MIM / PIM Application:
Laboratory Mixer, Compounding, Rheology

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The world leader in serving science

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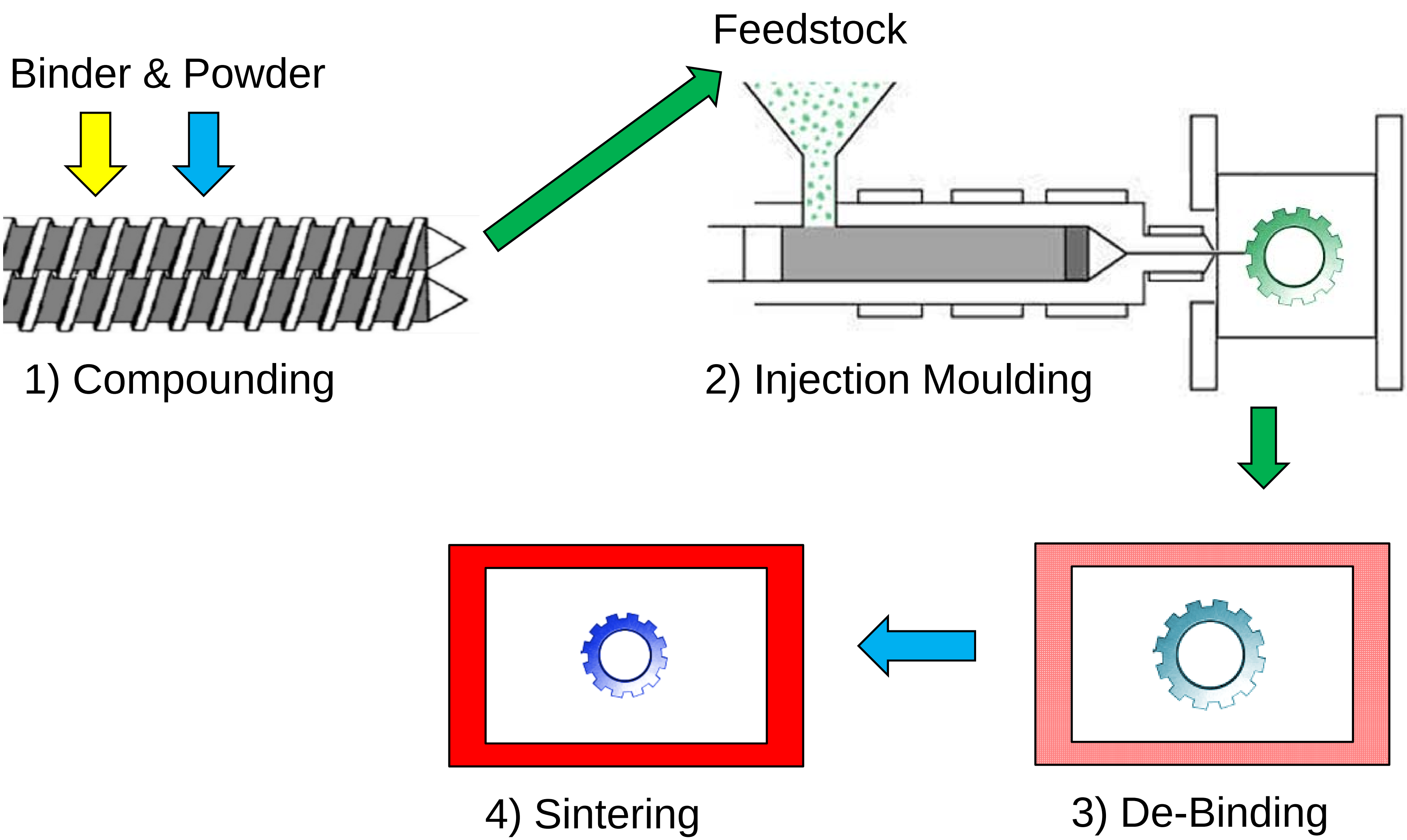
- **Capillary Rheology**

  - Rheological Characterization for optimized Flow Properties
- **Questions**

INTRODUCTION

POWDER INJECTION MOLDING PROCESS

Principle PIM-Process:



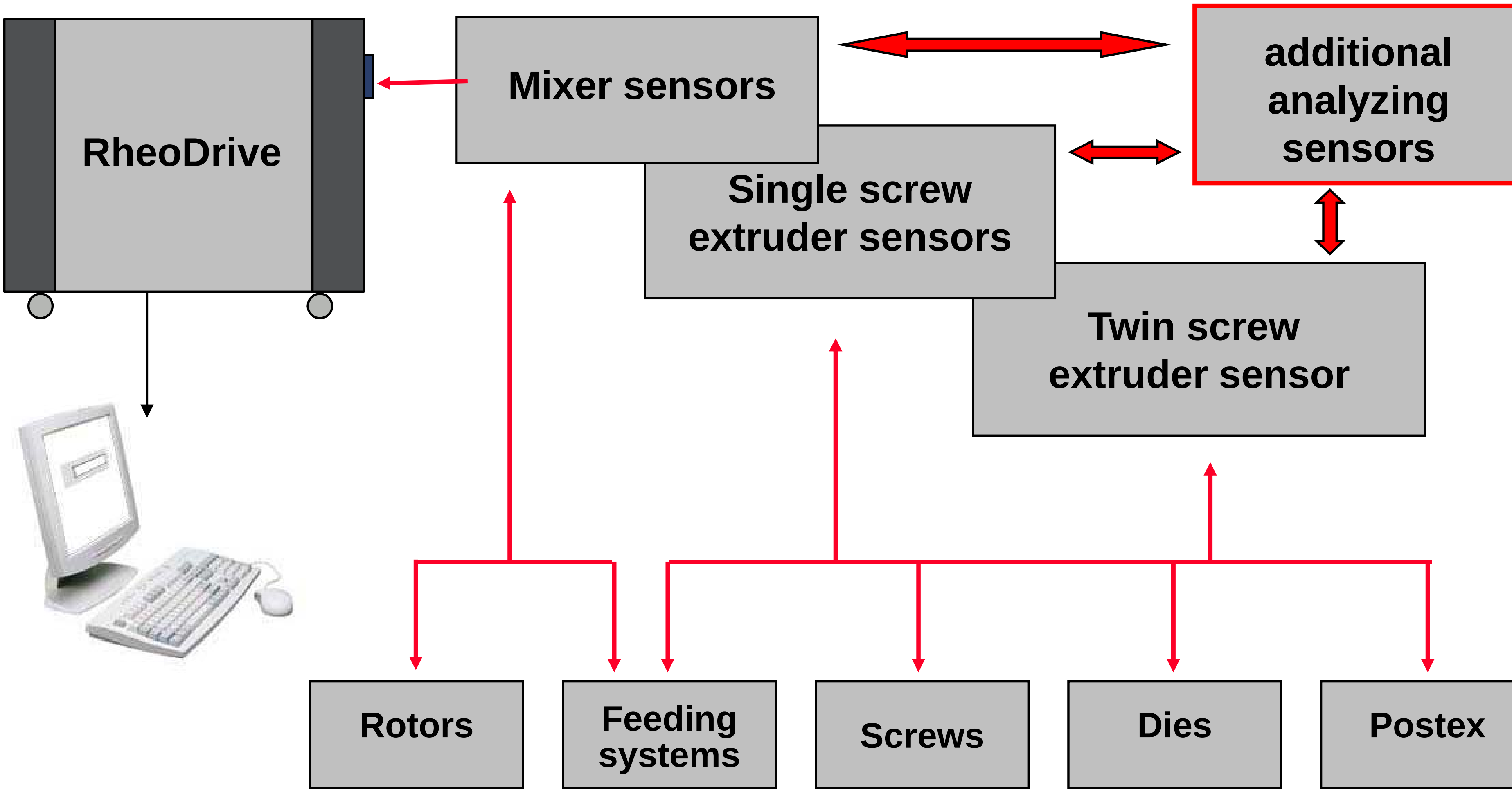
Powder-Injection-Moulding – Strength & Limitations

- **The typical PIM product combines a relatively small size, high complexity of shape, high strength and moderate to close dimensional tolerances.**
- **PIM is highly competitive where costly machining operations can be avoided, and when the number of parts is high enough, to justify the cost of a mold.**
- **The PIM process allows a high degree of automation and is therefore most competitive in volume production.**
- **PIM parts can be designed with extremely thin walls, fine bore holes, threads and other details. This helps to save weight and material cost.**
- **PIM is limited by the relatively high raw material cost, compared to cast metals and alloys. Because of that, PIM parts are usually rather small, where the material cost of a small part have a smaller portion of the total manufacturing cost.**

LABORATORY MIXERS
FOR COMPOUND DEVELOPMENT

RheoDrive + Mixer/Extruder Sensor

= *PolyLab System*



History: Old Torque Rheometer Models

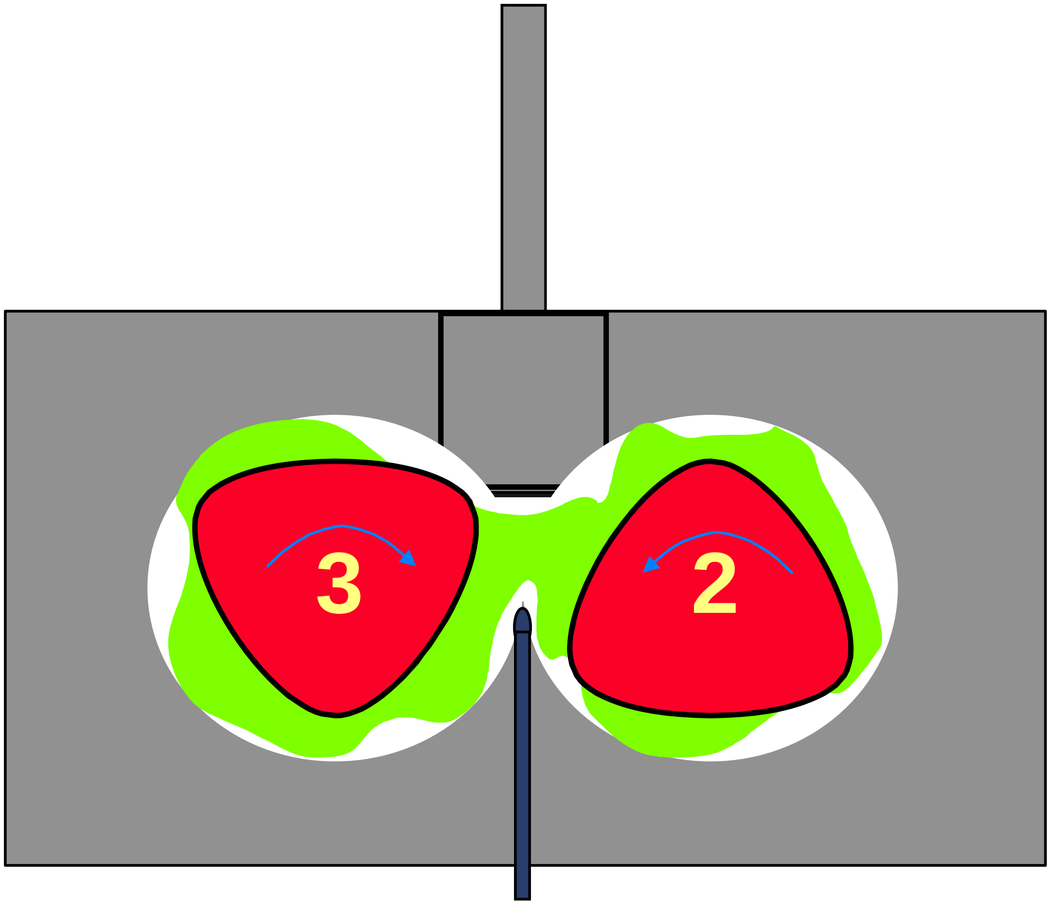
PolyLab OS – New Design

Proprietary & Confidential

PolyLab OS – New Design

PolyLab OS – Flexible Design

Laboratory Mixers - *measuring principle*



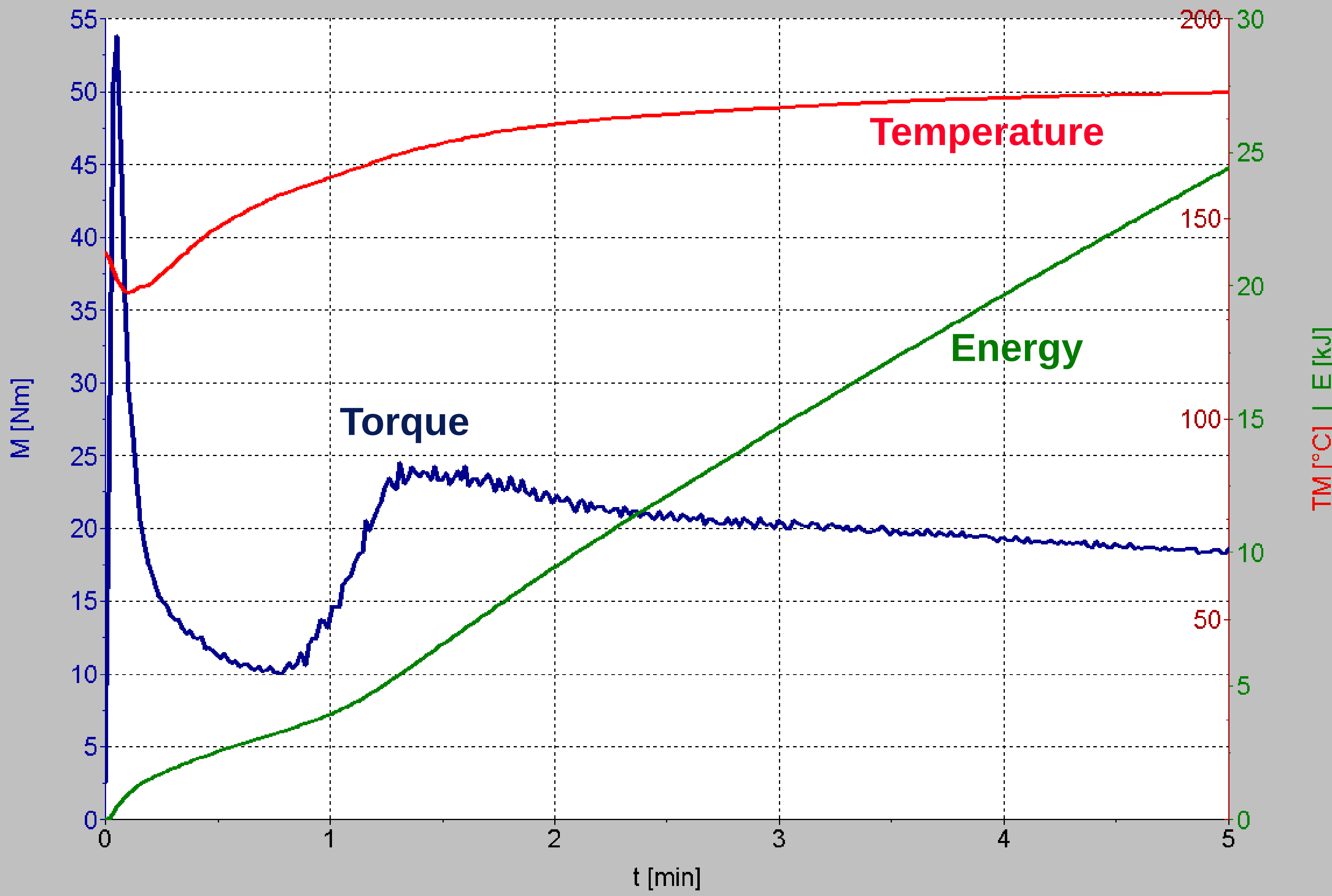
Shearing of a test sample in a heated mixing chamber with counter rotating rotors

<u>Test results:</u>

➔ Torque

➔ Melt temperature

Laboratory Mixers - *Rheogram*

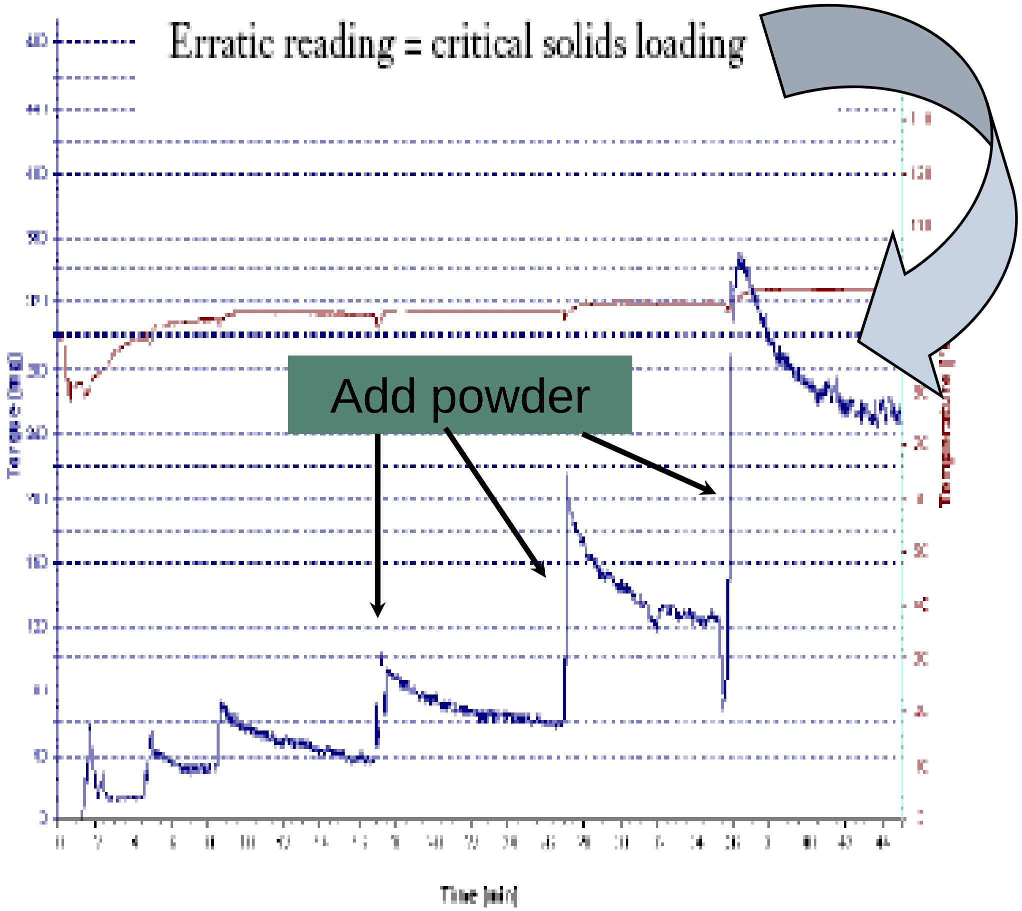


Laboratory Mixers - *Testing*

Rotors and Applications

Roller Rotors		Melting and mixing of thermoplastics, like Polyolefines, PVC, engineering plastics
Banbury Rotors		Mixing of elastomers and thermoplastic polymers with higher content of powder.
Cam Rotors		Thermoplastics, less axial distribution, ceramic compounds, food (sticky, higher torque)

