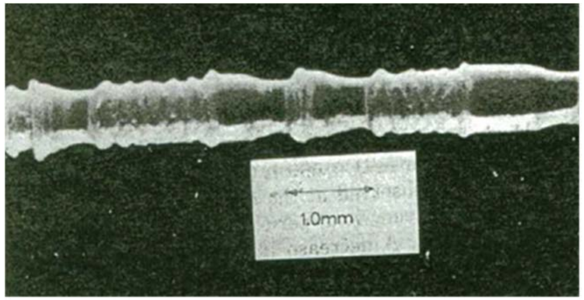
Zwick testXpo 2018

Capillary Rheometry – A Method to Predict Flow Properties under Processing Conditions

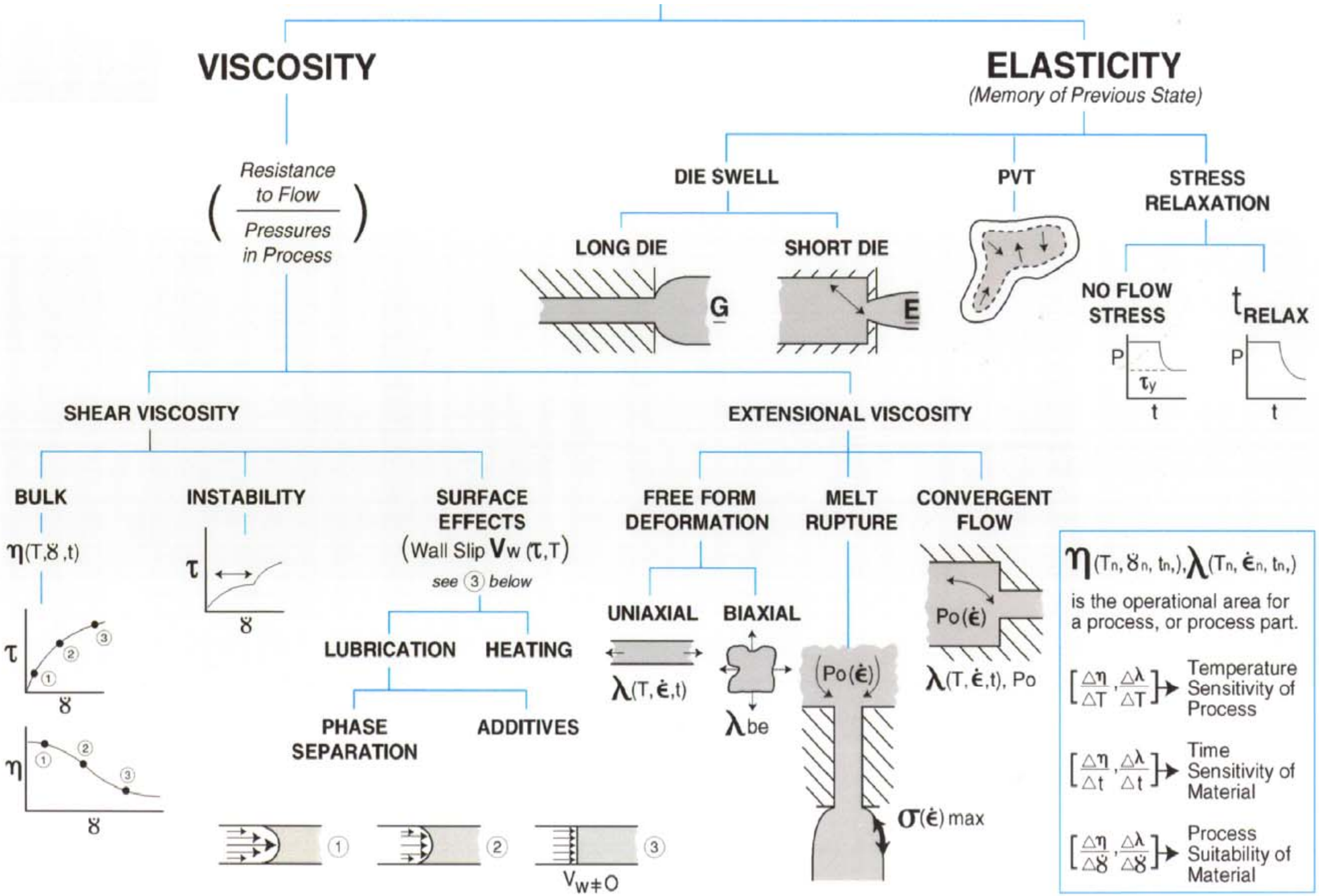
Torsten Remmler, Malvern Panalytical GmbH



Overview

- Range of Applications for Capillary Rheometry
- Introduction into capillary rheometry: Principle of Operation and theoretical background
- Test results on LDPE: Complete Capillary Characterisation
- Advanced Test Types: Relaxation, Thermal Degradation etc.

Capillary Rheometry: Main Applications



Typical Shear Rates

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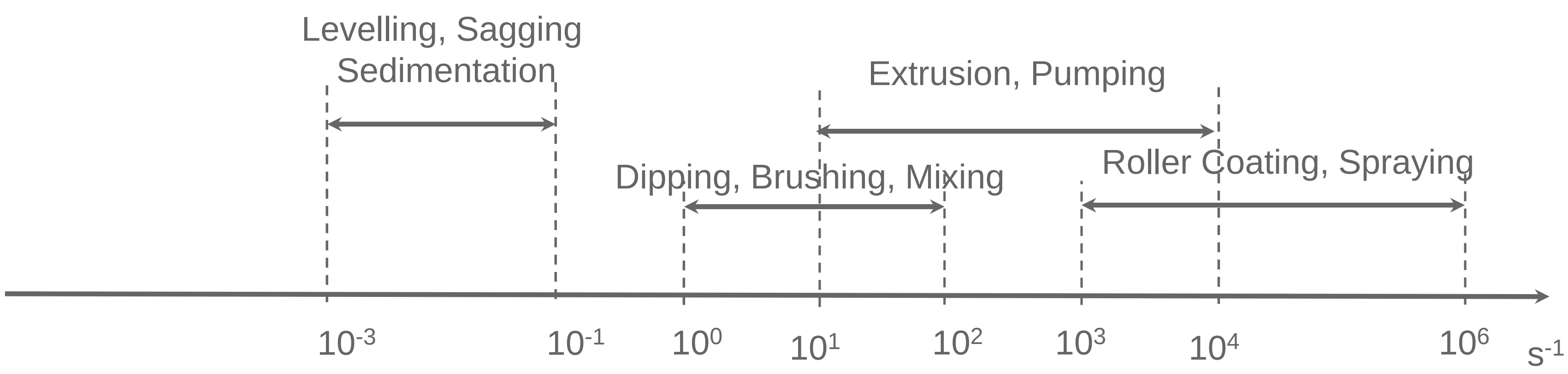


Rotational-Rheometer

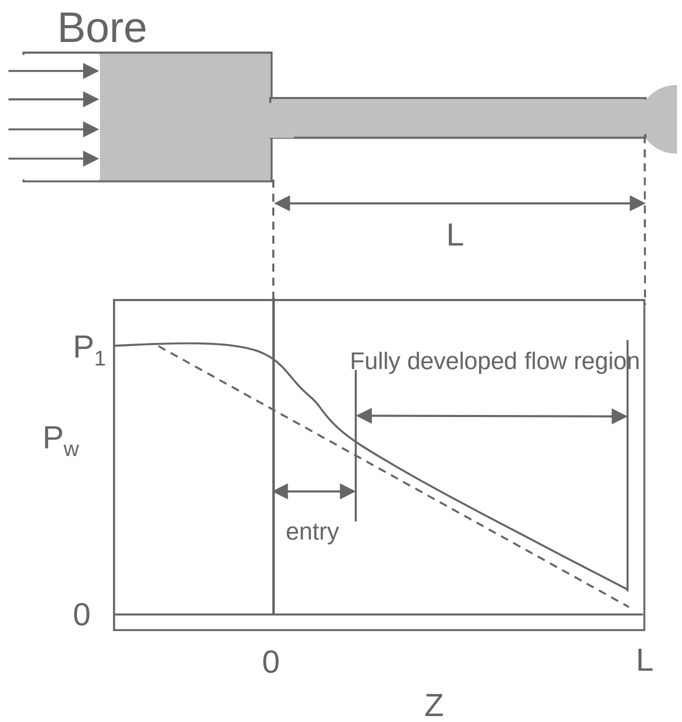
Sample: Water up to solids
Results: Shear-Viscosity, Yield Stesses, Visco-Elasticity, Relaxation...

