High Speed RTM Materials and Processing Technology Advancements for Affordable Lightweight Composites

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Dow Vision: “Affordable Lightweighting”

- **Durable, Stable Matrix Material**
- **Characterisation & Simulation for Optimised Parts**
- **Engineering & Design**
- **Material Efficiency**
  - Required Thickness Increase
    - Front Roof Bow
    - 80°C
    - CF PA6
    - CF Epoxy
- **Processing Efficiency**
  - 00:59s
- **Assembly Efficiency**
  - Fast Cure Resins
- **Large Part Consolidation & Adhesive optimisation**
- **Secondary Value**
  - kg
  - $€
- **Mass Decompounding**
- **Legislative Penalties**
- **Secondary Value**
- **Source: SEMA**
  - Legislative Penalties
- **Source: Multimatic**
  - Engineering & Design
  - Material Efficiency
  - Processing Efficiency
  - Assembly Efficiency
  - Secondary Value
  - Mass Decompounding
  - Large Part Consolidation & Adhesive optimisation
  - Source: SEMA
  - Source: Multimatic
Dow Automotive Lightweighting Portfolio

Dow VORAFORCE™
Thermoset Matrices

Optimised substrate-adhesive interface
Minimised composite overdesign
Minimised surface preparation
Corrosion control

Maximise resin-fiber interface performance

DowAksa AKSACA™
Carbon fiber

Optimised carbon fiber content via local foam reinforcement

Dow BETAMATE™
and BETAFORCE™
Structural Adhesives

Cavity sealing and Acoustic optimisation

Dow BETAFOAM™
Structural Foam
High-Pressure Resin Transfer Molding (HP-RTM)

- Highly coupled multi-physics process
- Fabric preforming affects the flow
- Permeability characterization is essential
- Cure kinetics modeling for ultrafast cure resin systems

**Resin Requirements:**
- Low viscosity and long gel time to fill mold and impregnate fiber mat
- Fast cure to reduce cycle time
- Complete/quick resin conversion

**Process requirements (for large volume automotive):**
- Infusion time 10-40 sec
- Demold time down to 40 sec
- Total cycle time 80-100 sec
Ultra fast cure Epoxy System 81 sec Cycle Time

- 50vol% carbon fiber composite, 540 x 290 x 2mm

**On screen Timer**

<table>
<thead>
<tr>
<th>Time</th>
<th>Step description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0:00</td>
<td>Process start (press closes)</td>
</tr>
<tr>
<td>0:13</td>
<td>Press is closed (mold is evacuated)</td>
</tr>
<tr>
<td>0:20</td>
<td>Injection starts (approx. 10 secs)</td>
</tr>
<tr>
<td>0:30</td>
<td>Injection complete, cure begins</td>
</tr>
<tr>
<td>1:10</td>
<td>Mold opens (40 secs after end of injection – this is “demold time”)</td>
</tr>
<tr>
<td>1:21</td>
<td>Press cycle time (press is open and in safe position, part ejects)</td>
</tr>
</tbody>
</table>
Development Materials

- Carbon fiber
  - DowAksa A 42 – 24k
  - 50% fiber volume fraction
  - 2x2 weave

- Epoxy Resin
  - VORAFORCE™ 5300 – Ultra fast cure epoxy system
Cure Window Development

Conversion as a function of mold temperature and cure time for ultra fast cure

<table>
<thead>
<tr>
<th>Mold temperature</th>
<th>Cure time</th>
<th>DSC, Conversion (from 1st heat)</th>
<th>DSC, mid point Tg (from 2nd heat)</th>
</tr>
</thead>
<tbody>
<tr>
<td>110° C</td>
<td>120 seconds</td>
<td>94.4 %</td>
<td>125 °C</td>
</tr>
<tr>
<td>120° C</td>
<td>60 seconds</td>
<td>93.9 %</td>
<td>123 °C</td>
</tr>
<tr>
<td>120° C</td>
<td>120 seconds</td>
<td>98.9 %</td>
<td>122 °C</td>
</tr>
<tr>
<td>130° C</td>
<td>60 seconds</td>
<td>98.9 %</td>
<td>122 °C</td>
</tr>
<tr>
<td>130° C</td>
<td>120 seconds</td>
<td>Approx. 100 %</td>
<td>123 °C</td>
</tr>
<tr>
<td>140° C</td>
<td>30 seconds</td>
<td>99.5 %</td>
<td>120 °C</td>
</tr>
<tr>
<td>140° C</td>
<td>60 seconds</td>
<td>Approx. 100 %</td>
<td>120 °C</td>
</tr>
</tbody>
</table>

All values generated from composite HP-RTM panels (50% fiber volume fraction, DowAksa fiber)

- At or above 130 °C, cure times of approximately 60 seconds are achieved with acceptable conversion (>98%)
- Demolding is possible within 120 seconds at 110 °C; however, a post-cure is recommended to achieve full conversion
- At 120 °C a cure time of >90 seconds is recommended to achieve acceptable conversion
Viscosity Curves

