1.0 INTRODUCTION

The purpose of this annual progress report is to summarize the work effort and results accomplished from 7/87 through 7/88 on NASA Research Grant NAG-1-659 entitled Micromechanics of Composite Laminate Compressive Failure. The report contains (1) the objective of the proposed research, (2) the summary of accomplishments during this contract year, (3) a more extensive review of compression literature, (4) the planned material (and corresponding properties) received to date, (5) the results for three possible specimen geometries, experimental procedures planned, and current status of the experiments, and (6) the work planned for the next contract year.

2.0 OBJECTIVE

The objective of this proposed research program is to develop a better understanding of the factors that determine the notched compressive strength and failure mode of graphite/polymeric composite laminates with multi-directional stacking sequences.

3.0 SUMMARY OF ACCOMPLISHMENTS
07/01/87 THROUGH 08/01/88

The major accomplishments of this research grant from 7/87 through 7/88 include the following:

3.1 The completion of the initial phase of compression testing (E. Gail Guynn's M.S. Thesis) characterizing the micromechanisms of compression failures in
open hole composite laminates.

3.2 Ms. Guynn's thesis has been converted to three publications, all presently accepted to be published in technical journals. The two most recent publications are included in this report. The first paper was sent to Dr. John D. Whitcomb in June 1987.

3.3 The completion of a very comprehensive literature review detailing the compressive failure modes and models, the effect of matrix resin properties on compressive strength, the effect of fiber properties on the compressive strength of composite materials, the effect of fiber properties on the compressive strength of composite materials, the effect of interfacial bonding on compressive strength, microbuckling and kinking, and post-impact compressive strength. This review has been extended from 38 to 100 references. However, only the list of references is included in this report.

3.4 The design of a material test matrix which includes systematic variation of many factors (supporting fiber orientation, fiber waviness, interfacial bonding, and presence of neat resin interleaf) to try to determine the individual role of each factor on the compressive strength of composite materials. Imperial Chemical Industries, Inc. (David Leach) has agreed to supply all material for these tests, and the material has been ordered. Approximately, one-half of this material has been received to date.

3.5 Three specimen geometries have been evaluated for optimum observation of stable damage growth. These geometries include the open hole specimen (from initial phase of compression testing), compact tension-like specimen, and a four-point bend specimen. Two additional geometries to be tested include (1) a specimen similar to the open hole compression specimen but with semi-circular
edge notches rather than the center hole and (2) a compression specimen with a tapered gage section, but no notch, to provide a simpler stress distribution in the specimen.

3.6 Test methods (include 70°F to 270°F) required for this program have been evaluated, and fixtures have been designed to either meet the new requirements or to improve the present test procedures.
4.0 LITERATURE REVIEW

A fairly comprehensive literature review detailing the compressive failure modes and models, the effect of matrix resin properties on compressive strength, the effect of fiber properties on the compressive strength of composite materials, the effect of fiber properties on the compressive strength of composite materials, the effect of interfacial bonding on compressive strength, microbuckling and kinking, and post-impact compressive strength was included in the 1987-88 semi-annual report. This review has been extended from 38 to 101 references as a part of Ms. Guynn's dissertation proposal (and future dissertation), one of the university requirements toward a Ph.D. This complete list of references is included in Section 8.0 of this report. It should be noted that this list does not at the present time include all of the relevant articles cited in the 1986-87 annual progress report. A copy of the complete dissertation proposal will be sent to the contract monitor, Dr. John D. Whitcomb, upon its completion and committee approval.
5.0 MATERIAL TEST MATRIX

The composite material system selected for this investigation was AS4/APC-2 (Aromatic Polymer Composite), a thermoplastic. The necessary material has been promised to be supplied by Imperial Chemical Industries, Inc. in Arizona, USA. Dr. David Leach is the contact for this material order, and the order was placed through Dr. Leach in early January, 1988.

In order to evaluate the factors that affect the compressive failure strength, the laminate stacking sequence was systematically varied. Initially, industry personnel were contacted to help choose an “in-service” lay-up. However, complications such as unsymmetric laminates and very complex stacking sequences inhibited systematic variation of the factors in this investigation. Thus, a relatively simple baseline stacking sequence, [(±45, 0\textdegree)\textsubscript{3}/±45/0\textsubscript{3}], was selected. Systematic variations of this stacking sequence will allow for a careful study of the effects of interfacial bond strength, fiber waviness, supporting fiber orientation, and resin rich regions. Note that for consistency, these variations are made through the entire laminate thickness. Appendix A contains Tables I–VI detailing the variation of the stacking sequences. Additionally, the panels of material received to date are highlighted on these tables. Material properties and data supplied by ICI are included in Appendix B. Laminate analyses for the stacking sequences selected are included in Appendix C.

Table I details the entire test matrix, including all variables (supporting fiber orientation, fiber waviness, interfacial bonding, and neat resin interleaf). Table II details the 3 stacking sequences used to vary the support to the 0\textdegree fibers. In this case, the ±45’s in the baseline stacking sequence are replaced with either ±15 (almost vertical support), ±75, or 90\textdegree (entirely horizontal support).

Table III lists the 3 stacking sequences used to determine the effects of fiber
waviness. In this case, the theoretically straight 0° fibers in the baseline stacking sequence are replaced with (0/90) woven plies two weaves (one relatively tight and one relatively loose) will be tested. However, due to availability, it may only be possible to obtain only one weave.

Interfacial bonding will be varied using two panels with the same baseline stacking sequence but one panel will be made with APC-2 to provide a good interfacial bond and the other panel with APC-1 to provide a poor fiber/matrix bond, as shown in Table IV. If APC-1 resin is not available, an alternative approach will be to use untreated fibers to give a poor fiber/matrix interface. The effect of the neat resin interleaf (Table V) will be studied by adding an interleaf (of two different thicknesses) at each interface through the thickness of the baseline panel. To determine $G_{12}$ (Table VI), a panel of $[\pm45]_{2s}$ will be tested in tension and $[\pm45]_{8s}$ will be tested in compression to more carefully characterize the material system. These tests will be run over a range of temperatures (70°F through 270°F).

The dependent variables to be studied in this research program will be compressive strength of a reduced gage compression specimen, compression strength of a compression specimen with two semi-circular edge notches (on opposite free edges) midway along the gage section, and load at damage initiation for compact tension specimens loaded in compression. Both the load at initial damage initiation in the zeros and the subsequent development of damage will be monitored as a function of the independent variables previously described.
6.0 EXPERIMENTAL PROCEDURES

Preliminary experimental work will be described in this section.

6.1 Open Hole Compression Tests

Four open hole compression tests were conducted in a specially designed high axial alignment Material Test System (MTS) machine in the Materials and Structures Laboratory of Texas A&M University. Figure 1a shows this test apparatus. Figure 1 is a close-up of the specimen configuration in the machine grips with the stereomicroscope in the background. The specimens tested were loaded in compression to failure in the servo-controlled hydraulic test stand at a relatively slow rate in strain gage control to provide more stable growth of the shear crippling zone. The strain rate was approximately 1000με/min. The purpose of these tests was to verify that reducing the gage length would not give significant end effects in these tests, and yet increase the stability (with respect to Euler buckling) of these tests.

The specimens tested were quasi-isotropic [(0/ ± 45/90)_4], IM6/HST-7 specimens (remaining from the initial phase of this grant). Each specimen contained a 0.0625 in. diameter center hole this specimen geometry is shown in Fig. 2. Longitudinal strain gages (designated Control & Opposite) were placed on each specimen front and back, on the horizontal centerline at the edge of the specimen, as shown in Fig. 2b. These specimens were 1.0 in. wide, each with a different gage length. The gage lengths tested were 2 in., 1.5 in., 1.0 in., and 0.5 in. The stress-strain data for each of the 2.0 in., 1.5 in., and 1.0 in. gage lengths are shown in Figs. 3-5, respectively. Comparison of the parallel curves in Fig. 5 with Figs. 3 and 4 show that the 1.0 gage length specimen has less Euler type bending than the longer
Fig. 1 Compression test facilities.
   a) Compression test set-up with stereomicroscope and video attachments.
   b) Specimen configuration in the hydraulic grips with the stereomicroscope in the background.
Fig. 2. Open hole compression test specimen configuration.

a) Compression test specimen.

b) Strain gage specimen configuration.

- $r = \{0.08 \text{ cm}, 0.16 \text{ cm}, 0.32 \text{ cm}\}$
- $2a = 2.54 \text{ cm}$
- $2b = 12.7 \text{ cm}$
- $2c = 6.35 \text{ cm}$
- $2d = 3.81 \text{ cm}$

- Opposite
Fig. 3  Typical stress-strain data for a center notched specimen.
Hole diameter is 0.0625 in.
Gage length is 2.0 in.
OPEN HOLE COMPRESSION

GL=1.5 in.

LEGEND

- Control
- Opposite

Fig. 4  Typical stress-strain data for a center notched specimen.
Hole diameter is 0.0625 in.
Gage length is 1.5 in.
Fig. 5 Typical stress-strain data for a center notched specimen. Hole diameter is 0.0625 in. Gage length is 1.0 in.
gage lengths. It should be noted that there is a greater offset in the strain gage data shown in Fig. 5, compared to the offset shown in Figs. 3 and 4. This offset is attributed to the gripping process of the MTS. A fixture to hopefully decrease this offset has been designed, and this fixture is described in subsection 6.4. The control strain gage data for these 3 tests is then summarized in Fig. 6, and the opposite strain gage data for these 3 tests is summarized in Fig. 7. These stress-strain plots indicate that decreasing the gage length from 2.0 in. to 1.0 in. will not introduce significant end effects and will minimize the incidence of Euler buckling.

Results from the 0.5 in. gage length specimen are not presented because it was not possible to test this specimen. This shorter gage length is not long enough to allow proper instrumentation and observation of the damage growth.

From these tests it was concluded that the gage length for future open hole compression tests will be reduced from 2.0 in. to 1.0 in. Additional alterations of this specimen design include making the same specimen but replacing the center hole with two semi-circular edge notches to facilitate microscopic observation of damage. An unnotched, tapered gage section specimen will also be tested to see if stable damage growth prior to catastrophic failure can be obtained in an unnotched specimen. This specimen would have the advantage (over the specimens with center or edge holes) of a simple stress distribution within the gage section.

6.2 Compact Tension-Like Specimens
Loaded in Compression

A second type of specimen geometry to test and directly observe the failure process in graphite/polymeric composites is a compact tension-like specimen (see Fig. 8), but modified to give a rounded notch rather than sharp crack tip. The chevrons and sidegrooves have also been omitted. To obtain the optimum design,
Fig. 6 Summary of stress-control strain data for the three different gage length tests.
Fig. 7 Summary of stress-opposite strain data for the three different gage length tests.
Fig. 8  Compact tension-like specimen configuration.
1\* C.T.
E.M.C.T.
for these specimens, a number of iterations from the standard 1" Compact Tension (C.T.) specimen and Electron Microscopic (E.M.) Compact Tension specimen (designed at Texas A&M University) designs have been performed. These specimens were loaded in compression in displacement control at a rate of 0.01 in./min. in a servo-controlled hydraulic MTS as shown in Fig. 9. Load-displacement data was recorded and will be presented later in this section.

