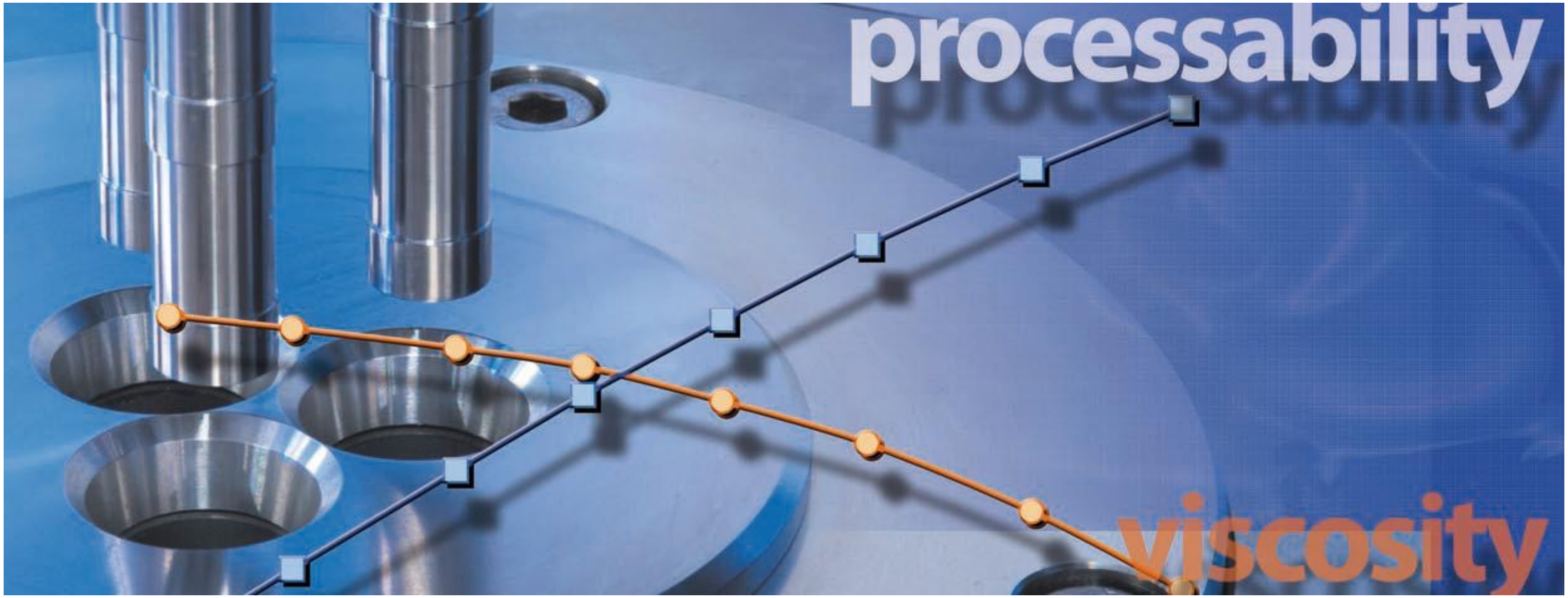
Thermoset Conference – SPE - 2023
Presentation by Tim Haake

RCR / TCR
Capillary Rheometer



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THIS IS RHEOLOGY

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Basics: Aims of rheological Analysis

