CURE MONITORING OF HIGH-TEMPERATURE RESINS FOR ENHANCING THE MANUFACTURING OF ADVANCED COMPOSITES

Dr N. Pantelelis, Synthesites SNC, Belgium
W. Gerrits, R. Klomp-de Boer, NLR, The Netherlands
A. Johnston, S. Wilson, A. McKibbin, Bombardier Aerospace, UK
C. Brauner, F. Schadt, L. Amirova, M. Grob, FHNW, Switzerland
On-going

**Recotrans**: (2017-2020) Multimaterial recyclable manufacturing for the transportation industry
RTM and pultrusion, Glass fibre, reactive thermoplastic for automotive and rail applications
Partners: Aimplas (CO), Fraunhofer, Daimler, Far UK, Stadler, INEA, Istanbul University, Arkema

Partners: DLR (CO), Bombardier, Aernnova, NLR, ONERA, Apodius, Rockwell Collins

Completed

**Ecomise**: First-time right composites manufacturing (2013-2016)
Partners: DLR (CO), Faser I., Bombardier, Hutchinson, Airborne, Loop, Dassault Systemes, NLR
RTM and RTI, Glass and carbon fibre, epoxy

**Coaline**: Injection pultrusion with microwave curing and injection of coatings (2013-2017)
Fraunhofer ICT, Aimplas, Resoltech, Rescoll, Acciona

**iREMO**: intelligent Reactive Moulding (2009-2012)
RTM, Light RTM and Infusion, Glass and carbon fibre, epoxy and polyester

**MAC-RTM**: Microwave curing (2011-2013), Fraunhofer ICT and Aimplas
Advantages of Process Monitoring in composites manufacturing

- Check resin quality and adjust process accordingly
- Detect accurately resin arrival at critical locations
  - Open/close valves based on sensors’ feedback
- Monitor viscosity changes and decide when start heating
- Identify minimum viscosity and decide about pressure
- Detect unexpected events and follow alternative routes
- Improve simulation accuracy and design intelligent strategies
- Real-time decision of the cure cycle based on Tg and degree of cure (depends on the resin) rather than time
Intelligent automation in composites processing

Injection machine

Control Actions

Temperature controller(s)

DAQ + Control

Inline Sensors

Mould

Composite part

In-mould Sensors

Monitoring Systems
- 4 temperature and resin arrival sensors
- Resistance-based measurements and RTD temperature
- Continuous connection checking
- One relay output for process automation

- **In-mould**
  - Durable

- **Gate sensor**
  - flat areas
  - possible mark
  - ideal for vacuum infusion in oven/autoclave (gates, pipelines, pots etc.)

- **Flexible disposable**
  - Curved surfaces
  - In the laminate for development
  - Over the peel-ply
  - Suitable for very long parts
  - no extra protection for Carbon Fibre Preforms

- **FloWire sensors**

---

**New**

**Curved Durable**
Real-time measuring of
- Resin’s electrical resistance (from 0.1 MOhm up to 50 TOhm)
- temperature (pt100 sensor with 0.1°C accuracy)

Input of external signals e.g. pressure sensors

**process monitoring sensor** = electrical resistance + RTD sensors

<table>
<thead>
<tr>
<th>Sensor Type</th>
<th>Features</th>
</tr>
</thead>
<tbody>
<tr>
<td>Durable sensor</td>
<td>High Temp RTM, Resin arrival, Viscosity rise, Gelation, End-of-cure</td>
</tr>
<tr>
<td>Flexible sensor</td>
<td>VI and RT cure, Resin arrival, Viscosity rise, Gelation, End-of-cure</td>
</tr>
<tr>
<td>Inline sensor</td>
<td>Avoid pipe cleaning, Adjust cycle, Mixing ratio check</td>
</tr>
<tr>
<td>Pot sensor</td>
<td>Mixing ratio, Resin Quality, Resin aging, Adjust cycle</td>
</tr>
</tbody>
</table>

CF In-mould Durable
Vacuum Bag Sensor

New

New
A typical RTM6 cure cycle as measured with Optimold

- Resin arrival
- End-of-cure
- Resistance
- Cooling
- Viscosity increase
- Gelation
- Temperature
- Minimum viscosity

Time (min)
More than 25 resins have been calibrated for the whole range of advanced composites manufacturing.
Verification of the real-time estimated $T_g$

Overview of the $T_g$ estimated online with the ORS software and $T_g$ measured right after demoulding by DSC and the difference between them for several isothermal and realistic test cases which shows that the $T_g$ online estimation is within the DSC accuracy.

