Viscoelastic Measurement

as a

Method for Process Control
Long Term Goal

Change From:
Time and Temperature Goals with Cure State Assumed

To:
Cure State Measured with Time and Temperature Managed
Lab Equipment Contemporary to Legacy Specification Development ca. 1971

The result of a modern chemical analysis is often a graph from a strip-chart recorder.

The modern chemistry laboratory uses automatic equipment to do what had previously been done by eye.
State of the Art Test Equipment and Process Controls
Why Viscoelastic Measurements?

- **Before Cure**
  - Storage time & out time
  - Handling and Layup
  - Staging time

- **During Cure**
  - Infusion
  - Volatile Release
  - Consolidation in staged pressure cures
  - Achieve ‘full cure’

- **After Cure**
  - Glass transition temperature
  - ‘Spring back’ & ‘Fit-up’
  - Fatigue
Visco-elastic Data Used To Develop Specifications but Requirements Not Typically Included In The Specification
Pre-Production Process Development

Testing to set limits

➢ Visco-Elastic boundaries

Time

Temp
Information Conveyed in Specs, T.O.’s and Shop Procedures
Experimental Setup

Control System and Rheometer
Method of Gathering Data

Production Control System modified to:

- Provide set-points to analytical instruments
  - in real time during a controlled production run
  - post run
  - from imported data

- Control of the processing equipment and the instrument using data from each
CSS300 Control System
Test Unit & Specimen
Test Cell

Upper Die is a load cell and Lower Die cycles torque on sample to a specified strain or load. G’ & G” reported. Dies are heated and grooved for gripping.
<table>
<thead>
<tr>
<th>Materials</th>
<th>Viscosities of Common Materials</th>
<th>Consistency</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Centipoise (cps)</td>
<td>Poise (ps)</td>
</tr>
<tr>
<td>Air</td>
<td>1.00E-03</td>
<td>1.00E-05</td>
</tr>
<tr>
<td>Water</td>
<td>1.00E+00</td>
<td>1.00E-02</td>
</tr>
<tr>
<td>Olive Oil</td>
<td>1.00E+02</td>
<td>1.00</td>
</tr>
<tr>
<td>Glycerin</td>
<td>1.00E+03</td>
<td>1.00E+01</td>
</tr>
<tr>
<td>Golden Syrup</td>
<td>1.00E+05</td>
<td>1.00E+03</td>
</tr>
<tr>
<td>Polymer Melts</td>
<td>1.0E+05-</td>
<td>1.0E+03-</td>
</tr>
<tr>
<td>Pitch</td>
<td>1.00E+12</td>
<td>1.00E+10</td>
</tr>
<tr>
<td>Glass</td>
<td>1.00E+24</td>
<td>1.00E+22</td>
</tr>
</tbody>
</table>

1000 cps = 10 poise = 1 Pa.s = 1 PI
Typical Cure Path G’

vs

Time and Temperature

Cure Path of G'
TC25 of selected Cure

Softens & Grips

Softens as Heats

G’ rises as cools

Cures over Time

Temperature Profile Target[Temperature(°F)]
Run Time[Time (mins)]
Typical Data from Replication of Shop Process in Autoclave
Check of Repeatability

- APA1 G p[BMS-8-276-Real - 1]
- APA1 G p[BMS-8-276-Real - 4]
- APA1 G p[BMS-8-276-Real - 2]
- TCO01[BMS-8-276-Real - 1]

Graph showing the check of repeatability with various data points and lines representing different conditions.
As Receive vs Staged for 120Min

Offset of X axis by 120 minutes

- APA1 G p[BMS-8-276-Real - 16]
- APA1 G p[BMS-8-276-Real - 1]
- TC001[BMS-8-276-Real - 1]

Temperature vs Run Time (°C)
End

Questions
Experimental Goals

Execute Cure State Feedback Scenarios

- Manage viscosity for infusion
  - Keep viscosity constant by increasing temperature

- Correlate Di-Electric Analysis to Viscosity
  - Provide greater relevance to process

- Manage Post-cure
  - Develop heat rate data that would prevent re-softening of the part or tool
Logic for Managed Infusion

Viscosity

1. Raise temperature until viscosity (G”) drops to a specified viscosity minimum
2. Stop temperature rise until cure causes viscosity to raise to a specified maximum
3. Resume heating and repeat steps 1&2 until maximum cure temperature is reached.
4. Stop

If this were an actual part, the run would be continued until the part were fully cured
Managed Viscosity Cure

Temperature Profile to Maintain a Constant Viscosity
Cytec 977-2

- RDA3 Oven Temperature
- RDA3 Viscosity

AvPro -- NCAMP May 17 2006
Control Logic for DEA Calibration

1. Temperature data from a cure cycle that had been monitored by DEA was provided.
2. Data was loaded into the CSS300 Post Process Analyzer.
3. The temperature cycle was duplicated on the rheometer.
4. Viscosity data was plotted on graph with DEA data.
Temperature Replication of Cure Monitored by DEA

Comparison of 977 Film between RDA-III and DEA Data

RDA-III and DEA Readings

Rheometer Viscosity in Pa.sec

DEA

Boeing Temperature Readings

Avpro Temperature Readings
Cure Monitored by DEA

Rescaled to Match Graphs

Viscosity Comparison of 977 Film between RDA-III and DEA Data

- Toffee Like
- Syrup
- Glycerin
- Olive Oil
- Wetting of Sensor

On RDA-III
Frequency – 1 Hz
Gap – 1.5 mm
On DEA
Frequency – 1 Hz

AvPro -- NCAMP May 17 2006
Tooling/Prototype Cure Cycle

1. Initial cure was set at 200F (to avoid problems with soft tooling and steam out-gassing)
2. Hold at 200F until G’ near maximum value at specified temperature
3. Raise temperature until a small drop in G’ is noted
4. Begin a hold until G’ recovers to near maximum
5. Repeat steps 3 & 4 until final cure temperature is reached

For actual parts the cure would be continued as specified to achieve full cure.
Maintain G’ Modulus on Post Cure
Summary

Time/Temperature
- Used to control properties but there is no visibility on the properties controlled

Visco-Elastic Monitoring & Control
- Time and temperature still critical to managing material state
- Viscoelastic feedback provides opportunity for process improvement and corrective action
- MSM provides a basis for time and temperature adjustments without compromising quality
Conclusions

- Tools exist to measure and manage viscoelastic properties
- Tools have immediately value for development, tooling and discrepancy disposition
- No significant additional shop requirements
- Certified structures are not harmed by MSM procedures

Certification of structures based on material state properties desirable but will require additional work and substantiation