300	<u>+</u> 7.6(<u>+</u> .009)	+15.3(+.017)	<u>+</u> 25.1(<u>+</u> 173)	+1.0(+ 7)
20% Fiber D/	88.6(0.101)	95.9(0.110)	130.4(899)	163.5(1127)
40% T-300	+11.1(+.013)	+17.4(+.020)	<u>+</u> 36.2(<u>+</u> 250)	<u>+</u> 4.2(<u>+</u> 29)
60% T-300	57.7(0.066)	58.2(0.067)	231.7(1598)	208.5(1438)
	<u>+</u> 7.1(<u>+</u> .008)	+6.5(+.007)	<u>+</u> 33.0(<u>+</u> 228)	+6.4(+ 44)
Humidity-Aged				
50% Fiber D	108.2(0,124)	111.7(0.128)	79.4(547)	96.2(663)
	+4.2(+.005)	<u>+</u> 7.3(<u>+</u> .008)	+5.8(+40)	+3.1(+21)
40% Fiber D/	83.3(0.095)	86.2(0.099)	114.5(789)	$129.1(890) \\ +3.5(+24)$
20% T-300	<u>+</u> 4.9(<u>+</u> .006)	+3.3(+.004)	<u>+</u> 9.6(<u>+</u> 66)	
20% Fiber D/	88.7(0.101)	90.2(0.103)	119.5(824)	130.6(900)
40% T-300	+9.2(+.011)	+3.4(+.004)	<u>+</u> 6.3(<u>+</u> 43)	<u>+</u> 1.2(<u>+</u> 8)
50% T-300	47.7(0.055)	47.7(0.055)	187.4(1292)	187.6(1294)
	+0.4(<u>+</u> .0005)	+0.4(+.0005)	<u>+</u> 7.9(<u>+</u> 54)	<u>+</u> 6.5(<u>+</u> 45)

IMPACT BEHAVIOR OF UNIDIRECTIONAL FIBER D/THORNEL®-300/BP-907 COMPOSITES AS DETERMINED BY INSTRUMENTED CHARPY TEST*

*All values are the average of 3 determinations.

IMPACT BEHAVIOR OF UNIDIRECTIONAL FIBER D/FIBER FP/BP-907 COMPOSITES AS DETERMINED BY INSTRUMENTED CHARPY TEST*

	Impact Energy	Per Unit Area	Flexure	Strength
	ft-lb/in	2 _{(J/mm} 2)	ksi	(MPa)
Composite	Dial	Oscilloscope	Charpy	<u>Quasi-Static</u>
As-Fabricated				
60% Fiber D	120.4(0.138)	130.5(0.149)	94.3(650)	101.9(703)
	<u>+</u> 7.5(<u>+</u> .009)	<u>+</u> 9.9(<u>+</u> .011)	<u>+</u> 4.5(<u>+</u> 31)	<u>+</u> 2.5(<u>+</u> 17)
40% Fiber D/	76.0(0.087)	87.2(0.100)	128.1(883)	162.3(1119)
20% Fiber FP	<u>+</u> 7.1(<u>+</u> .008)	<u>+</u> 15.5(<u>+</u> .018)	+3.0(+21)	<u>+</u> 8.2(<u>+</u> 57)
20% Fiber D/	39.2(0.045)	37.8(0.043)	94.5(652)	128.8(888)
40% Fiber FP	+2.1(+.002)	+2.8(+.003)	+9.9(+68)	+2.8(+19)
Humidity Aged				
60% Fiber D	108.2(0.124)	111.7(0.128)	79.2(547)	96.2(663)
	<u>+</u> 4.2(<u>+</u> .005)	<u>+</u> 7.3(<u>+</u> .008)	<u>+</u> 5.8(<u>+</u> 40)	<u>+</u> 3.1(<u>+</u> 21)
40% Fiber D/	80.8(0.092)	91.9(0.105)	109.5(755)	107.5(741)
20% Fiber FP	+3.7(+.004)	+14.3(+.016)	<u>+</u> 23.8(<u>+</u> 164)	<u>+</u> 3.7(<u>+</u> 26)
20% Fiber D/	47.9(0.055)	47.3(0.054)	59.7(412)	55.5(383)
40% Fiber FP	<u>+</u> 3.6(<u>+</u> .004)	+2.3(+.003)	+2.9(+20)	<u>+</u> 3.5(<u>+</u> 24)

*All values are the average of three determinations



Fig. 1 TYPICAL STRESS-STRAIN CURVE FOR FIBER D RESIN IMPREGNATED STRANDS



Fig. 2 SPECIFIC TENSILE STRENGTH AND SPECIFIC TENSILE MODULUS OF REINFORCING FIBERS



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240X

475X

Fig. 4 CROSS SECTION OF RESIN IMPREGNATED STRAND



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Fig. 5 SEM PHOTOGRAPHS OF FIBER D



FIBER D - THERMOGRAVIMETRIC ANALYSIS IN N₂ ATMOSPHERE

Fig. 6

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Fig. 7 FIBER D - THERMOGRAVIMETRIC ANALYSIS IN AIR

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Fig. 8 FIBER D - DIFFERENTIAL THERMAL ANALYSIS