PIM: Critical (powder) loading of feedstock



Ref. LR45-e, Joseph A. Krudys, ThermoHAAKE 2002

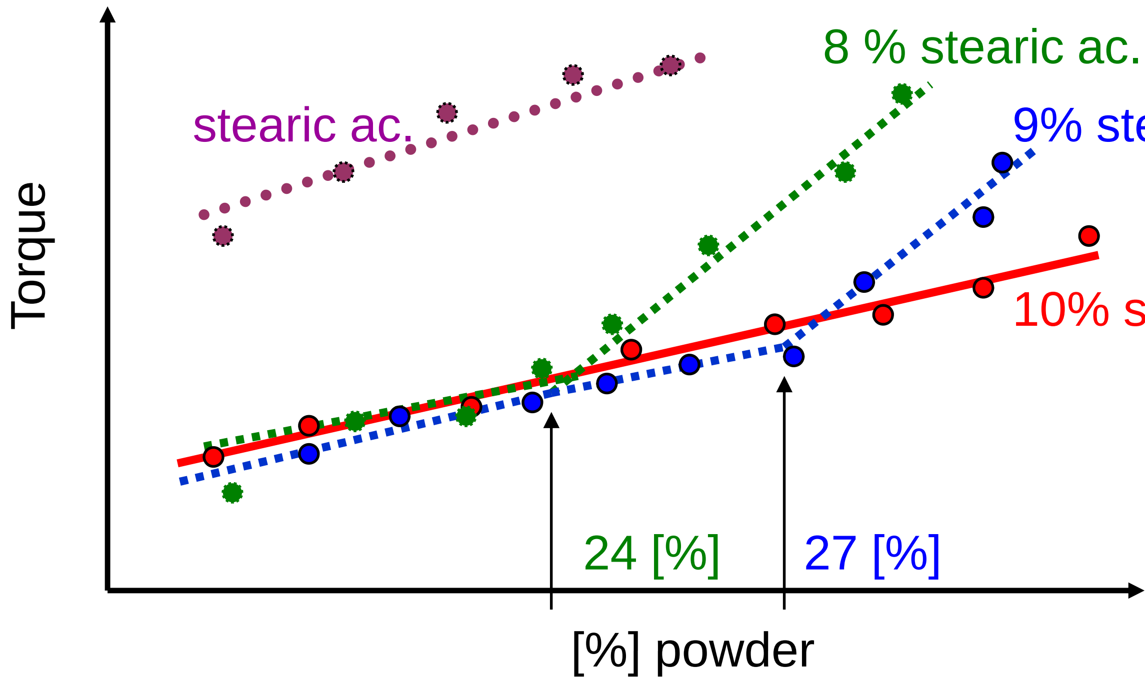
The erratic reading in the steps for increased powder load show the limiting powder [%] in the mixture.

Contact free and fast, high resolution torque sensors have a big advantage over old style “dynamometer” where the reaction of a heavy motor is measured, the erratic reading (here key feature) is damped or smoothed

Nanoscale Ceramic Al_3O_2 Powders for PIM

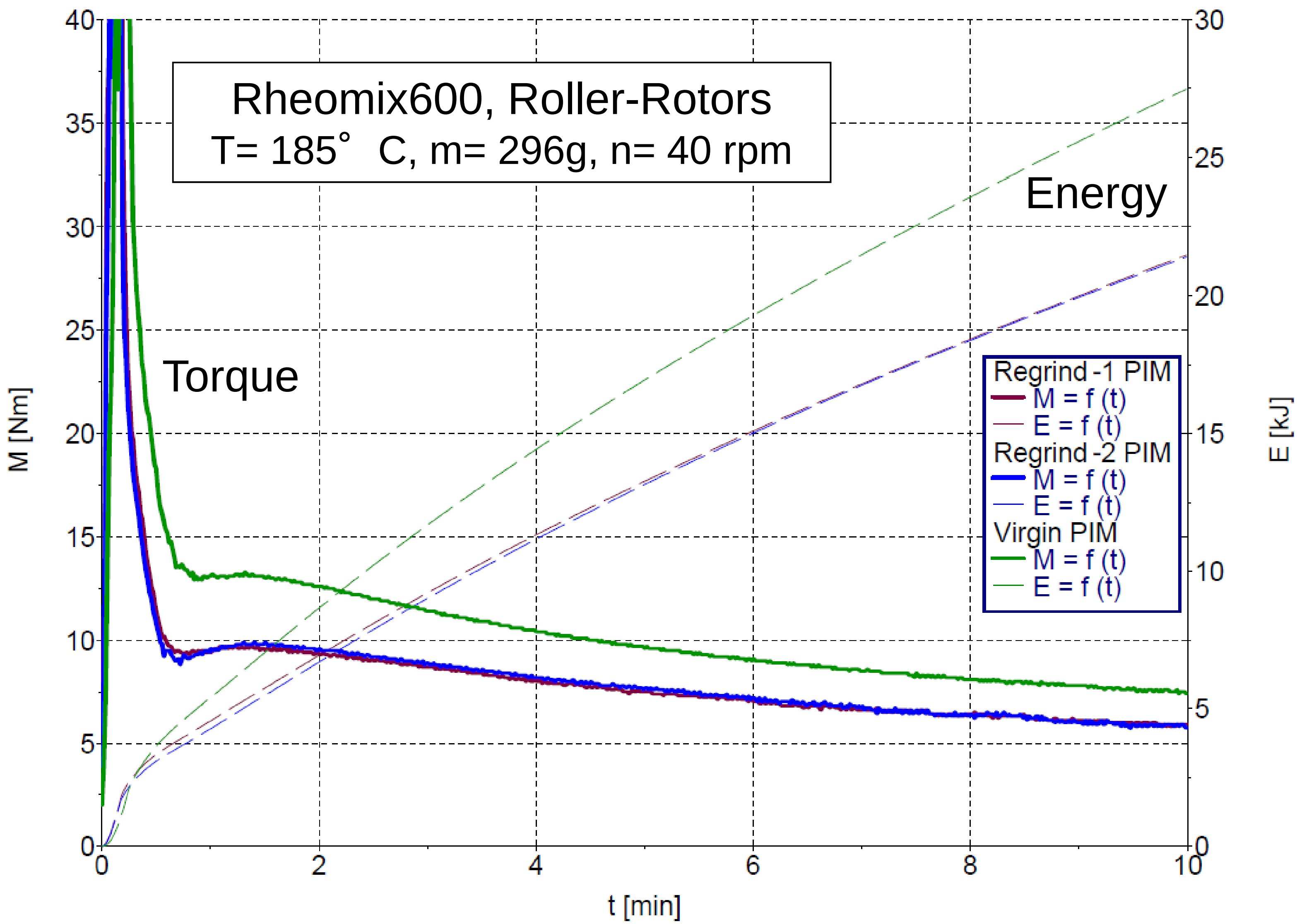
Juliane Kraus (Ref.) describes in detail how to optimize a binder system (stearic acid & stearic acid amide) using a laboratory mixer (Rheomix600 with Roller Rotors).

- **Type modifier (better: lower torque with stearic acid amide)**
- **Minimum content of this modifier (Torque depends only on [%]powder)**



Ref. (in german): Juliane Kraus
Spritzgießen nanoskaliger keramischer Pulver
am Beispiel des Degussa Aluminiumoxid C
Hochschulschrift: Saarbrücken, Univ., Diss., 1999

Differentiation of fresh and re-grinded PIM compounds



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TWIN-SCREW COMPOUNDING

FOR FEEDSTOCK PRODUCTION

Extrusion Principle:

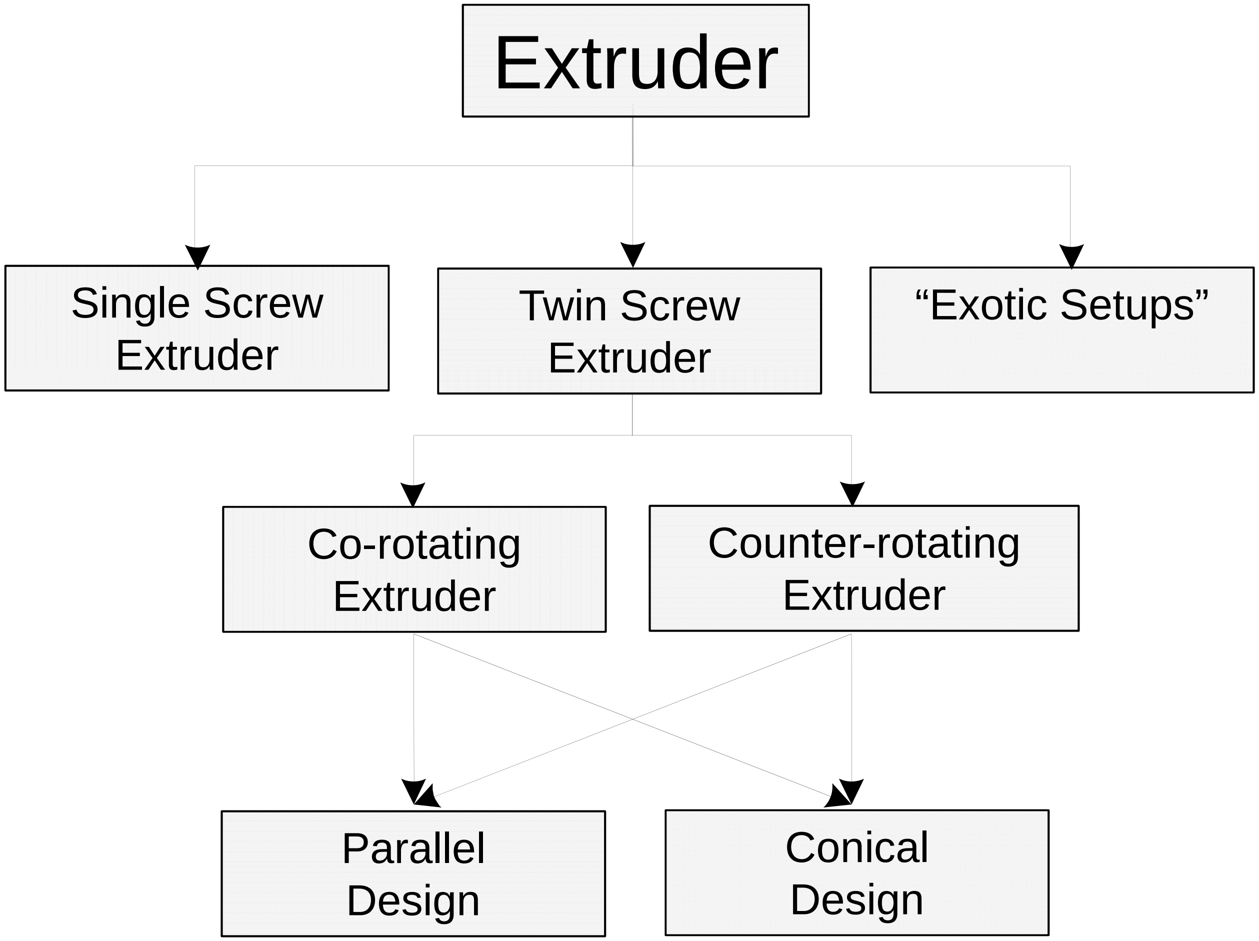
Extruder types

Single Screw Extruder:

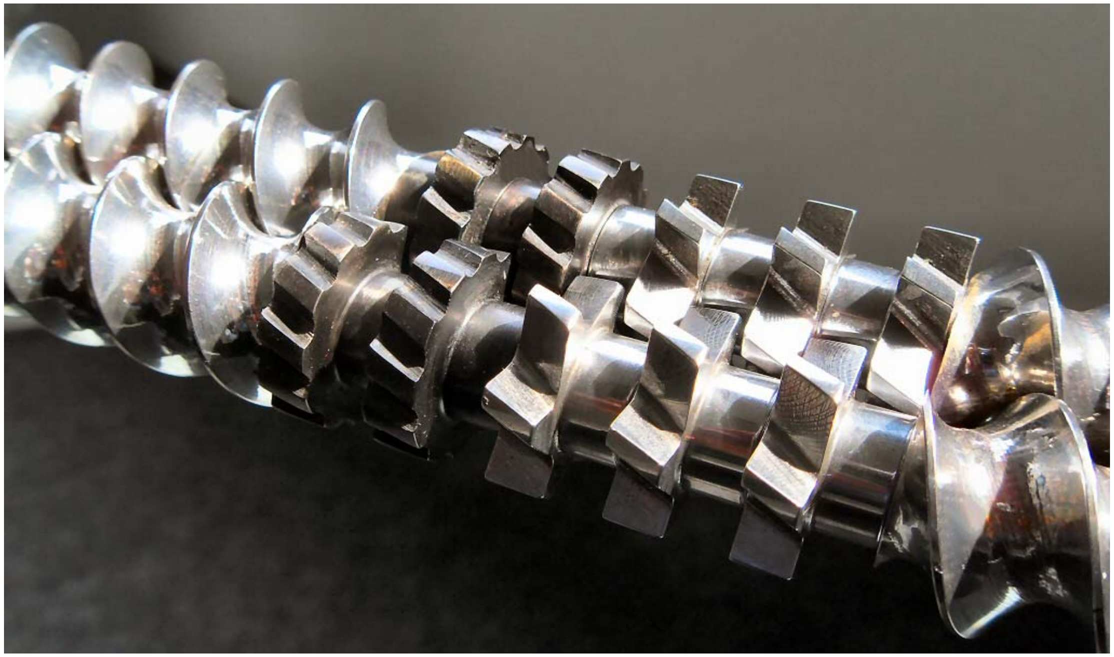
+ low cost
- bad compounding

Twin Screw Extruder:

- **Co-rotating**
  + flexible machine
  + good compounding
  - feeding system required
- **Counter-rotating**
  + fixed residence time (PVC)
  + high pressure built-up
  + low shear-heating
  - limited mixing
- **Conical screws**
  + easier gearbox
  + high torque
  - limited length
  - no segmented screw
- **Parallel screws**
  + segmented screw possible
  - sophisticated (expensive) gearbox

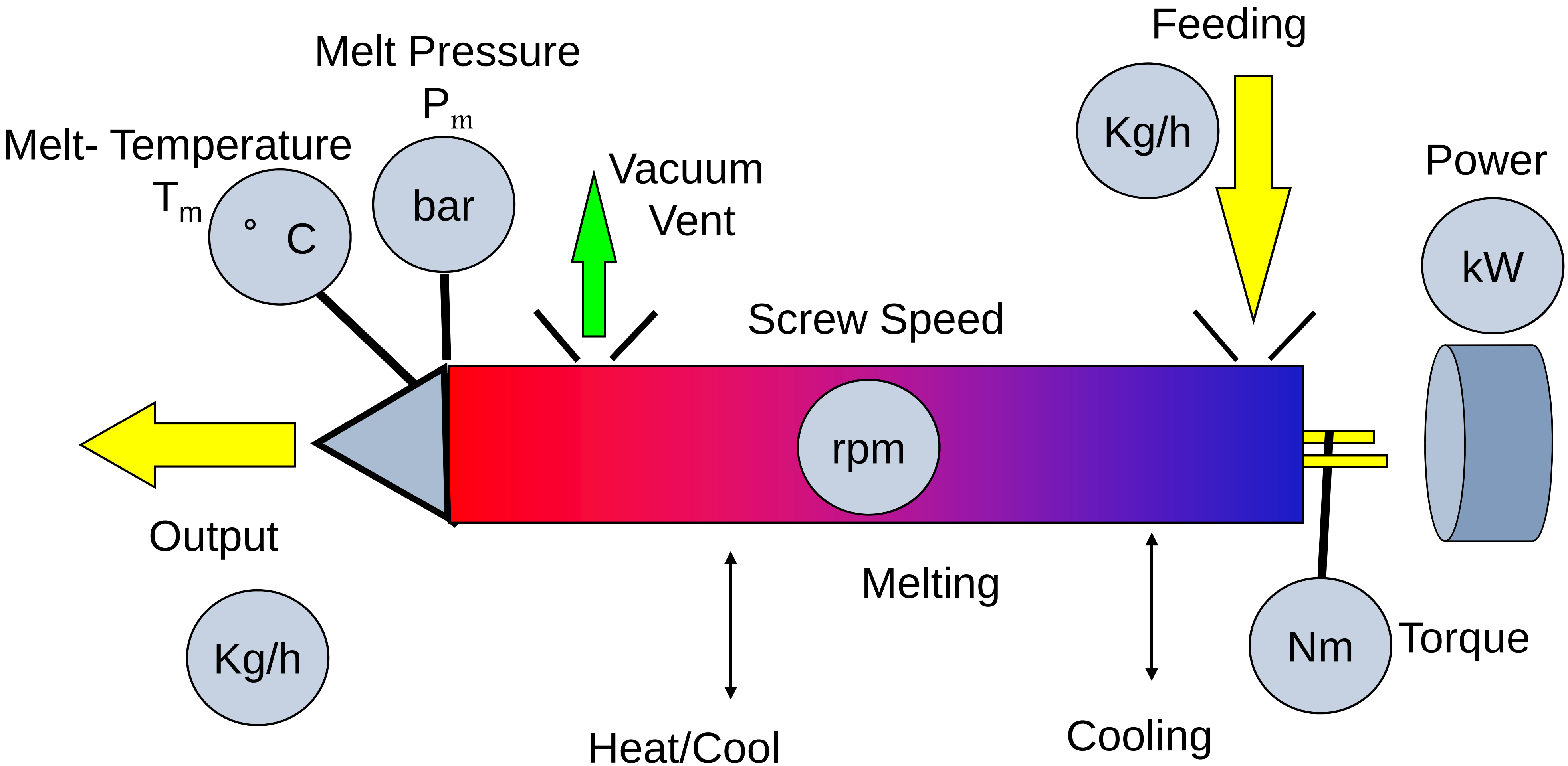


Twin-Screw Extruders – Typical Processing Tasks



- **Melting / Plasticizing**
- **Mixing / Homogenising**
- **Cooking**
- **Alloying of different polymers**
- **Chemical Reactions**
  Reactive Extrusion
  Polymerisation
- **Incorporation of fillers such as:**
  Talcum, $CaCO_3$, Carbon Black, …
- **Degassing / Venting of Monomers, Solvents, …**
- **Incorporation of reinforcing additives such as:**
  Carbon fibres, CNT, Glass fibres, Nano Clay, …
- **Dispersion of Pigments**
- **Modifying Polymers by incorporation of Plasticizers, cross-linking Agents, Flame Retardants, UV-Stabilisers, …**

