Thermoset Conference – SPE - 2023
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RCR / TCR
Capillary Rheometer
Basics: Aims of Rheological Analysis

Material consideration:

• Product development
• Compound development
• Product observance
• Quality control

Machine and process consideration:

• Development and design of processing and process techniques
• Development and design of machines and machine elements
Capillary Rheometer Model

- Reservoir
- Entrance
- Laminar Flow
- Exit
- Force Mmt
- Piston
- Barrel
- Pressure Mmt
- Capillary Die
Capillary Rheometer

- Protection hood
- Test piston
- Test chamber with capillary (Option)
- Lower test chamber half with mold (Option)
- Touch-screen panel for controlling and test data display
- Machine body with test piston drive, pre-compressing device, test chamber closure, hydraulic aggregate etc.
TCR – THERMOSET CAPILLARY RHEOMETER

1-piston
2-capillary with reservoir
3-mould with rectangular (slit) geometry

Devided Capillary

2-5mm ø-reservoir 20mm

Mould upper half
Mould lower half
TCR – Special Mold:

- Force sensor
- Piston
- Capillary

- Pressure transducers
- Temperature gradient sensors

Optional Ultrasonic Sensor
**Test Methods – CP / CS**

**Constant Pressure**

- Mainly used test method.

**Constant Speed**

- Typical capillary rheometer test to determine the viscosity.

<table>
<thead>
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<th>Determination of viscosity capillary mould</th>
<th>Piston pressure - P0</th>
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<tbody>
<tr>
<td>P1-P0</td>
<td>P2-P1</td>
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<tr>
<td>P3-P2</td>
<td>P4-P3</td>
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Resin Curing with RPA

- Cure temperature 160°C.
- Sample 2 and 3 have about the same minimum, minimum of sample 1 is much higher.
- Probe 1 has a significant higher cure time as sample 2 and 3.
TCR – Phenolic Resin – Injection at CS

Comparison Injection Die PSt>P0 vs P3 >P4

- In opposite to the minimum of cure curve sample 3 has the highest viscosity
- Sample 1 and 2 are better differentiated in constant speed mode
- Lowest viscosity of sample 2 at high shear rates
- Sample 1 and 3 show a similar behavior in the mould
TCR – Phenolic Resin Injection at CP

Evaluation of Curing P0 > P1

Calculation of the cross-linking curve from the pressure loss between the pressure sensor at the injection point P0 and the 2nd pressure sensor P1 provides similar results to RPA.

- The minimum for sample 1, similar to the RPA measurement, is significantly higher than for samples 2 and 3.
- Sample 1 has the fastest crosslinking.
TCR – Determination of Shrinkage

Material flows over the pressure sensor during the injection. Crosslinking increases the viscosity and eventually stops the flow at Gel point.

As material crosslinks further, it shrinks and the pressure sensor senses the pressure reduction.

Batch differences can be detected caused by shrinkage.
The temperature gradient sensors detect a heat flow by measuring a temperature difference over a reference material:

\[ Q = -k_n \frac{\Delta T}{L} \]

\[ \Delta T = T_0 - T_2 \]

The samples also show batch differences in the temperature peak.

The differences in positive heat flow (from the sample) correlates to gel time.

The negative temperature peak is determined by heating up and activation of the crosslinking.
SUMMARY

- Testing under conditions similar to injection molding at constant pressure or speed.
- Viscosity data at high strain rates show a different ranking of flow properties than when testing crosslinking in the RPA (vulcameter).
- At low deformation speeds, the ranking is similar to that in the RPA.
- A cross-linking curve can be determined from the slope of the pressure difference of the first two pressure sensors in the mold. The curve and the ranking of the samples is similar to the curve averaged in the RPA.
- The TCR provides a clear differentiation between different batches of different material groups with additional identification numbers.
- Filling time, defined as the time to reach a certain pressure sensor.
- Gel time, defined as the time it takes for material flow to stop.
THANK YOU FOR YOUR TIME ... ANY QUESTIONS?