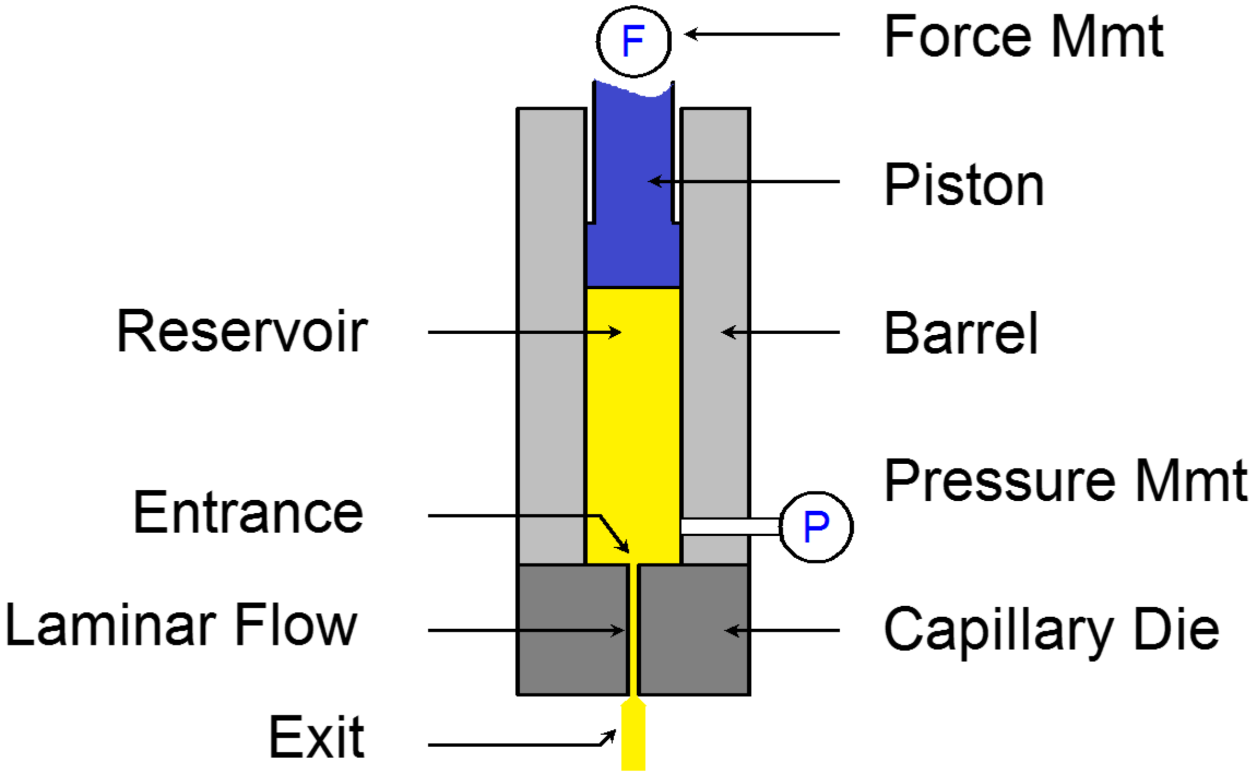
Material consideration:

- 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