Twin Screw Compounding



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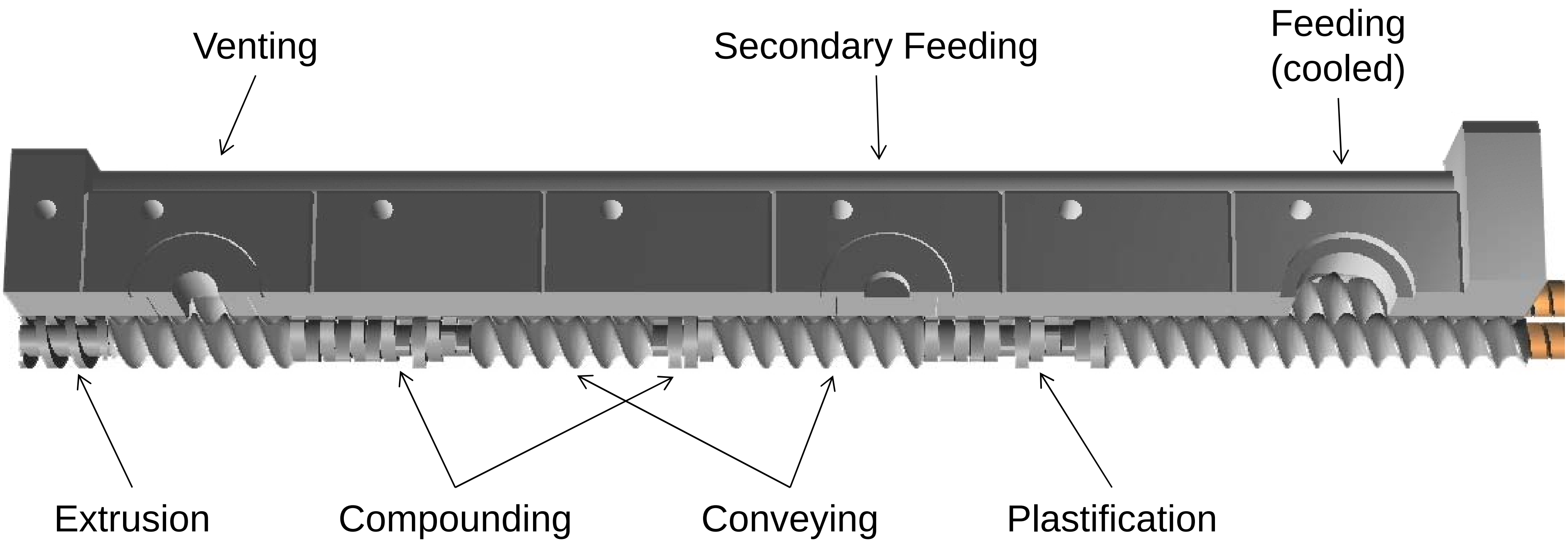
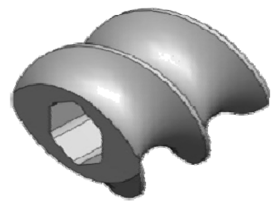
Twin Screw Compounding



Segmented Screw Design

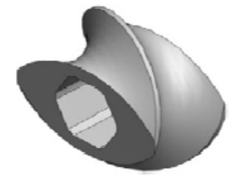
Discharge Element

Conveying Elements:

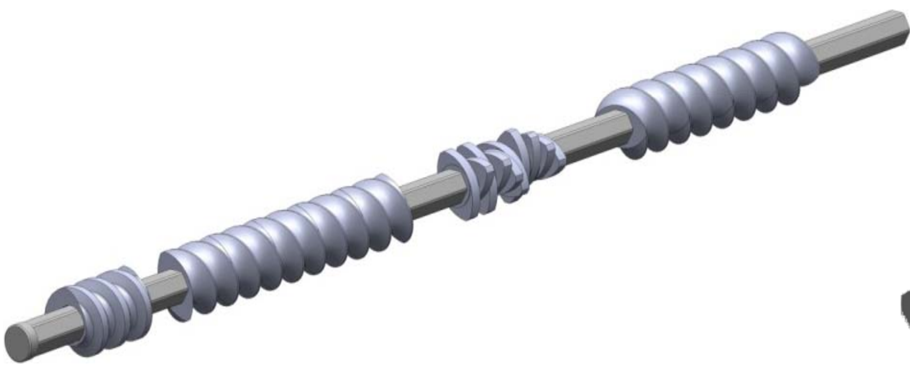


1 L/D

1/2 L/D

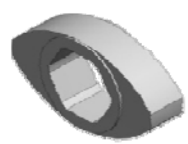


1/2 L/D reverse

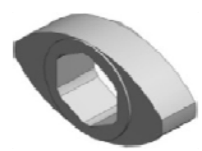


Easy change for different applications

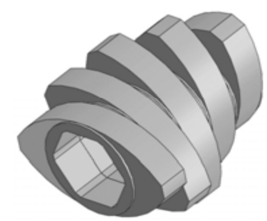
Mixing Elements:



0°



90°



Mixing Blocks

Parallel twin-screw extruder - Screw Elements:

Conveying elements:

Profiles with long helix are used:

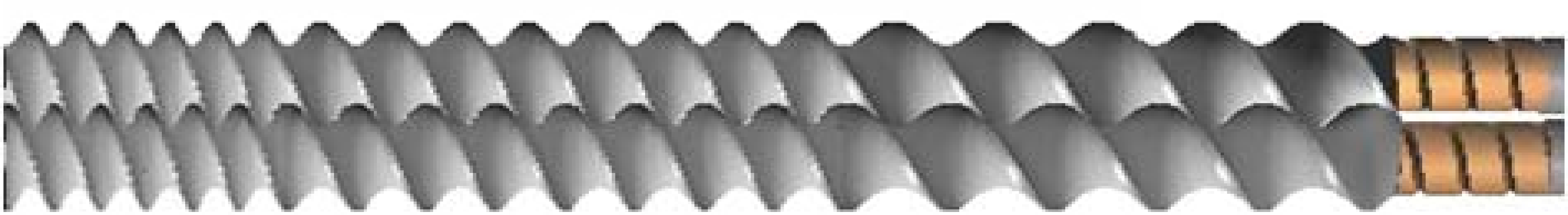
- in the feeding sections
- for melt exchange (longitudinal mixing)
- for degassing (venting)

Profiles with short helix are used:

- for pressure built up
- in front of kneading elements

Screw Elements: Conveying Elements

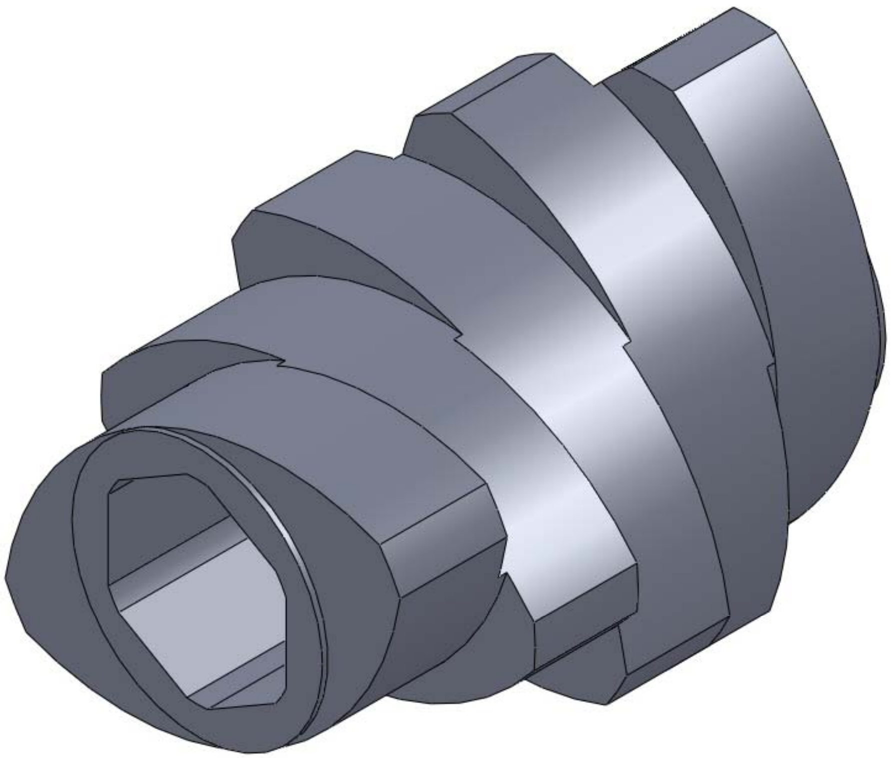
- **Self wiping geometry**
- **Only partial filled**
- **Different pitches**
  - 1 L/D: - Standard conveying
  - 1.5 L/D: - In the feed zones
  - 2/3 L/D: - Building up pressure
    - For compressing material with low bulk density



2/3 L/D **1 L/D** **1.5 L/D**

Parallel twin-screw extruder - Screw Elements:

Mixing Elements :



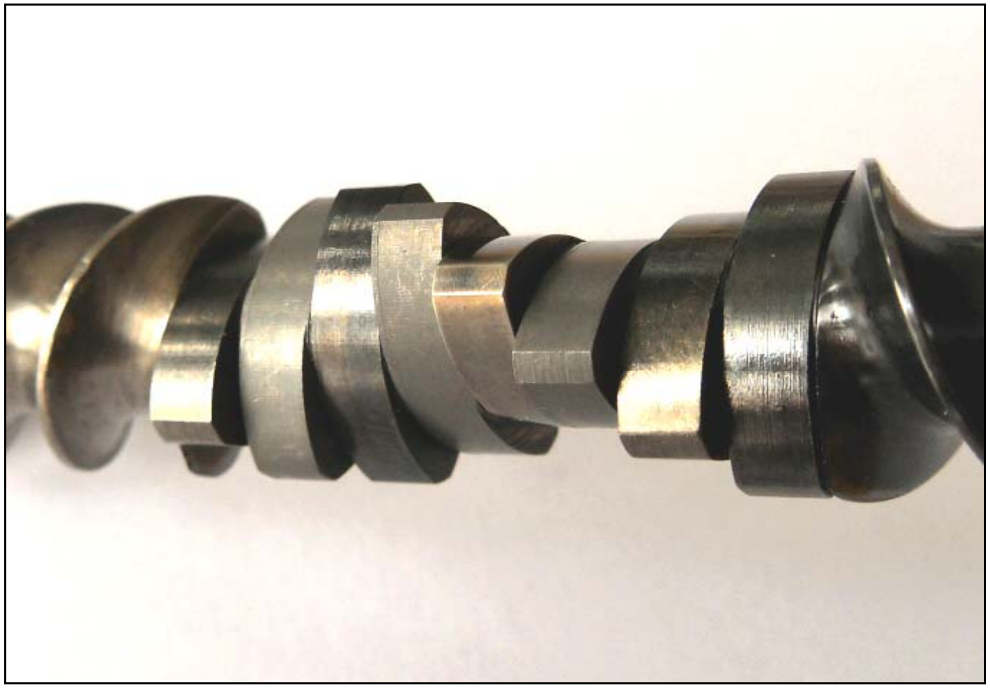
- **Mixing Elements are used to:**

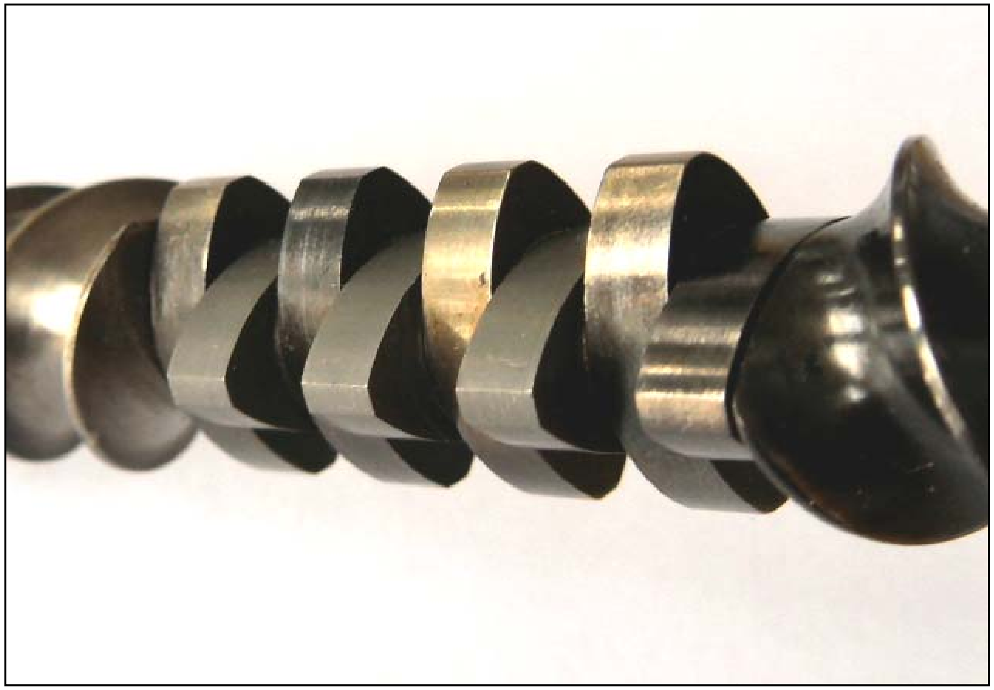
  - introduce shear energy
    to the extruded materials

- **The disks are arranged in different offset angles used for:**

  - melting
  - shearing
  - mixing
  - dispersing

Screw Elements: Mixing Elements

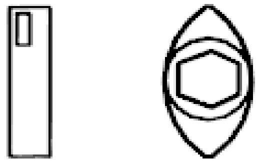




30° Offset – conveying
Shorter residence time
Lower shear

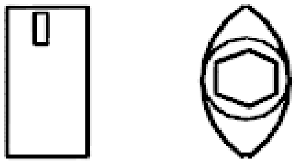
90° Offset – not conveying
Longer residence time
Higher shear

Dispersive & Distributive Mixing



Mixing Element
040-0104
¼ L/D

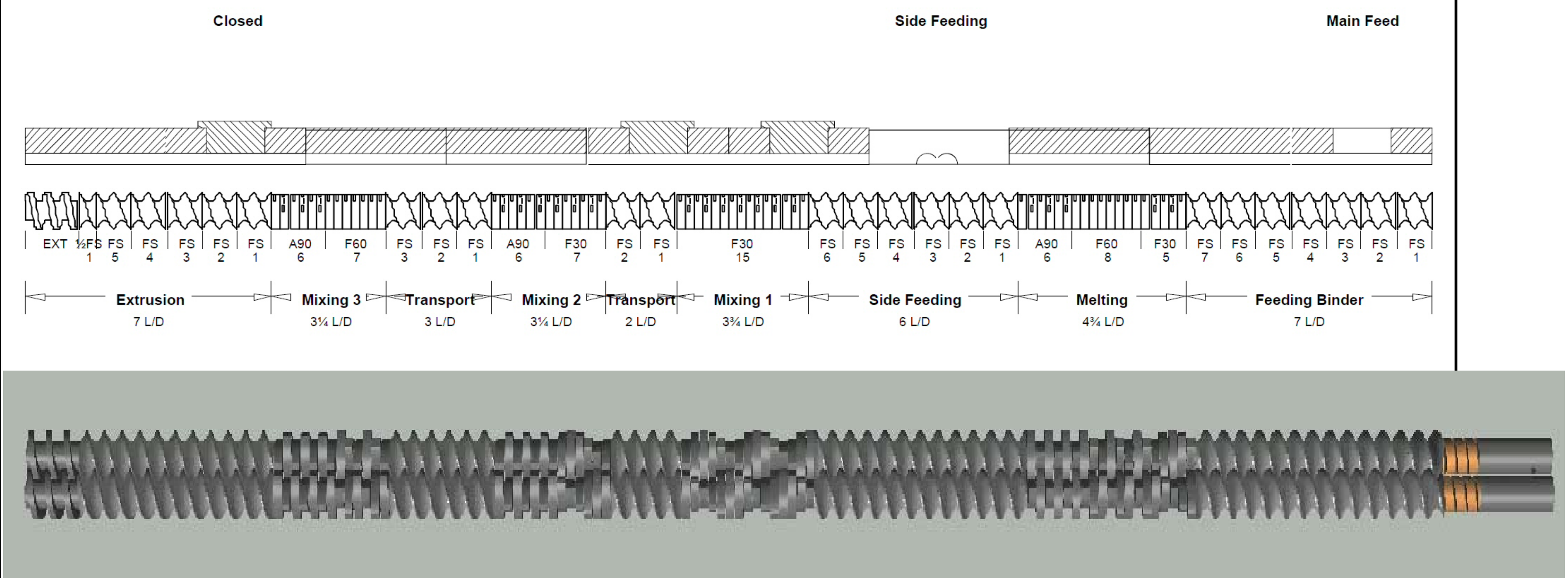
- Narrow Disks:
  - melt division
  - distributive mixing



Mixing Element
041-2632
½ L/D

- Wider Disks:
  - extensional shear
  - dispersive mixing

Typical screw configuration – PIM application



General Assembly

2 x 040-1575	Coupling Retaining Screw
2 x 040-1573	Gland Bush
2 x 040-1574	Coupling
2 x 045-1137	24 mm Shaft, 40 L/D

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Screw Design PIM

TSE 24 MC D: 24 mm - L/D: 40

Material	CPM	
Date		
Issue		matthias.jaehrling

Generated with Twin Screw Configurator, Version: 1.01 (13)

