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LAWRENCE LIVERMORE LABORATORY

University of California/Livermore, California

FABRICATION AND TESTING
OF GRAPHITE FIBER/POLYIMIDE COMPOSITES

L. E. Lorensen
C. M. Walkup
T. T. Chiao

March 21, 1975

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24

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CONTENTS

Summary and Conclusions	1
Introduction	1
Discussion	2
Selection of Materials	2
Fabrication Methods for Unidirectional Laminates	3
Testing for Solvent and Fiber Content	7
Test Results	8
Acknowledgments	17
References	18
Appendix A: Statistical Examination of Test Results and Test Result Tables	19

FABRICATION AND TESTING OF GRAPHITE FIBER/POLYIMIDE COMPOSITES

SUMMARY AND CONCLUSIONS

In response to a contractual agreement to examine graphite-fiber reinforced polyimide systems for relatively high-temperature aircraft applications, available materials were surveyed, and two polyimides (Hexcel HX580 (F-178)* and Kerimid 601) and two fibers (Thornel 300 and Hercules AS) were selected for examination. Processes were worked out to fabricate prepreg with good drape and tack from these materials. Fiber content was determined by means of a KOH/ethylene glycol hydrolysis method. A series of mechanical tests was run in conformance with the contractual requirements. The most important measurements were flexural strength and modulus changes with long-time heating at 232° C (450° F). Based on these and other results, the HX580/AS system is the most promising. Optimizing a post cure for this system might provide improved properties.

INTRODUCTION

The Navy requires high-performance materials with favorable strength-to-weight ratios for use in modern aircraft construction. To help find appropriate materials, Lawrence Livermore Laboratory, under contractual agreement with the Navy Air Systems Command, studied several graphite fiber/polyimide composites. The Navy specified the following requirements.

Fabrication considerations were:

- It must be possible to make a resin-impregnated, unidirectional, carbon-fiber tape (prepreg) which is sufficiently flexible to be formed to contoured shapes before cure (have good "drape"). Once part layup begins, the prepreg should be sufficiently self-adhesive to stay in place until cured (have good "tack").
- It should be possible to cure the part formed from prepreg at 177° C (350° F) or less by (1) enclosing it in a close fitting, flexible bag (a vacuum bag) which can be evacuated to aid in removal of volatiles, (2) pressurizing the part, with vacuum bag in place, to 0.69 MPa (100 psi) in an autoclave, and (3) heating it using a maximum rate of 2.8° C (5° F) per minute.
- It should be possible to posture the resulting composite unrestrained with no dimensional change.

The composite must:

- have excellent initial strength and low void content,
- have sufficient thermal stability to withstand 200 hr of operation at 232° C (450° F) with good strength retention, and
- exhibit good strength retention at elevated temperatures after long exposure to moisture.

* Reference to a company or product name does not imply approval or recommendation of the product by the University of the product by the University of California or the U.S. Energy Research & Development Administration to the exclusion of others that may be suitable.

As part of the contract, LLL will write a purchase specification covering the most promising candidate system. Actual production is the responsibility of General Dynamics, San Diego, California.

In this report, we describe our experimental work with the polyimide composites, test results, and conclusions.

DISCUSSION

Selection of Materials

Our first task was to select two fibers and two resins.

Selection of Fibers

We selected Thornel 300 (T300) and Hercules Type AS (AS) fibers because of their favorable cost and good tensile strength.* At first there was some concern that these low modulus fibers would lose strength due to oxidation at high temperatures. Hercules provided data from two AS fiber samples taken at the high and low ends of the modulus specification. These samples were aged in a positive flow of air for 312 hr at 260° C (500° F). The one with the higher modulus (33.7×10^6 psi) lost 1.14% weight and the one with the lower modulus (30.2×10^6 psi) lost 0.88% weight. About 0.5% of this weight could be loss of absorbed water. Union Carbide reports that similar low numbers result from heating T300 at 260° C (500° F) in air. Assuming the weight loss correlates with loss in strength, it would appear that at the temperatures of concern the effect of oxidation is small.

The Hercules fiber is fluffier than Thornel (which has a slight twist) and thus is somewhat more difficult to use. It has no added finish but has been chemically treated. T300 was ordered with Kerimid 601 finish.

Selection of Resin

The selection of polyimide resins for use in this program involved a survey of available materials which is described in Ref. 1. Based on our investigation, we chose Kerimid 601 (K601) and Hexcel HX580 (now called F178) resins. These resins were selected because they had favorable processing temperatures and pressures, low water pickup, and what appeared to be adequate temperature resistance.

A K601 solution was freshly prepared for each laminate by dissolving 50 wt% resin in N-methylpyrrolidone (NMP). HX580 (F178) is a solvent-free system for hot melt application.

* A delay in obtaining the T300 resulted in some preliminary work with Thornel 400 which was on hand.

Fabrication Methods for Unidirectional Laminates

We considered three methods of fabricating prepregs:

1. The impregnated strands are wound onto a flat aluminum mandrel with a filament winding machine so that a unidirectional laminate or prepreg forms on each side. The solvent is removed under vacuum and the laminate is pressed and cured while still on the mandrel.

2. Impregnated strands are wound in a single layer on a 14-in. drum and the solvent is removed by evaporation with the aid of heat or vacuum. The layer is then cut and removed, trimmed to size, and a stack of such layers is cured in a mold.

3. Winding is done on an open rack with flat sides, forming one layer only on each side. The solvent is removed in a forced air oven and the laminate is cut to size and stacked in a mold for curing.

