Research on Graphite Reinforced Glass Matrix Composites

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Graphite fiber reinforced glass matrix composites which offer excellent structural performance at temperatures up to 875 K, low density, excellent environmental stability, and low cost have been synthesized. Further, these composites can be made so that consistent properties are obtained together with 93% retention in flexural strength after exposure to air at 813 K for 100 hrs or complete strength retention after 100 hrs in air at 723 K. The oxidation resistance of a Hercules HMS graphite fiber reinforced C.G.W. 7740 +2% SiO₂ glass matrix composite is in contrast with the result found at the end of the first year's research for similar Hercules HMS graphite fiber glass matrices where only 72% of their original strength was retained after 4 hrs exposure to air at 833 K. The increased oxidation resistance of the Hercules HMS graphite fiber reinforced glass matrix composite is believed to be due both to the introduction of a new slurry and the fact that the composite is formed at a higher temperature, 1723 vs 1473 K. These changes also yielded a Hercules HMS graphite fiber reinforced glass matrix with higher flexural strength; its three-point bend strength is 1030 MPa (150 000 psi) and the four-point flexural strength is 964 MPa (140 000 psi), and a higher percent strain to failure (up to 0.52%) as well as a composite modulus of 200 GPa or 29 million psi. Results obtained with Hercules HTS, Thornel 300 and Thornel Pitch, and Celanese Type DG-102 graphite fibers reinforced C.G.W. 7740 and 7740 +2% SiO₂ glass matrix composites are also included but are not yet as impressive as are the results with the Hercules HMS graphite fiber reinforced C.G.W. 7740 +2% SiO₂ glass matrix composites.

The work of characterizing the Hercules HMS graphite fiber reinforced C.G.W. 7740 +2% SiO₂ and of developing the process for making it has started. At the current stage of development, graphite fiber reinforced borosilicate glass matrix composites have exhibited a strength which increases with temperature up to 875 K (the softening point of the glass), excellent fracture toughness, no influence of 100 thermal cycles from 383 to 833 K in argon or 100 flexural fatigue cycles from low load to high load on the residual strength, and no strength degradation when painted with sea salt concentrates and thermally cycled in argon to 833 K. The Hercules HMS graphite fiber reinforced C.G.W. 7740 glass matrix composite made with one of the new slurries but without added silica has an average interlaminar shear strength of 39.8 MPa (5780 psi). Such graphite fiber reinforced glass matrix composites can be made as uniaxial, biaxial, or multiaxially reinforced.

*C.G.W. is Corning Glass Works; C.G.W. 7740 is their code for a borosilicate glass or one of their Pyrex compositions

Use of commercial products or names of manufacturers in this report does not constitute official endorsement of such products or manufacturers, either expressed or implied, by the National Aeronautics and Space Administration.
composites. They can also be made from tapes containing sufficient glass that no additional glass between layers need be added, and these tapes can be transferred from take-up spool to die without loss of glass. Further, three thin composites can be hot pressed at one time just as easily as one thicker composite, and using the presently available equipment at the Research Center, composites can be formed as large as squares 10 cm x 10 cm. Again, tapes with 12 layers of slurry impregnated graphite fiber can be used as readily as the usual uni-layer tapes to form composites. When the coefficient of thermal expansion of uniaxial graphite fiber reinforced glass matrix composite is measured in the 90° direction, the value obtained for the coefficient of thermal expansion is only one-eighth that of a similar graphite fiber reinforced epoxy resin indicating the greater dimensional stability of the glass matrix composite. Despite these advances, much work remains to be done to more completely characterize the Hercules HHS graphite reinforced C.G.W. 7740 + 2% SiO₂ glass matrix composite and similar composites and in further simplification of the process for forming the composite.
Fiber reinforced composites are widely accepted as structural materials because of their desirable attributes of high strength, high modulus and low density. At the inception of this program the sales of high performance fiber reinforced composite materials exceeded a million pounds yearly. In general, most of these composites were organic polymer (epoxy resins, polyimides, polycarbonates, and similar materials) matrices reinforced with a great variety of fibers including Kevlar*, carbon, graphite, fused silica, glass, and boron. In general, almost all of these composites were limited to use temperatures not exceeding 575 K and many of these to temperatures not exceeding 425 K.

At the start of this contract, there were no reinforced glass matrix composites commercially available except the age-old wire reinforced glass used for improving the burglar resistance of homes and stores and the AVCO developed tungsten mesh reinforced fused silica intercontinental ballistic missile nose cones. Yet if one conceives of glass as just a high-temperature thermoplastic, the substitution of a glassy matrix for the low temperature polymers in composite materials seems a natural way to proceed. This concept, however, had attracted sparse attention in the past. Upon examining the technical literature, only 12 references by British and American scientists (Refs. 1-12) could be found. Since these references were discussed in detail in the first annual report on this subject (Ref. 13), it is sufficient to indicate that the fabrication approach has followed along the directions suggested by Sambell, et al (Ref. 4) and Levitt (Ref. 7) and has emphasized the use of a slurry in forming the graphite fiber reinforced glass matrix composite to the virtual exclusion of the other possible procedures. The materials and process are considered in more detail in the next sections.

*Aramid Fiber, trademark of DuPont*
EXPERIMENTAL PROCEDURE

Materials

Glass

The types of glasses which were considered for use on this program are shown in Table I. Just as the graphite fibers show individual characteristics, the glasses also vary widely in their nature possessing different coefficients of linear expansion, different chemical compositions, varied environmental stability, and, of course, different temperature working ranges. Although all the glasses in the table have relatively low thermal expansion coefficients, only the titanium silicate glass has an expansion coefficient as low as that of the graphite fibers.

Just how different the working characteristics of these glasses are is shown in Fig. 1. It is apparent, therefore, that any resultant graphite fiber-glass matrix composites will have their own fabrication conditions. The glasses as they actually arrive at UTRC are shown in the scanning electron micrograph of Fig. 2. Although each glass is purchased solely on the basis that 90% must pass through a 360 mesh screen; actually all glasses contain numerous fine particles under one micron in size, and it is believed that these fine particles contribute greatly to the fabrication process to be described in the next section.

Graphite Fiber

The types of fibers readily available in this country for a research program on the generation of new types of graphite fiber reinforced glass matrix composites are shown in Table II. It will be noted that they vary widely in several important characteristics such as the number of fibers in each, the strength and modulus of the fiber, the precursor used to make the fiber, and the price. It is perhaps not so obvious that the use of each fiber presents a distinct surface chemistry problem, but if one considers the finish on the fiber surface and the chemical elements found on the fiber surface as shown in Table II the problem is evident. This is particularly true if one examines the distinctive shape of each carbon fiber as shown in Fig. 3. It is apparent, therefore, that any given glass matrix reinforced with a given graphite fiber will show its own characteristic behavior.

To broaden the investigation as much as practicable, UTRC purchased some of each carbon fiber shown in Table III.
<table>
<thead>
<tr>
<th>Type of Glass</th>
<th>Nature of Glass</th>
<th>Strain Point $10^{11}$</th>
<th>Anneal Point $10^{12}$</th>
<th>Soften Point $10^{12}$</th>
<th>Liquidus</th>
<th>Working Point $10^{3}$</th>
<th>Density $kg/m^3$</th>
<th>Index of Refract.</th>
<th>Dielectric Constant</th>
<th>Coef. Linear Expansion $cm/cm K \times 10^{-7}$</th>
<th>Modulus GPa</th>
</tr>
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<tr>
<td>C.G.W. 7740</td>
<td>Boro-silicate</td>
<td>833 K</td>
<td>833 K</td>
<td>1094 K</td>
<td>1290 K</td>
<td>1525 K</td>
<td>2230</td>
<td>1.476</td>
<td>4.6</td>
<td>32.5</td>
<td>63</td>
</tr>
<tr>
<td>C.G.W. 1723</td>
<td>Alumino-silicate</td>
<td>938</td>
<td>983</td>
<td>1181</td>
<td>1343</td>
<td>1441</td>
<td>2640</td>
<td>1.574</td>
<td>6.3</td>
<td>46</td>
<td>88</td>
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<tr>
<td>Ferro S</td>
<td>Magnesio-alumino-silicate</td>
<td>1033</td>
<td>1083</td>
<td>1243</td>
<td>1323</td>
<td>2490</td>
<td>1.547</td>
<td>5.2</td>
<td>29</td>
<td>85</td>
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<tr>
<td>C.G.W. 7913</td>
<td>High silica</td>
<td>1163</td>
<td>1293</td>
<td>1803</td>
<td>1973</td>
<td>2180</td>
<td>1.658</td>
<td>3.8</td>
<td>5.5</td>
<td>68</td>
<td></td>
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<tr>
<td>C.G.W. 7940</td>
<td>Pure silica</td>
<td>1229</td>
<td>1357</td>
<td>1853</td>
<td>1973</td>
<td>2200</td>
<td>1.455</td>
<td>3.8</td>
<td>3.5</td>
<td>72</td>
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<tr>
<td>C.G.W. 7971</td>
<td>Titanium silicate</td>
<td>1273</td>
<td>1773</td>
<td>1873</td>
<td></td>
<td>2210</td>
<td>1.484</td>
<td>4.0</td>
<td>-2</td>
<td>68</td>
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*Viscosity, N-s/m²*
Figure 1. Viscosity - Temperature Curves

LOG VISCOSITY, N·S/m²

TEMPERATURE, K

SOFTENING POINT

STRAIN POINT

ANNEALING POINT

WORKING POINT

BOROSILICATE (7140)

HARDLIME (7140)

96% SILICA (7280)

