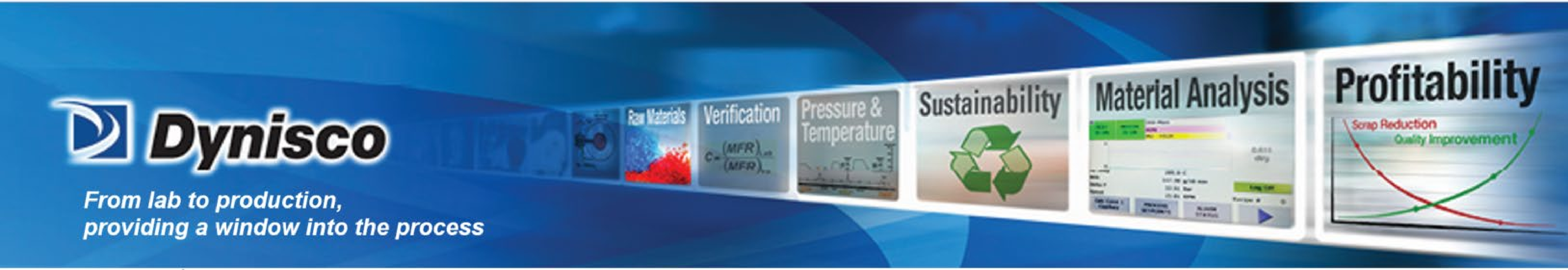


Practical Rheology
LCR 7001 Capillary Rheometer

Azadeh Farahanchi
Rheological Scientist

38 Forge Parkway, Franklin, MA 02038

Tel: 508.541.9582

Cell: 774.287.2366

Azadeh.Farahanchi@dynisco.com

Dynisco

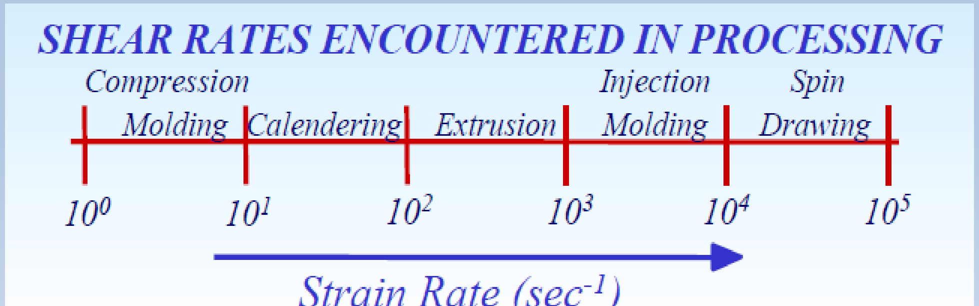
Outline

1. Introduction
2. Shear Sweep Test (Polymer Flow Behavior)
3. Rabinowicz and Bagley Corrections
4. Extensional Viscosity Measurements
5. Wall Slip Velocity Calculation
6. Time Sweep Test (Thermal Stability of Polymers)
7. Die Swell Measurements
8. MFR Correlation
9. Intrinsic Viscosity of PET
10. LCR Dies Information

Introduction

Why Capillary Rheometer?

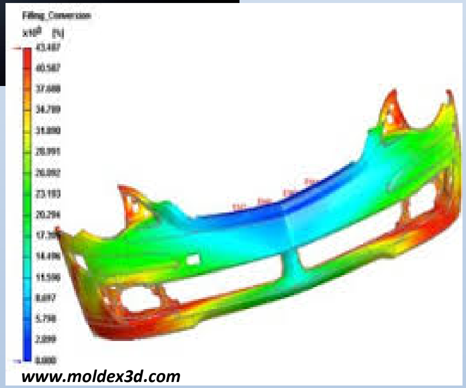
- ❖ The most common melt rheometer to analyze flow behavior of polymers under processing condition (shear rate, time, temperature).
- ❖ Duplication of processing parameters for design, simulation, and trouble shooting purposes in a faster way.
- ❖ Predict optimal operating condition based on correlation of rheological data from capillary rheometer to processing parameters.
- ❖ Analyze different materials for various applications and design.



http://slideplayer.com

www.directindustry.com/industrial-manufacturer/pp-extrusion-line

www.polymersni.com/industry/extrusion-film



www.moldex3d.com

APPLICATIONS

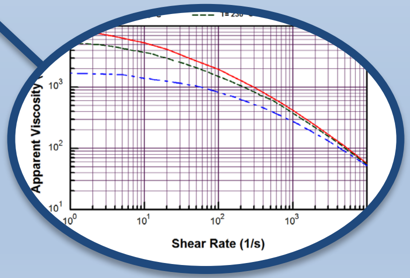
- ✓ Polymer viscosity at wide deformation rate range
- ✓ Polymer melt flow behavior
- ✓ Shear thinning behavior
- ✓ Polymer stability over time/temperature
- ✓ Elastic properties (die swell, wall slip)

Dynisco®LCR 7001

Force measurements
from **Load Cell**

Pressure measurement
from **Pressure Transducer**

RESULTS:
Polymer flow curve
(LabKars Software)



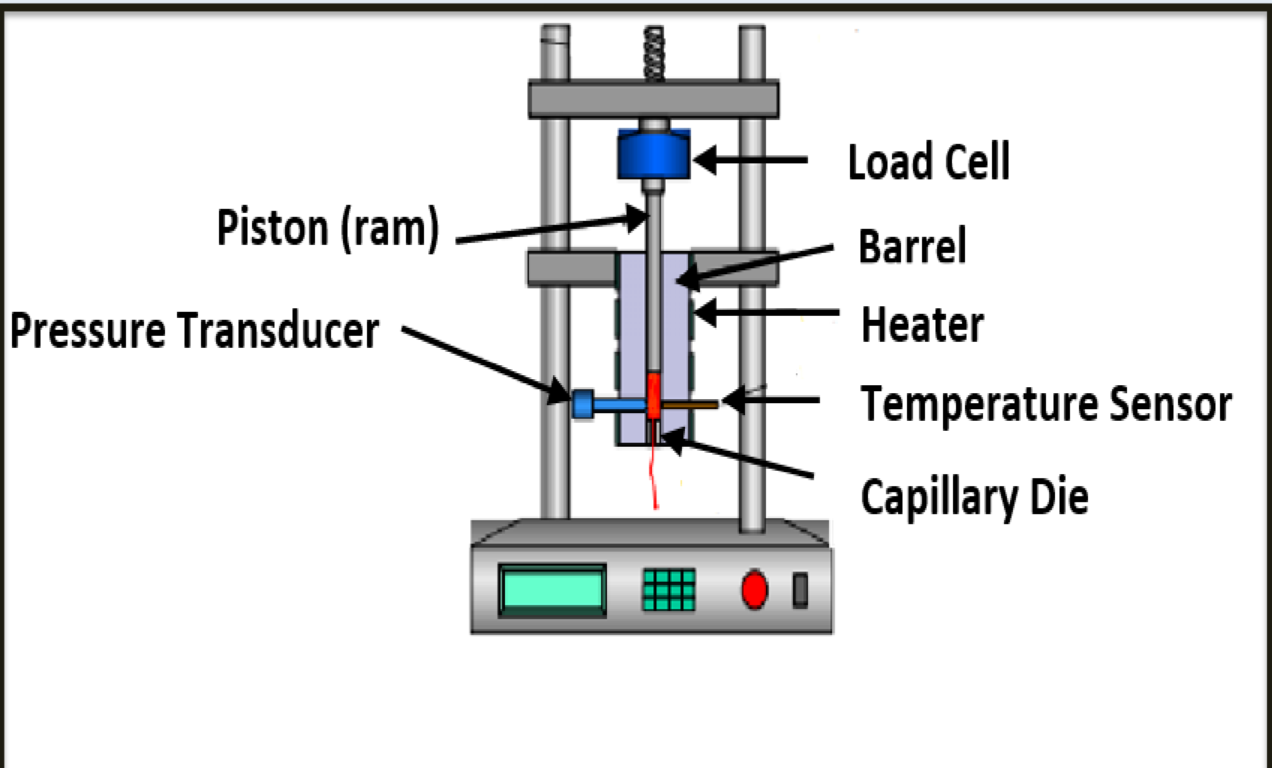
❖ **MEASURES:**

- ✓ Force
- ✓ Pressure
- ✓ Ram rate
- ✓ Time
- ✓ Temperature
- ✓ Expansion of extrudate

❖ **CALCULATES:**

- ✓ Shear stress
- ✓ Shear rate
- ✓ Shear/extensional Viscosity
- ✓ Die swell ratio

General Specification

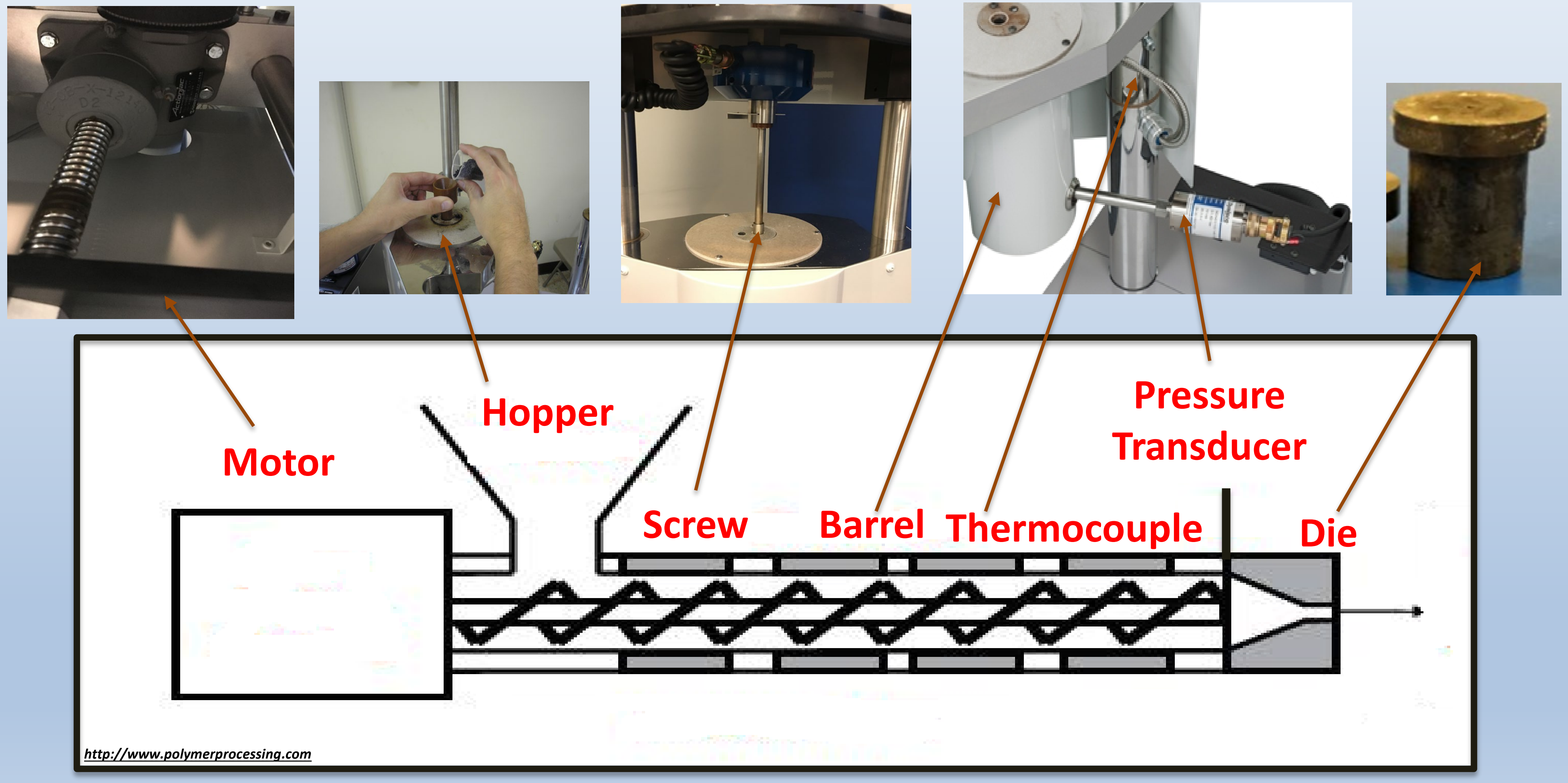


Based on **ASTM-D3835** *"Standard Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer"*

❖ **LCR7001 SPECIFICATION**

- ✓ Temp range: 25-500 °C
- ✓ Shear rate range: 1-100000 1/s
- ✓ Barrel diameter: 9.55 mm
- ✓ Available length: 227 mm
- ✓ Working length: 125 mm
- ✓ Min piston speed: 0.03 mm/min
- ✓ Max piston speed: 650 mm/min
- ✓ Max force measurement from load cell: 10 KN
- ✓ Max pressure measurements: 1400 Bar
- ✓ Accuracy of test-to-test~1.5-2%
- ✓ Accuracy of rheometer-to-rheometer~8%

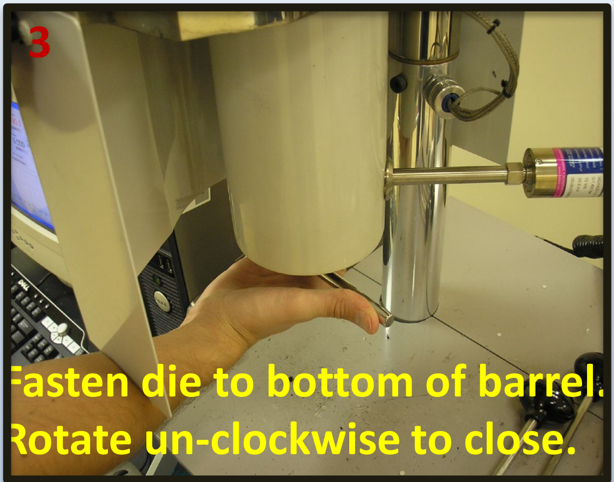
Extrusion & Capillary Rheometer



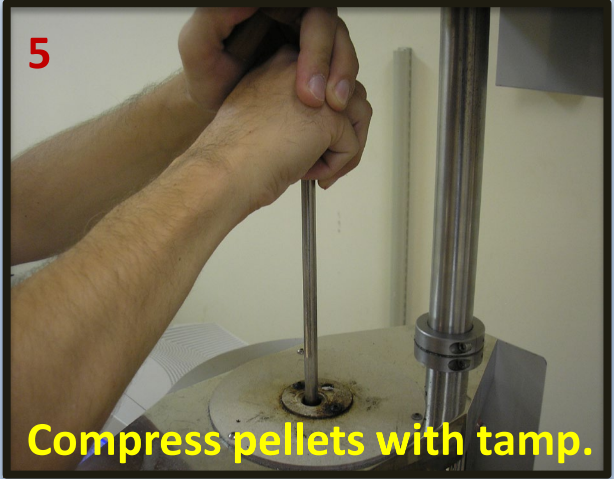
LCR Preparation Before Running the Test


1 Swing out barrel.

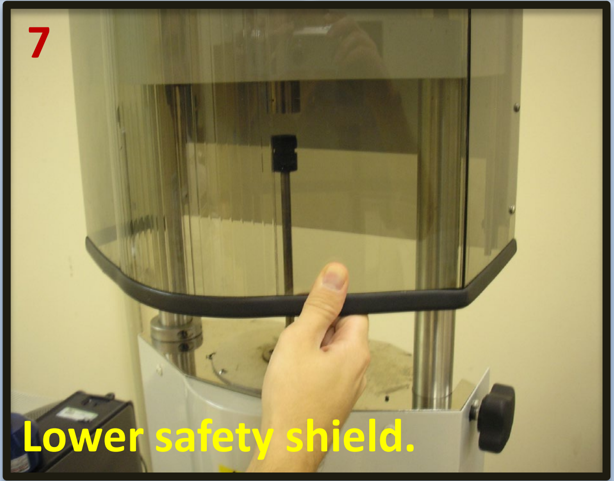

2 Insert the die in the die holder and die fastener.


3 Fasten die to bottom of barrel. Rotate un-clockwise to close.

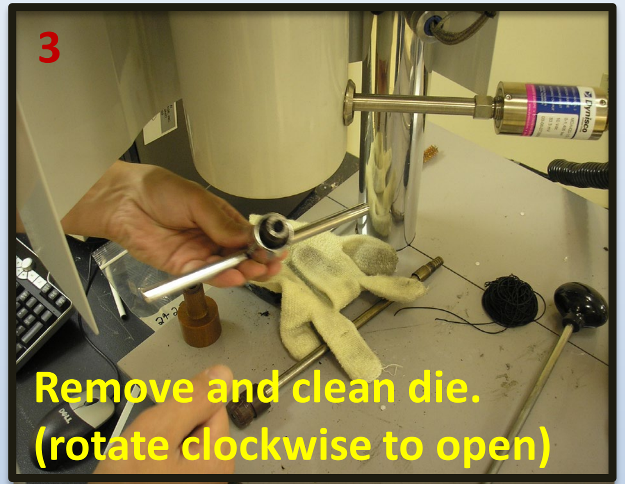

4 Pour sample into funnel – about 10 grams.

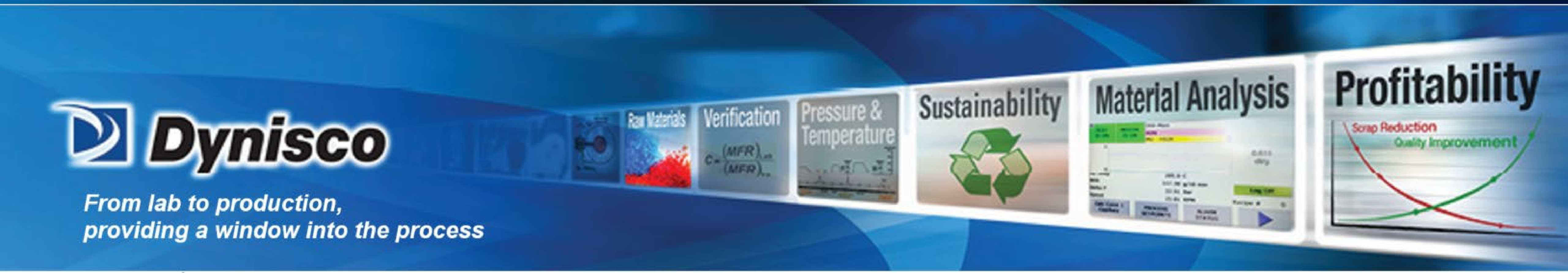

5 Compress pellets with tamp.


6 Place the piston inside barrel and lock it.


7 Lower safety shield.

LCR Cleaning After the Test

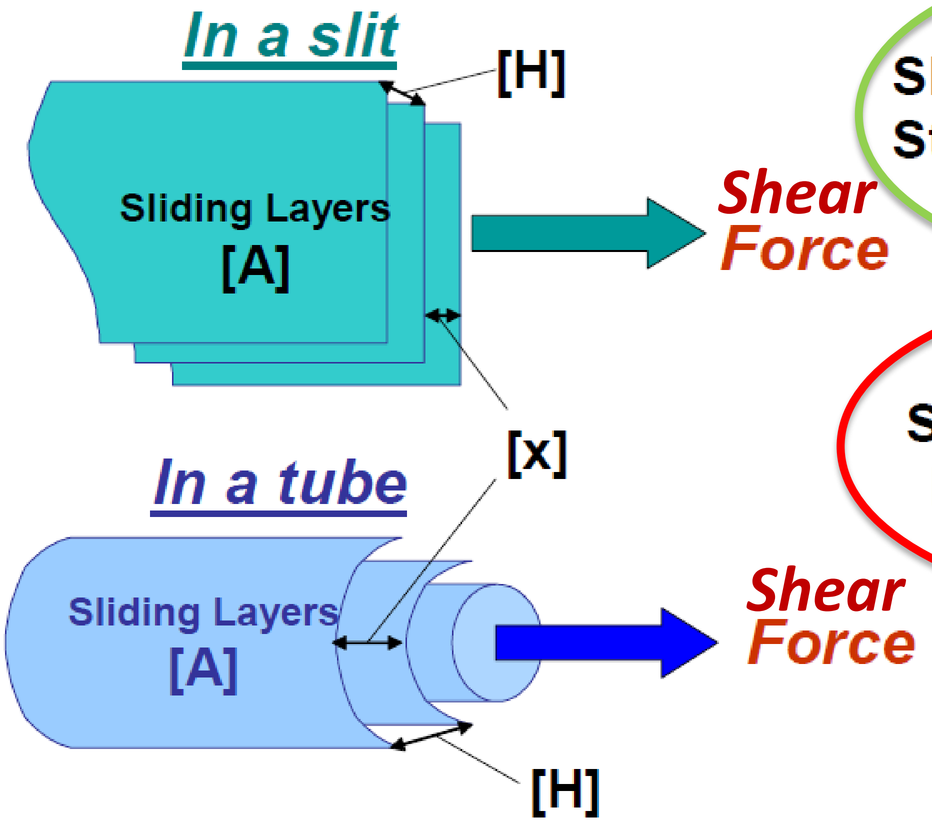




Shear Sweep Test

(Polymer Flow Behavior)

What is Shear?