Only methods 1 and 3 were actually tried, and as will be noted later, method 3 was selected for use. In both methods, the roving was wound with minimum tension using normal fiber spacing. When the solvent-containing K601 was used, roving was led sequentially over two wheels to insure fiber wetting. Resin was applied to the first wheel by means of a small applicator wheel which dipped into a resin pot. For the solventless HX580, two schemes were tried for impregnating the fiber with hot melt. In one, resin was applied stepwise to alternate sides of a flattened roving. In the second scheme, which worked best, resin was applied to both sides simultaneously. The roving was led downward between two 3/4-in. rollers spaced 0.010 in. apart and heated to 71-82° C (160-180° F). The space between the two rolls above the gap was kept filled with warm resin. After leaving these rolls, the wetted roving was led over two heated pins to assist resin impregnation before passing over the guide wheel.

The particular aircraft structural applications toward which this program was aimed demanded prepreg with considerable drape and tack. Usually these qualities are achieved by allowing a minimum amount of solvent to remain, which results in some diminution of final properties due to voids, retained solvent, or solvent reaction products. As criteria for drape and tack, we used test methods described by General Dynamics.²

Following the preparation of a suitable prepreg, unidirectional panels were prepared. To gain experience while awaiting delivery of T300 some preliminary work was done with Thornel 400 with a UC307 finish which was on hand. Three points emerged from this initial work. First, this epoxy finish is incompatible with the polyimides, which emphasizes the need to carefully match finish and resin when using carbon fibers. Second, the required 50% resin content (an excess which is used to assist in removing air and filling voids by outflow during processing) could not be achieved until the flat solid aluminum mandrel was replaced by the open rack. Third, in changing from the solid to the open rack type mandrel, solvent content calculated by mass balance decreased only from 7% to 6% with "boardy" characteristics still present. We then changed to the oven drying technique which gave lower, but still probably inaccurate, results due to the tenacity of NMP. This will be discussed later.

Preparation of the laminates from the four materials selected is described below.

Kerimid 601/Thornel 300 (with Kerimid 601 finish)

Prepreg with a resin content of 38 wt% and a solvent content of 12 wt% was made. This was achieved by oven drying for 1/2 hr at 79° C (175° F) and resulted in material with suitable drape and tack. Even lower solvent contents would probably have provided the desired drape and tack, but lack of time prevented investigation.

Swatches of prepreg were stacked in a 22.9 cm × 22.9 cm (9 in. × 9 in.) square mold (Fig. 1). Figure 2 shows the mold in position in the press. This mold apparatus was used for each resin-fiber composite. A vacuum bag layup was used in the press with the Kerimid composites since remaining solvent had to be removed. The layers were arranged from the top as follows:

- Vacuum bag, 0.0508-mm (2-mil) Capran
- Fiberglass breather, 2 plies of Hexcel 2A460 cloth
- Mold top, release coated with Garan 225
- Bleeder cloth, 6 plies of Hexcel 1-P28 glass cloth
- Bleeder cloth, 1 ply Du Pont perforated Armalon TG0003
- Graphite prepreg layup
- Release film, 0.0762-mm (3-mil) Kapton
- Mold bottom, matched die male/female
- Vacuum bag bottom, 0.0508-mm (2-mil) Capran.

A coil spring wrapped with a glass cloth was wrapped around the side of the mold to assist vapor transmission. The bag and vacuum line entry were sealed with zinc chromate sealing tape. Stops 2.159 mm (0.085 in.) thick prevented complete mold closure and excessive resin loss.

The cure cycle for the K601/T300 system was as follows: heat to 110° C (230° F) at contact pressure, then hold at 110° C (230° F) for 45 min under a vacuum of 5-20 mm Hg. Remove vacuum and press at 1.379 MPa (200 psi), then take to 180° C (356° F), and hold for 1 hr. Remove from the mold and postcure for 24 hr at 250° C (480° F).

Hexcel HX580/Thornel 300 (with Kerimid 601 finish)

Fiber impregnation with the hot melt system and open rack gave a prepreg with roughly 55 wt% resin content, the desired excess. Following cutting and stacking in the press (no vacuum bag), the layup was cured as follows: heat from 25 to 105° C (77-221° F) in 20 min, hold at 105° C (221° F) for 20 min, apply 1.379 MPa (200 psi), heat to 180° C (356° F) during 10 min. Hold at 180° C (356° F) for 1 hr. Postcure 3 hr at 250° C (480° F) between glass plates.

Hexcell HX580/Hercules AS

The fluffiness and lack of added finish of the Hercules fiber could make uniform fiber tension difficult. However, laminate appearance was good. The calculated prepreg fiber content was 52 wt%. The cure cycle was the same as that described above for the HX580/T300.

Fig. 1. Mold used to prepare unidirectional laminates.

Fig. 2. Laminating mold in position in press.

Kerimid 601/Hercules AS

The prepreg was oven dried for 1/2 hr at 79° C (175° F) and had suitable drape and tack. The cure cycle was the same as that described for the K601/T300. Some K601/AS prepreg was dried for 1 hr at 127° C (260° F), which gave boardy material with no drape and tack. Panels were made from this prepreg using the following cure cycle: heat from 25 to 150° C (77-302° F) in 15 min apply 1.379 MPa (200 psi) pressure, heat to 180° C (356° F) during 5 min, continue at 180° C (256° F) for 1 hr. Postcure 24 hr at 250° C (480° F) between glass plates. Preparation of these two sets of panels allowed comparison of the same materials with and without drape and tack.

Testing for Solvent and Fiber Content

Complete removal of the high-boiling NMP solvent is difficult. Thus, measuring weight loss from solvent evaporation is of dubious value in calculating solvent contents. We calculated the solvent content by a materials balance method, which used the fiber weight, the concentration of resin solution, and the newly prepared and final prepreg or part weight. We initially assumed that no NMP was lost by evaporation during prepregging, and an evaporation rate test with the pure NMP suggested that this might be valid. However, later experience showed that small evaporative losses occurred. This could account for our higher-than-expected solvent content. Therefore, reported solvent contents were determined by weight losses after 30 min at 300° C (572° F).