Three different notch types were tried in each of the one-inch and E.M. C.T.'s. The three notches were (1) 0.0625 in. square tip, (2) 0.0625 in. diameter slot and radius at the tip, and (3) 0.03125 in. diameter slot and radius at the tip.

The square crack tip was eliminated because it behaved like 2 sharp notches at the corners of the crack tip. Very little fiber microbuckling was observed in the notch. Furthermore, ply delamination was observed at the compressive free edge, probably due to the stress concentration at the loading points, in one specimen.

The 0.03125 in. diameter notch and crack tip radius was eliminated because of inaccessibility to observe the fiber microbuckling process. The 0.0625 in. slot and crack tip radius was determined to be the optimum design.

Because the C.T.-like specimen is loaded in compression, the notch at the free edge (closer to the loading pins) always closed during loading, well ahead of failure. Thus, the material from the load-line to the free-edge was cut away at a 45° angle (see Fig. 8) to increase allowable displacement and allow a much larger damage zone to propagate from the crack tip. However, one anticipated problem with this geometry is the interaction between the stress field at the crack tip and the stress field near the tensile free edge.

Load-displacement data for two 1" C.T. specimens (HST-7 material) are shown in Fig. 10. The surfaces were polished to better observe the fiber microbuckling
Fig. 10  Typical load-displacement curves for two HST-7 compact tension -like specimens.
process. The inherent stability of damage growth in this specimen geometry in tension is also noted for compressive loading. Very stable damage growth was observed from the crack tip to across approximately two-thirds of the ligament width, as indicated by the specimen that was included with the 1987-88 semi-annual report. Also, as seen in Fig. 10, the results are very repeatable. Catastrophic failures (in fact, specimen failures of any kind) were not observed in any of these specimens because the notch at the free edge closes (thus, requiring unloading) before the damage zone reaches a critical size.

The E.M.C.T. was tested with two different notch lengths, 0.550 in. and 0.350 in. In most of the specimens with the shorter notch, ply delamination was again observed at the free edge nearest the loading pins, and in the E.M.C.T. with the longer notch, tensile failures were observed at the tensile free edge farthest from the loading pins. Consequently, the modified 1” C.T. (Fig. 8a) is considered the superior specimen geometry.

Since damage growth in this specimen was very stable, we are hopeful to test the specimen in the scanning electron microscope (S.E.M.) and observe, in-situ, the fiber microbuckling process. However, the load limitation (100 lb) of the SEM tensile stage has temporarily inhibited these observations. Presently we are considering ways to scale down the C.T. so that failure will occur within the 100 lb load limitation. An alternate design for the C.T. fixture is also being considered. This design would increase the allowable applied load, but would only allow intermittent observation of the damage growth. The specimen would be loaded in this fixture (outside the column of the S.E.M.) in small incremental amounts, and then placed in the S.E.M. (after each incremental loading) for observation of the damage growth.
6.2.1 Scanning Electron Microscopy

To verify the uniformity of the damage zone through the thickness of the C.T.'s, two HST-7 C.T.'s were sectioned. One 1" C.T. was sectioned through the thickness of the laminate, examining (X-Y plane, see Fig. 8) only the 0° plies. The damage zone in each of the 0° ply groups measured 0.21", 0.26", 0.19", 0.20", and 0.18", respectively through the thickness. Additionally, one 1" C.T. was sectioned across the ligament width to just behind the notch tip (examining the Y-Z plane, see Fig. 10). The damage through the thickness of this C.T. is shown in Fig. 11. Figure 12a enlarges details of the 0° shear crippling zone and matrix microcracking. Fiber/interleaf debonding and resin microcracking (due to severe deformation) are shown in Fig. 12.

6.3 Four-Point Bend Tests

Two types of four-point bend specimen geometries have been tested thus far. These specimens are shown in Fig. 13. Notice the direction of the plies in this figure. The material used for specimens was quasi-isotropic IM6-HST-7 (remaining from the initial phase of this grant). Two types of notches (specimen geometry in Fig. 13a) have been tested unsuccessfully, so far. First, a deep, blunt notch was tested. In these test failure was initiated by fiber breakage on the tensile surface. Second, a shallow, sharp notch was tested. In this test failure was initiated by ply delamination on the compressive free edge, near the notch.

The second four-point bend specimen geometry (Fig. 13b) has been tested on two stacking sequences ([(±45/0₂)₃/ ± 45/0₂₃] and ([±75/0₂)₃/ ± 75/0₂₃] of newly acquired AS4/APC-2 material. Both specimen geometries were failed by tensile failures on the tension side of the specimens. Since the material tensile
Fig. 11 Through the thickness damage (Y-Z plane) in an HST-7 compact tension-like specimen.
Fig. 12 Details of the shear crippling zone and matrix microcracking.
Fig. 13 Four-point bend specimen geometry.
strengths are typically higher than the compression strengths, a compression failure was expected. Since the modulus in compression is less than that tension, the neutral axis has probably shifted, and thus, the stresses on the compressive surface of the specimen may be significantly less than the stresses on the tensile surface of the specimen. This stress distribution could account for the unexpected failures on the tensile side of the specimen. Presently, we are in the process of testing some strain-gaged AS4/APC-2 and IM6-HST-7 specimens to see if they will provide some stable growth of the fiber microbuckling process.

6.4 Shim Casting for Open Hole Compression Tests

The specially designed MTS (previously described, shown in Fig. 1) contains a collet type grip arrangement. The collet inserts which grip the specimen are machined for an ideal thickness, e.g. 0.210±0.001 in. However, due to the nonuniform thickness of laminated composites, most specimens in previous compression tests were shimmed symmetrically with precision brass shims.

To further improve the alignment and gripping support provided to the notched compression tests a fixture has been designed to allow resin shims to be cast onto the specimen ends. The cast shims will be within the thickness tolerances required by the collet inserts and will also be uniformly bonded to the specimen ends. The importance of the shimming process is mentioned in many literature citations in the Test Methods literature, and this importance will be described in the dissertation proposal that you will receive. Drawings for this fixture are included in Figs. 14–16.

6.5 High Temperature Tests

Test methods for this proposed research work include testing in the temperature
Fig. 14 Shim casting fixture, assembly drawing.

NOTES:

DRAWN TO SCALE

*SEE ATTACHED DETAILS

2 NON-ROTATING SPINDLE MIOMETER
HEADS - M. HUTONO (153-194) * $4.80 EA

**THUMBSCREW
TIP DETAIL

END CAPS
ALUMINUM

INSERT
BLOCKS
ALUMINUM

STEEL
(AEROCOAT)

0.250" 0.250" 0.750"

0.210"

0.500"

0.500"

0.875"

1.750"

2.000"

2.500"

1.000"

0.500"

1.250"

2.500"
END CAPS
FOR SHIM CASTING FIXTURE

NOTES:
TWO PIECES
MATERIAL: ALUMINIUM

Fig. 15 End caps for shim casting fixture.
**INSERT BLOCKS**

FOR SHIM CASTING FIXTURE

---

**Notes:**

**These blocks must have a good fit in the slots provided in the shim casting fixture.**

**Two pieces.**

**Material: Aluminum**

---

**Fig. 16** Insert blocks for shim casting fixture.
range from 70°F through 270°F. This range will provide variations in the degree of
softness of the APC-2 resin and thus, variations in the support provided to the 0°
fibers.

To provide easier observation and access to the specimen, a heat gun with a
controller, rather than an environmental chamber, will be used to heat the specimen
gage section. In addition to easier access, the heat gun heats the specimen to
test temperature much faster (10 min. compared to 1 hour) than the traditional
environmental chamber which also has to heat the MTS grips. The heat gun idea
was first suggested to Ms. Guynn in a class (Designing With Composites) at Texas
A&M University. In fact, General Dynamics in Fort Worth, TX uses the heat gun
because it more realistically simulates the heat spikes that an aircraft typically
experiences.

Some preliminary tests have been completed on the orthotropic \([([\pm 45/0_2])_2/\pm 45/0/90)_2\) AS4/PEEK specimens which remain from the initial phase of this
grant. In these tests, the thermocouple was embedded in the center of the specimen
thickness. Results indicate that these high temperature tests will need to be run in
displacement, rather than strain gage, control because of the instability (caused by
the air flow) in the strain gages. Additionally, the thermocouples will be embedded
in dummy specimens for the actual tests. The dummy specimens will be located so
they receive the same hot air flow as the actual specimen. Presently, we are trying
to work out the details of calibrating (e.g. similar heat losses) the dummy specimen
with the test specimen. It was also determined from embedded thermocouples that
when the gage section is 270°F, the gripped ends are approximately 200°F. Tabs
were also bonded to these trial specimens with different adhesives to attempt to
find a room temperature cure adhesive which will bond to the APC-2 material.
The tension $G_{12}$ tests will require tabbed ends in this temperature range. The best adhesives were found to be Loctite Super Bonder 495, Elmer's Epoxy Cement, and PC-7 (Protective Coating Co., Allentown, PA). However, all of these adhesives are too viscous to be used as cast resin shim material. An epoxy supplied by Dow Chemical, Freeport, TX will be investigated for this purpose.
7.0 SUMMARY OF CURRENT STATUS

The test matrix of independent variables and dependent variables to be measured has been defined. The material has been ordered from ICI (early January, 1988), and the material received to date is included in this report. Three specimen geometries have been verified to allow observation of the fiber microbuckling process. A fixture has been designed to allow resin shims to be cast onto the specimen ends. These shims should improve alignment during compression loading. Methods for high temperature testing have been evaluated. A heat gun capable of 500°F–700°F has been purchased, and a temperature controller is being incorporated with the gun. Two new publications (included in Appendices D and E) from the 1986-88 research grant period have been accepted for journal publication.

Presently, Ms. Guynn is in the process of completing her dissertation proposal for committee approval. Upon approval, this proposal (which includes a detailed literature survey) will be sent to Dr. John Whitcomb. Additionally, specimens for the initial testing using the new specimen geometries, shim casting fixture, and heat gun are presently being machined.
8.0 REFERENCES

8.1 Previous Grantee Publications


8.2 Test Methods


8.3 Failure Modes


8.4 Composite Failure Models


8.4.1 Fiber Microbuckling Models


8.4.2 Matrix Nonlinearity & Fiber Waviness/Curvature


8.4.3 Fiber Shear and Longitudinal Splitting


8.4.4 Micromechanics and Damage Approaches to Compressive Strength


8.4.5 Euler Buckling Analyses


8.5 Microbuckling and Kinking


8.6 Effects of Resin Properties on Compression Strength


8.7 Effects of Fiber Properties on Compression Strength


8.8 Effects of Interfacial Bonding on Compression Strength


Gray, R.J., "Experimental Techniques for Measuring Fibre/Matrix Interfacial Bond Shear Strength," The University of British Columbia, Vancouver, B.C., Canada.


8.9 Environmental Effects


APPENDIX A

MATERIAL TEST MATRIX
Table I. Composite Laminate Material Test Matrix.