File: C:\Programme\ScrewConfig\setups\24-40_PIM.scd

Twin Screw Solutions

MiniLab

5 -10 ml

11 mm

0.02 - 2 l/h

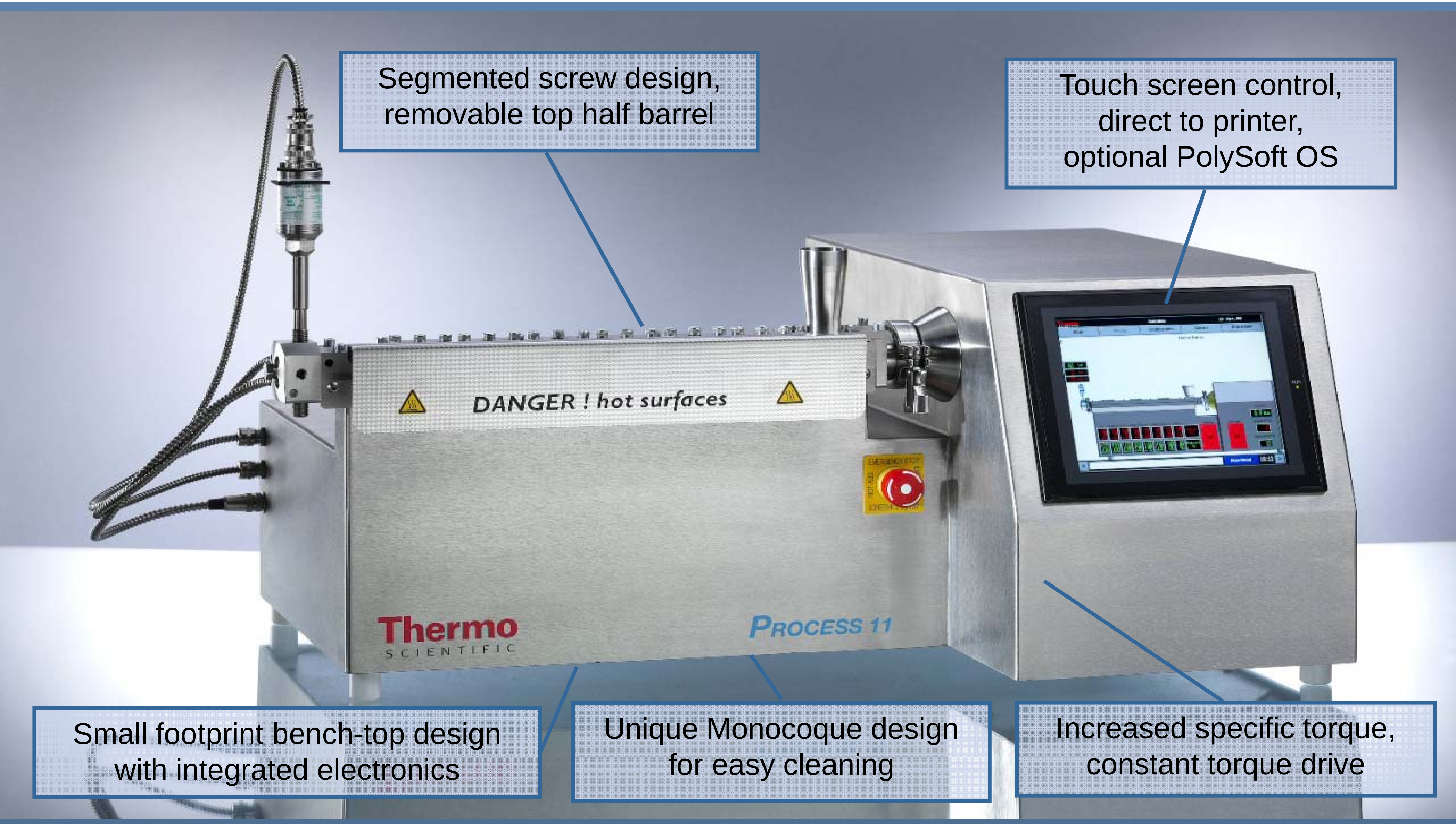
16 mm

0.2 - 5 l/h

24 mm

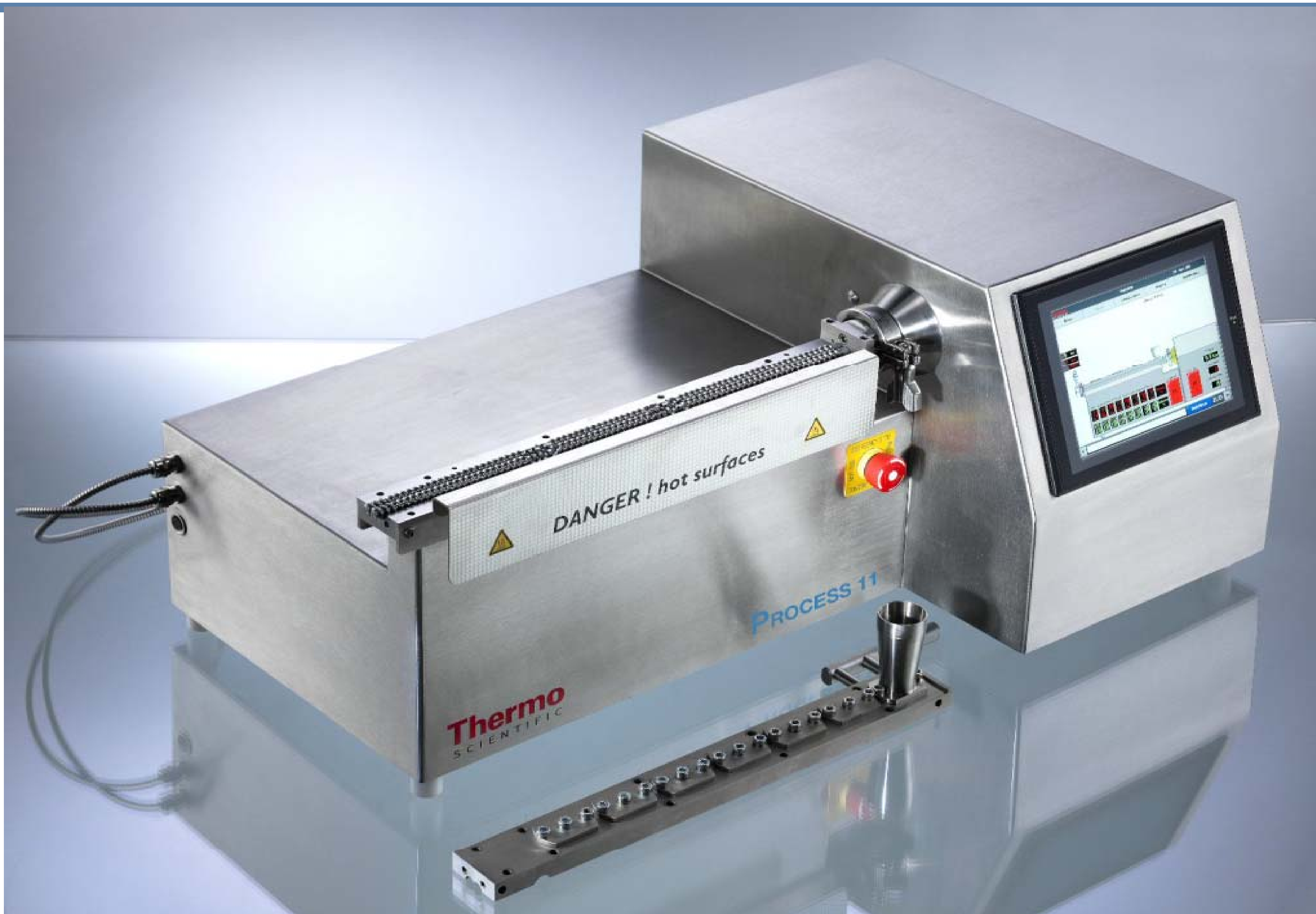
1 - 20 l/h

Process 11: Highlight Features



Bench-Top Design with integrated Electronics

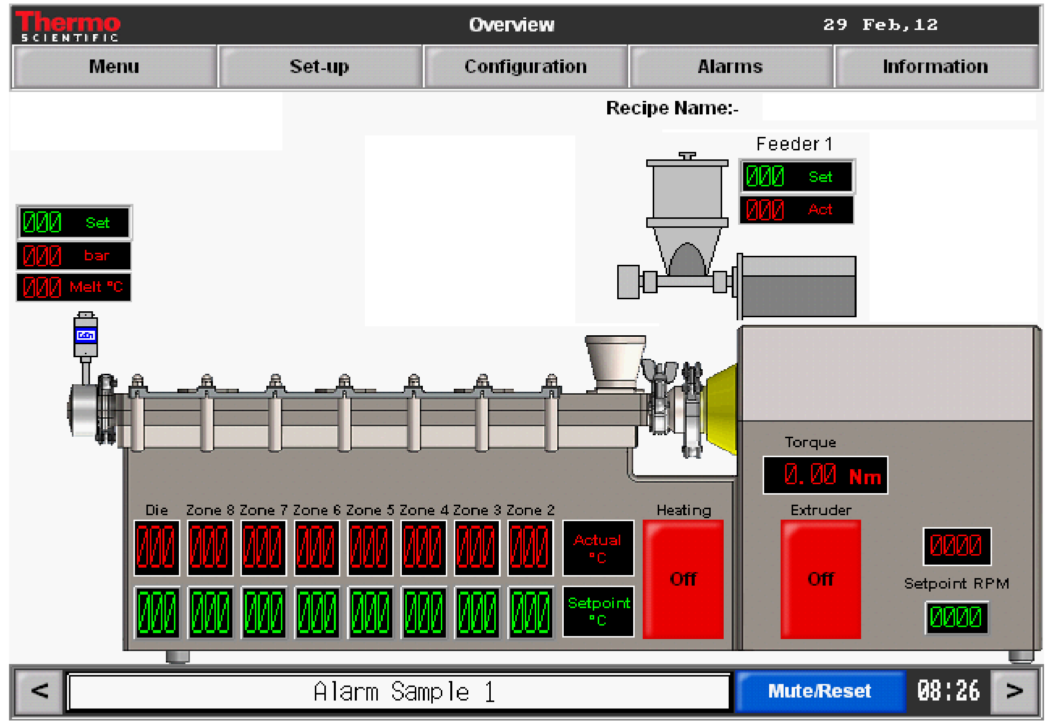
Process 11 TSE: Removable Top Half Barrel



Process 11 TSE: Segmented Screws

Process 11 – Easy to Use Touchscreen

- **All Set-Points:**
  - Temperatures
  - Screw Speed
  - Feed rate(s)
- **Processing data**
  - Torque
  - Pressure
  - Melt Temperature
- **Processing trend**
  - M, n, p, T_M, FR vs. time
- **Temperature trend**
- **Temperature profile**
- **Recipe storage**
- **Alarm history**



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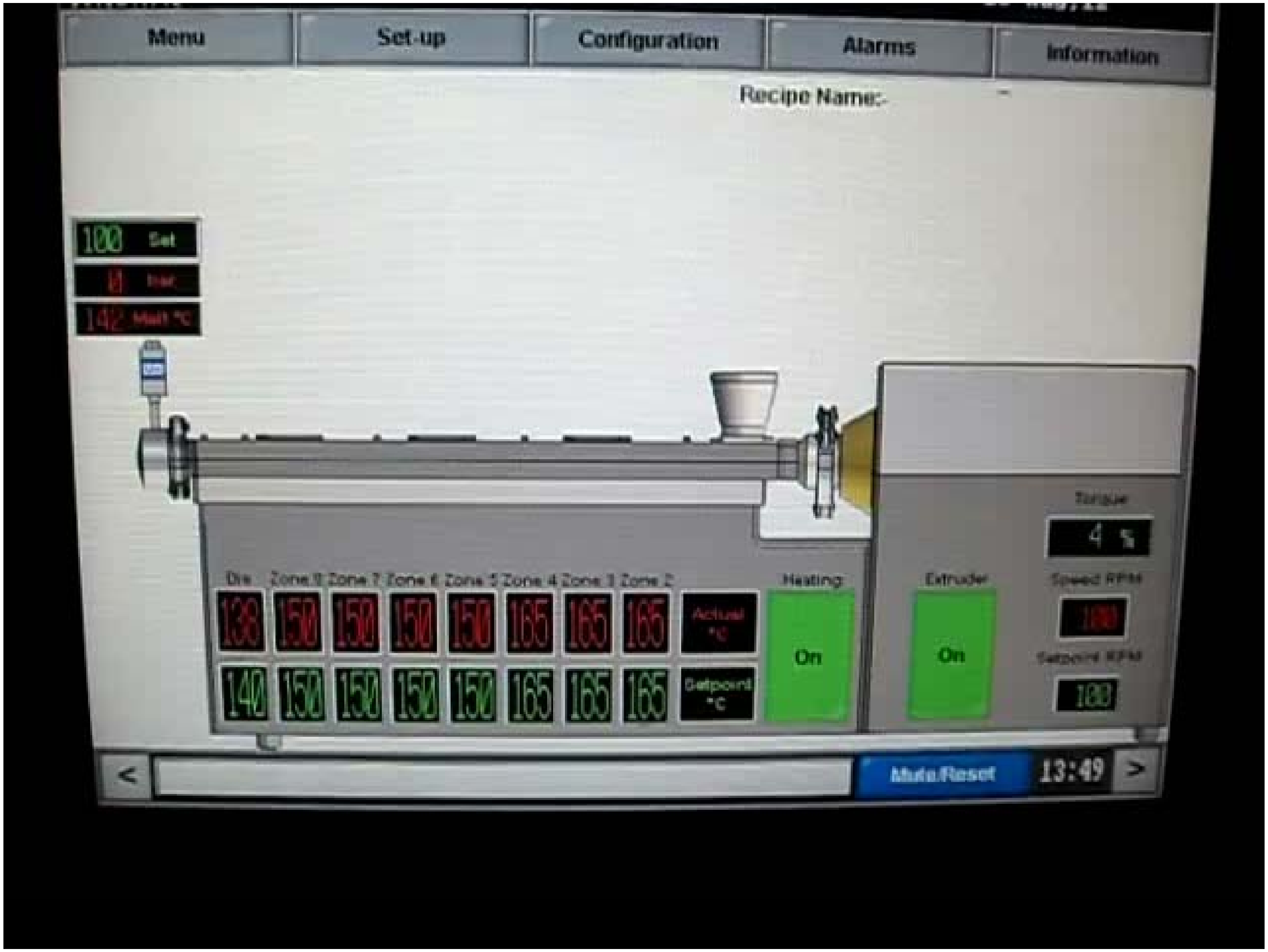
Thermo Scientific Process 11: PIM-Compounding



Example: Ceramics for PIM – Feeding options (1)

A HAAKE PolyLab OS System with RheoDrive16, a Rheomex PTW16/25 XL parallel twin screw extruder and two HAAKE metering feeder were used to compound a polyethylene wax based binder with Zirconium-Oxide (85/15 % wt/wt) :

•**Two different feed methods were used:**

1) Split feed with two feeders, both in the main feed port of the extruder

2) Split feed with the first feeder (binder) in the main feed port and feeding of the ceramic powder into a secondary feed port along the extruder barrel

Typical PIM part above

This is material production for Micro PIM parts (left)

Ref.: Picture: ARC Seibersdorf research center
Micro PIM part

Example: Ceramics for PIM – Feeding options (2)

Two different feed methods were used:

1) Split feed with two feeders, both in the main feed port of the extruder

2) Split feed with the first feeder (binder) in the main feed port and feeding of the ceramic powder into a secondary feed port along the extruder barrel

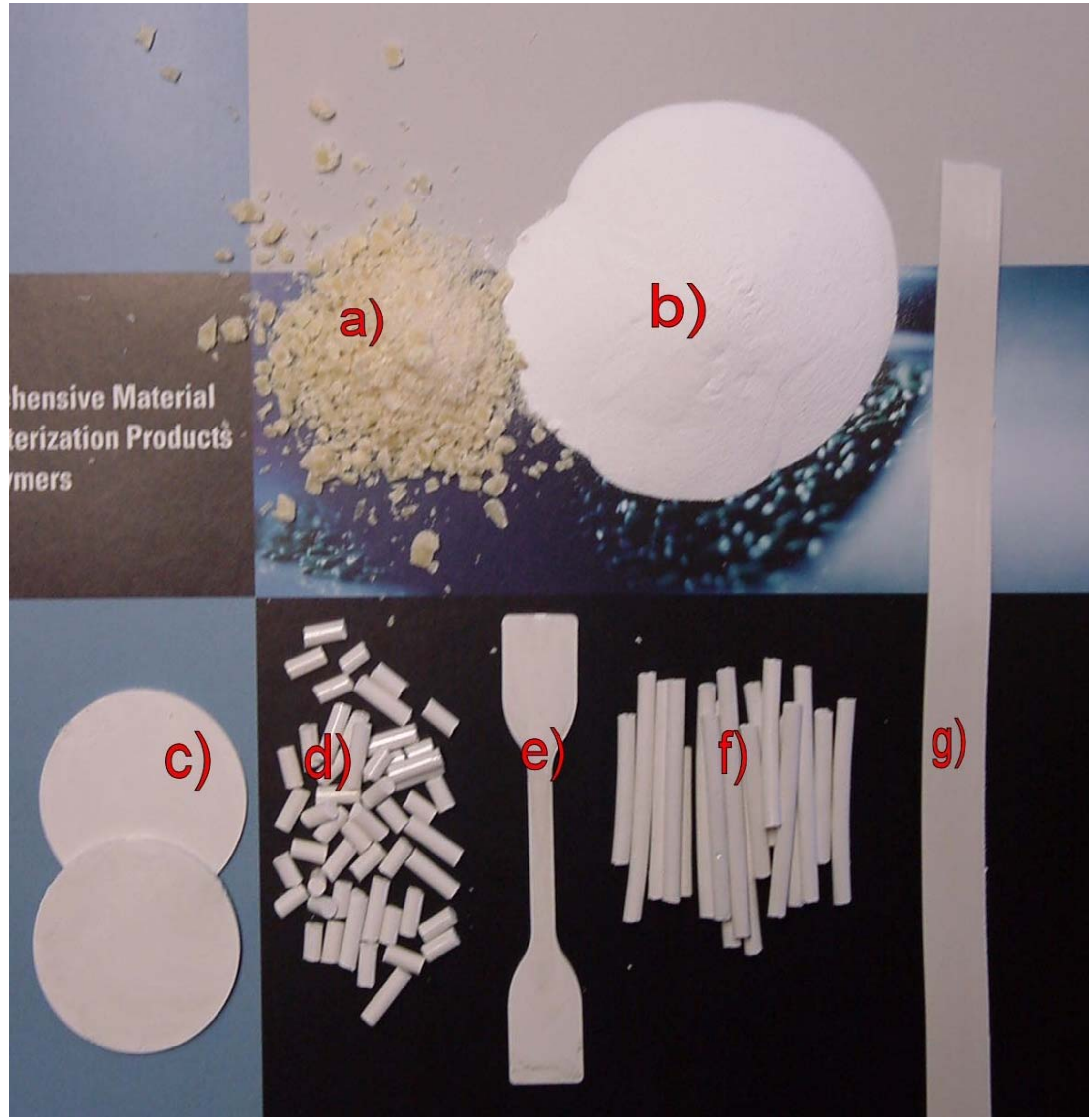
Result:
The separate feeding of wax and powder gave the better homogeneity (less agglomerates) and will reduce wear for extruder and extruder screw.

