3M™ Nextel™ 610 Towpreg: A new pathway for oxide ceramic matrix composite fabrication

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Automated fiber placement

- Automated Fiber Placement (AFP) is a digital additive manufacturing process where multiple tows can be independently added or cut in the fabrication of a composite

- AFP combines the advantages of Filament winding + automated tape layup + 3D printing

<table>
<thead>
<tr>
<th>Technique</th>
<th>Fabrication rates</th>
<th>Geometric constraints</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filament winding</td>
<td>Fast</td>
<td>Requires constant convex curvature</td>
</tr>
<tr>
<td>Automated tape layup</td>
<td>Fast</td>
<td>Simple shapes with no compound curvature</td>
</tr>
<tr>
<td>3D printing</td>
<td>Slow</td>
<td>Minimal</td>
</tr>
<tr>
<td>Automated fiber placement</td>
<td>Fast</td>
<td>Lower contour parts and male tools</td>
</tr>
</tbody>
</table>
Fabrication of composites using AFP

**Advantages vs hand layup**

- Lower scrap
- Lower material costs
- Automated process
  - More consistent and higher quality parts
  - High layup rates
  - Digital process (inspection, digital twin, etc)
- Process scales and is flexible
  - Many different part geometries and sizes can be produced via the process

**Limitations**

- Better suited for lower contour parts and male tools
- Minimum feature size
  - Radius ≥500 mm for 180° tool
- Minimum part size
  - Min segment length of 4”

**Potential applications for CMC structures**

- Thermal shielding
- Exhaust cone
- Combustion liner
**3M™ Nextel™ 610 Towpreg – Experimental Product**

**Nextel 610 Spread tow**
- Controlled width and thickness
- Water soluble sizing

**Ceramic matrix**
- All alumina, water based composition
- Organic additives to provide tack

**Release liner**
- Polyethylene
- Carrier for tacky towpreg

**Coating**
- Slurry infused into fiber tow to provide controlled matrix content
- Water soluble sizing removed from fiber surfaces

**Drying**
- Water removed from slurry to produce a dry matrix
  - Target < 1 wt.% water
  - Room temperature stable matrix
  - Humidity can result in slightly higher moisture content (1.5 to 2 wt.%)

**Winding**
- Level wound roll
- Interleaved liner
  - Smooth, stable unwind of dry towpreg
**3M™ Nextel™ 610 Towpreg – Experimental Product**

**Nextel 610 Towpreg**
- Controlled width and thickness
- Controlled matrix content
  - Dry, room temperature stable
  - Heat-activated tack

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<table>
<thead>
<tr>
<th>Fiber</th>
<th>Nextel 610</th>
</tr>
</thead>
<tbody>
<tr>
<td>Denier (g/9,000m)</td>
<td>10,000</td>
</tr>
<tr>
<td>Nominal width (mm)</td>
<td>6.35</td>
</tr>
<tr>
<td>Nominal thickness (um)</td>
<td>200</td>
</tr>
<tr>
<td>Ceramic matrix content (wt%)</td>
<td>51</td>
</tr>
<tr>
<td>Oxide particle composition</td>
<td>alumina</td>
</tr>
<tr>
<td>Liner</td>
<td>polyethylene</td>
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</table>

Towpreg properties are representative and not for specification purposes.
A new pathway for simple geometry parts

Fabric prepreg model

- Fiber manufacturing
- Sizing application
- Weaving
- Sizing removal (Burn off)
- Prepregging
- Part layup
- Firing

Tow preg enabled model

- Fiber manufacturing
- Fiber spreading
- Towpreg manufacturing
- Part layup
- Firing

- We are enabling another, more simple supply chain with a potential for the lowest cost RM for oxide CMCs
- Enables customers to pursue lower cost manufacturing, including AFP, for simple geometries
Panel fabrication

Poor tack

Early prototypes showed insufficient tack and peeled up after placement

Excellent tack

Nextel layup rates on flat part

- Ply #1: 100-200 in/min
- Ply #2: 800-1000 in/min

Optimized carbon typically runs at 3000 in/min (qualified production rate)

IR heater applying heat to the part during material layup
Physical and mechanical properties