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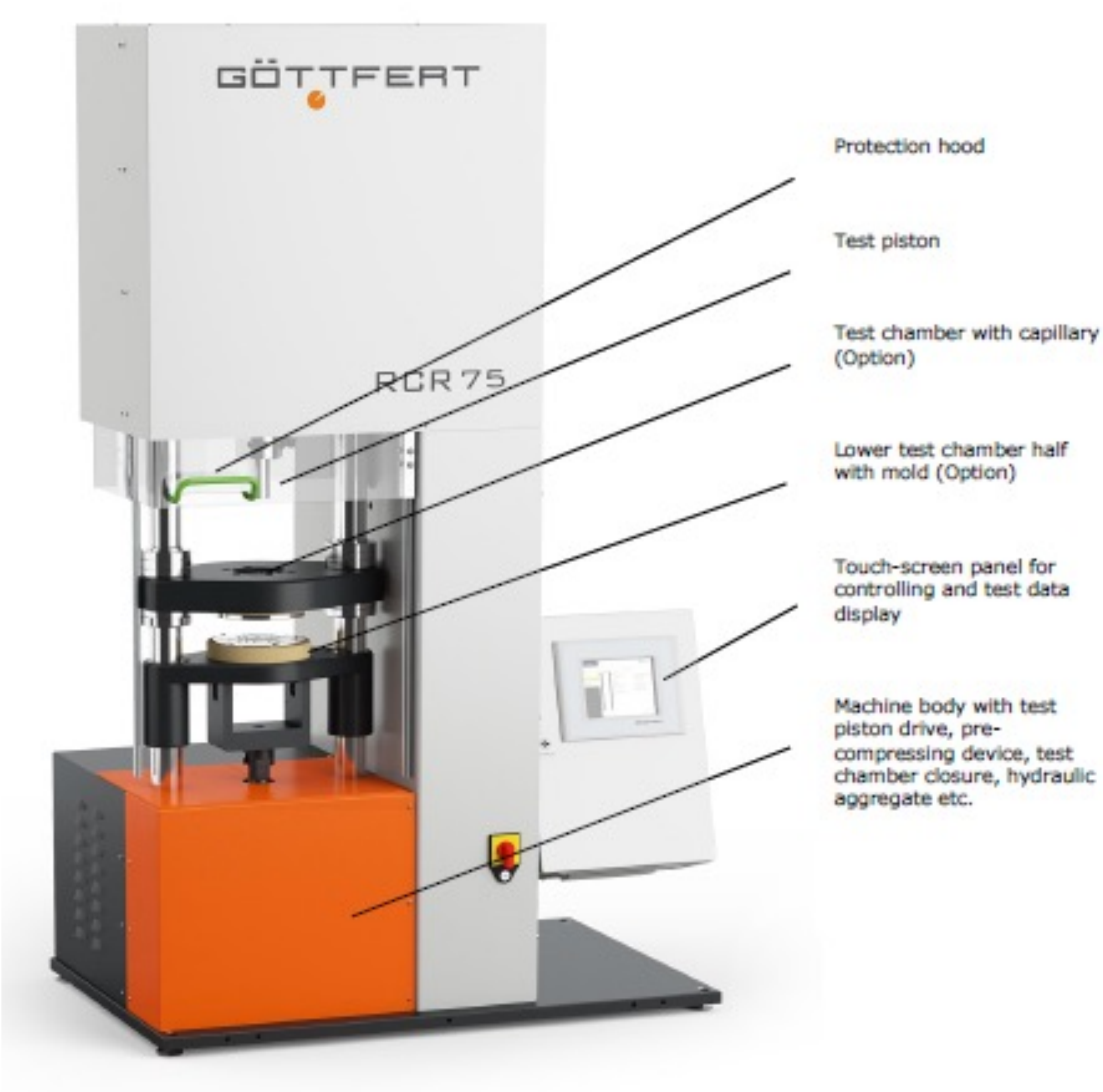
Capillary Rheometer