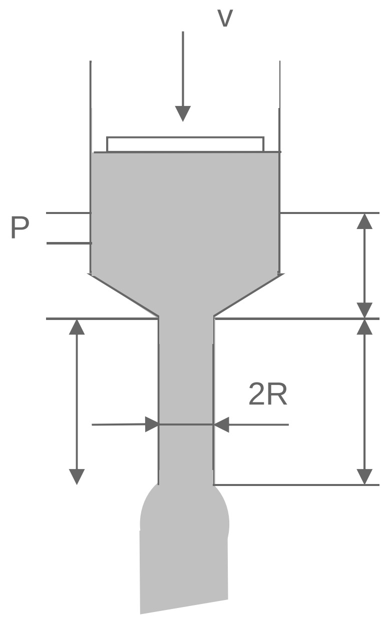
High Pressure Capillary-Rheometer

Sample: Water up to high viscous
Results: Shear-Viscosity, Elongational-Viscosity, Wall Slip...

Principle of Operation

Given quantity: piston speed $\Rightarrow$ wall shear rate
Measured quantity: pressure drop $\Rightarrow$ wall shear stress





Full pressure drop

=

Entrance pressure drop

+

Shear pressure drop

$\Rightarrow$ small ram extruder

Laminar Pipe Flow

Isothermal, stationary Flow of an incompressible fluid

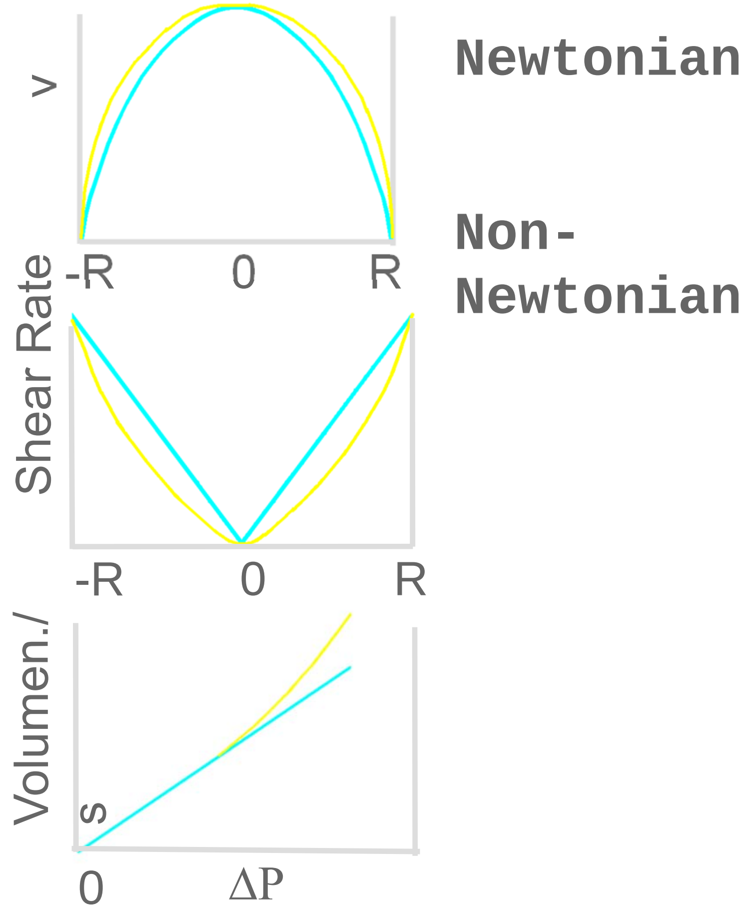
Newtonian

$$\dot{\gamma}_{app} = \frac{4 \cdot Q}{\pi R^3}$$

$$\sigma_{app} = \frac{R \cdot \Delta P}{2 \cdot L}$$

Q=Volume Flux, R= Die Radius

L=Die Length, ΔP=Pressure Drop



What are we doing to get flow curves?

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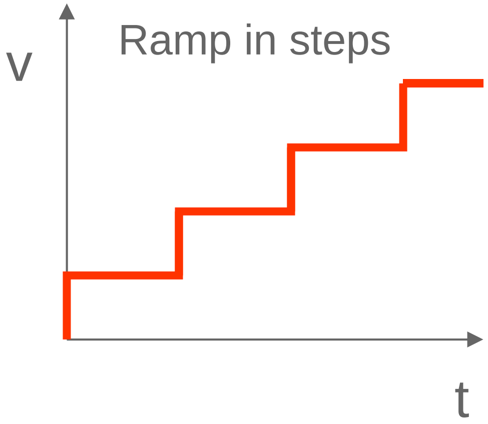
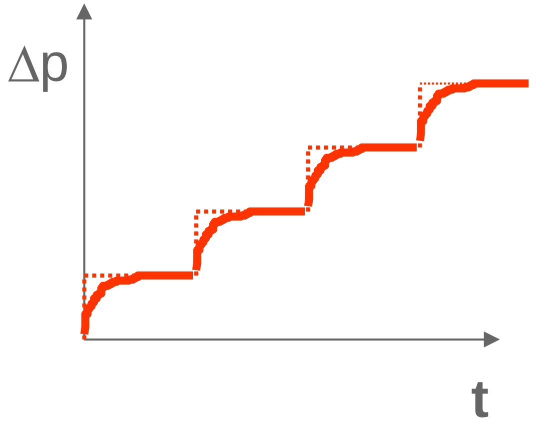
measurement :