Test panels were fabricated with Nextel Towpreg using AFP. Measured properties are given below. The image to the right shows the cross section of the composite, highlighting the consistent distribution of fiber and matrix throughout the composite.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber orientation</td>
<td>0/90 cross-ply</td>
</tr>
<tr>
<td>Ply count</td>
<td>20</td>
</tr>
<tr>
<td>Per ply thickness (mm)</td>
<td>0.11</td>
</tr>
<tr>
<td>Fiber content (vol%)</td>
<td>41.3</td>
</tr>
<tr>
<td>Areal density (gsm)</td>
<td>303</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>290</td>
</tr>
<tr>
<td>Tensile modulus (GPa)</td>
<td>108</td>
</tr>
<tr>
<td>Flex strength (MPa)</td>
<td>382</td>
</tr>
<tr>
<td>Flex modulus (GPa)</td>
<td>122</td>
</tr>
<tr>
<td>Interlaminar shear strength (MPa)</td>
<td>20</td>
</tr>
</tbody>
</table>

Towpreg composite properties are representative and not for specification purposes.
Room temp performance comparison

<table>
<thead>
<tr>
<th>Material</th>
<th>3M</th>
<th>CHI</th>
<th>Axiom</th>
<th>COIC</th>
<th>WPX</th>
<th>Keramikblech</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber</td>
<td>N610</td>
<td>N610</td>
<td>N610</td>
<td>N610</td>
<td>N610</td>
<td>N610</td>
</tr>
<tr>
<td>Matrix</td>
<td>alumina-silica</td>
<td>alumina-silica</td>
<td>alumina-silica</td>
<td>alumina</td>
<td>alumina-zirconia</td>
<td></td>
</tr>
<tr>
<td>Fiber content (vol%)</td>
<td>41</td>
<td>41</td>
<td>46</td>
<td>40</td>
<td>39</td>
<td>42</td>
</tr>
<tr>
<td>Porosity (vol%)</td>
<td>27</td>
<td>23</td>
<td>21</td>
<td>26</td>
<td>28</td>
<td></td>
</tr>
</tbody>
</table>

Compared to other commercial CMCs, composites generated with Nextel Towpreg using AFP show in family performance at room temperature.
Steering trials

- Steering evaluated with arcs of Nextel Towpreg on carbon and PET substrates
- Evaluation of radius from 500-2500 mm
- No defects for radius greater or equal to 1000 mm
  - As a comparison, optimized carbon typically runs at 2000 mm for ¼”

Examples of steering defects

- Buckles/bubbles
- wrinkles
Complex geometries

A variety of tooling has been explored to identify capabilities and limitations of the Nextel Towpreg with AFP.

**Cone**
- 20” diameter, 22” height demo cone
- Multiple layup configurations evaluated (0°, 90°, 45°, 30°, etc)

**Angled radius**
- 5 sided tool to represent multiple features relevant to CMC fabrication
- 90° and 120° features ranging from 10-40 mm radius
- Layup orientation: 0° / +45° / -45°
Angled tool trials

- 90° and 120° features ranging from 10-40 mm radius
- Tool wrapped with polyimide film (gold color)
- Layup orientation: 0° / +45° / -45°
- 22 plies

Example of 120°, 30 mm radius layup
Angled tool layup results – 90° radius

Minor defects observed during layup, most notably in 45° plies. These could likely be corrected with additional AFP tuning.

- Ply 4: 0°
- Ply 5: +45°
- Ply 6: -45°
Composite processing – CT analysis – 10 mm, 90°

Parts were scanned using CT after layup, autoclaving, and sintering process steps. Thickness was measured at 7 positions along the part profile and reported as an average along the length of the part.

[Images of layup, autoclaved, and sintered parts with grid of measurement locations and thickness measurements graph]
Wrinkling defect

- Wrinkle observed in both 10 and 20 mm parts (red arrow)

- Likely the result of excessive part contraction during consolidation

- Using thickness measurements, a bulk factor of 130-140% was calculated. Plot to the right shows bulk factor for each measurement location across the part profile.

- High bulk factor can be addressed through
  - AFP parameters
  - Tooling / debulking
  - Towpreg formulation
Challenges with AFP rollers – Metal abrasion

Driven feed roll (440C SS)  Passive feed roll (Bronze 954)  Potential solution: PVD coated rollers

Black contamination on panels (red arrows) identified as:
• Copper from passive rollers
• Iron from active rollers

Coating polished, but much less debris observed on panels

Drive rolls polished by processing Nextel Towpreg

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Conclusions

• Introducing a new experimental product: Nextel 610 Towpreg
• Unique dry matrix providing room temperature stability and heat-activated tack
• AFP compatibility
• In family room temperature mechanical performance

Next steps

• Continue to evaluate product performance, including small volume sampling
  • Process window/constraints for AFP
  • Demonstration part(s), in-service testing
  • Composition/construction development
    • Mechanical data set for elevated temperature performance

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