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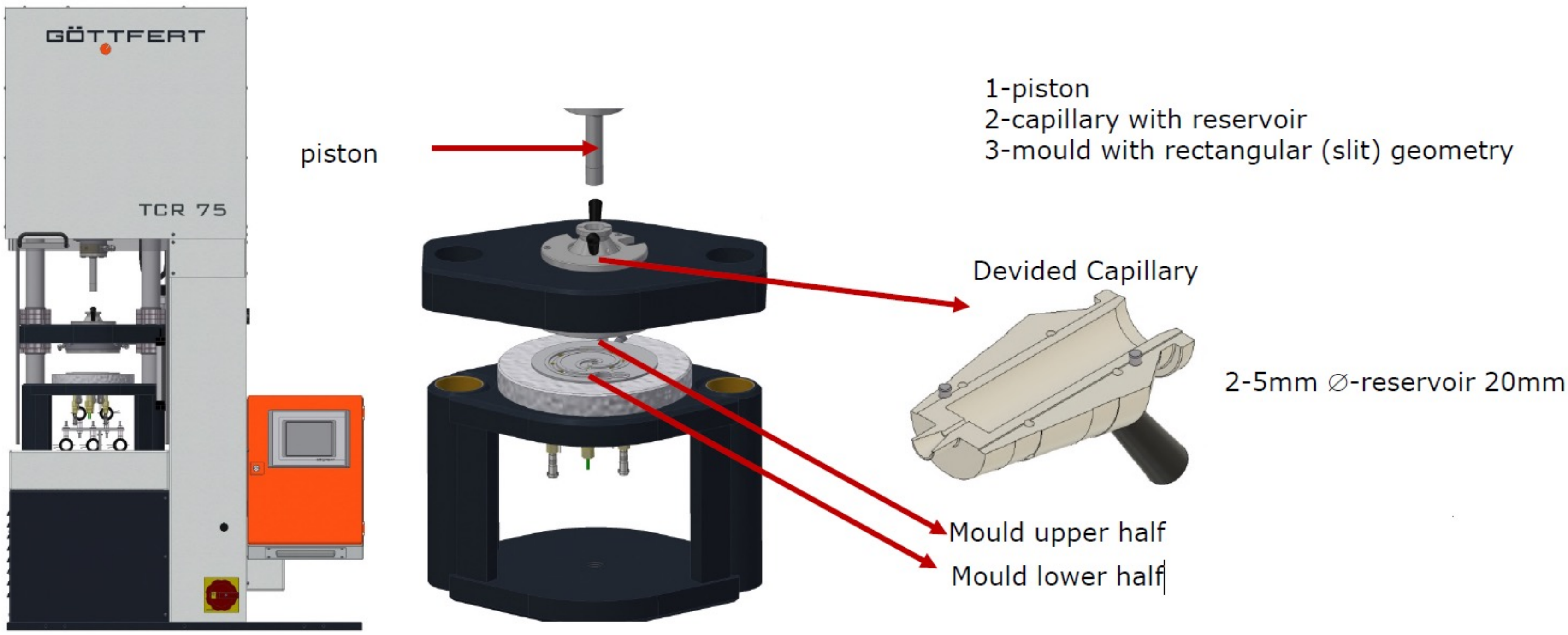
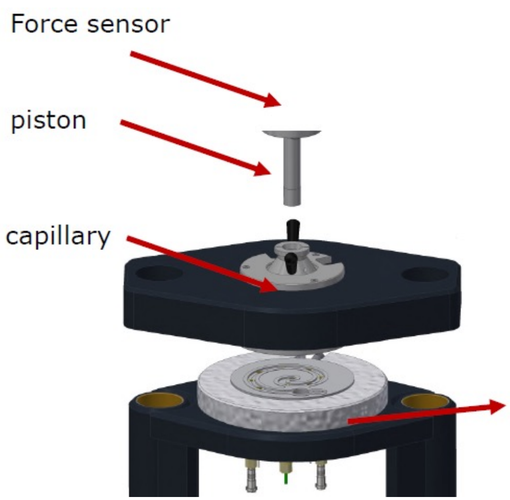
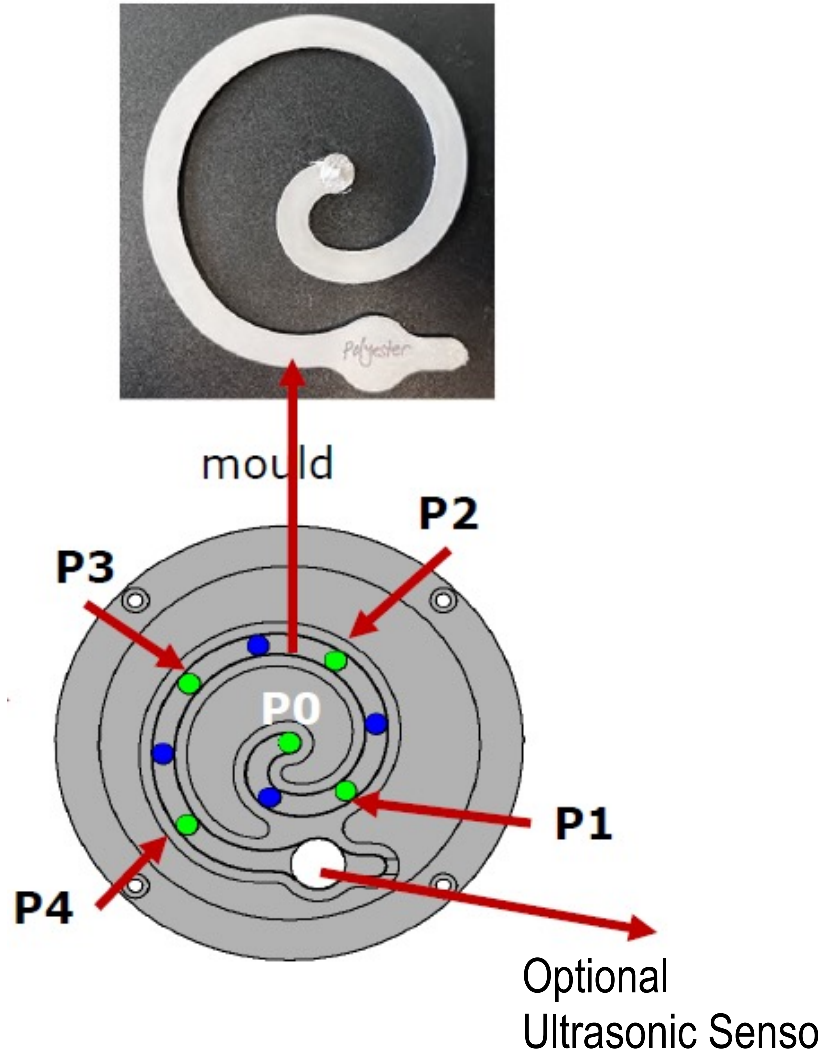
TCR – Thermoset Capillary Rheometer