Cure time can be adjusted over a wide temperature spectrum:

- High production volumes require processing temperatures >105 °C
Flow Improvements

Flow Comparison (50% carbon fiber vol., 2 mm thick)

[VSS] 65 cm Filled
29 Seconds (viscosity limit reached)

Competitive Product (demold time 4-5 minutes)

[SSS] 122 cm Filled
46 Seconds (viscosity limit reached)

VORAFORCE 5300 (demold time down to 40 seconds)
Internal mold release (IMR) agents were screened for efficacy on RTM equipment:

- External mold release applied for first 3 releases simulating start-up of production.
- Mold release tested at 1 pph resin and at 2 pph resin with similar results (on panel geometry).
- Recommendation 2 pph resin for complex geometries.
- Currently >60 releases demonstrated with no increase in demold force.
Internal Mold Release Agent

Robustness of thermal properties with variation in IMR:

<table>
<thead>
<tr>
<th>IMR pph resin</th>
<th>Panel type</th>
<th>DSC, % Conversion (from 1st heat)</th>
<th>DSC, mid point Tg (from 2nd heat)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.6</td>
<td>Clear cast</td>
<td>100</td>
<td>126, 124, 126</td>
</tr>
<tr>
<td>1.8</td>
<td>Clear cast</td>
<td>100, 100, 100</td>
<td>125, 126, 126</td>
</tr>
<tr>
<td>2.0</td>
<td>Clear cast</td>
<td>100, 100</td>
<td>123, 123, 123</td>
</tr>
<tr>
<td>2.2</td>
<td>Clear cast</td>
<td>100, 100</td>
<td>123, 124, 124</td>
</tr>
</tbody>
</table>

Multiple values indicate repeat measurements
All values generated from HP-RTM resin panels (mold temp. = 120 °C, Demold time = 120 sec.)
Integrated Predictive Engineering Capability Build

- Fibre
- Textile
- Preforming

- Resin

- Drapability Simulation
- RTM Simulation
- Structural Analysis

- Epoxy Rheo-Kinetics Model
- Process Optimization:
  - Tool conditions
  - Tool temperature
  - Geometry
  - Injection location
  - Fabric temperature
  - Vent location
  - Resin temperature
  - Flow rate
  - Pressure limits

Predict to Avoid Dry-Spots & High Pressure Gradients
Preform - Draping of Carbon Fibre Fabrics

Excellent visual agreement between experimental results and Abaqus.

Ref: Sherwood
Preform - Draping analysis – Complex tool

- Prediction of orientation
- Rotation of the fibers
- Shell thickness variation during draping

Thickness distribution of the Preform

Fiber Orientation & Rotation
Preform - Permeability Measurements on Stack-Ups

Significant impact of Fiber Volume on Permeability

Ref: M. Arnold e.a. IVW Kaiserslautern
The Integrated Model Suite

- Raw material database
- Formulation explorer
  - Virtual product development and testing
- Product Property Models
  - Thermal ($T_g$, CTE)
  - Mechanical ($K_{1c}$, Modulus)
  - Electrical properties ($D_k$)
- Product Processing Models
  - Rheokinetic behaviour
  - Exotherm
  - Optimize cure cycle
Complex HP-RTM tool

Instrumented Tool:
- Pressure Sensors
- Temperature Sensors

Different Gate Locations Possible
PAM-RTM Model

- 2.5D & 3D Models
  - 2.5D for isothermal analysis
    » Mapped Permeability & Fiber Volume
  - 3D for non-isothermal analysis
    » Permeability/Fiber Volume Zones

Ultra-fast cure epoxy
- Viscosity vs. time

Non-Isothermal:
- Effect of Resin at different temp. versus tool
- Exotherm effect
- eCURE™ Model for viscosity & cure

Isothermal:
- Viscosity & Cure as function of time
Filling profile – short shots

DowAksa Carbon fiber

- 24K tows
- 50% volume fraction
- 6 layers +/-45
- 15% short shot

- Slight lag in filling the dome area
Filling profile – short shots

Carbon fiber Package

• 24K tows
• 50% volume fraction
• 6 layers +/-45
• 25% short shot

• Lag in filling the dome area not replicated in actual part
• Corners- effect seen in actual part
Final Part Results

- PAM-RTM analyses show
  - Non-Isothermal effects
  - State of cure
  - Flow Pattern
  - Pressures to fill

- But…
  - Pressure readings in tests have significant variability

State of Cure at end of Fill

Filling Profile

Temperature profile at end of Fill
Conclusion

• Ultra fast cure epoxy system enable fast cycle time composite molding

• Preform simulation is matched by actual results reducing risks in manufacture of preform tools

• Advanced Rheo-Kinetic eCURE™ models coupled with PAM-RTM correlates well to molding process

• Flow modeling tools match short shot tests allowing for improved tool design

• Combination of ultra fast epoxy resin materials and modeling/engineering tools makes epoxy materials viable for large volume automotive applications