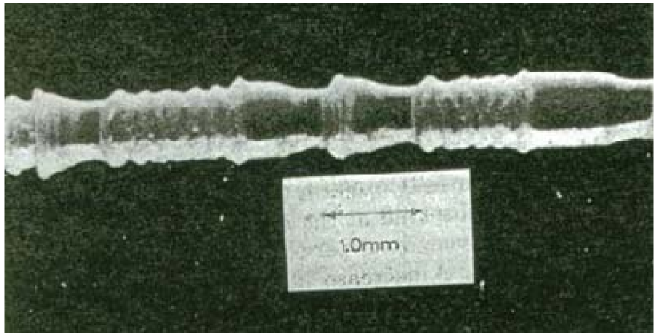
Brno, 28-29th march 2012 – School of Rheology

# Part II: Capillary Rheometry

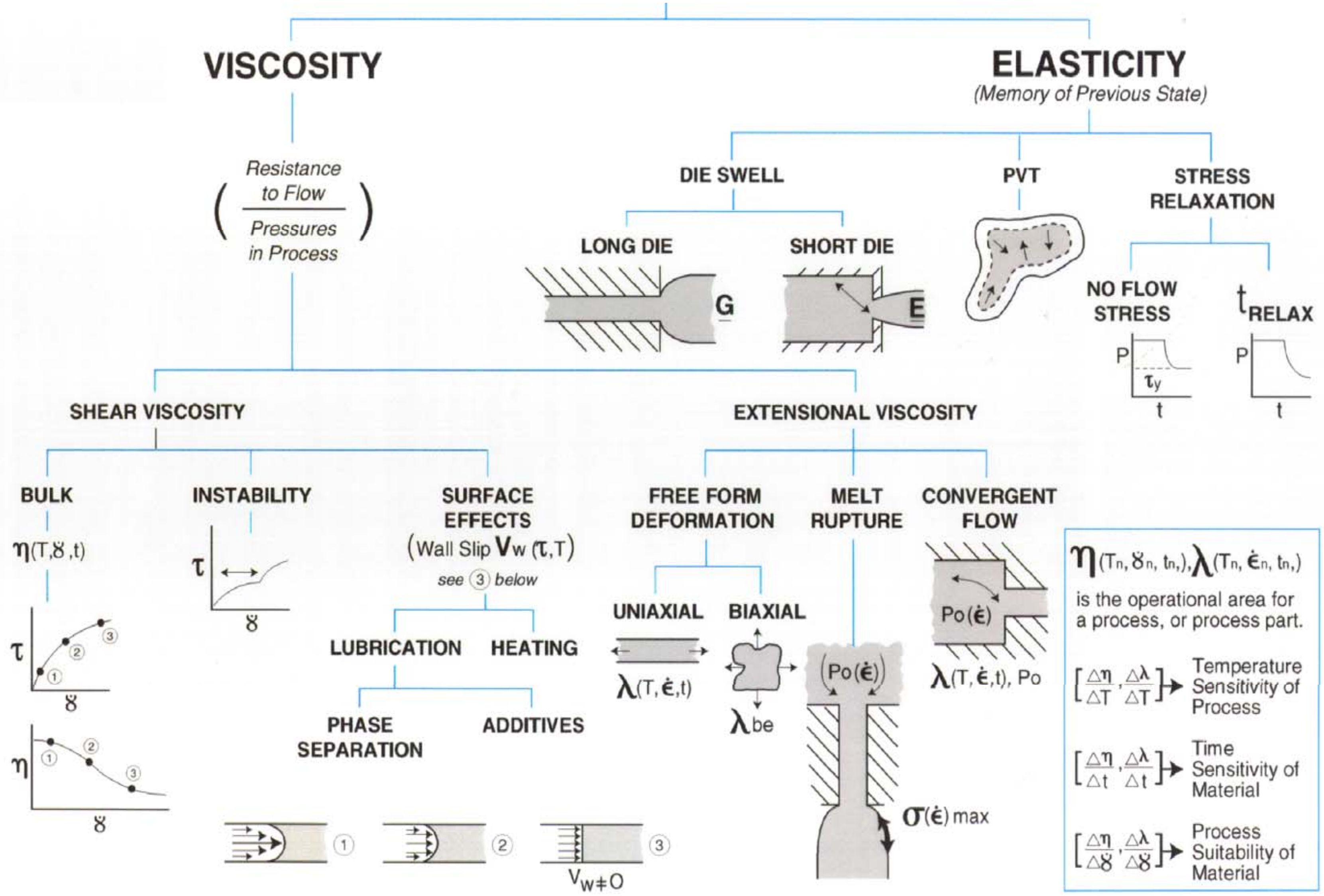
# A method to predict flow properties under processing conditions



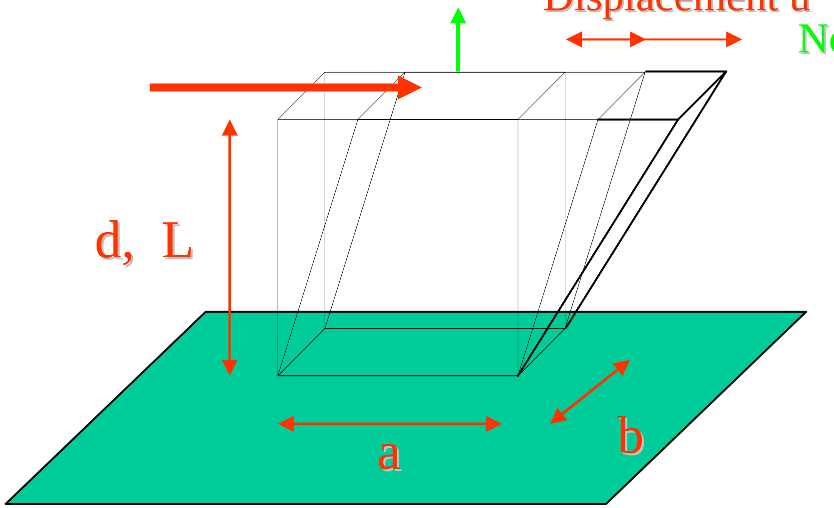
# Outline

- 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: pVT, Relaxation, Thermal Degradation etc.

# Capillary Rheometry: Main Applications



# Repeat from the previous session: Basic Terms




area A = a · b
Height = d
Initial length = L

$$\gamma = \frac{u}{d}$$ Strain []

$$\varepsilon = ln \frac{l}{L}$$ Extension []

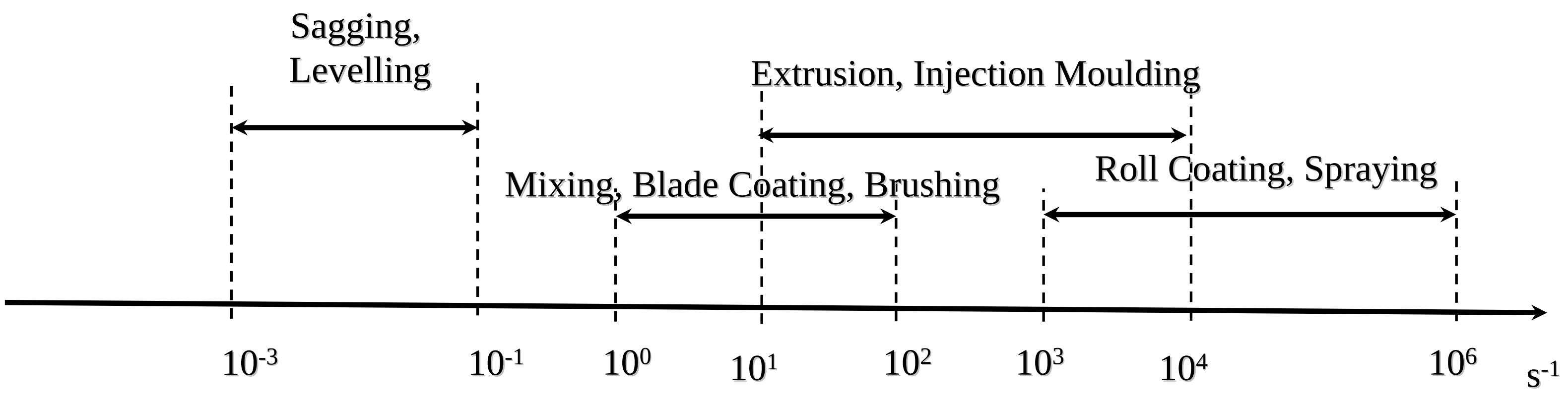
$$\dot{\gamma} = \frac{d\gamma}{dt}$$ Shear Rate [1/s]

$$\dot{\varepsilon} = \frac{1}{L}\frac{dl}{dt}$$ Extensional Rate[1/s]

$$\tau = \frac{F_{tan}}{A}$$ Shear Stress [Pa]

$$\sigma = \frac{F_{nor}}{A}$$ Extensional Stress [Pa]

# Typical Shear Rate Ranges




**Rotational-Rheometer**

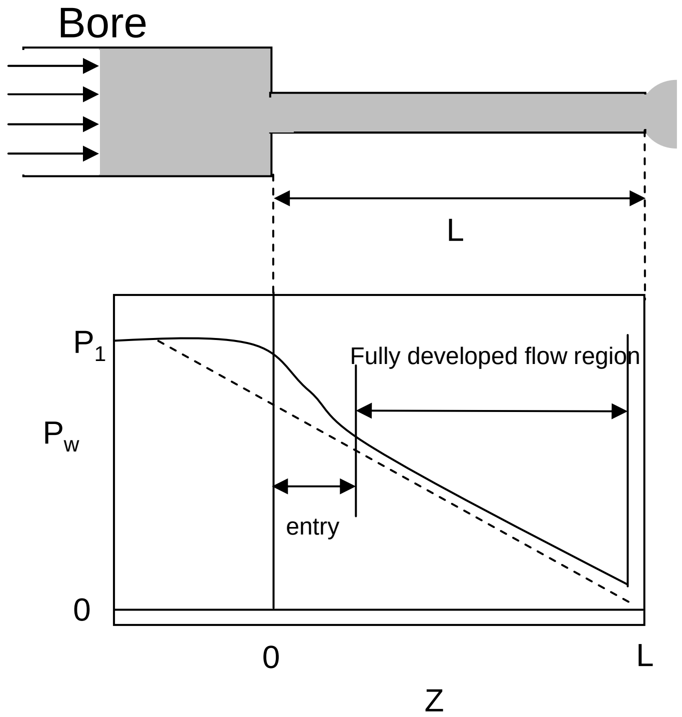
**Sample: Water up to solids**
**Results: Shear-Viscosity, Yield Stesses, Visco-Elasticity, Relaxation...**