$$\dot{\gamma}_{app} = \frac{4 \cdot Q}{\pi R^3}$$

$$\sigma_{app} = \frac{R \cdot \Delta P}{2 \cdot L}$$

corrections →

$$\eta = \frac{\sigma_{true}}{\dot{\gamma}_{true}}$$

Correction:
Entrance zone of a capillary die

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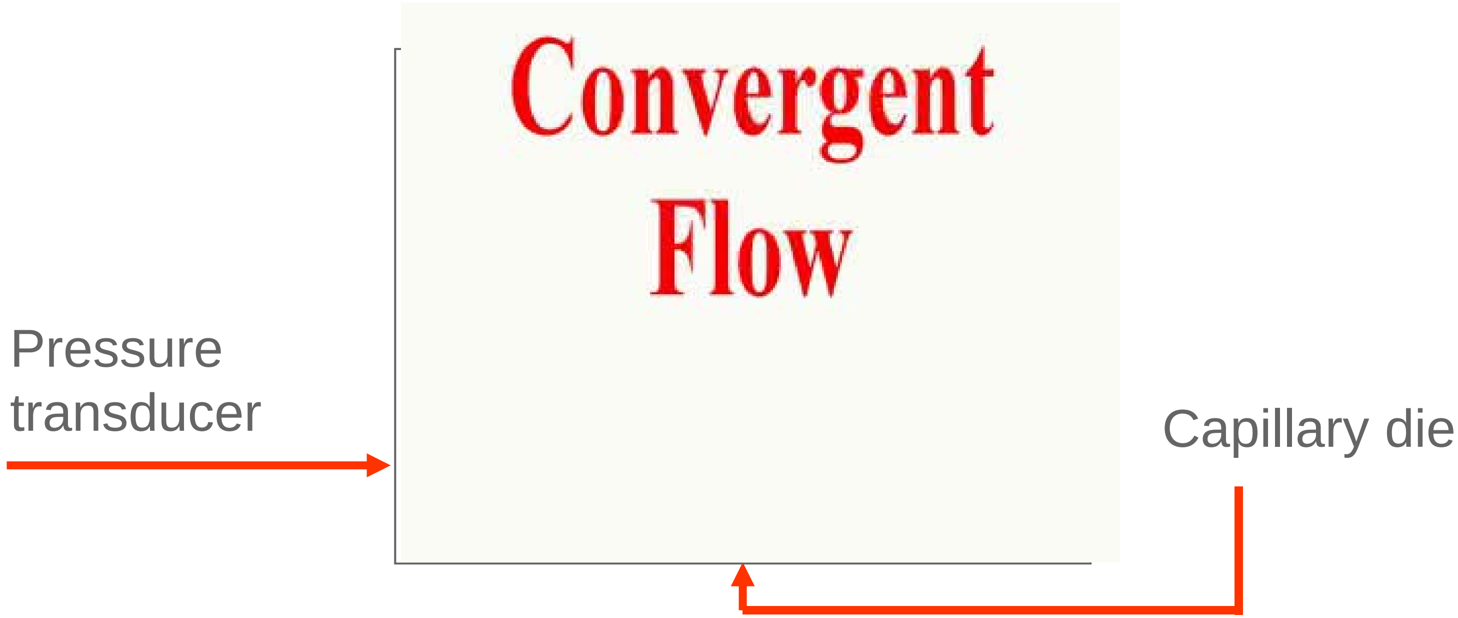
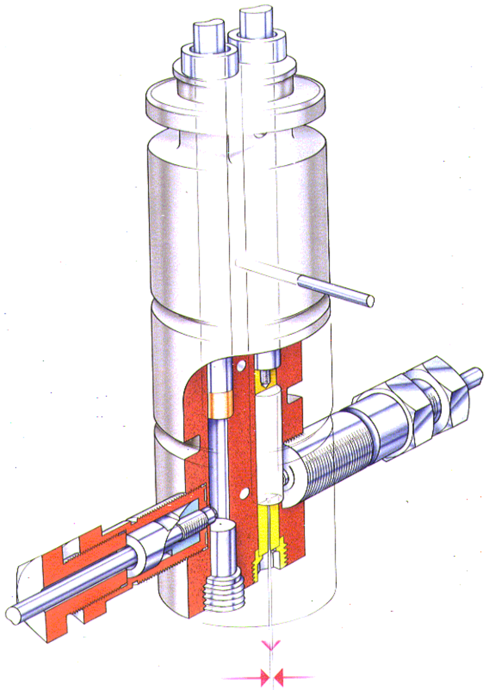
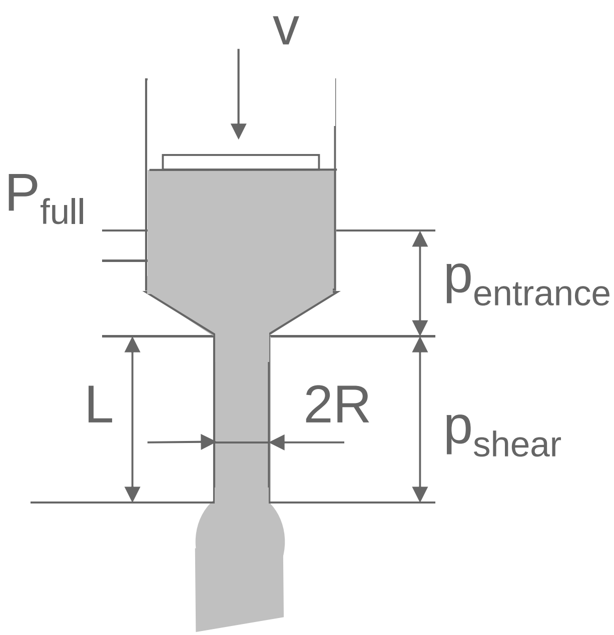
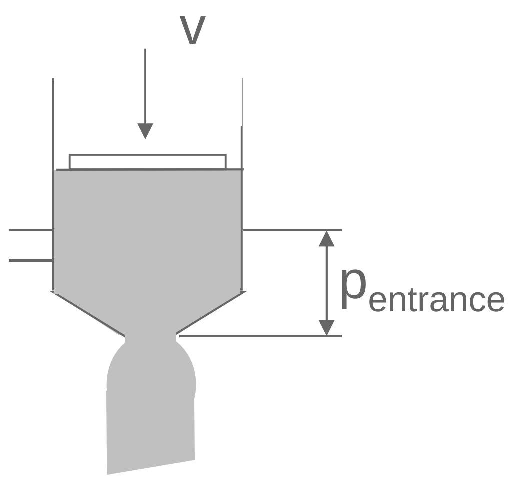


Aim of the test: to separate entrance pressure and shear pressure drop!

Rosand Twin Bore Principle







$P_{full} = P_{shear} + P_{entrance}$

left: capillary

right: orifice

How do we get the Extensional Viscosity?

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Cogswell`s Convergent Flow Model ⇒ Extensional Viscosity

$$P_{full} = P_{shear} + P_{entrance} \longrightarrow \lambda = \frac{9\,(n+1)^2\,(P_e)^2}{32\,\eta\,\dot{\gamma}^2}$$

- **Special Orifice Die according to Uni Zlin Model enables characterisation of very small extensional rates too.**

$$n = \frac{d\,(\log \sigma)}{d\,(\log \dot{\gamma})}$$

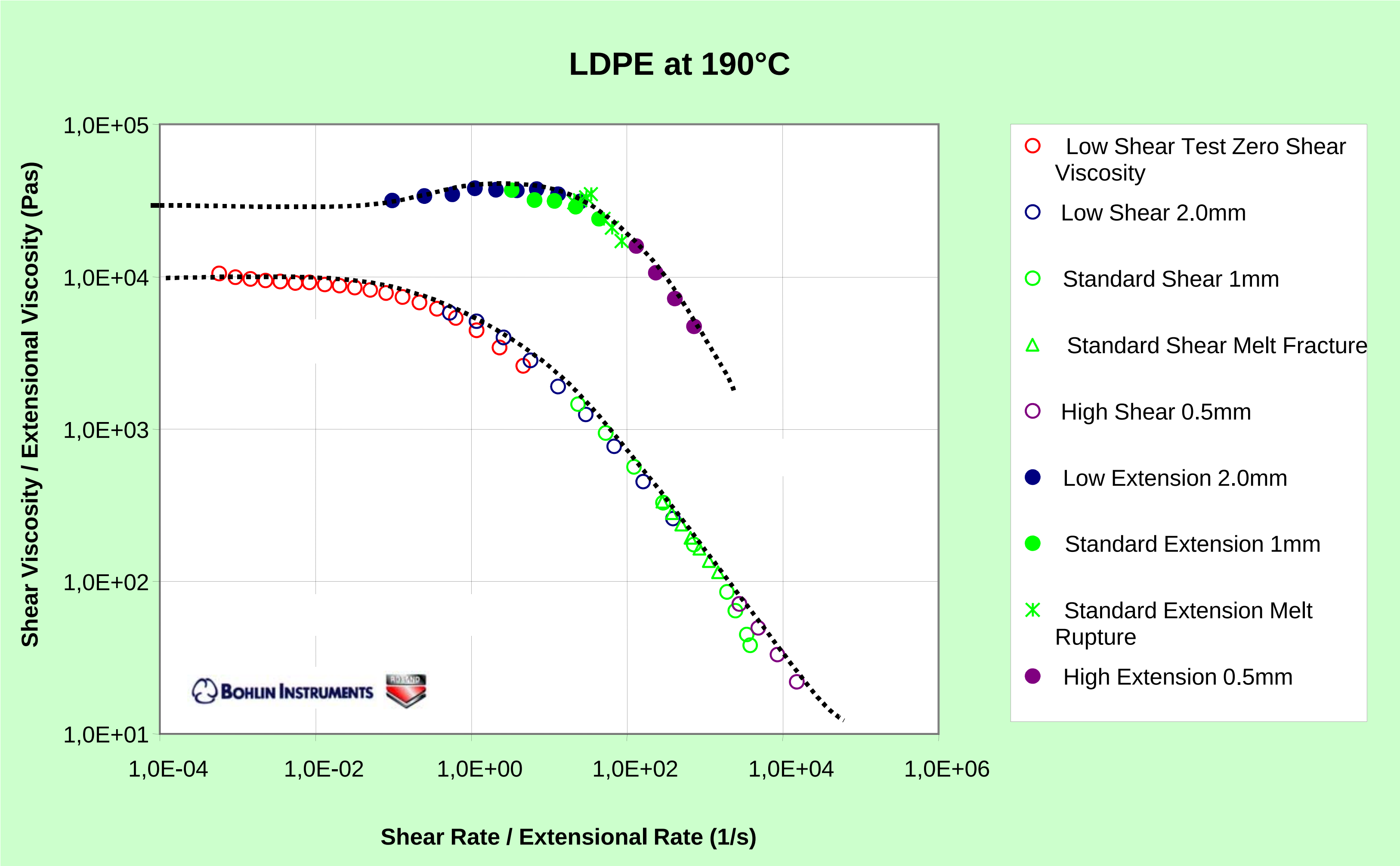
Non-Newtonian Index (Ostwald-de Waele)