Shear Stress $= \tau = F / A$ $(Pa \ or N/m^2)$

Shear Rate $= \dot{\gamma} = \frac{\Delta x / \Delta t}{H}$ $(1/S)$

Where:

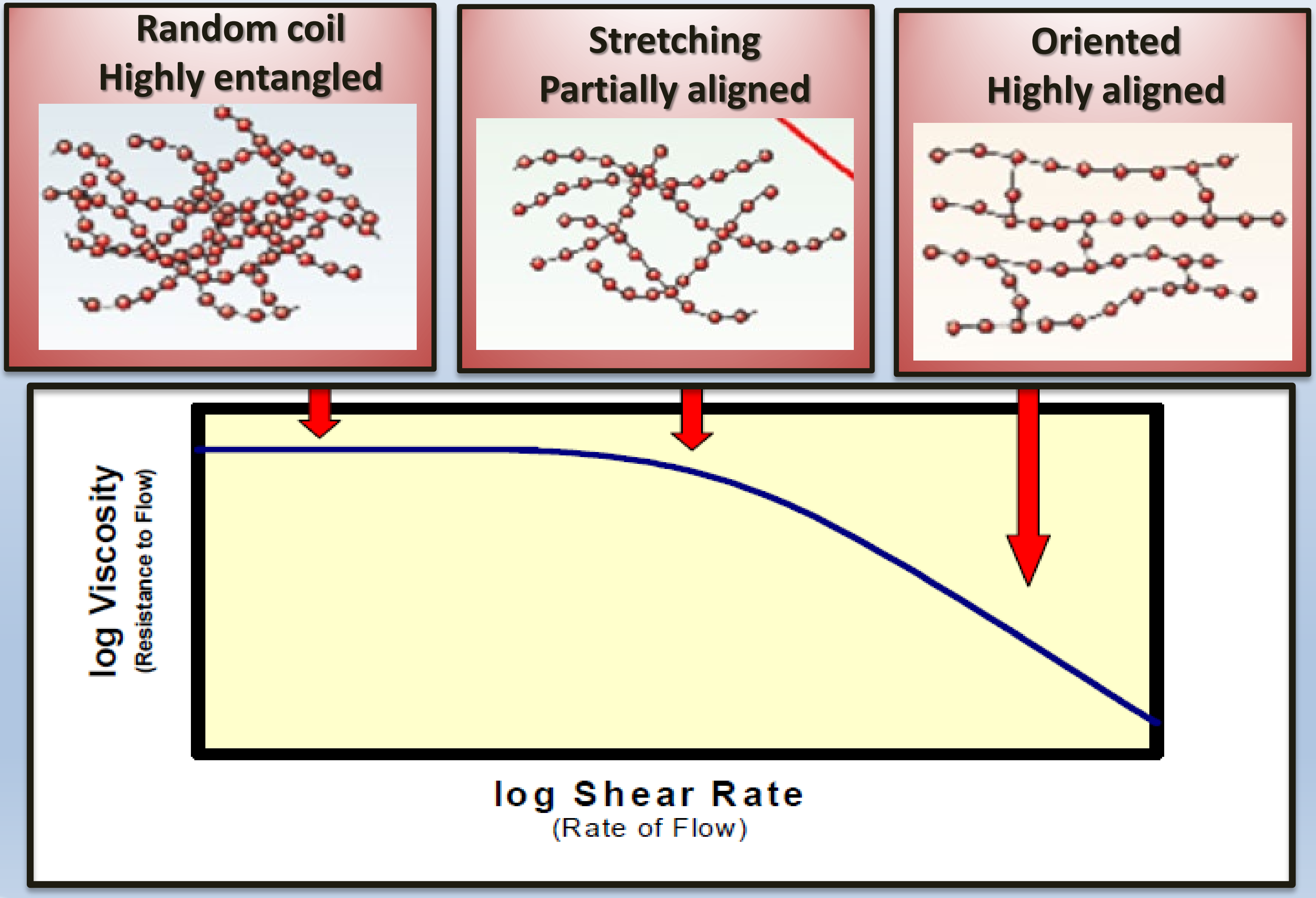
F = Force
A = Area of layer
H = Gap between layers
x = Displacement of layer
T = Time

Calculation of Rheological Data in LCR

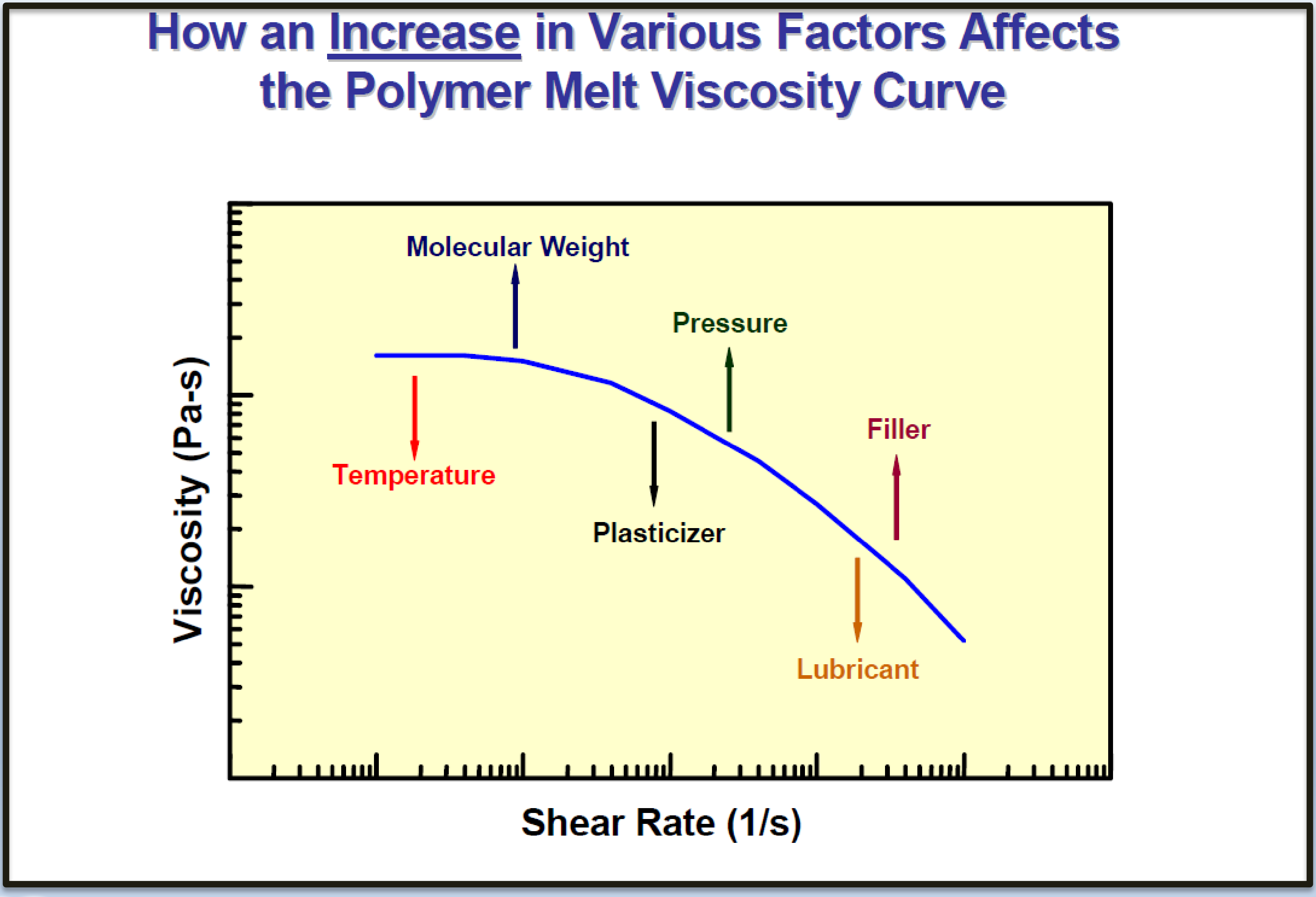
Apparent shear rate (based on piston speed)	❖ $\dot{\gamma}_a = \frac{4Q}{\pi R_c^3}$ ❖ $Q = S(\frac{\pi D_b^2}{4})$	➢ ***$\dot{\gamma}_a$ (1/s):*** apparent shear rate ➢ ***Q (mm³/sec):*** volumetric flow rate ➢ ***S (mm/min):*** piston speed ➢ ***D_b (mm):*** barrel diameter ➢ ***R_c (mm):*** die radios
Wall shear stress (based on force or driving pressure)	❖ $\tau_w = \frac{F/\pi D_b^2}{4(L/D)die}$ ❖ $\tau_w = \frac{P}{4(L/D)die}$	➢ ***τ_w (Pa):*** wall shear stress ➢ ***F (N):*** force from "load cell" on piston ➢ ***D_b(mm):*** barrel diameter ➢ ***L/D:*** length to diameter ratio of the die ➢ ***P_d (Pa):*** driving pressure at the die entrance from "pressure transducer"
Apparent shear viscosity	❖ $\eta_a = \frac{\tau_w}{\dot{\gamma}_a}$	➢ ***η_a (Pa-s):*** apparent shear viscosity ➢ ***τ_w (Pa):*** wall shear stress ➢ ***$\dot{\gamma}_a$(1/s):*** apparent shear rate

➢ **Assumptions: 1:** Fully developed, isothermal, steady state, and Laminar Flow - **2:** No radial or circumferential velocity components - **3:** Incompressible fluid - **4:** No slip at the wall of the die

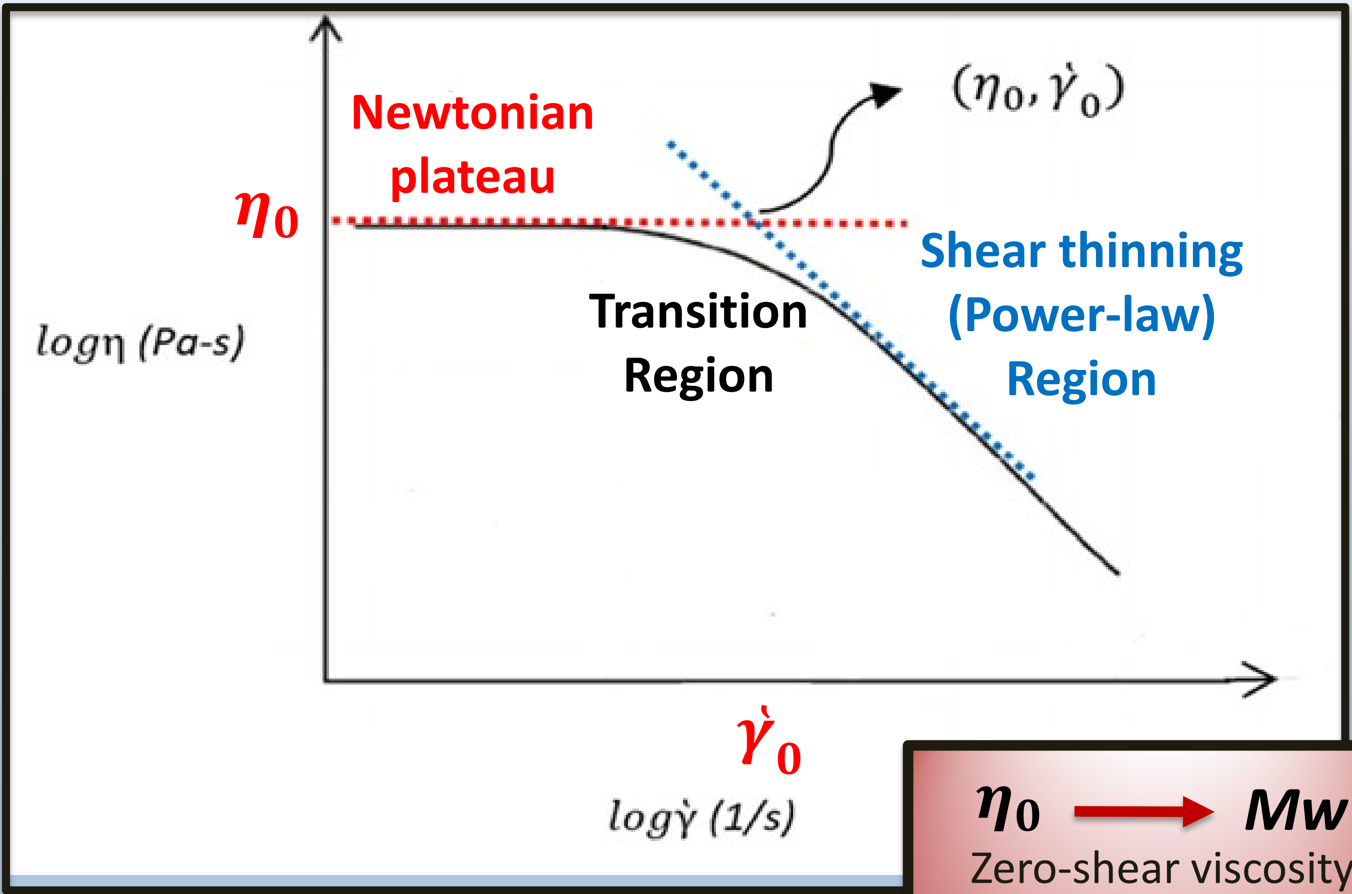
Shear Flow in Viscoelastic Polymer Melts



Effect of Various Factors on Polymer Flow Curve



Flow Curve of Polymer Melts

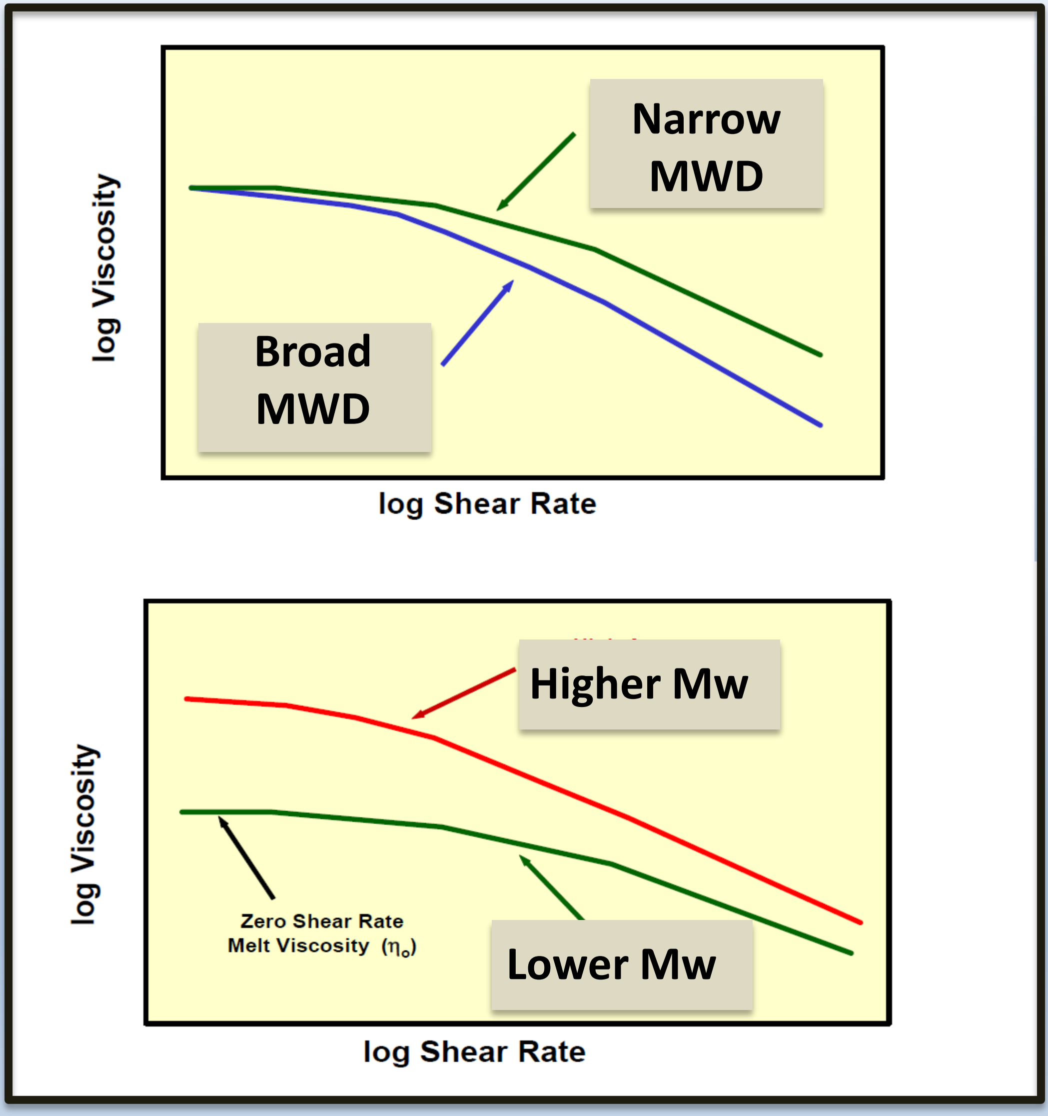


$\eta_0 \longrightarrow$ ***Mw***

Zero-shear viscosity

$\dot{\gamma}_0 \longrightarrow$ ***MWD***

Characteristic shear rate

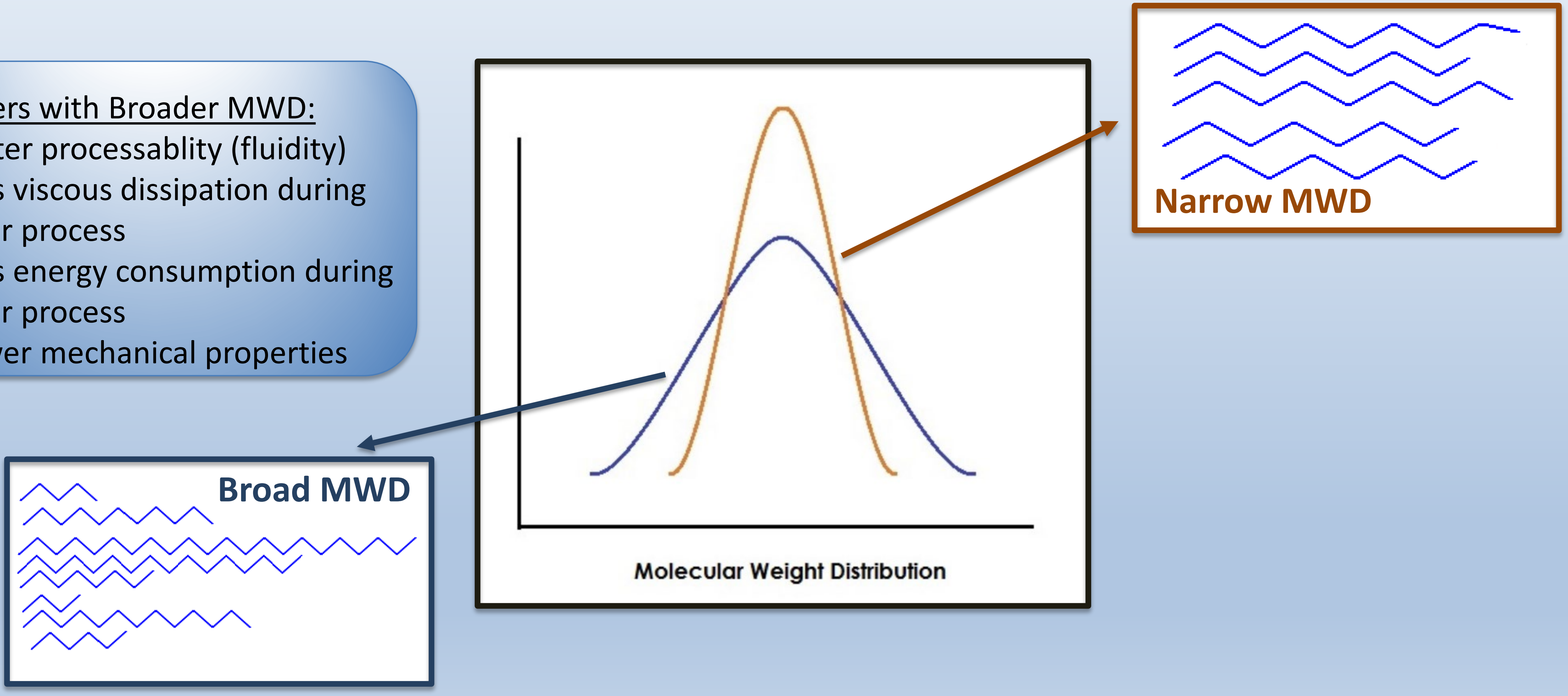


Dynisco

Molecular Weight Distribution (MWD)

Polymers with Broader MWD:

- ➢ Better processablity (fluidity)
- ➢ Less viscous dissipation during their process
- ➢ Less energy consumption during their process
- ➢ Lower mechanical properties



Analyzing Polymer Degradation from Flow Curve

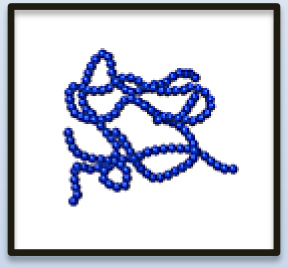
Extrusion Condition

Heat
- ➢ Barrel temp

Mechanical shear
- ➢ Screw speed

Residence time
- ➢ Flow rate

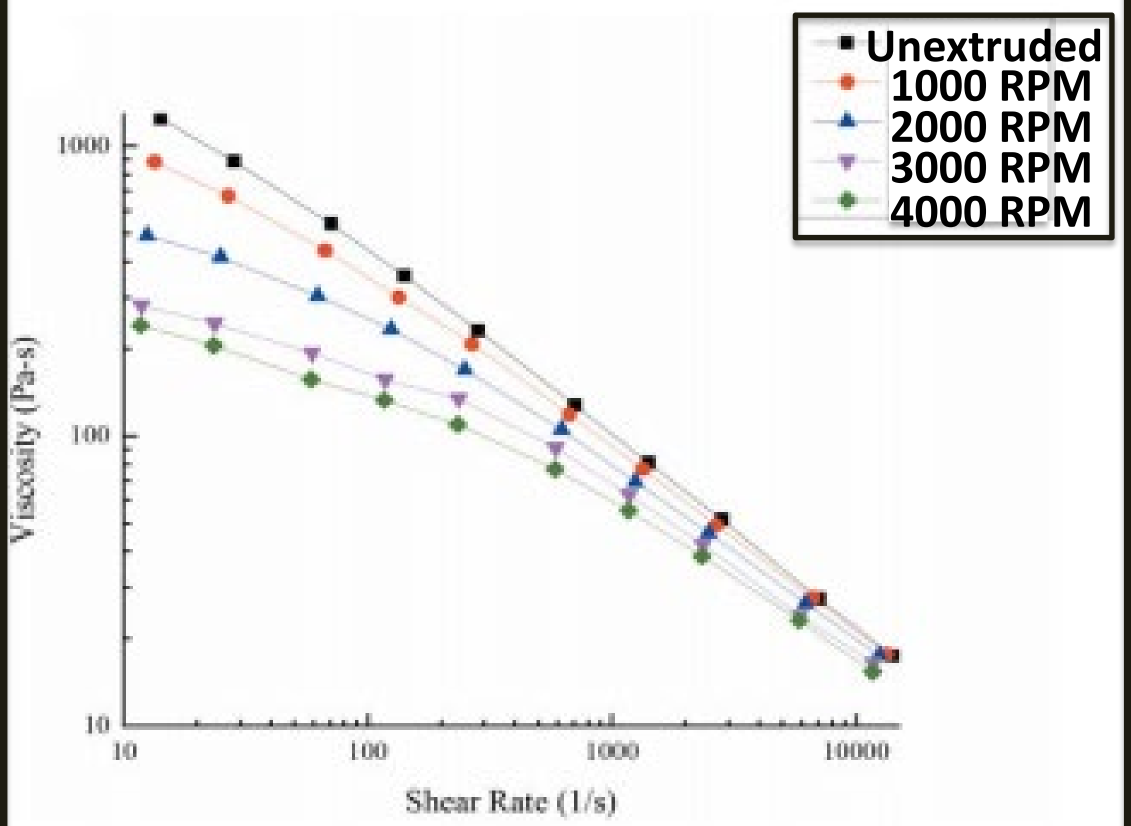
Higher η_0 and Mw



Lower η_0 and Mw

Degradation

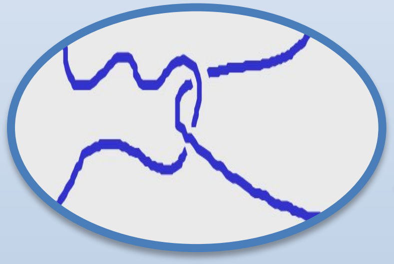
Flow curve of plastic samples before process and after process at various screw speeds in an extrusion