<table>
<thead>
<tr>
<th>Stacking Sequence</th>
<th>Variable Investigated</th>
</tr>
</thead>
<tbody>
<tr>
<td>[±45/(0/90)/ ± 45/(0/90)/ ± 45/(0/90)/ ± 45/0],</td>
<td>Baseline APC-2 resin</td>
</tr>
<tr>
<td>[±45/(0/90)/ ± 45/(0/90)/ ± 45/(0/90)/ ± 45/0],</td>
<td>Interchange 0's and 45's to observe surface 0's</td>
</tr>
<tr>
<td>[±45/(0/90)/ ± 45/(0/90)/ ± 45/(0/90)/ ± 45/0],</td>
<td>Replace 0_2 with woven (0/90) - tight weave</td>
</tr>
<tr>
<td>[±45/(0/90)/ ± 45/(0/90)/ ± 45/(0/90)/ ± 45/0],</td>
<td>Replace 0_2 with woven (0/90) - loose weave</td>
</tr>
<tr>
<td>[±45/0_2/ ± 45/0_2/ ± 45/0_2/ ± 45/0],</td>
<td>APC-1 resin, bad fiber/matrix interface</td>
</tr>
<tr>
<td>[±15/0_2/ ± 15/0_2/ ± 15/0_2/ ± 15/0],</td>
<td>Vary support to 0's</td>
</tr>
<tr>
<td>[±45/0_2/ ± 45/0_2/ ± 45/0_2/ ± 45/0],</td>
<td>Vary support to 0's</td>
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<tr>
<td>[±45/0_2/ ± 45/0_2/ ± 45/0_2/ ± 45/0],</td>
<td>Vary support to 0's</td>
</tr>
<tr>
<td>[±45/0_2/ ± 45/0_2/ ± 45/0_2/ ± 45/0],</td>
<td>Vary neat resin interleaf thickness, t=0.001in.</td>
</tr>
<tr>
<td>[±45/0_2/ ± 45/0_2/ ± 45/0_2/ ± 45/0],</td>
<td>Vary neat resin interleaf thickness, t=0.003in.</td>
</tr>
<tr>
<td>[0]_{24}</td>
<td>Measure ( G_{12} ) - tension tests</td>
</tr>
<tr>
<td>[0]_{24}</td>
<td>Measure ( G_{12} ) - compression tests</td>
</tr>
<tr>
<td>[0]_{24}</td>
<td>Baseline ( G_{fc} ) measurements - teflon insert</td>
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Table II. Effects of Supporting Fiber Orientation.

<table>
<thead>
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<th>Stackign Sequence</th>
<th>Variable Investigated</th>
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<tr>
<td>[±15/0 ± 15/0 ± 15/0 ± 15/0],</td>
<td>Vary support to 0's</td>
</tr>
<tr>
<td>[±15/0 ± 15/0 ± 15/0 ± 15/0],</td>
<td>Vary support to 0's</td>
</tr>
<tr>
<td>[±45/0 ± 45/0 ± 45/0 ± 45/0],</td>
<td>Baseline lay-up</td>
</tr>
<tr>
<td>[±15/0 ± 15/0 ± 15/0 ± 15/0],</td>
<td>Vary support to 0's</td>
</tr>
<tr>
<td>[±15/0 ± 15/0 ± 15/0 ± 15/0],</td>
<td>Vary support to 0's</td>
</tr>
</tbody>
</table>

Table III. Effects of Fiber Waviness.

<table>
<thead>
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<th>Stackign Sequence</th>
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<tbody>
<tr>
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<td>Baseline lay-up</td>
</tr>
<tr>
<td>[±45/0/90/ ± 45/0/90/ ± 45/0/90/ ± 45/0],</td>
<td>Replace 02 with woven (0/90) - tight weave</td>
</tr>
<tr>
<td>[±45/0/90/ ± 45/0/90/ ± 45/0/90/ ± 45/0],</td>
<td>Replace 02 with woven (0/90) - loose weave</td>
</tr>
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</table>

Table IV. Effects of Interfacial Bonding.

<table>
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<th>Variable Investigated</th>
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<tbody>
<tr>
<td>[±45/0 ± 45/0 ± 45/0 ± 45/0],</td>
<td>Baseline APC-2 resin</td>
</tr>
<tr>
<td>[±45/0 ± 45/0 ± 45/0 ± 45/0],</td>
<td>APC-1 resin, bad fiber/matrix interface</td>
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</table>
Table V. Effects of Neat Resin Interleaf.

<table>
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<th>Variable Investigated</th>
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</thead>
<tbody>
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<td>Baseline APC-2 resin, no interleaf</td>
</tr>
<tr>
<td>±45/0₂/±45/0₂/±45/0₂/±45/0₂</td>
<td>Vary neat resin interleaf thickness, t=0.001in.</td>
</tr>
<tr>
<td>±45/0₂/±45/0₂/±45/0₂/±45/0₂</td>
<td>Vary neat resin interleaf thickness, t=0.003in.</td>
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Table VI. Determination of $G_{12}$.

<table>
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<th>Stacking Sequence</th>
<th>Variable Investigated</th>
</tr>
</thead>
<tbody>
<tr>
<td>±45/0₂</td>
<td>Measure $G_{12}$ - tension tests</td>
</tr>
<tr>
<td>±45/0₂</td>
<td>Measure $G_{12}$ - compression tests</td>
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</tbody>
</table>

Table VII. DCB ($G_{fc}$) Test Matrix.

<table>
<thead>
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<th>Stacking Sequence</th>
<th>Variable Investigated</th>
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</thead>
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<tr>
<td>[0]₂₄</td>
<td>Baseline $G_{fc}$ measurements</td>
</tr>
<tr>
<td>[±45/0₂ ±45/0₂ ±45/0₂ ±45/0₂]</td>
<td>Baseline APC-2 resin</td>
</tr>
<tr>
<td>[±45/(0/90)/±45/(0/90)/±45/(0/90)/±45/0]</td>
<td>Interchange 0's and 45's to observe surface 0's</td>
</tr>
<tr>
<td>[±45/(0/90)/±45/(0/90)/±45/(0/90)/±45/0]</td>
<td>Replace 0₂ with woven (0/90) - tight weave</td>
</tr>
<tr>
<td>[±45/0₂/±45/0₂/±45/0₂/±45/0₂]</td>
<td>Replace 0₂ with woven (0/90) - loose weave</td>
</tr>
<tr>
<td>[±15/0₂/±15/0₂/±15/0₂/±15/0₂]</td>
<td>APC-1 resin, bad fiber/matrix interface</td>
</tr>
<tr>
<td>±45/0₂ ±45/0₂ ±45/0₂ ±45/0₂</td>
<td>Vary support to 0's</td>
</tr>
<tr>
<td>±45/0₂ ±45/0₂ ±45/0₂ ±45/0₂</td>
<td>Vary support to 0's</td>
</tr>
<tr>
<td>±45/0₂ ±45/0₂ ±45/0₂ ±45/0₂</td>
<td>Vary neat resin interleaf thickness, t=0.001in.</td>
</tr>
<tr>
<td>±45/0₂ ±45/0₂ ±45/0₂ ±45/0₂</td>
<td>Vary neat resin interleaf thickness, t=0.003in.</td>
</tr>
</tbody>
</table>
APPENDIX B

MATERIAL PROPERTY LITERATURE
APC-2 AROMATIC POLYMER COMPOSITES
PEEK/CARBON FIRE

APC-2 PEEK/carbon fibre, aromatic polymer composites, have opened up new horizons for lightweight structural materials.

The benefits of engineering with composites utilising high performance fibres of carbon, boron, and so on have been known for some years.

APC-2, a thermoplastic system, benefits from an engineering approach to fabrication processes and the economics of rapid, automated and controlled part production that this brings.

APC-2 is based on carbon fibre and Victrex® PEEK aromatic polymer from ICI. Compared with other types of thermoplastic matrix PEEK has a higher operating temperature and is unaffected by solvents.

But the real secret of APC-2 is the specially developed interface science which, together with a process designed to ensure thorough impregnation of the carbon fibres, results in negligible void content and maximises the matrix dominated properties. By providing effective stress transfer, the PEEK matrix allows the full properties of carbon fibre to be obtained.

APC-2 PEEK/CARBON FIBRE

* 30% lighter in weight than aluminium.
* uses rapid, automated, economic fabrication processes.
* high impact toughness and excellent damage tolerance.
* good creep and fatigue resistance.
* high temperature performance — Tg 143°C (290°F).
* excellent hot/wet strength.
* low water absorption and excellent solvent resistance.
* outstanding fire resistance, negligible smoke generation.
* easily repairable.

APC-2 the most cost-effective route to second generation advanced composites.

CONTACT: DNE LEESER (602) 750-2000
\( \nu = 0.3 \)
APC-2: PEEK®/carbon fibre composite is an advanced structural composite of a proprietary high strength, continuous carbon fibre in PEEK matrix. The carbon fibres are well dispersed and thoroughly wetted in the PEEK matrix to give a fibre content of 61% by volume and 68% by weight.

APC-2 is available in a variety of forms covering prepreg tape and tow and consolidated sheet. The following are typical standard forms:

**UNIDIRECTIONAL PREIMPREGNATED ("PREPREG") TAPE**

1. **Prepreg tape — fibre content 68% ± 2% by weight**
   - Standard widths: 305 mm (12.0 in)
   - Nominal thickness: 0.125 mm (0.005 in)
   - Nominal weight: 213 gm/m² (0.044 lb/ft²)
   - Areal weight of carbon: 145 gm/m² (0.030 lb/ft²)
   - Standard lengths: 150 m (450 ft)
   - Supplied wound on 153 mm (6.0 in) diameter reels.

2. **Slit prepreg tape (for tape laying etc.)**
   - Standard widths: 75 mm (3 in)
   - Lengths up to: 50 m (166 ft)
   - Supplied wound on 153 mm (6.0 in) diameter reels

3. **Prepreg tow**
   - Now at an advanced development stage for filament winding, weaving and braiding.

Properties (fibre volume, areal weight etc) as for prepreg tape.

<table>
<thead>
<tr>
<th>Widths</th>
<th>6 K tow</th>
<th>3 mm</th>
<th>(0.120 in)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>12 K tow</td>
<td>6 mm</td>
<td>(0.240 in)</td>
</tr>
</tbody>
</table>

| Standard lengths | 1000 m   | (3000 ft) |

Pre-impregnated tape and filament can be laminated and consolidated by remelting the PEEK matrix and applying a low pressure for a short time. See Data Sheet 2.

**CONSOLIDATED SHEET**

Produced by laminating prepreg under controlled conditions to ensure that optimum properties are attained (see Data Sheet 2). Void content is less than 0.5% and typically around 0.1%.

4. **Unidirectional sheet**
   - Standard size: 355 mm x 255 mm (14 in x 10 in)
   - In multiples of 0.125mm(0.005 in) single ply prepreg

5. **Quasi Isotropic Sheet**
   - Custom lay-ups available.
   - Typically in lay-up configuration [+45°/90°/-45°/0°]s repeated.
   - (To obtain flat sheet lay-up configuration must be symmetrical and balanced.)
   - Standard sizes: 405 mm x 405 mm (16 in x 16 in)
   - (Other sizes will be cut from and priced as standard sizes).