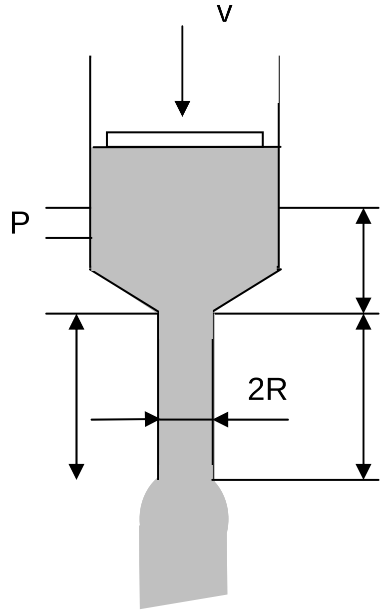
**High Pressure Capillary-Rheometer**

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

# Principle of Operation

Given quantity: piston speed ⇒ wall shear rate
Measured quantity: pressure drop ⇒ wall shear stress




Full pressure drop

=

Entrance pressure drop

+

Shear pressure drop

⇒ small ram extruder

# Laminar Pipe Flow

Isothermal, stationary Flow of an incompressible fluid

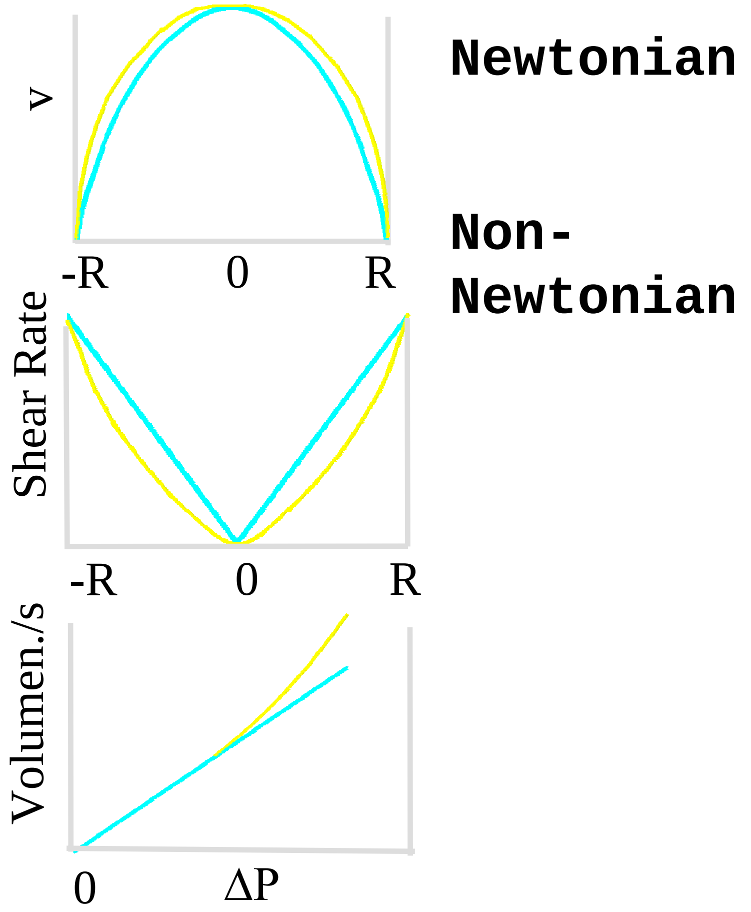
## Newtonian

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

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

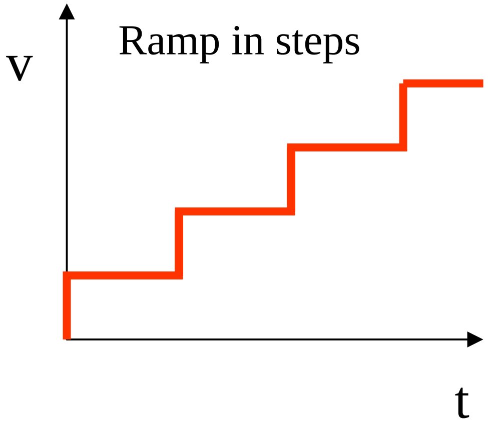
$$n = \frac{d(\log \tau)}{d(\log \gamma)}$$

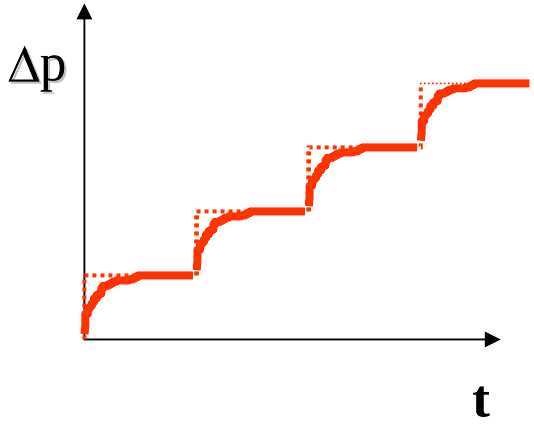
Non-Newtonian Index (Ostwald-de Waele)



# What are we doing to get flow curves?

measurement :



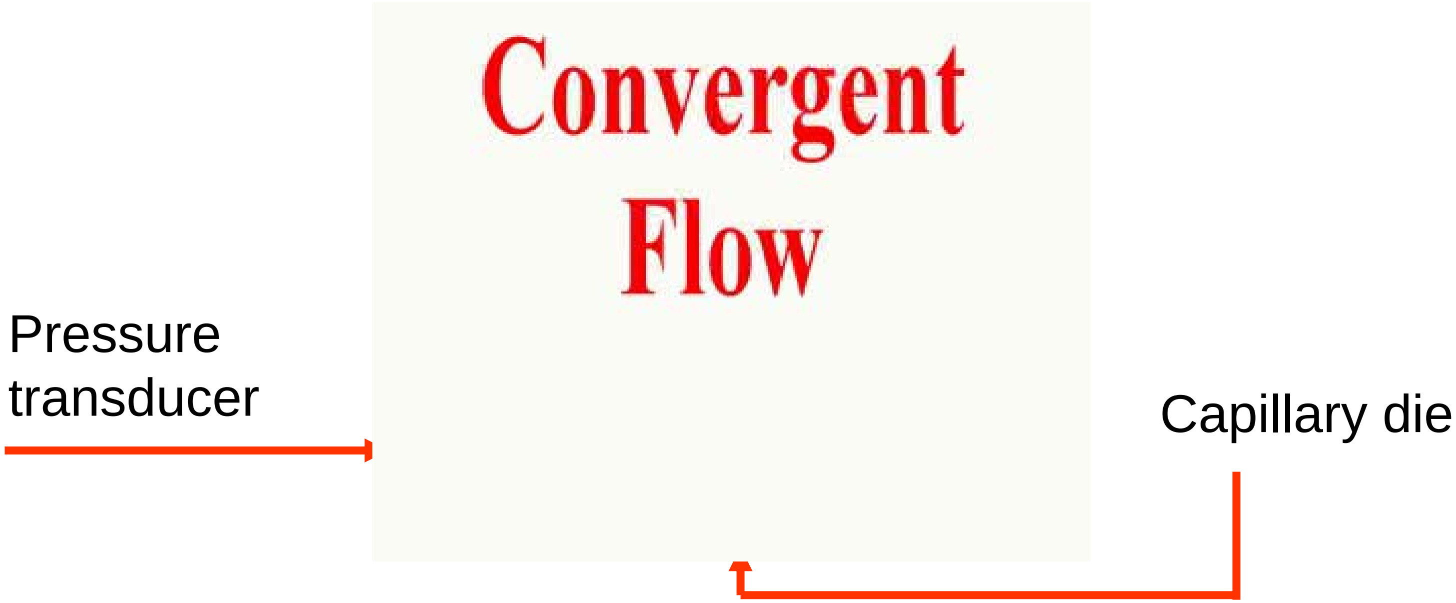

$$\dot{\gamma}_{app} = \frac{4 \cdot Q}{\pi R^3}$$

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

corrections →

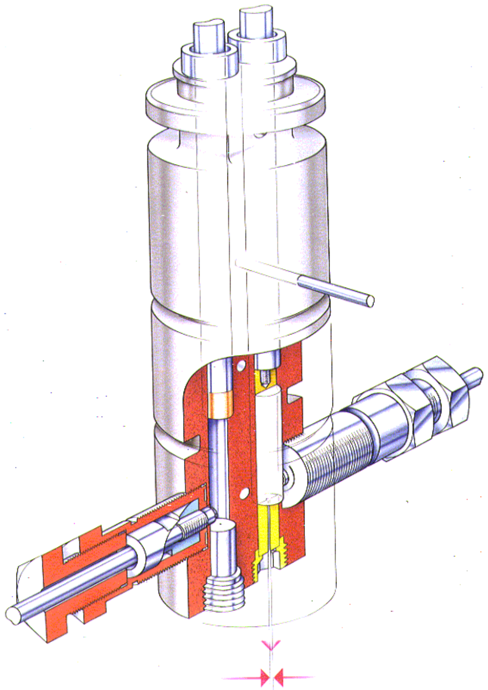
$$\eta = \frac{\tau_{true}}{\dot{\gamma}_{true}}$$

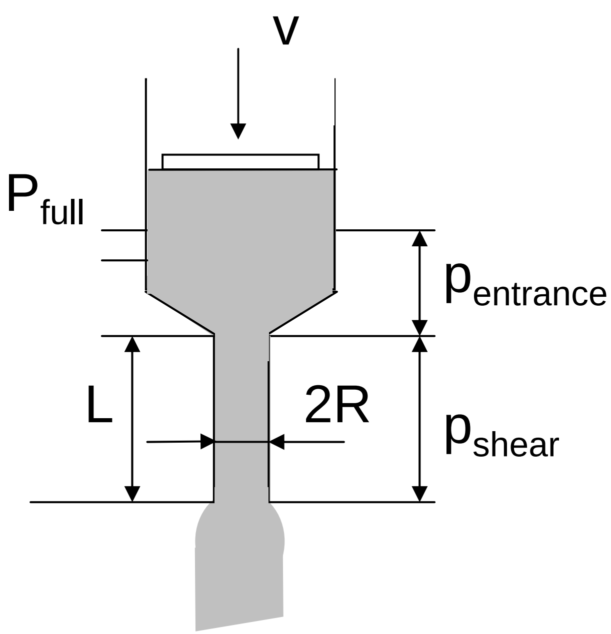
# Correction: Entrance zone of a capillary die

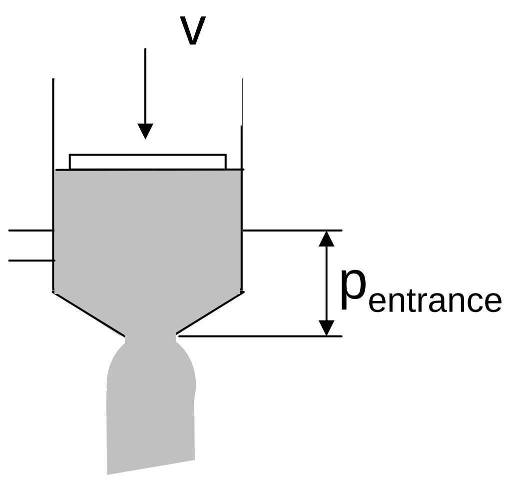



Aim of the test: to separate entrance pressure and shear pressure drop!