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FOR FIBER D/BP-907 EPOXY COMPOSITE IN 3-POINT BENDING



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Fig. 12 ULTRASONIC C-SCAN OF FIBER D/BP-907 LAMINATE NO. 8266-3

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- 64 -





Fig. 17 MICROSTRUCTURE OF FIBER D/FIBER FP/BP-907 COMPOSITES

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× 67 ×







- 69 -

Fig. 20 FIBER D/FIBER FP/BP-907 EPOXY HYBRID COMPOSITES, FLEXURAL MODULUS vs COM-POSITION



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AS FABRICATED

ICATED





vs COMPOSITION



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40% Fiber D/ 20% T-300 30X



20% Fiber D/ 40% T-300 30X

Fig. 28 MICROSTRUCTURE OF FIBER D/T-300/BP-907 COMPOSITES

ULTRASONIC C-SCAN OF 40% FIBER D/20% T-300/BP-907 the second of the second s -____-م تحکیم کے ایک میں میں کارون میں ایک میں -7 ್ಯ ಸ್ಟರ್ಗೆ ಕ್ರಮ ಆಗ್ರಾಂಗ್ ಸರ್ಕಾರಗಳು . . . 7 ·. T. - - <u>-</u> -... ÷÷; مىسىيىسى مىي .. - 2--- --atoreal (* 1997) 1. augusta (* 1997) 1. augusta (* 1997) - 1 ----and the second secon ente Deter ---------an an anns Anns Anns Anns • • • • i e ------. 29 Fig. ---. ÷ ÷ jer niş <u> 27</u>... ' ÷ 11 de 19

HYBRID COMPOSITE LAMINATE

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Fig. 31 ULTRASONIC C-SCAN OF 60% T-300/BP-907 LAMINATE

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SHORT BEAM SHEAR STRENGTH vs COMPOSITION



Fig. 36 SCHEMATIC REPRESENTATION OF CHARPY IMPACT TEST

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60% FIBER D

Load: 50 lb/div (22.7 kg/div) Load: 100 lb/div (45.5 kg/div) Energy: 2 ft-lb/div (2,7 J/div) Energy: 2 ft-lb/div (2,7 J/div)

40% FIBER D/20% T-300



20% FIBER D/40% T-300

Load: 100 lb/div (45,5 kg/div) Energy: 2 ft-lb/div (2.7 J/div) 60% T-300

Load: 200 lb/div (90,9 kg/div) Energy: 2 ft-lb/div (2,7 J/div)

Fig. 37 TYPICAL OSCILLOSCOPE RECORDS FOR INSTRUMENTED CHARPY IMPACT TESTS OF AS-FABRICATED FIBER D/T-300/BP-907 COMPOSITES



60% FIBER D

Energy: 2 ft-lb/div (2.7 J/div) Energy: 2 ft-lb/div (2.7 J/div)



40% FIBER D/20% T-300 Load: 50 lb/div (22.7 kg/div) Load: 100 lb/div (45.5 kg/div)



20% FIBER D/40% T-300

60%_T-300

Load:	100 lb/div (4	45.5 kg/div)	Load:	200 lb/div	(90.9 kg/div)
Energy:	2 ft-lb/div (2	2.7 J/div)	Energy:	2 ft-lb/div	(2.7 J/div)

Fig. 38 TYPICAL OSCILLOSCOPE RECORDS FOR INSTRUMENTED CHARPY IMPACT TESTS ON HUMIDITY-AGED FIBER D/T-300/BP-907 COMPOSITES

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20% Fiber D/ 40% Fiber P/ 20% Fiber D/ 20% Fiber D/ 20% Fiber D/ 40% T-300



HUMIDITY-AGED







Fig. 39 CHARPY IMPACT SPECIMENS AFTER TEST

60% T-300

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60% Fiber D

40% Fiber D/ 20% Fiber FP







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60% FIBER D

Load:	50 lb/div (22.7 kg/div)
Energy:	2 ft-lb/div (2.7 J/div)

40% FIBER D/20% FIBER FP

Load:	100 lb/div (4	45.5 kg/div)
Energy:	2 ft-lb/div	(2.7 J/div)



LOAD

20% FIBER D/40% FIBER FP Load: 100 lb/div (45.5 kg/div) Energy: 2 ft-lb/div (2.7 J/div)

Fig. 42 TYPICAL OSCILLOSCOPE RECORDS FOR INSTRUMENTED CHARPY IMPACT TESTS OF AS-FABRICATED FIBER D/FIBER FP/BP-907 COMPOSITES



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60% FIBER D

Load:	50 lb/div ((22.7 kg/div)
Energy:	2 ft-lb/div	′ (2.7 J/div)

40% FIBER D/20% FIBER FP

Load:	100 lb/div (45.5	kg/div)
Energy:	2 ft-lb/div (2.7	J/div)

20% FIBER D/40% FIBER FP

Load: 50 lb/div (22.7 kg/diy) Energy: 2 ft-lb/div (2.7 J/div)

Fig. 43 TYPICAL OSCILLOSCOPE RECORDS FOR INSTRUMENTED CHARPY IMPACT TESTS OF HUMIDITY-AGED FIBER D/FIBER FP/BP-907 COMPOSITES



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