FUSED SILICA
Figure 2. Scanning Electron Micrographs of Glass Powders
<table>
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<tr>
<th>Type of Fiber</th>
<th>No. of Fibers in Tow</th>
<th>Finish Used</th>
<th>Precursor and Diameter of Fiber (microns)</th>
<th>Modulus GPa</th>
<th>Strength MPa</th>
<th>Density kg/m³</th>
<th>Coeff. Linear Expansion cm/cm K (axial)</th>
<th>Cost per Pound $</th>
<th>Result of Spectroscopic Examination</th>
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<tr>
<td>Hercules HM/PVA</td>
<td>1 000</td>
<td>PVA 0.86</td>
<td>PAN</td>
<td>385</td>
<td>2427</td>
<td>1850</td>
<td>-5.7 x 10^-7</td>
<td>300</td>
<td>High Na, high Si, Cr</td>
</tr>
<tr>
<td>Hercules HNS</td>
<td>10 000</td>
<td>Oxidized</td>
<td>PAN 7.3</td>
<td>351</td>
<td>2703</td>
<td>1808</td>
<td>-3.8 x 10^-7</td>
<td>90</td>
<td>High Na, very high K</td>
</tr>
<tr>
<td>Hercules HTS</td>
<td>10 000</td>
<td>Oxidized</td>
<td>PAN 7.6</td>
<td>256</td>
<td>2830</td>
<td>1658</td>
<td>75</td>
<td>Very low Na, highest Fe, Si, Ti, Zr</td>
<td></td>
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<tr>
<td>Celanese DG-102</td>
<td>384</td>
<td>Oxidized</td>
<td>PAN 8</td>
<td>531</td>
<td>1724</td>
<td>1960</td>
<td>250</td>
<td>Very high Na, high Cu, high Sn, Zn</td>
<td></td>
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<tr>
<td>Thornel 300 Grade WYP30 1/0</td>
<td>3 000</td>
<td>UC 309</td>
<td>PAN 6.9</td>
<td>234</td>
<td>2482</td>
<td>1760</td>
<td>40</td>
<td>Very high Na, high Cu, high Sn, Zn</td>
<td></td>
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<tr>
<td>Thornel 300 Grade WYP90 1/0</td>
<td>1 000</td>
<td>UC 309</td>
<td>PAN 8.4</td>
<td>228</td>
<td>2655</td>
<td>1750</td>
<td>32</td>
<td>Moderate Na, high Mg, Sn, P</td>
<td></td>
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<td>Thornel 75 Grade WYL160 1/2</td>
<td>720</td>
<td>PVA</td>
<td>Rayon 6.0</td>
<td>538</td>
<td>2620</td>
<td>1800</td>
<td>385</td>
<td>Intermediate Na, very high P, high Ca, Ta, Zn</td>
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<td>Thornel 50 Grade WYGI30 1/2</td>
<td>720</td>
<td>PVA</td>
<td>Rayon 6.6</td>
<td>393</td>
<td>2172</td>
<td>1660</td>
<td>320</td>
<td>Intermediate Na, very high P, high Ca, Ta, Zn</td>
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Figure 3. Typical fibers as seen in Scanning Electron Micrograph.
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<th>Tensile Strength MPa (ksi)</th>
<th>Young's Modulus GPa (10^6 psi)</th>
<th>Cost ($/lb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hercules HTS</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 000</td>
<td>2979 (432)</td>
<td>234 (34)</td>
<td>75</td>
</tr>
<tr>
<td>Hercules HTS Special</td>
<td>1 000</td>
<td>2875 (417)</td>
<td>34</td>
</tr>
<tr>
<td>Hercules HM (μ) – PVA</td>
<td>1 000</td>
<td>2427 (352)</td>
<td>300</td>
</tr>
<tr>
<td>Hercules HMS – 10 K</td>
<td>10 000</td>
<td>2344 (340)</td>
<td>34</td>
</tr>
<tr>
<td>Hercules HMS – 3 K</td>
<td>3 000</td>
<td>2330 (338)</td>
<td>300</td>
</tr>
<tr>
<td>Thornel 50 WYG 130 1/2</td>
<td>1 540</td>
<td>2172 (315)</td>
<td>320</td>
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<tr>
<td>Thornel 75 WYL 160 1/2</td>
<td>1 540</td>
<td>2620 (380)</td>
<td>385</td>
</tr>
<tr>
<td>Thornel 300 WYP 30 1/2</td>
<td>3 000</td>
<td>2482 (360)</td>
<td>40</td>
</tr>
<tr>
<td>Thornel 300 WYP 90 1/0</td>
<td>1 000</td>
<td>2655 (385)</td>
<td>32</td>
</tr>
<tr>
<td>Thornel Pitch VS 0022-1</td>
<td>2 160</td>
<td>1145 (166)</td>
<td>55</td>
</tr>
<tr>
<td>Thornel Pitch VS 0022-2</td>
<td>2 160</td>
<td>945 (137)</td>
<td>55</td>
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<td>Thornel Pitch VS 0022-3</td>
<td>2 160</td>
<td>993 (144)</td>
<td>55</td>
</tr>
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<td>Thornel Pitch VS 0032-1</td>
<td>720</td>
<td>1282 (186)</td>
<td>270</td>
</tr>
<tr>
<td>Thornel Pitch VS 0032-2</td>
<td>720</td>
<td>1083 (157)</td>
<td>270</td>
</tr>
<tr>
<td>Celanese DG-102</td>
<td>384</td>
<td>1724 (250)</td>
<td>250</td>
</tr>
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</table>
Composite Fabrication

Slurry Technique

While several methods exist for coating the fiber as required in the construction of a fiber reinforced glass composite, much the simplest and lowest cost method, if it can be made to work, consists of pulling the graphite fiber tow through a slurry containing a suspension of the finely ground glass particles.

In the slurry process for coating the graphite fiber with glass, the graphite fiber is unwound from the spool and pulled through an agitated organic solution containing a suspension of fine glass particles. The process is shown schematically in Fig. 4. The slip may be composed of 40 grams of powdered glass and 3 grams of polyvinyl alcohol dissolved in 100 grams of water to which 2 grams of ethylene glycol is added as a plasticizer. Alternately, the slip may comprise 85 grams of glass in 225 ml of toluene to which 5 grams of polystyrene and 5 drops of tergitol have been added. Excess glass and solvent are removed by pressing a squeegee against the drum as it winds. The ground glass used is sized, so that 90% passes through a 325 mesh sieve. After the tape is dry (sometimes heating with a radiant heat source is required to remove excess solvent) it is cut and removed from the drum and then cut into strips or squares which are layed up to give unidirectional or cross-plied fiber alignment and then hot pressed.

The effect of the amount of glass in the slurry used to form the graphite fiber-glass tape is shown in Table IV where it is shown that doubling the glass added to the slurry results in a decrease of strength. Similar tests run with 45 grams of glass and with 135 grams of glass in the slurry confirm that the best choice seems to be 85 grams of glass i.e. 200 grams of isopropyl alcohol, 10 grams of polyvinyl alcohol and 5 drops of a wetting agent such as tergitol H.D. 527.

Slurry Variation

The procedure just described was used in all of the earlier work, i.e. up to and including (cf Appendix A) sample LB 174. As mentioned, the isopropyl alcohol furnished the solvent or suspension vehicle and the polyvinyl alcohol formed the plasticizer part of the suspension and is Slurry A. This process worked well until a specific supply of polyvinyl alcohol was exhausted. Then, although polyvinyl alcohol, reagent grade, under the same formula number from Baker Chemicals was reordered, the new supply of polyvinyl alcohol would not either dissolve or stay in suspension in the isopropyl alcohol with a consequence that all the new graphite fiber-glass powder tapes made lacked green strength and lost copious
Figure 4 Slurry Method of Coating Graphite Fiber

- Graphite Fiber
- Supply Wheel
- Guide Wheel
- Guide Wheel
- Coater
- Take-Up Wheel covered with Mylar film over Teflon
### Table IV

**Effect of Amount of Glass in Slurry A on Achieved Flexural Strength of Graphite Fiber Reinforced Glass Composite**

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Glass</th>
<th>Fiber</th>
<th>Temp. of Hot Press °C</th>
<th>Pressure MPa</th>
<th>3-Point Flexural Strength MPa psi</th>
<th>Glass in Slurry gms</th>
<th>Glass Added Between Layers gms</th>
</tr>
</thead>
<tbody>
<tr>
<td>LB 75-2</td>
<td>7740</td>
<td>Thornel 300</td>
<td>1323 argon</td>
<td>13.8</td>
<td>287 41 700</td>
<td>170</td>
<td>0.11</td>
</tr>
<tr>
<td>-3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>277 40 200</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>242 35 000</td>
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<td></td>
</tr>
<tr>
<td>-6</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>261 37 800</td>
<td></td>
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<td>-9</td>
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<td></td>
<td></td>
<td></td>
<td>306 44 400</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>274 39 700</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LB 78-1</td>
<td>7740</td>
<td>Thornel 300</td>
<td>1323 argon</td>
<td>13.8</td>
<td>418 60 600</td>
<td>85</td>
<td>0.063</td>
</tr>
<tr>
<td>-2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>326 47 300</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-4</td>
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<td></td>
<td></td>
<td></td>
<td>504 73 100</td>
<td></td>
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<td>355 51 500</td>
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<td></td>
<td>456 66 200</td>
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<tr>
<td>-8</td>
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<td></td>
<td></td>
<td>505 73 400</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>427.5 63 000</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
amounts of glass powder on handling. Since then a number of organic solvents and plasticizers have been tried without finding anything as useful as the old polyvinyl alcohol-isopropyl alcohol combination. As a result of these efforts, however, a superior slurry consisting of isopropyl alcohol, polyethylene glycol and a small amount of polystyrene was discovered. This combination of ingredients yields a tape with considerable "green" strength which can be handled without shedding much of its glass powder. Further, the amount of glass picked up by the graphite fiber with these organic materials present in the slurry is so great that no glass powder need be added between the layers of material as they are charged in the die for hot pressing. Elimination of this step of adding carefully weighed amounts of glass powder between the tape layers both greatly speeds up the process of filling the die and eliminates a significant factor in the variability due to nonuniform distribution of the glass powder between layers. This newer slurry was used for samples GC 256 through GC 291 (cf Appendix A). The remarkable success achieved with this slurry, hereafter called slurry B, in achieving a uniform distribution of glass powder on the tape is shown in Table V where the weight of 12.7.62 cm squares of tape cut from a larger tape are compared. It will be noted that these squares vary in weight by less than 1%. Slurry B specifically consisted of 85 grams of glass in 260 ml of isopropyl alcohol, 24 grams of polyethylene glycol and 2 grams of polystyrene with 5 drops of a wetting agent added.

Further investigation of slurry modification has resulted in a third slurry which is similar to that used previously except that it contains 6 grams of DuPont Company's Ludox* H.S. 30 in place of the 2 grams of polystyrene. The results obtained with this slurry to which only 6 grams of Ludox H.S. 30, containing approximately 2 grams of silica, have led to remarkable improvements in graphite fiber reinforced glass composites. This slurry hereafter called slurry C has been used in preparing all composites from GC 292 to GC 360 inclusive.

Hot Pressing Procedures

All of the specimens prepared in the second year of this contract were fabricated using a Centorr Hot Press shown in Fig. 5. The equipment can be used for the fabrication of specimens as large as 15 x 10 x 2.5 cm (7.6 cm thick before hot pressing). The press can provide a load of 530 kN, temperatures up to 3373 K, and a vacuum of 21 μm/m² (10^-6 Torr) or alternately it can operate in argon. The press is double acting in contrast to the single acting presses used during the first year of this program.

*Registered trademark, E. I. DuPont de Nemours Corporation, Wilmington, Delaware
Table V
Comparative Weight of 7.62 cm Squares of Graphite Fiber-Powdered Glass Cut from One Large Tape, GT 234 (Slurry B)

<table>
<thead>
<tr>
<th>Square</th>
<th>Weight (grams)</th>
<th>Difference from Average (grams)</th>
<th>% Variation from Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.727</td>
<td>+0.027</td>
<td>0.57</td>
</tr>
<tr>
<td>2</td>
<td>4.728</td>
<td>+0.028</td>
<td>0.60</td>
</tr>
<tr>
<td>3</td>
<td>4.683</td>
<td>-0.017</td>
<td>0.36</td>
</tr>
<tr>
<td>4</td>
<td>4.676</td>
<td>-0.024</td>
<td>0.51</td>
</tr>
<tr>
<td>5</td>
<td>4.737</td>
<td>+0.037</td>
<td>0.79</td>
</tr>
<tr>
<td>6</td>
<td>4.713</td>
<td>+0.013</td>
<td>0.28</td>
</tr>
<tr>
<td>7</td>
<td>4.706</td>
<td>+0.006</td>
<td>0.13</td>
</tr>
<tr>
<td>8</td>
<td>4.642</td>
<td>-0.058</td>
<td>1.23</td>
</tr>
<tr>
<td>9</td>
<td>4.771</td>
<td>+0.071</td>
<td>1.51</td>
</tr>
<tr>
<td>10</td>
<td>4.729</td>
<td>+0.029</td>
<td>0.62</td>
</tr>
<tr>
<td>11</td>
<td>4.644</td>
<td>-0.056</td>
<td>1.19</td>
</tr>
<tr>
<td>12</td>
<td>4.642</td>
<td>-0.058</td>
<td>1.23</td>
</tr>
</tbody>
</table>

Average: 4.700 +0.75
In actual operation the HMS graphite fiber C.G.W. 7740 glass matrix composites made from slurry B tapes were usually hot pressed at temperatures of 1473 K, 13.8 MPa, 1 hr dwell time, argon atmosphere, and the pressure was not released until the sample had cooled to 773 K. Composites reinforced with Hercules HTS or Celanese DG-102 were prepared similarly. On the other hand, the HMS graphite fiber reinforced C.G.W. 7740 + 2% SiO₂ glass matrix composites, slurry C, were most generally prepared by hot pressing at 1723 K, 6.9 MPa, argon atmosphere, 1 hr dwell time and no release of pressure until the sample was cooled to 773 K. The Hercules HTS, Thornel Pitch, and Celanese DG-102 graphite fiber reinforced C.G.W. 7740 + 2% SiO₂ (slurry C tapes) were prepared in the same manner. As may be seen from Fig. 6, where the relative motion of the die plunger after application of full hot pressing pressure is shown, since most of the plunger motion takes place earlier than the usual 60 min dwell time, it may be possible to appreciably speed up the hot pressing operation. It is not known, however, whether the last relatively small motion of the plunger removes the last traces of porosity or whether it is caused by the extrusion of glass out of the die cavity. These questions would need to be studied before any drastic change in dwell time could be justified. All composites shown on this figure were formulated with slurry A and hot pressed at 1473 K, GC 221 and 223 at 13.8 MPa and GC 221 at 6.9 MPa.