LR-56e Example PIM results

Raw material (a,b) and
Feedstock Product samples (c-g):

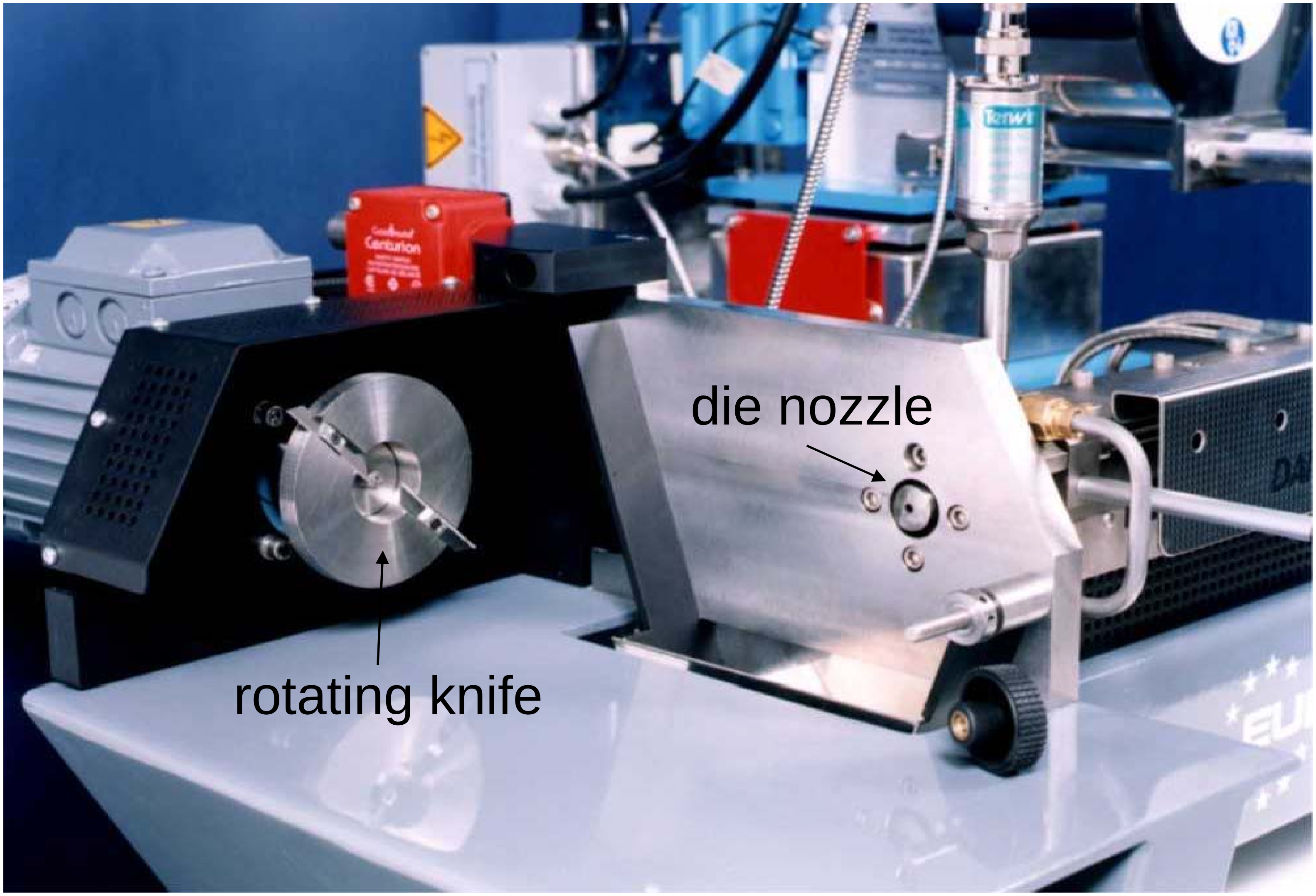
a) wax (PE)
b) ceramic powder (ZrO_2)

c) disks
d) pellets
e) tensile bar
f) strands
g) sheet



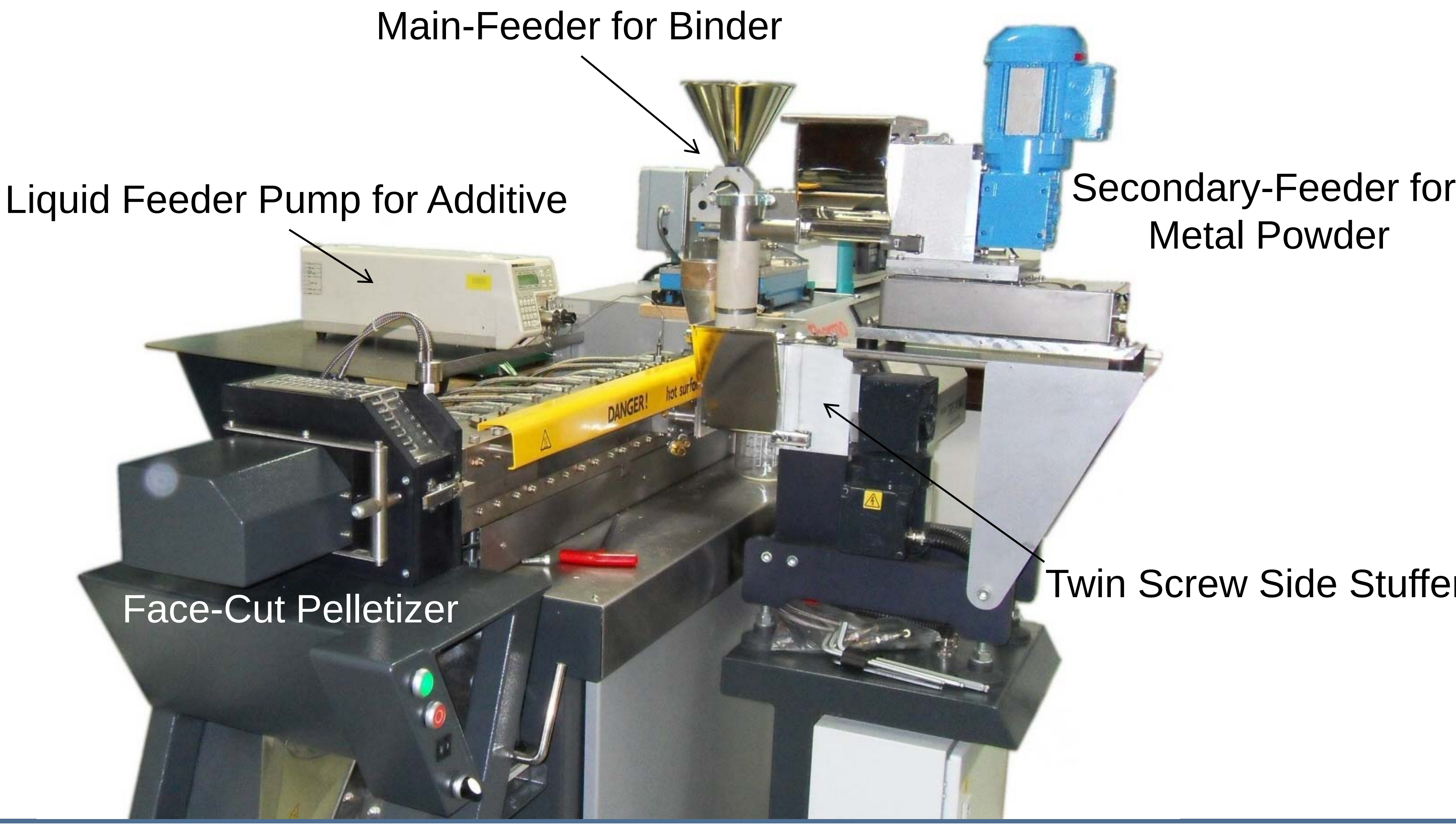
Classic Strand Pelletizing

Face-Cut Pelletizing



Face-Cut Pelletizing

PIM compounding with TSE24MC



Example: Compounding of a PIM-Compound

MICRO INJECTION MOLDING

SMALL SCALE SAMPLE-PREPARATION

HAAKE MiniJet Pro

Micro Injection Molding Machine for the production of specimens for tests like:

- Tensile tests (i.e. ASTM D638, ASTM D1708, ISO 178, ISO 527–2)
- Charpy Impact Strength (ISO 179)
- Izod Impact Testing (ISO 180, ASTM D256, ASTM D4508, ASTM D4812)
- Rheological tests
- DMA
- Distortion Temperature (ASTM E2092)
- Colour matching
- Tablets for bioavailability studies
- Customized solutions

HAAKE MiniJet – vertical alignment

Numerical controlled

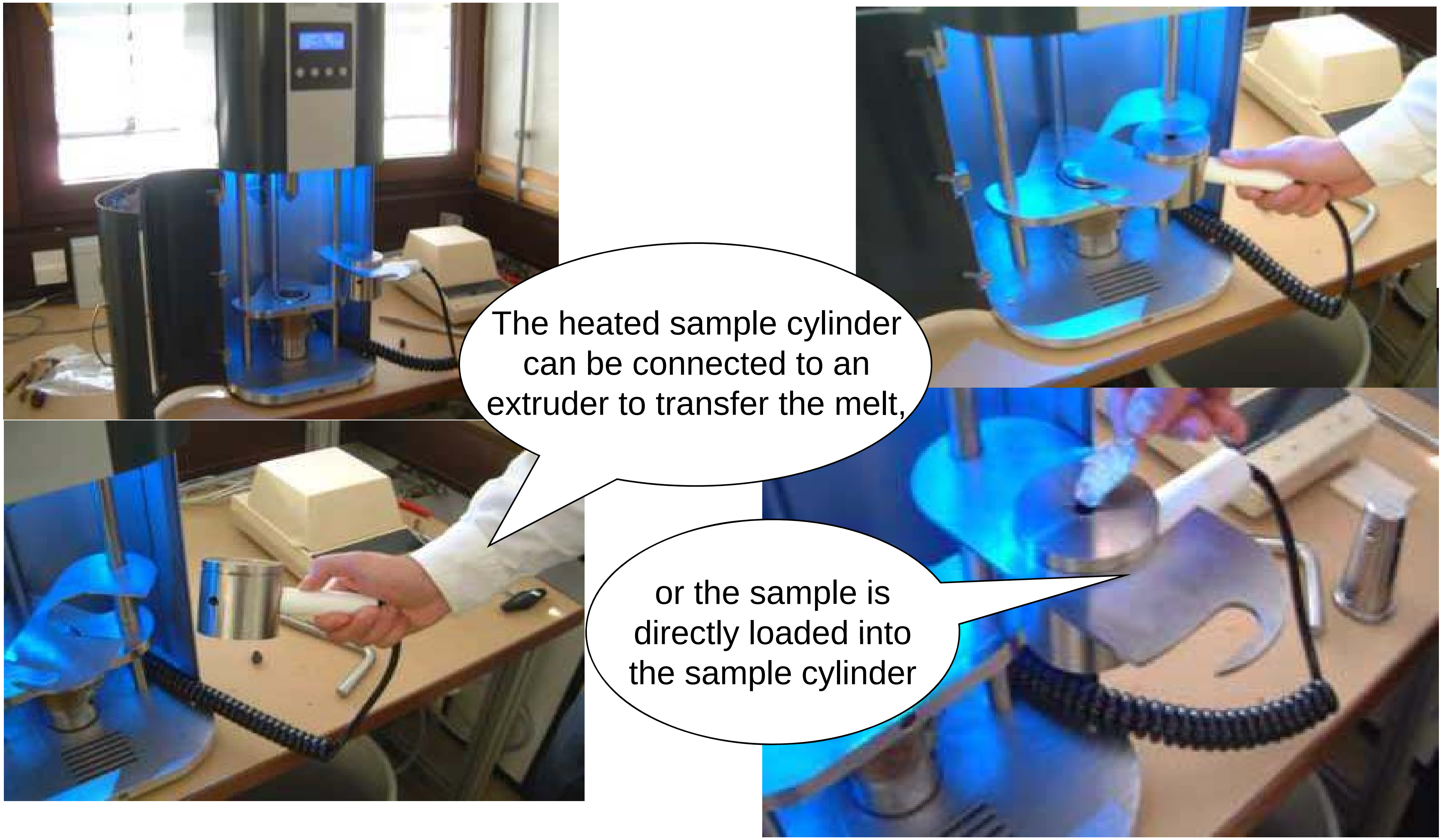
- 2 Temperatures controlled
- Pressure controlled (0.1 bar)
- Process controlled
  1. Injection pressure & duration,
  2. Post pressure & duration
- All parameter can be stored
- Language: English, German
- Units: bar, psi - ° C, F, K

Improved handling

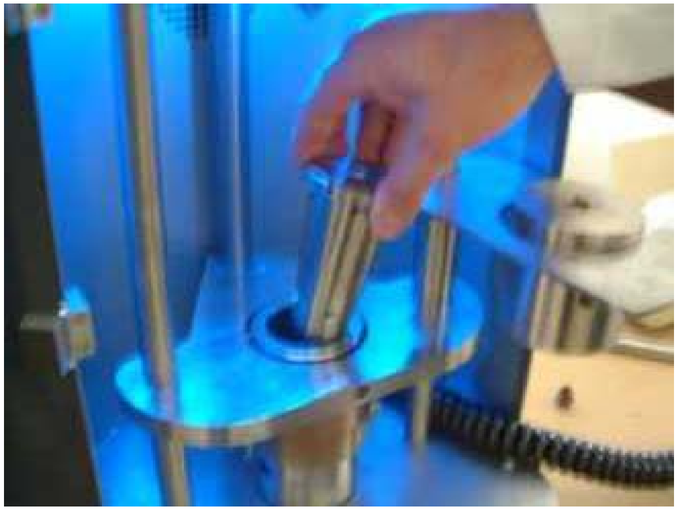
- No pressing lever necessary
- Easy filling of pellet samples
- Active liquid cooling option

Sample Volume	**2 - 12.5 ml**
Melt temperature	**max. 450 °C**
Mould temperature	**max. 250 °C**
Liquid Cooling Option	**max. 80 °C**
Injection pressure	**max. 1200 bar**
Electrical power	**230V / 110V**
Max air supply	**10 bar**