A direct determination of fiber content is desirable. Industry has not yet settled on an optimum method. Current methods include using hydrogen peroxide in nitric acid,³ or using sulfuric acid. A New method uses KOH hydrolysis. We initially chose the nitric acid digestion method because of previous experience and safety considerations. The effect of nitric acid on neat fibers was measured using the standard test conditions and little change was noted. The Hercules AS fiber actually gained slightly (less than 0.5 wt% average) and the T300 lost less than 0.5 wt%, possibly due to removal of the K601 finish.

In some nitric acid digestions, it was noted that small amounts of organic residues remained which resisted the action of the acid. It was found that strong organic bases such as diethylene triamine would attack such residues, but at this point we abandoned this method in favor of a newer method involving sample digestion in hot KOH/ethylene glycol. This has been proposed to ASTM by the Fiberite Corporation.⁴

The KOH hydrolysis method appeared promising and we used it for a number of determinations. It is much faster than the acidic treatment, but small amounts of white precipitate were present in fiber residues. These are probably a silicate or borate, etc., from the glass, insoluble in the glycol or washing solutions used.

To rapidly estimate fiber content (required for uninterrupted progress during prepreg and laminate preparation), both volume and gravimetric methods were used. Knowing the mold cross-sectional area, individual fiber cross-sections, and fibers per roving, the proper number of rovings for a desired fiber content could be placed in the mold. Following mold closure and cure, the desired thickness was verified. In the

gravimetric method, 100.0-in. lengths of roving were weighed, giving a weight per linear inch. The number of turns of rovings per part was recorded, the part trimmed so that accurate roving length was known, and the total roving weight per part could then be calculated, giving another estimate of fiber content. The fiber contents determined from both methods were usually within a few percent of those obtained chemically so were regarded as sufficiently reliable for guidance. Since the calculated values bracketed those obtained by KOH hydrolysis, the precipitate noted above while very undesirable probably had only a minor effect on results. This may have been the reason for the spread (reported in Table A-1). The hydrolysis method, while in need of a change to eliminate precipitate formation, appears quite attractive and was used for the bulk of the study.

Test Results

The series of tests performed to evaluate these polyimide composites is listed in Table A-2, along with general test information and modifications used. Figure 3 shows examples of the various test specimens. Complete test results are given in the tables in the appendix.

The test results were evaluated statistically by checking for 2 and 3 σ overlap to see if differences in the data were real. The materials were ranked 1 (best), 2, 3, etc. The rank numbers were then totalled for the 35 tests performed on the 15 fabricated panels. These totals, as well as subtotals, for six of the major types of mechanical tests are given in Table 1.

Table 1. Ranking of results for mechanical tests performed on laminates. Low number indicates preferable material.

Composite	Tensile tests	Compressive tests	Flexural tests	Flexural tests, exposure to moisture	Short beam shear strength	Fatigue tests	Total
K601/AS	7	2	35	15	3	9	71
HX580/AS	9	2	18	8	2	9	48
HX580/T300	10	2	26	12	2	5	57
K601/T300	7	2	21	10	2	9	51

The HX580/AS system with the lowest value of 48 is the best candidate, followed by the K601/T300 system with a value of 51. A more critical way of comparing these two systems would be to examine results from tests which measure qualities of greatest importance to the Navy, i.e., resistance to heat and moisture (assuming all other properties were acceptable). The flexural tests listed in Table 1 measure these qualities. Summing the subtotals for HX580/AS gives 26, and for K601/T300, 31, values which increase the relative difference between these two materials somewhat. A more complete discussion of this treatment is given in the appendix.

Fig. 3. Test specimens used. Left to right (excluding ruler): longitudinal tensile, transverse tensile, compression, flexural, and interlaminar (short beam) shear.

In the following sections, specific comments for individual tests are made and particularly significant results are mentioned. However, the reader is urged to refer to Table A-3 in the appendix where the combinations are ranked. This allows both inter- and intra-group comparisons of materials and performance in different tests at a glance.

Tensile strength and modulus at room temperature (Table A-4)

Longitudinal—Failure and failure events seemed to occur randomly with all samples. This made it impossible to get ultimate strain, for each failure jarred the strain gage out of position. However, the initial modulus could be obtained before fiber breakage began. The HX580/T300 combination gave poor ultimate properties (as it did later in some of the flexural testing). The K601/AS material, tested in two panels with different fiber contents, performed best.

Transverse—The two K601/AS panels with different resin contents gave different results here. The high resin samples exhibited surprisingly high strength in marked contrast to the low resin samples. It is not known whether differences in void content, degree of fiber wetting, or simple resin starvation were involved. Another possibility is that the extra resin allowed some of the fluffy AS fibers to deviate from the assumed unidirectional placement resulting in multidirectional reinforcement, although resin flow during molding was deliberately encouraged in the fiber directions to minimize this possibility. Some specimens broke outside the gage length next to the edge of the tab. The high resin K601/AS material gave highest values.

Compressive Strength and Modulus at Room Temperature (Table A-5)

Values obtained from compressive tests were comparable to the tensile moduli shown in Table A-4. Some bending of specimens was recorded during compression. Average curves were used to calculate modulus values.

A Celanese test fixture was used (Figs. 4 and 5). This test proved very time consuming. It was necessary to bond the small strain gages to each side of each sample because of limited space. Two were required to detect and measure bending. In place calibration had to be done on each gage. The steel grip tabs also failed occasionally, showing that great care must be taken with bead blasting, degreasing, and bonding.