Composite Characterization

Sample Preparation

The majority of the experimental composites prepared in the second year were of two sizes, either 7.6 cm x 7.6 cm x 0.25 cm or 6.67 cm x 2.22 cm x 1 cm. These composite plates were then cut into individual samples using diamond grit cutting and grinding wheels. In every case, except where specifically noted in this report, the surfaces of all specimens were ground flat and parallel thus exposing graphite fibers on all sides.

Flexural Testing and the Effect of Span-to-Depth Ratio

Flexural tests have been used for determining the strength properties of these composites. This technique has been chosen because of simple shapes (bars) required and because it avoids all the difficulties associated with gripping the specimen.

The fracture of a composite specimen during three-point flexural strength testing can be controlled by the applied flexural stresses or the applied shear stresses. The relative levels of these stresses, and the relative levels of inherent composite tensile and shear strength determine material performance. This complex type of behavior can be expressed using an interaction diagram
Figure 6. Relative Motion of Die Plunger After Application of Full Pressure During Hot Pressing, Slurry B
approach. By this method both the flexural strength and shear strength of each composite specimen are calculated and plotted as a function of test specimen span-to-depth ratio (L/h). At low values of L/h specimen failure should be controlled by shear deformation, while at high values of L/h the flexural strength of the specimen should control. This type of representation has been successfully used in the past for a wide range of composites (Refs. 14,15).

A uniaxially reinforced specimen containing 50 vol % HMS fiber in 7740 glass was fabricated and tested over an L/h range of 10 to 40. All specimen surfaces were machined prior to testing and the resultant data are presented in Figs. 7 and 8. The shear interaction diagram, Fig. 7, was obtained by calculating the maximum shear stress applied to each specimen based on the maximum applied load at fracture. Also included in the figure are two calculated curves that would represent the maximum applied shear stress of specimens that failed in flexure with actual material flexural strengths (σf) of 689 MPa and 550 MPa.

The same specimen data used in Fig. 7 are replotted in Fig. 8; however, in this case flexural strengths were calculated and plotted as (L/h). Again, calculated lines are included for specimens failing by flexure at 689 MPa and 550 MPa. In addition, the line for a specimen failing by shear at a level of 16.2 MPa was plotted. This shear strength is equal to the average value of shear stress calculated for L/h = 10 from Fig. 7.

Although the data do exhibit the trends expected, it is also clear that the scatter in data is very significant. Additional testing during the later phases of this program will provide additional data; however, the practice in this program of testing specimens for flexural strengths at (L/h) values above 30 in general is amply justified.

Oxidation Studies

The investigation of the resistance to oxidation of various graphite fiber reinforced glass matrix composites was carried out by heating samples prepared for flexural testing to the test temperature in air. The temperature was controlled within ± 5 K at temperatures of 723, 813 and 833 K for times of 4 hrs, 24 hrs, and 100 hrs. At the end of these exposures the flexural strength of the sample was measured at room temperature.

Thermal Expansion

The thermal expansion of the composite specimen was measured using a Dilatronic III High Resolution dilatometer purchased from Theta Industries Inc. of Port Washington, New York. In this instrument the change in length of the specimen is measured in reference to an NBS fused silica standard (SRM #739).
Figure 7. Calculated Values of Maximum Applied Shear Stress at Failure for Three Point Bend Testing as a Function of Specimen Span (L) to Depth (h) Ratio
Figure 8. Calculated Values of Maximum Applied Flexural Stress at Failure for Three Point Bend Testing as a Function of Specimen Span (L) to Depth (h) Ratio
The absolute expansion is determined by correcting the measured curve for the expansion of the standard and for the expansion of the specimen holder if the sample and standard are not the same length. The change in length is referred to the initial length of the specimen at 293 K.

Exposure to Sea Salt

To simulate exposure to spray with sea water and sea air damage it was necessary to improvise since UTRC does not at this time possess a standard salt spray controlled humidity cabinet. This was done by painting standard flexural test specimens with a solution containing six times the normal concentration of sea salt, drying, repainting to give a total of eight coats of sea salt. The coated flexural specimen was then sealed in a silica tube in an argon atmosphere and thermally cycled from 383 to 833 K for 100 cycles at the rate of 1 cycle every eight minutes.

Cycling Under Flexural Load

Cycling under a flexural load which drops to 1/20th of the assessed strength of the sample and rises to 8/10ths of this load was carried out under a four-point bend mode.

Instrumented Impact

A Charpy test is an impact failure conducted in three-point bending. This test is a measure of the toughness of a material at moderate impact velocities (up to 3.6 meters/sec). While the value of the energy required for fracture is dependent on the size of the specimen, and therefore is not an intrinsic material value, the tests are useful for comparative purposes. The instrumented Charpy test in particular has been a valuable asset for the comparison of material toughness and for illustration of the fracture modes under dynamic conditions. The impact machine is instrumented with strain gages so a record can be made of the force as a function of time.

Thermal Cycling

Premachined flexural test specimens were cycled between 383 and 833 K while encapsulated in glass tubes containing argon. The use of an inert atmosphere permitted the exclusion of any effects due to specimen oxidation.
RESULTS AND DISCUSSION

In the appendices are tabulated specific data that are not needed for an understanding of the results obtained in this study. In Appendix A, a summary listing of the graphite fiber reinforced glass matrix composites is given. In Appendix B, data which are mainly represented in the report by figures are tabulated. A paper based on this sponsored research entitled "Glass Matrix Composites I - Graphite Reinforced Glass" and authored by K. M. Prewo and J. F. Bacon was presented at the Second International Conference on Composite Materials (Ref. 14).

Digest of First Year's Research

Since an annual report has been issued (Ref. 13) detailing the first year's research on graphite fiber reinforced glass matrix composites, this section of the report contains only those results which serve to characterize results of these composites which were not repeated in the second year's research.

Strength, Distribution of Strength, High Temperature Strength

Strengths as high as 977 MPa or 142 ksi (Appendix B - Table 1) had been achieved by the end of the first year with HMS graphite fiber reinforced 7740 glass matrix composites made with slurry A using the old press at 1273 K and 13.8 MPa. More usually, composites made with HMS graphite fiber and 7740 glass matrix under the same conditions would have an average strength of 689 MPa or 99.98 ksi (Appendix B, Table 1). Such composites showed a very narrow strength distribution as shown in the probability paper plot of Fig. 9 and tabulated in Appendix B, Table 1. These HMS graphite fiber reinforced 7740 glass matrix composites can be used to temperatures of 873 K. The slurry A data of Fig. 10 and Table 2, Appendix B, demonstrate that a typical HMS fiber-7740 glass composite has an average strength of 825 MPa (120 ksi) at room temperature and increases in strength to 1213 MPa (175 ksi) at 873 K when studied by means of three point bend tests, and only at 973 K does the specimen deform excessively. A similar increase in strength with temperature is shown for slurry A composites of Celanese DG-102 graphite fiber reinforced C,G,W. 7740 glass matrix composites (Table 3, Appendix B). These Celanese DG-102 graphite fiber 7740 composites start out lower in strength, however, and never approach the level of the HMS graphite fiber reinforced composites.
Figure 9. Three Point Bend Strength of Specimen Tested at 295 K, Slurry A
Figure 10. Flexural Strength of Hercules HMS - 10 K Fiber Reinforced 7740 Glass as a Function of Test Temperature, Slurry A
The manner in which these HMS graphite fiber reinforced C.G.W. 7740 glass matrix composites fail is shown by the four-point flexural test data of Fig. 11. Again these composites were made with slurry A, old press, temperature 1273 K and pressure of 13.8 MPa. Both totally linear and combined linear-nonlinear deflections are shown; however, in all cases specimens did not fracture completely but instead cracks were diverted in the interior of the specimen so that failed specimens remained visibly intact after test.

Comparison of Graphite Fiber Reinforced Glass Matrices with Other Composites

Three-point bend data for the Hercules HMS graphite fiber reinforced C.G.W. 7740 glass matrix composites have been compared with graphite fiber reinforced thermoplastics, graphite fiber reinforced aluminum, and BORSIC* fiber reinforced titanium alloy.

The data used to compare graphite reinforced thermoplastics with HMS graphite fiber reinforced C.G.W. 7740 glass matrix composites (slurry A) are shown in Fig. 12 and were obtained from a NASA-sponsored program at UTRC**. The fiber is the same Hercules HMS fiber used in this program and if one compares Figs. 12 and 10 it can be seen that the resin matrix composite maximum flexural strength at room temperature is approximately equal to the strength of the HMS-7740 composite at 873 K although greater at room temperature. The comparison also shows that the rate of strength decrease in the vicinity of the resin glass transition temperature is rapid and similar to that for the glass matrix composite above 923 K.

To compare the flexural strength of graphite reinforced glass matrix composites (slurry A) with that of graphite fiber reinforced aluminum, the recently published data of W. Harrigan of Aerospace Corporation shown in Fig. 13 was used. Again in comparing these data which are for wire and plates with the data of Fig. 10 for the HMS graphite fiber reinforced C.G.W. 7740 glass, it can be seen that the glass matrix composite is equal or superior to the aluminum matrix composite over the entire test temperature. As Fig. 13 also shows on a specific property basis, the glass matrix composite offers an additional advantage. The specific strength comparison of the graphite fiber reinforced aluminum with the HMS graphite fiber C.G.W. 7740 matrix specimen is shown in Fig. 14. The maximum use temperature of the glass matrix composite is significantly higher than that of the graphite aluminum.

*Trademark, United Technologies Corporation
Figure 11. Load - Deflection Traces for the 4 - Pt. Bend Tests on Hercules HMS - 10 K Reinforced 7740, Slurry A
Figure 12. Flexural Strength of Hercules HMS — 10 K Fiber Reinforced Thermoplastics as a Function of Test Temperature.

* UTRC CONTRACT NAS3–20077, 1976
Figure 13. Flexural Strength Comparison
Figure 14. Specific Flexural Strength Comparison
The only metal matrix composite with a possible use temperature in the range of room temperature to 773 K (excluding the much higher density, reinforced super alloys) is Boracic fiber reinforced Ti-6Al-4V. It is important, therefore, to compare the HMS graphite fiber reinforced C.G.W. 7740 matrix, slurry A, with this titanium alloy system at temperatures above 573 K. The data are shown in Fig. 15. Unfortunately, the flexural data over the temperature range of interest are not available for the titanium alloy so that Boracic-Ti tensile data are used. To provide a more realistic comparison the Boracic-Ti tensile strengths were multiplied by a factor of 1.4, a factor experimentally determined at UTRC for the ratio of flexural strength to tensile strength at room temperature for the Boracic-Ti composite. In addition, the data were divided by composite density to obtain the specific flexural strength comparison shown in Fig. 16. At temperatures above 423 K the glass matrix composite is clearly superior to the titanium matrix composite.

The elastic modulus for HMS graphite fiber reinforced 7740 glass slurry A is compared with values of E, characteristic of other composite systems in Table VI. The higher value for the glass matrix composite stands out.

Comparison of 3 Point and 4 Point Flexural Data for Graphite Fiber Glass Matrix Systems

The nature of the flexural strength test, i.e. whether it is a three-point or a four-point flexural strength test might be expected to affect the validity of a flexural-strength test for screening composite progress, but as Table VII shows, there is a definite relationship between the two tests.

Fracture Toughness Characteristics of HMS Fiber Reinforced 7740 Glass Matrix

Notched three-point bend systems were fabricated and tested in an attempt to characterize the fracture toughness of the HMS-7740 system. The specimen geometry is given in Fig. 17 and specimen dimensions with resultant data in Table VIII. As may be seen from the data of Table VIII, the fracture toughness does decrease with increasing temperature in a manner opposite to the variation in strength with temperature. It will be noted, however, that even the values obtained at the higher temperatures are favorable when compared to other composites and more conventional engineering alloys, Table IX.

