POLYIMIDE COMPOSITE PROPERTIES OF RTM370 FABRICATED BY VACUUM ASSISTED RESINS TRANSFER MOLDING (VARTM)

RTM370 imide resin based on 2,3,3′,4′-biphenyl dianhydride (a-BPDA), 3,4′-oxydianiline (3,4′-ODA) with 4-phenylethynylphthalic (PEPA) endcap has shown to exhibit high Tg (370 °C) and low melt viscosity (10-30 poise) at 280 °C with a pot-life of 1-2 h. Previously, RTM370 resin has been fabricated into composites with T650-35 carbon fabrics by resin transfer molding (RTM) successfully. RTM370 composites exhibit excellent mechanical properties up to 327°C (620°F), and outstanding property retention after aging at 288°C (550°F) for 1000 hrs. In this presentation, RTM 370 composites will be fabricated by vacuum assisted resins transfer molding (VARTM), using vacuum bags without mold. The mechanical properties of RTM370 composites fabricated by VARTM will be compared to those of RTM370 made by RTM.
Polyimide Composites Properties of RTM370 Fabricated by VARTM

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Objectives

♦ Fabricated composite panels with RTM370 imide resin (~10-30 poise) by vacuum assisted resin transfer molding (VARTM)

♦ Compare mechanical properties of VARTM panels to RTM panels at 288-315°C (550-600°F)
### VARTM vs RTM

<table>
<thead>
<tr>
<th>VARTM</th>
<th>RTM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Use vacuum bag</td>
<td>Need a mold (expensive)</td>
</tr>
<tr>
<td>Use vacuum only</td>
<td>Use pressure and/or vacuum</td>
</tr>
<tr>
<td>15-20 psi</td>
<td>200 psi</td>
</tr>
</tbody>
</table>
RTM370 Imide Resins

Advantages of imide resins containing α-dianhydrides

- Lower melt viscosities
- Higher $T_g$’s

Advantages of imide resins containing α-dianhydrides:

- Lower melt viscosities
- Higher $T_g$’s
# Physical Properties of Imide Oligomers/Resins Based on a-BPDA and 4-PEPA

<table>
<thead>
<tr>
<th>Resin</th>
<th>Diamine</th>
<th>Oligomer Min. η @280 °C by Brookfield&lt;sup&gt;1&lt;/sup&gt; (Poise)</th>
<th>Oligomer Min. Complex [η]&lt;sup&gt;−1&lt;/sup&gt; @280 °C&lt;sup&gt;2&lt;/sup&gt; (Poise)</th>
<th>Cured Resin T&lt;sub&gt;g&lt;/sub&gt; (°C) NPC&lt;sup&gt;3&lt;/sup&gt; by TMA&lt;sup&gt;5&lt;/sup&gt;</th>
<th>Cured Resin T&lt;sub&gt;g&lt;/sub&gt; (°C) PC&lt;sup&gt;4&lt;/sup&gt;@ 650 °F</th>
</tr>
</thead>
<tbody>
<tr>
<td>RTM370</td>
<td>3,4′ -ODA</td>
<td>8.8</td>
<td>6.5</td>
<td>342</td>
<td>370&lt;sup&gt;5&lt;/sup&gt;</td>
</tr>
<tr>
<td>RTM370 Composite</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>338 (DMA)&lt;sup&gt;6&lt;/sup&gt;</td>
<td>350 (DMA)&lt;sup&gt;6&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>1</sup> Absolute viscosity measured by Brookfield Viscometer at 280 °C.
<sup>2</sup> Complex viscosity measured by Aries Rheometer, using parallel plates.
<sup>3</sup> NPC = No Post cure
<sup>4</sup> PC = Resin Postcured at 343 °C (650 °F) for 16 hrs while composite postcured @ 650°
<sup>5</sup> TMA = Thermal mechanical analysis heated at 10 °C/min, using expansion mode.
<sup>6</sup> DMA = Dynamic mechanical analysis were performed at 5 °C/min heating rate, using single cantilever.