$$\dot{\varepsilon} \approx 10^{-1} - 10^{3}\ s^{-1}$$

F. Cogswell, “Polymer Melt Rheology”, Woodhead Publishing Limited (1981)

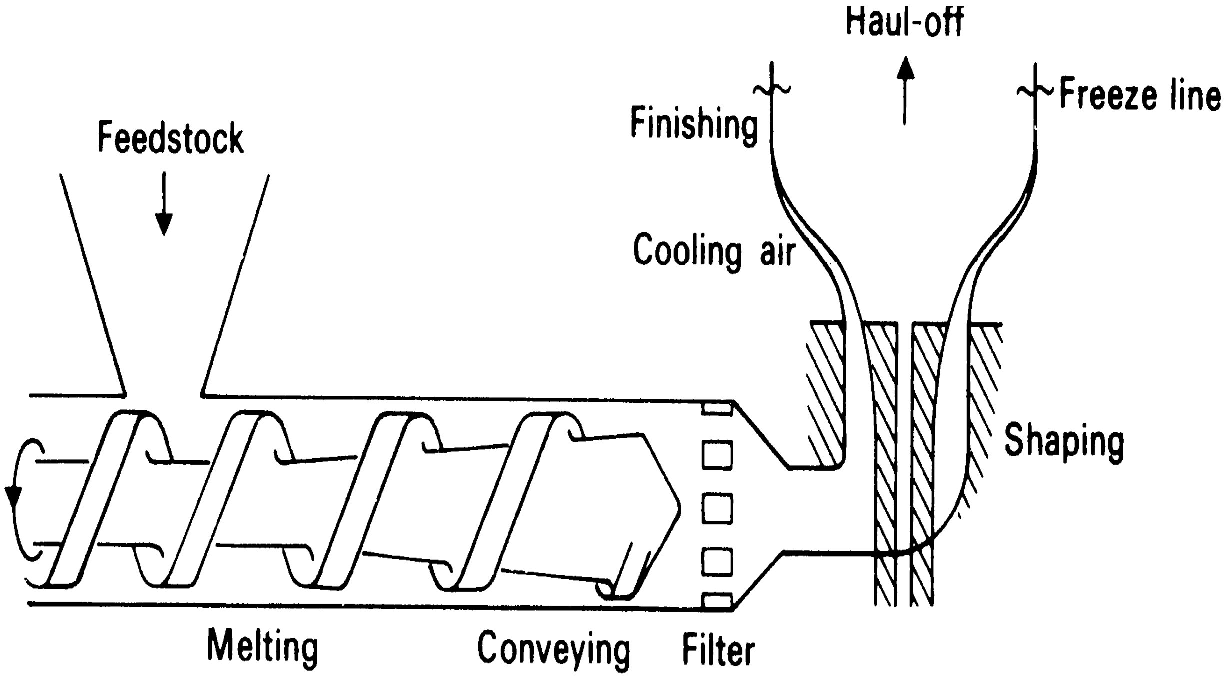
Zatloukal, Vlcek, Tzoganakis, Saha *J. Non-Newtonian Fluid Mech*. **107** (2002) 13–37

Example LDPE



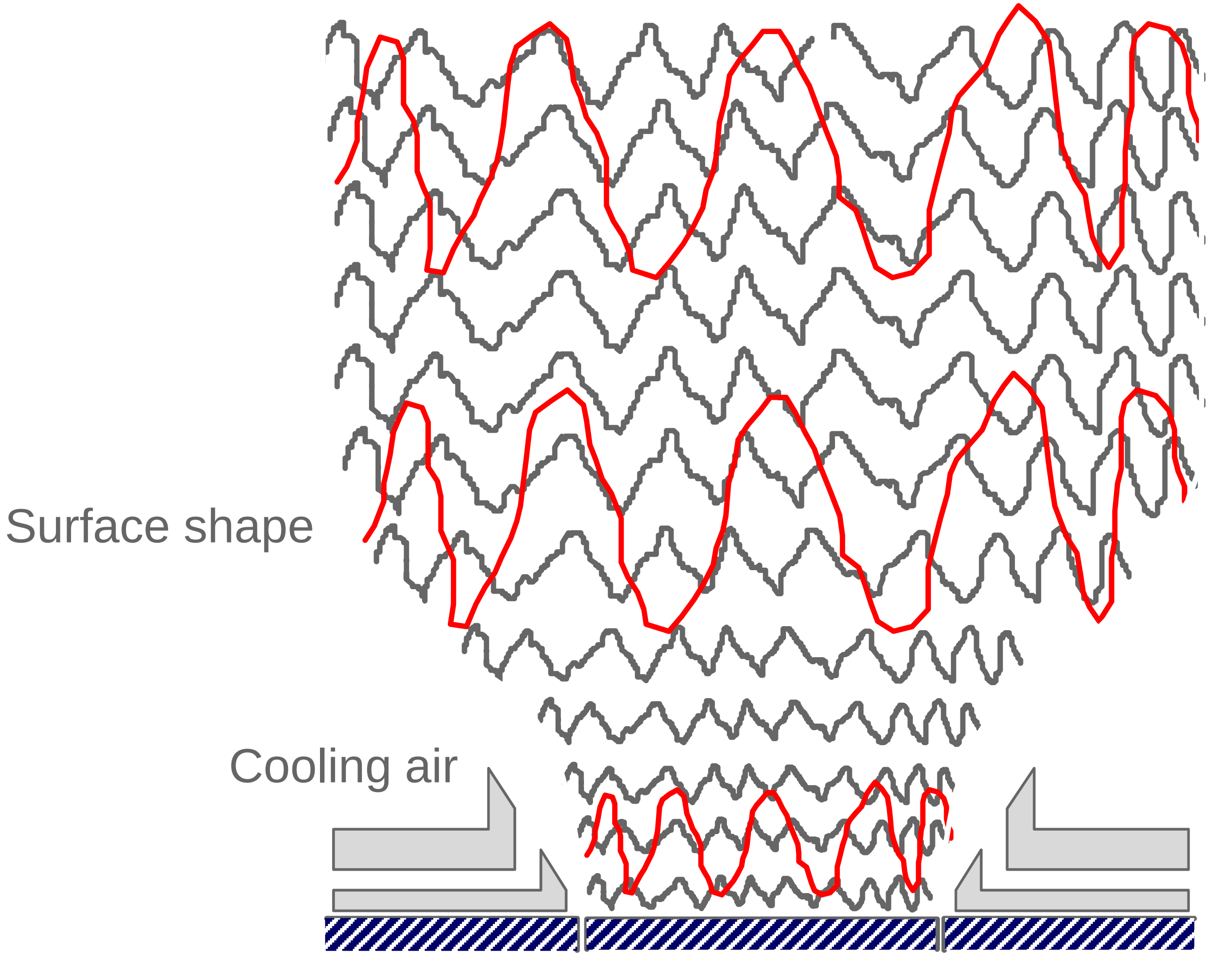
Extensional Rheology of LDPE

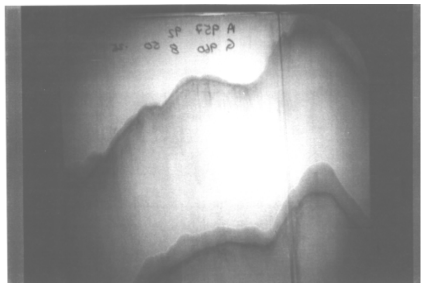
Blow Moulding



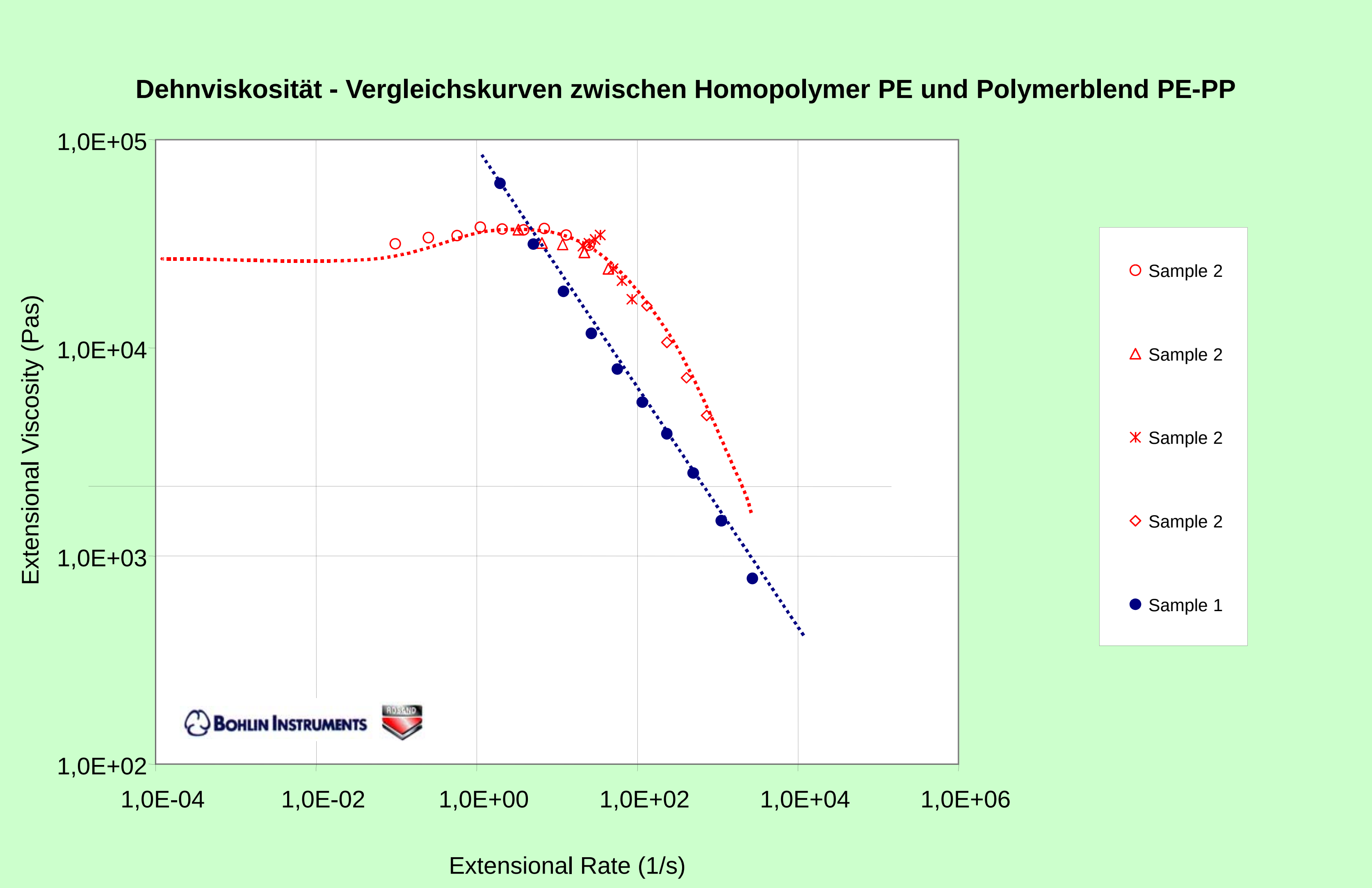
⇒ Blow Moulding is mainly influenced by Extension!