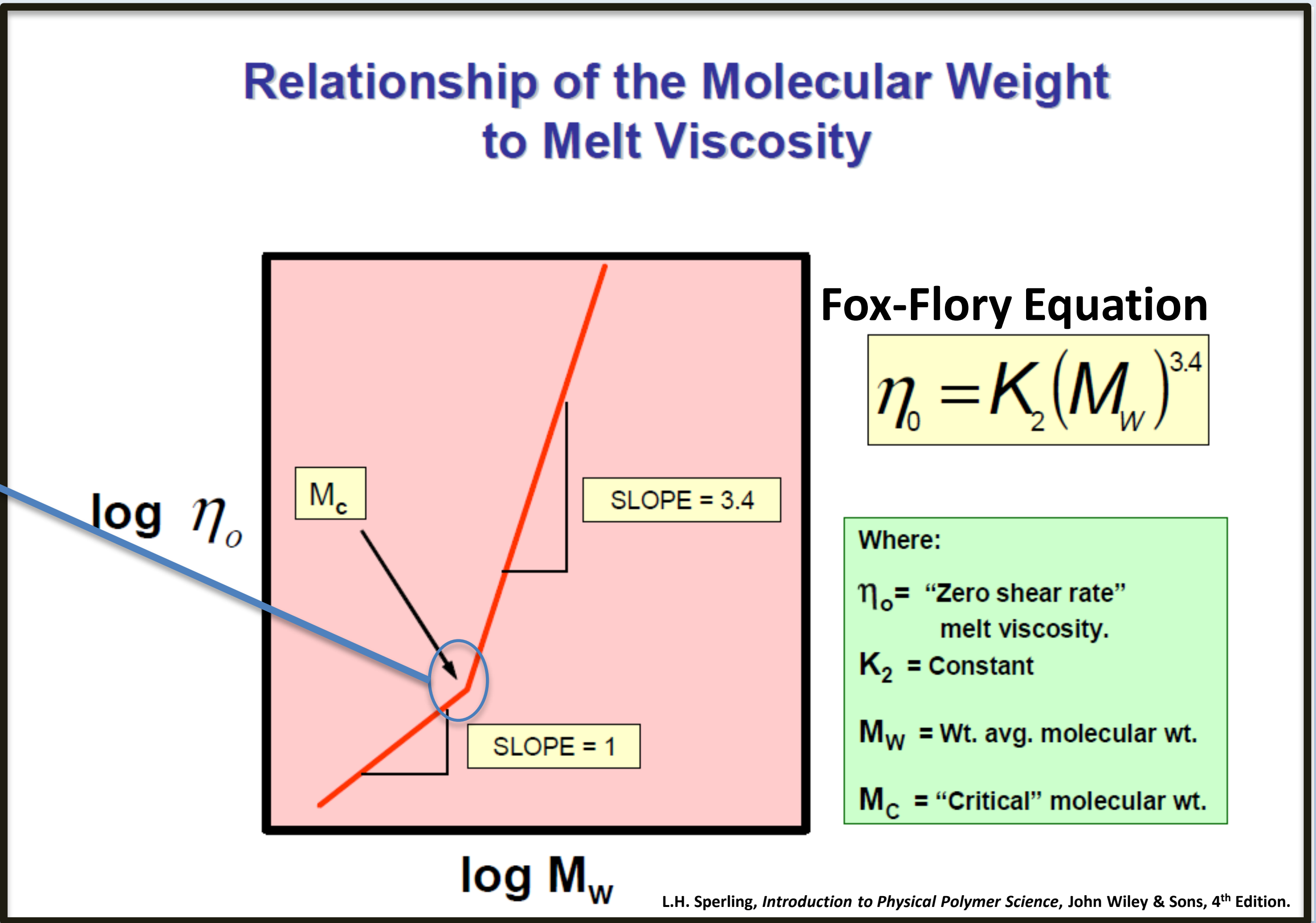


Farahanchi, A., Malloy, R., Sobkowicz, M.J. (2016), *Polymer Engineering & Science* 56: pp. 743-751

Mw Dependence of Zero-Shear Viscosity

Critical Molecular wt. (M_C)
molecular entanglements occur

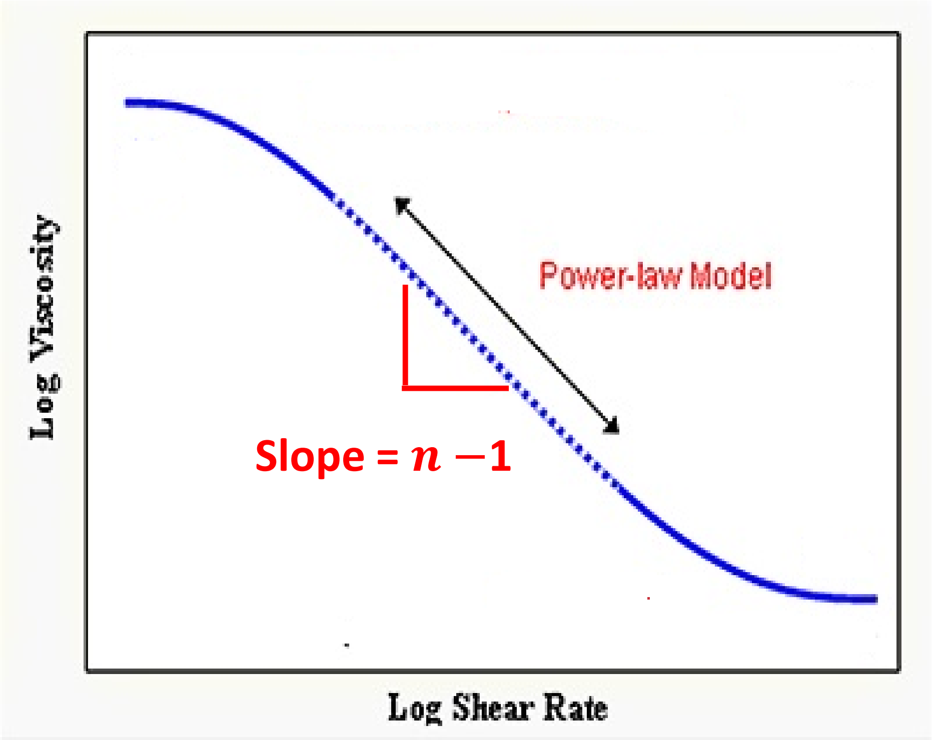




Polymers with higher Mw:
- ✓ Higher intermolecular entanglement
- ✓ Higher strength
- ✓ Higher chemical resistance

Dynisco

Quantitative Relationships for the Dependence of Viscosity upon Shear Rate



Power-law Model

$$\eta(\dot{\gamma}) = k\dot{\gamma}^{n-1}$$

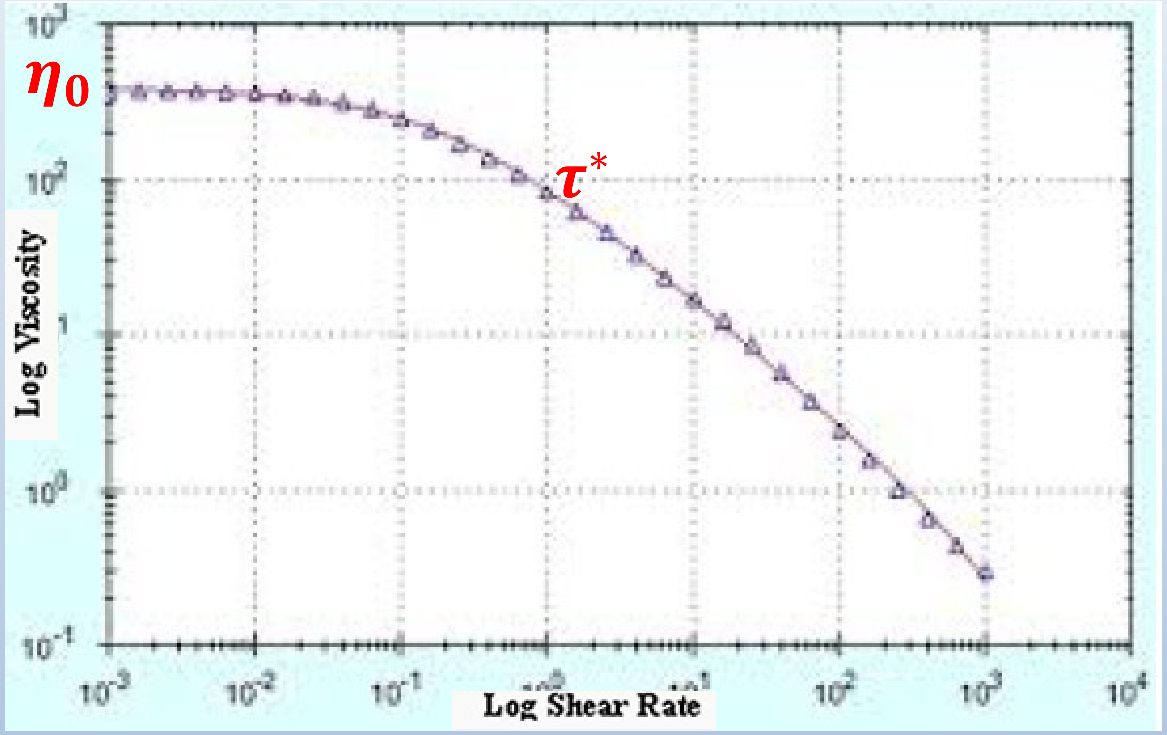
- k (Pa-s): Consistency
- n: Power-law index

- ❖ "Only" fits the shear-thinning (power-law) portion of the curve.
- ❖ Shear-thinning exponents dependent on intermolecular forces.
- ❖ n ranges from 0.2-0.9 depending upon the type of polymer.
- ❖ n equals to 1 for Newtonian materials.
- ❖ n represents the processability (shear-thinning intensity).
- ❖ Polymers with lower n are more sensitive to the shear rate.
- ❖ n decrease with broader MWD.
- ❖ k has temperature dependency and controlled by Mw.

Dynisco

J.M. Dealy, K.F. Wissbrun, *Melt Rheology and its Role in Plastics Processing: Theory and Applications*, Van Nostrand Reinhold, New York (1990).

Quantitative Relationships for the Dependence of Viscosity upon Shear Rate



Modified Cross Model

$$\eta(\dot{\gamma}) = \frac{\eta_0}{1 + \left(\frac{\eta_0 \dot{\gamma}}{\tau^*}\right)^{1-n}}$$

- η_0 (Pa-s): Zero-shear viscosity
- τ^* (Pa): Critical shear stress
- n: Power-law index

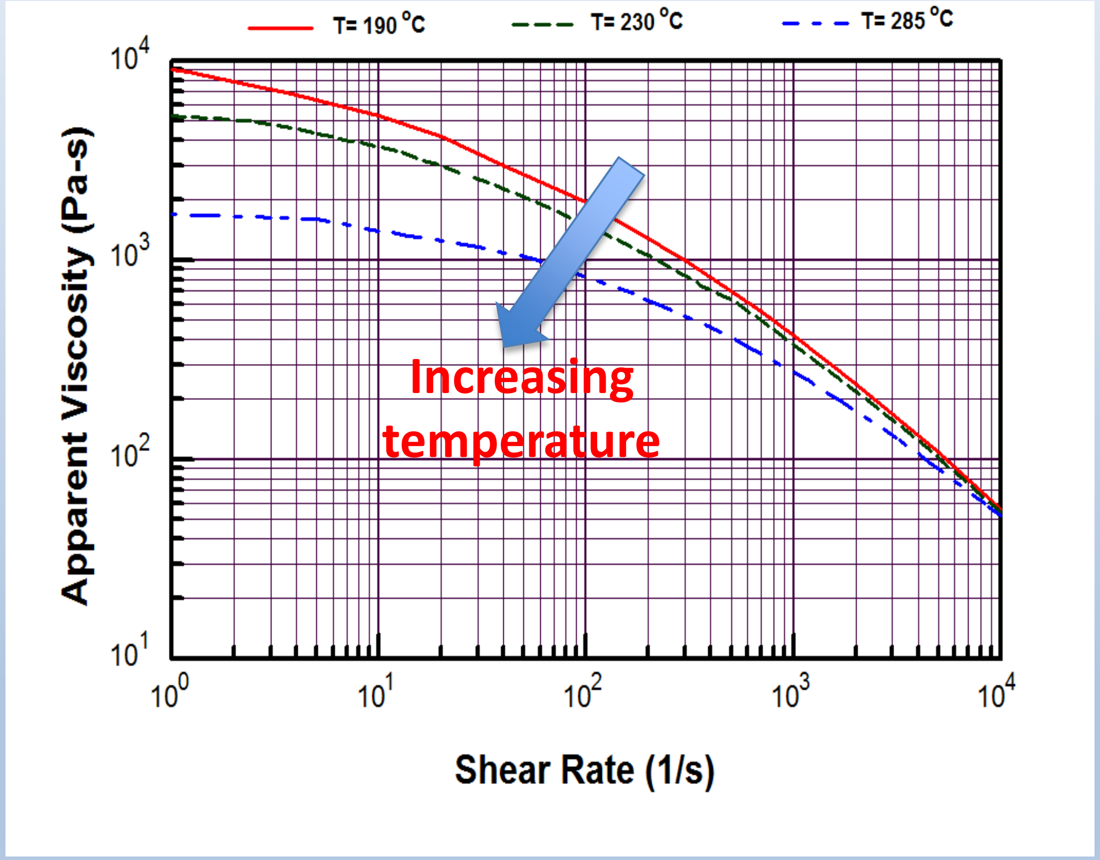
- ❖ Combines the power-law and Newtonian regions.
- ❖ Also fits the low shear Newtonian plateau.
- ❖ η_0 is controlled by molecular weight.
- ❖ τ^* is stress at the beak in curve and controlled by MWD.
- ❖ Broader MWD (by blending or branching) cause earlier τ^*.

Dynisco

J.M. Dealy, K.F. Wissbrun, *Melt Rheology and its Role in Plastics Processing: Theory and Applications*, Van Nostrand Reinhold, New York (1990).

Viscosity-Temperature Dependence

Window into the process



- Williams-Landel-Ferry (WLF) model: $\boldsymbol{(T_g < T < T_g + 100)}$

$$\log(a_T) = \frac{\eta_0(T)}{\eta_0(T_{ref})} = [\frac{-C_1(T - T_g)}{C_2 + T - T_g}]$$

$$C_1 = 17.44$$
$$C_2 = 51.6\ K$$

- Arrhenius model: $\boldsymbol{(T > T_g + 100)}$

$$a_T = \frac{\eta_0(T)}{\eta_0(T_{ref})} = \exp[\frac{E_a}{R}\left(\frac{1}{T} - \frac{1}{T_{ref}}\right)]$$

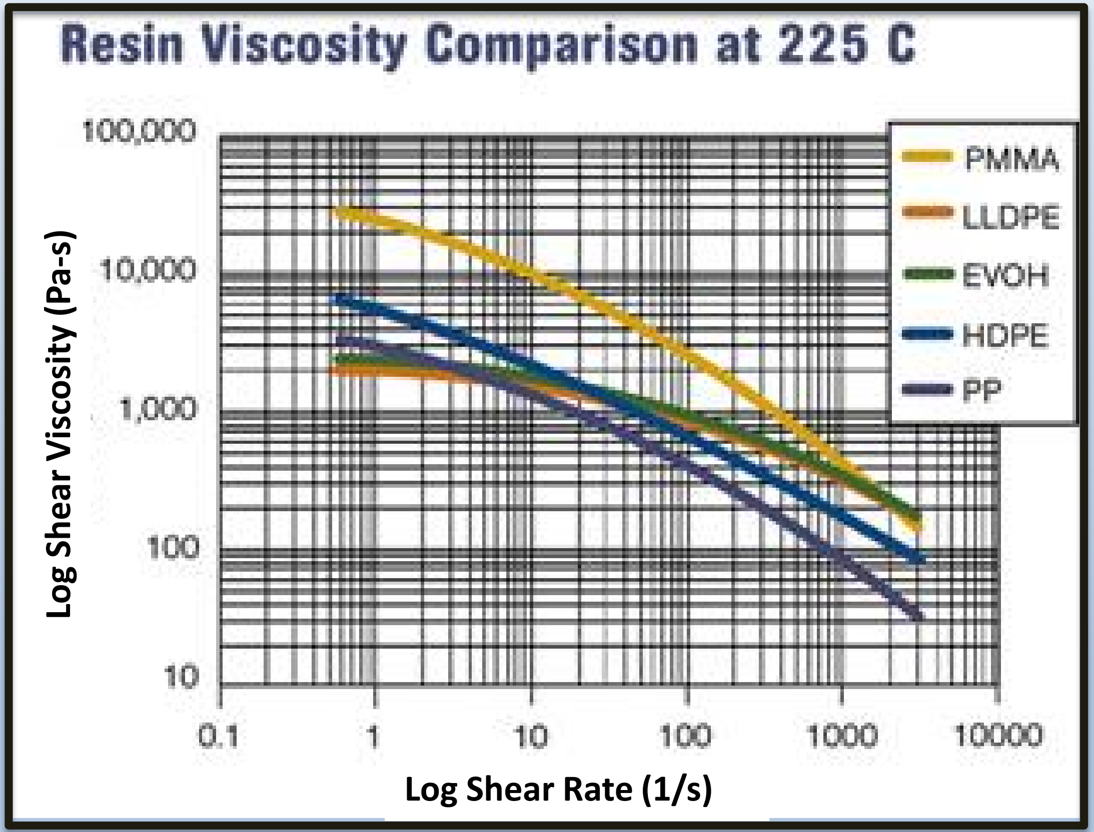
$E_a: Activation\ Energy$

$R: Universal\ gas\ constant = 8.314 \times 10^{-3}\ {}^{kJ}/_{mol.K}$

Dynisco

J.M. Dealy, K.F. Wissbrun, *Melt Rheology and its Role in Plastics Processing: Theory and Applications*, Van Nostrand Reinhold, New York (1990).

A Window into Your Process!

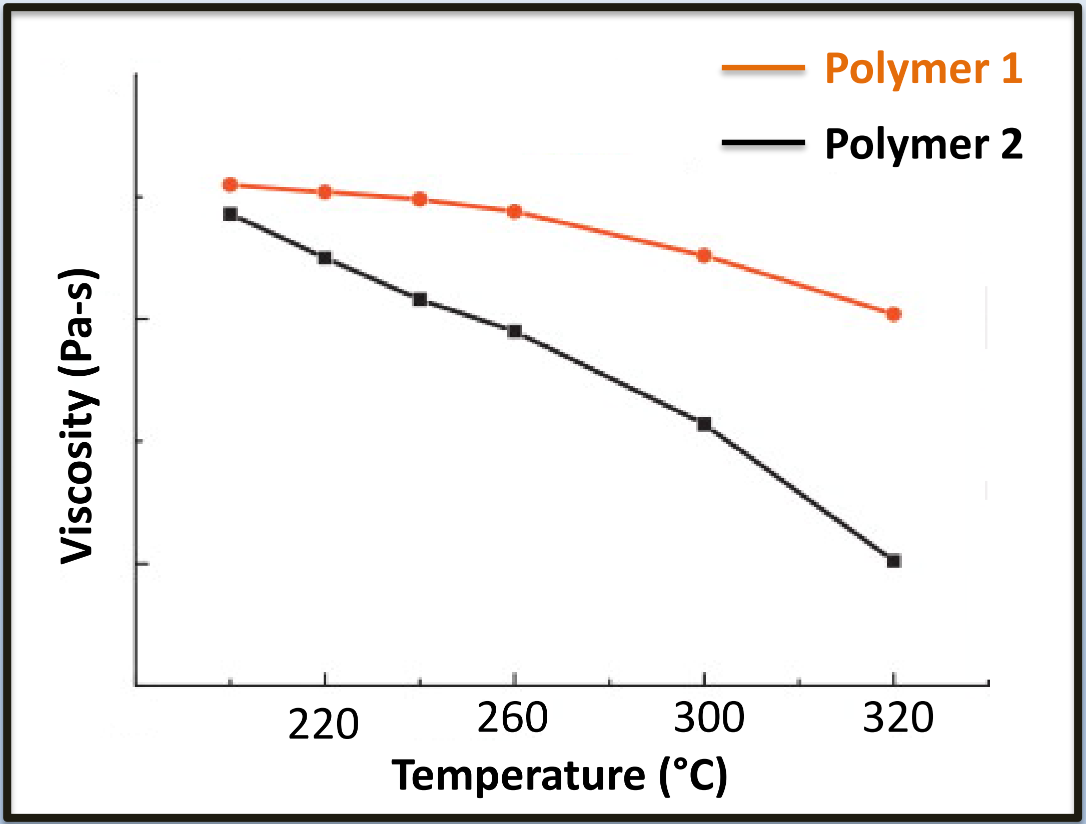


https://www.ptonline.com/columns/how-to-spec-a-flat-die

- At shear rate 100-1000 1/s (common at normal RPM in extrusion), the processing of which polymer cause higher torque and head pressure, and viscous heating in the extrusion?
- Which polymer has higher shear-thinning behavior (easier process-ability)?
- With increasing screw speed which polymer will have the highest flow rate?
- Which polymer has the highest molecular weight?
- Which polymer had the Broader MWD?
- Which polymer might have linear might have long chain-branching and which one might have linear structure?

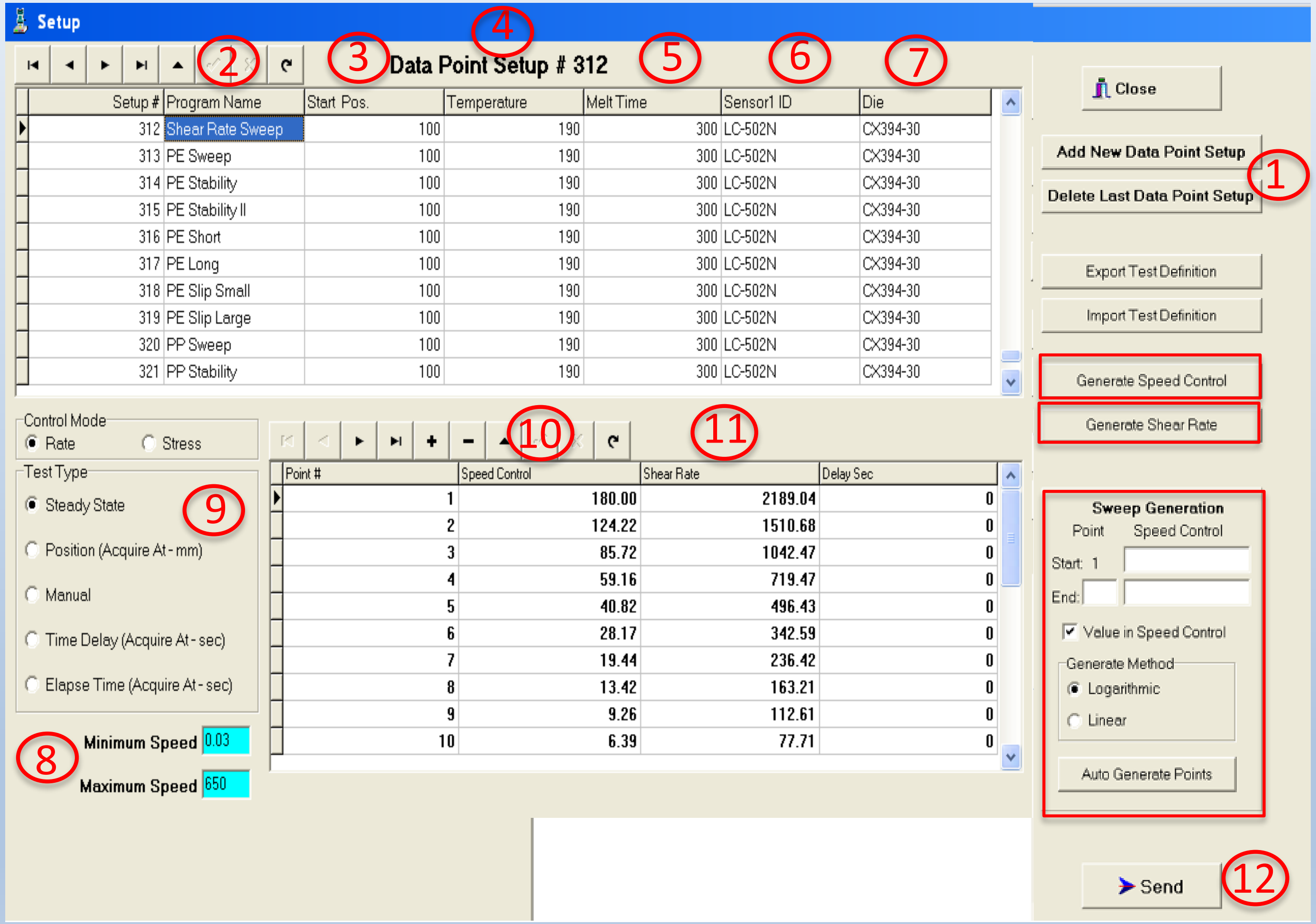
Dynisco

A Window into Your Process!