3,4′ -ODA = 3,4′ -Oxydianiline
Rheology of APS’s RTM370 Imide Resin
2 hr hold at 280 °C
# RTM370 Composite Property Comparison

## VARTM vs RTM (T650-35/HT sizing)

<table>
<thead>
<tr>
<th>Test Temp.</th>
<th>OHC Strength (MPa) @550°F</th>
<th>OHC Modulus (GPa) @550°F</th>
<th>SBS Strength (MPa) @550°F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Initial</td>
<td>500h</td>
<td>1000h</td>
</tr>
<tr>
<td>VARTM</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>550°F</td>
<td>233</td>
<td>194</td>
<td>120</td>
</tr>
<tr>
<td>600°F</td>
<td>186</td>
<td>197</td>
<td>135</td>
</tr>
<tr>
<td>620°F</td>
<td>182</td>
<td>---</td>
<td>118</td>
</tr>
<tr>
<td></td>
<td>184</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>RTM</td>
<td>269</td>
<td>287</td>
<td>230</td>
</tr>
<tr>
<td>550°F</td>
<td>242</td>
<td>244</td>
<td>198</td>
</tr>
<tr>
<td>600°F</td>
<td>231</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>620°F</td>
<td>241</td>
<td>---</td>
<td>---</td>
</tr>
</tbody>
</table>
RTM370 OHC Strength VARTM vs RTM (T650-35/8HS/HT Sizing)

Open-Hole Compression Strength (MPa)

- RTM370 by RTM
- RTM370 by VARTM
- BMI-5270-1 by RTM

Temperatures:
- RT (RTM)
- 288°C (550°F)
- 315°C (600°F)
- 327°C (620°F)
RTM370 OHC Modulus VARTM vs RTM (T650-35/8HS/HT Sizing)
RTM370 SBS Strength VARTM vs RTM
(T650-35/8HS/HT Sizing)

RTM370 by RTM
RTM370 by VARTM
BMI-5270-1 by RTM

Short Beam Shear Strength (MPa)

- RTM370 by RTM
- RTM370 by VARTM
- BMI-5270-1 by RTM

RT 288°C 315°C 327°C

0 10 20 30 40 50 60

RTM370 by RTM
RTM370 by VARTM
BMI-5270-1 by RTM
Open-Hole Compression Strength of RTM370 Composites Subjected to Isothermal Aging at 550°F for 1000 h
Open-Hole Compression Modulus of RTM370 Composites Subjected to Isothermal Aging at 550°F for 1000 h

- Initial Property by RTM
- Initial Property by VARTM
- 500 h@550°F by RTM
- 500 h@550°F by VARTM
- 1000 h@550°F by RTM
- 1000 h@550°F by VARTM

<table>
<thead>
<tr>
<th>Temperature</th>
<th>RTM</th>
<th>VARTM</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT</td>
<td>50</td>
<td>40</td>
</tr>
<tr>
<td>288°C</td>
<td>45</td>
<td>42</td>
</tr>
<tr>
<td>315°C</td>
<td>40</td>
<td>38</td>
</tr>
<tr>
<td>327°C</td>
<td>35</td>
<td>35</td>
</tr>
<tr>
<td>550°F</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>600°F</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>620°F</td>
<td>20</td>
<td>20</td>
</tr>
</tbody>
</table>
Short-Beam Shear Strength of RTM370 Composites Subjected to Isothermal Aging at 550°F for 1000 h
VARTM vs RTM

SEM of RTM370 made by VARTM
Void content = ~6.5% after postcured at 650°F/8 h
Resin=35-38 wt%, Fiber volume=49-53%

RTM370 Made by RTM
Void content= ~1%
Resin = 35-36 wt%
Fiber volume=53-56%

<table>
<thead>
<tr>
<th>VARTM</th>
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</tr>
</thead>
<tbody>
<tr>
<td>15-20 psi</td>
<td>200 psi</td>
</tr>
<tr>
<td>Higher void content</td>
<td>Lower void content</td>
</tr>
</tbody>
</table>

Process Improvement:
♦ Injection at 260°C instead of 280°C ⇒ Longer pot-life, but longer injection time
♦ Add hold time at ~300°C, instead of direct ramp from 280°C to 371°C
Summary

Demonstrated RTM370 imide resin can be processed by VARTM out of autoclave.

VARTM panels have higher void content than those made by RTM, due to lack of pressure.

⇒ VARTM panels appeared to have slightly lower OHC, but comparable SBS at high temp. & retained good mechanical properties after aging.

Need process development to reduce void content to < 2% for aerospace applications.

Need to lower the resin content of VARTM panels from 50% to 35-40%.
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