<table>
<thead>
<tr>
<th>Thicknesses</th>
<th>8 ply</th>
<th>1 mm</th>
<th>(0.040 in)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>16 ply</td>
<td>2 mm</td>
<td>(0.080 in)</td>
</tr>
<tr>
<td></td>
<td>24 ply</td>
<td>3 mm</td>
<td>(0.120 in)</td>
</tr>
<tr>
<td></td>
<td>32 ply</td>
<td>4 mm</td>
<td>(0.160 in)</td>
</tr>
<tr>
<td></td>
<td>40 ply</td>
<td>5 mm</td>
<td>(0.200 in)</td>
</tr>
</tbody>
</table>
New forms of Aromatic Polymer Composites are continuously under development. Please check with your nearest Sales Office.

The following new products are at an advanced stage of development.

(a) Larger sized APC-2 sheets up to 2000 mm x 1200 mm (84 in x 48 in).

(b) Braided tubes of APC-2 made from single tow prepreg up to 200 mm (8 in) diameter or slit to roll forming of stiffners etc. These braided structures can include constructions with fibre orientations incorporating ± 45° as well as 0° and 90° tapes.

(c) Continuous unidirectional S-glass/PEEK composite, with good dielectric properties, higher strain to failure and better damage tolerance. Limited development quantities soon available.

(d) APC-2: — Wide unidirectional tape — continuously seam welded up to 60” wide.

The illustration is a magnified section cut through consolidated quasi isotropic sheet and illustrates the complete, void free impregnation and fibre wetting typical of APC-2 materials.

All values quoted for properties of Fiberite's Aromatic Polymer Composites are results of tests on representative samples and do not constitute a specification.

Information contained in this publication (and otherwise supplied to users) is based on our general experience and is given in good faith, but we are unable to accept responsibility in respect of factors which are outside our knowledge or control. Freedom under patents, copyright and registered designs cannot be assumed.

• PEEK is an ICI product sold under the trade mark Victrex®

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SHEET PRODUCTION FROM PREPREG

Sheet production involves stacking layers of APC-2: PEEK/carbon fibre, prepreg between press plates, heating to 380°C(720°F) under low pressure, applying a consolidating pressure, and cooling rapidly. This will ensure that the best properties are attained.

CONTROL OF CRYSTALLINITY AND MORPHOLOGY

In order to optimise the performance of APC-2 it is necessary to control the crystallinity and morphology of the PEEK polymer. This is exercised by controlling the rate at which the composite is permitted to cool down from the molten state at 380°C-400°C(720°F-750°F) to a temperature of 150°C(300°F).

It is recommended that the rate of this cool down is in the range 10°C-70°C/minute (18°F-126°F/minute). There is little variation in crystallinity in this range and optimum composite properties result. If APC-2 is cooled at rates less than 10°C/minute (18°F/minute), there is an increase in crystallinity which will result in some reduction in composite toughness. At cooling rates in excess of 70°C/minute (126°F/minute), the polymers spherulitic growth will not achieve completion and the optimum level of crystallinity will not be reached. This may cause some reduction in stiffness and resistance to hostile solvents. However this state can be corrected by subsequently annealing the composite at between 200°C-300°C (390°F-570°F) for 20 minutes to achieve the optimum level of crystallinity and performance.

DETAILS OF PROCEDURE

The laying up of prepreg prior to pressing is carried out at room temperature and requires careful control to obtain good results. The following points should be taken into account.

1. The lay up must be balanced and symmetrical about the neutral axis (or centre line) of the sheet to avoid the bowing due to differential thermal contraction on cooling that occurs with APC-2 as with other composite materials.

2. Lay up should take place under clean room conditions.

3. Where several pieces of prepreg are required to form one layer, their edges should be straight and closely butted (maximum gap 0.5 mm 0.02 in) with no overlap. The join between these pieces should then be seam welded using a soldering iron to melt the PEEK resin, applying light hand pressure to form the weld.

4. The individual plies can then be assembled into a prepreg stack of up to 8 plies. Each ply should be tacked to the one below it by a series of welds around the periphery of the stack. Any laminate of greater than 8 plies should be made from a convenient number of individual stacks assembled together to form the final pack.

A soldering iron with a flat elliptical bit approximately 25 mm² in area and a temperature of 400°C—500°C(750°F—930°F) and a power rating of approximately 250 watts is adequate for all the welding assembly work.

It may be found advantageous to press in a 'picture frame'. This will help to stop the flow of material near the edges of the panel so that thickness and fibre direction are better controlled. In this case a stainless steel frame should be used with a border width of approximately 50 mm(2 in). The thickness of the frame should be about 0.254 — 0.380 mm(0.010 — 0.015 in) thinner than the thickness of the finished plaques.

The inner dimensions of the picture frame should be 3 mm(0.12 in) larger than the desired panel size for easy removal.

5. The laid up stack of prepreg should be placed between sheets of 0.10—0.15 mm(0.004—0.006 in) high temper aluminium foil pretreated with Freekote® FRP mould release agent sprayed on as recommended by the manufacturer.

6. The whole sandwich should then be placed between mirror finish press plates and put into a hot platen...
the platen should not be greater than 390°C (730°F) and the coldest spot must not be less than 370°C (700°F). Contact pressure of only 0.5 MPa (70 psi) should be applied at this stage, sufficient to ensure good heat transfer without stressing the fibres before the PEEK matrix is soft enough to be compliant. PEEK melts at 343°C (650°F).

7 Heating times required depend upon thickness and approximately 5 minutes per 8 plies of prepreg is usually sufficient to achieve a thermal equilibrium at 380°C (720°F), subject to an overall maximum of 30 minutes.

8 When thermal equilibrium at 380°C (720°F) has been achieved, a consolidation pressure of 1.4 MPa (200 psi) should be applied for 5 minutes.

9 Post consolidation cooling should preferably be rapid: 380°C (720°F) down to 200°C (390°F) in less than 5 minutes. This is best achieved by transferring the sandwich from the hot press to a ‘cool’ press operating at about 190°C (370°F), and applying a consolidation pressure of 2 MPa (300 psi) immediately. Transfer from hot to ‘cool’ press should be achieved in less than 20 seconds and the laminate may be removed from the cool press after approximately 5 mins.

10 Other constructions such as woven prepreg can be pressed into sheet using the same technique.

11 Unidirectional fibre sheet must be manufactured in a slightly different way to prevent excessive transverse fibre movement during pressing. The laid up stack requires no welding but is laid directly into a matched metal mould between aluminium foil treated with Frekote FRP. If the prepreg is of insufficient width to cover the mould cavity, joins should be staggered from layer to layer. The press cycle is as described in 5—8 above, with the exception that the heating and cooling times should be approximately doubled to account for the thermal inertia of the matched metal mould.

12 Inevitably there are differences between presses which may result in some variation. If difficulties are experienced, such as poor surface finish, the pressure in steps 8 and 9 should be increased in stages of up to 0.3 MPa (50 psi).

13 It is important that the pressure and glazing plates are flat and parallel. Depressions as small as 0.7 microns (0.002 in) can adversely affect the surface quality of the pressed sheet.

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Data Sheet 2

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Telex: 8529202 TRIB
APC-2: PEEK®/carbon fibre composite is an advanced structural composite of a proprietary high strength, unidirectional continuous carbon fibre in a PEEK matrix.

**PHYSICAL PROPERTIES**

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Method</th>
<th>Temperature</th>
<th>Density</th>
<th>COEFFICIENT OF THERMAL EXPANSION</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td></td>
<td></td>
<td>1.6 g/cm³</td>
<td>0.5 x 10⁻⁶°C C</td>
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<tr>
<td>Carbon fibre volume fraction</td>
<td></td>
<td></td>
<td>(0.058 lb/in³)</td>
<td>(0.28 x 10⁻⁶°F)</td>
</tr>
<tr>
<td>Carbon fibre weight fraction</td>
<td></td>
<td></td>
<td>61%</td>
<td>1.0 x 10⁻⁶°C C</td>
</tr>
<tr>
<td>Carbon fibre areal weight</td>
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<td></td>
<td>68%</td>
<td>(0.56 x 10⁻⁶°F)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>145 g/m³</td>
<td>0.5 x 10⁻⁶°C C</td>
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</tbody>
</table>

**MECHANICAL PROPERTIES**

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Method</th>
<th>Temperature</th>
<th>Tensile strength</th>
<th>Tensile modulus</th>
<th>Tensile strain to failure</th>
<th>Flexural modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>0° Tensile strength</td>
<td>ASTM D-3039</td>
<td>23°C (73°F)</td>
<td>2130 MPa</td>
<td>134 GPa</td>
<td>1.45 %</td>
<td>121 GPa</td>
</tr>
<tr>
<td>Tensile modulus</td>
<td></td>
<td></td>
<td>1100 MPa</td>
<td>160 ksi</td>
<td>273 ksi</td>
<td></td>
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<tr>
<td>Tensile strain to failure</td>
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<td></td>
<td>1880 MPa</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Compressive strength</td>
<td>IITRI Test</td>
<td>23°C (73°F)</td>
<td>80 MPa</td>
<td>11.6 ksi</td>
<td>1.0 %</td>
<td></td>
</tr>
<tr>
<td>Flexural strength</td>
<td>ASTM D-790</td>
<td>23°C (73°F)</td>
<td>8.9 GPa</td>
<td>1.29 mpi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flexural modulus</td>
<td>Span-to-depth ratio 60:1</td>
<td></td>
<td>8.9 GPa</td>
<td>1.29 mpi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>90° Tensile strength</td>
<td>ASTM D-3039</td>
<td>23°C (73°F)</td>
<td>80 MPa</td>
<td>11.6 ksi</td>
<td>1.0 %</td>
<td></td>
</tr>
<tr>
<td>Tensile modulus</td>
<td></td>
<td></td>
<td>8.9 GPa</td>
<td>1.29 mpi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tensile strain to failure</td>
<td></td>
<td></td>
<td>8.9 GPa</td>
<td>1.29 mpi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flexural strength</td>
<td>ASTM D-790</td>
<td>23°C (73°F)</td>
<td>137 MPa</td>
<td>19.9 ksi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flexural modulus</td>
<td>Span-to-depth ratio 25:1</td>
<td></td>
<td>8.9 GPa</td>
<td>1.29 mpi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>± 45° Tensile strength</td>
<td>ASTM D-3518</td>
<td>-73°C (-99°F)</td>
<td>267 MPa</td>
<td>38.7 ksi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tensile modulus</td>
<td>-73°C (-99°F)</td>
<td></td>
<td>21.9 GPa</td>
<td>3.18 mpi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tensile strain to failure</td>
<td>-73°C (-99°F)</td>
<td></td>
<td>19.9%</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
In-Plane shear modulus, $G_{12}$

<table>
<thead>
<tr>
<th>Open-Hole tensile strength</th>
<th>Specimen width</th>
<th>23°C(73°F)</th>
<th>5.1 GPa</th>
<th>0.74 msi</th>
</tr>
</thead>
<tbody>
<tr>
<td>$[-45/0/+45/90]_s$</td>
<td>38mm (1.50in)</td>
<td>23°C(73°F)</td>
<td>429 MPa</td>
<td>62 ksi</td>
</tr>
<tr>
<td>$[(+45/90/-45/0)]_s$</td>
<td>6.35mm (0.250in.)</td>
<td>23°C(73°F)</td>
<td>843 MPa</td>
<td>122 ksi</td>
</tr>
<tr>
<td>Short beam shear strength</td>
<td>ASTM D-2344</td>
<td>23°C(73°F)</td>
<td>105 MPa°</td>
<td>15.2 ksi°</td>
</tr>
</tbody>
</table>

° Specimens do not fail in shear.