Cyclic Testing of HMS Fiber Reinforced 7740 Glass Matrices

As in all the tests described to date, cyclic testing was carried out using uniaxial specimens of HMS fiber reinforced 7740 glass matrix composites. The thermal cycle interval was 8 min and 20 sec as shown in Fig. 18 and as may be seen from Fig. 19 and Table 4 of Appendix B the flexural strength of the HMS
Figure 15. Flexural Strength Comparison
Figure 16. Specific Flexural Strength Comparison
Table VI
Room Temperature Elastic Modulus Comparison

<table>
<thead>
<tr>
<th>System with Calculated Fiber Contents</th>
<th>Density $\rho$ (kg/m$^3$)</th>
<th>Density $\rho$ (lb/in$^3$)</th>
<th>Elastic Modulus $E$ (GPa)</th>
<th>$E_{11}/\rho$ (GPa/kg/m$^3$)</th>
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</thead>
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<tr>
<td>50 v/o Celanese DC-102-7740</td>
<td>2020</td>
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<td>296</td>
<td>43</td>
</tr>
<tr>
<td>50 v/o HMS-7740</td>
<td>1980</td>
<td>0.071</td>
<td>193</td>
<td>28</td>
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<tr>
<td>42 v/o T50-Al</td>
<td>2300</td>
<td>0.083</td>
<td>207</td>
<td>30</td>
</tr>
<tr>
<td>50 v/o B-Al</td>
<td>2700</td>
<td>0.097</td>
<td>227</td>
<td>33</td>
</tr>
<tr>
<td>43 v/o B-Ti</td>
<td>3700</td>
<td>0.133</td>
<td>234</td>
<td>34</td>
</tr>
</tbody>
</table>
### Table VII

Comparison of 3-Point Bend and 4-Point Bend Test Data for Several Graphite Fiber-Glass Composites

<table>
<thead>
<tr>
<th>Sample</th>
<th>3-Point Flexural Strength</th>
<th>4-Point Flexural Strength</th>
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<td>648</td>
<td>94 000</td>
</tr>
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<td>- 6 tests</td>
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<td>977</td>
</tr>
<tr>
<td>LB 135F - 9 tests</td>
<td>977</td>
<td>142 000</td>
</tr>
<tr>
<td>LB 97 - 9 tests</td>
<td>342</td>
<td>49 600</td>
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<tr>
<td>LB 97D - 9 tests</td>
<td>7740</td>
<td>342</td>
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Figure 17. Fracture Toughness Specimen
Table VIII

Fracture Toughness Specimen Dimensions

<table>
<thead>
<tr>
<th>Specimen</th>
<th>C (cm)</th>
<th>B (cm)</th>
<th>D (cm)</th>
<th>Test Span (cm)</th>
<th>L (cm)</th>
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<tbody>
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<td>0.990</td>
<td>0.691</td>
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<td>0.305</td>
<td>0.990</td>
<td>0.691</td>
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<td>LB-157-1</td>
<td>0.330</td>
<td>0.954</td>
<td>0.852</td>
<td>3.98</td>
<td>5.0</td>
</tr>
<tr>
<td>-2</td>
<td>0.330</td>
<td>0.954</td>
<td>0.852</td>
<td>3.98</td>
<td>5.0</td>
</tr>
</tbody>
</table>

Specimen Test Data

<table>
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<tr>
<th>Specimen</th>
<th>Test Speed (cm/min)</th>
<th>Test Temp K</th>
<th>$K_c$ $10^3$ psi/$\sqrt{\text{in}}$</th>
<th>Energy Per Unit Area Joules/m²</th>
<th>Energy Per Unit Area ft-lbs/in²</th>
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<tr>
<td>LB-140-1</td>
<td>20 000</td>
<td>295</td>
<td>21.4</td>
<td>23 500</td>
<td>11.3</td>
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<td>0.127</td>
<td>295</td>
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<td>LB-157-1</td>
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<td>873</td>
<td>15.8</td>
<td>10 600</td>
<td>5.1</td>
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<td>-2</td>
<td>20 000</td>
<td>923</td>
<td>15.0</td>
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Table IX
Fracture Toughness Comparison at 295 K

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<tr>
<td>$0^\circ$-HMS/7740, Slurry A</td>
<td>21.7</td>
<td>19.8</td>
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<tr>
<td>$0/90$-AS Graphite/AR 288*</td>
<td>14.25-1.973</td>
<td>13-18</td>
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<tr>
<td>Morganite II/Epoxy**</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$0^\circ$</td>
<td>35</td>
<td>32</td>
</tr>
<tr>
<td>$90^\circ$</td>
<td>1.75</td>
<td>1.6</td>
</tr>
<tr>
<td>$45^\circ$</td>
<td>2.52</td>
<td>2.3</td>
</tr>
<tr>
<td>$+45^\circ$</td>
<td>19.73</td>
<td>18</td>
</tr>
<tr>
<td>$0/\pm45^\circ/90$</td>
<td>24.1</td>
<td>22</td>
</tr>
<tr>
<td>2014-T6 Aluminum Alloy***</td>
<td>21.92</td>
<td>20</td>
</tr>
<tr>
<td>6061-T651 Aluminum Alloy***</td>
<td>29.6</td>
<td>27</td>
</tr>
</tbody>
</table>

*UTRC data using compact tension specimens
***Damage Tolerant Design Handbook NCIC-HB-01
EXCEEDED TARGET TEMPERATURE
BY 33 K IN OVERNIGHT RUN

Figure 19. Flexural Strength of HMS – 7740 Glass Composite After Thermal Cycling, Slurry A
graphite reinforced 7740 glass matrix composite was unaffected by a hundred such cycles. Similarly, 100 cycles of flexural fatigue between 0.8 and 0.08 of the as-fabricated four-point bend strength left the graphite fiber glass matrix composite unaffected. Again, painting the composite specimens with six times normal sea salt concentration and thermally cycling the samples between temperatures of 383 and 833 K (encapsulated in silica tubes) left the composites unaffected.

Discussion of Recent Advances

The progress made since the last annual report is summarized below. Processing effects on the microstructural characteristics and flexural strength properties, the shear, transverse, and cross-ply strengths, the thermal expansion response and the oxidation characteristics of these composites are topics that will be discussed in turn.

Processing Effects

A positive effect of very high temperature hot pressing on the flexural properties of graphite fiber reinforced 7740 containing Ludox (slurry C) has been discovered as will be described below. In order to make a qualitative assessment of the graphite-glass composites, a comparison of the microstructures of HMS graphite fiber reinforced 7740 pressed, slurry B, at 1473 K and the Ludox containing 7740 pressed at 1723 K, slurry C, can be made by considering Figs. 20-23. In both samples the glass is seen to surround each graphite fiber. The tape maps (Figs. 22,23) of the composites suggest that there is somewhat less memory of the tows in the slurry C composite pressed at 1723 K, more intimate contact of the adjacent tows and therefore fewer glass rich areas.

The three-point flexural properties of samples processed in a fashion similar to those represented in Figs. 20-23 are shown in Tables X and XI; these data are also represented in the cumulative failure probability plot of Fig. 24. Taking the data from the samples in each curve as belonging to a single population, the average strength and standard deviations are for the composites hot-pressed at 1473 K (no silica), slurry B, 785 ± 118 MPa and at 1723 K with silica addition, slurry C, 1023 ± 104 MPa.

Specimens of HMS graphite fiber reinforced 7740 + 2% SiO2, slurry C, hot pressed at 1723 K (GC 326), have also been flexurally tested in four-point bending. The data for these tests are listed in Appendix B, Table 8. The stress-strain behavior of one of these specimens is displayed in Fig. 25. Its ultimate strength is 1105 MPa, its modulus is 217 GPa and its failure strain is 0.53%. Although the average failure strain of HMS graphite (0.0077) is not achieved in this test, 70% of this value is achieved which is especially significant and impressive for a brittle matrix-brittle fiber composite combination.
Figure 20. Microstructure of HSiS Graphite Fiber Reinforced 7740 Glass Matrix (G.C. 281) Hot Pressed at 1473 K, Slurry B
Figure 21. Microstructure of HMS Graphite Reinforced 7740 + 2% SiO₂ (GC 326)
Hot Pressed at 1723 K, Slurry C
Figure 22. Tape Map of Cross Section of HMS Graphite Fiber Reinforced 7740 Glass Matrix (GC 281) Hot Pressed at 1473 K. Slurry B
Figure 23. Tape Map of Cross Section of HMS Graphite Reinforced 7740 + 2% SiO₂ Glass Matrix (GC326)
Hot Pressed at 1723 K, Slurry A
Table X

Three Point Flexural Strengths for Several Samples of HMS Graphite Fiber Reinforced 7740 Glass Matrix (no silica), Hot Pressed at 1473 K, 13.8 MPa, Slurry B

<table>
<thead>
<tr>
<th></th>
<th>GC 281</th>
<th></th>
<th>GC 289 Top</th>
<th></th>
<th>GC 289 Middle</th>
<th></th>
<th>GC 289 Bottom</th>
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<tbody>
<tr>
<td></td>
<td>MPa</td>
<td>ksi</td>
<td>MPa</td>
<td>ksi</td>
<td>MPa</td>
<td>ksi</td>
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<tr>
<td>1</td>
<td>965</td>
<td>140</td>
<td>660</td>
<td>95.8</td>
<td>852</td>
<td>124</td>
<td>746</td>
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<tr>
<td>2</td>
<td>674</td>
<td>97.7</td>
<td>895</td>
<td>130</td>
<td>839</td>
<td>122</td>
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<td>3</td>
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<td>99.8</td>
<td>928</td>
<td>135</td>
<td>939</td>
<td>136</td>
<td>799</td>
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<tr>
<td>4</td>
<td>752</td>
<td>109</td>
<td>759</td>
<td>110</td>
<td>908</td>
<td>132</td>
<td>799</td>
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<tr>
<td>5</td>
<td>786</td>
<td>114</td>
<td>652</td>
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<td>83.0</td>
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<td>6</td>
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<td>7</td>
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<td>9</td>
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<td>680</td>
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<tr>
<td>10</td>
<td></td>
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<td>757</td>
<td>110</td>
<td>879</td>
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<td>841</td>
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</table>

Mean 765   111 | 779   113 | 814   118 | 779   113
Std. Dev. 91.7 13.3 | 130  18.9 | 131   19.0 | 128   18.5
Std. Error 30.6 4.44 | 40   5.78 | 41    6.00 | 40    5.85
Table XI
Verification of Consistency of Results Obtained with New Type Slurry, HMS Graphite Reinforced 7740 Glass (Silica Addition) Made at 1723 K 6.9 MPa, 1 hr dwell time, argon atmosphere, slurry C

<table>
<thead>
<tr>
<th>Sample</th>
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<th>ksi</th>
<th>Sample</th>
<th>Three-Point Flexural Strength MPa</th>
<th>ksi</th>
<th>Sample</th>
<th>Three-Point Flexural Strength MPa</th>
<th>ksi</th>
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<td>GC 329</td>
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<tr>
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<td></td>
<td>A2</td>
<td>866.9 125.7</td>
<td></td>
<td>A2</td>
<td>1102 160</td>
<td></td>
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<tr>
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<td>1150 166</td>
<td></td>
<td>A6</td>
<td>859.6 124.7</td>
<td></td>
<td>A5</td>
<td>1005 146</td>
<td></td>
</tr>
<tr>
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<td>1240 180</td>
<td></td>
<td>A8</td>
<td>1061.6 154.0</td>
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<td>A7</td>
<td>954 138</td>
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<td></td>
<td>A12</td>
<td>1046.1 151.7</td>
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<td>A10</td>
<td>1107 161</td>
<td></td>
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<td>1060 153</td>
<td></td>
<td>B2</td>
<td>1109.9 161.0</td>
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<td>B2</td>
<td>982 142</td>
<td></td>
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<td>931 135</td>
<td></td>
<td>B6</td>
<td>1199.7 174.0</td>
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<td>B5</td>
<td>961 139</td>
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<td></td>
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<td>1149.8 166.8</td>
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<td>B7</td>
<td>935 136</td>
<td></td>
</tr>
<tr>
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<td>B10</td>
<td>1069 155</td>
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<td></td>
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<td>935.0 135.6</td>
<td></td>
<td>C2</td>
<td>1015 147</td>
<td></td>
</tr>
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<td>823 119</td>
<td></td>
<td>C6</td>
<td>999.5 146.2</td>
<td></td>
<td>C5</td>
<td>1020 148</td>
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<td></td>
<td>C8</td>
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<tr>
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<td></td>
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<td>1016.4 147.4</td>
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<td>Avg</td>
<td>1019 148</td>
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<td>Std Dev</td>
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Figure 24. Failure Probability of HMS Graphite Fiber Reinforced CGW 7740 Glass with and without Silica.
Figure 25. Stress Strain Curve of HMS Graphite Fiber Reinforced 7740 + 2% SiO$_2$ + Slurry C, Determined by Four Point Bend Test

E = 217 GPa
SPEC. GC 326-C7
Prior to the discovery of the advantages of very high temperature hot pressing, other variables which might influence composite behavior were explored. One such variable was the relationship between hot pressing pressure at a temperature of 1473 K and resultant strength in HMS graphite fiber reinforced 7740 matrix composites, slurry B. The strength data from the four pressures evaluated, i.e. 4.1, 6.1, 10.1 and 13.8 MPa, are listed in Appendix B, Table 7, the average flexural strengths are plotted as a function of pressure in Fig. 26. The strength increases in a linear fashion with pressure, reaching a value of 765 MPa at a 13.8 MPa pressure.