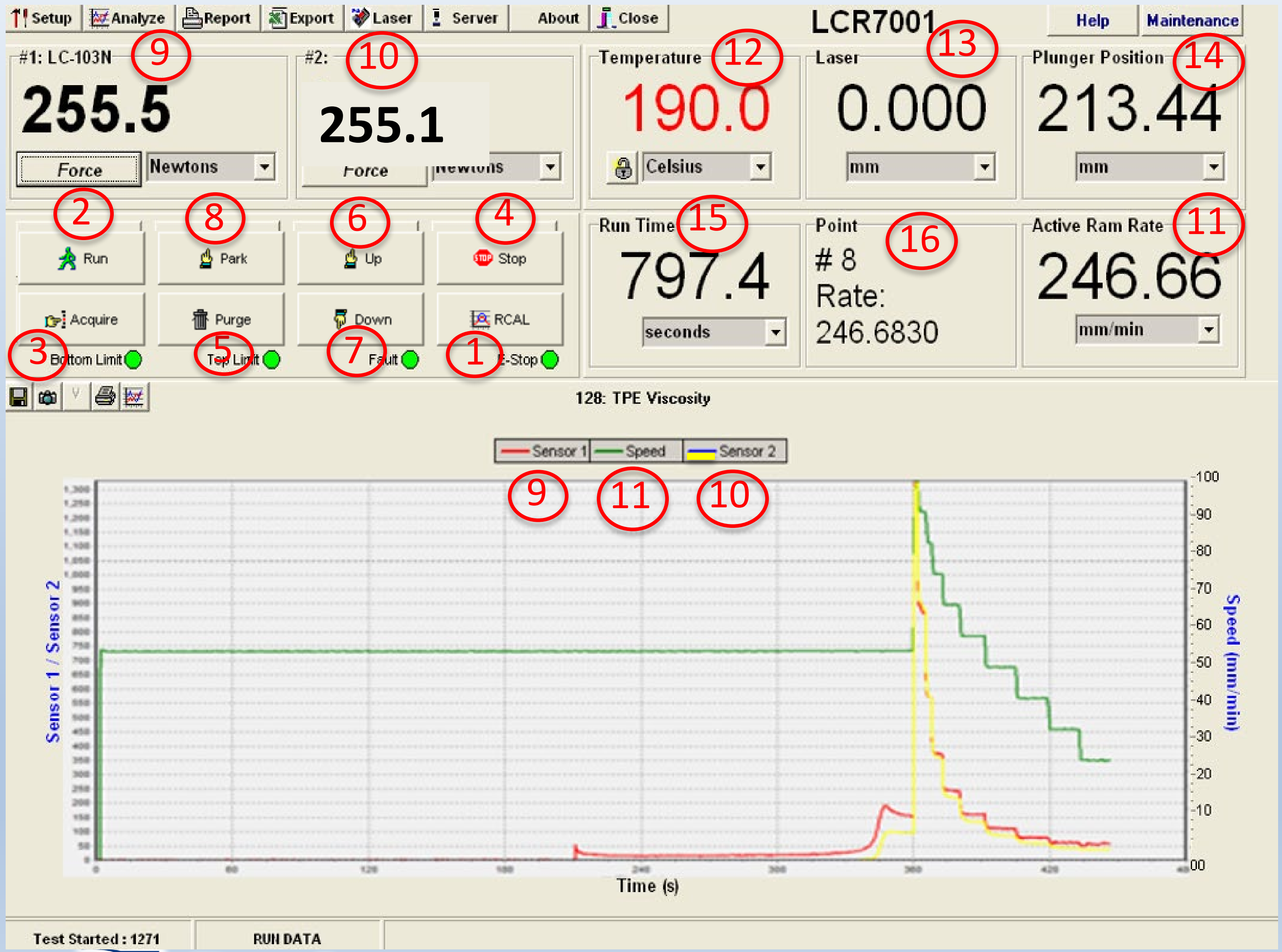
- Which polymer might face higher thermal degradation with increasing temperature?
- Which polymer has wider processing temperature?

Shear-Sweep Test Setup in LabKars



1. **Add/delete Data Point Setup**
2. **Program Name**
3. **Start Position (mm):** 89-100 mm
4. **Temperature (°C):** barrel temperature set point
5. **Melt time (sec):** 180-360 sec
6. **Sensor1 ID:** LC-103N
7. **Die:** choose entrance angle, D and L/D from list
8. **Minimum/Maximum Speed (mm/min):**
   Min at 0.03 and Max at 650 mm/min
9. **Data Acquisition mode:**
   Steady state, Position, Time Delay, Manual
10. **Speed Control range (mm/min)**
11. **Shear Rate range (1/s)**
12. **Send:** sending test setup to rheometer

LabKars Software (Control Screen)



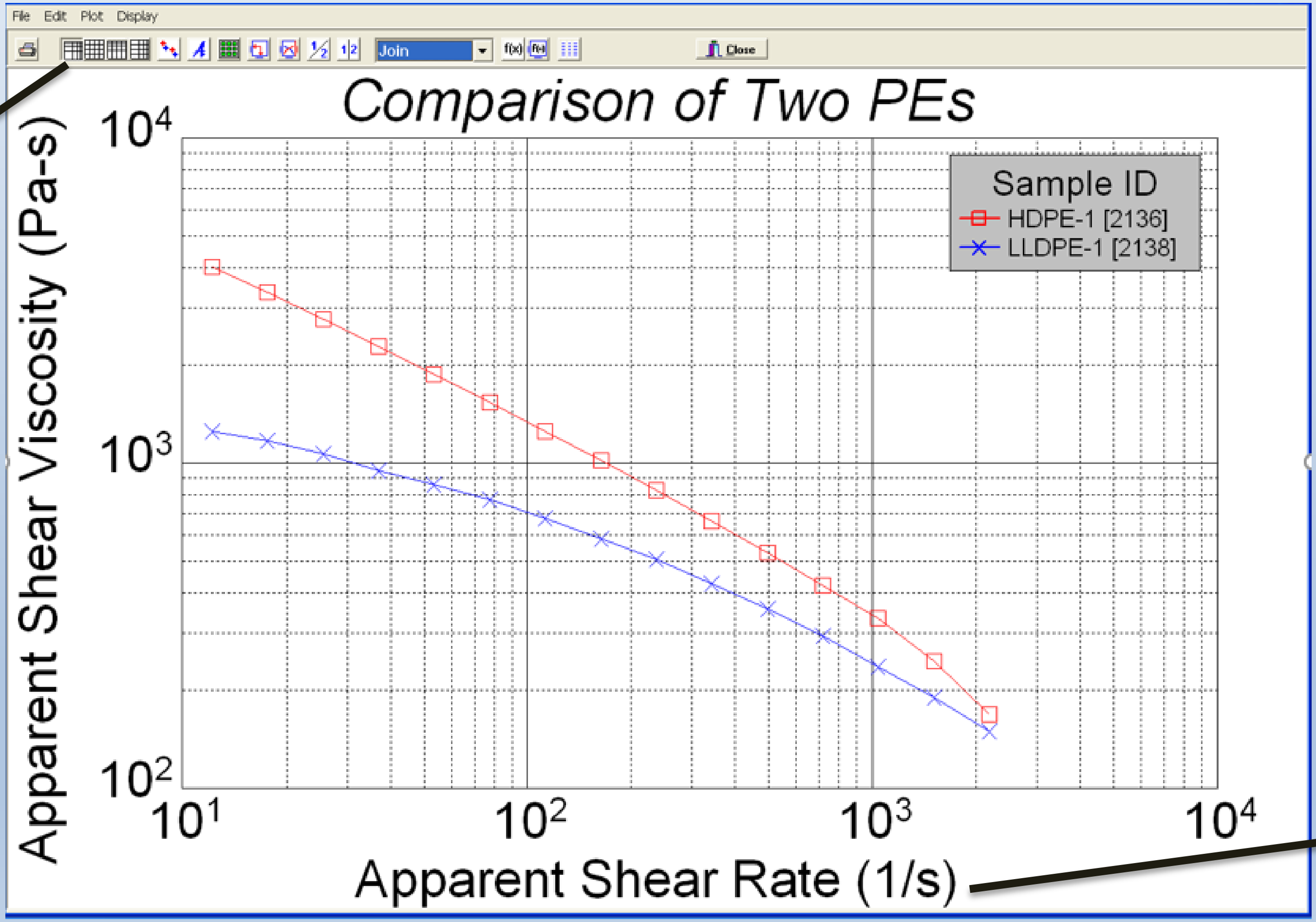
Real-time data graph during the test

1. **RCAL**: balance transducers!
2. **Run**: run the test!
3. **Acquire**: collect data!
4. **Stop**: stop the test!
5. **Purge:** purge all the material in barrel!
6. **Up**: move plunger upward!
7. **Down**: move plunger downward!
8. **Park**: go to park position (25mm)!
9. **Sensor #1 (N):** force on load cell
10. **Sensor #2 (N)**: force on pressure transducer
11. **Active Ram Rate (mm/min):** piston speed
12. **Temperature (°C):** actual barrel temperature
13. **Laser (mm):** die swell detection
14. **Plunger position (mm)**
15. **Run time (sec)**
16. **Collected point**: at each shear rate

Data Analysis

Plot of log apparent viscosity versus apparent shear rate

Click to make logarithmic scale

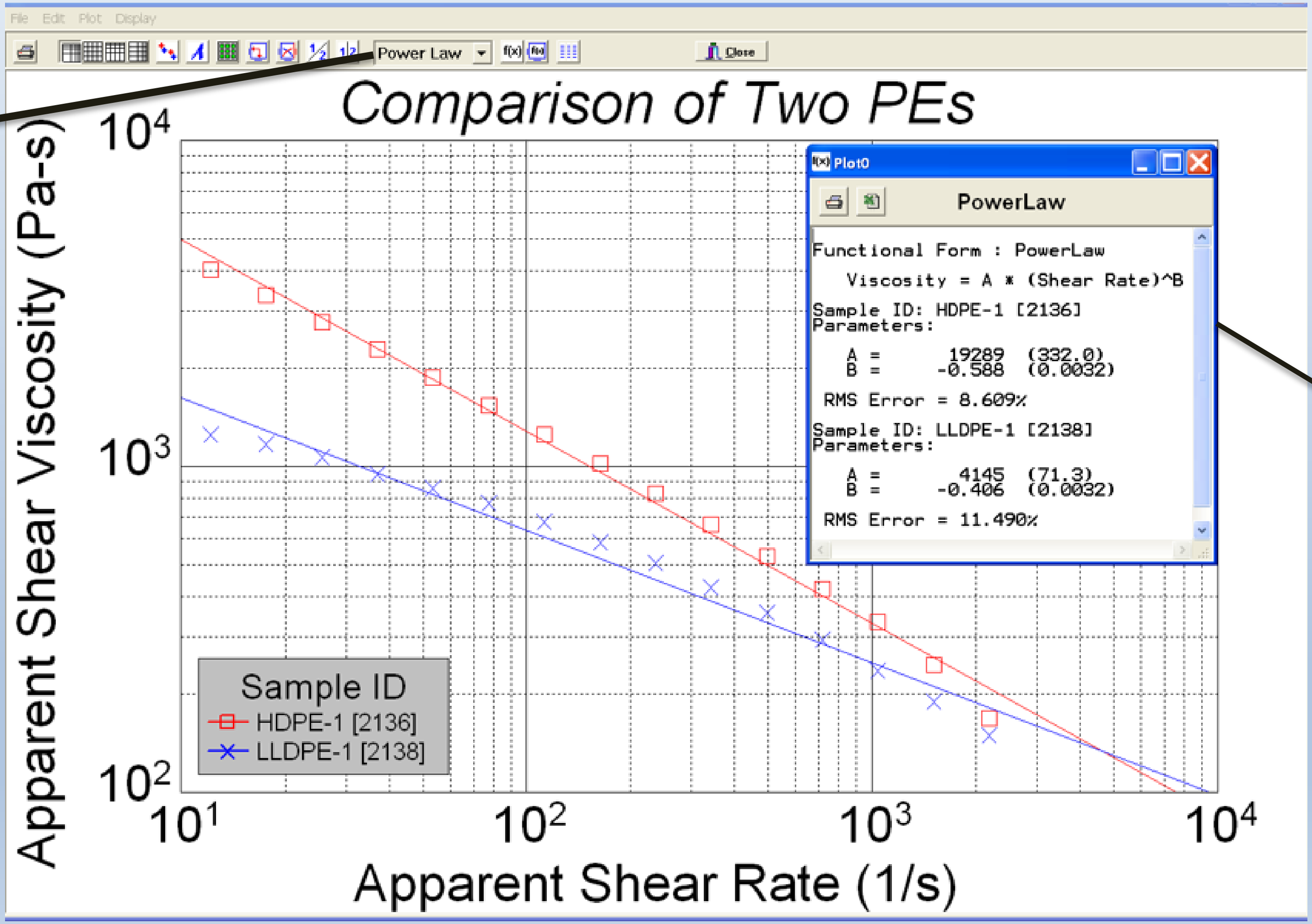


Right Click to change the axis

Data Analysis

Power-law model analysis

Choose "Power Law"

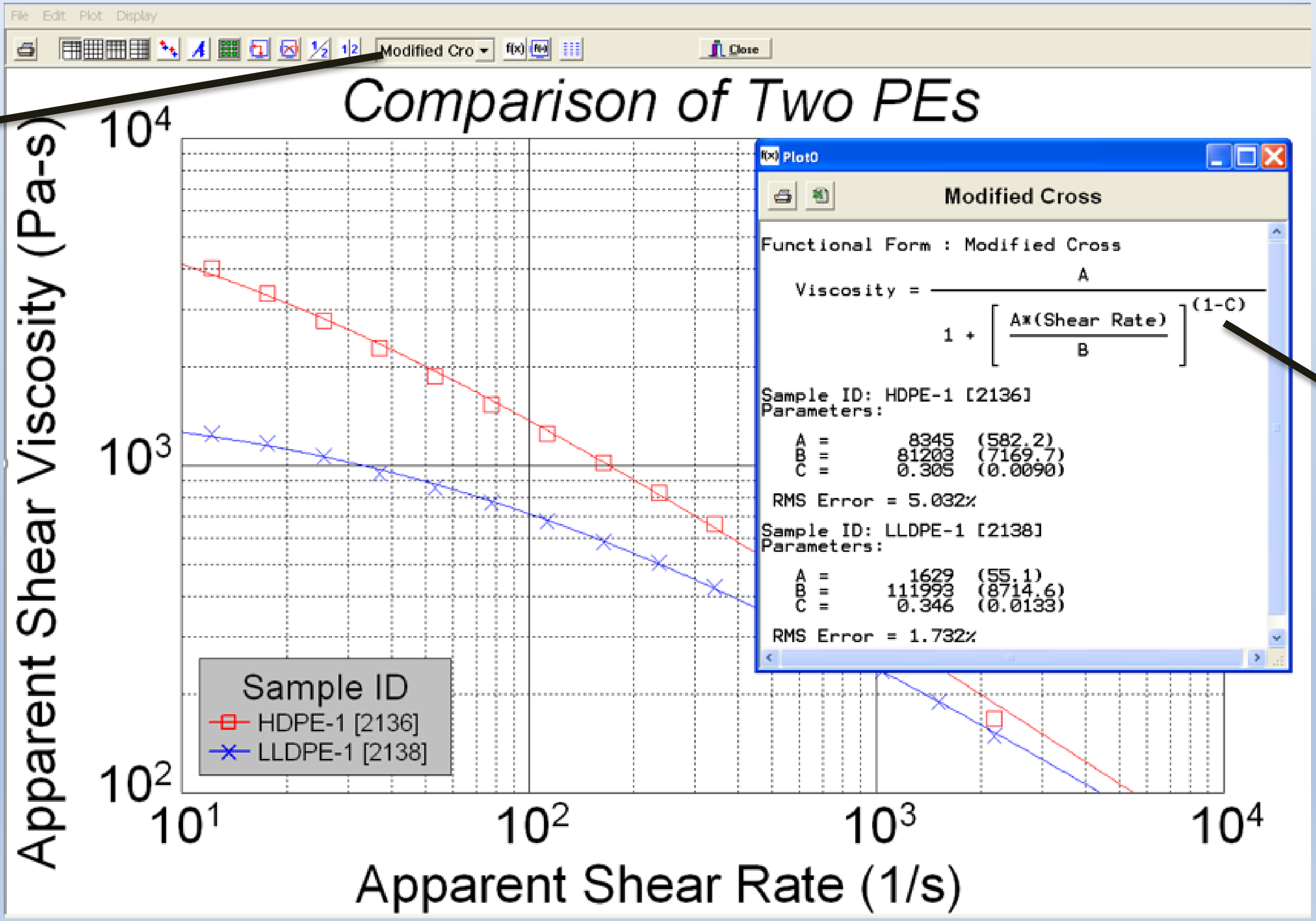


Power law fitting data

Data Analysis

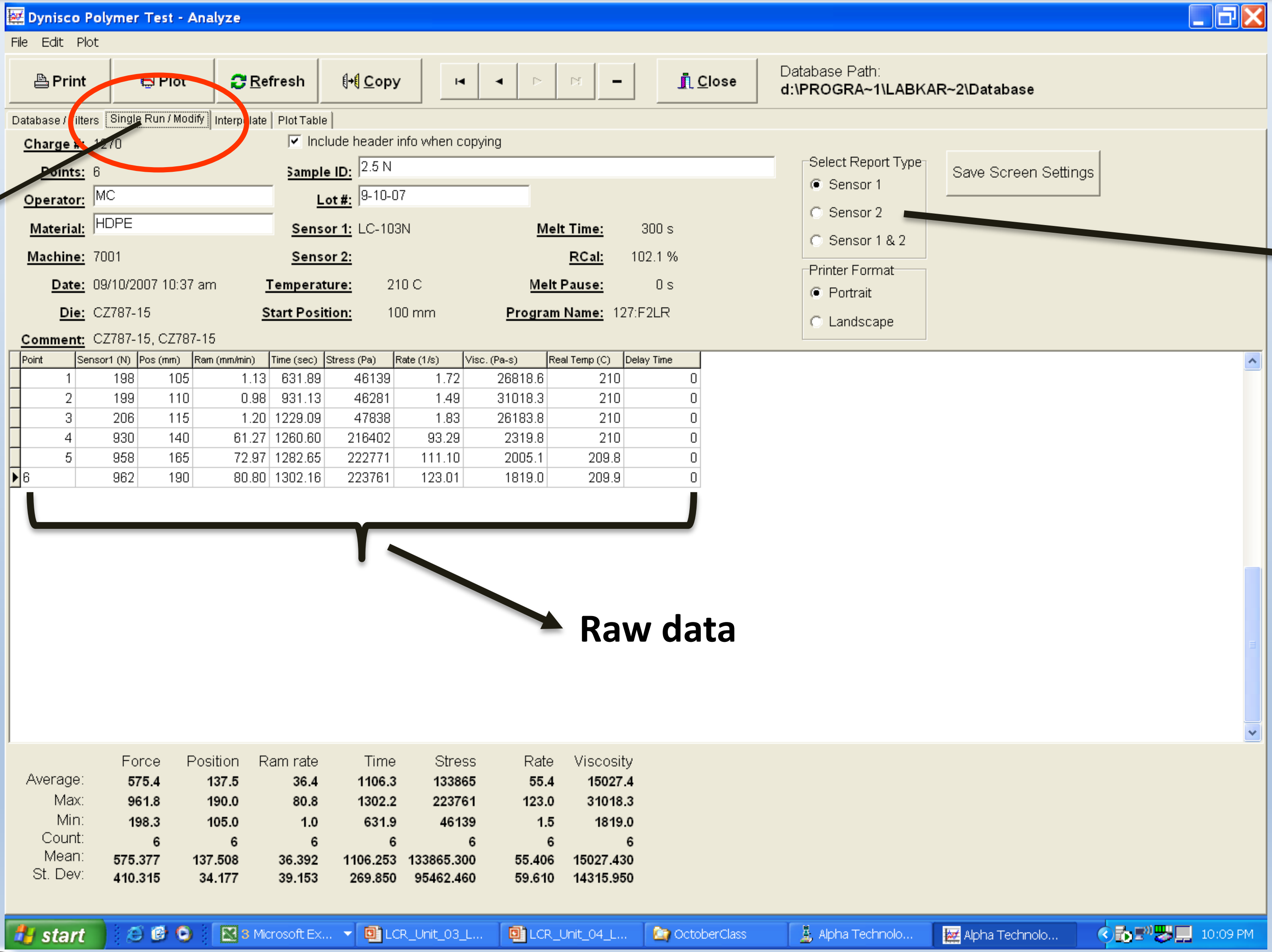
Modified cross model analysis

Choose "Modified Cross"



Modified cross fitting data

Raw Data in LabKars



Select "Single Run/Modify" to see the raw data

Select the type of sensor

- Sensor 1: Load cell
- Sensor2: Pressure transducer

Raw data

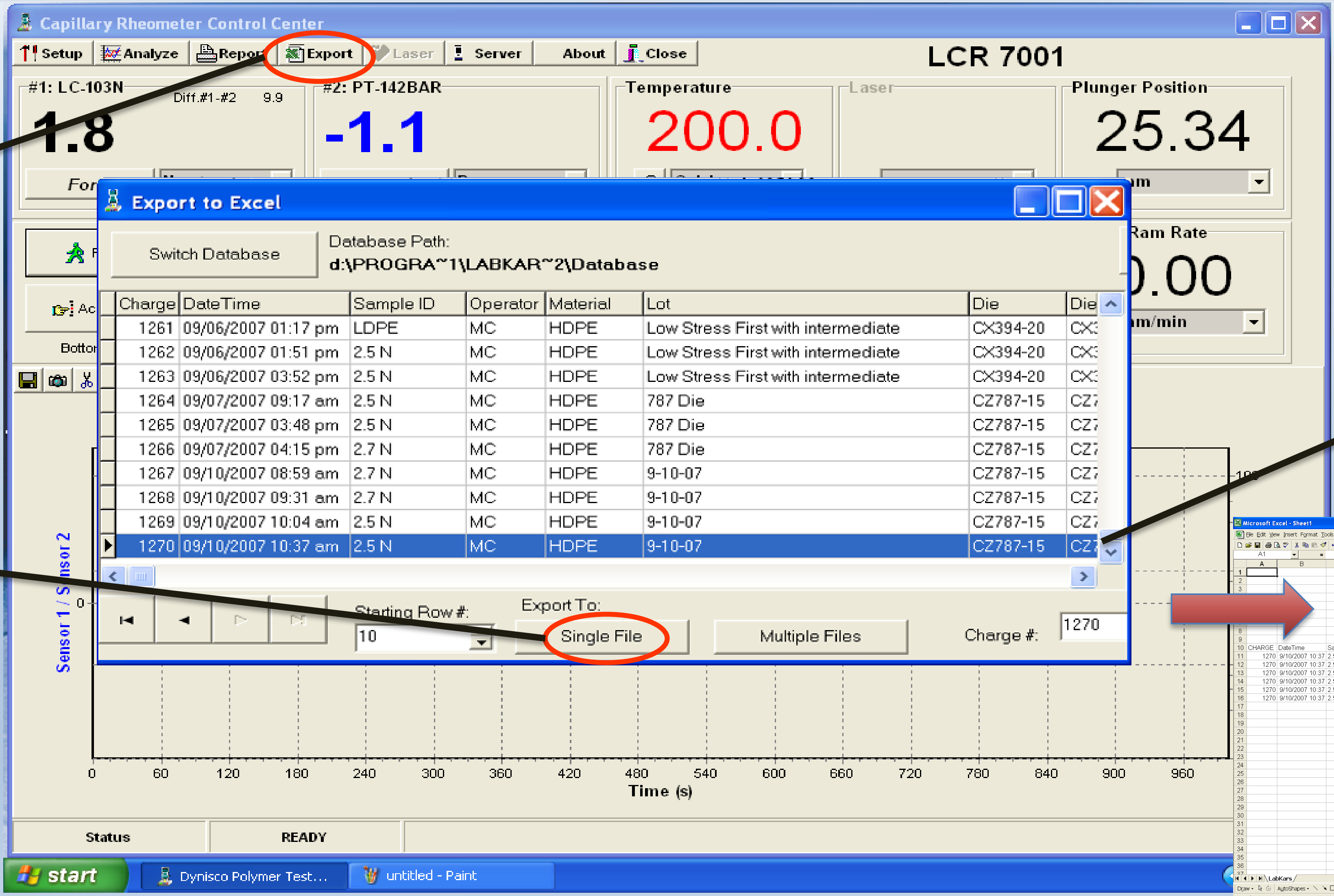
Max, min, average, mean, and standard deviation information of all parameters