TCR – Special Mold:



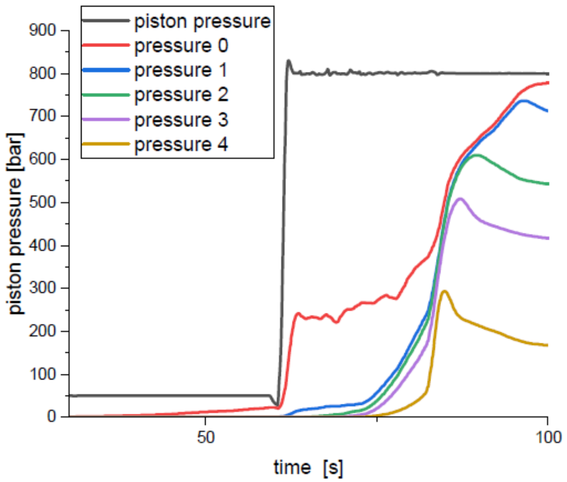


Pressure transducers

Temperatur gradient sensors

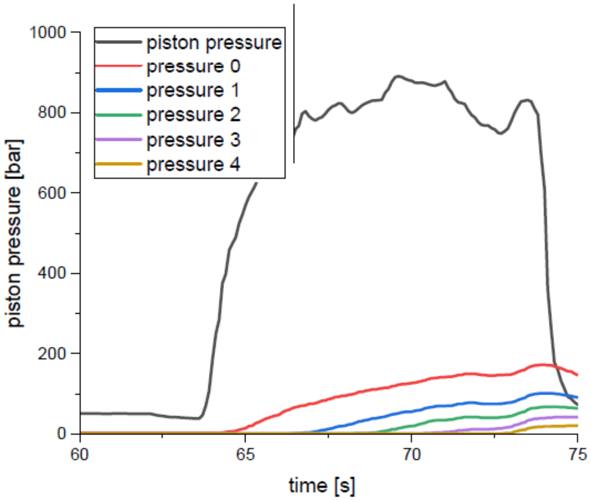
TEST METHODS – CP / CS

Constant Pressure



Mainly used test method

Constant speed

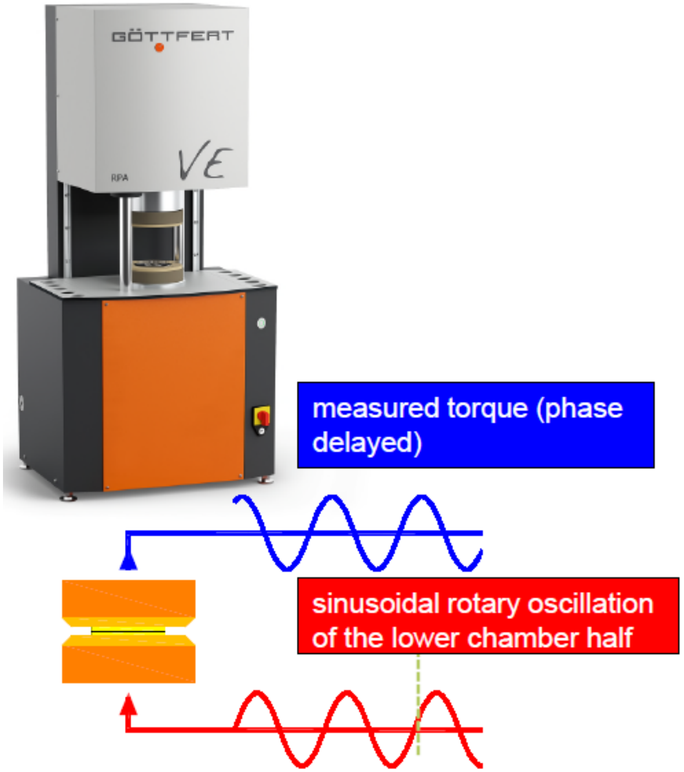


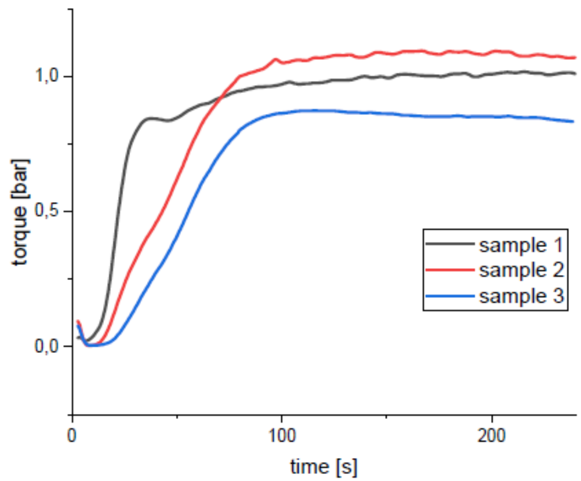
Typical capillary rheometer test to determine the viscosity