Another variable explored was the effect of hot pressing the samples (in series) simultaneously. At a hot pressing temperature of 1473 K (slurry B) and a pressure of 13.8 MPa, the average flexural strengths from the samples were 113, 118 and 113 MPa for the top, center, and bottom, respectively (Appendix B, Table 8).

After the observation was made of the positive effect of increasing the hot pressing temperature to greater than 1473 K, a study was made to determine the optimum temperature for the HMS-7740 + 2% SiO2 composite system, slurry C. The relationship between temperature and strength, which are listed in Appendix B, Table 9 and which are plotted in Fig. 27, show an optimum temperature of 1723 K with a corresponding strength of 1034 MPa. Furthermore, the strength of the system pressed at 1473 K is only 225 MPa; in comparison, the 7740 matrix without the silica additive, pressed at 1473 K, would be expected to exhibit a strength of ~550 MPa when pressed at 6.9 MPa (Fig. 26). Therefore, not only does the colloidal silica influence the green strength of the composite, but also has a definite influence on the composite consolidation.

The consistency of strength results obtained in HMS graphite fiber reinforced 7740 + 2% SiO2 glass matrix composites (slurry C) hot pressed at 1623 and 1723 K at a pressure of 6.9 MPa are demonstrated by comparing the data listed in Table 12 of Appendix B and Table XI of the text and the averages mentioned below. At 1623 K, the average strengths from three separate samples were 616, 639 and 651 MPa; at 1723 K, the average strengths of three separate samples were 1016, 1019 and 1034 MPa. This corresponds to an approximately 5 and 2% difference between extremes in the two cases respectively.

Holding the pressure constant, the effect of fiber type on the flexural strength properties of graphite fiber reinforced glass matrix composites made with a matrix of C.G.W. 7740 plus added silica (slurry C) was evaluated. The HTS fiber reinforced matrices had a flexural strength of 455 MPa when hot pressed at 1473 K and 731 MPa when hot pressed at 1623 K, Table XII. In comparison the average flexural strength of a similar system hot pressed at 1373 K but without silica addition (slurry B) was 370 MPa. It is evident, therefore, that the silica addition which permits hot pressing at a higher temperature by reducing fluidity of the glass is beneficial. However, the optimum temperature of hot pressing this fiber has not been achieved.
COMPOSITE IS HMS GRAPHITE FIBER REINFORCED 7740 GLASS MATRIX PREPARED 1473 K, ONE HOUR DWELL TIME, SLURRY B

Figure 26. Relation of Hot Pressing Pressure to Three-Point Flexural Strength of Composite
Figure 27. Relationship of Three-Point Flexural Strength of HMS-7740 + 2% SiO₂ Composite to Temperature at whichComposite is not Pressed, Slurry C
Table XII

Effect of Change in Slurry and Hot Pressing Temperature on Three-Point Flexural Strength of HTS Fiber Reinforced 7740 Composites

<table>
<thead>
<tr>
<th>Sample</th>
<th>3-Point Flexural Strength</th>
<th>Sample</th>
<th>3-Point Flexural Strength</th>
<th>Sample</th>
<th>3-Point Flexural Strength</th>
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<td></td>
<td>MPa</td>
<td>ksi</td>
<td></td>
<td>MPa</td>
<td>ksi</td>
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<td>LB 148 Old Slurry A 1373 K (13.8 MPa)</td>
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<td>408</td>
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<tr>
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<td>70.7</td>
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<td>111</td>
<td>A7 651</td>
<td>94.4</td>
<td>A12 681</td>
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<td>8 379</td>
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<td>69.1</td>
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<td>B12 844</td>
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<td>C2 789</td>
<td>114</td>
<td>C7 708</td>
</tr>
<tr>
<td></td>
<td>11 343</td>
<td>49.8</td>
<td>12 407</td>
<td>59.1</td>
<td>13 319</td>
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<tr>
<td></td>
<td>Mean 370</td>
<td>63.7</td>
<td>455</td>
<td>66.0</td>
<td>731</td>
</tr>
<tr>
<td></td>
<td>Std Dev 42.4</td>
<td>6.15</td>
<td>26.5</td>
<td>3.80</td>
<td>59.8</td>
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<td></td>
<td>Std Err 11.0</td>
<td>1.59</td>
<td>8.83</td>
<td>1.27</td>
<td>19.9</td>
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</tbody>
</table>
In the case of Celanese DG-102 fiber reinforced 7740 + 2% SiO₂ (slurry C), flexural strength data have been obtained from samples hot pressed at 1473, 1623 and 1723 K, Table XIII. The average respective strengths were 209, 417 and 460 MPa. The positive influence of higher hot pressing temperature is again apparent. The flexural strength of DG-102 reinforced 7740 prepared using the previous slurry B process method (7740 pressed at 1473 K) was 340 MPa.

An experiment was also conducted with Thornel pitch VS 0032 fiber in slurry C 7740 + 2% SiO₂. After hot pressing at 1623 K, the average flexural strength of such specimens was 448 MPa. The data are given in Table 11 of Appendix B.

For composites utilizing a matrix of 7740 + 2% SiO₂ (slurry C) and hot pressed at the nonoptimum temperature of 1623 K, the following comments with respect to fiber type are appropriate. The HTS fiber reinforced system was strongest (731 MPa) as might be expected from the nominally higher strength of this fiber. The HMS reinforced system displayed the next highest strength (635 MPa) in keeping with HMS being the next strongest fiber. The pitch and DG-102 fiber reinforced systems were weakest (448 and 417 MPa, respectively). Although the strength of both fibers is relatively low with the pitch fibers being the weaker, the strength of the composites derived from these fibers is in reverse order.

Composites employing higher percentages of Ludox have also been prepared (GC 225 and 226). Tapes have demonstrated excellent green strength and were easily handled without the loss of glass powder. Specimens prepared from these samples which were hot pressed at 1473 K were weaker than comparison samples without Ludox. In retrospect, based on the most recent data, a much higher hot pressing temperature might provide composites from these type compositions with equivalent or superior flexural strengths to the comparison composites pressed at 1473 K.

Shear, Transverse and Cross Ply Strengths

Shear Strength

The interlaminar shear strength was determined in three-point bending using a 3.5 to 1 span-to-depth ratio. The strength for an HMS graphite fiber reinforced 7740 glass matrix hot pressed at 1473 K, slurry B, was 39.8 MPa. This low value of shear strength illustrates the poor bonding that exists between matrix and fiber. Data for specimens hot pressed at 1723 K have not, as yet, been obtained and specimens made with 2% added silica, slurry C, have not yet been evaluated.
**Table XIII**

Effect of Hot Pressing Temperature on Three Point Flexural Strength of Composites

Formed from Celanese DC-102 Fiber Reinforced C.G.W. 7740 + 2% SiO₂ Glass

Matrix Hot Pressed Using Newest Slurry, 6.9 MPa, 1 hr Dwell Time

**Slurry C**

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Flexural Strength</th>
<th>Specimen</th>
<th>Flexural Strength</th>
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<td>221</td>
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Transverse Strength

Hercules HMS fiber unidirectionally reinforced 7740 glass matrix composites were fabricated from slurry B. Two composite thicknesses of 4 ply and 16 ply were used and the resultant panels were cut into 90° oriented specimens for three-point bend testing. The specimen surfaces were glass rich and this layer was not removed prior to testing since its presence simulates a surface protected against fiber oxidation.

The resultant three-point flexural strength data are listed in Appendix B, Table 12; the average flexural strengths were 9.7 and 14.8 MPa for specimens of 4 and 16 plys, respectively. In every case the specimens appeared to fracture at the tensile surface and crack propagation occurred across the specimen thickness. Fracture did not occur, however, at the mid-span in each case. This was particularly true of the 4 ply thick composite specimens (GC 208) and can be readily understood when one considers the importance of microstructure and the location of weaker and stronger material regions on transverse properties. Calculated composite strengths ranged from approximately 6.8 MPa (1000 psi) to 22.2 MPa (3240 psi) with the single high value occurring for a thicker specimen. These low levels of transverse strength are expected because of the low bond strength between the HMS graphite fiber and 7740 glass.

HTS and Celanese DG-102 fiber unidirectionally reinforced 7740 specimens were fabricated for 90° flexural strength measurement. The data for the HTS containing specimens are also presented in Table 12, Appendix B; the average transverse strength was 10.8 MPa and ranged from 4.7 to 18.0 MPa. As expected, the transverse strengths measured were quite low. The DC-102 fiber composite data were not obtained because the fabricated panels fractured during handling and cutting, thus also evidencing a very low transverse strength. As was previously noted, these data point clearly to the importance of multiaxially reinforced specimens for most applications. Furthermore, since these composites were prepared using a 7740 matrix hot pressed at 1473 K, the data should be viewed as preliminary. Improvements might be obtained from composites pressed at 1723 K, the optimum temperature for longitudinal strength.

Cross-Ply Composites

Cross ply constructions of HMS and Celanese DG-102 fiber reinforced composites were fabricated by the same procedures used for uniaxially reinforced specimens (hot pressed at 1473 K). The composite lay-up consisted of a sequence of alternating 2 ply thick strata that were stacked symmetrically with respect to the composite mid plane. The abbreviated notation for this lay-up sequence is \([(90_2, 0_2)_2]_s\) and consists of a total of 16 plys.
The microstructure of the composite containing DG-102 fiber is shown in Fig. 28. Besides the obvious fiber distribution, the most notable feature is the presence of many microcracks running through the plies. Many of these cracks are evenly spaced, run at $90^\circ$ to the ply plane, and may be associated with the mismatch in thermal expansion between the two ply orientations. On a macro scale, however, the as-fabricated composites looked excellent and uncracked. The $90^\circ$ direction has a higher coefficient of thermal expansion and thus, on cooling from the hot pressing temperature, it could crack because of the tensile stresses induced within the ply and at $90^\circ$ to the fiber direction. Careful examination of composites, however, has demonstrated that any observed cracks are due more to the preparation of the metallographic sections rather than any other reason. Thus, specimens which were cut and polished by the standard technique exhibited a large population of microcracks, while those which were surface ground and polished to remove approximately $0.25$ cm of material after cutting, were almost completely crack free. This is shown in the cross-ply composite, GC 215, in Fig. 29 which was prepared with a ply lay-up scheme of $[(0/90),]_s$. In addition, the matrix was leached out of samples of each composite and the freed fibers examined to ascertain whether any fiber fragmentation had occurred. This could conceivably occur during the application of pressure at the contact points of neighboring crossed fibers. No evidence of fiber fragmentation was found, however, and all of the extracted fibers exhibited lengths equivalent to the dimensions of the original specimens.