Dynisco

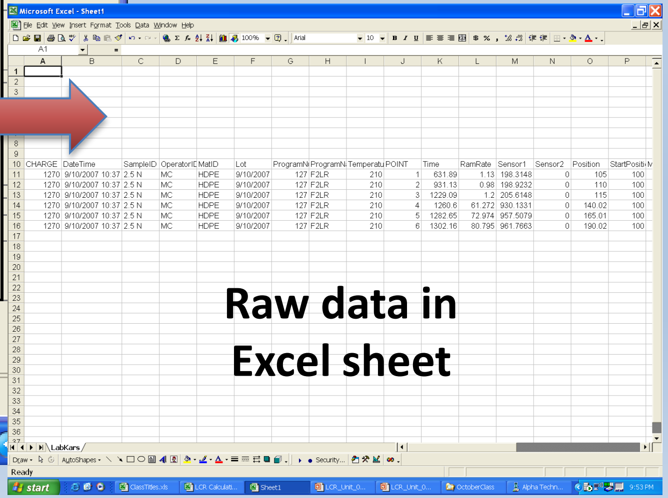
Export Raw Data in Excel

Select "Export"

Select "Single File" to export the data in Excel



Choose your data set



Raw data in Excel sheet

Rabinowicz and Bagley Corrections

Corrections

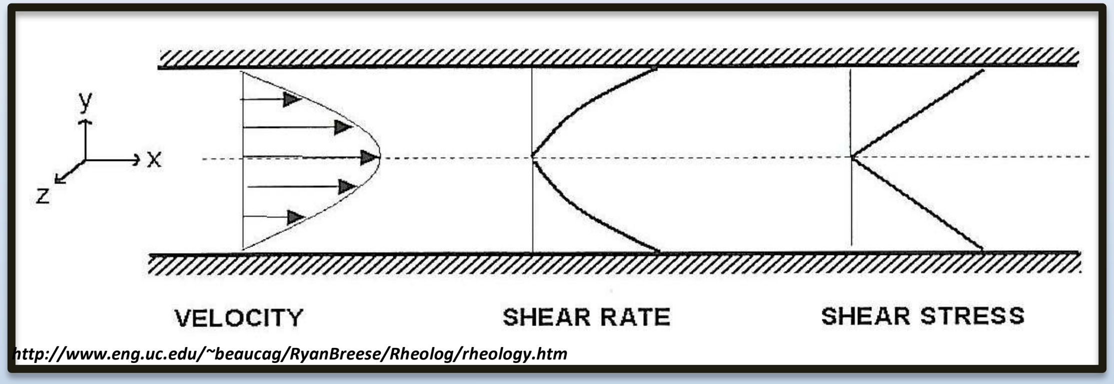
- All calculations we discussed are "Apparent" values since they assume Newtonian behavior and that the entire pressure drop occurs inside through die. (Assuming no die entrance/exit effect)

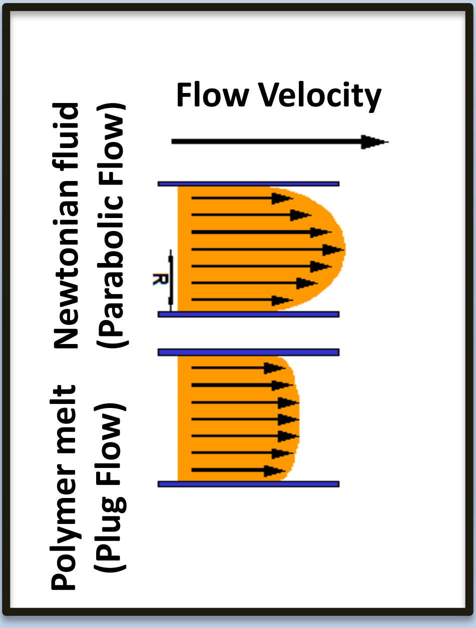
- **Rabinowicz correction** needs to be applied in order to rectify the data for non-Newtonian character of polymer melt. (Calculation of **corrected shear rate**)

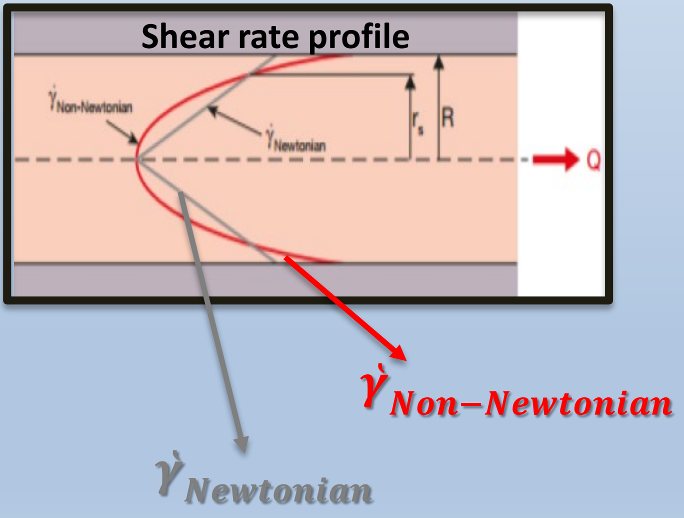
- **Bagley correction** needs to be applied to consider the extra pressure drop that may happen at the entrance/exit of the die.(Calculation of **corrected shear stress**)

Why Rabinowicz Corrections?

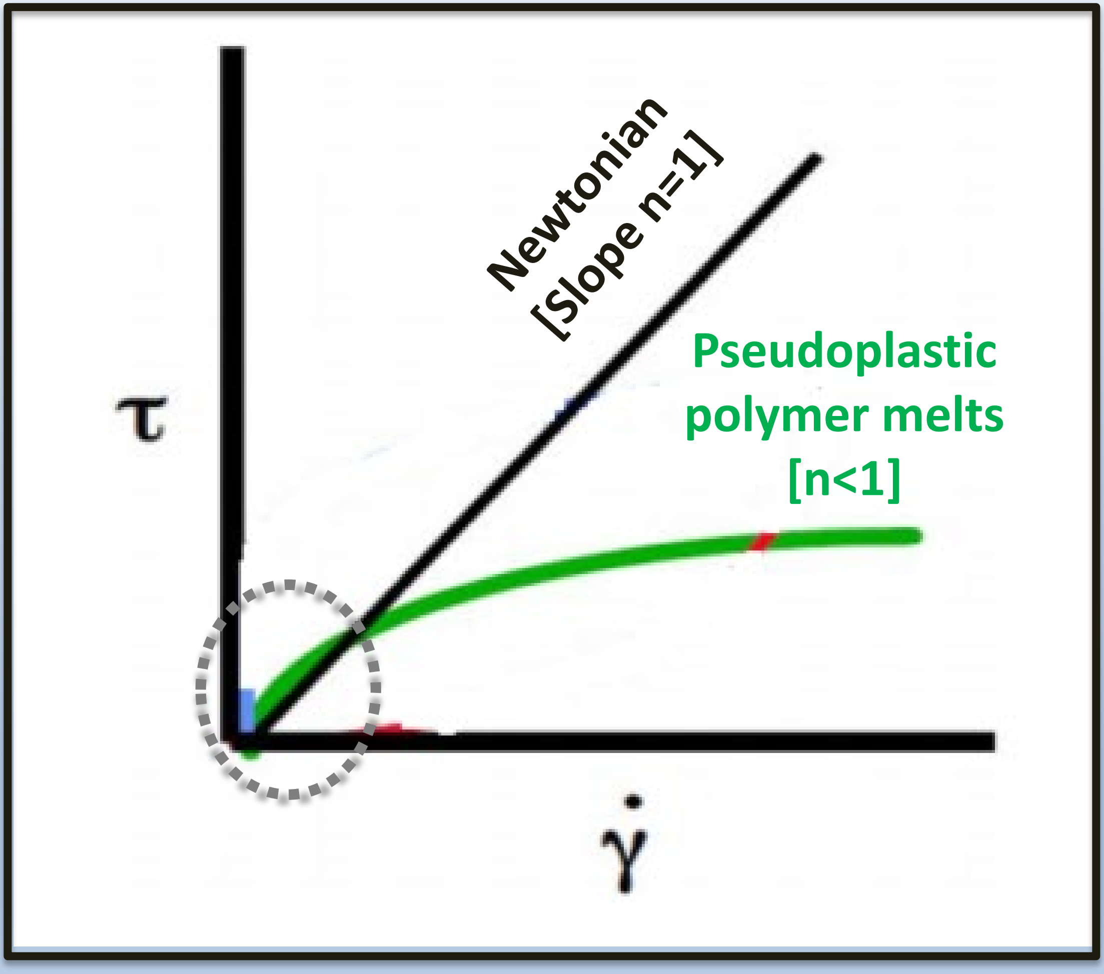
Flow through a die







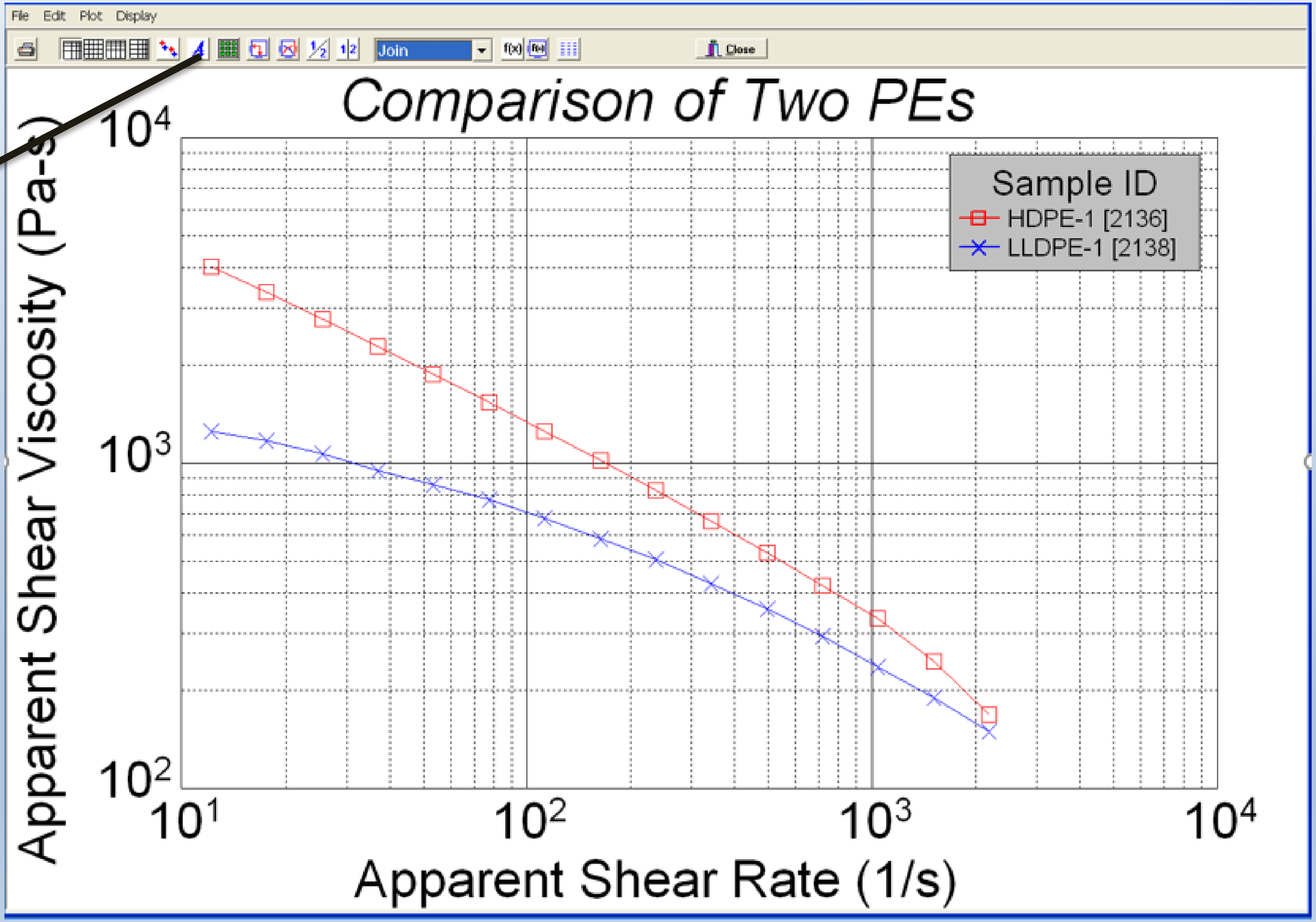
Rabinowicz Corrections



$$\dot{\gamma}_{corrected} = \left(\frac{3n+1}{4n}\right)\dot{\gamma}_{apparent}$$

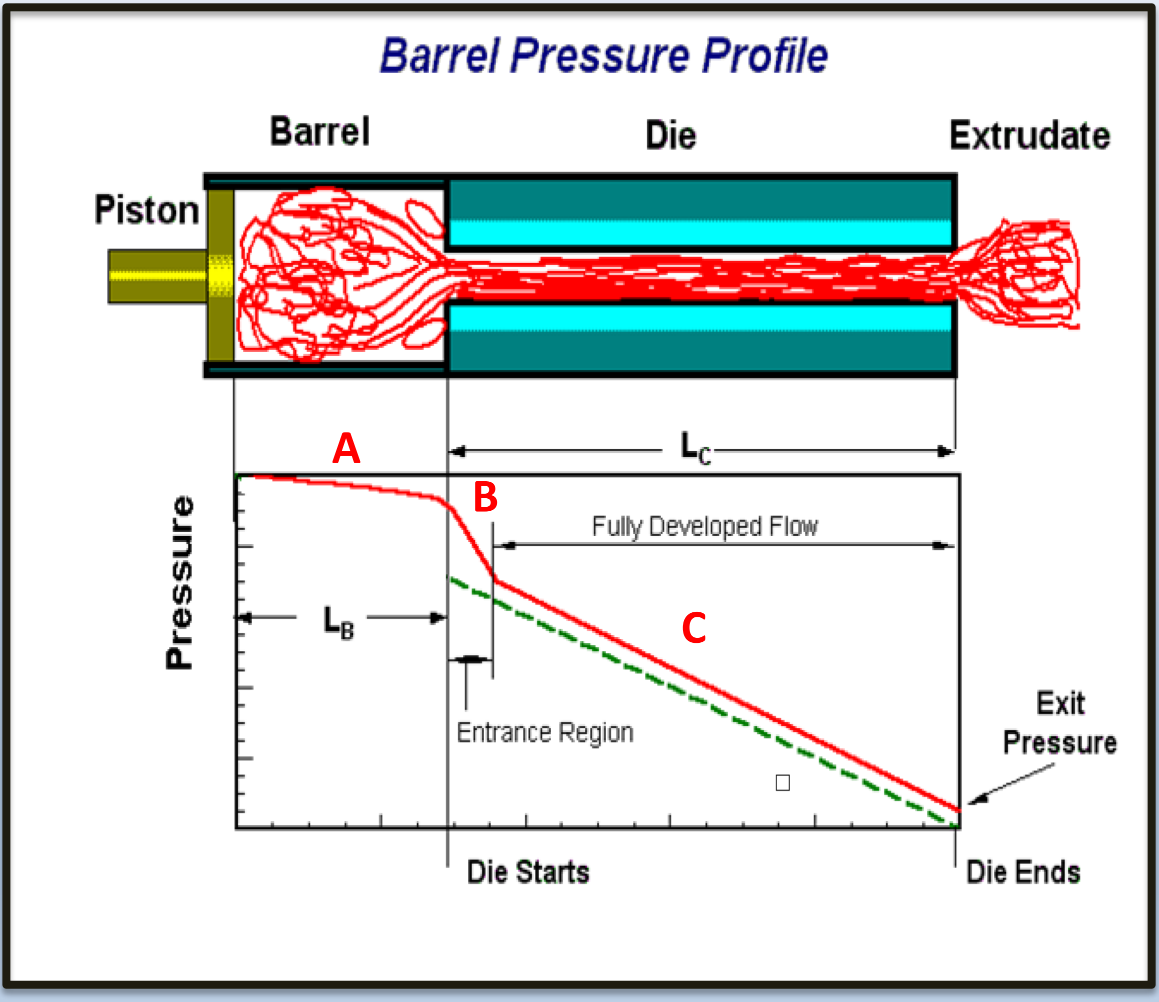
Correction Factor

How to Apply Rabinowicz Corrections in LabKars?



Click to plot viscosity versus "corrected" shear rate

Why Bagley Corrections?



- $A = \Delta p_{Barrel}$: Very minor pressure drop in barrel
- $B = \Delta p_{Entrance}$: Excess pressure drop in die entrance
- $C = \Delta p_{Capillary}$: Fully developed flow region in capillary die

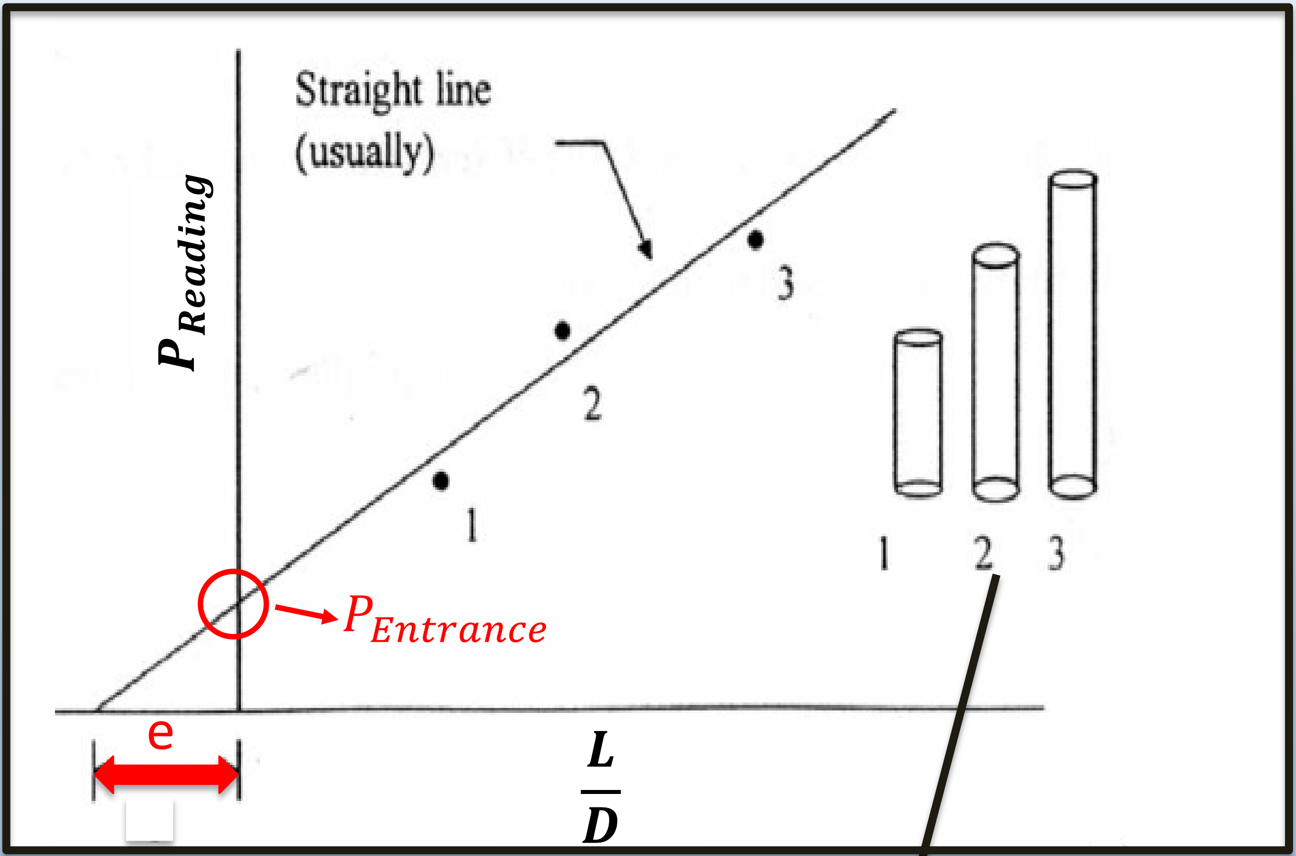
- Large pressure drop associated with the flow entrance region due to viscoelasticity of polymers.
- After entrance region, The pressure gradient approaches a constant value (fully developed flow region)
- In reality:

$$\Delta P_{total} = \Delta P_{Entrance} + \Delta P_{capillary}$$

- Bagley correction needs to be applied to calculate the entrance pressure drop

Dynisco

How to Calculate the Entrance Pressure?



