CHAPTER II

EXPERIMENTAL
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2.1 MATERIALS

Cellulose acetate butyrate (CAB 381-20), henceforth denoted as CAB, was obtained in powder form from Eastman Chemical Company, Kingsport, Tennessee. The average acetyl and butyryl contents were 13.5 and 38.1 wt.\% and the falling-ball viscosity was 20 seconds (76 poise). The number average molecular weight (Mn) was 69,600 with a molecular weight distribution (MWD) of 1.83.

Lyocell fabric and continuous filament tows were supplied by Acordis, The Netherlands. The fabric was plain weave, 3.5K continuous filament cloth, with a basis weight of 450 gm/m². The individual yarns had 3500 filaments, with individual filaments having 1.49 dTex. The tows consisted of 16,700 fibers, each 12 \( \mu \)m in diameter and a specific gravity of 1.32 (see Appendix 1, Chapter 2 for calculations). The fibers were spun with a line speed of 60m/min.
Surfactant (TRITON X-100™) was obtained from Aldrich Chemical Company, St. Louis, MO (Catalog number 23,472-9). Hydroxypropyl xylan (HPX) was prepared in-house, following the procedure of Jain et al. [61].

2.2 METHODS

2.2.1 Composite Prepregging

i. Powder Suspension Process

The matrix (CAB 381-20) powder suspension was prepared in water with two different dispersants, TRITON X-100™ and Hydroxypropyl Xylan (HPX). CAB powder was added to distilled water at room temperature, and 2% by weight (of total solution) of dispersant was added to the mixture. The suspension was stirred for 30 min with a magnetic stir bar. The concentrations of the suspension varied from 10-20% by weight. Samples were collected from the suspension and the particle size distribution was determined. Some of the suspensions were sonicated with a Tekmar model TM 300 sonic disrupter at a power level of 75 W for 2 min.

Particle size distribution of the suspensions was determined with a Shimadzu Centrifugal Particle Size Analyzer Model SA-CP3 from Shimadzu Corporation, Kyoto, Japan. Particle sizes were measured by the sedimentation method based on Stoke’s Law. Sedimentation from an initially uniform suspension was employed. The photometric method adopted in the SA-CP3 utilizes the fact that the intensity of transmitted light increases as the particle concentration decreases.

Cellulose fiber tow was impregnated with CAB powder using a fiber prepregging machine from Kamitsu Seisakusho Ltd., Japan [Fig.2.1a]. The same process of matrix
application applies to the solution process also. The tension of the fiber tow was controlled by a load cell in order to spread the fiber tow sufficiently in the suspension bath [Fig.2.1b]. The line speed was varied to obtain sufficient residence time of the fiber tow in the bath. The horizontal displacement on the winding drum controlled the degree of overlap of the fiber tows [Fig.2.1c]. After the prepregging operation, the prepreg was left on the drum to dry for 2 hours before it was taken off the drum and cut into 6”x 6” panels. Before the panels were consolidated, the panels were dried in a convection oven at 100°C for 24 hours and then in a vacuum oven at 40°C under 27.5 inch of Hg for 12 hours.

Figure 2.1a: Schematic illustration of the prepregging machine used for matrix application on continuous lyocell fibers.
Figure 2.1b: Picture depicting the fiber tow passing through the solution bath and spreading of the tow.

Figure 2.1c: Picture depicting the fiber tow being wound up on the take-up drum after passing though the drying tower. The overlap of the tow is controlled by the horizontal movement of the take-up drum.
ii. Melt Process

In case of melt processing of the composites, thin sheets of CAB (thickness of approximately 1-1.5 mm) were prepared by compression molding CAB powder in a hot press at 210°C. The CAB sheets were then placed in between the fiber mats so as to form a sandwich preform. The sandwich was consolidated in a hot press at 210 – 220°C. Pressure was applied to some of the composites in order to squeeze out matrix to achieve a greater matrix content in the composite.

iii. Solution Process

Solutions of CAB were prepared in MEK at room temperature. The solution concentrations varied from 5% to 12% of CAB by weight. The fiber mats (fabric) were dipped in the solution and then dried at room temperature for 12-24 hours. For some prepregs the panels were dipped several times (not more than 3 times) to obtain the required amount of CAB (ca. 40% by weight of CAB, measured by the weight gain of the prepregs after dipping in CAB solution) in the prepeg. The prepregs were kept in a vacuum oven at 40°C for 12 hours to remove any solvent.