**MODE I INTERLAMINAR FRACTURE TOUGHNESS ($G_{IC}$)**

| Test method | Temperature | Cleavage Mode Cleavage Mode Ductile Mode Ductile Mode |
|-------------|-------------|----------------|----------------|----------------|
| Straight-sided double cantilever beam test. | 23°C(73°F) | 1.5 kJ/m² | 2.0 kJ/m² | 8.5 in² | 11.3 in² |

Data reduction by Area Method

**FIGURE I DAMAGE TOLERANCE**

Residual compressive strain to failure vs applied impact energy/unit thickness

**DAMAGE TOLERANCE**

Residual compressive strain to failure vs applied impact energy/unit thickness
DAMAGE TOLERANCE

Residual compressive strength vs applied impact energy/unit thickness.

FIGURE II DAMAGE TOLERANCE

FIGURE III INTERPOLATED CREEP DATA FOR ±45° LAY-UP AT 23°C (73°F)

±45° CREEP DATA

ORIGINAL PAGE IS OF POOR QUALITY
INSTRUMENTED FALLING WEIGHT IMPACT DATA VERSUS TEMPERATURE

(75mm x 75mm) plaques, 50mm diameter support, 12.5mm impactor nose, impact velocity 5m/s


All values quoted for properties of Fiberite’s Aromatic Polymer Composites are results of tests on representative samples and do not constitute a specification.

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Data Sheet 3a

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This data is from initial testing of a development material and should not be used as design data.

<table>
<thead>
<tr>
<th>PHYSICAL PROPERTIES</th>
<th>Carbon fibre areal weight</th>
<th>150 g/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon fibre volume fraction</td>
<td>61%</td>
<td></td>
</tr>
<tr>
<td>Carbon fibre weight fraction</td>
<td>68%</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>MECHANICAL PROPERTIES</th>
<th>Property</th>
<th>Test Method</th>
<th>Temperature</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0°</td>
<td></td>
<td>23°C (73°F)</td>
<td></td>
</tr>
<tr>
<td>Tensile strength</td>
<td>ASTM D-3039</td>
<td></td>
<td>2891 MPa</td>
<td>419 ksi</td>
</tr>
<tr>
<td>Tensile modulus</td>
<td></td>
<td></td>
<td>169 GPa</td>
<td>24.5 ksi</td>
</tr>
<tr>
<td>Tensile strain to failure</td>
<td>IITRI Test</td>
<td></td>
<td>1.60 %</td>
<td></td>
</tr>
<tr>
<td>Compressive strength</td>
<td>ASTM D-790</td>
<td></td>
<td>1139 MPa</td>
<td>165 ksi</td>
</tr>
<tr>
<td>Flexural strength</td>
<td>Span-to-depth ratio 60:1</td>
<td></td>
<td>2040 MPa</td>
<td>296 ksi</td>
</tr>
<tr>
<td>Flexural modulus</td>
<td></td>
<td></td>
<td>148 GPa</td>
<td>21.5 msi</td>
</tr>
<tr>
<td></td>
<td>90°</td>
<td></td>
<td>23°C (73°F)</td>
<td></td>
</tr>
<tr>
<td>Tensile strength</td>
<td>ASTM D-3039</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tensile modulus</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tensile strain to failure</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flexural strength</td>
<td>ASTM D-790</td>
<td></td>
<td>157 MPa</td>
<td>22.8 ksi</td>
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<tr>
<td>Flexural modulus</td>
<td>Span-to-depth ratio 25:1</td>
<td></td>
<td>9.3 GPa</td>
<td>1.35 msi</td>
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<tr>
<td></td>
<td>Short beam shear strength</td>
<td>ASTM D-2344</td>
<td>23°C (73°F)</td>
<td>110 MPa°</td>
</tr>
<tr>
<td></td>
<td>Specimens do not fail in shear.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
### MODE I INTERLAMINAR FRACTURE TOUGHNESS (G\textsubscript{IC})

<table>
<thead>
<tr>
<th>Test method</th>
<th>Temperature</th>
<th>Cleavage Mode</th>
<th>Ductile Mode</th>
<th>Cleavage Mode</th>
<th>Ductile Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straight-sided double cantilever beam test. Data reduction by Area Method</td>
<td>23°C(73°F)</td>
<td>2.5</td>
<td>3.3</td>
<td>14.3</td>
<td>18.8</td>
</tr>
</tbody>
</table>

### DAMAGE TOLERANCE

<table>
<thead>
<tr>
<th>Impact Energy</th>
<th>Residual compressive strength</th>
<th>Residual strain to failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>in lb/in</td>
<td>MPa (ksi)</td>
<td>%</td>
</tr>
<tr>
<td>1000</td>
<td>4.45</td>
<td></td>
</tr>
<tr>
<td>1500</td>
<td>6.68</td>
<td></td>
</tr>
<tr>
<td>2000</td>
<td>8.90</td>
<td></td>
</tr>
</tbody>
</table>

All values quoted for properties of Fiberite's Aromatic Polymer Composites are results of tests on representative samples and do not constitute a specification.

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Data Sheet 3b
SUMMARY

APC-2: PEEK*/carbon fibre composite has very good resistance to burning. It is difficult to ignite and exhibits low flame propagation rates by standard laboratory tests. The smoke emission from APC-2 during a fire is amongst the lowest of all plastics.

Sheets of APC-2 material show significantly longer flame penetration times than aluminium sheets at temperatures above 1000°C (1830°F).

Values quoted for properties are the result of limited tests on representative samples. These values do not constitute design data. Additional and more comprehensive data will be published in due course after further testing.

IGNITABILITY*

A commonly used test method for determining ignitability is the Oxygen Index Test (ASTM D2863, ISO/IS4S89). This measures the minimum percentage of oxygen necessary in an atmosphere to support combustion. APC-2 has an Oxygen Index of 67%. This value is high by comparison with other composites.

The difficult ignitability of PEEK resin alone has also been demonstrated in other tests. Self-ignition and flash-ignition temperatures were determined using the Setchkin hot-air ignition furnace (ASTM D1929-75). Results in the range 575°—600°C (1070°—1100°F) were obtained in these tests.

FLAME PROPAGATION*

The Underwriters Laboratories Standard 94 is used to evaluate the flammability of materials. APC-2 readily achieves the lowest flammability rating of V-0 when tested in accordance with UL94, Vertical Burning Test.

The flame spread characteristics have been measured in accordance with FAR 23.853. The results obtained from this test carried out on APC-2 are:

<table>
<thead>
<tr>
<th>Mode</th>
<th>Burn Rate</th>
<th>Average Burn Length</th>
<th>Average Flaming Time after Removal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Horizontal mode</td>
<td>0</td>
<td>14 mm (0.6 in)</td>
<td>0 secs</td>
</tr>
<tr>
<td>Vertical mode</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

SMOKE*

APC-2 is one of the lowest smoke emitting materials evaluated in the NBS Smoke Chamber. This test (ASTM E662, BS 6401) provides a measure of the obscuration of visible light by smoke in units of specific optical density. The NBS smoke test is normally carried out in two modes; flaming and non-flaming. Under the conditions of the flaming mode, the maximum specific optical density obtained for APC-2 was:

\[ D_m(\text{corr.}) = 1 \]

FIRE RESISTANCE*

Many components of an aircraft structure are required to be fire-resistant. As defined by the ISO Standard DIS2685, this means that structural material should have a flame penetration time at 1100 ± 50°C (2000 ± 120°F) which is at least equal to aluminium alloy sheet. Experiments have been carried out to determine the penetration times of APC-2 versus aluminium alloy sheet to a propane-air burner at 1050°C (1900°F). (Fig. 1).

Figure I
APC-2 PEEK/CF COMPOSITE FAA FLAME PENETRATION TEST

28 in. Square (700 mm)
Front Surface 2000°F (1090°C)
Rear Surface 700°F (370°C)
Propane Flame 2000°F (1090°F)
0.080 in. thick (2 mm)
Burn through time 15—18 mins.
sheets 3 mm (0.120 in) thick show significant advantages over aluminium sheets of the same thickness:

(i) APC-2 sheets have much lower thermal conductivity than aluminium; it takes 3 times longer for the back of the APC-2 sheet to glow red compared with aluminium sheet. With the front face of APC-2 at 1100°C (2000°F) the rear face temperature was 275°C, (530°F).

(ii) APC-2 sheets resist flame penetration much longer.

(iii) APC-2 sheets retain their mechanical properties in the high temperature flame whilst the aluminium sheet softens at 600°C, (1100°F) and is rapidly reduced to a weak oxide film at the higher temperatures (1000°C, 1830°F) used in these tests.

TYPICAL FIRE PROPERTIES OF APC-2*

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>Units</th>
<th>Results</th>
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</thead>
<tbody>
<tr>
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<td></td>
<td>mm (in)</td>
<td>3.1 (0.120)</td>
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<tr>
<td>Density</td>
<td></td>
<td>kg m⁻³, lb ft m⁻³</td>
<td>1657 104</td>
</tr>
<tr>
<td>Ignitability:-</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oxygen Index</td>
<td>ASTM D2863, ISO/IS4589</td>
<td>%</td>
<td>67</td>
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<tr>
<td>Self-ignition temperature (PEEK resin)</td>
<td>ASTM D1929</td>
<td>°C, (°F)</td>
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<tr>
<td>Flash-ignition temperature (PEEK resin)</td>
<td>ASTM D1929</td>
<td>°C, (°F)</td>
<td>575 (1070)</td>
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<tr>
<td>Flammability:-</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>UL 94 Vertical</td>
<td></td>
<td>Minutes</td>
<td>V-0</td>
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<tr>
<td>Fire resistance:-</td>
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<td></td>
</tr>
<tr>
<td>Flame penetration time at 1100°C</td>
<td>ICI propane torch</td>
<td>Minutes</td>
<td>15</td>
</tr>
<tr>
<td>Smoke:-</td>
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<td></td>
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<tr>
<td>NBS Smoke chamber</td>
<td>ASTM E662</td>
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<td>1</td>
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<tr>
<td>Optical Density Dₘ (corr) in flaming mode</td>
<td></td>
<td></td>
<td></td>
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</tbody>
</table>

* Tests were carried out under ISO standard conditions, but as yet not by ISO appointed test laboratories.

All values quoted for properties of Fiberite's Aromatic Polymer Composites are results of tests on representative samples and do not constitute a specification.

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Data Sheet 4
APC-2 is supplied as pre-impregnated tape, tow on consolidated sheet. Based on PEEK, reinforced with carbon fibres it is thermoplastic in nature and fabrication processes involve the following steps:

— Heating to soften and melt the PEEK matrix.
— Pressure to provide shape and consolidation.
— Cooling to solidify.

As a true thermoplastic material considerations of 'shelf life' and 'cure time' do not apply to APC-2. It is unaffected by storage conditions and as no chemical changes occur during heating or fabrication APC-2 composites can be reheated and softened a number of times without detrimental effect.