Three dies, same diameter
but different lengths
(e.g. L/D: 10, 20, and 30)

$$\tau_{Corrected} = \frac{P_{Reading} - P_{Entrance}}{4(\frac{L}{D})}$$

where

$$P_{Entrance} = P\,(at\,\frac{L}{D} = 0\,)$$

OR

$$\tau_{Corrected} = \frac{P_{Reading}}{4(\frac{L}{D} + e)}$$

where

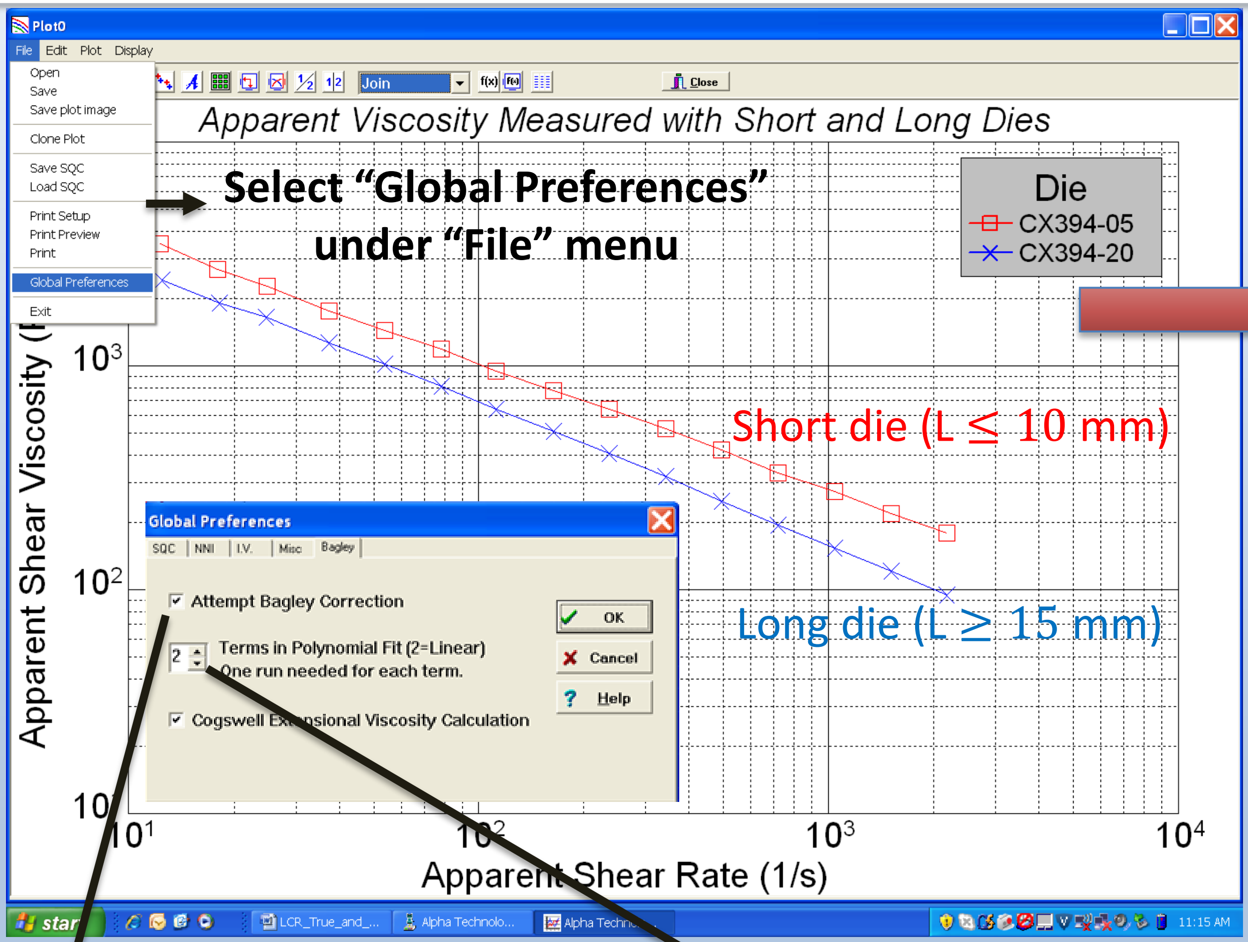
$$\boldsymbol{e = end\ correction = \frac{-L_{Entrance}}{D}}$$

Dynisco

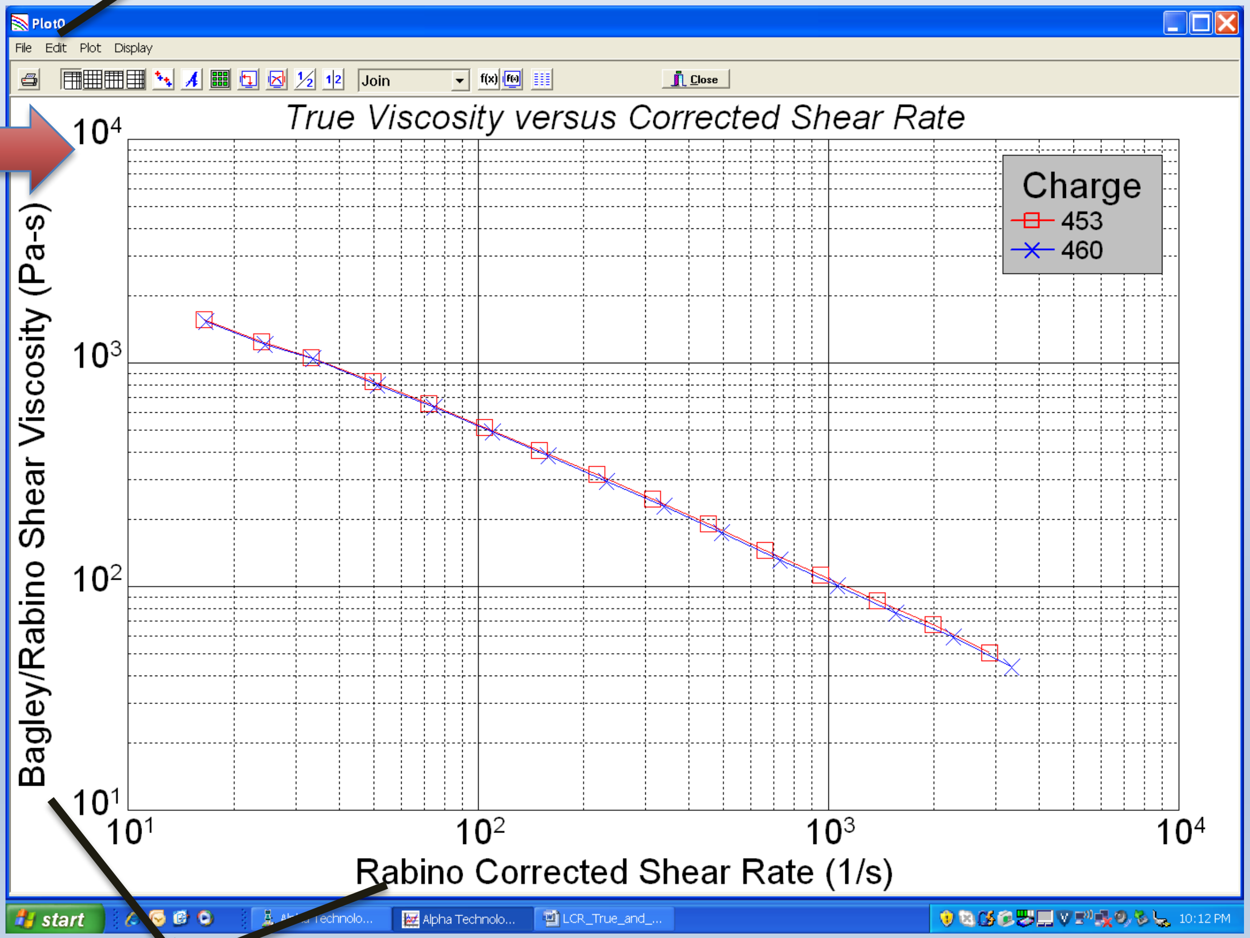
J.M. Dealy, K.F. Wissbrun, *Melt Rheology and its Role in Plastics Processing: Theory and Applications*, Van Nostrand Reinhold, New York (1990).

How to Apply Bagley Corrections in LabKars?

Select “Global Preferences” under “File” menu



Select “Copy Table to Clipboard” under “Edit” menu to get the raw data



Select “Attempt Bagley Correction”

Insert number “2”

Right click on each axis and select the appropriate item to plot

How to Calculate True Viscosity?

$$\eta_{True} = \frac{\tau_C}{\dot{\gamma}_C}$$

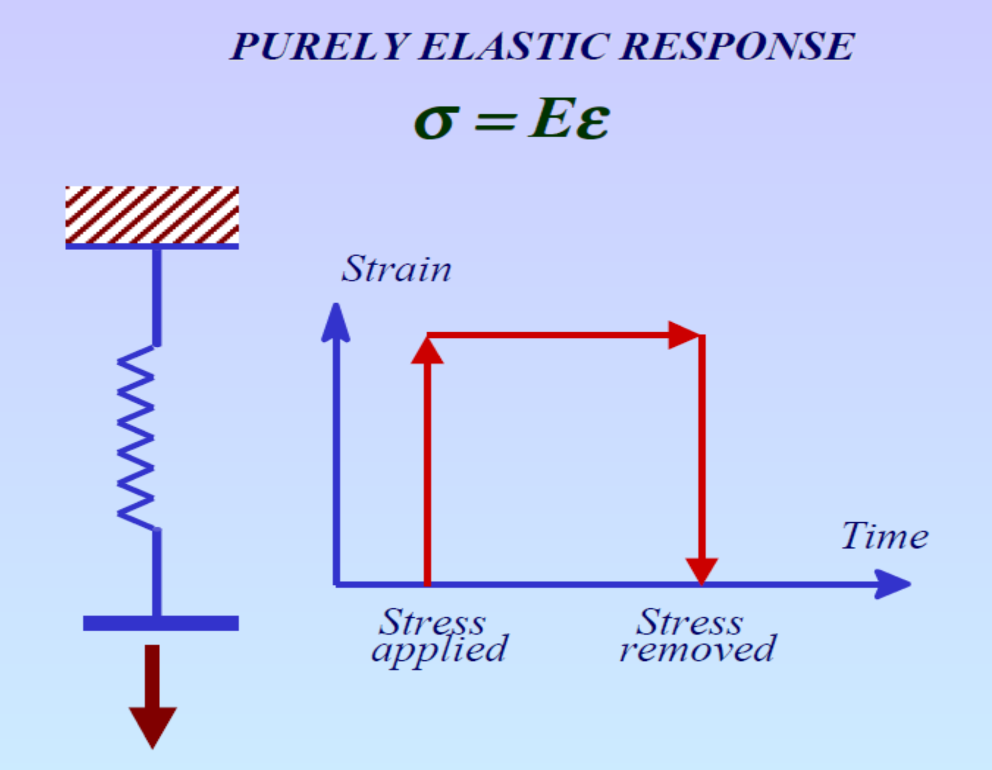
where

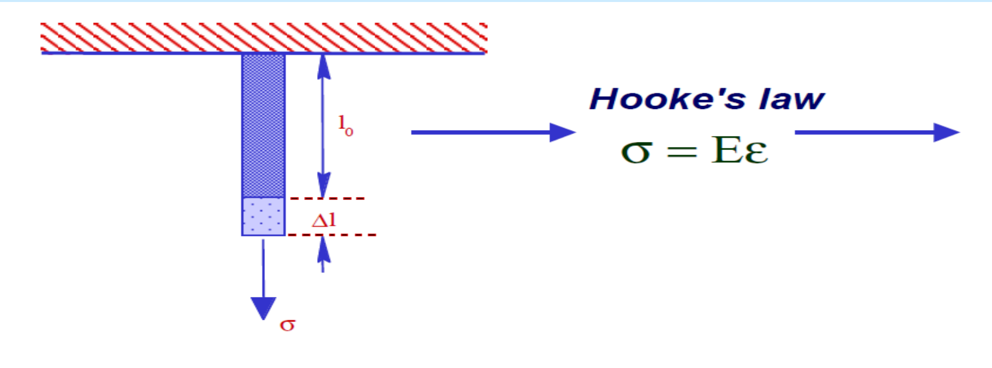
- η_{True} (Pa-s): Viscosity with Bagley and Rabinowicz Corrections
- τ_C (Pa): Bagley corrected shear stress
- $\dot{\gamma}_C$ (1/s): Rabinowicz corrected shear rate

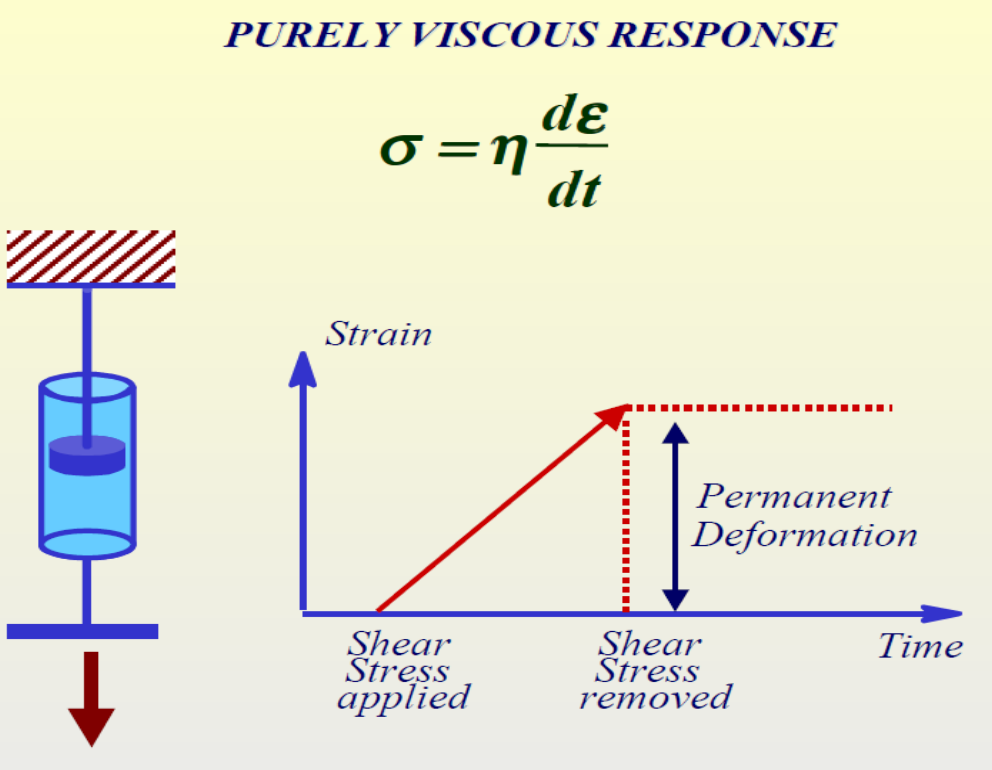
Dynisco

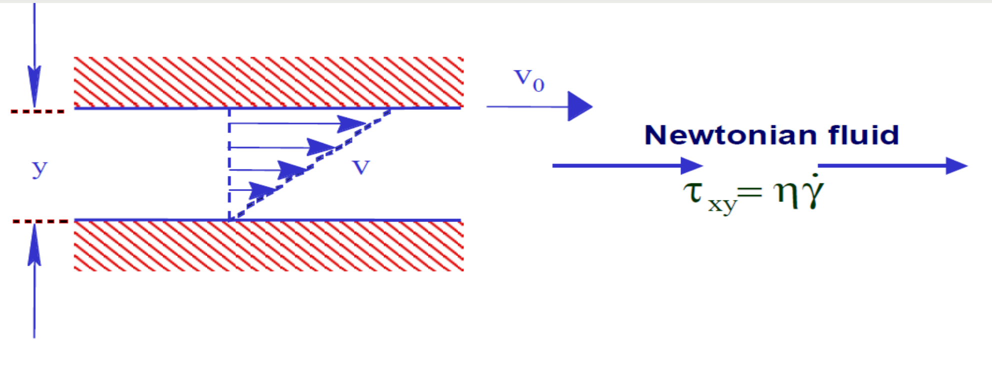
Viscoelasticity

Mechanical Models of Viscoelastic Behavior

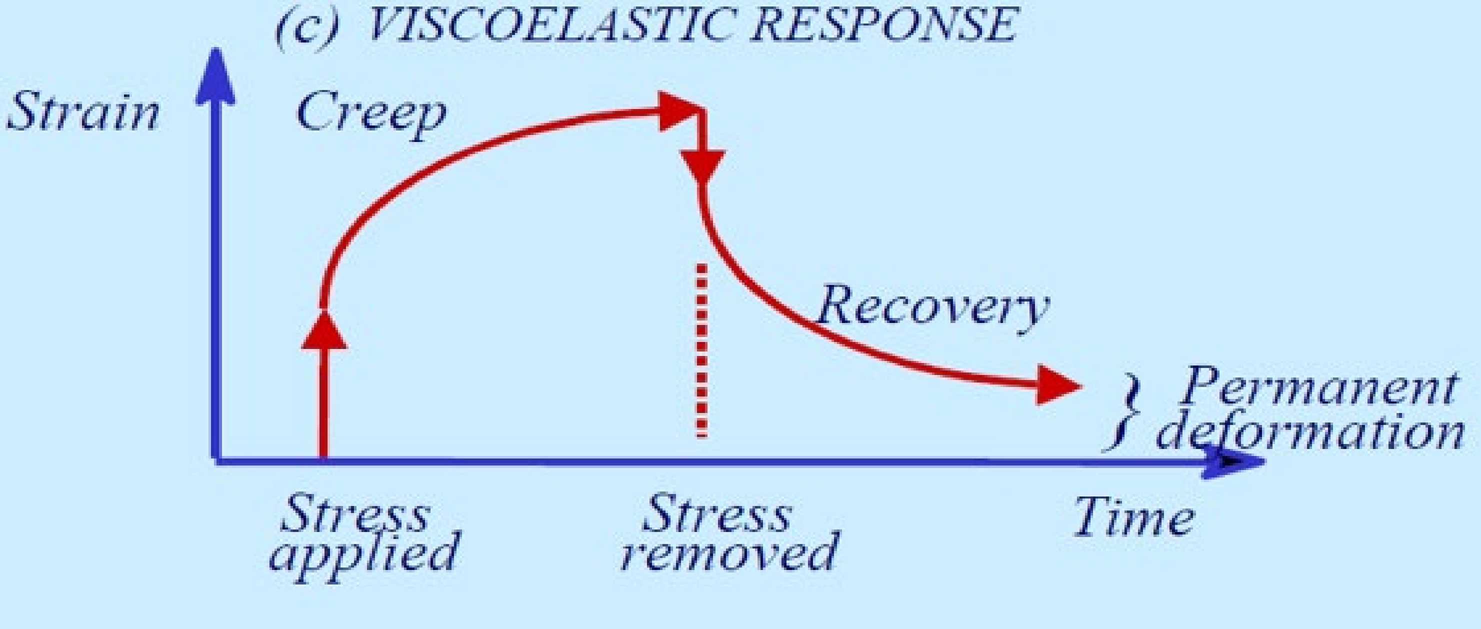








Mechanical Models of Viscoelastic Behavior

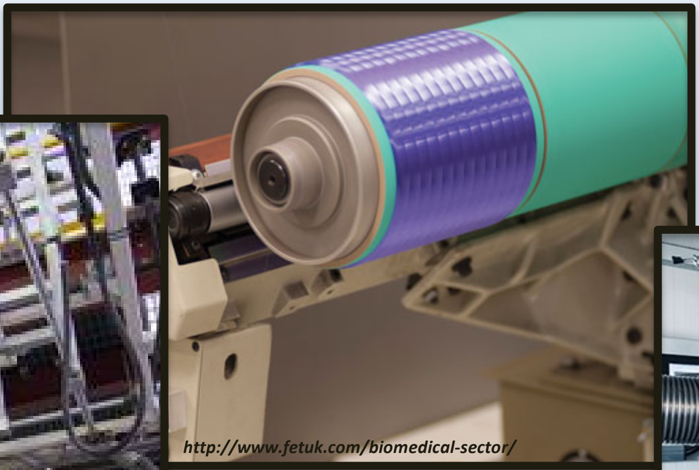


Dynisco

Extensional Viscosity Measurements

When Extensional Viscosity is important?

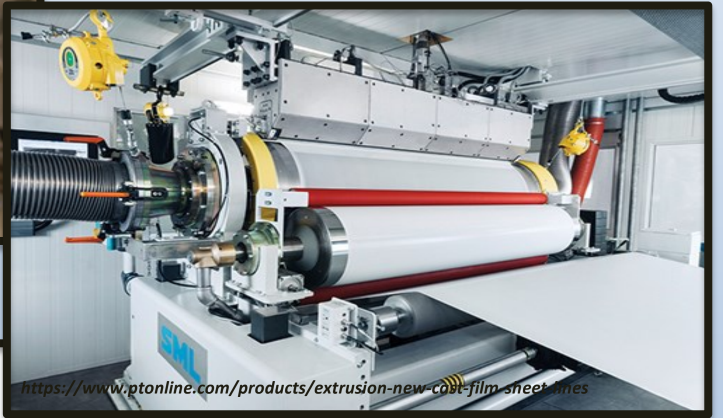
http://www.polymersni.com/industry/extrusion-film



http://www.fetuk.com/biomedical-sector/

http://www.smg3d.co.uk/images/pc_bottle_master_blow_molding_resized.jpg

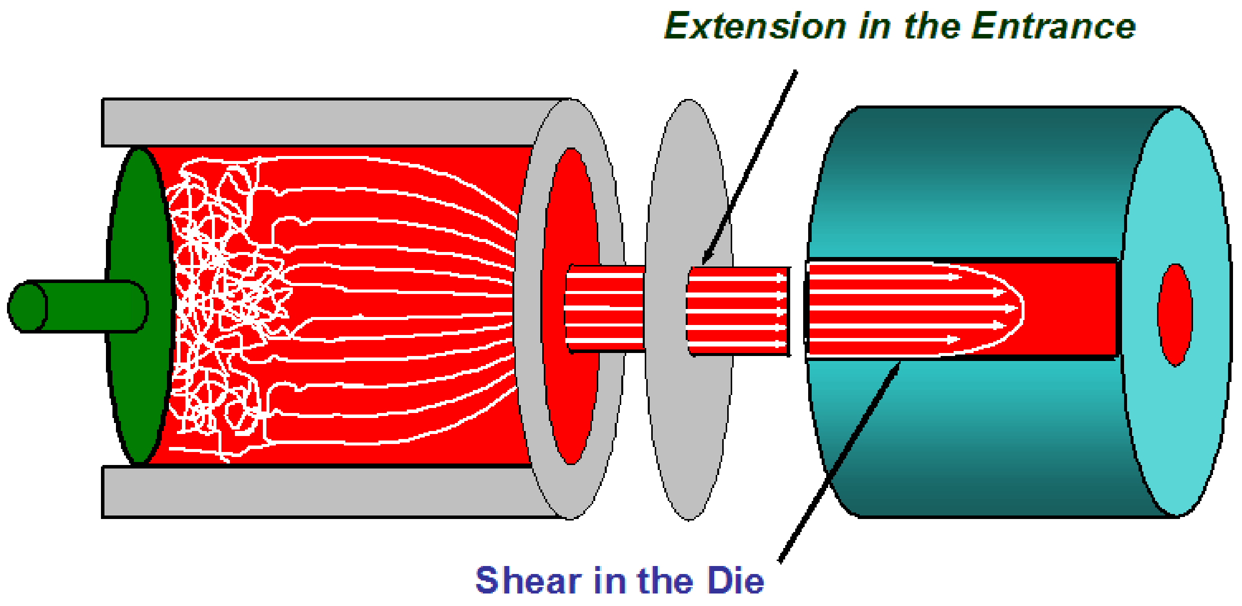
Any process that has stretch!

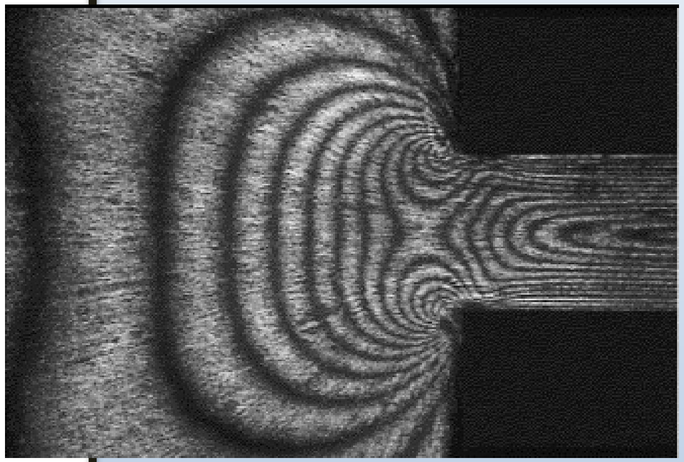


https://www.ptonline.com/products/extrusion-new-cost-film-sheet-lines

http://www.directindustry.com/prod/amut/product-20400-1445327.html

What is Extensional (Elongational) Viscosity?