For continuous fiber prepregging, the continuous fiber tow was guided through a bath of CAB solution in MEK (conc. varying from 8% to 12% by weight) using a fiber prepregging machine from Kamitsu Seisakusho Ltd., Japan [Fig. 2.1]. The tension of the fiber tow was controlled by a load cell in order to spread the fiber tow. The line speed was kept at 0.75 to 1.00 m/min to obtain sufficient residence time of the fiber tow in the bath. The horizontal displacement on the winding drum controlled the degree of overlap of the fiber tows. After the prepregging operation, the prepreg was taken off the drum and
cut into 6”x 6” panels for consolidation. The prepregs were kept in a vacuum oven at 40ºC for 12 hours to remove any solvent. The individual prepregs were stitched transverse to the fiber direction using a sewing machine in order to avoid dis-alignment of fibers during processing and consolidation. A high strength nylon fiber was used for stitching.

2.2.2 Composite Consolidation

i. For Fabric Reinforced Composites (Panels B1 to B5)

The composite panels or prepregs in each of the above cases were stacked into layers and consolidated under heat and pressure in a hot press. The temperature was kept constant at 210-220 ºC (measured by a thermocouple placed in between the two flat molds) while the pressure was varied for each process as shown in Table 2.1. The molds were preheated to the desired temperature before the prepregs were consolidated.

ii. For Continuous Fiber Reinforced Composites (Panels C1 to D4)

The prepregs were subjected to a ramp and hold temperature-pressure cycle as shown in Fig. 2.2. The temperature and pressure in the consolidation cycle varied for each composite and the values are mentioned in Tables 2.2 and 2.3. The cycle time was 10 min and the platens were preheated to the desired temperatures. The criteria for the consolidation conditions (the consolidation cycle) was not optimized in this study. The consolidation cycle was chosen as shown in Fig. 2.2 because adequate flow of matrix (reduction in viscosity) was desired before any pressure was applied. The pressure was applied after 4-5 ins so that the composite panel reached the constant temperature of the
hot platens within this time and adequate reduction of matrix viscosity is achieved. The prepregs were stacked into layers between two flat metal plates, which were at room temperature before being inserted into the hot press. Several prepreg assemblies contained CAB-films that were inserted in between layers of fiber prepregs so as to allow the manufacture of composites with greater matrix fraction (Table 2.3). The consolidation was carried out under a vacuum of 27.5 inches of Hg. Other details of the processing stages are specified in Tables 2.2 and 2.3.
2.2.3 Composite Characterization

i. C-Scan

After consolidation, the composite panels were evaluated with a Sperry Corporation S-80 C-scan ultrasonic scanning unit. This nondestructive test determines the voids and uniformity of matrix dispersion in the panels. The panels were dipped in water. Only one panel was evaluated with this method since the panels absorbed water while testing. The image of the C-scan is shown in Fig.2.3 The densities of the matrix and the fiber being very close to each other, C-Scan image analysis failed to provide clear indications of any presence of voids. It was difficult to distinguish between matrix filled regions and void-filled regions as the contrast for these regions was not sharp. Moreover, the specimens were immersed in a bath containing water and so water penetration into the
composite led to further problems in the analysis. Water filled domains are seen in the four corners of the panel in Fig. 2.3. Therefore, further analysis by C-Scan was not carried out for the other composites. Other viable methods of analyzing the composite panels may be the x-ray technique.