Fibre areal weight and fibre positioning can be very accurately controlled as there is no “bleed out” of resin and little ‘fibre wash’ during processing.

APC-2 process times can be quite rapid with heating to the shaping temperature of 400°C(750°F) taking minutes and shaping and consolidation taking a few seconds at only moderate pressures. This is followed by a rapid cooling cycle of 5 minutes or so. A total cycle time of minutes can be achieved, compared with the hours often taken for thermosetting systems.

CONTROL OF CRYSTALLINITY AND MORPHOLOGY

In order to optimise the performance of APC-2 it is necessary to control the crystallinity and morphology of the PEEK polymer. This is exercised by controlling the rate at which the composite is permitted to cool down from the molten state at 380°C-400°C(720°F-750°F) to a temperature of 150°C(300°F).

It is recommended that the rate of this cool down is in the range 10°C-700°C/minute (18°F-1260°F/minute). There is little variation in crystallinity in this range and optimum composite properties result. If APC-2 is cooled at rates less than 10°C/minute (18°F/minute), there is an increase in crystallinity which will result in some reduction in composite toughness. At cooling rates in excess of 700°C/minute (1260°F/minute), the polymers spherulitic growth will not achieve completion and the optimum level of crystallinity will not be reached. This may cause some reduction in stiffness and resistance to hostile solvents. However this state can be corrected by subsequently annealing the composite at between 200°C-300°C (390°F-570°F) for 20 minutes to achieve the optimum level of crystallinity and performance.

Manufacturing processes for APC-2 fall into two main categories.

1 Processes using preconsolidated sheet feedstock such as:-
   - Hydro rubber forming
   - Press forming
   - Roll forming
   - Diaphragm shaping
   - Matched die moulding.

2 Processes using pre-impregnated tape and tow feedstock such as:-
   - Filament winding
   - Tape laying
   - Braiding
   - Pultrusion
   - Autoclave and vacuum bag moulding
   - Diaphragm moulding.
SHAPING FROM PRECONSOLIDATED SHEET

There are some general considerations that apply to all APC-2 sheet shaping processes.

Heating

APC-2 is a good 'black body absorber' and can be heated quite rapidly in a number of ways. The most effective method is radiant heating using quartz infra-red heaters. Ideally, heating should be from both sides simultaneously using 75 kW/m² (7.5 kW/ft²) energy density. To effect this the APC-2 sheet can be supported on a fine wire shelf midway between 2 banks of quartz infra-red heaters placed approximately 25 mm (12 in) from the material surface. With such an arrangement heating takes only a minute or so (see Fig 1).

Shaping has to take place whilst the PEEK matrix is above its melting point of 343°C (650°F) and the whole thickness of the sheet should ideally be heated to approximately 400°C (750°F).

PEEK exhibits a super-cooling effect and some shaping is possible for a very short time after APC-2 has fallen below the PEEK melting point by 20°C (70°F) or so.

PEEK is an excellent hot melt adhesive and will bond firmly to itself and to other materials including metals (moulds etc) under only contact pressure, if both surfaces are at or above the melting point of 343°C (650°F). An optimum tool temperature is 180°C-200°C (350°F-390°F) to give immediate solidification, optimum crystallisation, and to minimise bonding to the tool.

Shaping Mechanisms

As the continuous carbon fibre reinforcement is, to all intents and purposes, inextensible, shaping processes have to be capable of accommodating fibre movement. One of the main mechanisms in sheet shaping is interply slippage which can occur above the PEEK melting point (Fig 2).

Hydroforming and Rubber Block Forming

Hydroforming and rubber block forming are similar processes in which sheet material is formed to the shape of a single sided mould by the application of pressure, either by a hydraulically pressurised rubber diaphragm (hydroforming) or by a mechanically pressurised and deformed rubber block.

Hydroforming is a well-known metal forming process, used widely in the aircraft industry, and APC-2 has been successfully shaped using pre-heated blanks and a standard process, 3 variants of which are shown in Fig 2.

FIGURE 1: TYPICAL APC2 HEATING CURVES vs BLANK THICKNESS

![Typical APC2 Heating Curves vs Blank Thickness](image)
This process has advantages for forming APC-2 composite as it can accommodate the interply slip required to avoid fibre wrinkling in areas being stretched, and allows some thickening by fibre 'bunching' in those areas, such as flanges, being compressed. It also applies a more gradual folding and feeding action during shaping than can be achieved in matched die moulding. The compliant pressurising medium applies a relatively even pressure over the surface area of the moulding minimising fibre damage in high spots and sharp corners.

Infra-red heating has proved to be useful for heating the APC-2 blanks to forming temperatures of 380°C(720°F), but any means of heating capable of reaching this temperature could be used. It is important that the blank remains adequately hot until the moment of forming, so a rapid transfer of blank from oven to mould is essential; typically less than 10-15 seconds.

Hydroforming APC-2 uses the same tooling as for conventional sheet metal forming and moulds made from aluminium and aluminium filled epoxy have been used.

Cold aluminium alloy tools can be used, although depending on the nature of the formed item and the other process details, the use of moulds heated to 80°C-150°C(176°F-300°F) may be advantageous in reducing premature chilling of the APC-2 blank before forming.

It is important that the mould has a well polished surface to allow blank slippage during forming and the use of suitably positioned vent holes of .75 mm(.030 in) diameter is recommended, with adequate channels leading away from the vents. This will ensure the evacuation of all the air trapped between the blank and the mould and will reduce the occurrence of surface blisters caused by entrapped air.

Roll Forming

Roll forming is a widely used metal-forming process used to produce long structural sections such as 'top-hat' or 'Z' stiffeners. APC-2 has been successfully roll formed at production speeds of up to 15 m/min(50 ft/min) using a standard cold roll process with pre-heating of the APC-2 blank.

The APC-2 strip feedstock must be heated to 400°C(750°F) immediately prior to forming, and this can be achieved by the use of conveyors using infra-red elements, or hot air heating. Tunnel length will be dictated by strip thickness, rolling speed and residence time.

Selected masking of the APC-2 blank is helpful when using IR heating to produce cold (and hence stiff) areas to aid alignment and feeding to the forming rolls.

A minimum of 5 rolls is recommended (4 to form and 1 to repeat and cool the full section) at a spacing of 150 mm(6 in). It is desirable to have the facility to drive top and bottom rolls separately (to restrict unwanted inter-layer slip within the hot sheet). Alignment and support devices are necessary for the pre-heated blank before the first stand and also between the first and second stands.

No roll lubrication or surface release agent is required during roll forming as a smooth polished cold roll will not encourage adhesion. The setting of the gaps between the mating faces of the rolls is of critical importance and is dependent on the blank composition and the section formed. Typical starting values for the roll gap are 0.075 mm(0.003 in) less than the average thickness of the cold blank.

Rolled sections have been circled to a 4 m(12 ft) diameter by the use of a final stripper plate.
**Pultrusion**

Because of its thermoplastic nature it is possible to reform APC-2 prepreg tape into other cross-sections by pultruding through a hot die. Die design needs to allow for heating the APC-2 to 400°C (750°F) whilst at the same time gradually changing the cross section as the tape proceeds through the die. As the carbon fibre is incompressible and there is little excess resin to bleed out, there is no compliance in APC-2. Some compliance may therefore have to be built into the die, in order to maintain a constant pressure.

To avoid die wipe causing fibre distortion the carbon fibre in the surface layer should run in the direction of pultrusion with the ±45° and 90° layers in the core. Braided APC-2 prepreg tow should provide a good basis for the core.

**FIGURE 3: DIAPHRAGM FORMING PRESS**

**Diaphragm Forming**

This process involves simultaneously heating, maintaining consolidation and shaping APC-2 sheet between 2 plastically deformable diaphragms, (Fig. 3). One proprietary process uses Supral® superplastic aluminium alloy as the diaphragm. Either preconsolidated sheet or unconsolidated ply lay up can be formed by this process (see Data Sheet 2).

Positive pressure is applied to the outside of the diaphragms (or vacuum between) to consolidate the plies and maintain consolidation whilst the whole sandwich is heated to 400°C (750°F) and moulded to the required shape, (Fig. 4). The diaphragms help to prevent splitting or wrinkling of the APC-2 during shaping, by providing elastic, tear resistant surfaces.

**FIGURE 4: DIAPHRAGM FORMING PROCESS**
This process can be applied to quite large area mouldings, e.g. 4 sq m (40 sq ft) and with a reasonable degree of double curvature. Ply 'drop off', change in section thickness and provision of stiffening ribs can be accommodated in this process.

**Autooclave and Vacuum Bag Moulding**

APC-2 prepreg tape requires only contact pressure for consolidation. Therefore if pressure can be applied in a constant compliant manner over the whole surface area, relatively low pressures can be used. Unlike rigid platten pressing (described in Data Sheet 2) where higher pressures are required to press out the high spots and fill in the troughs to mould a surface, pressures as low as 0.06 MPa (10 psi) can be used for consolidation, using temperature resistant compliant membranes.

The standard autoclaving and vacuum bagging techniques, well developed for production of thermoset composite components, can easily be adapted for APC-2.

The principal change is in the use of higher temperature bagging and sealing materials capable of operating at 400°C (750°F). Suitable bagging materials are Du Pont's KAPTON® Grade 200H film or Airtech's VACALLOY® Grade DD foil. A suitable vacuum sealant tape is A800®, also from Airtech.

Temperature must reach 400°C (750°F) during the autoclave cycle and pressure should be maintained down to 200°C (390°F) to ensure good consolidation.

**Tape Laying**

Large area, planar parts can be produced from APC-2 by a tape laying technique which involves ironing consecutive layers of prepreg tape onto a mould surface. This is a continuous process in which APC-2 prepreg tape is heated to 400°C (750°F) as it is being ironed or rolled down on to a mould surface, consolidation pressure being maintained for a few seconds whilst the tape cools and solidifies (see Fig. 5). Subsequent plies are welded to previously consolidated layers by further passes of the tape laying head. Tape laying speeds of around 2.5 m/min (100 inches/min) can be achieved with void free consolidation. Both the prepreg tape surface and the surface of the previous layer need to be above the melting point of PEEK to obtain effective welding and consolidation, but the heat capacity of the thin tape being laid is small compared to the mould and previous layers and cooling to solidification occurs rapidly. Tape laying allows the designer to predetermine the fibre direction within a moulding to optimise mechanical performance in service. Some contouring, local thickening and ply drop-off can be achieved by this process.

Proprietary tape laying machines are available for room temperature lay-up of epoxy thermosetting prepreg tape, and work to modify such machines for high temperature laying of thermoplastics is currently under way.

---

**FIGURE 5: CONTINUOUS TAPE PLACEMENT AND CONSOLIDATION WITH IN-LINE 100% QUALITY ASSURANCE**
Filament winding
APC-2 can be supplied in 3 and 6 mm (0.120 and 0.240 in) wide tow in lengths of 1000 m (3000 ft) for filament winding. The tow is heated to softening point (400°C—750°F) immediately prior to winding on to a mandrel. Consolidation pressure can be applied through filament winding tension, or pressure rollers (see Fig. 6). Again both surfaces need to be above the melting point of PEEK at the point of contact, but cooling to solidity is rapid due to the relatively low thermal capacity of the heated area. Proprietary machinery is being developed from existing filament winding equipment.