# Rosand Twin Bore Principle






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

left: capillary

right: orifice

# How do we get the Extensional Viscosity?

Cogswell`s Convergent Flow Model $\Rightarrow$ Extensional Viscosity

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

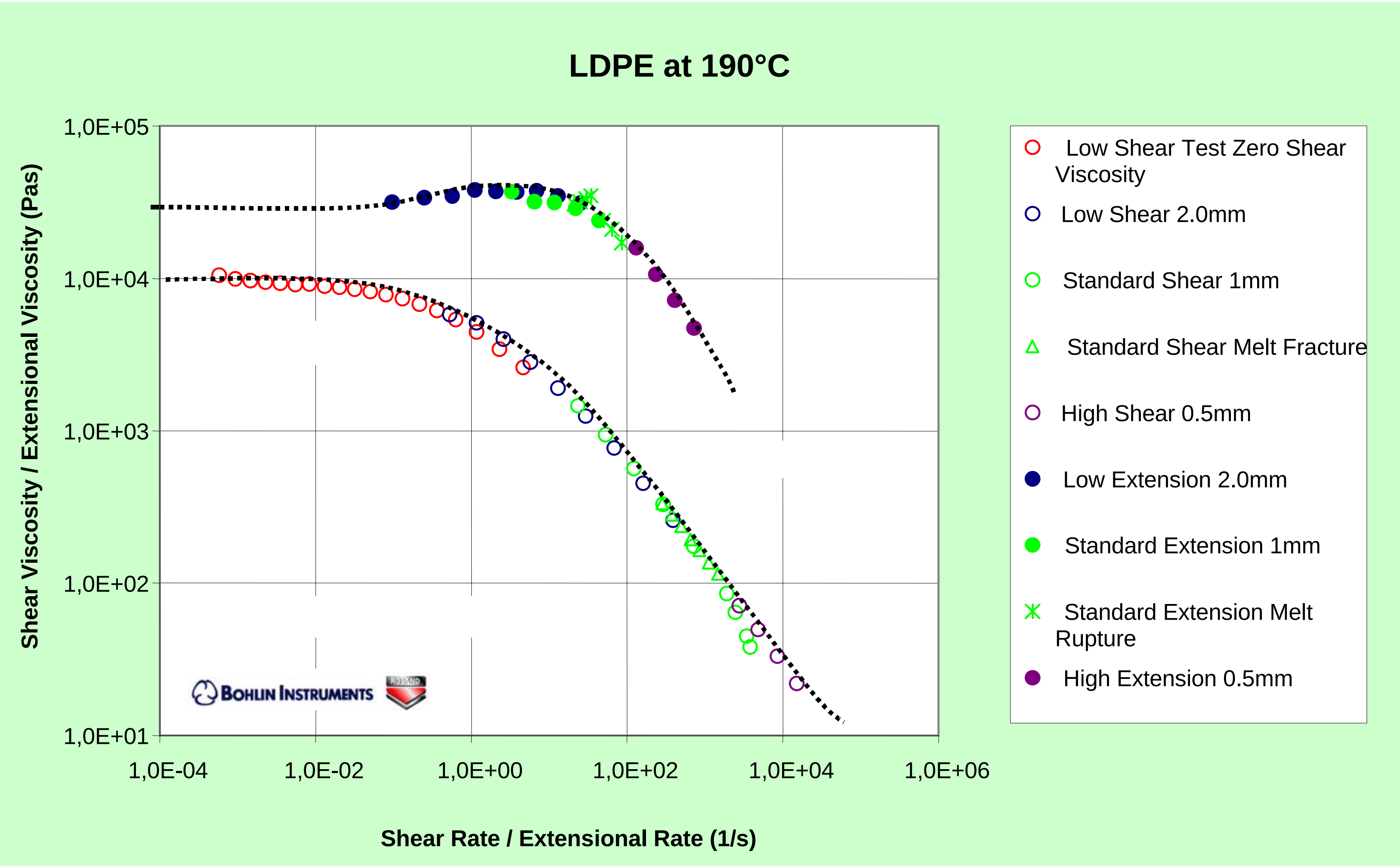
- **Special Orifice Die according to Uni Zlin Model enables characterisation of very small extensional rates too.**

$$n = \frac{d\,(\log \tau)}{d\,(\log \dot{\gamma})}$$ Non-Newtonian Index (Ostwald-de Waele)

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

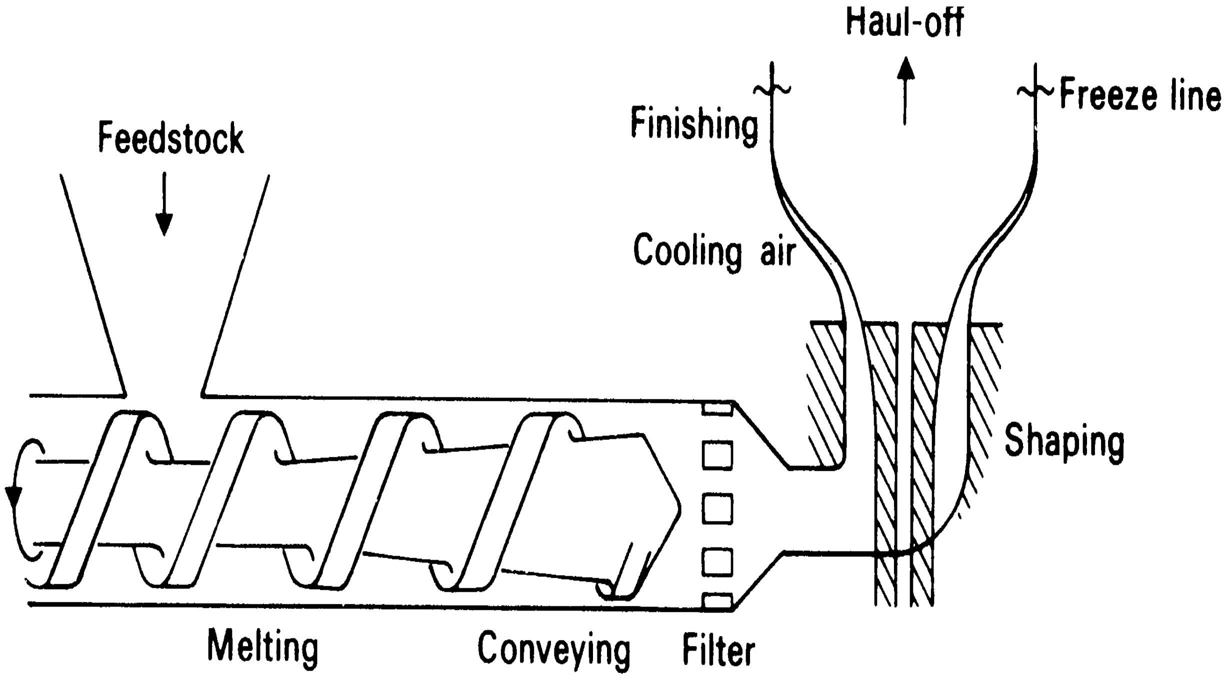
F. Cogswell, “Polymer Melt Rheology”, Woodhead Publishing Limited (1981)
Zatloukal, Vlcek, Tzoganakis, Saha *J. Non-Newtonian Fluid Mech*. **107** (2002) 13–37