Mechanical testing of samples was carried out in the three-point bend mode. The specimens were cut such that the surface ply fibers were parallel to the major span of the bend test apparatus and no surface preparation was used prior to testing in order to avoid grinding through the $0^\circ$ primary load bearing plies. Because of this lack of grinding, and also the fact that the as-fabricated panels were not very uniform in thickness, the bend tests were not well controlled. Nevertheless, the objective was to make a first attempt at gaining data for cross ply material, and this was achieved.

The measured strengths are given in Table XIV, along with other pertinent information. In the case of the Celanese fiber reinforced specimens, an average strength of 115 MPa (16,700 psi) is somewhat lower than that expected since the average room temperature bend strength of unidirectionally reinforced $0^\circ$ specimens has been approximately 270-320 MPa (40-45,000 psi). This difference, however, may be attributable to a lower fiber content (40%) than has been typical in the past (50-60% by volume). All of these specimens fractured in a tensile mode and, as previously reported for $0^\circ$ Celanese reinforced specimens, the fracture did not exhibit much fiber pull out. In contrast, the HMS fiber reinforced specimens failed by interlaminar cracking at regions generally remote from the mid-span of the specimens. These cracks then propagated along interlaminar ply bond lines toward the centers of the specimens. The specimens, however, did not separate into pieces and instead retained substantial fractions of their original...
Figure 28. Microstructure of Specimen GC 30 203 Celanese DG 102 Reinforced 7740, [(90₂ O₂)₂] s, Slurry B
Figure 29. Microstructure of Specimen GC 215–5 HMS Reinforced 7740, [(0/90)₄]₅ samples Surface Ground and Polished, Slurry B
Table XIV

Three Point Bend Data for Cross Ply Specimens
Slurry B

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1In these tests the outer 0^0 plys were oriented with fibers parallel to the
major span
2Span-to-depth ratio for 3 point bend test
3Specimens separated at mid point of span into two pieces
4Specimens remained in tact after test. Interlaminar cracks occurred between
0^0 and 90^0 layers. These cracks started at the free ends of the specimens
and propagated inward.
strength after test. This observed failure mode may have been aggravated by the use of a smaller value of span-to-depth ratio (14) than has been typical in the past (30). The tabulated specimen flexural strengths were not widely spread and averaged 217 MPa which again appears somewhat low when compared with a value of 550-700 MPa for all 0° material.

Composites GC 210, 214 and 215, slurry B, were fabricated using the [0(0/90)_4]_s lay-up scheme and HMS as the fiber. GC 214 was pressed in vacuum and GC 215 was slow cooled from 1473 K to minimize residual stresses. The strengths of these composites are also tabulated in Table XIV, were similar to the alternate HMS ply lay-up, and were not affected by changes in the fabrication procedures. A higher hot pressing temperature would be expected to improve the strength of the cross-plied composites.

**Woven Cloth**

A two dimensionally woven cloth of Thornel 300 fiber was used to fabricate cross ply specimens. The cloth contained 12 fibers per inch in both the 0° and 90° directions, woven in a simple weave, and was easily cut and handled. The fabrication procedure involved dipping the layers of cloth in a standard slurry of glass followed by stack up of plies and hot pressing. The resultant composite microstructure is shown in Fig. 30 where it can be seen that the fiber content is very high. Glass did, however, penetrate the woven cloth and this type of procedure will be pursued further in the coming months. The major difficulty is that the woven nature of the cloth prevents spreading of the individual tows and hence it is more difficult to get the glass slurry to penetrate the fiber bundle.

A specimen was also prepared wherein the PVA fiber sizing was removed prior to glass impregnation. The surface appearance of this composite is shown in Fig. 31. The fabric weave is clearly visible through the thin glass surface layer and the general appearance was excellent. The degree of internal densification and bonding was not measurably superior, however, to the specimens fabricated without a fiber precleaning step. The fiber-matrix bond was very weak, and the specimens failed by delamination at a shear stress on the order of 3 MPa. The specific data are shown in Table 13 of Appendix B.

**Thermal Expansion**

One of the most interesting and important aspects of graphite fiber reinforced composites is their coefficients of thermal expansion. Because of the very low axial coefficient of thermal expansion of graphite fibers, it has been possible to make resin matrix composites that are exceptionally dimensionally stable in thermal gradients. This has been accomplished despite the high thermal expansion of resin matrices. Based on this successful experience, glass matrix composites would be expected to offer superior dimensional stability due to the much lower thermal expansion of glass, as compared to resins.
Figure 30. Microstructure of Specimen GC 218-5 Thornel 300 Cloth Reinforced 7740, Slurry B
Figure 31. Transparent Glass Surface of Specimen GC 220 0° / 90 Thornel 300
Reinforced 7740 Fabricated Using Woven Cloth, Slurry B
Unidirectionally reinforced 7740 matrix composites were fabricated with Thornel 300 (LB 161E), Celanese DG-102 (LB 97L), and Hercules HMS (GC 216) reinforcements. Elongation of 0° and 90° oriented specimens was measured, and the resultant data are presented in Figs. 32-38 in the form of thermally induced specimen strain as a function of temperature. All tests were begun at room temperature and were repeatedly run up to approximately 823-848 K for at least two complete cycles. There did not appear to be any significant difference in material performance due to cycling so that the data presented, which were taken from the final cycle, are typical of stable material behavior.

The data in Figs. 32 and 33 are for 0° and 90° oriented Celanese DG-102 reinforced 7740, respectively. The 0° data are particularly interesting because they reflect the unique ability of graphite fibers to contract in the axial direction during heating. This negative expansion is reversed at about 573 K so that a net zero change in dimension is the resultant of heating to 823 K. The 90° thermal expansion is always positive and of much larger magnitude than the 0° characteristic primarily due to the glass which essentially controls the transverse expansion.

The thermal expansion of Thornel 300 reinforced glass specimens is presented in Figs. 34 and 35. The 0° data are nonlinear and small in value; however, no evidence of composite contraction was obtained illustrating the importance of fiber type in controlling expansion. As in the case of the Celanese fiber reinforced composite, the 90° expansion is considerably larger than that for the 0° orientation.

The thermal expansion data for the HMS reinforced 7740 are similar to those of the DG-102 reinforced glass. In the case of the 0° orientation, Fig. 36, the composite contracts initially on heating due to the negative thermal expansion of the fibers. The expansion is reasonably linear up to a temperature of approximately 423 K at which time the rate of contraction decreases with a complete reversal to expansion taking place between 573 and 623 K. Although the maximum temperature was 848 K, a net zero change in length would be expected at a temperature of 923 K. Because the glass is reasonably soft at this temperature, permanent deformation of the specimen would be expected, however. The transverse composite thermal expansion, Fig. 37, is much more linear, and positive at all temperatures. The value of coefficient of thermal expansion is dependent on both the radial fiber expansion, and the expansion of the matrix, with the latter having the predominant role.

Values of composite coefficients of thermal expansion were obtained from the above described data and are presented in Table XV. In the cases of non-linearly expanding 0° specimens, the coefficient of expansion varies significantly with temperature range and each calculation was based on drawing a straight line between the low temperature and high temperature strain values.
Figure 32. Thermal Expansion of $0^\circ$ Celanese DG – 102 Fiber Reinforced 7740, Slurry B
Figure 33. Thermal Expansion of 90° Celanese DG - 102 Fiber Reinforced 7740, Slurry B
Figure 34. Thermal Expansion of $0^\circ$ Thornel 300 Reinforced 7740, Slurry B
Figure 35. Thermal Expansion of 90° Thornel 300 Fiber Reinforced 7740, Slurry B
Figure 36. Thermal Expansion of 0° HMS Reinforced 7740, Slurry B
Figure 37. Thermal Expansion of 90° HMS Reinforced 7740 Spec. GC 216, Slurry B
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*Freeman & Campbell, ASTM-STP 497, p 121, 1972
Also presented in the table are data for several resin matrix systems. A comparison between resin and glass matrix composites reveals a major difference in $90^\circ$ values of $a$, with a factor of up to eight decrease in expansion ascribable to the 7740 matrix composites. In all cases the $0^\circ$ values are quite low. Another important point is that the glass matrix data extend up to a temperature of 823 K while resin matrix composite data are limited (by matrix properties) to a maximum temperature of about 423 K.

The above described data have confirmed the unique qualities of graphite reinforced glass matrix composites for use in the construction of dimensionally stable structures. They also confirm the major anisotropy of expansion which can cause microcracking in cross ply composites.

The thermal expansion of a cross ply (0/90$^\circ$) HMS reinforced 7740 composite was measured; the results are displayed in Fig. 38. The measurement was repeated several times, and in no case was there any permanent change in dimension after a full heating and cooling cycle. The measured thermal expansion of the cross ply material is quite low over the entire temperature range and particularly between 295 and 573 K. In this range, a hysteresis effect is also maximized. The hysteresis arises from the significant thermal expansion mismatch of the $0^\circ$ and 90$^\circ$ directions.

**Oxidation Properties**

The effect of oxidation on the strength of graphite fiber reinforced glass matrix composites is summarized in Fig. 39. The specimens from which these data were collected were nominally 0.2 x 0.5 x 6.4 cm in size. After oxidation exposure the surfaces of the specimens assumed a whitish color. Nonetheless, the most important path for oxidation is along the fiber direction. This accounts for the majority of the weight loss experienced by the composites. Therefore any application which minimizes edge exposure should exhibit superior oxidation resistance.

Specific data on the oxidation properties of the graphite fibers at 823 K, the flexural strength and weight loss in HTS and HMS graphite fiber reinforced 7740 and the flexural strengths of DG-102 and pitch type graphite fiber reinforced 7740 are tabulated in Appendix B, Tables 19-31.

As shown in Fig. 39 and the tabulated data, the HMS graphite fiber reinforced 7740 + 2% silica, hot pressed at 1723 K, exhibits superior resistance to oxidation. After 24 hrs at 813 K, the percentage weight loss is 5.6; after 100 hrs, 12.6. In comparison, this matrix reinforced by HTS fiber and hot pressed at 1623 K exhibits a weight loss of 9.7% after 24 hrs and 28.8% after 100 hrs. At an oxidation temperature of 723 K, the weight loss is accordingly much less for the HMS fiber reinforced composite. After 24 hrs, the loss is 0.6% and after 100 hrs, 1.8%.
Figure 38. Thermal Expansion of 0° / 90° HMS Reinforced 7740,
Bars Represent Confidence in these Data Points for this Cycle
Figure 39. Effect of Oxidation on Three-Point Flexural Strength of Graphite Fiber-7740 Glass Matrix Composites with added Silica, Slurry C
In the case of composites fabricated with fibers of the Thornel pitch and Celanese GC-102 types, strength losses of 57 and 68% were suffered after 100 hrs at 813 K. In both cases these results are disappointing since, based on oxidation studies of the bare graphite fibers (Appendix B, Tables 19 and 20), these materials were expected to yield better, not worse, oxidation resistance than found for the HMS graphite reinforced glass composites. These results are not conclusive, however, since composites pressed at 1723 K, found optimum for the HMS composites, need to be evaluated.