Braiding and Weaving
APC-2 prepreg tow 3 mm and 6 mm (0.120 and 0.240 in) wide can be used for braiding and weaving. Because the tow is a flat, stiff tape and cannot be bent back on itself without snapping, special braiding and weaving techniques have had to be developed.

Though the stiffness of APC-2 prepreg tow tends to produce a rigid product, the braided and woven forms exhibit a limited degree of "drape" which should assist in shaping double curvatures etc.

Braided and woven forms can either be preconsolidated by pressing into sheet or consolidated during manufacturing fabrication. These forms should prove advantageous for pultrusion, roll forming stiffeners etc.

Braided and woven forms of APC-2 will shortly be available in development quantities.

Adhesive bonding
Thermoset adhesives
APC-2 is renowned for its excellent environmental and chemical resistance but this causes a problem when it comes to trying to bond with adhesives, as it is difficult for the traditional aerospace thermosetting adhesives to form a chemical bond with the APC surface.

Whilst it is possible to bond APC-2 with traditional epoxy adhesives and obtain reasonable lap shear strengths the peel strength is generally low. However bonding can be improved by mechanically abrading the APC-2 surface or etching away the surface ‘Victrex’ PEEK with chromic acid or plasma etching to expose the carbon fibre surface.

The following thermoset adhesives have been found to give the best results:

- Ciba Geigy "Redux" 319A
- "Araldite" 2007
- 3M EC 3430

FIGURE 6: THERMOPLASTIC FILAMENT WINDING WITH CONTINUOUS CONSOLIDATION
Fusion bonding

Thermoplastic adhesives

Fortunately PEEK is also a superb hot melt adhesive and fusion bonding techniques work well.

APC-2 can be bonded to itself (and to other materials including metals) simply by heating the two surfaces to be mated to 400°C(750°F) and applying contact pressure whilst cooling to 200°C(390°F) or below. This requires of course that the two surfaces mate closely together to provide overall contact. The inclusion of a thin PEEK film (200 micron — 0.008 in) between the surfaces to give a degree of resin richness may help to mate slightly uneven surfaces.

Heating the APC-2 surface can be carried out in a number of ways:-

1. High intensity infra-red radiant heating to melt the surfaces of the areas to be bonded has been found satisfactory. This needs to be done rapidly to avoid delaminating subsurface plies. The mating surfaces must be brought together before the temperature falls below the melting point of PEEK.

2. Prepreg tape can be used as an adhesive layer between two APC-2 parts. The interface is heated by applying an electric current through the carbon fibres of the prepreg interlayer. The filaments act as resistance heaters and melt the PEEK matrix in situ, welding the two parts together.

   The prepreg tape needs to extend beyond the bonding area. The exposed ends should be treated to remove the PEEK matrix which otherwise acts as an insulant. This can be done by burning in an oxidising flame or by chemical etching in chromic acid.

   Electrodes should be fixed to the exposed carbon fibre. A current of approximately 15 amps/in of prepreg width at 30V DV has been found to provide sufficient heating effect in a minute or so. Contact pressure should be maintained during cooling.

3. Induction heating of a fine wire gauze at the interface can be used in a similar way. In this case a thin film of PEEK (200 microns (0.008 in)) is placed either side of the wire gauze and that sandwich is then placed between the APC-2 parts to be bonded. The whole assembly is placed in an induction field when the gauze heats up and fuses the PEEK film at the interface. Moderate pressure is required to obtain 'strike through' of the gauze and a void free bond line.

4. Ultra sonic heating can be used both to spot weld-APC-2 as well as to weld it continuously. It is necessary that the correct horn geometry is used which suits the configuration of the weld to be made.

5. Hot plate welding, often used to join large automotive components, can be used to join APC-2 structures.

Lap Shear joints have been produced by all the above mentioned thermo-bonding processes. Mechanical testing has demonstrated that the strength of the bonds made closely approaches the shear strength of the PEEK matrix itself.

MACHINING

APC-2 composite sheet can be machined using a wide variety of metal machining processes. However, tools need to be very sharp (usually diamond tipped), cutting speeds need to be high, using slow feed rates and a coolant to wash away swarf. Because of the composite (ie nonhomogeneous) nature of APC-2 a rough finish due to fibre pick out will occur unless the above precautions are observed.

It is better to think in terms of a high speed accurate grinding operation rather than the cutting or paring action of conventional metal machining tools.

Cutting and Sawing

1. APC-2 sheet can be cut on standard band saws and circular saws, the best results being achieved when diamond coated blades are used.

2. Water jet cutting has been successfully employed on APC-2 sheet provided satisfactory back surface support is given to prevent fibre pick out on the rear face.

3. Circular shapes can be cut by trepanning using diamond tipped tools.

Drilling and Countersinking

1. Holes can be drilled in APC-2 sheet using diamond tipped trepanning drills operating at high cutting speed and using a slow feed rate. High speed steel twist drills can be used but it is recommended to drill several holes of increasing size to achieve the final diameter. The paring action of a twist drill will tend to leave a rough surface on the inside of the hole.

2. Countersinking is best achieved by using a high speed grinding action at slow feed rates.

3. Threads can be tapped into APC-2 sheet using conventional tapping techniques. Very high bolt pull out strengths can be obtained with this fastening technique.

Guillotining and Stamping

Thin sections of APC-2 (generally up to 1 mm(0.04 in)) can be guillotined or stamped into quite intricate shapes.
All values quoted for properties of Fiberite's aromatic polymer composites are results of tests on representative samples and do not constitute a specification.

Information contained in this publication (and otherwise supplied to users) is based on our general experience and is given in good faith, but we are unable to accept responsibility in respect of factors which are outside our knowledge or control. Freedom under patents, copyright and registered designs cannot be assumed.

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Data Sheet 5

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SUMMARY

This note describes the precautions that should be taken in the interest of health and safety when handling and processing APC-2: PEEK*/carbon fibre composite when supplied as prepreg tape, prepreg tow or consolidated sheet. Its scope does not include discussion on the suitability of the material for applications, nor any precautions that may be necessary during the use in service of any product made from the material. Health and safety information is in accordance with OSHA requirements.

If further advice is needed on health and safety aspects of APC-2 it can be obtained from Fiberite Sales Offices.

Further information on APC-2 and Victrex® PEEK is published in other literature which is available from the same address.

INTRODUCTION

APC-2 is an advanced structural composite of a proprietary high strength, undirectional continuous carbon fibre (CAS 7782-42-5) in a Victrex® PEEK matrix (CAS 29568-26-2). The carbon fibres are well dispersed and thoroughly wetted in the Victrex® PEEK matrix to give a fibre content of 61% by volume and 68% by weight. Both Victrex® PEEK and carbon fibre appear on the EPA TSCA Inventory.

Victrex® PEEK is a linear polymer, which can be chemically described as a poly(aryletherketone), based on the following repeat unit:

![Figure I](image)

Carbon fibres comprise carbon in multifilament fibrous form. The individual filaments have diameters in the range 6-9 micrometers (0.23 — 0.32 x 10⁻³in) but are unlikely to present a hazard to the respiratory system beyond that associated with nuisance dusts. All carbon fibre derived products present a number of potential hazards under use. Precautionary processing practices are recommended to ensure the safe handling of these materials.

PHYSICAL DATA

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HEALTH HAZARD ASSESSMENT

General:
No toxicity information on this specific preparation is available; this health hazard assessment is based on information that is available on its components.

Dust inhalation:
Dust concentration limits of 10mg/m³ total dust, and 5mg/m³ respirable dust should be observed (Ref 1, Ref 2). In unavoidably dusty conditions, dust masks should be worn.

Eye contact:
No irritation is likely to develop following contact with human eyes. Mechanical irritation may develop from contact with dust.

Skin contact:
No irritation is likely to develop following contact with human skin. Mechanical irritation may develop from contact with dust. See also PROCESSING.

Skin absorption:
This product is not likely to be absorbed through human skin.
SPECIAL PROTECTION INFORMATION

**TLV or suggested control value:**
No TLV assigned. Minimise exposure in accordance with good hygiene practice.

**Ventilation:**
As needed to control exposures.

**Respiratory protection (specify type):**
If needed, use MSHA-NIOSH approved respirator for dusts.

**Protective clothing:**
Impervious gloves and apron to protect from thermal effects when handling molten material.

**Eye protection:**
Safety glasses with side shields.

**Other protective equipment:**
Eyewash station in work area.

STORAGE AND HANDLING

APC-2 is inert under normal storage conditions. APC-2 consolidated sheet can have sharp edges and APC-2 prepreg products can form multifilament carbon splinters. Protective gloves should therefore be worn when handling APC-2.

DUST HAZARD

There will not normally be a dust hazard with APC-2 products. However, Victrex® PEEK, carbon fibre, or composite dust may be generated through severe handling or more particularly through cutting, machining or grinding. Care should be taken to prevent accumulation or distribution of the dust. Fibre particles present in such dust may cause transient skin irritation.

**First aid procedures:**
Skin: Wash material off the skin with copious amounts of soap and water. If redness, itching or a burning sensation develops, get medical attention. For thermal burns, cool quickly with water and get medical attention. Do not peel off solidified material.

Eyes: Immediately flush with copious amounts of water for at least 15 minutes. If redness, itching or a burning sensation develops, have eyes examined and treated by medical personnel.

Ingestion: Give one or two glasses of water to drink. If gastrointestinal symptoms develop, consult medical personnel. (Never give anything by mouth to an unconscious person.)

Inhalation: Remove victim to fresh air. If cough or other respiratory symptoms develop, consult medical personnel.

ELECTRICAL PROBLEMS

Carbon fibre ‘fly’ is electrically conductive and can disturb electrical equipment installations and switchgear causing short circuiting. If dust cannot be prevented, electrical equipment should be protected by sealing to prevent the ingress of fibres or by isolating in clean air conditions. The provision of filtered ventilation maintained at a slightly positive pressure is recommended for particularly sensitive components.

PROCESSING

No particular hazards are associated with the processing of APC-2 if it is carried out according to good manufacturing practice and in accordance with our recommendation. However, molten APC-2 with its high processing temperature of 375-400°C (700-750°F) will cause severe burns and adhere strongly to the skin. Burns are the most common injury met within melt processing of thermoplastic materials and utmost care must be taken. Gloves, face shields and other suitable protective clothing should be worn when handling hot material.

NB: The interior of heated masses will remain hot for sometime. Especial care is therefore necessary when attempting to handle or dispose of such masses.

FIRE AND EXPLOSION HAZARD DATA

**Flash point (and method):** Not applicable
**Autoignition temp:** No data
**Flammable limits (STP):** Not applicable

**Extinguishing media:**
Water fog, foam, carbon dioxide, dry chemical, Halon 1211.

**Special fire fighting protective equipment:**
Self-contained breathing apparatus with full facepiece and protective clothing if involved in a fire of other materials.