# Example LDPE



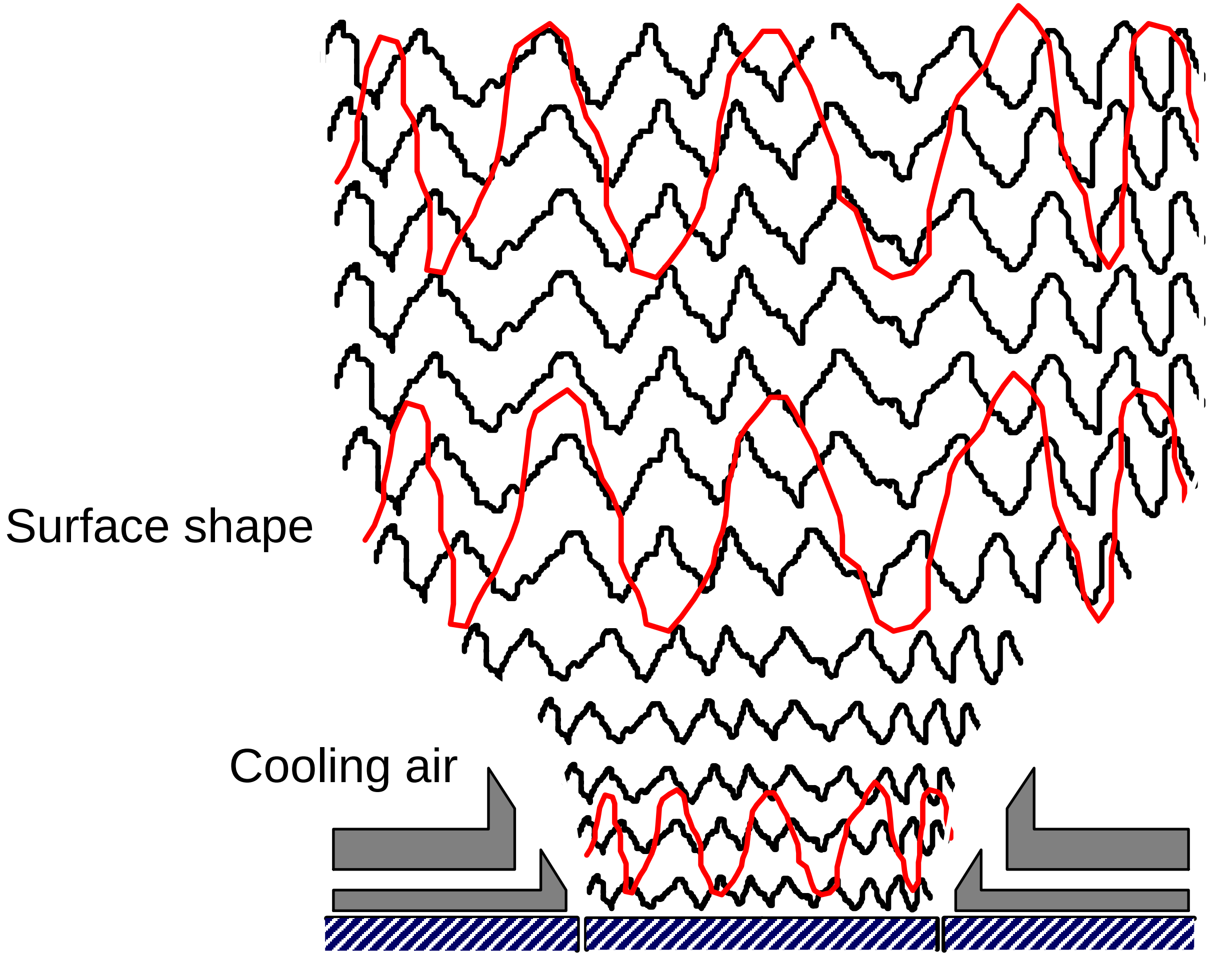
# Extensional Rheology of LDPE

Blow Moulding

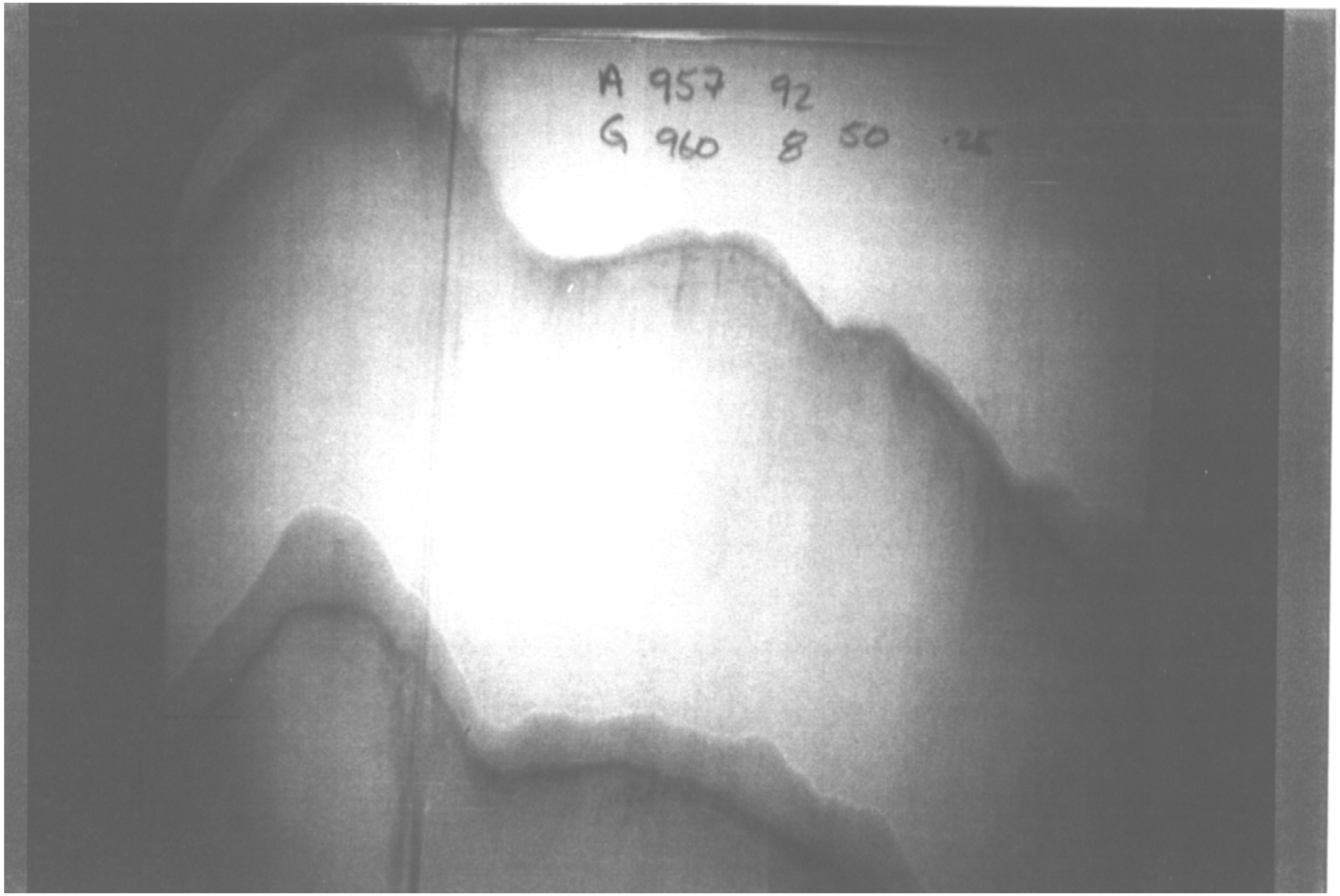



$\Rightarrow$ Blow Moulding is mainly influenced by Extension!

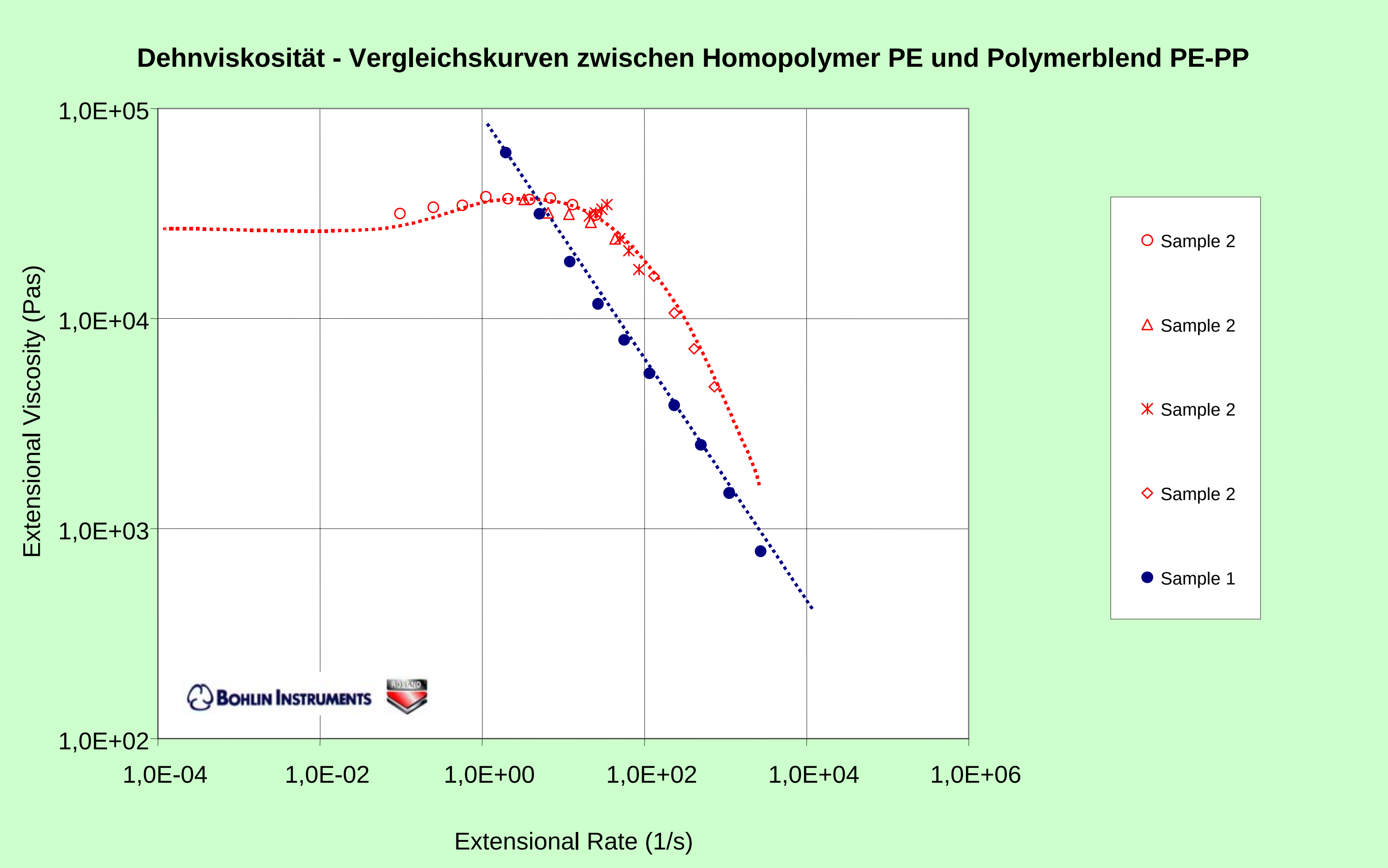
# Surface Instabilities LDPE



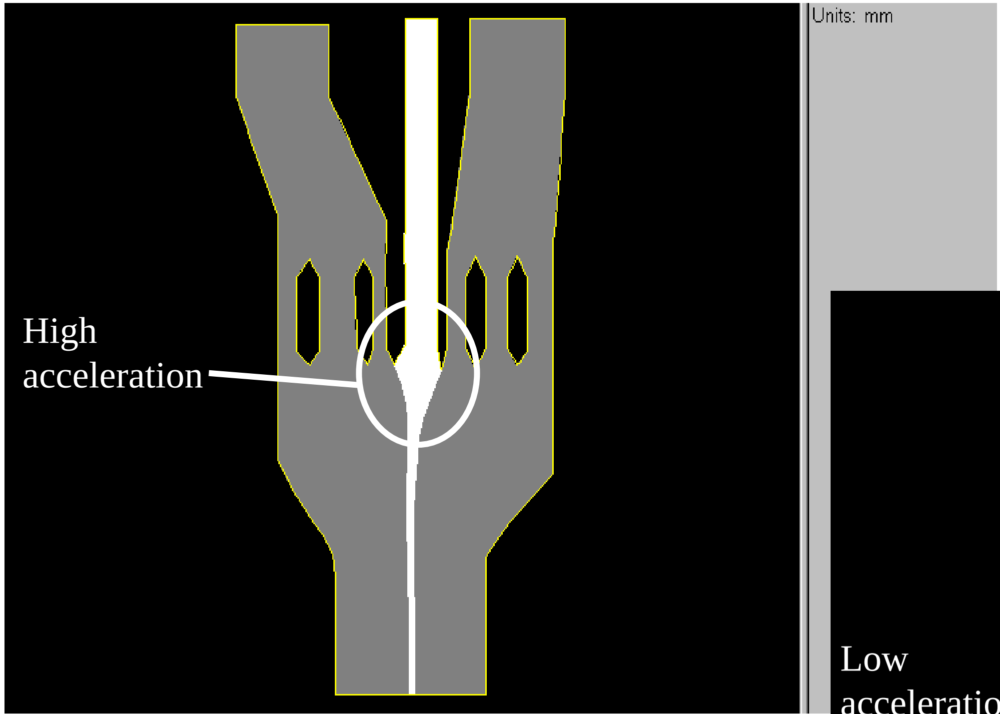
# Surface Instabilities LDPE



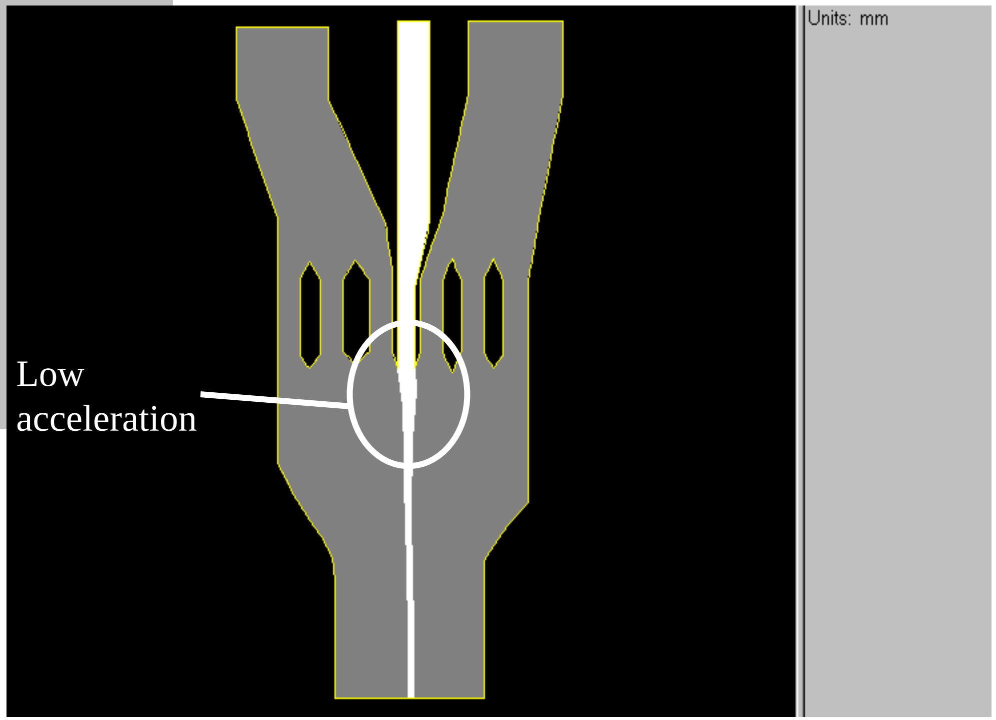
# How can the process be improved?