Surface Instabilities LDPE



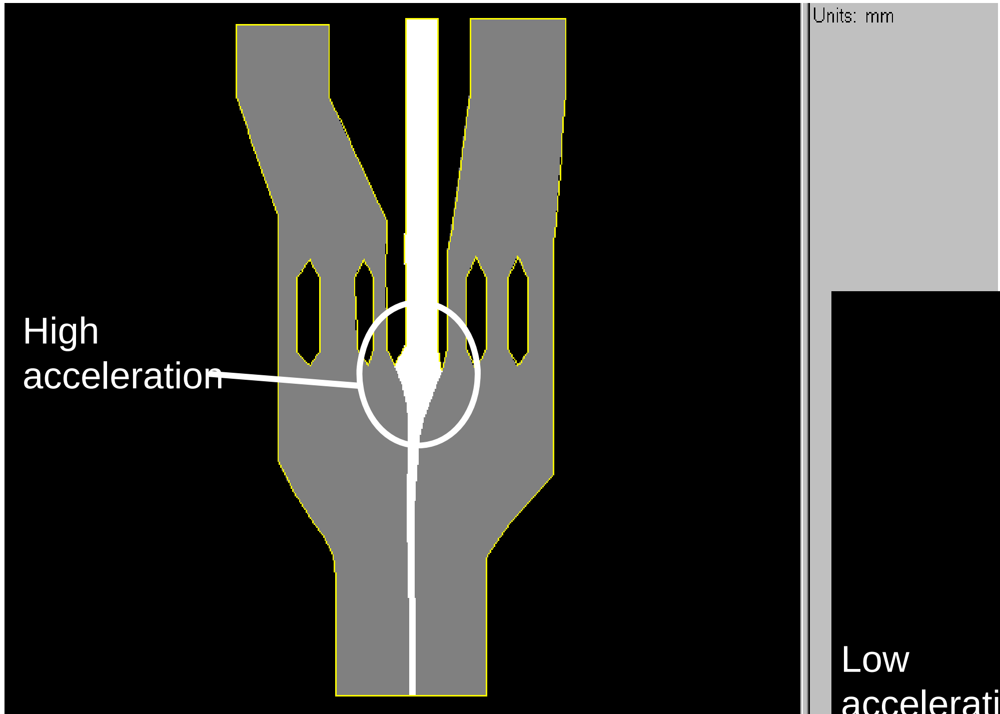


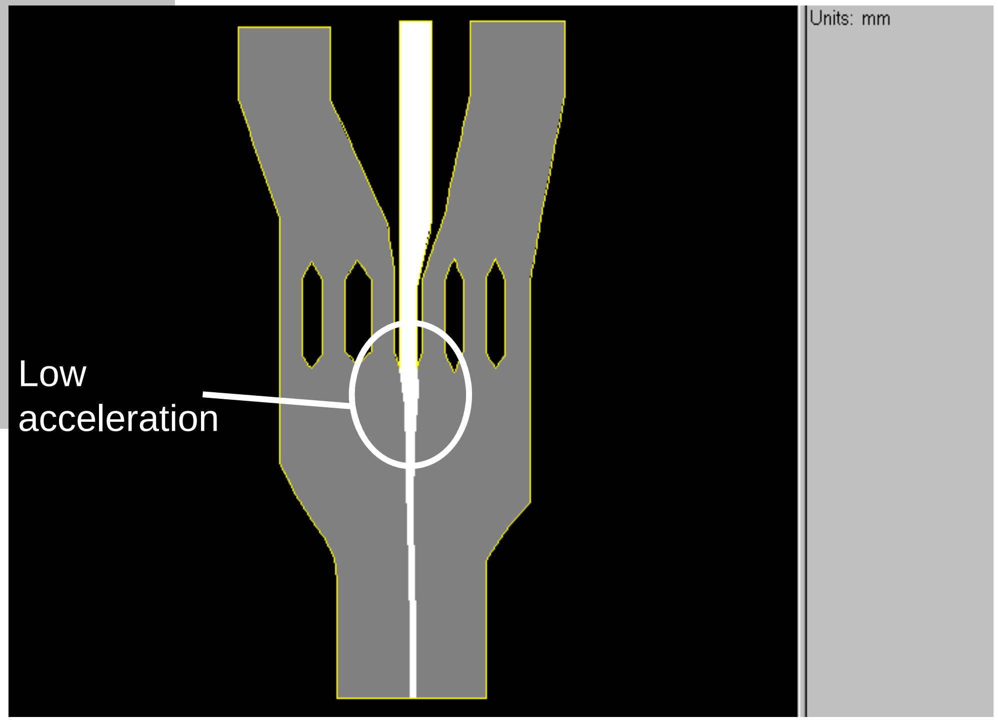
How can the process be improved?



Another Example: Co-Extrusion

Similar instabilites





LDPE in Co-Extrusion Die

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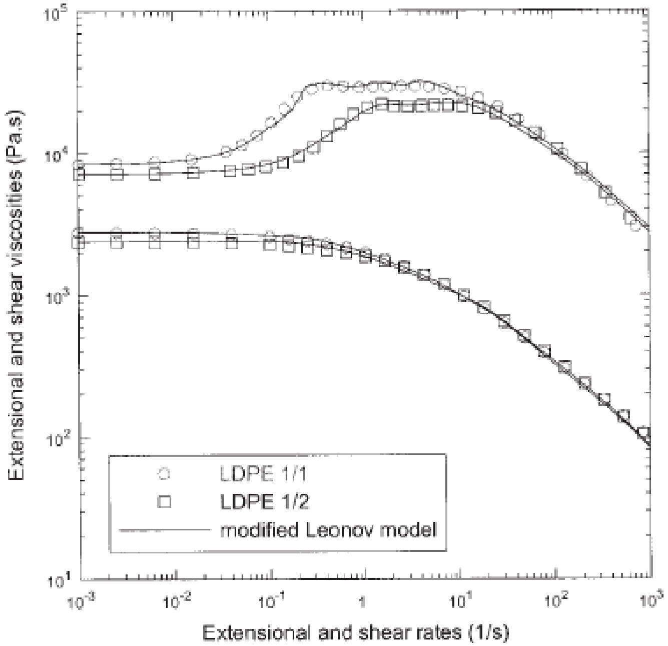
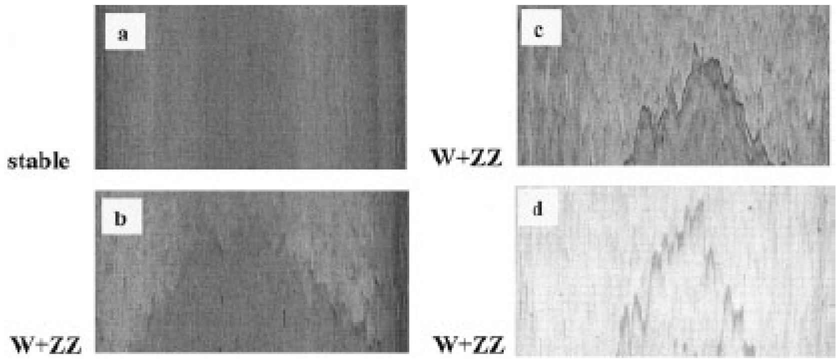


Figure 4 Extensional and shear viscosities for two different lots of LDPE 1, 210°C.