Flexural Strength and Modulus as a Function of Temperature and Thermal Aging (Table A-6)

Initial (Unaged) Ambient and 232° C (450° F) Flexural Properties (Table A-6a)—At room temperature, the HX580/T300 laminate failed catastrophically at low strength values. The K601/AS fabricated from prepreg without drape and tack was superior here to that from prepreg with drape and tack. This was true in a number of other tests, illustrating the expected sacrifice in properties as a result of residual solvent.

To help force outer fiber failure, four point loading or larger diameter heads should be tried to minimize undue concentration of load. Figure 6 is a photograph of the test setup, which is then shown in the oven in Fig. 7.

Fig. 4. Celanese compression test fixture, partly disassembled.

Fig. 5. Celanese compression test fixture.

Fig. 6. Flexural strength test apparatus.

Fig. 7. Flexural strength test apparatus in the oven.

At 232° C both composites made from K601 and AS fiber (i.e., with and without drape and tack in the prepreg) suffered drastic reductions in strength and modulus. The HX580/AS and K601/T300 combinations were best.

Room Temperature and 232° C Flexural Strength After 100 hr at 232° C (450° F)
(Table A-6b) — The changes from initial values at room temperature were insignificant. Only HX580/T300 showed a small strength loss at 232° C. All other composites increased in strength and modulus compared to the initial 232° C values. Cure appears to be continuing. This is particularly noteworthy in the case of HX580/AS which had the highest initial 232° C strength, which increased during the 100 hr to a value which is still 90% of the 100-hr room temperature value.

Room Temperature and 232° C Flexural Strength After 500 hr at 232° C (450° F)
(Table A-6c) — Relatively small changes in strength and modulus were observed at room temperature.

At 232° C, all strength and modulus values were still above those observed initially, with no significant changes since the 100 hr determinations. Table 2 gives the 232° C (450° F) flexural strengths. The HX580/AS system still retains top ranking.

Table 2. Composite high temperature flexural strengths vs heating time.

Composite	Initial, MPa (kpsi)	After 100 hr at 232° C (450° F), MPa (kpsi)	After 500 hr at 232° C (450° F), MPa (kpsi)
K601/AS ^a	0.121 (18)	0.193 (28)	0.212 (31)
HX580/AS ^b	0.939 (136)	1.52 (220)	1.33 (192)
HX580/T300 ^b	0.578 (84)	0.506 (73)	0.636 (92)
K601/T300 ^b	0.768 (111)	0.977 (142)	1.12 (163)
K601/AS ^b	0.157 (23)	0.230 (33)	0.231 (33)

^aNo drape and tack.

^bWith drape and tack.

Room Temperature and 232° C Flexural Strength After 10 hr at 316° C (600° F)
(Table A-6d) — At room temperature, both samples of K601/AS suffered drastic losses in strength and modulus. The HX580/T300 samples increased in strength very considerably, being essentially equivalent to the HX580/AS and K601/T300. Blistering and swelling of some samples distorted them such that the standard 32:1 span-to-depth ratio could not be achieved. Longer heating times at 316° C are needed before choices can be made.

At 232° C, all systems suffered marked decreases in strength and modulus except the HX580/T300 system. The two HX580 systems and the K601/T300 system were best.

Despite some variation in test results, it is worthwhile to note certain trends observed in this data. The "duplicate" samples of K601/AS gradually lose room temperature strength with heating time, but the 232° C (450° F) strength increases.

Whether this is the result of further solvent loss or of chemical changes is not known. This same thing is true for most of the other composites. If one considers all data for samples heated at 232°C, the HX580/AS system appears to be best with the K601/T300 a close second.

The average weight percent loss for each of the five materials after 500 hr at 232°C was: for K601/AS (no drape and tack), 2.91; for HX580/AS, 4.23; for the HX580/T300, 2.49; for the K601/T300, 1.93; and for the K601/AS (with drape and tack), 2.71. These losses may be a result of continuation of cure which appears to occur with prolonged heating.

Flexural Strength, Modulus Retention, and Weight Change after Exposure to Water (Table A-7)

Room Temperature and 232°C (450°F) Values After 1 Month at 49°C (120°F), 95% Relative Humidity – Essentially no changes in values or material rank order took place with measurements at room temperature.

At 232°C, a minor change in rank order was observed.

Room Temperature and 232°C (450°F) Values After 24 hr Water Boil – The K601/AS samples suffered some losses in ambient values. Values at 232°C were unchanged compared to values obtained with unexposed specimens.

It is not clear that one test is more severe than the other, although the samples that were boiled in water for 24 hr gained the most weight. Water at these exposure levels seems to have little effect upon composite performance. The HX580/AS system therefore remained first choice.

Interlaminar (Short Beam) Shear Strength on Unidirectional Laminates (Table A-8)

Respectable values were obtained for all systems except for the K601/AS system made from prepreg with drape and tack. This suggests that remaining solvent or solvent-reaction products were involved in lowering the shear strength compared to that seen with samples from prepreg having no drape and tack. A lower resin content or voids might have contributed also.

Flexural Fatigue Testing (Table A-9)

While not an established ASTM test procedure, we wanted to see if relative fatigue performance among the candidate materials could be determined. The usual prepreg materials were stacked alternately at 0° and 90°, cured, and specimens cut at a 45° angle. Initial flexural strength was determined, then fatigue testing done by cycling between 5 and 67% of the initial flexural values, at 8 cycles per second, which was slow enough not to increase temperature. The results showed considerable scatter, probably due to the great importance of edge effects. Moduli were recorded at intervals during the testing, and in general didn't change much until just before failure.

They are not reported because of inconsistencies. The K601/AS system (no drape and tack) had the highest flexural strength, and the HX580/T300 system had the most fatigue resistance.