# Another Example: Co-Extrusion




Similar instabilites



# LDPE in Co-Extrusion Die

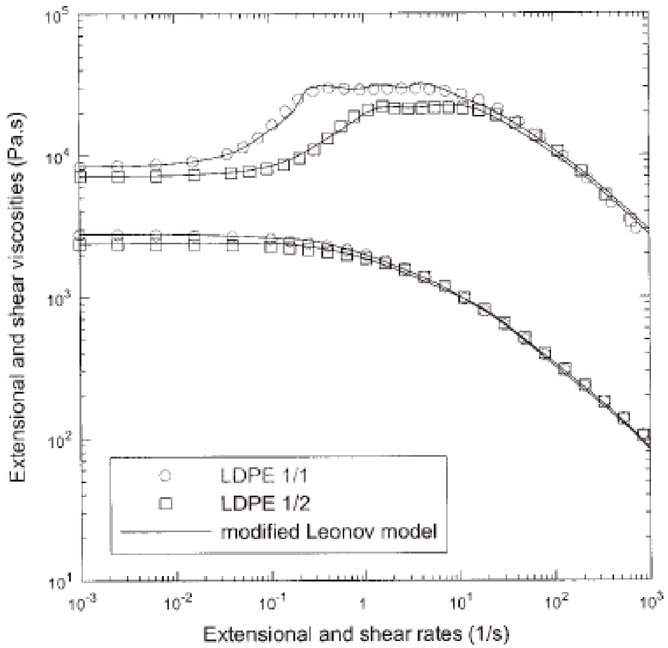



**Figure 4** Extensional and shear viscosities for two different lots of LDPE 1, 210°C.

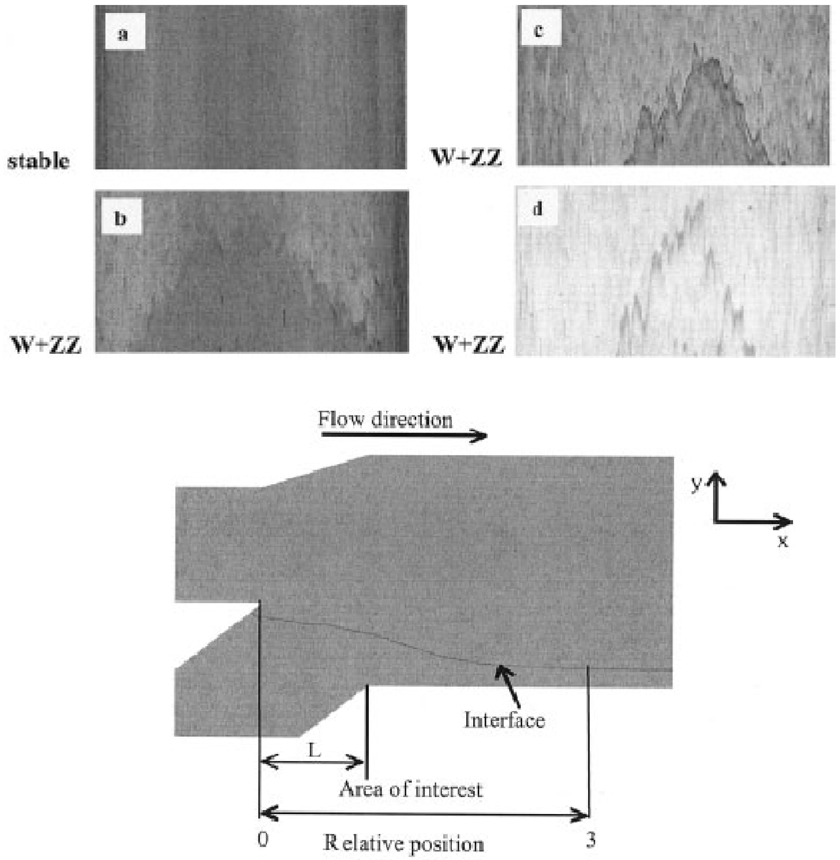
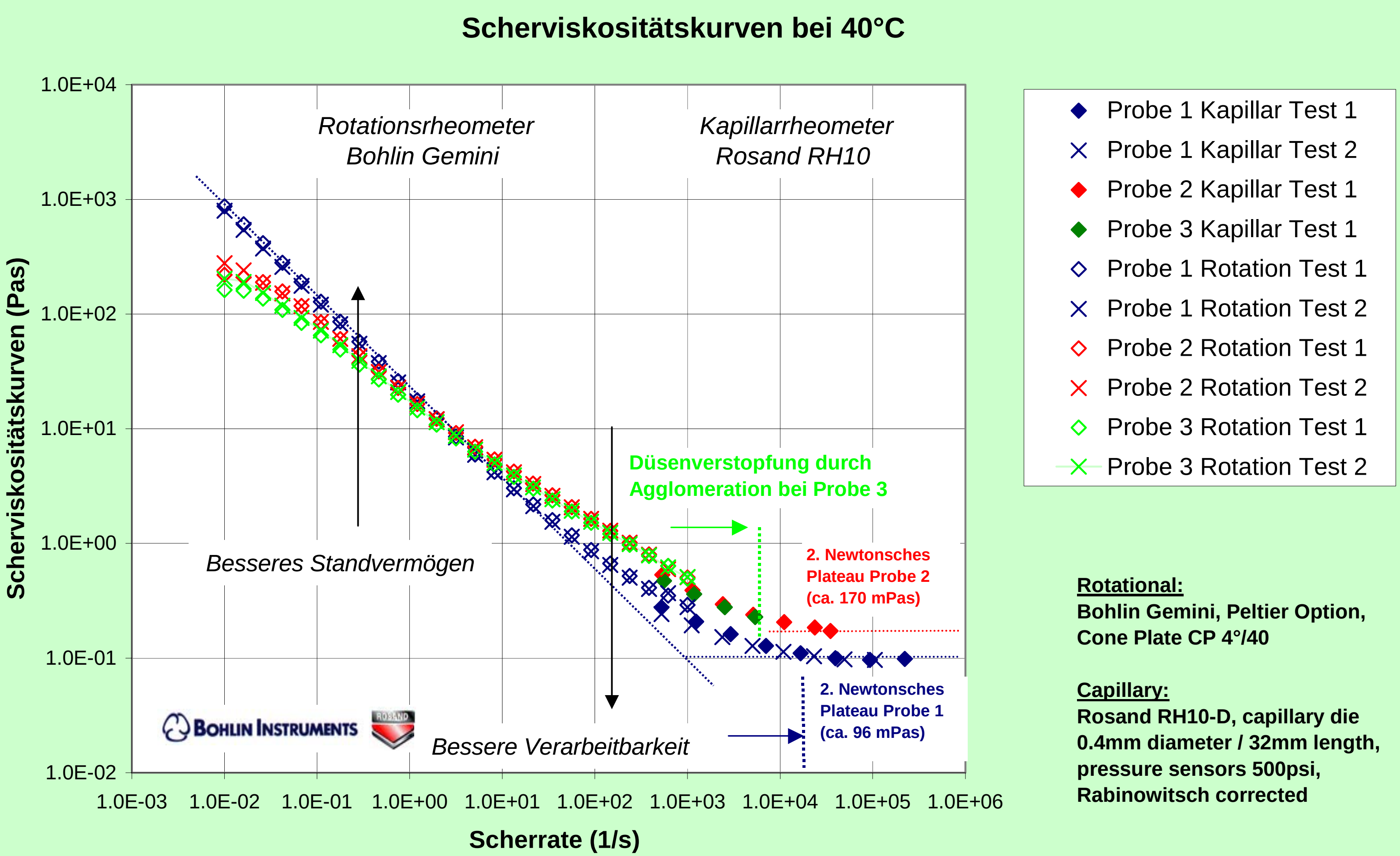