Determination of viscosity

capillary	piston pressure –P0
Mould	P1-P0
	P2-P1
	P3-P2
	P4-P3

Resin Curing with RPA

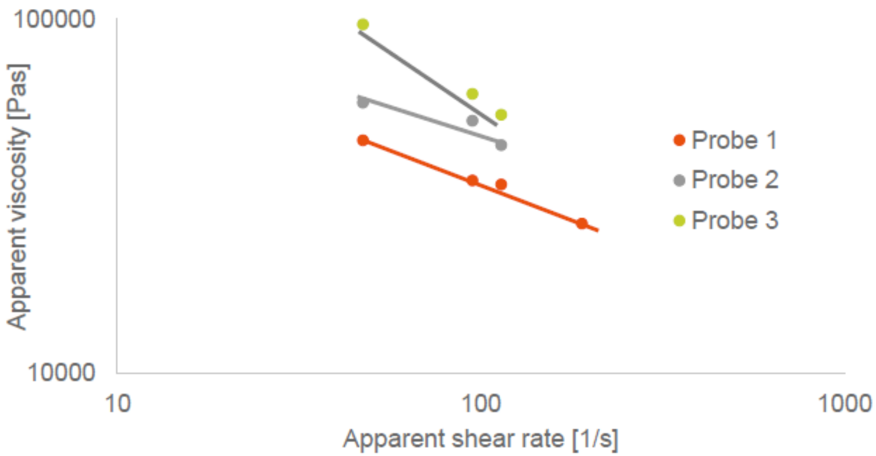


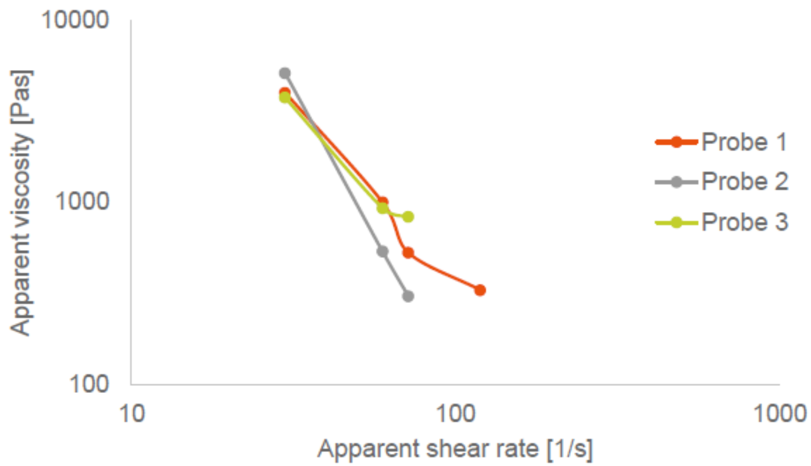


- Cure temperature 160°C.
- sample 2 and 3 have about the same minimum, minimum of sample 1 is much higher.
- Probe 1 has a significant higher cure time as sample 2 and 3