Izod Impact Strength, Room Temperature (Table A-10)

Some indication of the impact resistance of the candidate composites was desired. The composite panels were approximately 60 mils thick, so a standard 0.5-in.-thick specimen was unavailable. ASTM specifications do not cover subsize specimens for plastic materials. One alternative considered consisted of stacking seven 60-mil blanks, held together with double-backed tape, to give a standard-size specimen. Insufficient composite was on hand to allow assembling the required number of such test samples.

The actual tests were made with side-notched specimens, following ASTM E-23 which covers metallic materials including samples with subwidths and subthicknesses. The results obtained were then used to calculate an approximation of the Izod impact energy of a standard-size specimen. The results, arranged in order of increasing impact strength, are shown in Table A-10.

While these results cannot be offered as completely adequate substitutes for impact strengths determined in the standard manner, we feel that the relative rank order is valid.

Chemical Resistance to Aircraft Fluids (Table A-11)

The fluids used were a standard aircraft lubricant and a new hydraulic fluid which is undergoing qualification. Specific identification, sources, and results are given in Table A-11.

Testing was done as follows. A sample of laminate weighing approximately 2 g was weighed and placed on a coarse screen on the bottom of a 75-ml beaker, covered with approximately 30 to 40 cc of test oil and placed in an oven at 49°C (120°F) for 1 week. On removal, the specimens were carefully wiped and reweighed. The weight changes shown in Table A-11 are considered too small to be significant, so no further testing was done. K601/AS with drape and tack gained slightly while all others lost weight. The test samples as well as new samples were examined microscopically. All surfaces appeared to be unchanged. While some differences in void content could be noted in a very qualitative way, they did not appear to be related to the observed weight changes.

ACKNOWLEDGMENTS

Thanks are due Norriss Hetherington for advice on statistical treatment of the data, and to E. T. Mones and Iven Anderson for performing some of the tests. We

gratefully acknowledge constructive criticisms given the report itself by Lynn Penn, Linda Clements, T. T. Chiao, J. Lepper, Harry Rizzo, and Beth Bathgate.

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APPENDIX A

STATISTICAL EXAMINATION OF TEST RESULTS AND TEST RESULT TABLES

The test results were evaluated statistically. Values given in the tables are averages for a certain number of specimens. In some cases, considerable spread in the data is apparent. To see if the differences among five different types of specimens were real, the value limits of $\pm 3 \sigma$ were calculated, and the resulting ranges examined for overlap. If overlap was present, limits of $\pm 2 \sigma$ were calculated. If anything more than a very minimal overlap persisted, those values were considered to be the same. The five values (or fewer if overlap was present) were then ranked 1 (best material), 2, 3, etc. The rank numbers are listed in Table A-1 for 15 panels and 35 test values. Summation of all rank numbers for each system showed that the HX580/AS system with a total of 48 best met our criteria, followed by K601/T300 with 51, HX580/T300 with 57, and K601/AS with 71. This treatment recognizes no weighting for test importance, but this can be done in any way desired by using the rank numbers in Table A-1. The 35 test values have been divided into six categories with the sum of rank numbers given for each, i.e., tensile, compression, flexure as a function of heating, fluxure as a function of exposure to moisture, interlaminar (short beam) shear, and flexural fatigue. The above treatment does not include results from five additional tests, i.e., weight changes from exposure to 95% RH, weight changes from exposure to boiling water, resistances to aircraft fluids, weight losses after 500 hr at 232°C (450°F), and results from Izod impact tests. The observed differences were small, or insufficient data exists to warrant inclusion.

Table A-1. Composite resin contents.^a

Laminate I.D. No.	Sample No.			Average
	1	2 Resin, wt%	3	
W5-70	37.3	36.9	37.4	37.2
W5-74	32.8	33.8	34.4	33.7 ^b
W5-74	37.7	32.5	35.5	34.2
W5-85	21.3	23.8	27.0	24.0
W5-85	28.9	24.2	22.8	25.3
W5-89	24.2	26.2	24.4	24.9
W5-96	26.4	28.3	27.7	27.5
W5-100	29.8	28.0	29.2	29.0
W5-116	35.1	31.4	30.6	32.4
W5-122	34.4	35.5	33.1	34.3
W5-128	23.1	24.7	20.1	22.8
W5-129	24.2	24.9	24.9	24.7

^aAll values by KOH method except as noted.^bBy HNO₃ method.

Table A-2. Tests conducted to evaluate composites.

Test	Test direction to fiber angle	ASTM No.	Specimen size	Grips	Gage length	Test machine	Test speed	Notes
1. Tensile strength and modulus 22.8°C (73°F)	0°	D-3039-72	8 X 0.5 X 0.055-0.066 in.	2-1.5 X 1/8 in. beveled Synthane, epoxy bonded	5 in.	Instron Universal	0.1 cm/min	2-in. 50% strain gage attached to specimen, 1-in. strain gage on specimen.
	90°	D-3039-72	4.5 X 1.0 X 0.055-0.066 in.	2-1.5 X 1/8 in. beveled Synthane, epoxy bonded	1.5 in.	Instron Universal	0.1 cm/min	
2. Compressive strength and modulus 22.8°C (73°F)	0°	Proposed method of test for composite properties of oriented fiber composites	5.5 X 0.250 X 0.057 -0.067 in.	Steel tabs, epoxy bonded	0.5 in.	Instron Universal	0.1 in./in./min or 0.35 in./in./min	Used "celanese" fixture. Strain gages bonded to each side of specimen.
3. Flexural strength and modulus, (a) Room temp., and after high temp. aging, both (b) wet and (c) dry	90°	D-790-71 (modified)	2.5-2.75 X 0.5 X 0.055 -0.065 in.	No grips; used 3 point loading with span: depth ratio of 32:1. Loading heads 0.250 in. steel rod.	-	Instron Universal	0.1 cm/min	The 232°C (450°F) specimens equilibrated 20-30 min before testing. Wet specimens were tested within 30 min after removal from moisture source.
4. Weight change after exposure to water, 49°C (120°F) and 100°C (212°F)	-	-	2.5-2.75 X 0.5 X 0.055 -0.065 in.	-	-	-	-	-
5. Interlaminar (short beam) shear strength 22.8°C (73°C)	90°	D-2344-72 (modified)	0.75 X 0.250 X 0.055 -0.065 in.	No grips; used 3 point loading with span: depth ratio of 4:1. All loading heads were 0.250-in. diameter steel rod.	-	Instron Universal	0.1 cm/min	
6. Flexural fatigue 22.8°C (73°F)	45° to 0°/90° layup.	-	2.5-2.75 X 0.5 X 0.055 -0.065 in.	No grips; used 3 point loading with span: depth ratio of 32:1	-	Materials testing machines	8 cycles per sec., between 5 and 67% of initial flex strength.	Initial flex, strength determined on Instron.
7. Izod impact 22.8°C (73°F)	90°	Guided by ASTM E-23 (for metals)	0.055 X 0.515 X 2.165 in.	-	-	T.O. plastics impact testing machine	-	Used "side-notched" specimens.
8. Aircraft fluid exposure, change after 1 wk/49°C (120°F)	-	-	2.5-2.75 X 0.5 X 0.055 -0.065 in. (flex specimens)	-	-	(Measured weight change)	-	