**Figure 5** Merging area of the flat coextrusion die.

Instabilities caused by Extensional Flow Behaviour of LDPE

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

# Further Examples: Dispersions

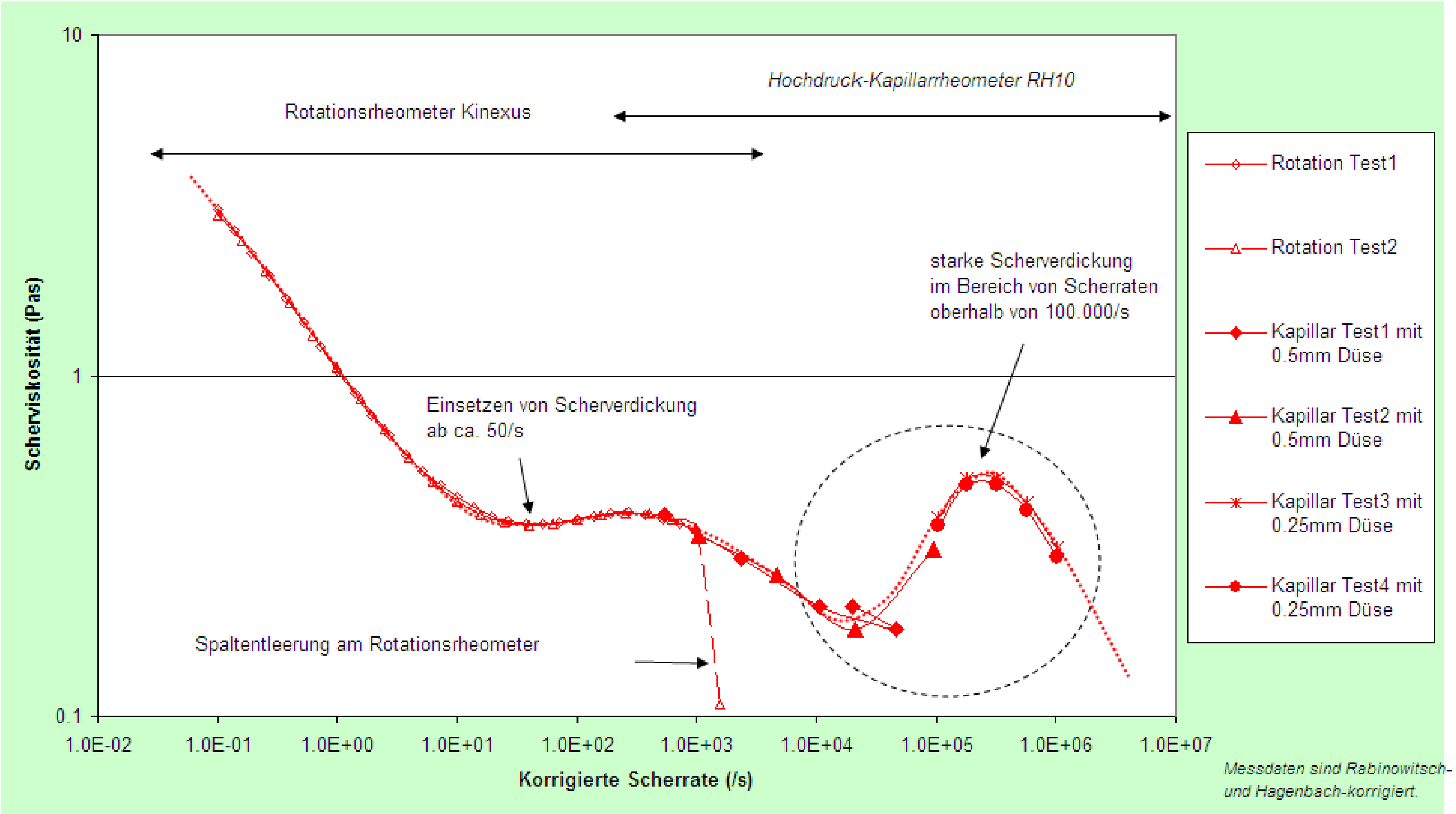



**Rotational:**
**Bohlin Gemini, Peltier Option, Cone Plate CP 4°/40**

**Capillary:**
**Rosand RH10-D, capillary die 0.4mm diameter / 32mm length, pressure sensors 500psi, Rabinowitsch corrected**

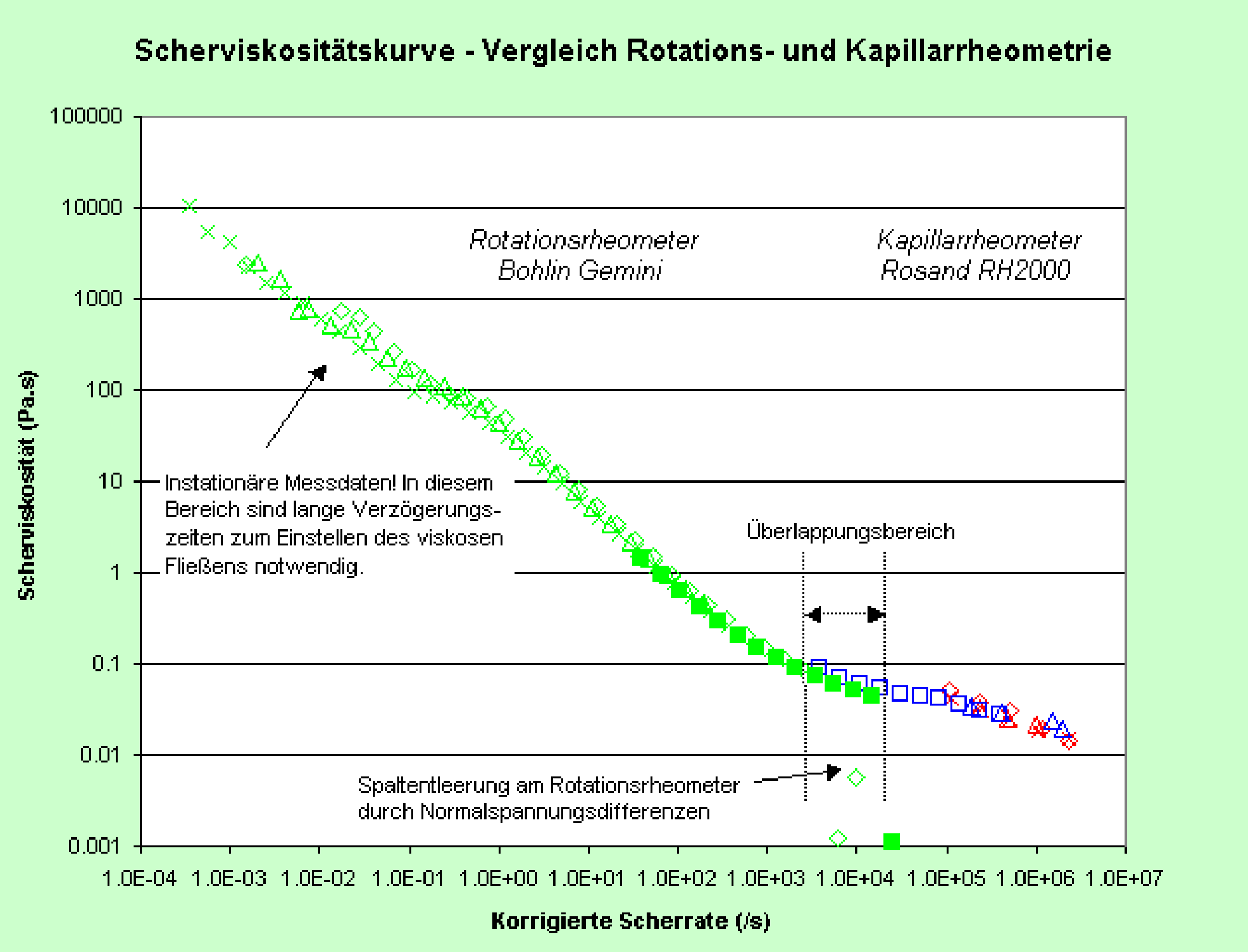
⇒ Capillary Rheometry can predict Die Blocking