- Since the diameter of the barrel versus die is very large, there is a high degree of stretching along streamlines at the entrance to the die
- Cogswell assumes that the pressure drop in entrance has two components, one due to shear and one due to extension

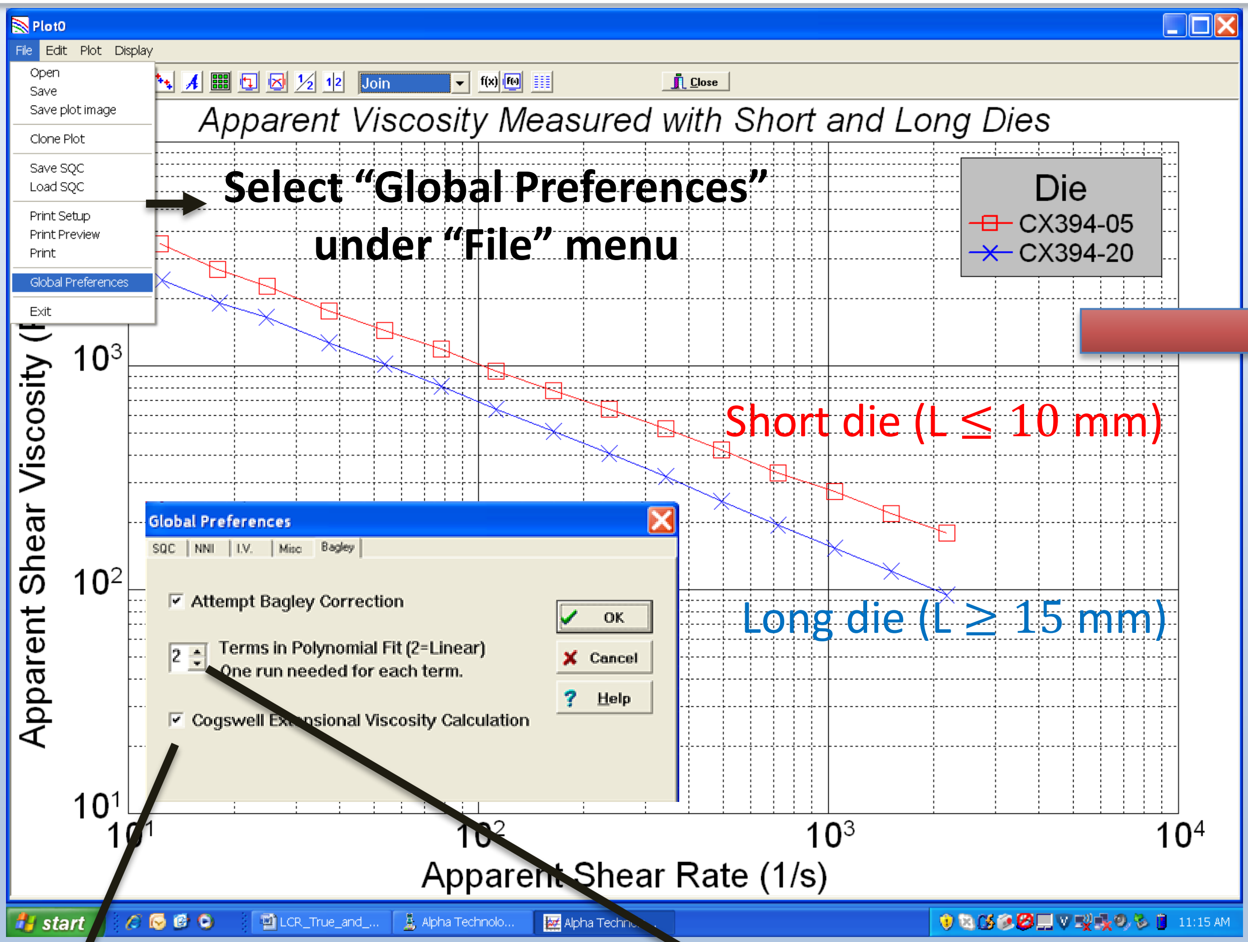
Dynisco

Cogswell`s Equations

Extensional Stress	❖ $\sigma_{Ext} = \frac{3}{8}(n+1)P_{Ent}$	➢ $\boldsymbol{\sigma_{Ext}}$ ***(Pa):*** Extensional stress ➢ $\boldsymbol{n}$***:*** Power law index ➢ $\boldsymbol{P_{Ent}}$ ***(Pa):*** Entrance pressure
Extensional Rate	❖ $\dot{\epsilon} = \frac{4\dot{\gamma}_a^2\eta_a}{3(n+1)P_{Ent}}$	➢ $\boldsymbol{\epsilon}$ ***(1/s):*** Extensional rate ➢ $\boldsymbol{\dot{\gamma}_a}$***(1/s):*** Apparent shear rate ➢ $\boldsymbol{P_{Ent}}$ ***(Pa):*** Entrance pressure
Extensional Viscosity	❖ $\eta_{Ext} = \frac{9(n+1)^2P_{Ent}^2}{32\eta_a\dot{\gamma}_a^2}$	➢ $\boldsymbol{\eta_{Ext}}$***(Pa-s):*** Extensional viscosity

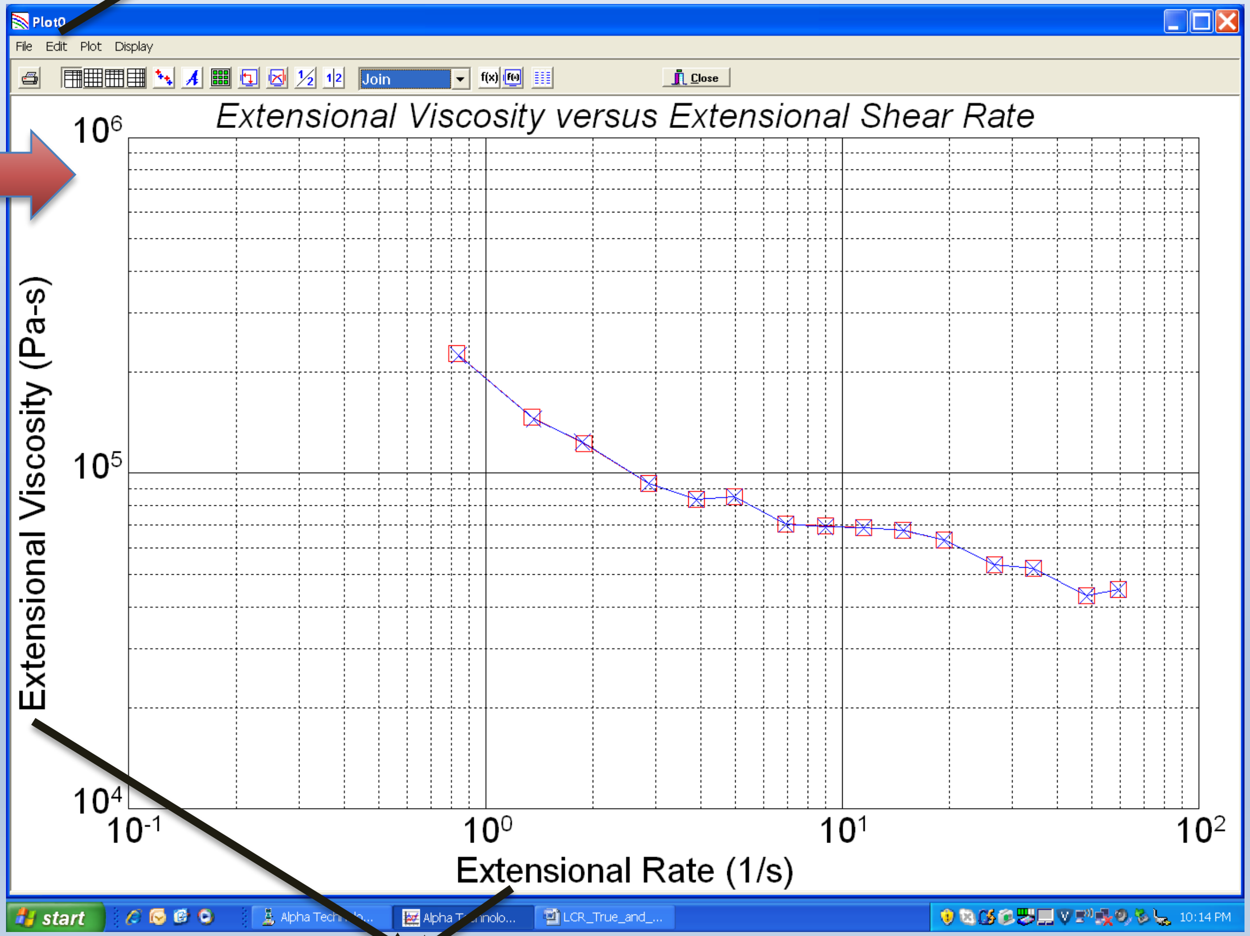
J.M. Dealy, K.F. Wissbrun, *Melt Rheology and its Role in Plastics Processing: Theory and Applications*, Van Nostrand Reinhold, New York (1990).

How to Perform Extensional Viscosity Measurements in LabKars?



Select "Global Preferences" under "File" menu

Select "Copy Table to Clipboard" under "Edit" menu to get the raw data



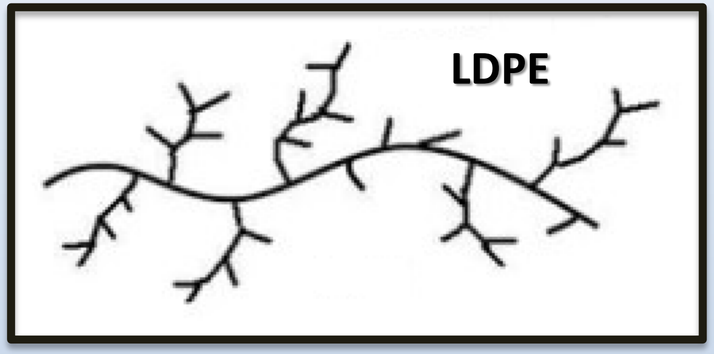
Select "Cogswell Extensional Viscosity Calculation"

Insert number "2"

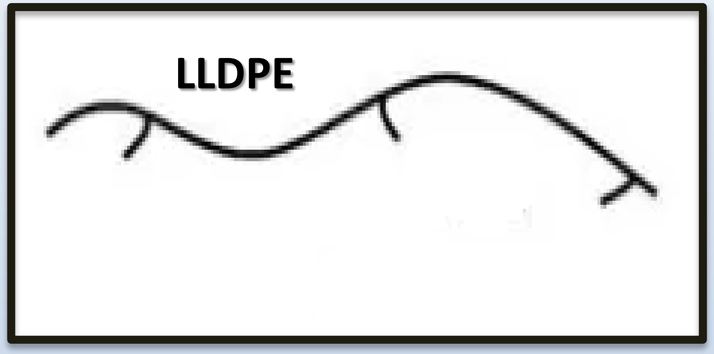
Right click on each axis and select the extensional type of axis

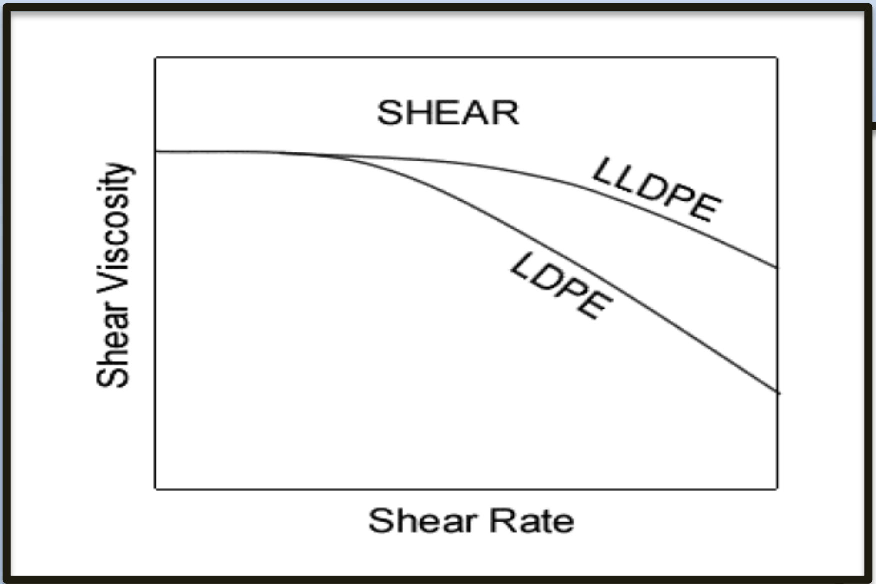
Dynisco

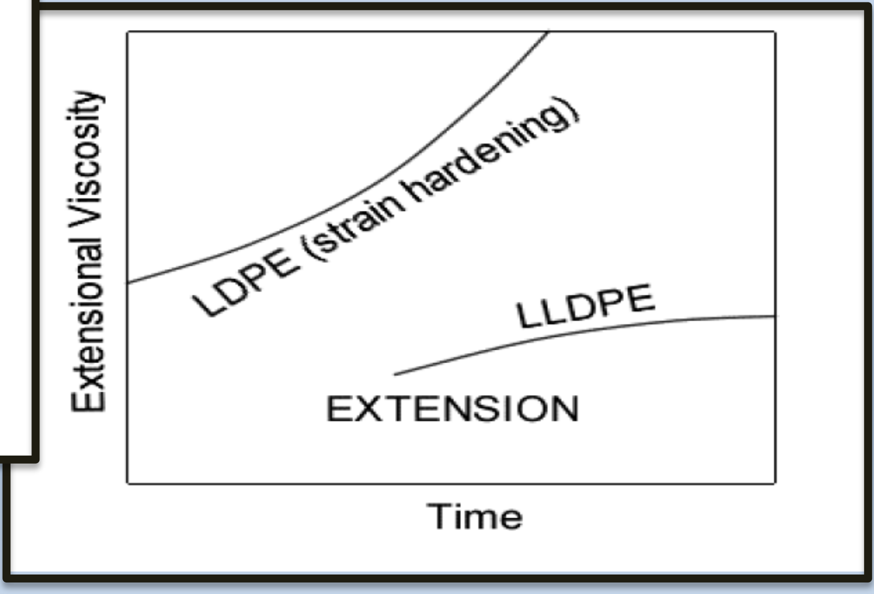
Example!

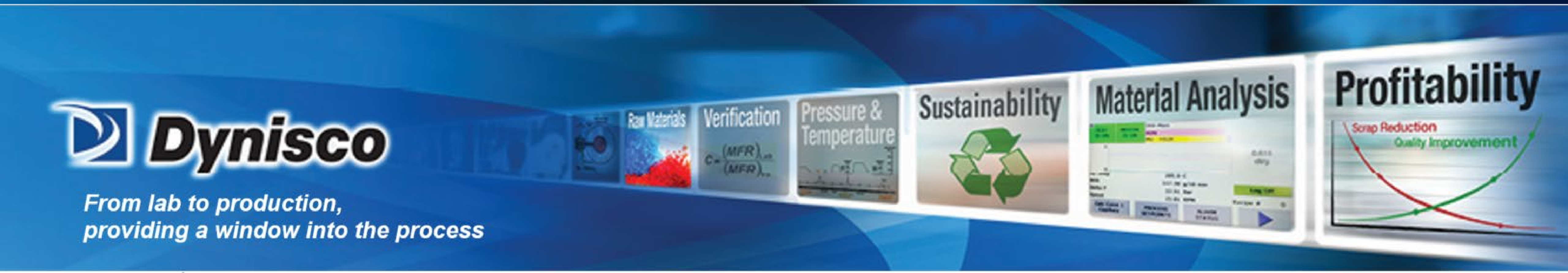


LLDPE (Linear structure) is stiffer than LDPE (branched structure) in shear but softer in extension



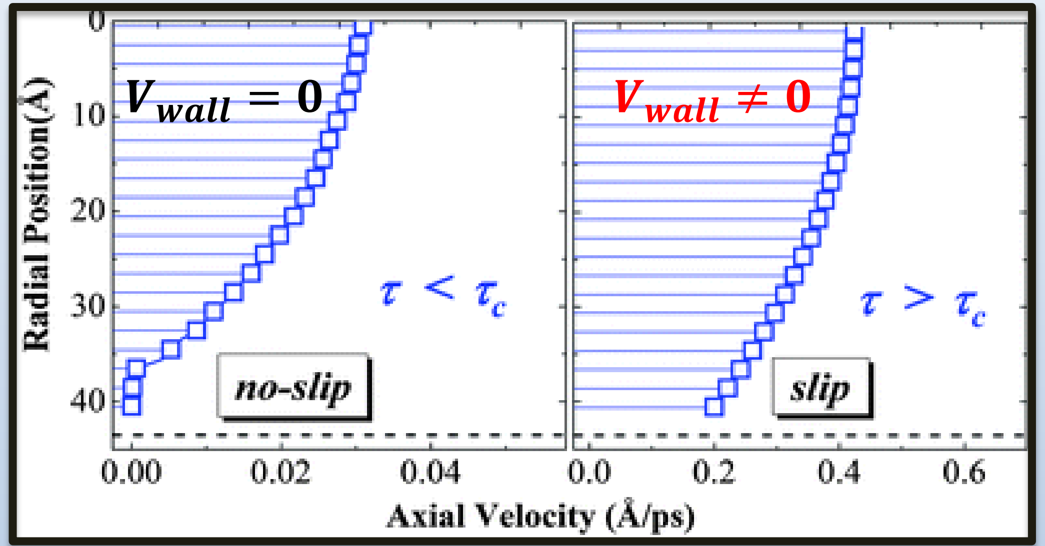


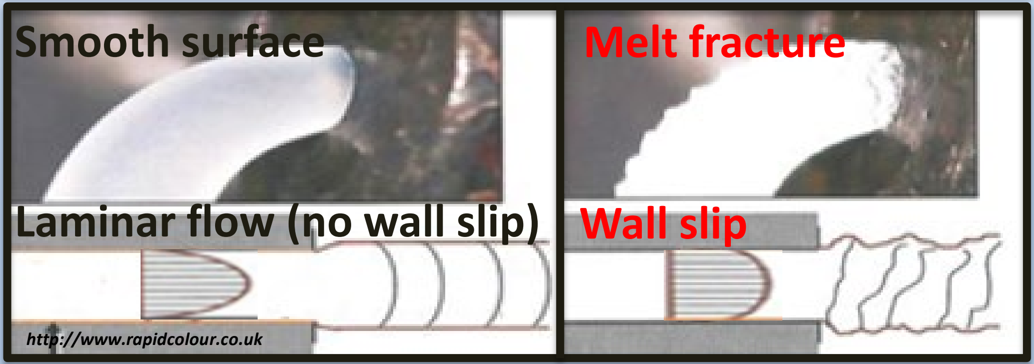




Wall Slip Velocity & Melt Farcture

Wall Slip in Capillary Flow

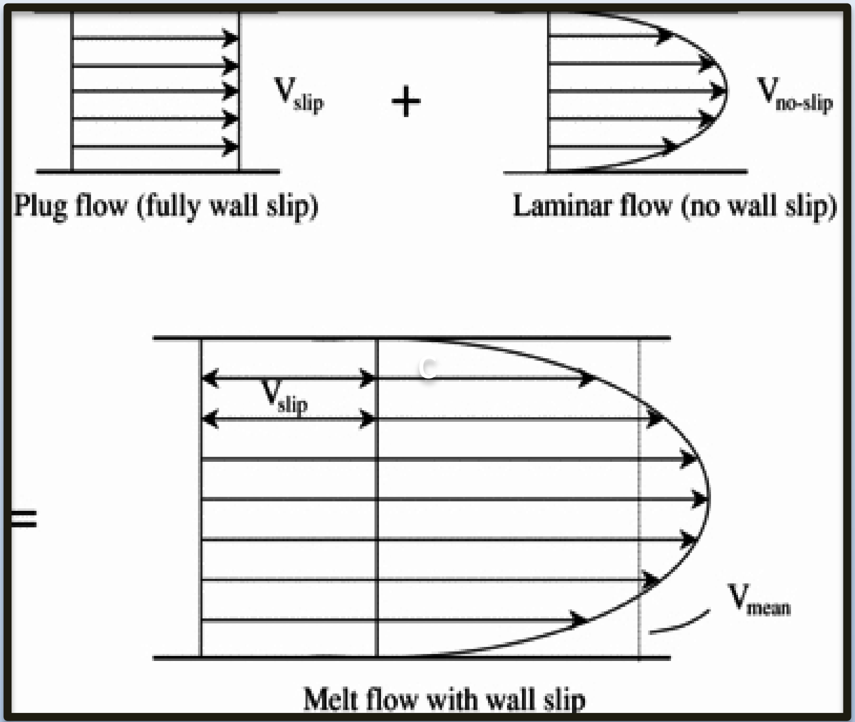




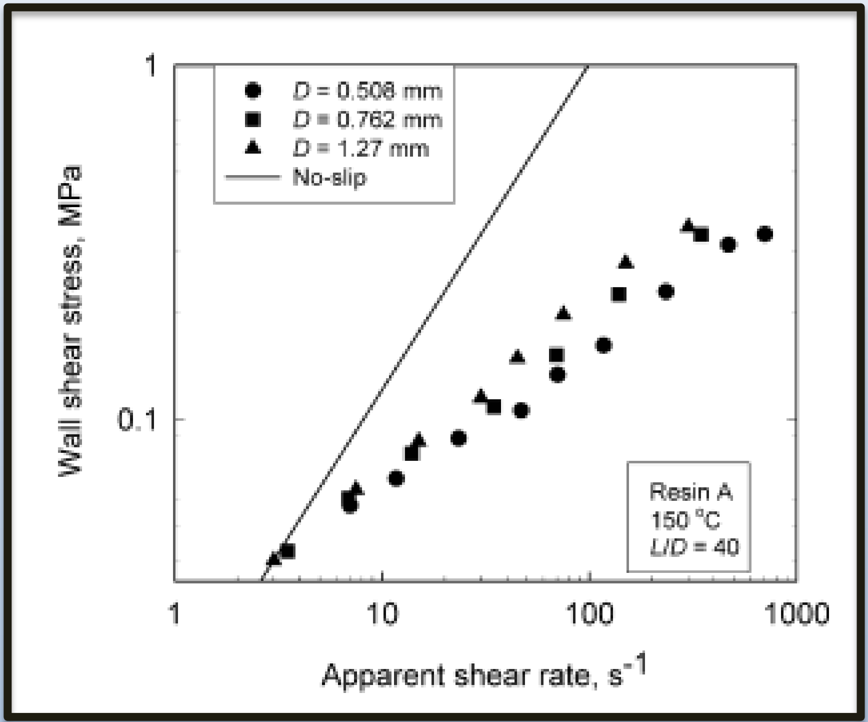
- Wall slip velocity increases dramatically above critical shear stress (~0.1 MPa).
- Slippage reduces apparent viscosity. Also, the surface of the extrudate begins to be rough **(melt fracture)**.
- Wall slip happens due to elastic properties of polymer materials.
- Critical shear stress is lower for polymers with higher molecular weight
- Trouble shooting by using larger die diameter, longer die, tapering the die, higher temperature, or lower shear rate.

Dynisco

Effect of Wall Slip on Capillary Rheometer Results



R.D. Chien, W.R. Jong, S.C. Chen, Study on Rheological Behavior of Polymer Melt Flowing Through Micro-Channels Considering the Wall-Slip Effect, *J. Micromech. Microeng.* 15, (2005), 1389



S. G. Hatzikiriakos, Wall Slip of Molten Polymers, *Progress in Polymer Science*, 37 (2012), 624-643

Wall slip causes formation of plug flow and a discontinuity in flow curve.