HAAKE MiniJet – Micro Injection Molding



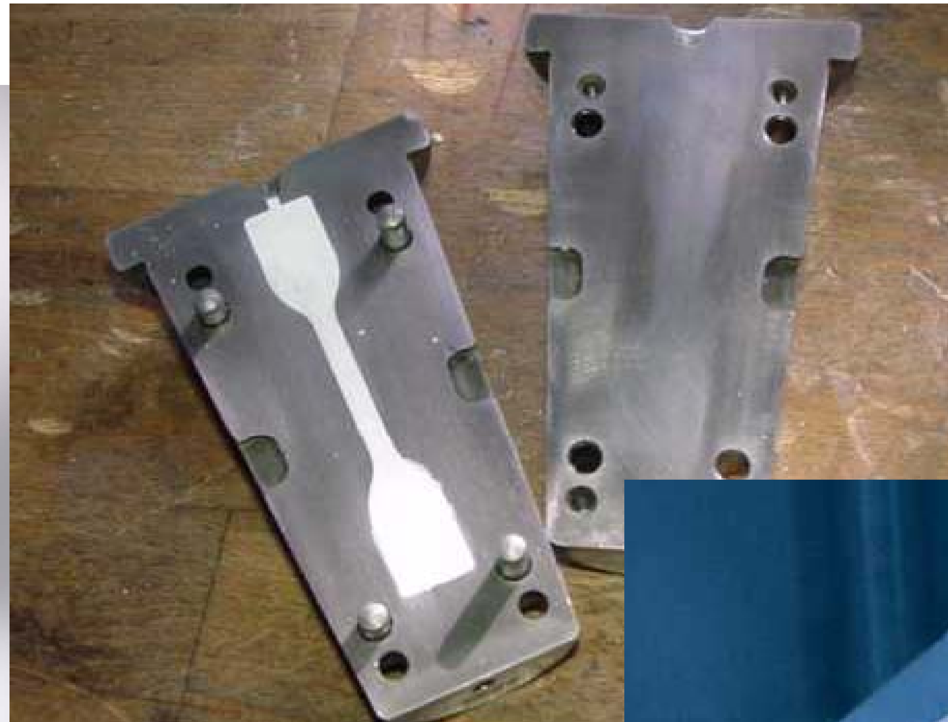
HAAKE MiniJet – Micro Injection Molding



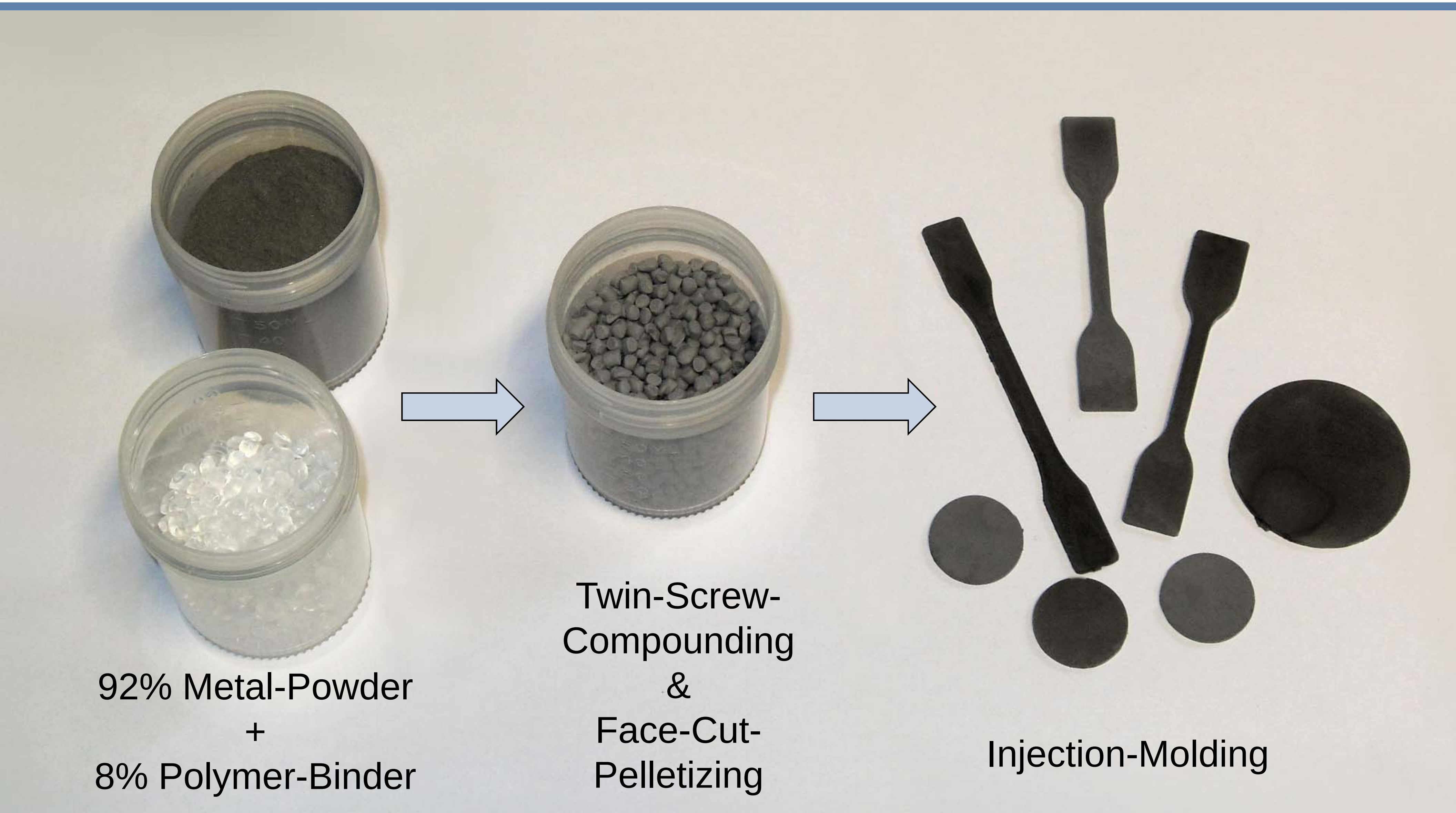
HAAKE MiniJet – Molds

HAAKE MiniJet – Molds

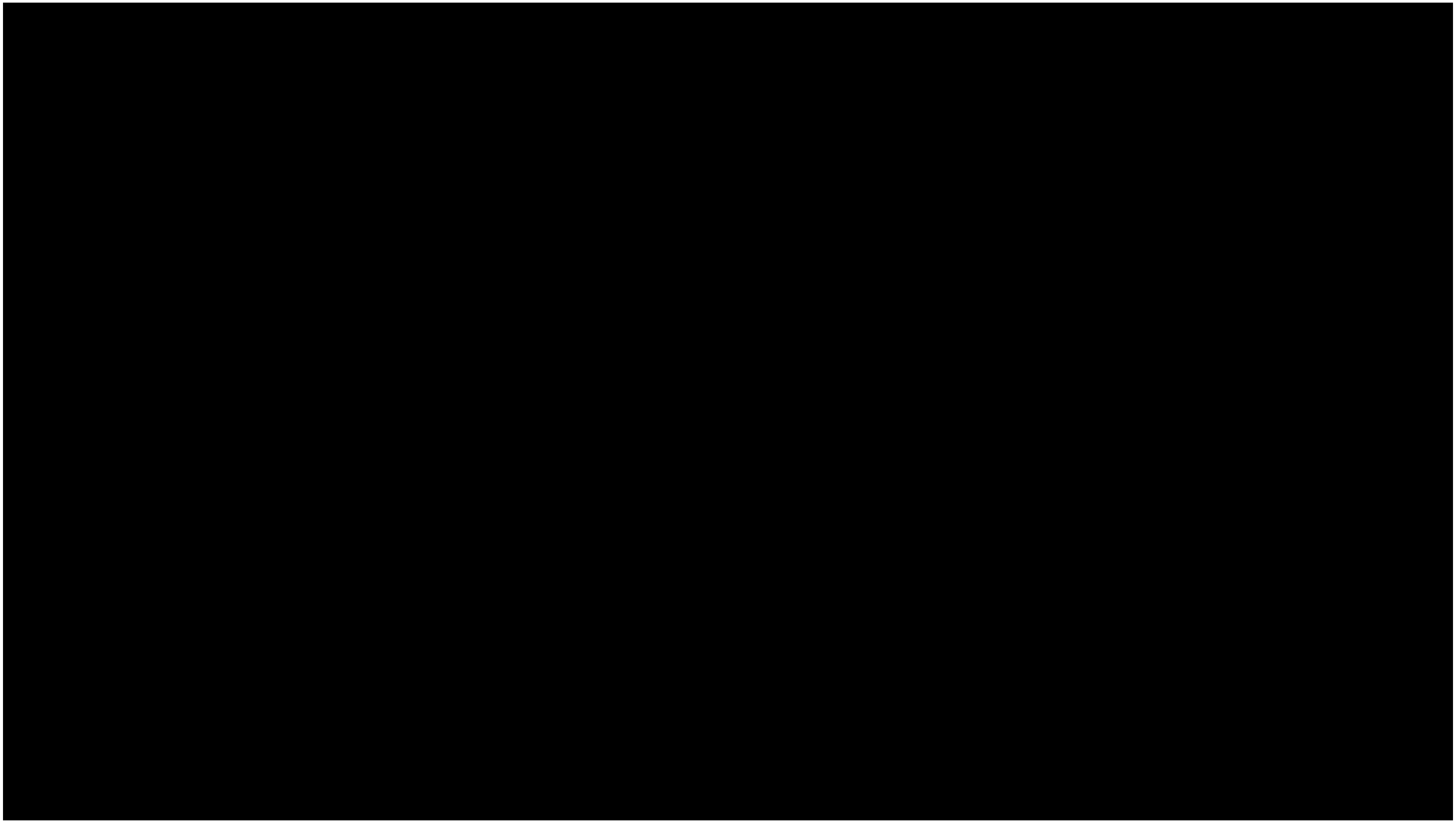
HAAKE MiniJet – Injection Molding with 2g – 15g feedstock



Example: MIM-Compounding



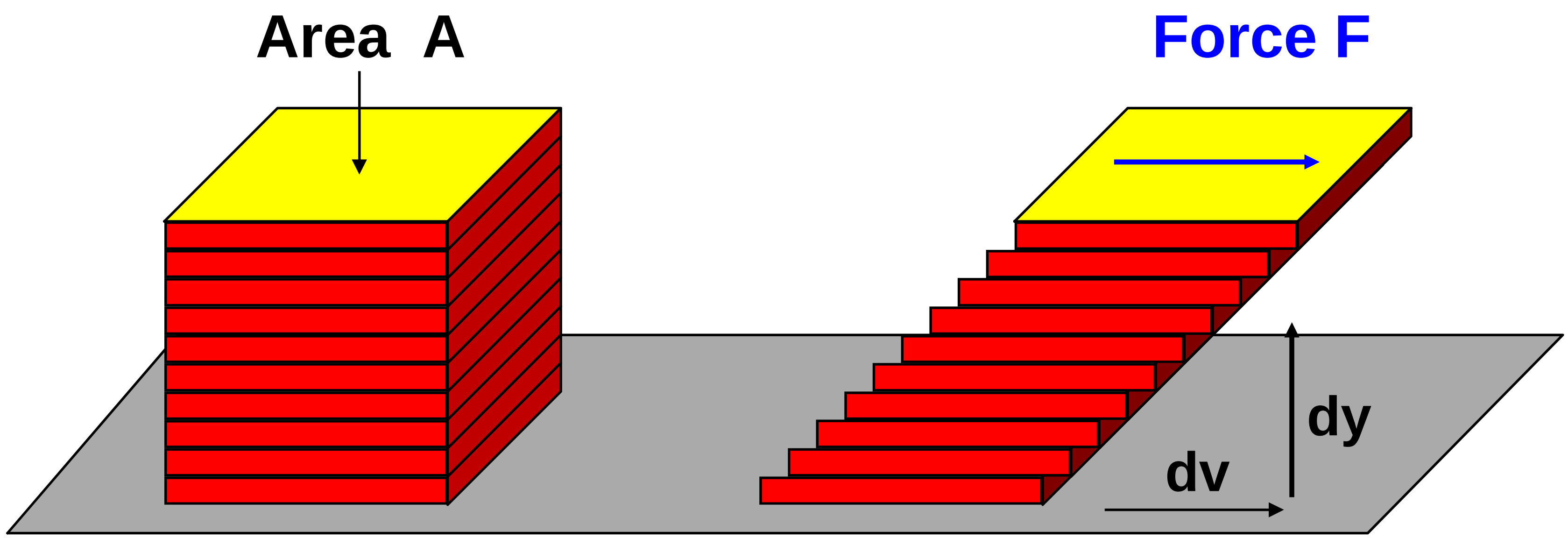
Thermo Scientific Process 11 & StrandLine & MiniJet



CAPILLARY RHEOLOGY

RHEOLOGICAL CHARACTERIZATION FOR OPTIMIZED FLOW PROPERTIES

Rheology *Newtonian plate model*



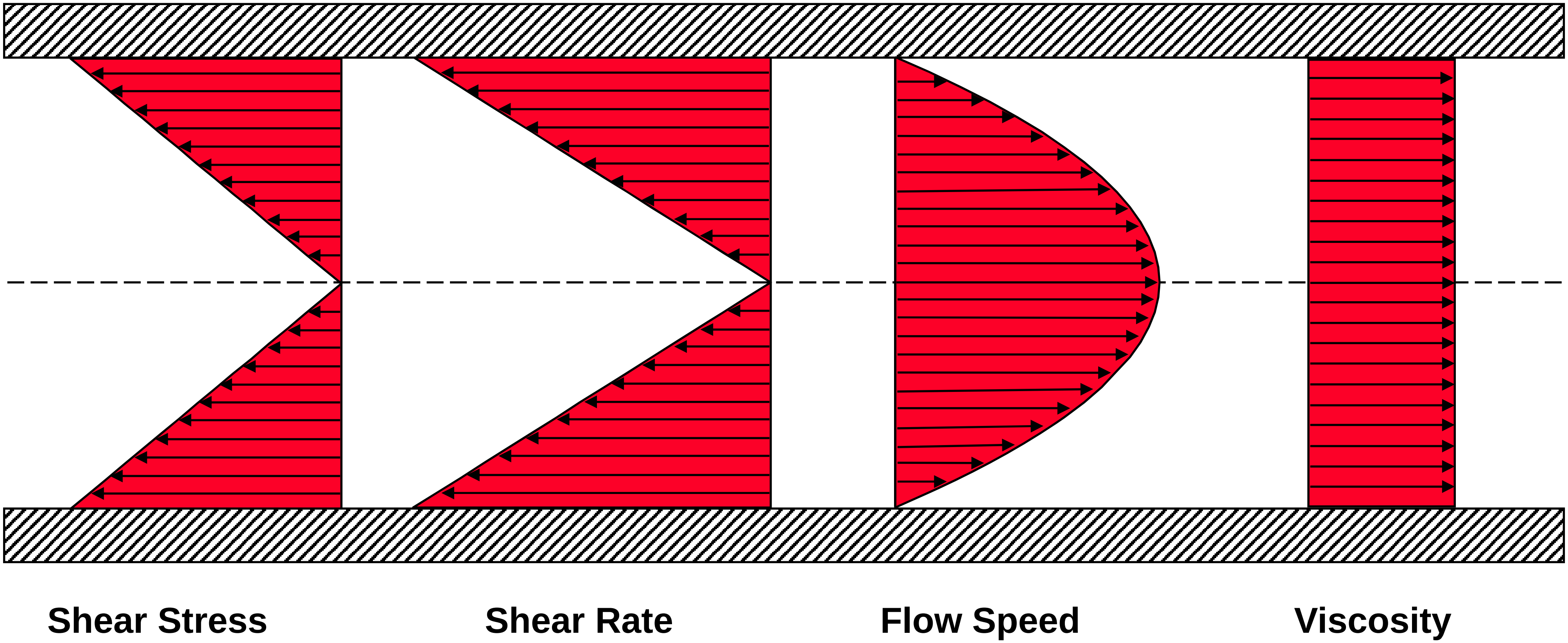
Shear Stress: $\tau = \frac{F}{A}$

Shear Rate: $\dot{\gamma} = \frac{dv}{dy}$

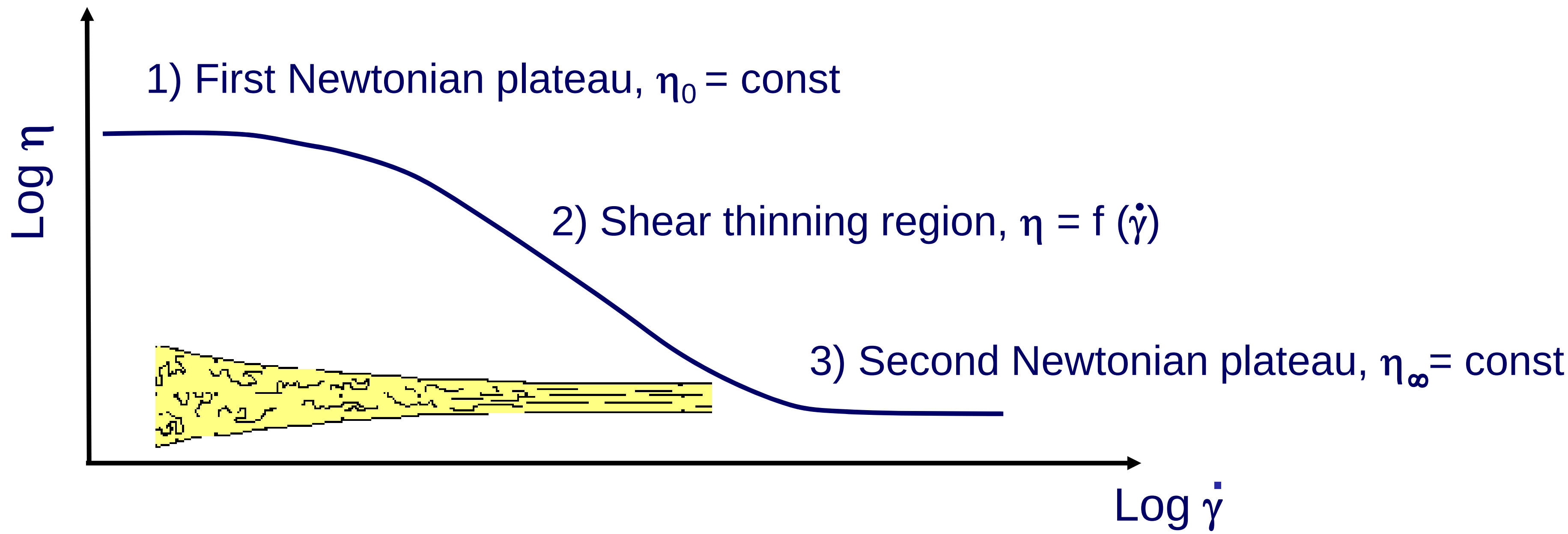
Viscosity: $\eta = \frac{\tau}{\dot{\gamma}}$

Pressure flow of a Newtonian liquid

Rod Capillary

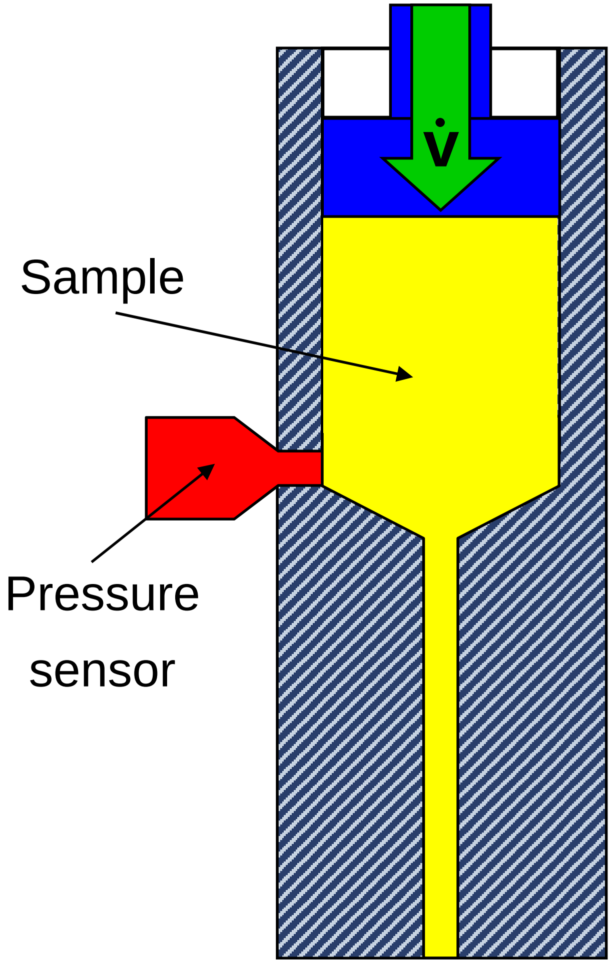


Typical viscosity function of a polymer melt



1) At rest each macromolecule can be found in a state of a three dimensional coil, that is entangled several times with their neighbor macromolecules.
2) With increasing shear the molecules are more and more orientated in the shear direction. The molecules disentangle to a certain extend, which lowers their flow resistance.
3) At infinite shear (theoretical!) the molecules are totally disentangled and aligned in the shear direction. Further increase in shear doesn't reduce their flow resistance anymore

Capillary Test - Measurement Principle

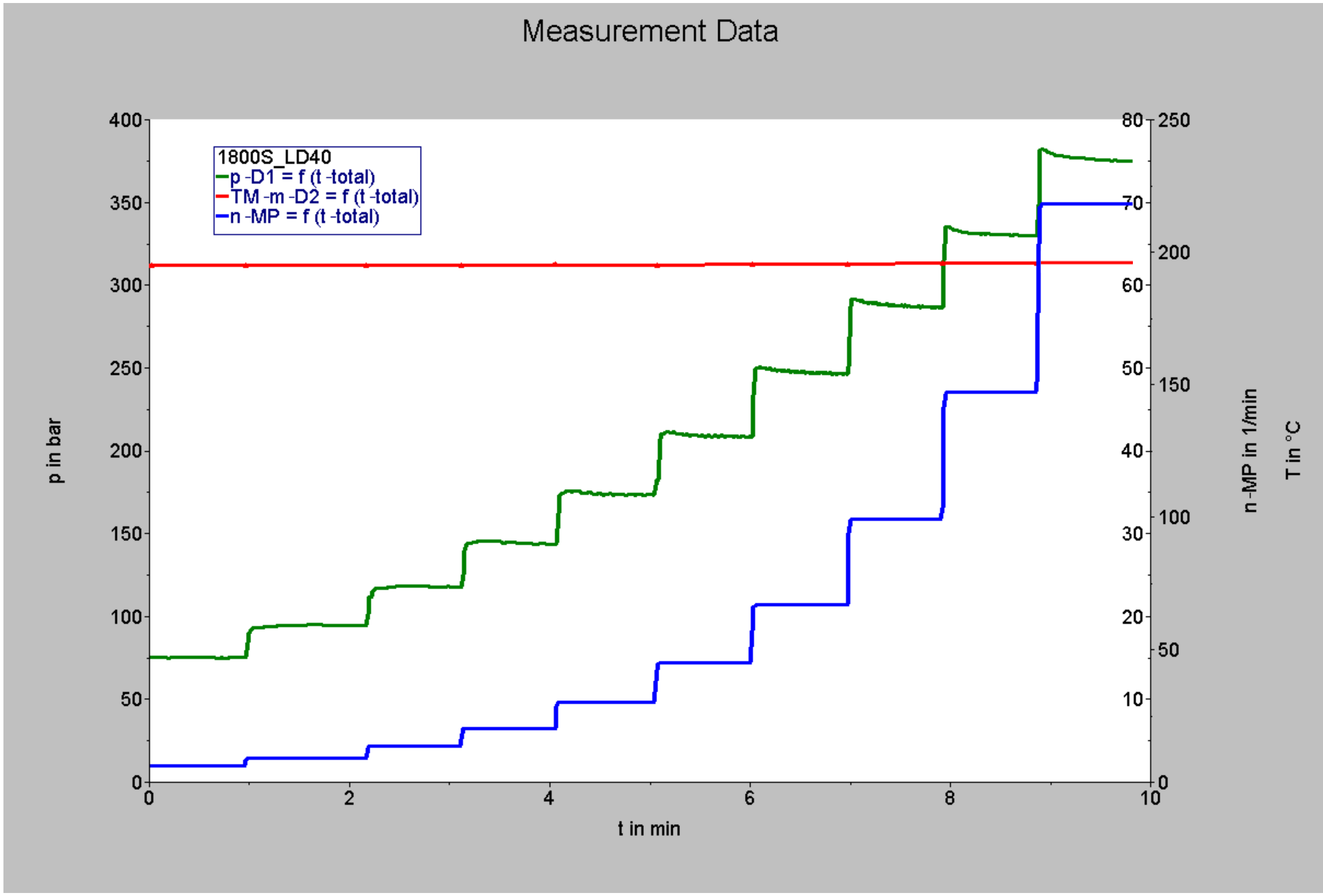


- **Sample is pressed with defined speed through the capillary.**
- **This volume flow (V̇) results in a constant shear rate in the capillary**
- **The higher the sample viscosity the higher it's resistance to flow out of the capillary.**
- **A higher viscous sample generates a higher pressure in front of the capillary.**
- **The measured pressure is related to the shear stress.**

$$\text{Viscosity} = \frac{\text{Shear Stress}}{\text{Shear Rate}}$$

Capillary Test - Measurement Principle

- To measure a viscosity curve the shear rate is changed by step wise changing the flow speed
- At each speed step the equilibrium pressure is measured and the shear stress is calculated
- From shear rate and shear stress the viscosity is calculated