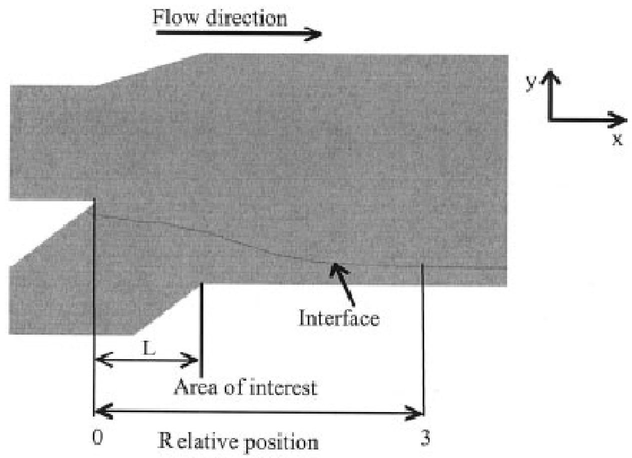


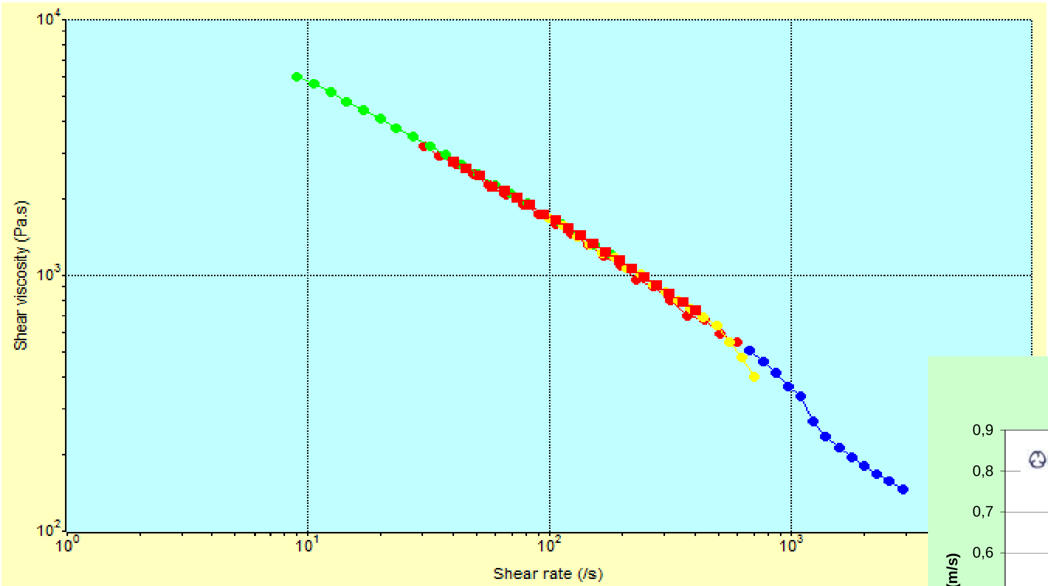
Figure 5 Merging area of the flat coextrusion die.

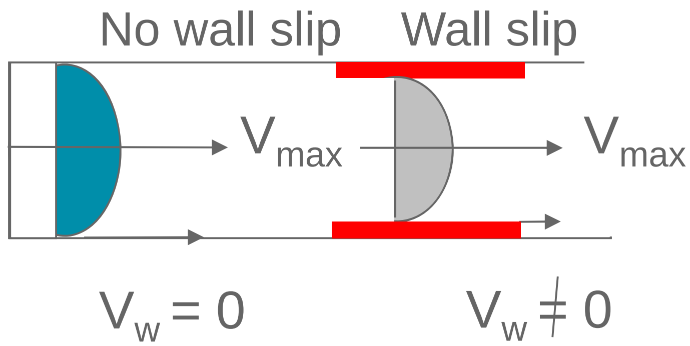
Instabilities caused by Extensional Flow Behaviour of LDPE

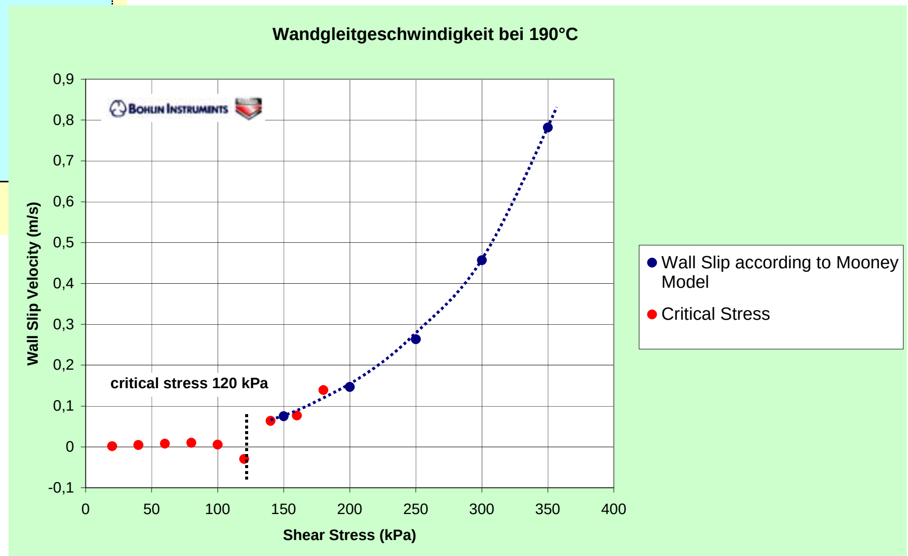
*Zatloukal et. al. Journal of Applied Polymer Science, **98** (2005) 153*

Further Applications: Wall Slip

- Wall Slip Velocity of chromium catalyzed HDPE at 190°C







- Wall slip velocity increases dramatically at just above 0.1 MPa.

Equilibrium Pressure: Homogeneity

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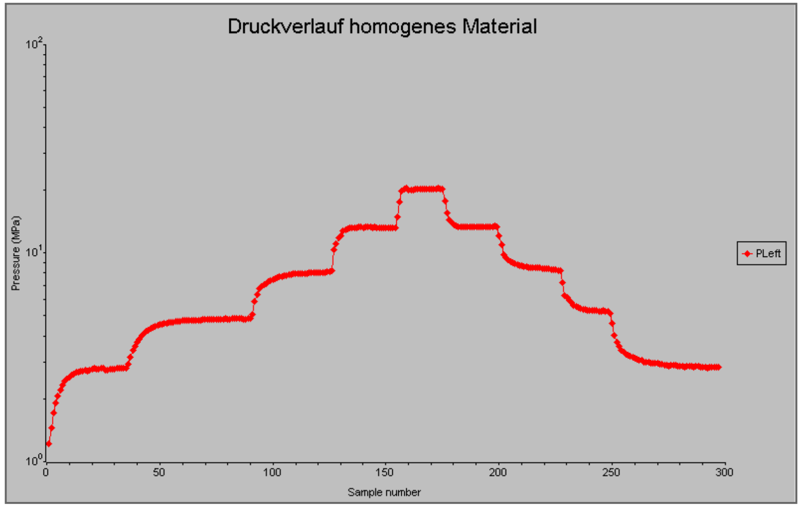
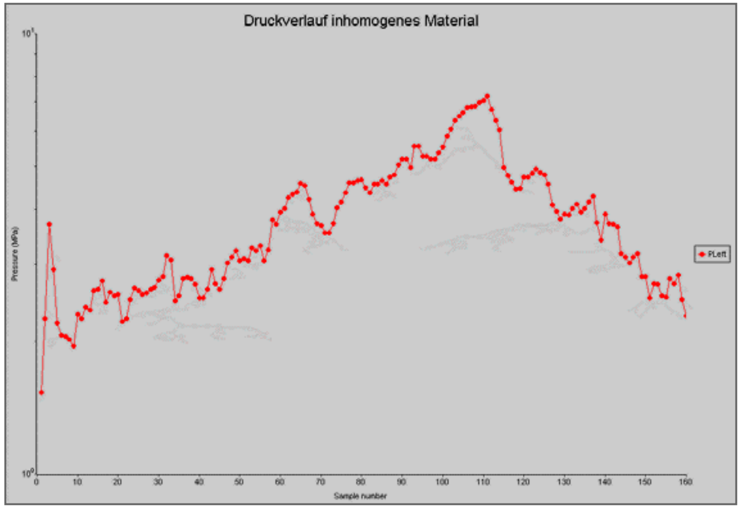
Pressure drop is important