**Unusual fire and explosion hazards:**
None known.

BURNING BEHAVIOUR

**Combustion products:**
Like many other materials APC-2 will burn to produce predominately carbon monoxide and carbon dioxide. Testing Victrex® PEEK to UK Ministry of Defence Test NES713 ‘Determination of the Toxicity Index of the Products of Combustion of Materials’ confirmed the presence of carbon monoxide and carbon dioxide but no hydrogen fluoride, sulphur dioxide, hydrogen cyanide or ammonia were detected.

Carbon fibre particles which may be released during incineration are easily circulated in the atmosphere representing a hazard to electrical installations as outlined in the previous section.
Conditions in a real fire cannot be predicted as many factors are involved, such as the location, the oxygen availability and the presence of other flammable materials.

CLEANING OF EQUIPMENT

Equipment is often cleaned by burning off excess polymer. During burning of APC-2, CO and CO\textsubscript{2} will be evolved and good ventilation is therefore necessary. Aluminium oxide fluid bed cleaning baths at 500-600°C (930-1100°F) have been found to be very effective in removing Victrex\textsuperscript{®} PEEK. Again good ventilation is required.

Care should be taken that no significant quantities of carbon fibre can be distributed into the environment during burning off. Carbon fibre dust is electrically conductive and can short circuit or otherwise disturb electrical equipment.

REACTIVITY DATA

Stability:
Stable under normal conditions.

Incompatibility (materials to avoid):
Dissolved by concentrated sulphuric acid.

Hazardous decomposition products:
Combustion products: Carbon dioxide, carbon monoxide.

Hazardous polymerisation:
Will not occur.

APC-2 should not be incinerated due to the possibility of generating carbon fibre 'fly' as indicated under 'combustion products' above. Collection and packaging for landfill is recommended. Hot water and detergent should be used to wash down and flush away spillage into a serviced waste water treatment facility. Discarded products are not a hazardous waste under RCRA 40 CFR 261.

SPECIAL PRECAUTIONS OR OTHER COMMENTS

None.

REFERENCES


2. Threshold Limit Values for Chemical Substances in the Work Environment 1982/84, ACGIH. (This publication is revised annually and the latest edition should be consulted).

Acknowledgement is due to Hysol Grafil Limited for carbon fibre health and safety information.
Values quoted for properties of Fiberite's Aromatic Polymer Composites APC-2 are results of tests on representative samples and do not constitute a specification.

Information contained in this publication (and otherwise supplied to users) is based on our general experience and is given in good faith, but we are unable to accept responsibility in respect of factors which are outside our knowledge or control. Freedom under patents, copyright and registered designs cannot be assumed.

*PEEK is an ICI product sold under the trade mark Victrex®

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West Germany
Tel: (02161) 58929
Fax: (02161) 570657
Telex: 8529202 TRIB
APPENDIX C

LAMINATE ANALYSES
The loading type for this laminate is NO LOADS.

The laminate contains 30 plies.

The laminate consists of 1 materials.

MATERIAL PROPERTIES, psi

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PLY K Z(K), in T(K), in THETA(K) MATERIAL(K) H, in

\[ \begin{bmatrix} (\pm 45/0)_{5} \end{bmatrix} \]

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| Ex     | = 0.1055E+08 psi |
| Ey     | = 0.3305E+07 psi |
| Gxy    | = 0.3017E+07 psi |
| NUxy   | = 0.6873       |
| NUyx   | = 0.2153       |
| ETAxy,x| = 0.0000       |
| ETAxy,y| = 0.0000       |
| ETAx,xy| = 0.0000       |
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LAMINATE ENGINEERING CONSTANTS:

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- Ey = 0.230E+07 psi
- Gxy = 0.301E+07 psi
- NUxy = 0.6873
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\[ \text{[ABBD]} \]

\[ \text{[ABBD] inverse} \]

\[ \left[ \begin{array} {ccc} 0.378E-06 & -0.263E-06 & 0.761E-25 \\ -0.263E-06 & 0.502E-05 & -0.145E-23 \\ 0.761E-25 & -0.145E-23 & 0.509E-05 \end{array} \right] \]

LAMINATE ENGINEERING CONSTANTS:

\[ \begin{align*} 
E_x &= 0.1763E+08 \text{ psi} \\
E_y &= 0.1329E+07 \text{ psi} \\
G_{xy} &= 0.1309E+07 \text{ psi} \\
N_{uxy} &= 0.6949 \\
N_{uxy} &= 0.0524 \\
E_{Axy},x &= 0.0000 \\
E_{Axy},y &= 0.0000 \\
E_{Axy},xy &= 0.0000 
\end{align*} \]
The loading type for this laminate is NO LOADS.

The laminate contains 30 plies.

The laminate consists of 1 materials.

### MATERIAL PROPERTIES, psi

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>E1</th>
<th>E2</th>
<th>G12</th>
<th>NU12</th>
<th>NU21</th>
<th>ALPHA 1</th>
<th>ALPHA 2</th>
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<tbody>
<tr>
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### PLY K Z(K), in T(K), in THETA(K) MATERIAL(K) H, in

<table>
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<th>PLY</th>
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<th>MATERIAL(K)</th>
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</table>

### LAMINATE ENGINEERING CONSTANTS:

| Ex   | 0.9788E+07 psi |
| Ey   | 0.9702E+07 psi |
| Gxy  | 0.1309E+07 psi |
| NUxy | 0.0979        |
| Vxy  | 0.0970        |
| ETAxy, x | 0.0000        |
| ETAxy, y | 0.0000        |
| ETAx, xy | 0.0000        |

\[
\begin{bmatrix}
(\pm 75/0^\circ) & (1/1)_{10}
\end{bmatrix}
\]

### [ABBD]

\[
\begin{bmatrix}
0.681E-06 & -0.667E-07 & 0.197E-24 & -0.360E-21 & 0.321E-22 & 0.173E-23 \\
-0.667E-07 & 0.687E-06 & -0.193E-25 & 0.302E-22 & -0.660E-21 & -0.877E-22 \\
0.197E-24 & -0.193E-25 & 0.509E-05 & 0.119E-23 & -0.110E-21 & -0.371E-20 \\
-0.360E-21 & 0.302E-22 & -0.119E-23 & 0.414E-03 & -0.393E-04 & -0.642E-06 \\
0.321E-22 & -0.660E-21 & -0.110E-21 & 0.439E-04 & 0.333E-03 & -0.559E-04 \\
0.173E-23 & -0.877E-22 & -0.371E-20 & 0.642E-06 & 0.559E-04 & 0.259E-02 \\
\end{bmatrix}
\]
The loading type for this laminate is NO LOADS.

The laminate contains 30 plies.

The laminate consists of 1 materials.

**MATERIAL PROPERTIES, psi**

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>E1</th>
<th>E2</th>
<th>G12</th>
<th>NU12</th>
<th>NU21</th>
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<th>ALPHA 2</th>
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<tbody>
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**PLY K** | **Z(K), in** | **T(K), in** | **THETA(K)** | **MATERIAL(K)** | **H, in** |
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\[
\begin{bmatrix}
0.144E-11 & 0.568E-13 & -0.212E-20 \\
0.568E-13 & 0.250E-11 & -0.122E-18 \\
-0.212E-20 & -0.122E-18 & 0.128E-12
\end{bmatrix}
\]

**[ABBD] inverse**

\[
\begin{bmatrix}
0.584E+05 & -0.200E-02 & -0.200E-02 \\
-0.200E-02 & 0.111E+06 & 0.111E+06 \\
-0.200E-02 & 0.111E+06 & 0.111E+06
\end{bmatrix}
\]

**LAMINATE ENGINEERING CONSTANTS:**

- \(E_x = 0.9786E+07\) psi
- \(E_y = 0.1100E+08\) psi
- \(G_{xy} = 0.7400E+06\) psi
- \(N_{uxy} = 0.0353\)
- \(N_{uyx} = 0.0397\)
- \(\eta_{xy,x} = 0.0000\)
- \(\eta_{xy,y} = 0.0000\)
- \(\eta_{x,xy} = 0.0000\)
The loading type for this laminate is NO LOADS.
The laminate contains 8 plies.
The laminate consists of 1 materials.

**MATERIAL PROPERTIES, psi**

<table>
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<tr>
<th>MATERIAL</th>
<th>E1</th>
<th>E2</th>
<th>G12</th>
<th>NU12</th>
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<td>1</td>
<td>0.194E+08</td>
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**PLY K**

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\[
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\pm 45 \end{bmatrix}
\]

\[
\begin{bmatrix}
0.246E+06 & 0.186E+06 & 0.000E+00 & -0.426E-13 & 0.142E-13 & -0.711E-14 \\
0.186E+06 & 0.246E+06 & 0.000E+00 & 0.142E-13 & -0.142E-13 & 0.000E+00 \\
0.000E+00 & 0.000E+00 & 0.200E+06 & -0.711E-14 & 0.000E+00 & 0.000E+00 \\
-0.426E-13 & 0.142E-13 & -0.711E-14 & 0.327E+02 & 0.248E+02 & 0.911E+01 \\
0.142E-13 & -0.142E-13 & 0.000E+00 & 0.248E+02 & 0.327E+02 & 0.911E+01 \\
-0.711E-14 & 0.000E+00 & 0.911E+01 & 0.911E+01 & 0.267E+02 & 0.911E+01 \\
\end{bmatrix}
\]

**[ABDD] inverse**

\[
\begin{bmatrix}
0.960E-05 & -0.729E-05 & 0.000E+00 & 0.499E-19 & -0.455E-19 & 0.105E-20 \\
-0.729E-05 & 0.960E-05 & 0.000E+00 & -0.452E-19 & 0.419E-19 & -0.798E-21 \\
0.000E+00 & 0.000E+00 & 0.499E-05 & 0.259E-20 & -0.190E-20 & -0.235E-21 \\
-0.499E-19 & -0.452E-19 & 0.259E-20 & 0.731E-01 & -0.536E-01 & -0.664E-02 \\
-0.455E-19 & -0.190E-20 & -0.536E-01 & 0.731E-01 & -0.664E-02 & 0.420E-01 \\
-0.105E-20 & -0.235E-21 & -0.664E-02 & -0.664E-02 & 0.420E-01 & 0.420E-01 \\
\end{bmatrix}
\]

**LAMINATE ENGINEERING CONSTANTS:**

\[
\begin{align*}
E_x &= 0.2603E+07 \text{ psi} \\
E_y &= 0.2603E+07 \text{ psi} \\
G_{xy} &= 0.5009E+07 \text{ psi} \\
N_{Lx} &= 0.7589 \\
N_{Lx} &= 0.7589 \\
E_{TAxy,x} &= 0.0000 \\
E_{TAxy,y} &= 0.0000 \\
E_{TAx,xy} &= 0.0000 \\
E_{TAy,xy} &= 0.0000 \\
\end{align*}
\]
The loading type for this laminate is NO LOADS.
The laminate contains 32 plies.
The laminate consists of 1 materials.

**Material Properties, psi**

<table>
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<tr>
<th>Material</th>
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<th>$E_2$</th>
<th>$G_{12}$</th>
<th>$N_{12}$</th>
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**Laminate Engineering Constants:**

- $E_x = 0.2603E+07$ psi
- $E_y = 0.2603E+07$ psi
- $G_{xy} = 0.5009E+07$ psi
- $N_{12} = 0.7589$
- $N_{21} = 0.7589$
- $\alpha_{xy} = 0.0000$