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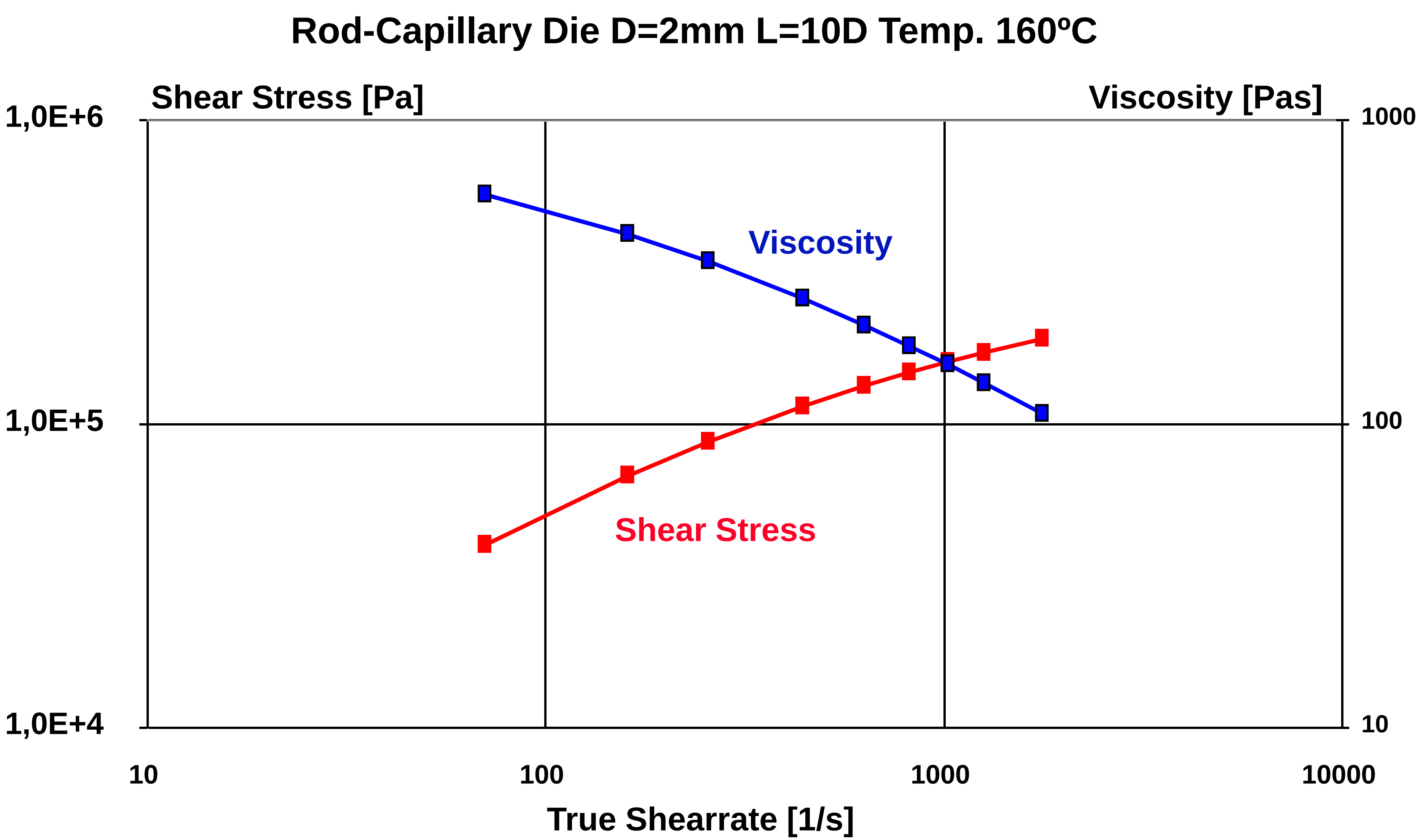
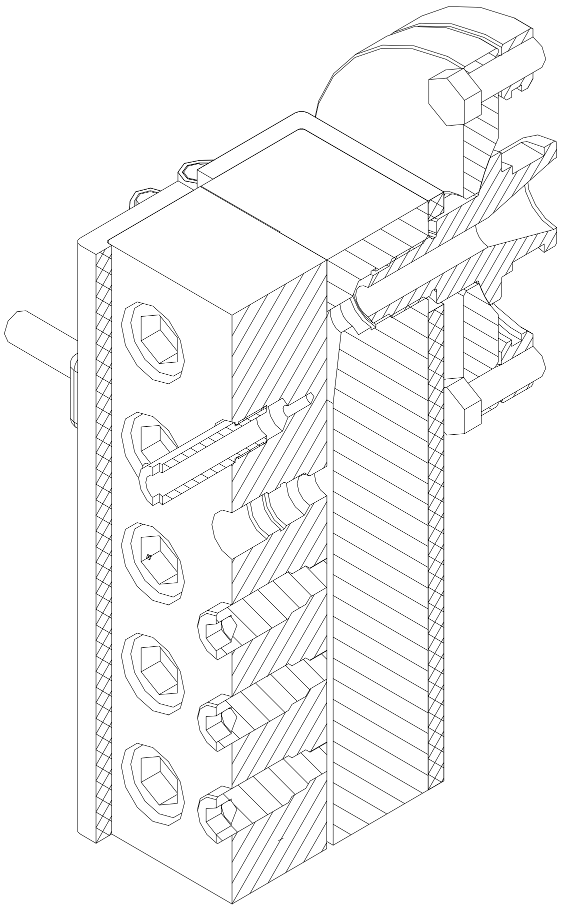
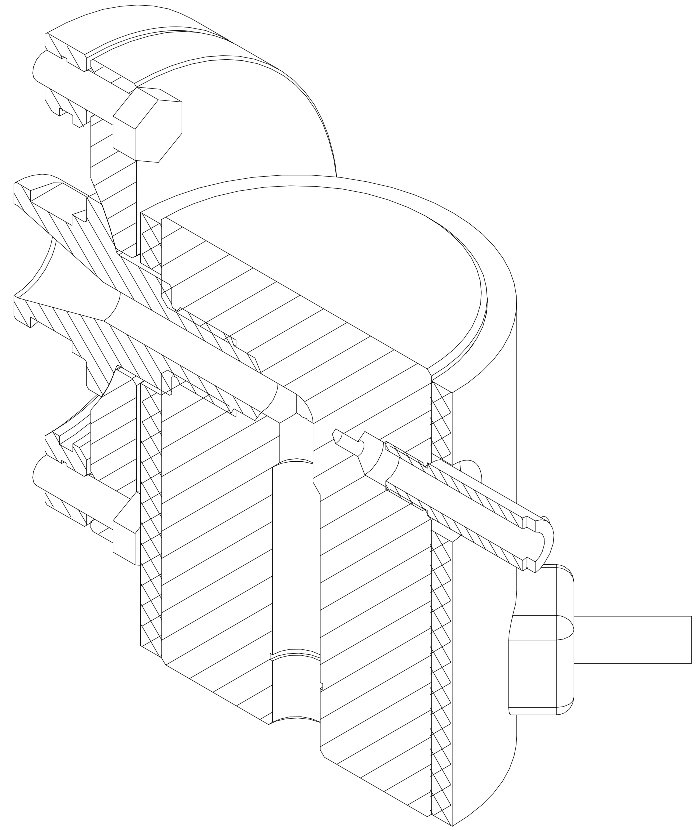
Viscosity measurements of ceramic material



Capillary Dies

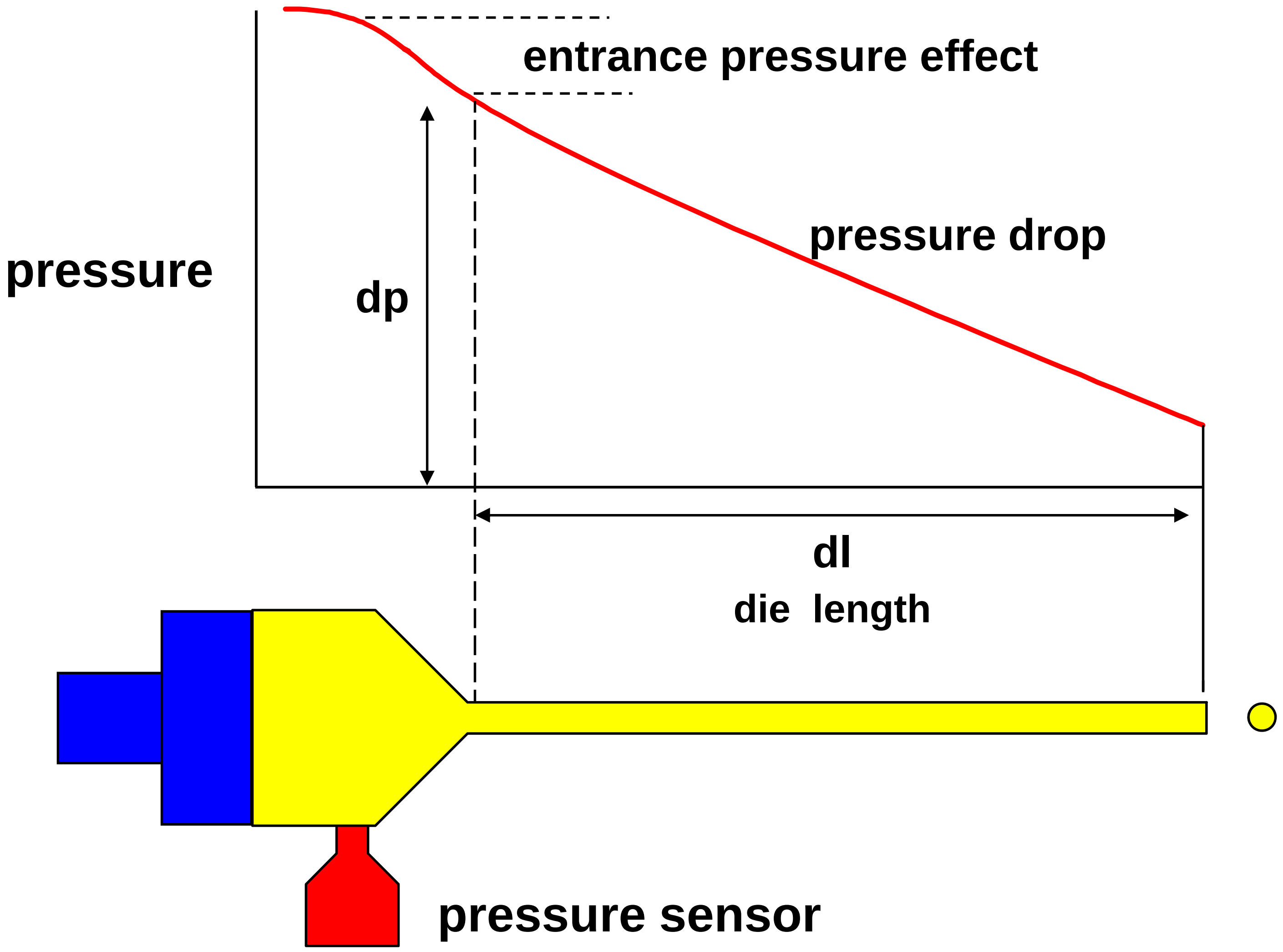


Slitcapillary Die



Rodcapillary Die

Rod Capillary Die



Rod Capillary Die

Calculations for Newtonian liquids:

Pressure Gradient: $p' = \frac{dp}{dl}$

Shear Rate: $\dot{\gamma} = \frac{4 \bullet Q}{\pi \bullet r^3}$

Volume flow: $Q = \frac{V}{t}$

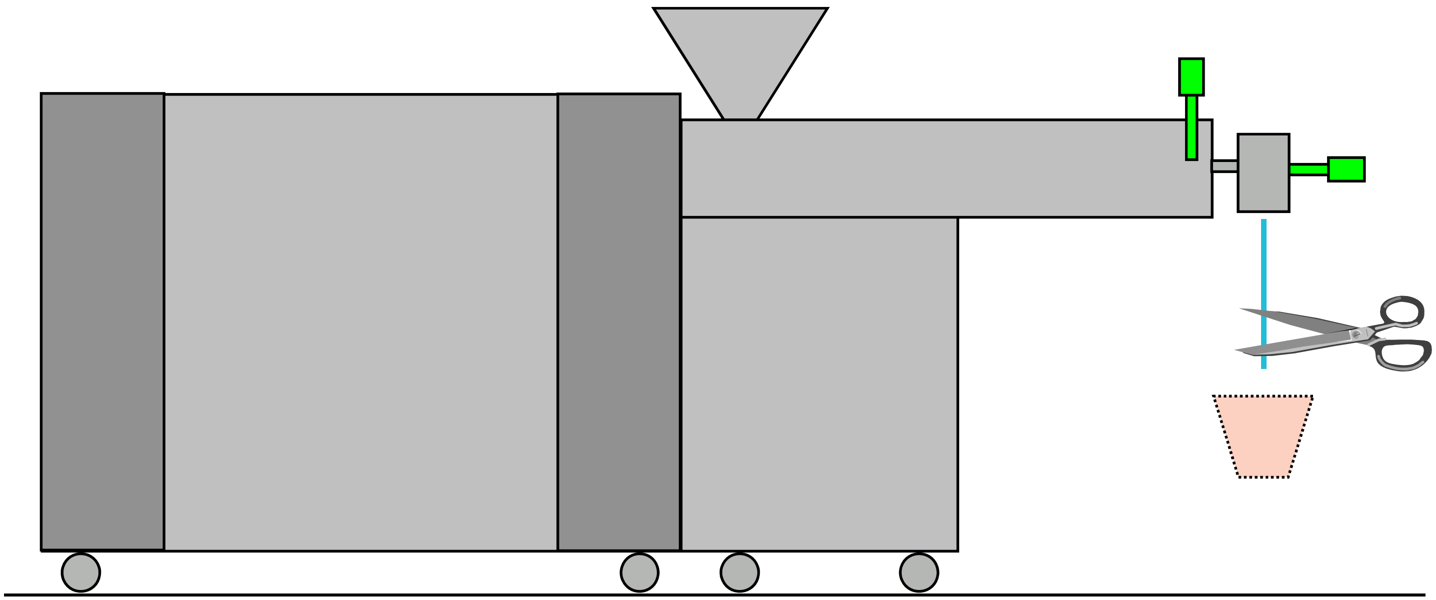
Viscosity: $\eta = \frac{\tau}{\dot{\gamma}}$

Shear stress: $\tau = \frac{r}{2} \bullet p'$

Assumptions:
- Laminar, stationary and isothermal flow
- Wall sticking
- *L/D > 10 (for laminar flow)*