In comparing the data in Table 27 of Appendix B which represents recently made samples of HMS graphite fiber reinforced C.G.W. 7740 glass + 2% silica with that of Table 28 of Appendix B for similar samples made a year ago and with a slurry without added silica, two facts must be considered. First, the making of strong graphite fiber reinforced C.G.W. 7740 + 2% SiO₂ has been a gradual learning process and secondly, not only did the older samples lack the beneficial effect of the 2% added silica but they were also hot pressed at 200 K higher temperature. These same two factors must also be considered in comparing Tables 29 and 30, Appendix B, for Celanese graphite fiber reinforced glass matrix composites where Table 29 represents year old data and here the 4 hr data of Table 29 is certainly less accurate than the 24 and 100 hr data of Table 30.
CONCLUSIONS

High strength graphite fiber reinforced borosilicate glass matrix composites can be prepared by hot pressing tapes of powdered glass which has been introduced into graphite yarn from a slurry. By employing colloidal silica as part of the slurry vehicle, a modified borosilicate glass which contains an additional 2% silica has been identified as a composite matrix. With such a matrix, Hercules HMS graphite fiber and a very high hot pressing temperature (i.e. 1723 K), uniaxially reinforced composites have exhibited room temperature flexural strengths on the order of 1000 MPa, elastic moduli of 200 GPa and failure strains of 0.005; furthermore, strengths have been retained after 100 hrs of air exposure at 723 K. These results represent substantial improvements over what has been previously accomplished with graphite fiber reinforced glass matrix composites.

From other data which have been obtained on graphite fiber reinforced glass matrix composites, the following conclusions can be made:

- the flexural strength of borosilicate glass matrix composites increases with temperature up to 875 K which is the softening point of the glass;
- the uniaxially reinforced composites exhibit surprisingly high values of fracture toughness only 40% less than that of a graphite reinforced epoxy composite;
- the composite strength is not reduced by flexural fatigue cycling or by short term thermal cycling between 383 and 833 K; and
- extremely low values of thermal expansion and attendant dimensional stability result from the properties of the composite components.

By suitable orientation of tapes, uniaxial, biaxial and multiaxially reinforced composites can be prepared in the graphite fiber reinforced glass matrix system.
REFERENCES


APPENDIX A

Summary of Graphite Fiber Reinforced Glass Matrix Composites Made
### Table A1

Summary of Graphite Fiber Reinforced Glass Matrix Composites

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<th>Fiber Type</th>
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<th>Temp. (K)</th>
<th>Density (kg/m³)</th>
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All samples with numbers lower than GC 175 are slurry A. Samples GC 175 through GC 294 are slurry B, higher numbered samples are slurry C. Exceptions are noted.
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APPENDIX B

Properties of Selected Graphite Fiber Reinforced Glass Matrix Composites
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<th>Sample Number</th>
<th>Thickness cm</th>
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<p>| LB 135FRC     | 0.150        | 1473                        |                    | 1070 155.0                       |
| CB            | 0.150        |                             |                    | 1010 146.0                       |
| LC            | 0.173        |                             |                    | 1050 152.0                       |
| CC            | 0.150        |                             |                    | 1140 165.0                       |
| TC            | 0.152        |                             |                    | 903 131.0                        |
| TR            | 0.157        |                             |                    | 946 137.0                        |
| TL            | 0.160        |                             |                    | 698 101.0                        |
| LB            | 0.152        |                             |                    | 1050 152.0                       |
| RB            | 0.155        |                             |                    | 983 143.0                        |
|               |              |                             | Average            | 977 142.0                       |</p>
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*Specimen highly deformed so that this number is not strictly valid.
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<td>Average</td>
<td>717</td>
<td>104</td>
</tr>
</tbody>
</table>

*Tested with major and minor spans of 5 cm and 1.27 cm

**Cycled while in argon containing glass tube between 863-903 K and 383 K
  Target temperature 833 K or annealing point of glass matrix

***The 100 cycles extended overnight with result that temperature exceeded
  target temperature (annealing point of glass matrix is 833 K or 1040°F) by 33K
<table>
<thead>
<tr>
<th>Condition</th>
<th>4 Point Bend Strength MPa</th>
<th>10^3 psi</th>
<th>Elastic Modulus GPa</th>
<th>10^6 psi</th>
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<tbody>
<tr>
<td>As Fabricated</td>
<td>548</td>
<td>79</td>
<td>200</td>
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<tr>
<td></td>
<td>532</td>
<td>77</td>
<td>195</td>
<td>28.3</td>
</tr>
<tr>
<td>After 3 cycles between 484 and 48 MPa</td>
<td>325</td>
<td>47</td>
<td>185</td>
<td>26.8</td>
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<tr>
<td>After 20 cycles between 337 and 34 MPa</td>
<td>519</td>
<td>75</td>
<td>186</td>
<td>26.9</td>
</tr>
<tr>
<td>After 20 cycles between 390 and 39 MPa</td>
<td>658</td>
<td>95</td>
<td>194</td>
<td>28.1</td>
</tr>
<tr>
<td>After 20 cycles between 450 and 45 MPa</td>
<td>574</td>
<td>83</td>
<td>210</td>
<td>30.5</td>
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<tr>
<td>After 20 cycles between 520 and 52 MPa</td>
<td>638</td>
<td>92</td>
<td>208</td>
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<tr>
<td>After 100 cycles between 430 and 43 MPa</td>
<td>575</td>
<td>84</td>
<td>207</td>
<td>30.0</td>
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<tr>
<td>After 100 cycles between 445 and 44 MPa</td>
<td>573</td>
<td>83</td>
<td>201</td>
<td>29.1</td>
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</table>
Table B6

Four Point Flexural Strength Data for HIMS Graphite Fiber Reinforced Glass Matrix Composite* (C.G.W. 7740 + SiO2) Made from New Slurry C Span 6.35 cm to 1.90 cm

<table>
<thead>
<tr>
<th>Sample</th>
<th>Flexural Strength</th>
<th>Flexural Modulus</th>
<th>Ω ef</th>
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<tbody>
<tr>
<td></td>
<td>MPa</td>
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<td>GPa</td>
</tr>
<tr>
<td>GC 326</td>
<td>896</td>
<td>130</td>
<td>195</td>
</tr>
<tr>
<td>B3</td>
<td>1131</td>
<td>164</td>
<td>144</td>
</tr>
<tr>
<td>B6</td>
<td>803</td>
<td>116</td>
<td>213</td>
</tr>
<tr>
<td>B9</td>
<td>931</td>
<td>135</td>
<td>207</td>
</tr>
<tr>
<td>B11</td>
<td>853</td>
<td>124</td>
<td>202</td>
</tr>
<tr>
<td>C2</td>
<td>950</td>
<td>138</td>
<td>211</td>
</tr>
<tr>
<td>C5</td>
<td>1105</td>
<td>160</td>
<td>217</td>
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<td>C7</td>
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<tr>
<td>Std Dev</td>
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<td>17.1</td>
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</tbody>
</table>

*Hot pressed at 1723 K, 6.9 MPa pressure, 1 hr dwell time in argon
Table B7

Relation of Hot Pressing Pressure to Three Point Flexural Strength of HMS Graphite Fiber Reinforced 7740 Glass Matrix Composites

All Samples Made at 1473 K

Slurry B

GC-280 - 4.1 MPa

<table>
<thead>
<tr>
<th>Sample</th>
<th>Flexural Strength MPa</th>
<th>Flexural Strength ksi</th>
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</thead>
<tbody>
<tr>
<td>TL</td>
<td>595</td>
<td>86.4</td>
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<tr>
<td>LC</td>
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<tr>
<td>LB</td>
<td>481</td>
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<tr>
<td>TC</td>
<td>387</td>
<td>56.5</td>
</tr>
<tr>
<td>CC</td>
<td>507</td>
<td>73.5</td>
</tr>
<tr>
<td>CB</td>
<td>471</td>
<td>68.3</td>
</tr>
<tr>
<td>RB</td>
<td>513</td>
<td>74.3</td>
</tr>
<tr>
<td>RC</td>
<td>489</td>
<td>70.9</td>
</tr>
<tr>
<td>TR</td>
<td>563</td>
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<tr>
<td>Avg</td>
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<td>73.5</td>
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</table>

GC-257 - 6.1 MPa

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<th>Flexural Strength ksi</th>
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<td>LC</td>
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<td>LB</td>
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<tr>
<td>TC</td>
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<td>CC</td>
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<td>CB</td>
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<td>RB</td>
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</table>

GC-278 - 10.1 MPa

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<tbody>
<tr>
<td>LC</td>
<td>644</td>
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<tr>
<td>TR</td>
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<tr>
<td>RC</td>
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<tr>
<td>RB</td>
<td>644</td>
<td>93.5</td>
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<tr>
<td>TL</td>
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</tr>
<tr>
<td>CB</td>
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<td>114</td>
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<tr>
<td>TC</td>
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<td>91.3</td>
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GC-281 - 13.8 MPa

<table>
<thead>
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<th>Flexural Strength MPa</th>
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<tbody>
<tr>
<td>RB</td>
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<tr>
<td>Avg</td>
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### Table B8

Properties of Three Composite Samples of HMS-7740 Produced Simultaneously  
(Hot Pressed at 1473 K, 13.8 MPa)  
Slurry B

<table>
<thead>
<tr>
<th>Specimen</th>
<th>GC-289 - Top Sample</th>
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<th>GC-289 - Center Sample</th>
<th>Specimen</th>
<th>GC-289 - Bottom Sample</th>
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<tbody>
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<td>Flexural Strength</td>
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<tr>
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<td>MPa</td>
<td>ksi</td>
<td>MPa</td>
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<td>572</td>
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Table B9

Effect of Hot Pressing Temperature on Three-Point Flexural Strength of HMS Fiber Reinforced-7740 Composites Made with New Slurry C at a Pressure of 6.9 MPa

<table>
<thead>
<tr>
<th>Material</th>
<th>Temperature (°C)</th>
<th>Flexural Strength (MPa)</th>
<th>Flexural Strength (kSI)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>1473</td>
<td>284</td>
<td>41.1</td>
</tr>
<tr>
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<td>372</td>
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<td>C1</td>
<td>1573</td>
<td>352</td>
<td>51.1</td>
</tr>
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<td>A1</td>
<td>1623</td>
<td>767</td>
<td>111</td>
</tr>
<tr>
<td>A2</td>
<td>1523</td>
<td>746</td>
<td>108</td>
</tr>
<tr>
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<td>1623</td>
<td>628</td>
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<td>1623</td>
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<td>89.2</td>
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<td>667</td>
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<th>Flexural Strength (kSI)</th>
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<tbody>
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<table>
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<th>Flexural Strength (MPa)</th>
<th>Flexural Strength (kSI)</th>
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<tr>
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</tbody>
</table>
Table B10

Check of Consistency of Test Results Obtainable in Measuring the Three-Point Flexural Strength of HHS Fiber Reinforced-774- Composites Made With 7740 + 2% Silica Slurry C at a Temperature of 1623 K, 6.9 MPa Pressure

<table>
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<th>Three-Point Flexural Strength</th>
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<td>MPa  ksi</td>
<td>MPa  ksi</td>
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<td>A1  767 111</td>
<td>B1  565 81.9</td>
<td>A1  700 102</td>
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<td>A2  746 108</td>
<td>B2  689 99.9</td>
<td>A2  755 110</td>
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<tr>
<td>A3  628 91.0</td>
<td>B3  623 90.4</td>
<td>A3  521 75.5</td>
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<td>B4  727 105</td>
<td>A4  563 81.7</td>
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<td>A6  630 91.4</td>
<td>B6  774 112</td>
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<td>A7  637 92.5</td>
<td>B7  466 67.7</td>
<td>A7  538 78.0</td>
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<td>A8  667 96.8</td>
<td>B8  586 84.9</td>
<td>A8  642 93.1</td>
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<td>A9  456 66.1</td>
<td>B9  818 119</td>
<td>A9  720 104</td>
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<td>A10 622 90.1</td>
<td>B10 536 77.7</td>
<td>A10 556 80.6</td>
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<td>A11 682 98.9</td>
<td>B11 635 92.1</td>
<td>A11 547 72.3</td>
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<td>A12 712 103</td>
<td>B12 733 106</td>
<td>A12 642 93.1</td>
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<td>Avg  639 92.7</td>
<td>Std Dev  87.6 12.7</td>
<td>Std Dev  87.9 12.6</td>
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<td>Std Err  18.5 2.68</td>
<td>Std Err  29.8 4.32</td>
<td>Std Err  25.1 3.64</td>
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Table B11