Table A-3. Composite ranking for various tests (unabridged).

Sample I. D. No.	Description	Fiber. wt%	Tests																														
			Longitudinal tensile, ultimate Modulus	Transverse tensile, ultimate Modulus	%elongation at failure	Subtotal	Compressive strength Modulus	R. T. flex. str., initial Modulus	R. T. flex. str., after 100 hr/232°C (450°F) Modulus after 100 hr/232°C (450°F)	R. T. flex. str., after 500 hr/232°C (450°F) Modulus after 500 hr/232°C (450°F)	R. T. flex. str., after 10 hr/316°C (600°F) Modulus	232°C (450°F) flex. str., initial Modulus, initial	232°C (450°F) flex. str., after 100 hr/232°C (450°F)	232°C (450°F) modulus, after 100 hr/232°C (450°F)	232°C (450°F) flex. str., after 500 hr/232°C (450°F)	232°C (450°F) modulus, after 500 hr/232°C (450°F)	232°C (450°F) flex. str., after 10 hr/316°C (600°F)	232°C (450°F) modulus, after 10 hr/316°C (600°F)	Subtotal	R. T. flex. str., after 30 da/95% RH Modulus, after 30 da/95% RH	R. T. flex. str., after 24 hr H ₂ O boil Modulus, after 24 hr H ₂ O boil	232°C (450°F) flex. str., after 30 da/95% RH	232°C (450°F) modulus, after 30 da/95% RH	232°C (450°F) flex. str., after 24 hr H ₂ O boil	232°C (450°F) modulus, after 24 hr H ₂ O boil	Subtotal	Short beam shear strength	Flexural strength (prefatigue) Modulus (prefatigue)	Cycles to failure	Subtotal			
5-70	K601/AS	62.8	1 ^a 2	1 ^a 1	1	6	1	1																									
5-89	HX580/AS	75.1	2 ^a 2	2 ^a 1	2	9	1	1																									
5-96	HX580/T300	72.5	3 ^a 2	2 ^a 1	2	10	1	1																									
5-116	K601/T300	67.6	2 ^a 1	2 ^a 1	1	7	1	1																									
5-128	K601/AS	77.2	1 ^a 2	2 ^a 1	2	8	1	1																									
5-74	K601/AS ^b	66						1	1	1	1	1 ^a 1	2	3	3	2	4	2	3	2	2	3	32	1 ^a 1	1	1	2	2	3 ^a 2	13	1 ^a		
5-85	HX580/AS	75						1	1	1	1	2 ^a 1	1	1	1	1	1	1	1	1	2	18	1 ^a 1	1	1	1	1	1 ^a 1	8	2 ^a			
5-100	HX580/T300	71						3	1	3	1	3 ^a 1	1	1	2	1	3	1	2	1	1	1	26	3 ^a 1	2	1	1	1	2 ^a 1	12	2 ^a		
5-122	K601/T300	65.7						2	1	2	1	2 ^a 1	1	1	1	1	2	1	1	1	2	21	2 ^a 1	1	1	1	1	2 ^a 1	10	2 ^a			
5-129	K601/AS	75						2	1	2	2	2 ^a 1	2	2	3	2	4	2	3	2	2	3	35	2 ^a 2	1	1	2	2	3 ^a 2	15	3 ^a		
6-12	HX580/T300																												2 ^a 2 ^a 1	5			
6-15	HX580/AS																												4 ^a 3 ^a 2	2			
6-17	K601/AS ^b																												1 ^a 1 ^a 5	5			
6-65	K601/T300																												2 ^a 3 ^a 4	4			
6-66	K601/AS																												3 ^a 3 ^a 3	3			

^aIndicates use of averages $\pm 2 \sigma$ instead of 3σ to determine overlap.^bNo drape or tack.

Table A-4. Tensile strength and modulus at room temperature.