**ii. Fiber Content Analysis**

**a. Soxhlet Extraction Method**

The fiber content analysis was performed by a soxhlet extraction, which is similar to the ASTM D 3171-76 method [50]. Specimens were cut from the composite panels, dried under vacuum at 35°C for 12 hours and kept in a dessicator for 48 hours to remove moisture. The weights of the specimens ($W_S$) and weight of each thimble ($W_T$) were noted and each specimen was placed in a ‘Watman cellulose thimble’, which was also kept in a dessicator for 48 hours to remove moisture. The thimbles were repeatedly immersed in acetone with continuous recycling of the solvent in a Soxhlet apparatus. The process was continued for 72 hours. The residue was filtered, dried first under vacuum for 12 hours and then kept in a dessicator for 48 hours. Total weight of the cellulose fiber and the thimble was measured. The residual weight ($W_R$) of the composite specimen after extraction was obtained by subtracting the weight of the thimble from the total weight measured. The weight content of fiber was determined by dividing the residual weight by the initial weight of the specimen.

\[
\text{Fiber Content (wt. %)} = \frac{(W_R - W_T)}{W_S} \quad (2.1)
\]

where,
WR = Residual Weight after extraction (includes thimble),

WT = Weight of thimble,

WS = Initial weight of sample

b. Aminolysis Method

The aminolysis method is based on quantitative determination of 1-butyrylpyrrolidine, which is obtained in the deacylation reaction between cellulose ester and pyrrolidine according to the following reaction mechanism provided by Mansson et al. [49]. Only the matrix contains butyryl groups.

\[
\text{R-C-OR} + \text{N} \rightarrow \text{R-C-N} + \text{RO}^{-}
\]

A stock solution was prepared by dissolving an internal standard, 1-hexylpyrrolidine, in a mixture of pyridine and pyrrolidine (1:1). The concentration of the internal standard was 10%. The stock solution (2ml) was added to weighed amounts of composite samples to be analyzed in a vial fitted with a plastic cap. The reaction mixture was kept at 80°C for 18 hours with continuous stirring. The solid material was filtered and the filtrate was analyzed in a gas chromatograph by Hewlett Packard model 6890. The response peak areas of butyryl and hexanoyl pyrrolidine derivative were noted and the CAB content in the composite was calculated as mentioned in Ref 49.
c. **Fiber Volume Fraction Determination**

Fiber content (volume %) is calculated on a voidless basis as follows:

\[
\text{Fiber Content (Vol. %)} = 100 \frac{W_f}{\rho_f} / \left[ \frac{W_f}{\rho_f} + \frac{W_m}{\rho_m} \right]
\]  

(2.2)

where,

- \(W_f\) = Weight fraction of fiber in composite,
- \(W_m\) = Weight fraction of matrix in composite,
- \(\rho_f\) = Density of fiber,
- \(\rho_m\) = Density of matrix.

Densities of Fiber and CAB matrix are 1.5 gm/cc and 1.2 gm/cc, respectively.

iii. **Theoretical Density Determination of Composites (Voidless Basis)**

The densities of the fiber and matrix were determined by the displacement method according to ASTM D 792-91 [53]. Specimens were weighed in air and then they were weighed after being immersed in distilled water (sp. gravity of 1.0) or isopropanol (sp.gravity of 0.785). The difference in the weights in air and liquid is the buoyancy force on the specimen. The buoyancy force equals the weight of the same volume of water as the volume of the specimen. Therefore, the volume of the specimen is

\[
V_S = \frac{\text{Weight in air} - \text{Weight in liquid}}{\rho_{\text{liquid}}}
\]  

(2.3)

where,

- \(\rho_{\text{liquid}}\) is the density of liquid.
The density of the specimen is then calculated by dividing the weight of the specimen by the volume. The theoretical density of the composite assumes that there are no voids present in the composite.

\[ T_d = \frac{100}{(M/\rho_m + F/\rho_f)} \]  

(2.4)

where:

\( T_d \) = Theoretical density (voidless basis),
\( M \) = Matrix in composite, weight %,
\( F \) = Fiber in composite, weight %,
\( \rho_m \) = Density of matrix,
\( \rho_f \) = Density of fiber.

iv. Void Content Determination

The densities of the composites were determined by the displacement method according to ASTM D 792-91 [53] discussed earlier. Void contents of the composites were determined according to the ASTM D 2734-94 standard procedure [54]. The void content was determined as follows:

\[ V = 100 \left( \frac{T_d - M_d}{T_d} \right) \]  

(2.5)

where:

\( V \) = Void Content, volume %,
\( T_d \) = Theoretical composite density, voidless basis,
\( M_d \) = Measured composite density.
v. Mechanical Tests

Tensile tests were performed according to the ASTM D 3039 procedure [51] using an Instron Tensile Testing Machine model 4204 with a load beam of 50 KN. The crosshead speed was kept at 1 mm/min for all tests. At least 5 tensile specimens were cut from the composite panels using a band saw. The specimen dimensions were approximately 1.12 mm in width and 15.24 mm in length. The tests were conducted at room temperature (23°C). The thickness of the specimens varied from one another according to the thickness of the panels. Each data point reported represents the average of at least 4 measurements.

vi. Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) of the composite specimens were performed using an AMRAY 180 D (Diffusion pump system) Scanning Electron Microscope at an operating voltage of 15 kV. Specimens of the composite panels were mounted on a flat aluminum surface and sputter coated with gold in a Denton vacuum DV 515 evaporator. The specimen surfaces investigated consisted of untested as well as the failure surfaces of the tested samples.

vii. Determination of Water Absorption

This method determines the relative rate of absorption of water by the composites when immersed in water. The tests were performed according to the ASTM D570-95 [ASTM D570-95, 1997] standard procedure for two and twenty four hour immersions (i.e. repeated immersion). Specimens of the composite panels were cut into dimensions of
76.2 mm (3 inch) in length by 25.4 mm (1 inch) in width by the thickness of the panel. The specimens were dried in an oven at 110°C, cooled in a desiccator and weighed. The conditioned specimens were immersed in distilled water maintained at 23°C. The specimens were fully immersed in water. The two-hour immersion method is used for materials having a relatively high rate of absorption and thin specimens, which can gain an equilibrium water content in 2 hours. For the composite panels under consideration, both the two hours and twenty-four hours methods were used to compare the rate of absorption of water. Therefore, the specimens were removed from water after 2 hours one at a time, all surfaces were wiped off, and weighed. They were again placed in water, and weighed again after another 22 hours. Since there are no water soluble materials present in the composites, there was no need for any reconditioning. The percentage of water absorbed was calculated as follows

\[
W_{\text{Absorbed}} = \left(\frac{W_{\text{Wet}} - W_{\text{Conditioned}}}{W_{\text{Conditioned}}}\right) \times 100 \tag{2.6}
\]

where,

- \(W_{\text{Absorbed}}\) = Amount of water absorbed (weight gain in %),
- \(W_{\text{Wet}}\) = Wet weight of sample,
- \(W_{\text{Conditioned}}\) = Conditioned weight of sample before immersed in water.

**viii  Differential Scanning Calorimetry (DSC)**

Thermal analysis of the samples was carried out using a Perkin-Elmer Model DSC-4 equipped with a Thermal Analysis Data Station (TADS) using standard aluminum pans. Measurements were made on ca. 5-20 mg samples in the temperature range between 23
and 240°C at a scanning rate of 10°C/min. Nitrogen was used as a sweeping gas. The instrument was calibrated with an indium standard. The glass transition temperature was reported from the second heating scan, unless otherwise indicated. The glass transition temperature ($T_g$) was taken as the temperature at the midpoint ($1/2 \Delta C_p$) of the transition.
Appendix 1: Conversion of Fiber Density from Textile Units

Linear density of a filament = 1.49 decitex = 0.149 tex = 0.149 gm/1000m of fiber = 0.149x10^{-3} gm/m of fiber.