# Example: Dispersion Adhesive for Spray Coating




⇒ Shear Thickening effect depends on the particle volume fraction

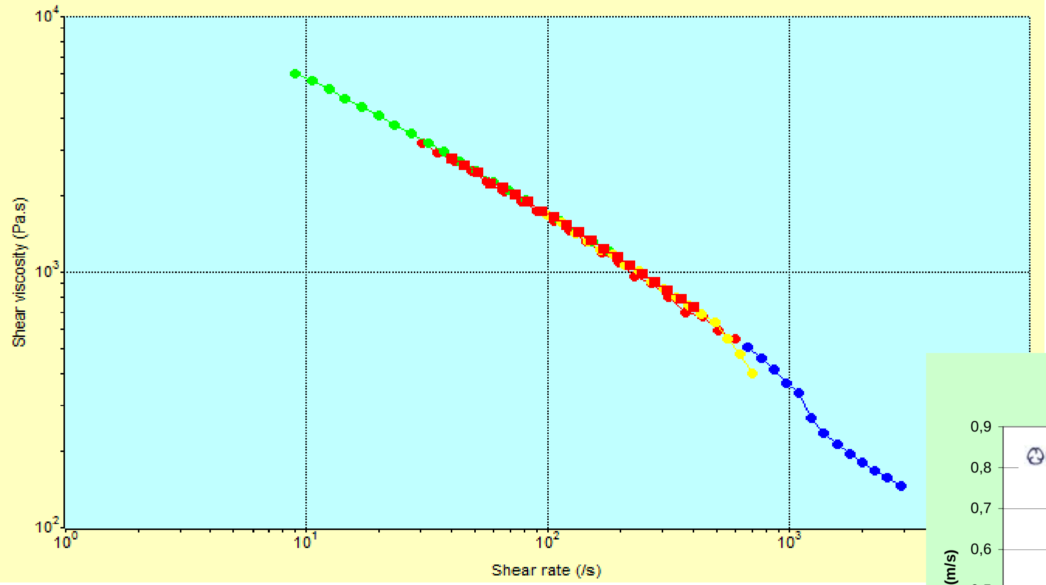
# Wide Shear Rate Range

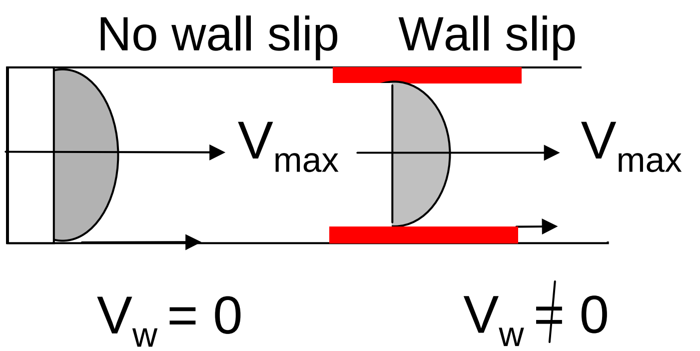


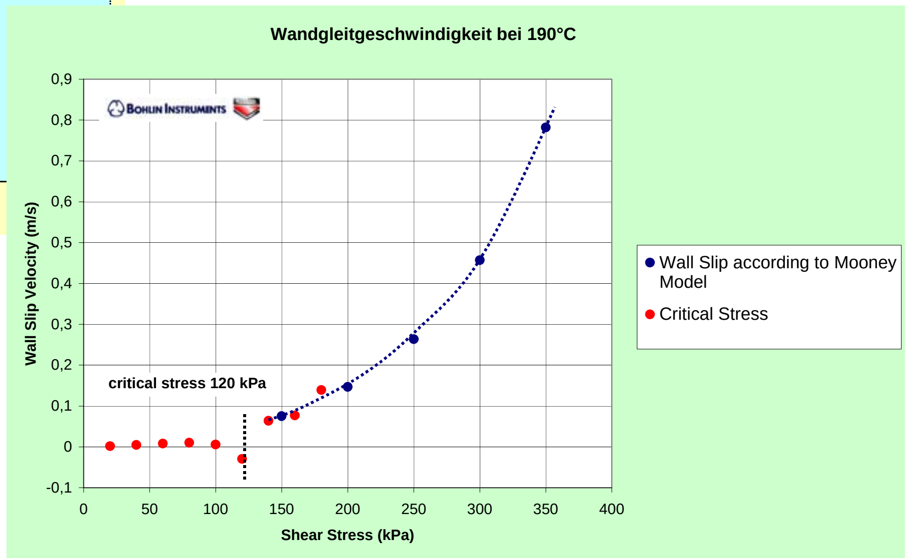

⇒ Rotational and Capillary Rheometry cover approx 13 decades in shear

# Further Applications: Wall Slip

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



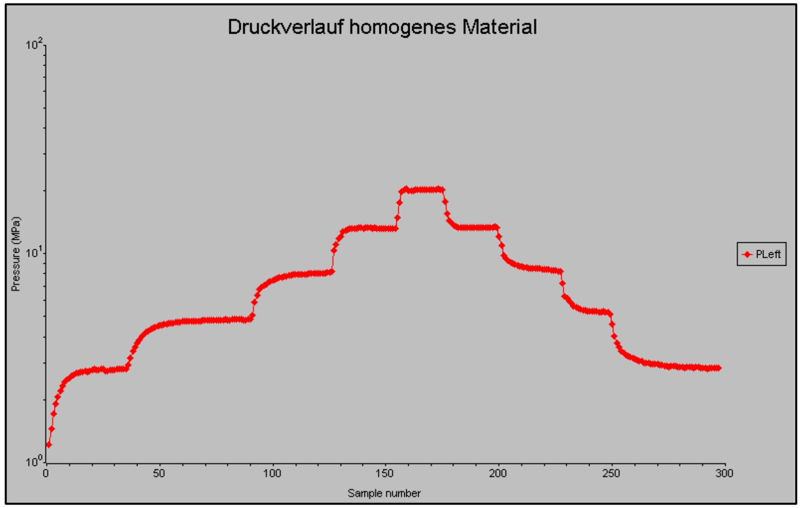



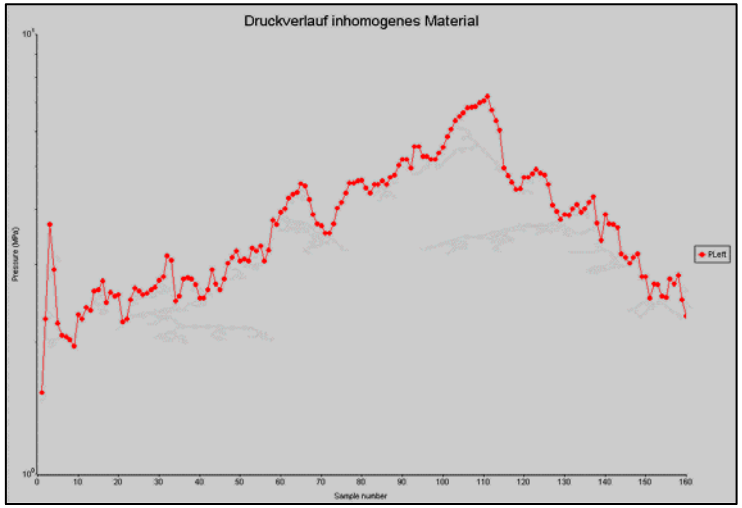

- Wall slip velocity increases dramatically at just above 0.1 MPa.

# Equilibrium Pressure: Homogeneity

Pressure drop is important




homogeneous

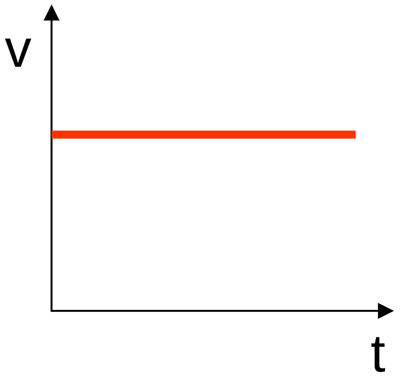


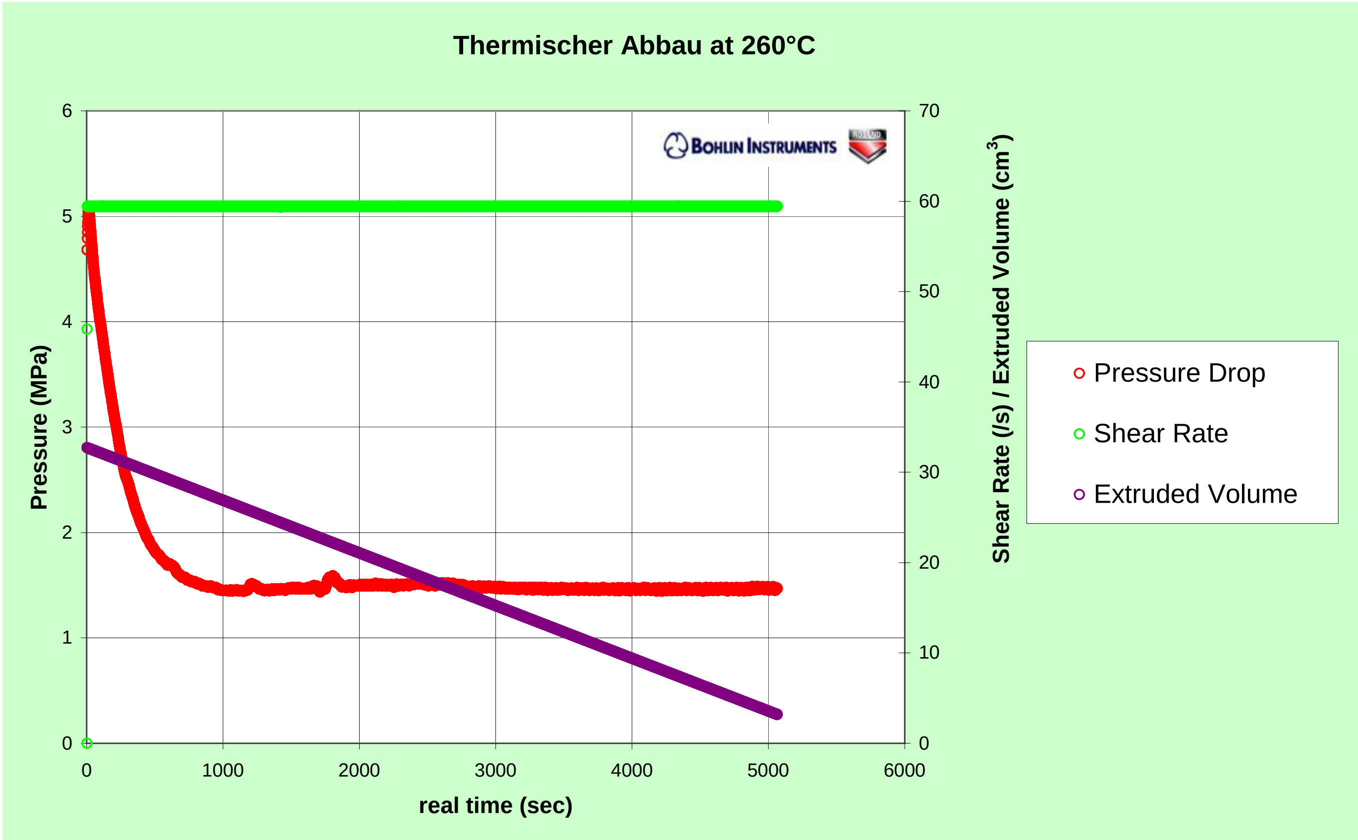

inhomogeneous

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

# Thermal degradation / Curing

Prinzip:



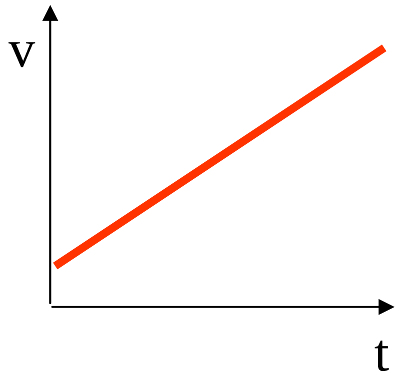



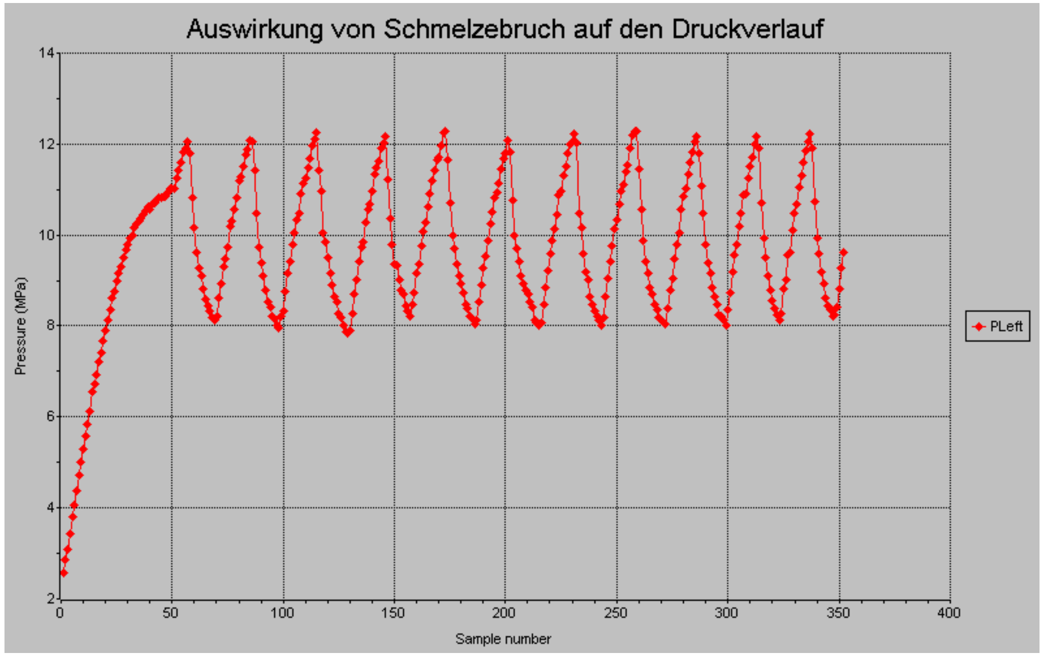

⇒ Gives max process time

# Stick-Slip

Flow Instabilities

Linear Ramp



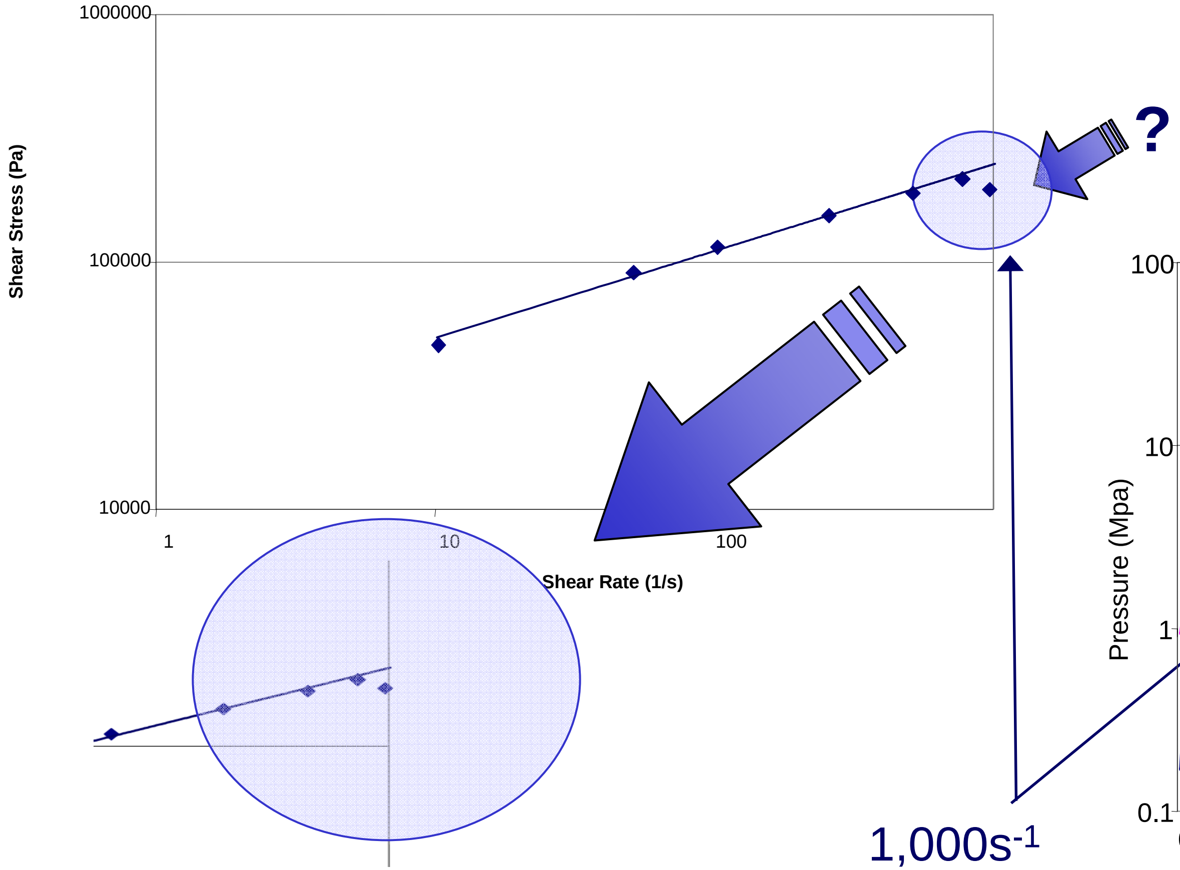



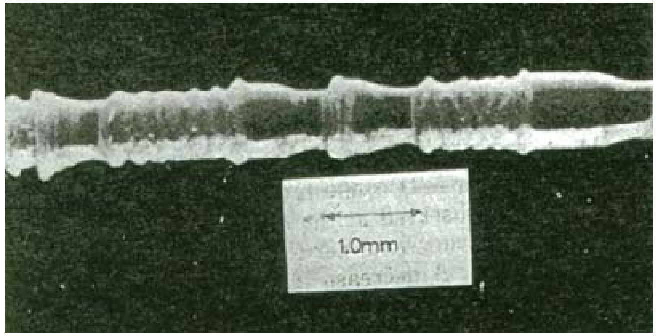

Melt fracture

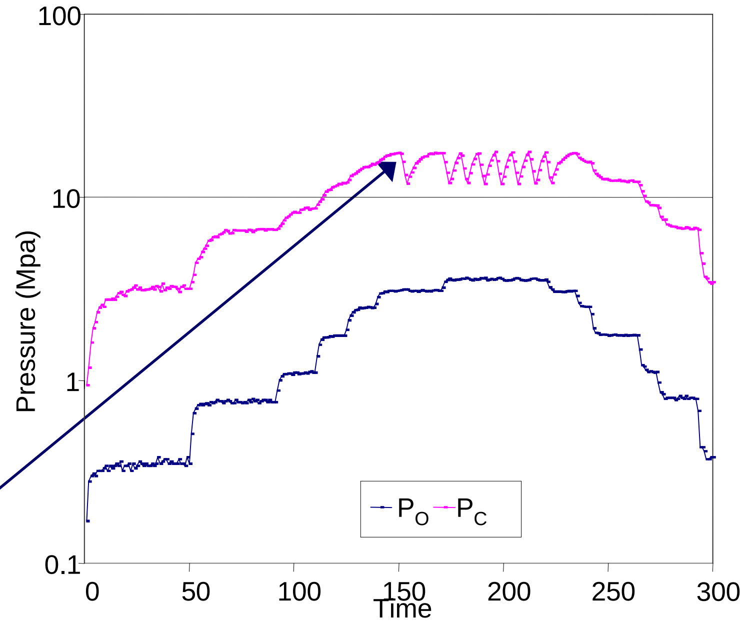
⇒ What is the max processing pressure / Shear Rate?

# Melt Fracture

- Unstable flow, poor product quality.



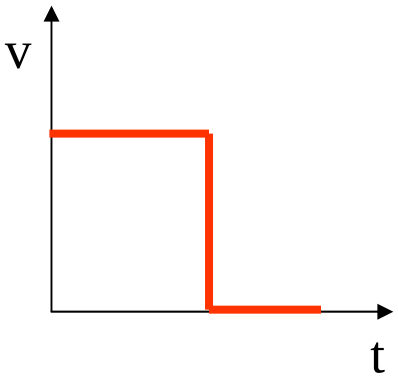



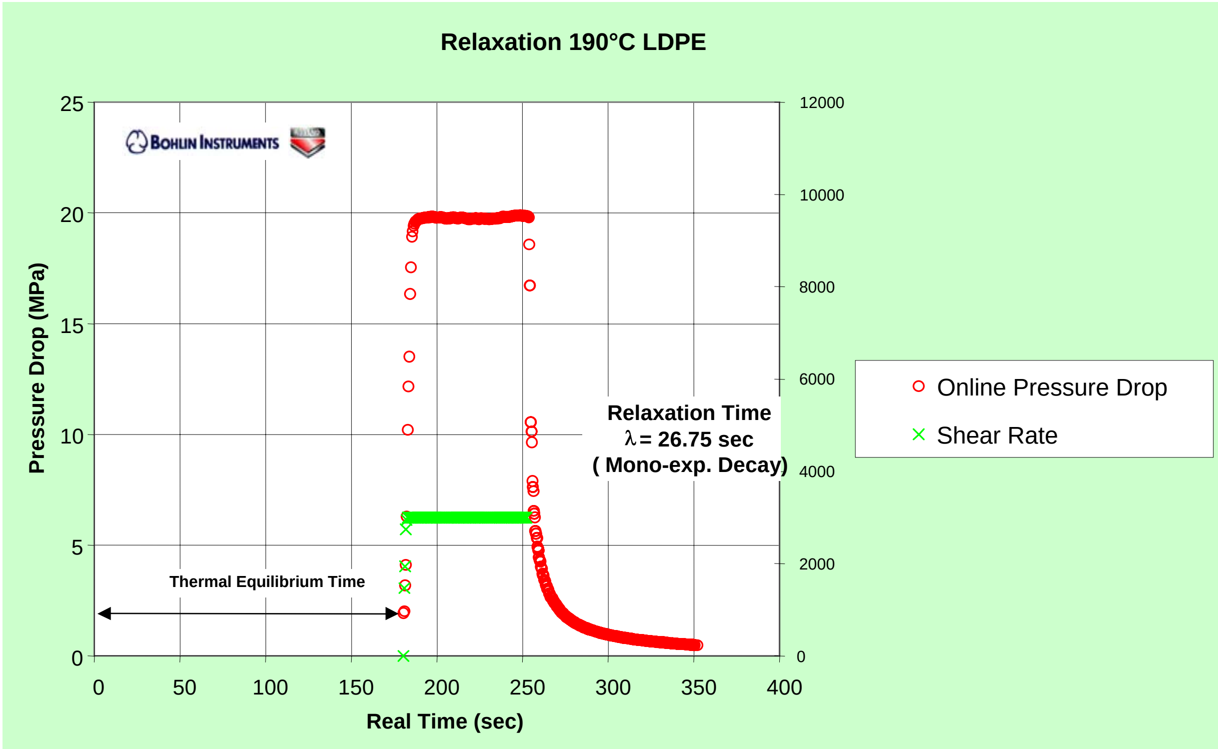



# Relaxation LDPE

What happens after processing

Prinzip:

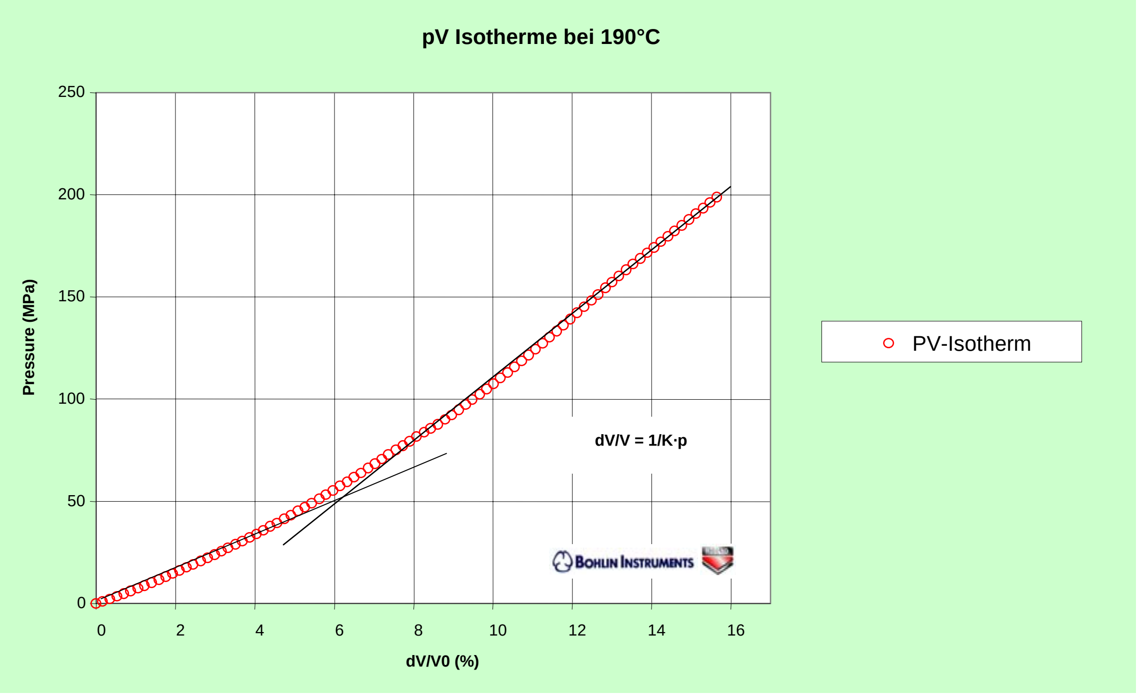



⇒ inner stresses can lead to surface crack (automotive industry)

# Compressibility

PV-Isotherm




PVT:
- Mainly needed for flow simulation

# Rheometer Types

Benchtop RH2000 and Floor Standing RH7/10


# Example: Test Run at RH7

# Conclusion

The complete flow behaviour under processing conditions

**Rosand Double Capillary System with Orifice Die**:

- direct measurement of the entrance pressure drop - no extrapolation needed
- calculation of extensional viscosity according Cogswell method
- flow curve up to very high shear end extensional rates
- ability to detect wall slip by Mooney's method
- correlation with structural changes during processing
- additional Options for detection of elastic behaviour (Die-Swell)

**Thank you for your attention.**