<u>Longitudinal tensile properties</u>							
Laminate I. D. No.	Description	Fiber, wt%	No. of specimens	Ultimate stress, 10^3 MPa (10^3 psi)	C. V., ^a %	Modulus, 10^6 MPa (10^6 psi)	C. V., %
W5-70	K601/AS	62.8	5	1.55 (225)	4	0.092 (13.4)	6
W5-128	K601/AS	77.2	4	1.33 (193)	10	0.101 (14.7)	2
W5-89	HX580/AS	75.1	3	1.01 (146)	5	0.098 (14.2)	3
W5-96	HX580/T300	72.5	3	0.375 (54)	16	0.100 (14.5)	1
W5-116	K601/T300	67.6	3	1.02 (148)	3	0.108 (15.6)	3
<u>Transverse tensile properties</u>							
W5-70	K601/AS	62.8	3	0.047 (6.8)	19	0.009 (1.3)	3
W5-128	K601/AS	77.2	5	0.005 (0.8)	45	0.006 (0.94)	13
W5-89	HX580/AS	75.1	5	0.015 (2.1)	14	0.010 (1.5)	11
W5-96	HX580/T300	72.5	5	0.014 (2.0)	33	0.009 (1.3)	2
W5-116	K601/T300	67.6	5	0.018 (2.7)	32	0.008 (1.2)	8

^a Coefficient of variation.

Table A-5. Room temperature compressive properties.

Laminate I. D. No.	Description	No. of specimens	Ultimate compressive strength, MPa (psi)	C. V., %	Modulus, 10 ⁶ MPa (10 ⁶ psi)	C. V., %
W5-70	K601/AS	3	0.805 (116)	6	0.104 (15)	4
W5-128	K601/AS	3	0.632 (92)	8	0.112 (16)	3
W5-89	HX580/AS	3	0.643 (93)	7	0.109 (16)	7
W5-96	HX580/T300	3	0.800 (116)	6	0.117 (17)	2
W5-116	K601/T300	3	0.813 (118)	6	0.112 (18)	3

Table A-6a. Initial flexural properties.

<u>Measured at room temperature</u>								
Laminate I. D. No.	Description	Wt% fiber found	Prepreg solvent content ^a	No. of specimens	Flexural strength, 10 ³ MPa (10 ³ psi)	C. V., %	Modulus, 10 ⁶ MPa (10 ⁶ psi)	C. V., %
W5-74	K601/AS ^b	66	7.0	5	1.82 (264)	4	0.114 (17)	11
W5-129	K601/AS ^c	75	30.2	5	1.22 (176)	10	0.096 (14)	6
W5-85	HX580/AS ^c	75	—	5	1.83 (266)	5	0.114 (16)	4
W5-100	HX580/T300 ^c	71	—	5	0.575 (83)	11	0.114 (17)	5
W5-122	K601/T300 ^c	65.7	23.0	5	1.02 (148)	10	0.110 (16)	5
<u>Measured at 232°C (450°F)</u>								
W5-74	As above	As above		5	0.121 (18)	16	0.017 (2.5)	14
W5-129				5	0.157 (23)	9	0.034 (5.0)	12
W5-85				5	0.939 (136)	8	0.105 (15)	7
W5-100				5	0.578 (84)	8	0.105 (15)	3
W5-122				5	0.768 (111)	20	0.104 (15)	7

^aSolvent content calculated by weight loss in oven and based on resin only.

^bNo drape and tack.

^cWith drape and tack.

Table A-6b. Flexural properties after 100 hr at 232°C (450°F).

<u>Measured after cooling to room temperature</u>						
Laminate I. D. No.	Description	No. of specimens	Flexural strength, 10 ³ MPa (10 ³ psi)	C. V., %	Modulus, 10 ⁶ MPa (10 ⁶ psi)	C. V., %
W5-74	K601/AS	4	1.68 (244)	5	0.095 (14)	7
W5-129	K601/AS	4	1.15 (166)	11	0.093 (13)	4
W5-85	HX580/AS	4	1.59 (231)	2	0.114 (17)	3
W5-100	HX580/T300	4	0.531 (77)	13	0.118 (17)	2
W5-122	K601/T300	4	1.08 (156)	3	0.112 (16)	1
<u>Measured at 232°C (450°F)</u>						
W5-74	As above	4	0.193 (28)	10	0.026 (3.8)	11
W5-129		4	0.230 (33)	12	0.040 (5.8)	16
W5-85		4	1.52 (220)	4	0.112 (16)	3
W5-100		4	0.506 (73)	13	0.113 (16)	4
W5-122		3	0.977 (142)	4	0.114 (17)	1

Table A-6c. Flexural properties after 500 hr at 232°C (450°F).

<u>Measured after cooling to room temperature</u>						
Laminate I. D. No.	Description	No. of specimens	Flexural strength, 10 ³ MPa (10 ³ psi)	C. V., %	Modulus, 10 ⁶ MPa (10 ⁶ psi)	C. V., %
W5-74	K601/AS	4	1.61 (234)	3	0.084 (12)	5
W5-129	K601/AS	4	1.14 (166)	16	0.085 (12)	10
W5-85	HX580/AS	4	1.36 (197)	9	0.118 (17)	5
W5-100	HX580/T300	4	0.625 (91)	4	0.115 (17)	2
W5-122	K601/T300	4	1.01 (147)	8	0.108 (16)	5
<u>Measured at 232°C (450°F)</u>						
W5-74	As above	4	0.212 (31)	15	0.023 (3.3)	18
W5-129		4	0.231 (33)	11	0.043 (6.2)	15
W5-85		4	1.33 (192)	7	0.116 (17)	3
W5-100		4	0.636 (92)	10	0.112 (16)	5
W5-122		4	1.12 (163)	7	0.110 (16)	3

Table A-6d. Flexural properties after 10 hr at 316°C (600°F).