Assuming a circular cross-section of each filament, from SEM images, the filament diameter (d) was found to be ca. 12µm = 12x10^{-6} m

Volume (V) of each filament (assuming a cylindrical cross-section) = \( \pi (d/2)^2 L \)
where, D = Diameter and L = Length of the filament

Therefore, \[ V = \pi (12\times10^{-6}/2)^2 \times 1 \text{ m}^3/\text{m of filament} = 1.131 \times 10^{-10} \text{ m}^3/\text{m of filament}. \]

Density of the filament = weight/volume = \( \frac{0.149\times10^{-3}}{1.131\times10^{-10}} \text{ gm/}\text{m}^3 = 1.32 \times 10^6 \text{ gm/m}^3 = 1.32 \text{ gm/cm}^3 \)

Specific gravity of fiber = 1.32
## Table 2.1: Specifications of the Fabric Reinforced Composite Panels.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Prepregging Process</td>
<td>Solvent (MEK)</td>
<td>Solvent (MEK)</td>
<td>Melt</td>
<td>Melt</td>
<td>Melt</td>
</tr>
<tr>
<td>Fiber Type</td>
<td>Fabric</td>
<td>Fabric</td>
<td>Fabric</td>
<td>Fabric</td>
<td>Fabric</td>
</tr>
<tr>
<td>Solution Conc (wt. %)</td>
<td>10</td>
<td>10</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>No of dips in solution</td>
<td>2</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>No of plies / layers of fibers</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>No of layers of CAB film.</td>
<td>-</td>
<td>-</td>
<td>2</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>Prepregging</td>
<td>Each ply separately(^1)</td>
<td>Both plies together(^2)</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Release cloth used</td>
<td>Kapton (polyimide)</td>
<td>Teflon</td>
<td>Kapton (polyimide)</td>
<td>Kapton (polyimide)</td>
<td>Kapton (polyimide)</td>
</tr>
<tr>
<td>Molding Temp (°C)</td>
<td>210-220</td>
<td>210-220</td>
<td>210-220</td>
<td>210-220</td>
<td>210-220</td>
</tr>
<tr>
<td>Molding time (sec.)*</td>
<td>80</td>
<td>45</td>
<td>180</td>
<td>240</td>
<td>240</td>
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<tr>
<td>Molding Pressure (psi)</td>
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<td>100</td>
<td>10</td>
<td>no pressure</td>
<td>no pressure</td>
</tr>
<tr>
<td>Approx fiber Content (wt.%)**</td>
<td>60.3</td>
<td>48.9</td>
<td>40.2</td>
<td>28.6</td>
<td>18.0</td>
</tr>
</tbody>
</table>