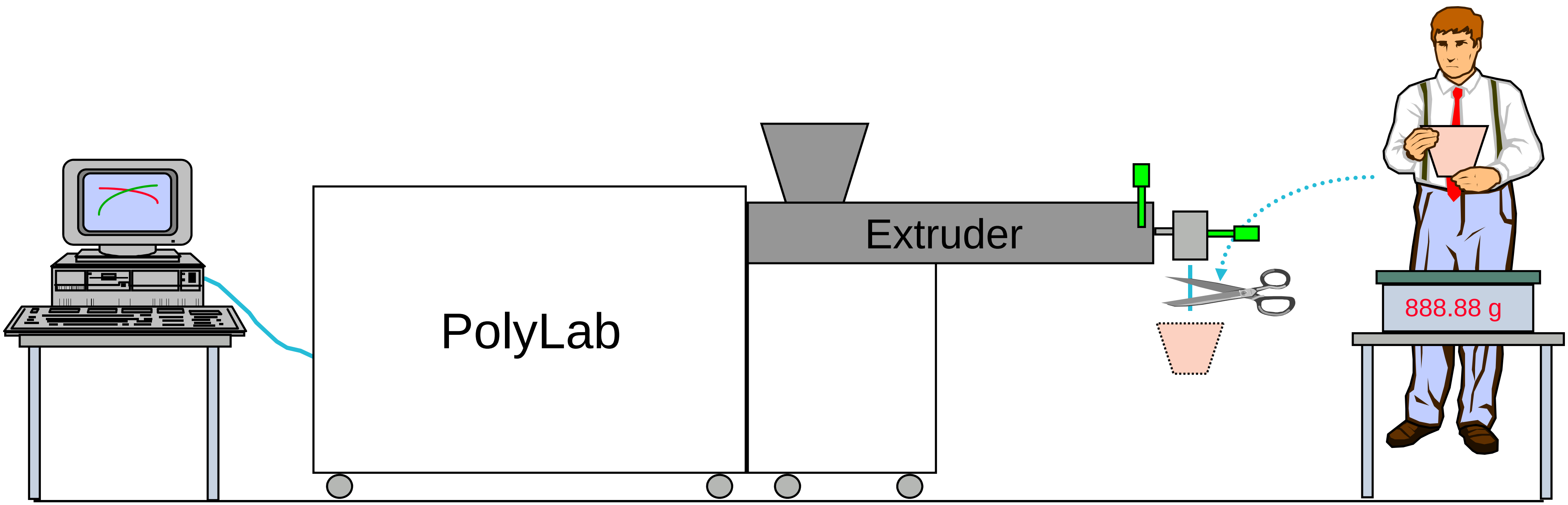
Extruder Capillary Rheology

- **Measuring Modes**



PolySoft Software – Measurement Modes

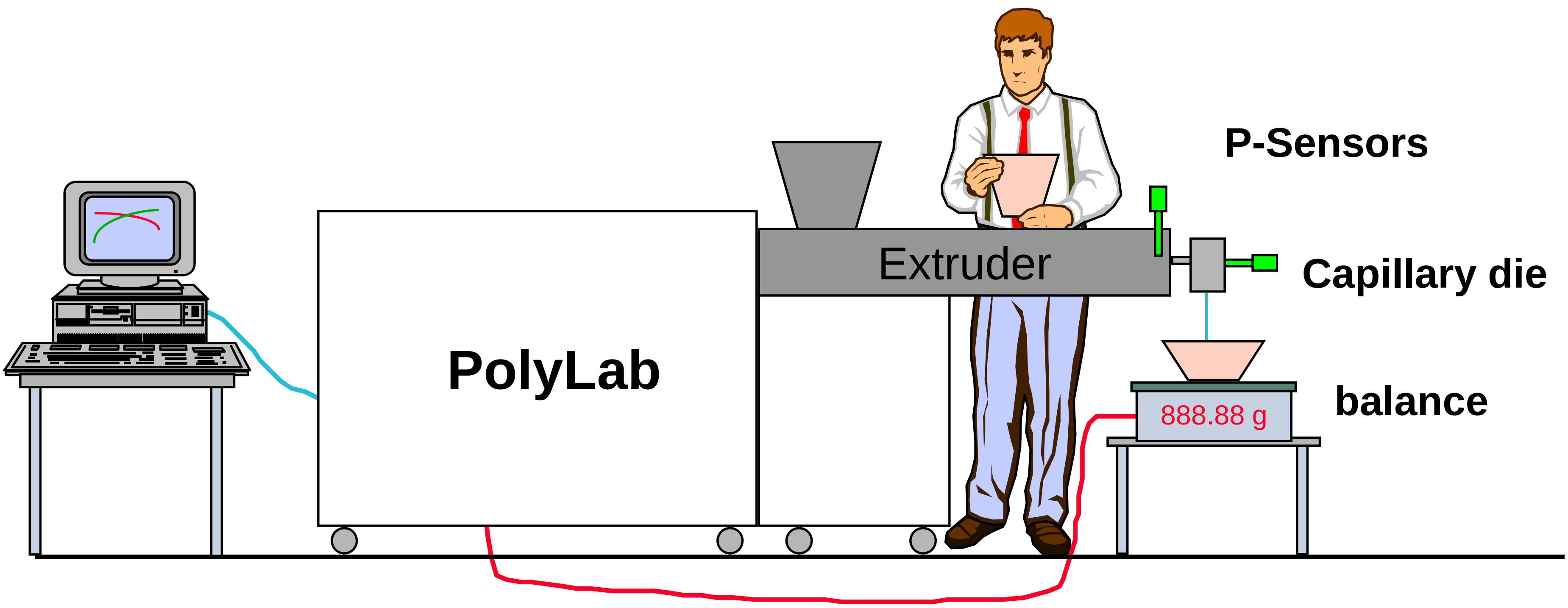
Cutting and manual entry (external balance)



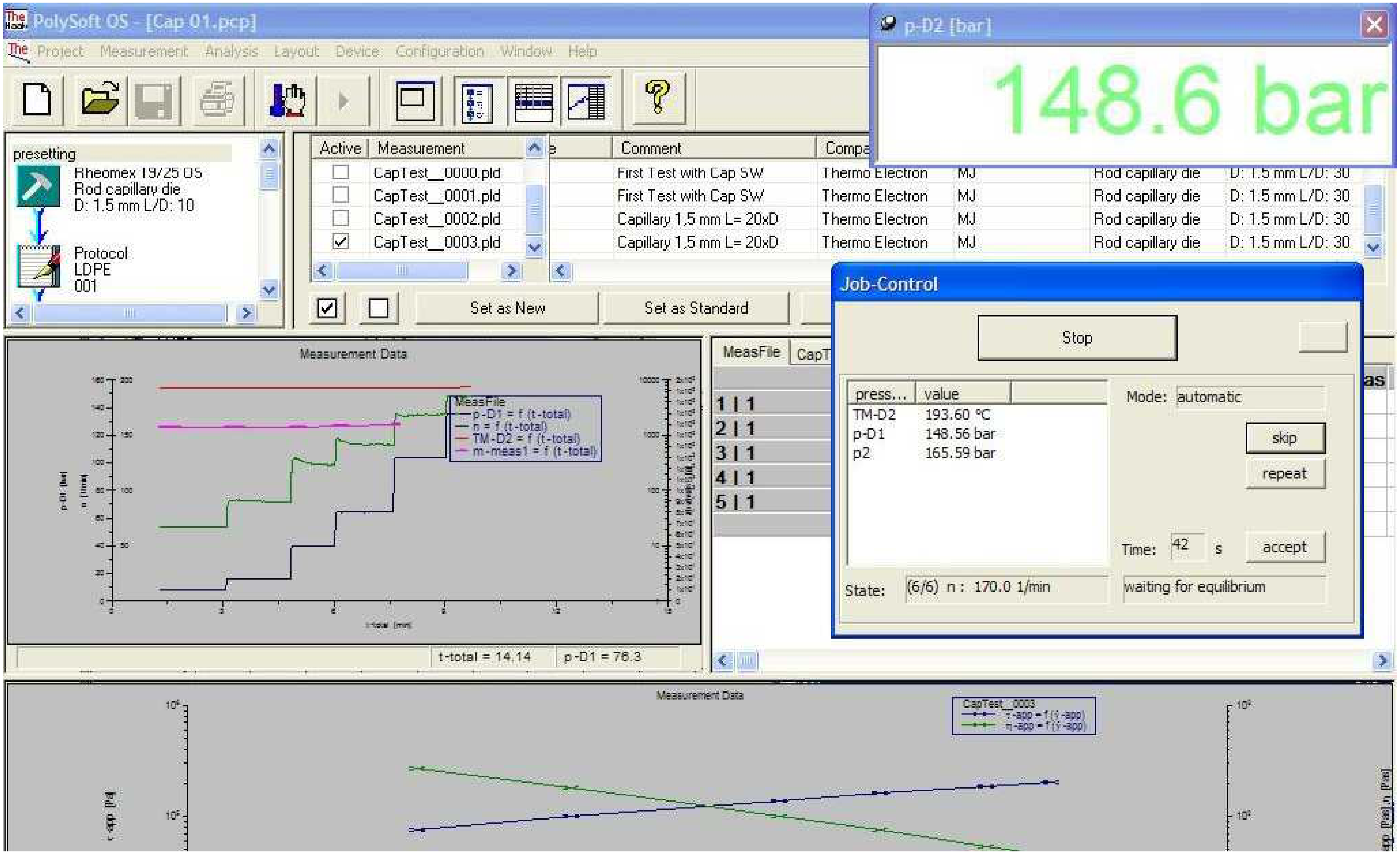
PolySoft Software – Measurement Modes

Automatic measurement

The software checks for the constant pressure reading and starts the measurement at each speed step automatically



PolyLab OS – PolySoft Job Controller



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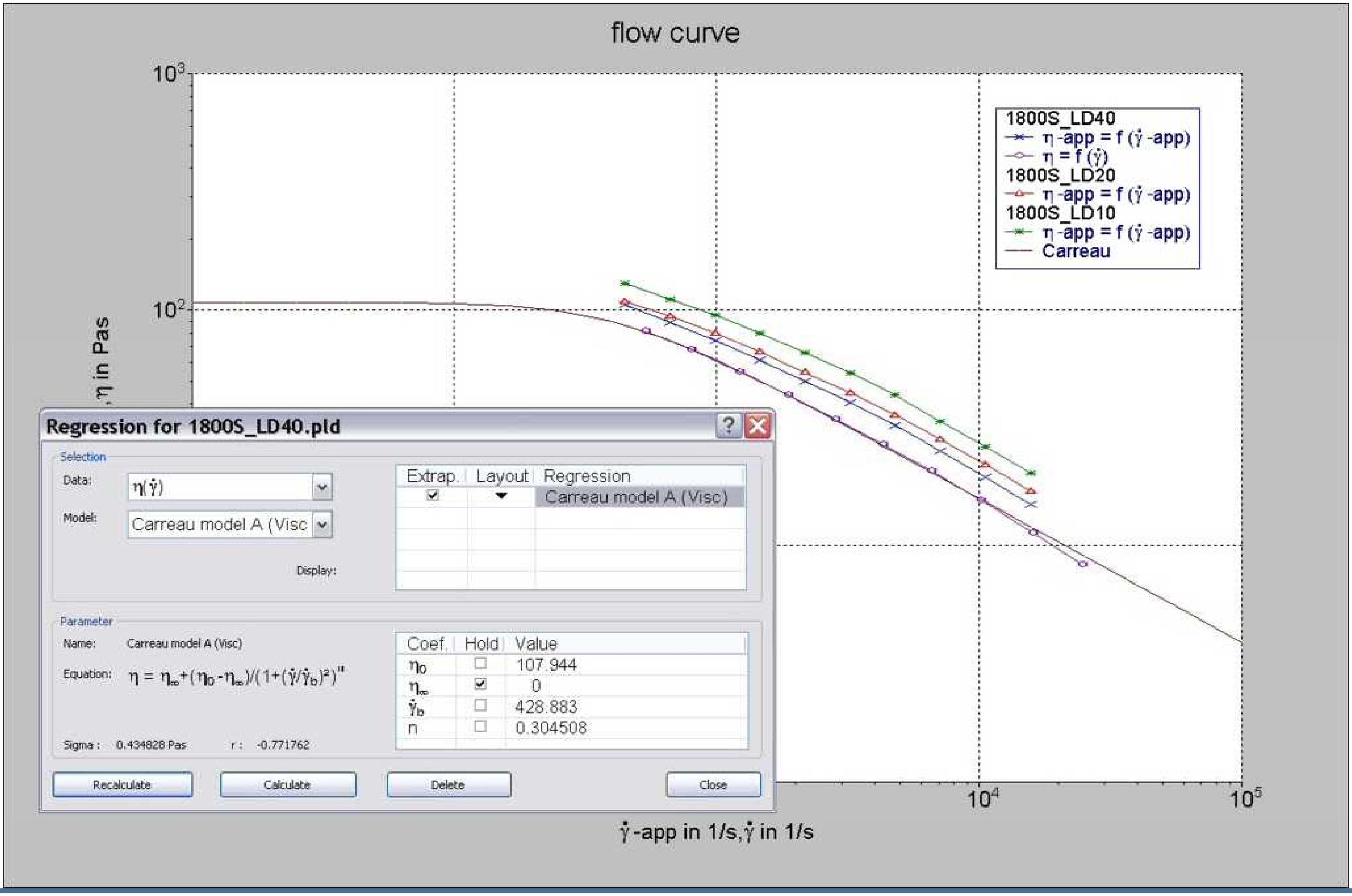
PolyLab OS – PolySoft Regression



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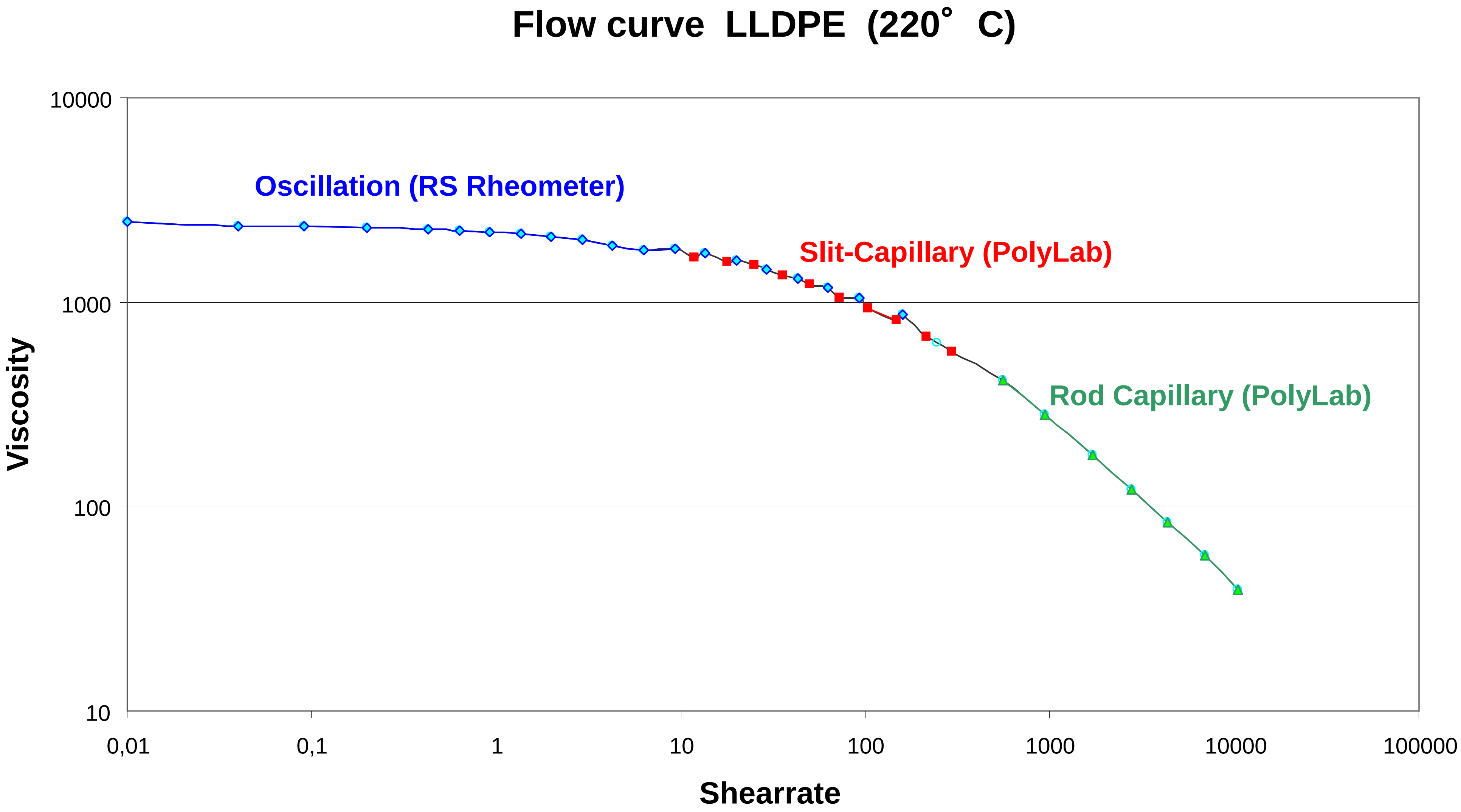
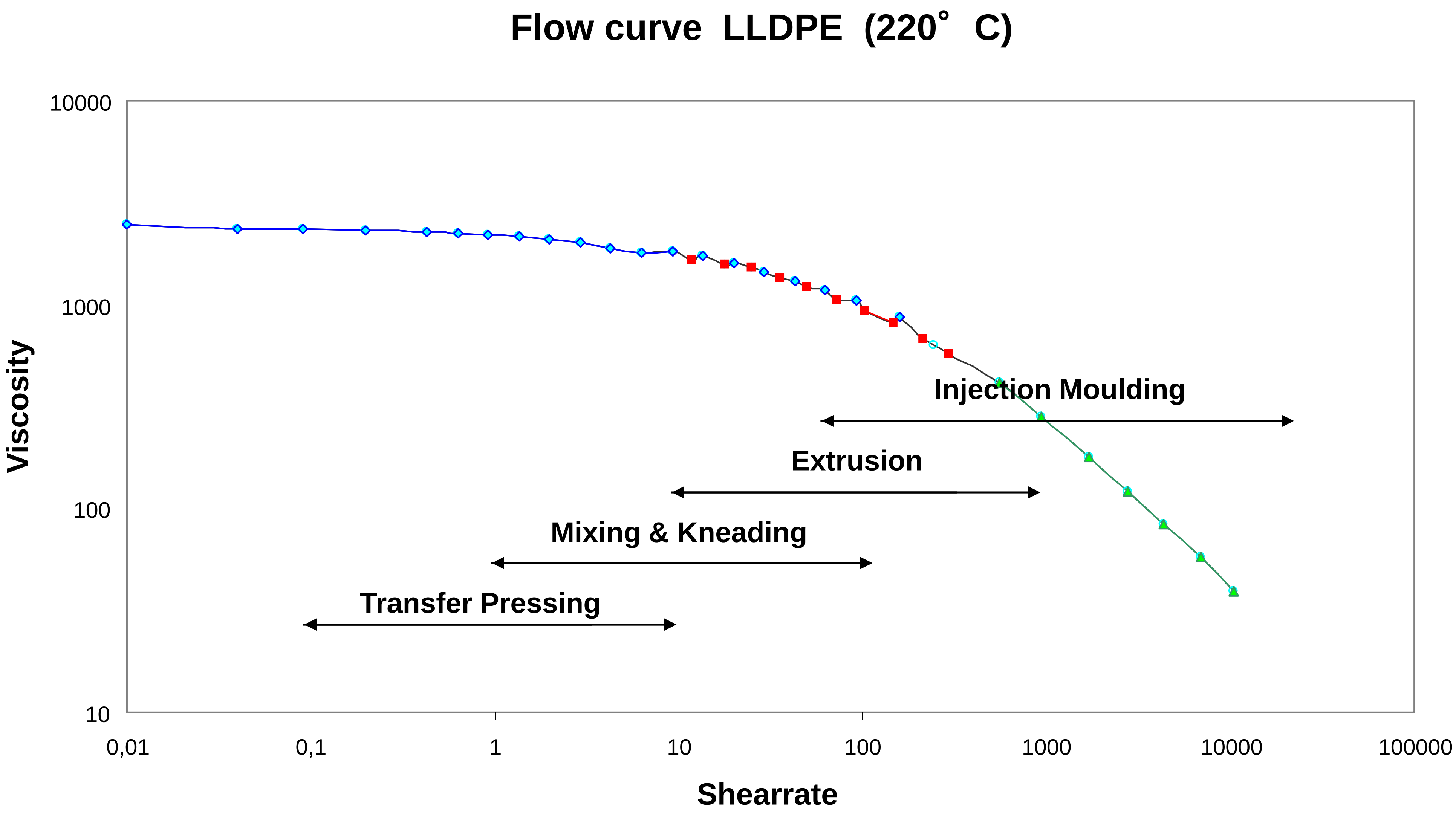
The whole Rheology



Relevant shear rates for some technical processes



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Questions ???