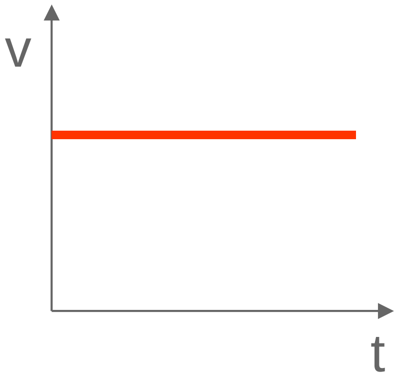
homogeneous

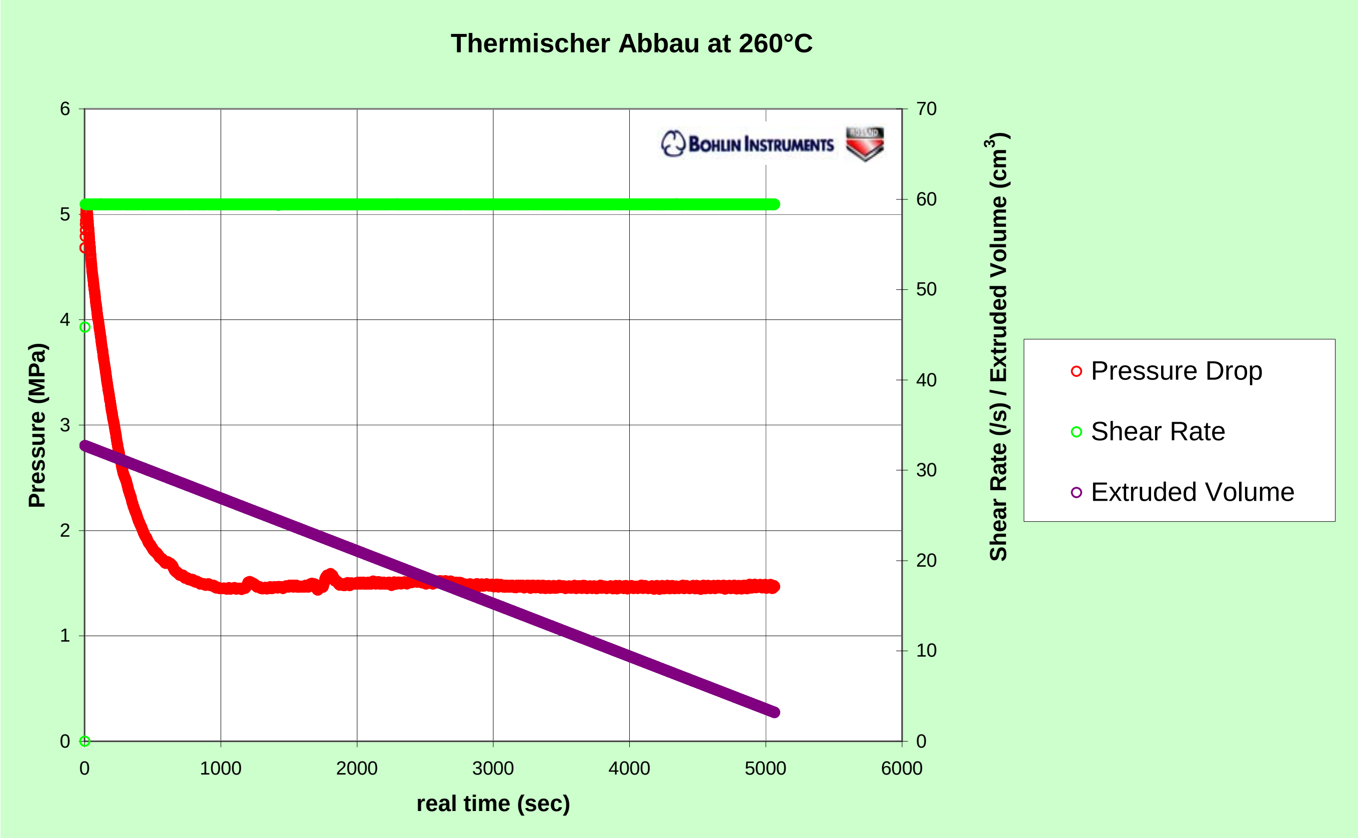
inhomogeneous

⇒ For polymer blens, filled polymers, suspensions, emulsions, composites etc.

Thermal degradation / Curing

Prinzip:



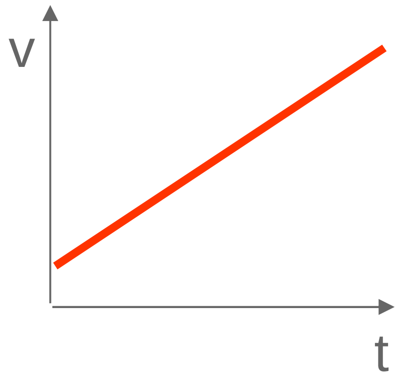


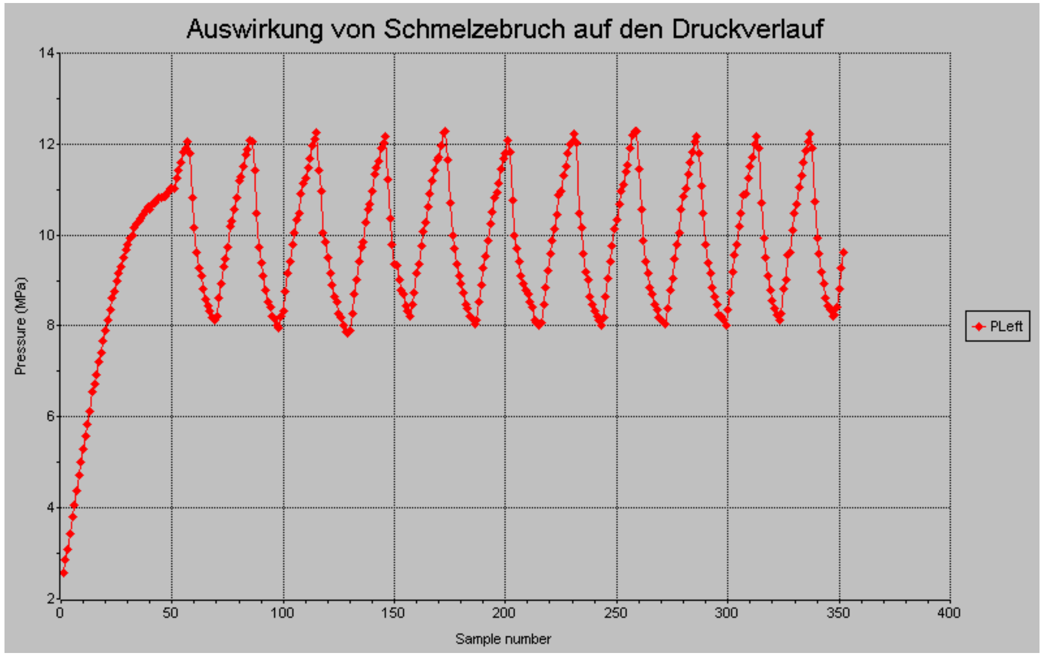
⇒ Gives maximum process times under high temperatures

Stick-Slip

Flow Instabilities

Linear Ramp





Melt fracture

⇒ What is the max processing pressure / Shear Rate?