Three Point Flexural Strength of Thorne1 Pitch VS 0032 Fiber Reinforced 7740 + 2% SiO$_2$ Glass Composite (GC-209)
Hot Pressed at 1623 K, 6.9 MPa, 1 Hr Dwell Time
Slurry B

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<td>A3</td>
<td>465</td>
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<tr>
<td>A5</td>
<td>491</td>
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<td>A7</td>
<td>579</td>
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<td>399</td>
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<td>A11</td>
<td>335</td>
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<td>B1a</td>
<td>574</td>
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<tr>
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<td>489</td>
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<tr>
<td>B7</td>
<td>523</td>
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<td>294</td>
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<td>B11</td>
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<td>Avg</td>
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<td>Std Dev</td>
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### Table B12

**Three Point Bend Data for Transversely Oriented Specimens**

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<tr>
<th>Specimen</th>
<th>Fiber</th>
<th>Lay-up</th>
<th>Max. Flex. Stress $10^3$ psi</th>
<th>S/D#</th>
<th>Fracture</th>
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<tbody>
<tr>
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<td>HNS</td>
<td>90°</td>
<td>1.4 9.3</td>
<td>40</td>
<td>Tensile</td>
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<tr>
<td></td>
<td></td>
<td>(4 plys in thickness) 13.8 MPa</td>
<td>1.3 9.1</td>
<td>40</td>
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<tr>
<td></td>
<td></td>
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<td>1.0 6.8</td>
<td>40</td>
<td>&quot;</td>
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<td></td>
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<td>2.0 13.6</td>
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<tr>
<td>Avg</td>
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<td>1.4 9.7</td>
<td></td>
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<tr>
<td>GC 209</td>
<td>HNS</td>
<td>90°</td>
<td>1.8 12.7</td>
<td>10</td>
<td>Tensile</td>
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<tr>
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<td>(16 plys in thickness) 13.8 MPa</td>
<td>3.2 22.2</td>
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<td>1.4 9.5</td>
<td>10</td>
<td>&quot;</td>
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<tr>
<td>Avg</td>
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<td></td>
<td>2.15 14.8</td>
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</tr>
<tr>
<td>GC 221**</td>
<td>HTS</td>
<td>90°</td>
<td>1.8 12.6</td>
<td>14</td>
<td>Tensile</td>
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<tr>
<td></td>
<td></td>
<td>10 plys thick</td>
<td>0.7 4.7</td>
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<td>1.3 9.1</td>
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<td>&quot;</td>
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<td></td>
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<td>1.4 9.8</td>
<td>14</td>
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<tr>
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<td>1.4 9.5</td>
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<tr>
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<td>90°</td>
<td>2.1 14.7</td>
<td>12</td>
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<td>10 plys thick</td>
<td>1.4 9.3</td>
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<td>1.8 12.1</td>
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<td>1.0 6.9</td>
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<td>&quot;</td>
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<td>2.6 18.0</td>
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<tr>
<td>Avg</td>
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<td>1.8 12.0</td>
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*Span-to-depth ratio for 3 point bend test*

**Fabricated using 13.8 MPa pressure**

***Fabricated using 6.9 MPa pressure***
Table B13
Three Point Bend Data for
Thorne 300 Fabric Reinforced 7740 Glass
Slurry B

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Precleaned Fiber</th>
<th>Flexural Strength MPa</th>
<th>Flexural Strength ksl</th>
<th>Shear Strength MPa</th>
<th>Shear Strength ksl</th>
<th>S/D*</th>
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<tr>
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<td>0.32</td>
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<td>13.5</td>
<td>2.05</td>
<td>0.30</td>
<td>5</td>
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<td>15.7</td>
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*Span-to-depth ratio of test
Table B14

Oxidized Fiber Strength Loss

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<th>HMS</th>
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<th>Celanese BG-102</th>
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<td>Vendor supplied fiber</td>
<td>2446</td>
<td>2873</td>
<td>1723</td>
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<td>Average UTS (MPa)</td>
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<tr>
<td>As-received average fiber</td>
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<td>2777</td>
<td>2074</td>
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<tr>
<td>UTS measured at UTRC (MPa)</td>
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<tr>
<td>Average fiber strength after exposure to air at 823 K for 1 hr (MPa)</td>
<td>1550*</td>
<td>792*</td>
<td>1054*</td>
</tr>
<tr>
<td>% of fiber UTS lost based on UTRC measurements</td>
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<td>71%</td>
<td>49%</td>
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<td>% weight loss of fiber after exposure to air at 823 K for 1 hr</td>
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<td>84%</td>
<td>16%</td>
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*Strength calculated based on original unexposed average fiber diameter
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<th>Fiber</th>
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<th>6 hours</th>
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<td>no data</td>
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<td>2446</td>
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<td>Celanese DG-102</td>
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<td>85.2</td>
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<td>1723</td>
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<tr>
<td>Thornei 50</td>
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<tr>
<td>Thornei Pitch Type</td>
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<td>17.3</td>
<td>47.3</td>
<td>413</td>
<td>*944</td>
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*Material currently shipped has strength greater than 1378 MPa
Table B16

Effect of Exposure to Air for 4 hrs at 833 K on the
3 Point Flexural Strength of HTS Graphite Fiber
Reinforced 7740, Hot Pressed at 1473 K
Slurry B

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<th>Specimen</th>
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<td>Exposed Fibers</td>
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<td>-CC</td>
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<tr>
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<td>Avg</td>
<td>334          48.5</td>
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Table B17

Effect of Heat Treatment in Argon and Air on 3 Point Strength of Hercules HTS Graphite Fiber in 7740 Glass Matrix Hot Pressed at 1473 K Slurry C
Table B18

Effect of Heat Treatment in Vacuum and in Air on 3 Point Flexural Strength of Hercules HTS Graphite Fiber in C.G.W. 7740 (Pyrex) Glass Matrix
1473 K - Hot Pressed, Slurry B

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Table B19

Effect of Air Exposure at 813 K on the Three Point Flexural Strength and Weight Loss of HTS Graphite Fiber Reinforced 7740, GC 304, Pressed at 1623 K, Slurry C

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### Table B21

Effect of Air Exposure at 813 K on Weight Loss and Flexural Strength of H/M Graphite Fiber Reinforced 7740 + 2% Silica, GC 326, Pressed at 1723 K and GC 327, Pressed at 1773 K Slurry C

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## Table B22

**Improved Oxidation Resistance of HMS Fiber-7740 Composites**

Made by Slurry C at 1673 K and 6.9 MPa

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<td>645                 93.5</td>
<td>C10            647        93.8</td>
<td>B10            642        93.1</td>
</tr>
<tr>
<td>A11</td>
<td>778                 113</td>
<td>C11            625        90.6</td>
<td>B11            650        94.2</td>
</tr>
<tr>
<td>A12</td>
<td>806                 117</td>
<td>C12            663        96.2</td>
<td>B12            559        81.1</td>
</tr>
<tr>
<td>Avg</td>
<td>834                 121</td>
<td>738            107</td>
<td>605            87.8</td>
</tr>
<tr>
<td>Std Dev</td>
<td>107                15.5</td>
<td>80.0           11.6</td>
<td>49.4           7.17</td>
</tr>
<tr>
<td>Std Err</td>
<td>32.2               4.67</td>
<td>23.0           3.34</td>
<td>14.3           2.07</td>
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Table B23

Effect of Air Heat Treatment on 4 Point Flexural Strength of Hercules HMS Graphite Fiber in 7740 Glass Matrix
Spans 1.25 cm and 5.0 cm
Slurry A

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Heat Treatment</th>
<th>4 Point Flexural Strength</th>
<th>Modulus</th>
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<tbody>
<tr>
<td></td>
<td></td>
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<td>ksi</td>
</tr>
<tr>
<td>LB 135T-LC</td>
<td>none</td>
<td>596</td>
<td>86.5</td>
</tr>
<tr>
<td>-BC</td>
<td></td>
<td>699</td>
<td>101</td>
</tr>
<tr>
<td>-CC</td>
<td></td>
<td>594</td>
<td>86.1</td>
</tr>
<tr>
<td>-TC</td>
<td></td>
<td>487</td>
<td>70.7</td>
</tr>
<tr>
<td>-RB</td>
<td></td>
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<td>66.3</td>
</tr>
<tr>
<td>Avg</td>
<td></td>
<td>566</td>
<td>82.2</td>
</tr>
<tr>
<td>-LB</td>
<td>4 hrs, 833 K, air</td>
<td>345</td>
<td>50.0</td>
</tr>
<tr>
<td>-RC</td>
<td></td>
<td>286</td>
<td>41.5</td>
</tr>
<tr>
<td>-TL</td>
<td></td>
<td>413</td>
<td>59.9</td>
</tr>
<tr>
<td>-TR</td>
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<td>476</td>
<td>69.1</td>
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<td>Avg</td>
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<td>380</td>
<td>55.3</td>
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Table B24

Effect of Heat Treatment in Argon on 4 Point Flexural Strength of Celanese DG-102 Graphite Fiber in 7740 Glass Matrix

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Heat Treatment</th>
<th>Flexural Strength (MPa)</th>
<th>Flexural Strength (ksi)</th>
<th>Modulus (GPa)</th>
<th>Modulus (10^6 psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LB 97E-TL</td>
<td>none</td>
<td>239</td>
<td>34.7</td>
<td>354</td>
<td>51.4</td>
</tr>
<tr>
<td>-LC</td>
<td></td>
<td>258</td>
<td>37.5</td>
<td>327</td>
<td>47.5</td>
</tr>
<tr>
<td>-LB</td>
<td></td>
<td>262</td>
<td>38.0</td>
<td>285</td>
<td>41.4</td>
</tr>
<tr>
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<td></td>
<td>253</td>
<td>36.7</td>
<td>322</td>
<td>46.8</td>
</tr>
<tr>
<td>LB 97E-TR</td>
<td>4 hrs, 833K, argon</td>
<td>289</td>
<td>41.9</td>
<td>277</td>
<td>40.2</td>
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<tr>
<td>-CR</td>
<td></td>
<td>301</td>
<td>43.7</td>
<td>269</td>
<td>39.0</td>
</tr>
<tr>
<td>-BR</td>
<td></td>
<td>336</td>
<td>48.7</td>
<td>271</td>
<td>39.3</td>
</tr>
<tr>
<td>Avg</td>
<td></td>
<td>308.7</td>
<td>44.8</td>
<td>272</td>
<td>39.5</td>
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Table B25

Apparent Failure to Improve Oxidation Resistance of Céianese DG-102 Graphite Fiber-7740 Glass Composites Made by Slurry C at 1623 K and 6.9 MPa

Compare Table B24

<table>
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<tr>
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<th>GC 331</th>
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</thead>
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<tr>
<td></td>
<td>As Made</td>
<td>After 24 hrs in Air @ 813 K</td>
<td>After 100 hrs in Air @ 813 K</td>
<td></td>
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<tr>
<td></td>
<td>Flexural Strength</td>
<td>Flexural Strength</td>
<td>Flexural Strength</td>
<td>Flexural Strength</td>
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<tr>
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<td>MPa</td>
<td>ksi</td>
<td>MPa</td>
<td>ksi</td>
<td>MPa</td>
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<td>221</td>
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<td>327</td>
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<tr>
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<td>60.3</td>
<td>A12</td>
<td>117</td>
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<td>351</td>
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<td>B3</td>
<td>306</td>
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<td>B9</td>
<td>326</td>
<td>47.3</td>
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<td>47.9</td>
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<td>C9</td>
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<tr>
<td>Std Err</td>
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- GC 331 Three Point Flexural Strength
- As Made
- After 24 hrs in Air @ 813 K
- After 100 hrs in Air @ 813 K

<table>
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<th>MPa</th>
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<td>Std Err</td>
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Table B26

Apparent Failure to Improve Oxidation Resistance of Pitch Type Fiber-7740 Composites Made by New Process at 1623 K and 6.9 MPa
Slurry C

<table>
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<tr>
<th>As Made</th>
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<td>GC 310 - Thornel Pitch Type Graphite Fibers</td>
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<td>MPa</td>
<td>ksi</td>
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