Trials and DSC performed by CARBON ROTEC COMPOSITE TECHNOLOGY

<table>
<thead>
<tr>
<th>Trial</th>
<th>Duration [h]</th>
<th>$T_g$-ORS (°C)</th>
<th>$T_g$-DSC (°C)</th>
<th>Difference (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>80DV1</td>
<td>3</td>
<td>73.17</td>
<td>73.34</td>
<td>-0.17</td>
</tr>
<tr>
<td>80DV3</td>
<td>2.5</td>
<td>70.30</td>
<td>70.91</td>
<td>-0.61</td>
</tr>
<tr>
<td>80DV4</td>
<td>2.5</td>
<td>73.45</td>
<td>72.49</td>
<td>0.96</td>
</tr>
<tr>
<td>80-120'</td>
<td>1.92</td>
<td>66.96</td>
<td>66.02</td>
<td>0.94</td>
</tr>
<tr>
<td>80-90-1</td>
<td>1.50</td>
<td>62.04</td>
<td>61.80</td>
<td>0.24</td>
</tr>
<tr>
<td>80-90-2</td>
<td>1.50</td>
<td>65.52</td>
<td>65.21</td>
<td>0.31</td>
</tr>
<tr>
<td>80-D2-2</td>
<td>1.50</td>
<td>61.88</td>
<td>60.59</td>
<td>1.29</td>
</tr>
<tr>
<td>60-260'</td>
<td>4.33</td>
<td>55.02</td>
<td>56.51</td>
<td>-1.49</td>
</tr>
<tr>
<td>70-190'</td>
<td>3.17</td>
<td>64.92</td>
<td>65.39</td>
<td>-0.47</td>
</tr>
</tbody>
</table>

Isothermal cases, mean difference: 1.61
Isothermal cases, standard deviation: 2.42

<table>
<thead>
<tr>
<th>Non-isothermal</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEB1-1</td>
</tr>
<tr>
<td>TEB1-2</td>
</tr>
<tr>
<td>TEB2-1</td>
</tr>
<tr>
<td>TEB2-2</td>
</tr>
<tr>
<td>LESW1-1</td>
</tr>
<tr>
<td>TESW1</td>
</tr>
<tr>
<td>Shell1-1</td>
</tr>
<tr>
<td>Shell1-2</td>
</tr>
<tr>
<td>Shell2-1</td>
</tr>
</tbody>
</table>

Non-isothermal cases, mean difference: 2.15
Non-isothermal cases, standard deviation: 1.26
Intelligent Process Control

Measuring resistivity, temperature, pressure

Online Estimating Tg, viscosity, degree of cure

Intelligent Process Control

Controlling temperature, demoulding time etc.

Injection machine

Press controller

Temperature controller(s)
Viscosity, Resistance and temperature vs. time for 4 resin batches of Cycom 890

Case 1

Case 2, 4

Case 3
Outside of the autoclave

Inside of the autoclave
Real-time Tg prediction and demoulding decision based on targeted Tg.
A new durable sensor was developed to allow to measure CFRP production without the need of any extra protection e.g. glass fibre

- Due to the unique surface measurement of the DC-based sensor no coating but specially shaped electrodes were used

Extensive trials in a C-HPRTM application have shown very good results confirming that the use of this sensor in CFRP production is feasible
A new higher-temp material was introduced in the disposable sensor to improve the high-temp performance of the sensor. First trials with a prepreg BMI sample sent by BAB demonstrated the improved performance of the sensor.
BMI Cytec 5250: Correlation between the expected Degree of Cure as simulated by a kinetic model and the recorded resistance during for the recommended temperature profile.
Within SuCoHS, Cyanate ester resins were also monitored for the first time successfully by FHNW.
The online cure monitoring and quality control for high temperature resins was applied and verified successfully.

The development of new high temperature sensors and calibration methods will lead to significant reduction of the curing time of advanced aerospace resins ensuring cure quality.

Further development with SuCoHS project will allow disposable and durable sensors to work at temperatures up to 300°C (first step) and ultimately at 350°C.
This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 769178.

www.sucohs-project.eu
https://www.linkedin.com/company/sucohs-project/
Contact

Belgium
SYNTESITES SNC
Av. du Lycée Français 5, Bte9
1180, Uccle
Phone: +32 (0) 472 201 382
e-mail: be@synthesites.com

Greece
SYNTESITES INNOVATIVE TECHNOLOGIES Ltd
Kyprion Agoniston 33,
GR-185 41, Piraeus
Phone: +30 210 42 12 274,
e-mail: info@synthesites.com

UK
SYNTESITES UK LTD
31 Arden Close,
Bristol, BS32 8AX
Phone: +44 333 01 2468 1
e-mail: uk@synthesites.com

www.synthesites.com