<u>Measured at room temperature</u>						
Laminate I. D. No.	Description	No. of specimens	Flexural strength, 10^3 MPa (10^3 psi)	C. V., %	Modulus, 10^6 MPa (10^6 psi)	C. V., %
W5-74	K601/AS	3	0.085 (12)	65	0.012 (1.7)	31
W5-129	K601/AS	3	0.234 (34)	37	0.040 (5.8)	17
W5-85	HX580/AS	3	1.21 (175)	13	0.105 (15)	4
W5-100	HX580/T300	3	1.27 (185)	2	0.109 (16)	5
W5-122	K601/T300	3	1.34 (195)	1	0.102 (15)	5
<u>Measured at 232°C (450°F)</u>						
W5-74	As above	3	0.108 (16)	22	0.015 (2.2)	23
W5-129		3	0.125 (18)	10	0.022 (3.2)	14
W5-85		3	0.573 (83)	8	0.087 (13)	6
W5-100		3	0.741 (107)	6	0.103 (15)	2
W5-122		3	0.743 (108)	20	0.083 (12)	9

Table A-7a. Flexural properties after 30 days at 95% relative humidity and 49°C (120°F).

Measured at room temperature

Laminate I. D. No.	Description	No. of specimens	Flexural strength, 10 ³ MPa (10 ³ psi)	C. V., %	Modulus, 10 ⁶ MPa (10 ⁶ psi)	C. V., %	%wt change
W5-74	K601/AS	4	1.67 (242)	7	0.101 (15)	2	+0.81
W5-129	K601/AS	4	1.19 (173)	12	0.100 (14)	3	+0.51
W5-85	HX580/AS	4	1.79 (259)	4	0.115 (17)	3	+0.73
W5-100	HX580/T-300	4	0.612 (89)	5	0.114 (16)	3	+0.74
W5-122	K601/T-300	4	1.15 (166)	4	0.114 (17)	1	+1.00

Measured at 232°C (450°F)

W5-74	As above	4	0.140 (20)	15	0.018 (2.7)	19	
W5-129		3	0.148 (21)	14	0.027 (3.9)	10	
W5-85		4	0.805 (117)	14	0.107 (15)	5	
W5-100		4	0.598 (87)	10	0.108 (16)	5	
W5-122		4	0.597 (87)	8	0.091 (13)	6	

Table A-7b. Flexural strength properties after 24 hr water boil.

Measured at room temperature

Laminate I. D. No.	Description	No. of specimens	Flexural strength, 10 ³ MPa (10 ³ psi)	C. V., %	Modulus, 10 ⁶ MPa (10 ⁶ psi)	C. V., %	%wt change
W5-74	AS	4	1.26 (183)	6	0.097 (14)	3	+1.96
W5-129		4	0.979 (142)	13	0.101 (15)	5	+4.22
W5-85		4	1.69 (246)	12	0.113 (17)	6	+0.79
W5-100		4	0.612 (89)	5	0.119 (17)	3	+0.86
W5-122		4	1.09 (158)	8	0.114 (17)	7	+1.97

232°C (450°F) flexural strength properties after 24 hr water boil

W5-74		4	0.135 (20)	21	0.017 (2.5)	24	
W5-129		4	0.161 (23)	6	0.036 (5.3)	9	
W5-85		4	0.955 (139)	11	0.104 (15)	6	
W5-100		4	0.673 (98)	5	0.114 (17)	2	
W5-122		4	0.609 (88)	4	0.094 (14)	4	

Table A-8. Interlaminar (short beam) shear test results.

<u>Measured at room temperature</u>				
Laminate I. D. No.	Description	No. of specimens	Short beam shear, 10^3 MPa (10^3 psi)	C. V., %
W5-74	K601/AS	10	0.111 (16)	3
W5-129	K601/AS	10	0.058 (8)	7
W5-85	HX580/AS	10	0.097 (14)	6
W5-100	HX580/T-300	10	0.078 (11)	15
W5-122	K601/T-300	10	0.093 (13)	6

Table A-9. Flexural fatigue properties.

<u>Measured at room temperature</u>								
Laminate I. D. No.	Description	No. of specimens	Flexural strength, 10^3 MPa (10^3 psi)	C. V., %	Modulus, 10^6 MPa (10^6 psi)	C. V., %	Cycles to failure	Range in cycles to failure
W6-17	K601/AS ^a	5	0.194 (28)	14	0.016 (2.4)	8	1,463	550-2,000
W6-66	K601/AS ^b	5	0.105 (15)	2	0.009 (1.3)	5	18,840	4,140-31,780
W6-15	HX580/AS ^b	4	0.086 (13)	7	0.008 (1.2)	6	27,910	6,600-40,130
W6-12	HX580/T300 ^b	4	0.136 (20)	7	0.012 (1.8)	1	37,512	28,000-48,570
W6-65	K601/T300 ^b	5	0.127 (18)	6	0.010 (1.4)	11	5,878	1,130-12,890

^aNo drape and tack.^bWith drape and tack.

Table A-10. Izod impact strength (calculated).

Laminate I. D. No.	Description	No. of specimens	Energy range (in. -lb/linear in.	Average of notch)
W5-96	HX580/T300	3	6.4-7.0	6.7
W5-128	K601/AS	3	142-195	175
W5-116	K601/T300	3	165-196	183
W5-70	K601/AS	3	337-408	362
W5-89	HX580/AS	1	—	464

Table A-11. Resistance to effects of aircraft fluids.

Laminate I. D. No.	Description	% wt change after 1 wk in 0-156 oil ^a at 49°C (120°F)	% wt change after 1 wk in Mil-H-83282 ^b at 49°C (120°F)
W5-74	K60/AS	-0.22	-0.22
W5-85	HX580/AS	-0.36	-0.42
W5-100	HX580/T300	-0.14	-0.22
W5-122	K601/T300	+0.11	-0.08
W5-129	K601/AS	+0.82	+1.02

^aMil-23699 aircraft engine oil, provided by Alameda, Calif., Naval Air Station, (Humble Oil and Refining Co. product.)

^bPurchased from Royal Lubricants Co., River Road, E. Hanover, N. J., 07936.

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