* It is suspected that the molding time can be reduced to 2-3 secs. when it is processed on an industrial scale. The processing time was kept high enough to ensure total melt flow of the matrix in these tests.  
** The fiber content was determined by soxhlet extraction (See Table 3.2).  
\(^1\) Each fabric ply was dipped and dried separately into the solution bath. They prepregs were then assembled together before consolidation.  
\(^2\) Both the fabric plies were dipped and dried as an assembly (together).
Table 2.2: Specifications of the Continuous Fiber Reinforced Composite Panels using Two Coats of CAB Matrix.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Panel C1</th>
<th>Panel C2</th>
<th>Panel C3</th>
<th>Panel C4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prepregging Process</td>
<td>Solvent (MEK)</td>
<td>Solvent (MEK)</td>
<td>Solvent (MEK)</td>
<td>Solvent (MEK)</td>
</tr>
<tr>
<td>Fiber Type</td>
<td>Tow</td>
<td>Tow</td>
<td>Tow</td>
<td>Tow</td>
</tr>
<tr>
<td>Solution Conc (wt. %)</td>
<td>8% (1st Coat)</td>
<td>8% (1st Coat)</td>
<td>8% (1st Coat)</td>
<td>8% (1st Coat)</td>
</tr>
<tr>
<td></td>
<td>12% (2nd coat)</td>
<td>12% (2nd coat)</td>
<td>12% (2nd coat)</td>
<td>12% (2nd coat)</td>
</tr>
<tr>
<td>No of dips/coats in solution</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Line Speed (m/min)</td>
<td>0.6</td>
<td>0.6</td>
<td>0.6</td>
<td>0.6</td>
</tr>
<tr>
<td>Tow Spacing (mm/rotation)</td>
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<td>1.7</td>
<td>1.7</td>
<td>1.7</td>
</tr>
<tr>
<td>No of plies/ layers of fibers</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Fiber orientation</td>
<td>Unidirectional</td>
<td>Crossply</td>
<td>Unidirectional</td>
<td>Unidirectional</td>
</tr>
<tr>
<td>No of layers of CAB film added before consolidation</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Release cloth used</td>
<td>Kapton (polyimide)</td>
<td>Kapton (polyimide)</td>
<td>Kapton (polyimide)</td>
<td>Kapton (polyimide)</td>
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<tr>
<td>Molding Temp (°C)</td>
<td>210</td>
<td>210</td>
<td>195</td>
<td>180</td>
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<tr>
<td>Molding time (min)*</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Molding Pressure (psi)*</td>
<td>27</td>
<td>27</td>
<td>27</td>
<td>27</td>
</tr>
<tr>
<td>Vacuum in molding chamber (inch of Hg)</td>
<td>27.5</td>
<td>27.5</td>
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<tr>
<td>Approx fiber Content (wt.%)**</td>
<td>68.3</td>
<td>72.9</td>
<td>71.3</td>
<td>67.8</td>
</tr>
</tbody>
</table>

* The molding time-temp and pressure cycle is shown in Fig. 2.1.
** The fiber content was determined by soxhlet extraction (See Table 3.2).
### Table 2.3: Specifications of the Continuous Fiber Reinforced Composite Panels using One Coat of CAB Matrix.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Panel D1</th>
<th>Panel D2</th>
<th>Panel D3</th>
<th>Panel D4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prepregging Process</td>
<td>Solvent (MEK)</td>
<td>Solvent (MEK)</td>
<td>Solvent (MEK)</td>
<td>Solvent (MEK)</td>
</tr>
<tr>
<td>Fiber Type</td>
<td>Tow</td>
<td>Tow</td>
<td>Tow</td>
<td>Tow</td>
</tr>
<tr>
<td>Solution Conc (wt. %)</td>
<td>12%</td>
<td>12%</td>
<td>12%</td>
<td>12%</td>
</tr>
<tr>
<td>No of dips/coats in solution</td>
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<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Line Speed (m/min)</td>
<td>0.6</td>
<td>0.6</td>
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</tr>
<tr>
<td>Tow Spacing (mm/rotation)</td>
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<tr>
<td>No of plies / layers of fibers</td>
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<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Fiber orientation</td>
<td>Unidirectional</td>
<td>Unidirectional</td>
<td>Unidirectional</td>
<td>Unidirectional</td>
</tr>
<tr>
<td>No of layers of CAB film added before consolidation</td>
<td>0 (between plies)</td>
<td>1 (on top and bottom)</td>
<td>2 (top, bottom and between plies)</td>
<td>3 (top, bottom and between plies)</td>
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<td>Kapton (polyimide)</td>
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<td>Molding Temp (°C)</td>
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<td>180</td>
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<tr>
<td>Molding time (min)*</td>
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<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Molding Pressure (psi)*</td>
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<tr>
<td>Vacuum in molding chamber (inch of Hg)</td>
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<td>27.5</td>
<td>27.5</td>
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<tr>
<td>Approx fiber Content (wt. %)**</td>
<td>74.2</td>
<td>45.8</td>
<td>32.8</td>
<td>25.5</td>
</tr>
</tbody>
</table>

*, ** Same as Table 2.2

1) The approximate thickness of each CAB film was in the range 0.75 to 1.5 mm.