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

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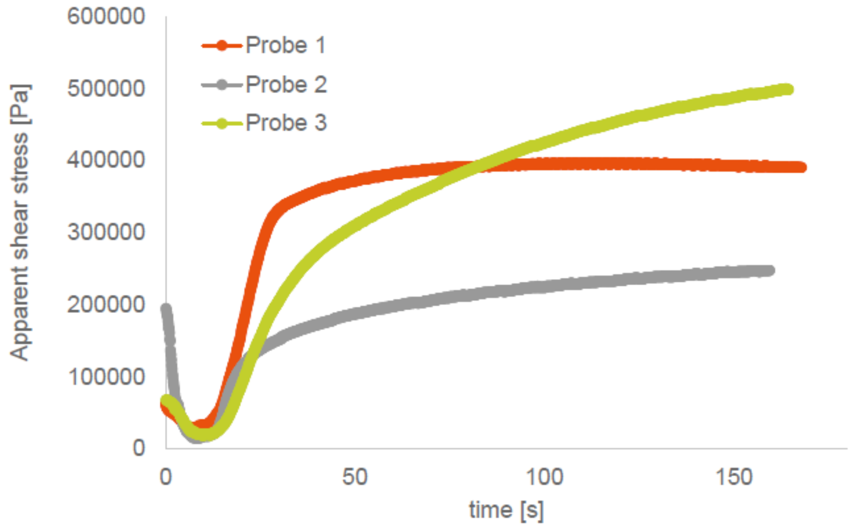
TCR – Phenolic Resin Injection at CP

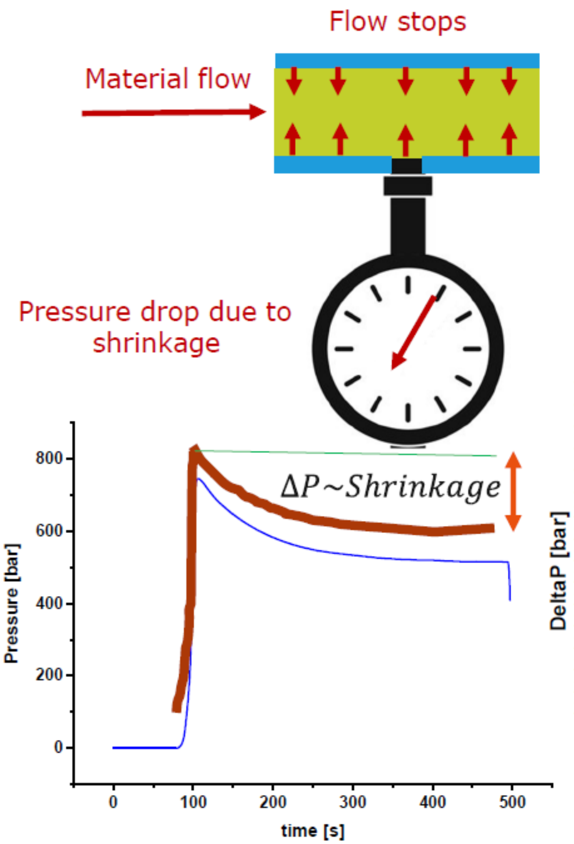
Evaluation of Curing P0 > P1



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

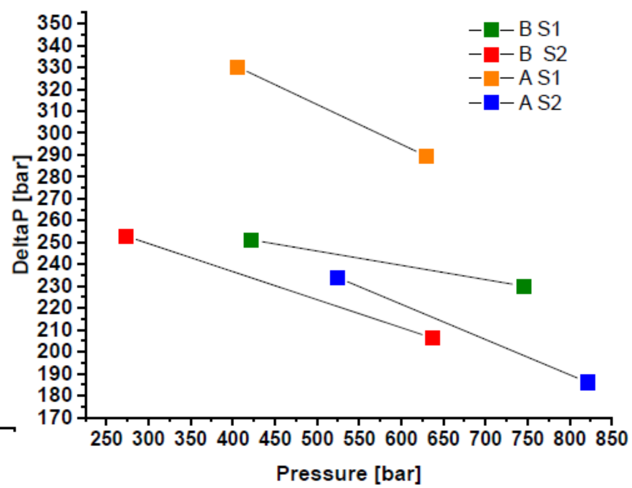
TCR – Determination of Shrinkage

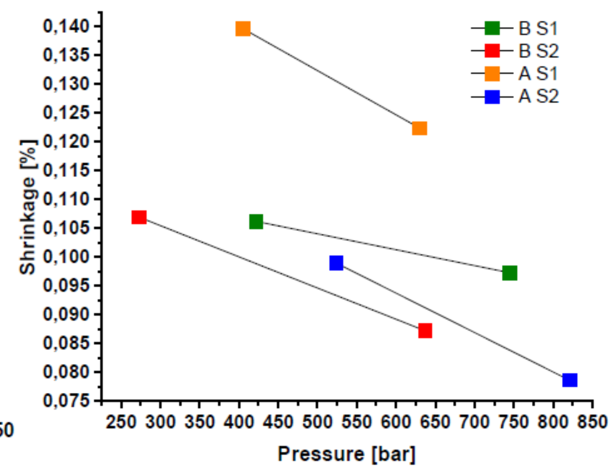


Material flows over the pressure sensor during the injection. Crosslinking increases the viscosity and eventually stops the flow at Gel point.