Melt Fracture

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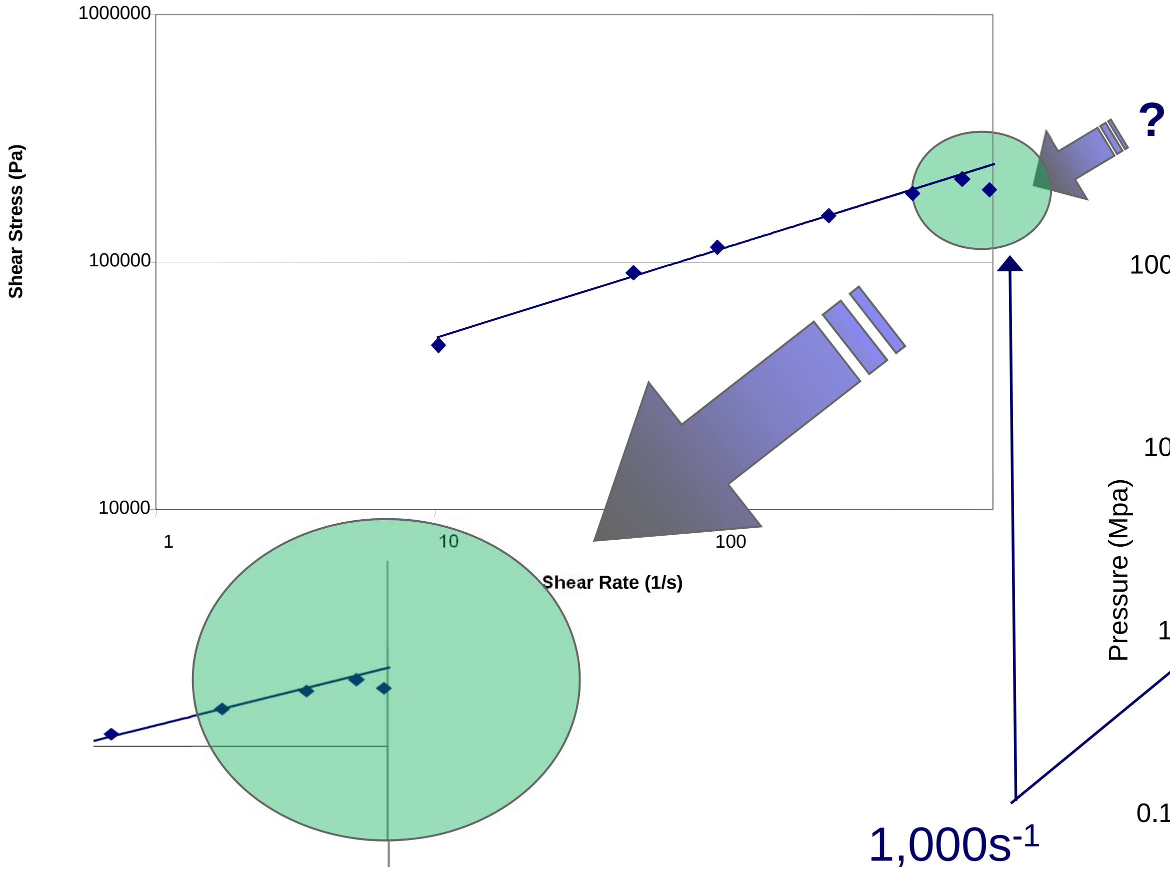
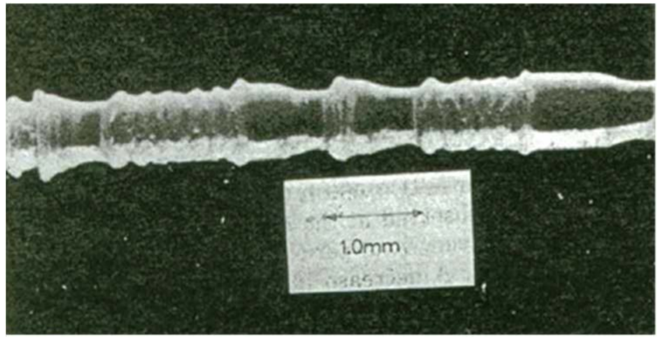
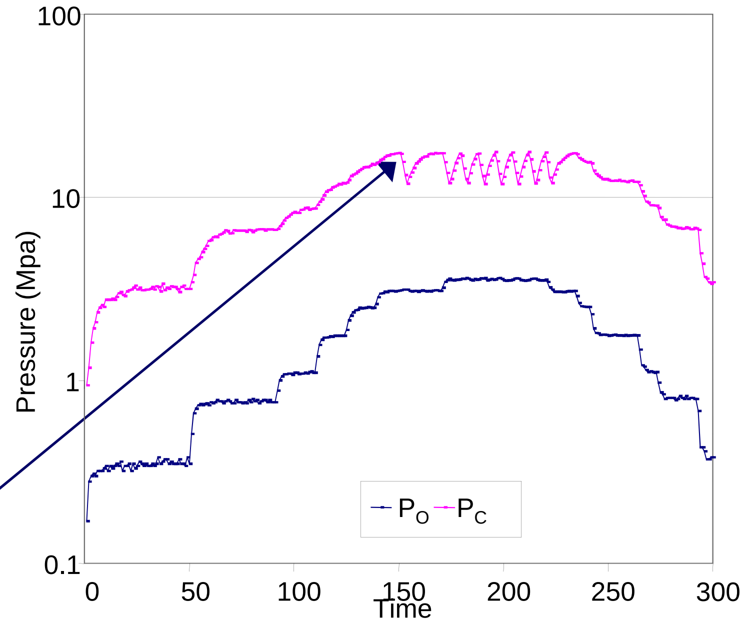
- Unstable flow, poor product quality.



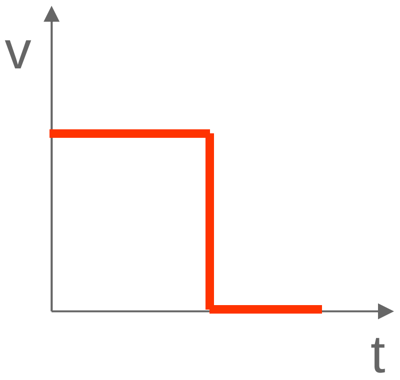


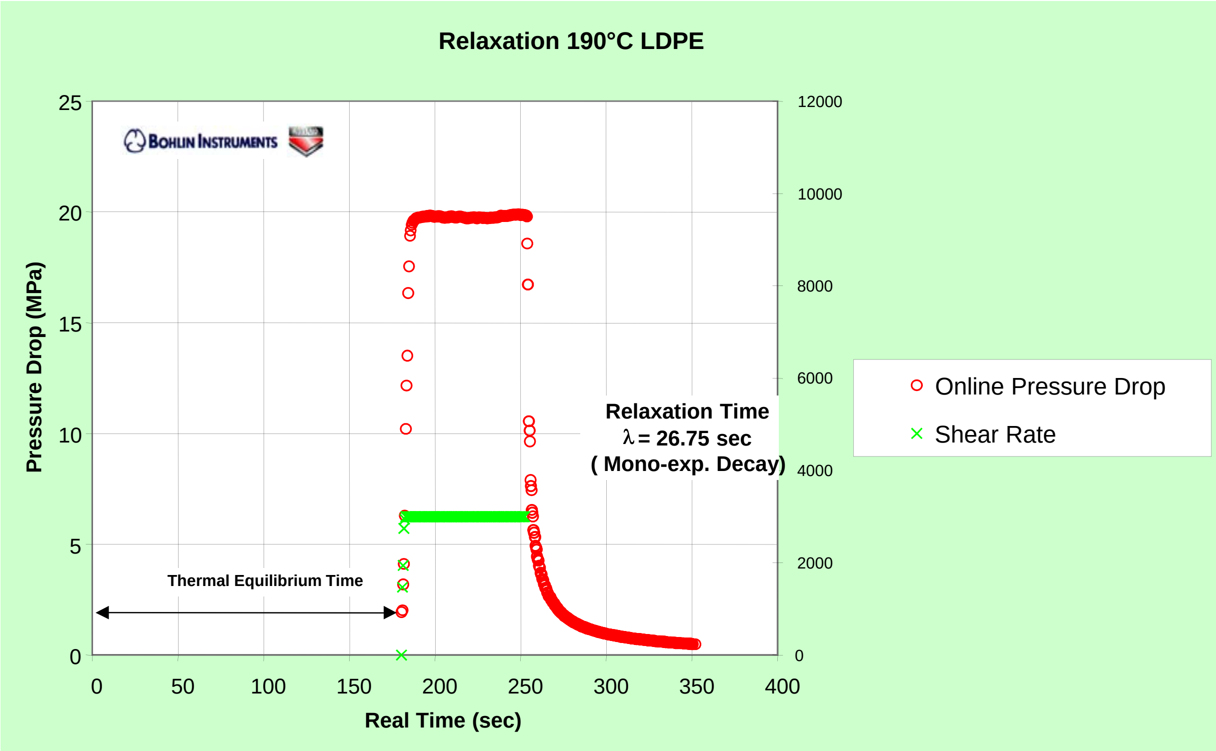


Relaxation LDPE

What happens after processing

Prinzip:





⇒ inner stresses can lead to surface crack (automotive industry)

Rheometer Types

Benchtop RH2000 and Floor Standing RH7/10

Summary

- Capillary Rheometry gives correlation with processing flow properties
- Calculation of extensional viscosity according Cogswell method
- Flow curves up to very high shear end extensional rates
- Prediction of flow instabilities
- Correlation with structural changes during processing

Thank you for your attention.

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