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

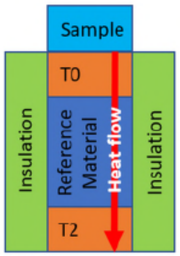
Batch differences can be detected caused by shrinkage





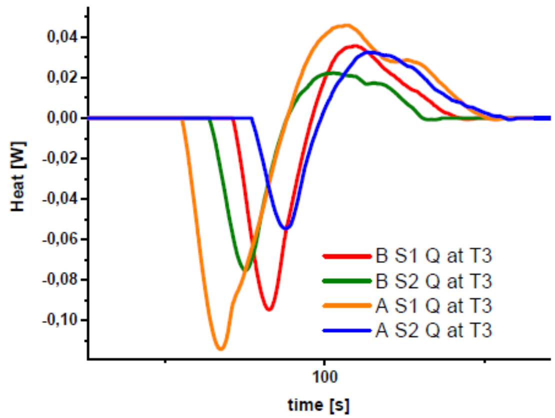
TCR – Thermal Behavior During Curing

The temperature gradient sensors detect a heat flow by measuring a temperature difference over a reference material

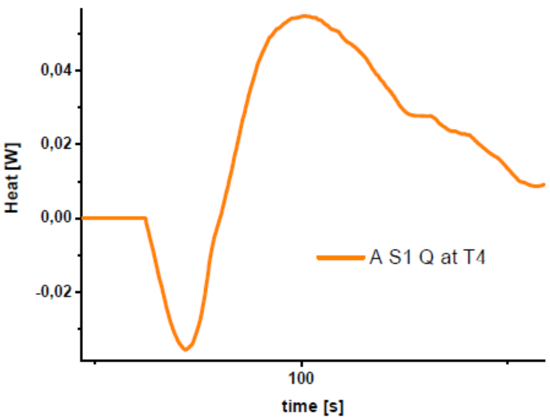


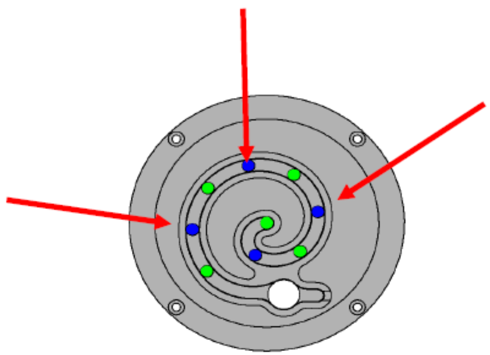
$$Q = -k_R A \frac{\Delta T}{L}$$

$$\Delta T = T0 - T2$$

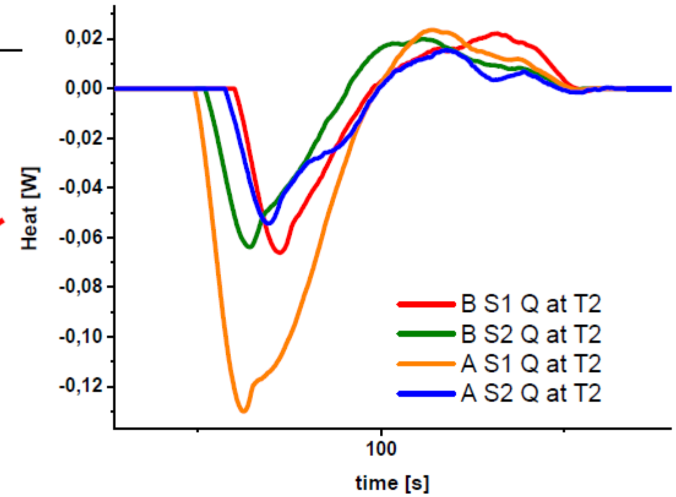


- The samples also show batch differences in the temperature peak.
- The differences in positive heat flow (from the sample) correlates to gel time.
- The negative temperature peak is determined by heating up and activation of the crosslinking.





Thermoset Rheometer



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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?