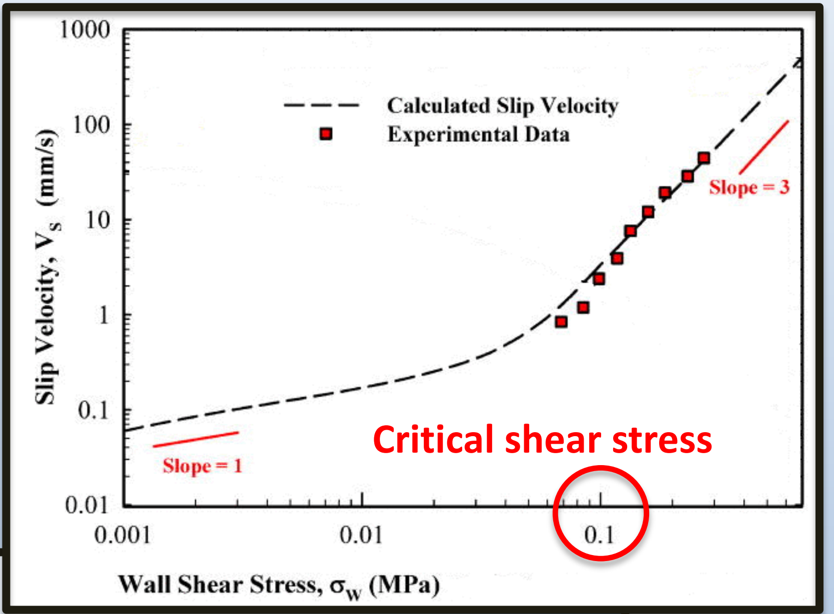
Dynisco

How to Calculate Wall Slip Velocity?

$$\dot{\gamma}_a = 4V_w\left(\frac{1}{R_C}\right) + X$$

where

- V_w **(mm/sec):** Wall slip velocity
- $\dot{\gamma}_a$**(1/s):** apparent shear rate at a given value of shear stress
- R_C**(mm):** Capillary die radios
- X**:** Dimensionless parameter (a function of the shear stress)



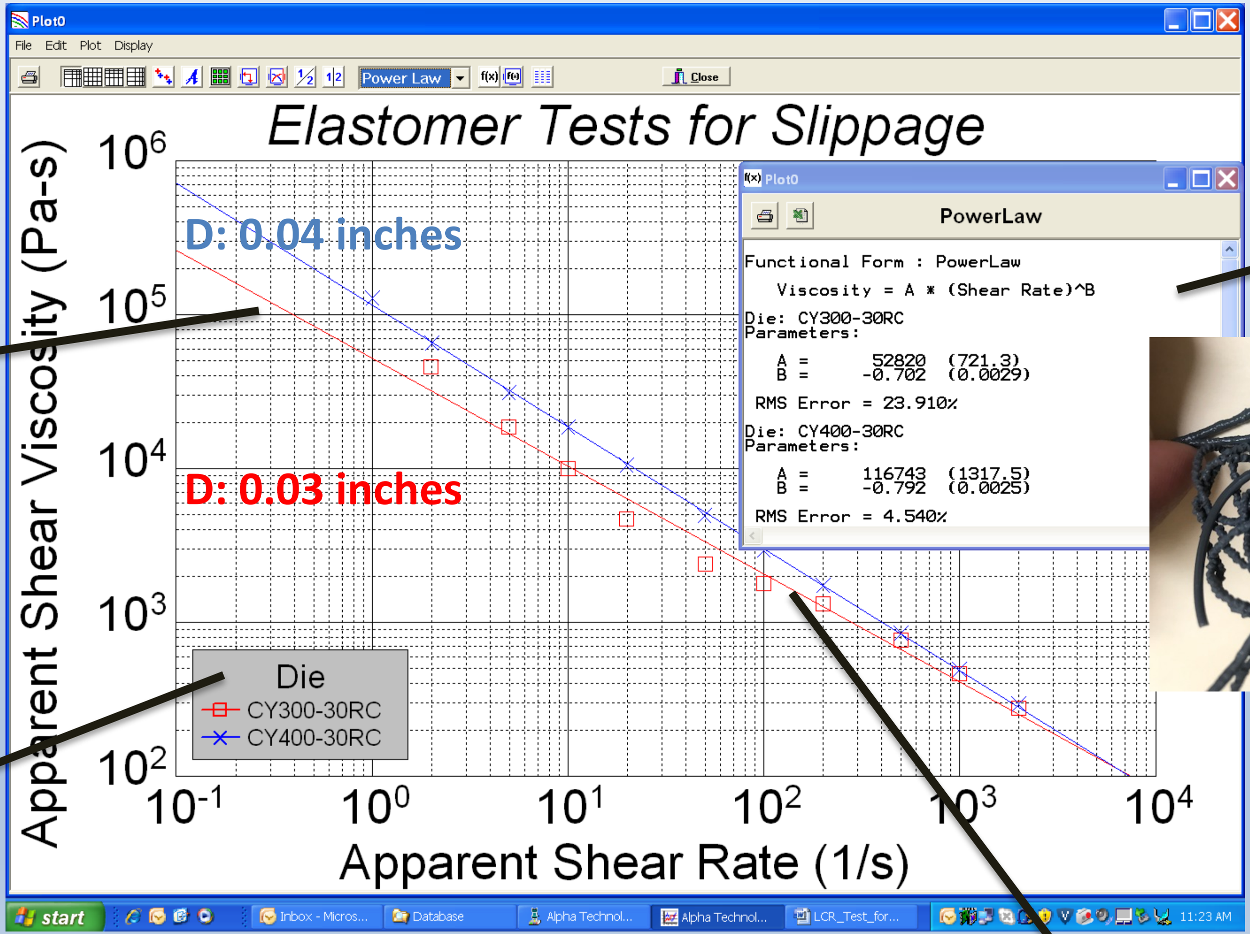
Steps:

1. Produce a series of flow curves using a set of dies of varying radius (R_C).
2. At a given value of shear stress, make a plot of apparent shear rate ($\dot{\gamma}_a$) versus inverse radius ($\frac{1}{R_C}$).
3. Slip velocity at a given shear stress will be one quarter of the slope of $\dot{\gamma}_a$-vs-$\frac{1}{R_C}$ plot.
4. Repeat the test at other shear stresses and calculate the slip velocity at each specific shear stress.
5. Make the plot of slip velocity versus shear stresse.

Dynisco

Reference:
J.M. Lupton, H.W. Regester, Melt Flow of Polyethylene at High Rates, *Polym. Eng. Sci.* **5**, 235-245 (1965)

How to Notice Wall Slippage from Flow Curve in LCR?



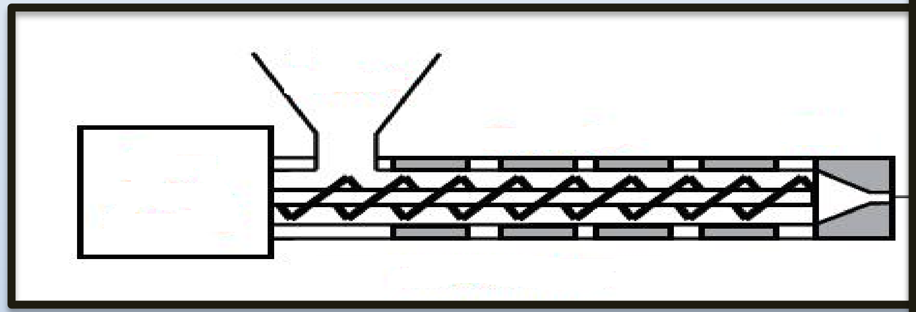
Slippage affect Power-Law model parameters

NOT overlapping of results from two dies means slippage happened at the smaller die

Perform shear rate sweep on two dies with same L/D but different diameters

Viscosity is lower when slippage happens in smaller die diameter.

Melt Fracture Trouble Shooting in Extrusion Process



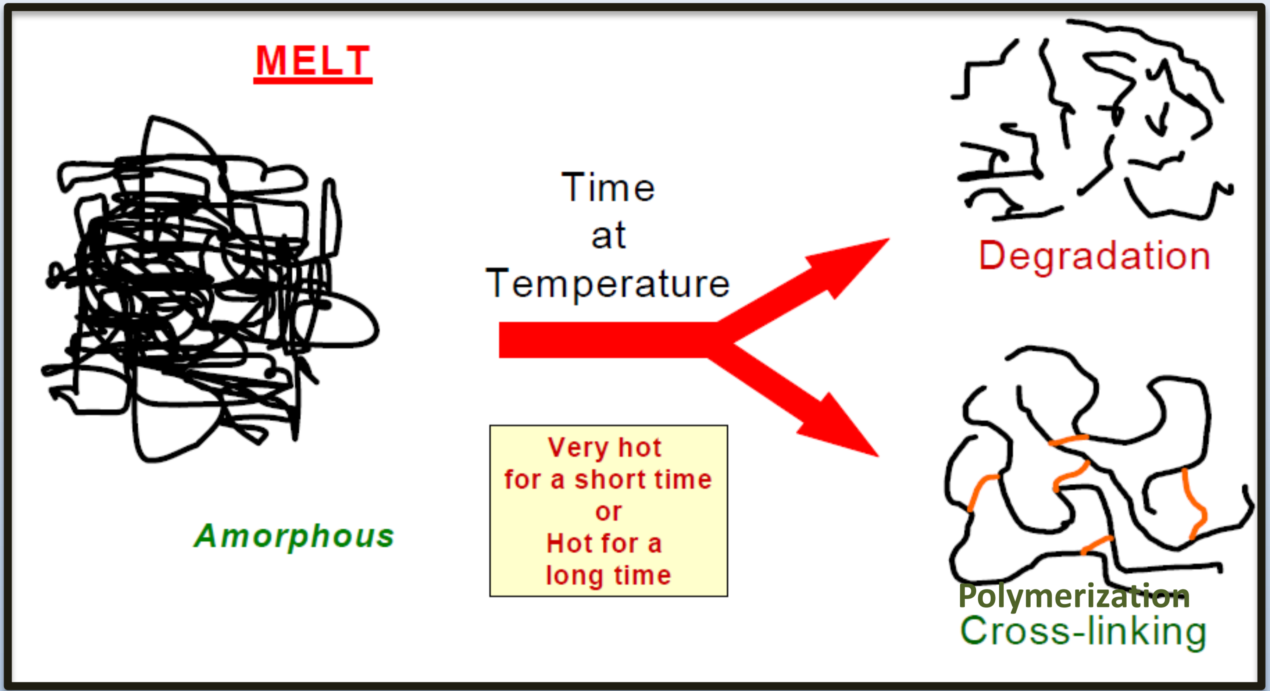
This phenomena happens due to elasticity of polymer melts. **Any procedure that reduces the melt elasticity will help to troubleshoot.** e.g.:

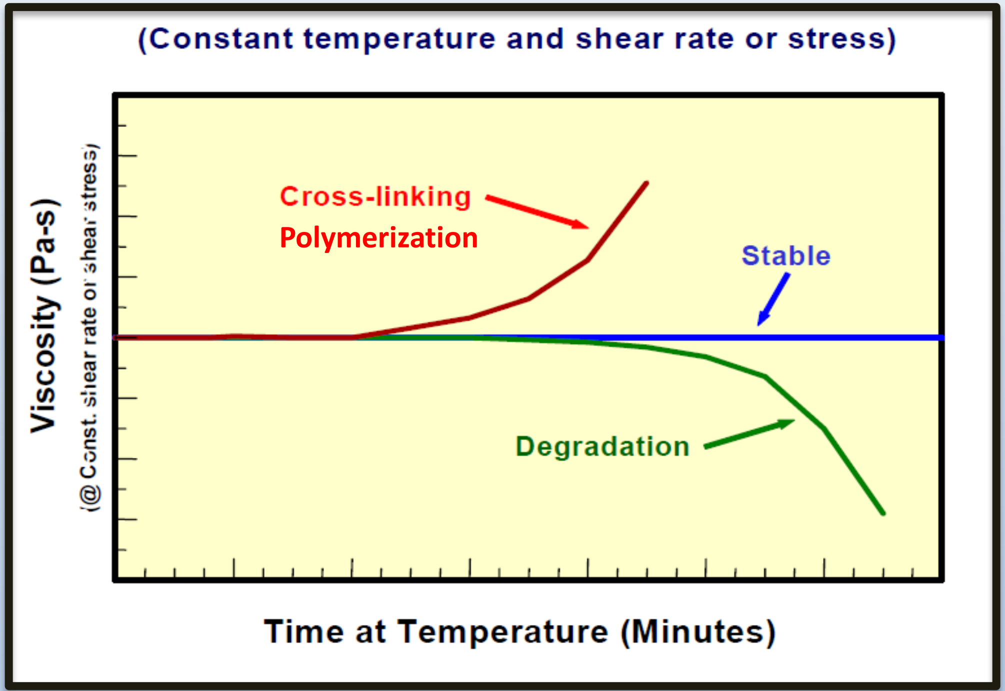
- ✓ Increasing temperature
- ✓ Reducing screw speed (shear rate)
- ✓ Increasing the die length or die diameter
- ✓ Tapering the die

Time Sweep Test

(Thermal Stability of Polymers)

Thermal Stability of Polymer Melt





- The stability test can determine the resistance of a material to a change in viscosity at the test temperature.
- The stability of polymer melts varies depending on temperature, time at temperature, formulation, and contaminants.
- This test can be used to show the presence of moisture or reactive chemicals in a polymer.
- This test can be used to measure the degradation rate or reactivity of a sample

Dynisco

How to Set up Thermal Stability Test (Time Sweep) in LabKars?

Setup

Data Point Setup # 11

Setup #	Program Name	Start Pos.	Temperature	Melt Time	Sensor1 ID	Die
2	7523 Control Grey PP	100	230	300	LC-103N	CY300-33
3	Polycarbonate	100	300	300	LC-103N	CY300-33
4	Position Based Test	90	190	300	LC-103N	CX300-33
5	Bagley Test	100	230	300	PT-142BAR-A	CX394-40
6	Thermalstability Test	100	285	120	LC-103N	CZ600-20
7	Nylon	90	240	240	LC-103N	CZ394-20
8	HDPE	90	90	360	LC-103N	CZ787-15
9	PEEK	100	400	300	LC-103N	CY400-15
10	PET I.V.	100	285	240	LC-103N	CX300-33
11	Nylon Stability	89	240	180	LC-502N	CZ394-20

Control Mode

- Rate
- Stress

Test Type

- Steady State
- Position (Acquire At - mm)
- Manual
- Time Delay (Acquire At - sec)
- Elapse Time (Acquire At - sec)

Minimum Speed 0.03

Maximum Speed 650

Point #	Speed Control	Shear Rate	Delay Sec	Time Min	Acquire At-mm
1	6.50	79.05			102.0
2	6.50	79.05			115.0
3	6.50	79.05			128.0
4	6.50	79.05			141.0
5	6.50	79.05			154.0
6	6.50	79.05			167.0
7	6.50	79.05			180.0
8	6.50	79.05			193.0
9	6.50	79.05			206.0
10	6.50	79.05			219.0

Select "Position" Test Type

Appling the same speed and shear rate multiple times

$$\Delta L = S \times \Delta t$$

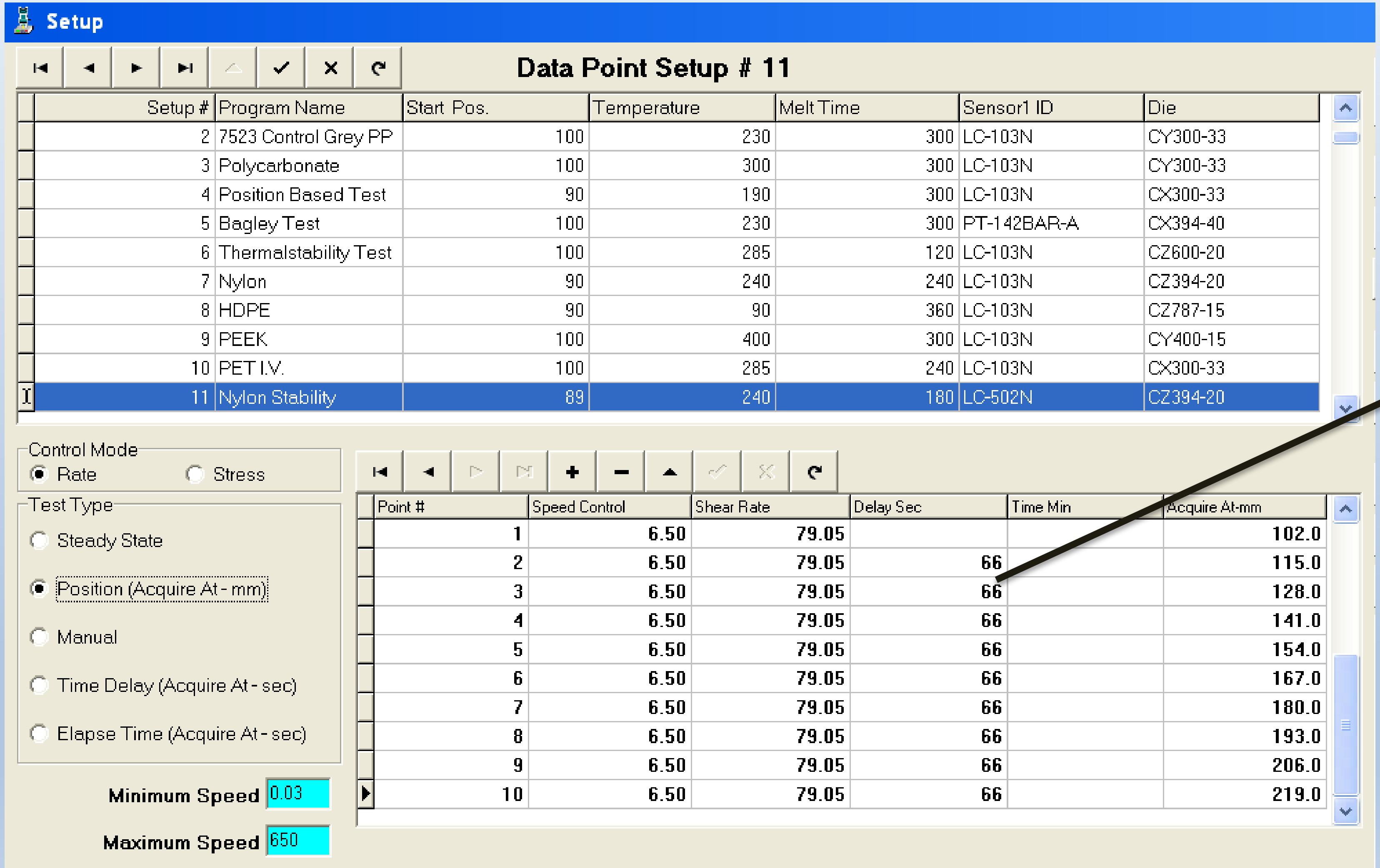
13 mm   6.5 mm/sec   2 sec

Where

- ΔL: **Piston travel distance between points**
- S: **Piston speed**
  $= \dfrac{\textit{Max pluner travel}\ (130\text{ mm})}{\textit{Test time}\ (\text{e.g.20 min})}$
- Δt: **Piston time travel between points**
  $= \dfrac{\textit{Test time}\ (\text{e.g.20 min})}{\#\ \textit{of points}\ (\text{e.g.10})}$

Dynisco

How to Set up Thermal Stability Test (Time Sweep) in LabKars?



Setup

Data Point Setup # 11

Setup #	Program Name	Start Pos.	Temperature	Melt Time	Sensor1 ID	Die
2	7523 Control Grey PP	100	230	300	LC-103N	CY300-33
3	Polycarbonate	100	300	300	LC-103N	CY300-33
4	Position Based Test	90	190	300	LC-103N	CX300-33
5	Bagley Test	100	230	300	PT-142BAR-A	CX394-40
6	Thermalstability Test	100	285	120	LC-103N	CZ600-20
7	Nylon	90	240	240	LC-103N	CZ394-20
8	HDPE	90	90	360	LC-103N	CZ787-15
9	PEEK	100	400	300	LC-103N	CY400-15
10	PET I.V.	100	285	240	LC-103N	CX300-33
11	Nylon Stability	89	240	180	LC-502N	CZ394-20

Control Mode
- Rate
- Stress

Test Type
- Steady State
- Position (Acquire At - mm)
- Manual
- Time Delay (Acquire At - sec)
- Elapse Time (Acquire At - sec)

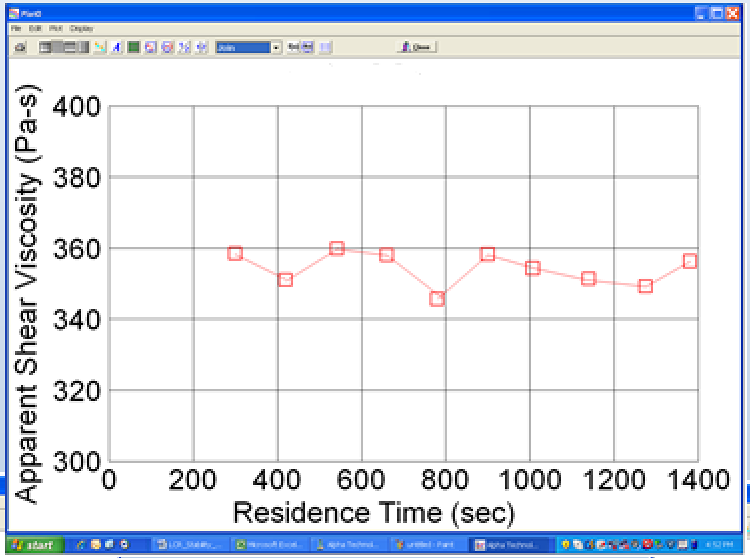
Minimum Speed 0.03

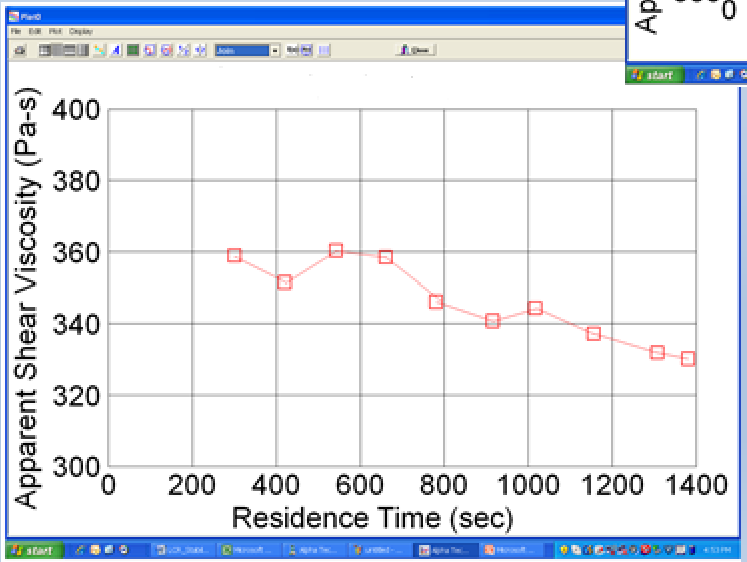
Maximum Speed 650

Point #	Speed Control	Shear Rate	Delay Sec	Time Min	Acquire At-mm
1	6.50	79.05			102.0
2	6.50	79.05	66		115.0
3	6.50	79.05	66		128.0
4	6.50	79.05	66		141.0
5	6.50	79.05	66		154.0
6	6.50	79.05	66		167.0
7	6.50	79.05	66		180.0
8	6.50	79.05	66		193.0
9	6.50	79.05	66		206.0
10	6.50	79.05	66		219.0

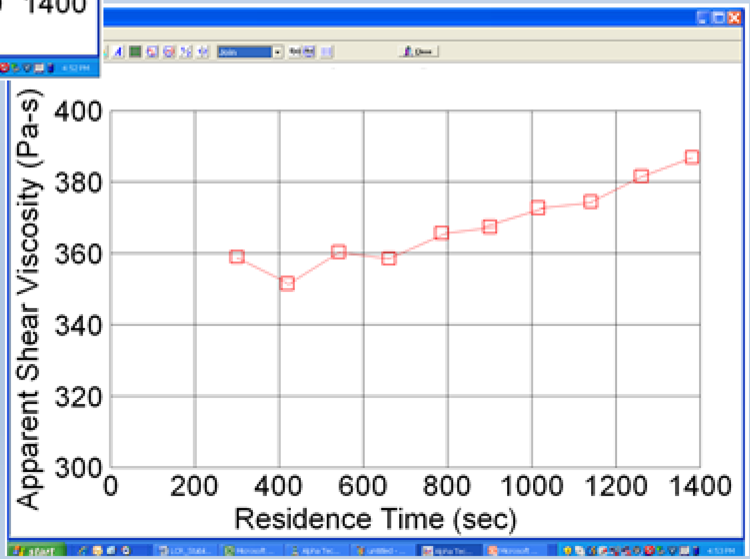
Add delay time to increase the stability test time while keeping shear rate constant

Thermal Stability Test Results (Time Sweep) in LabKars



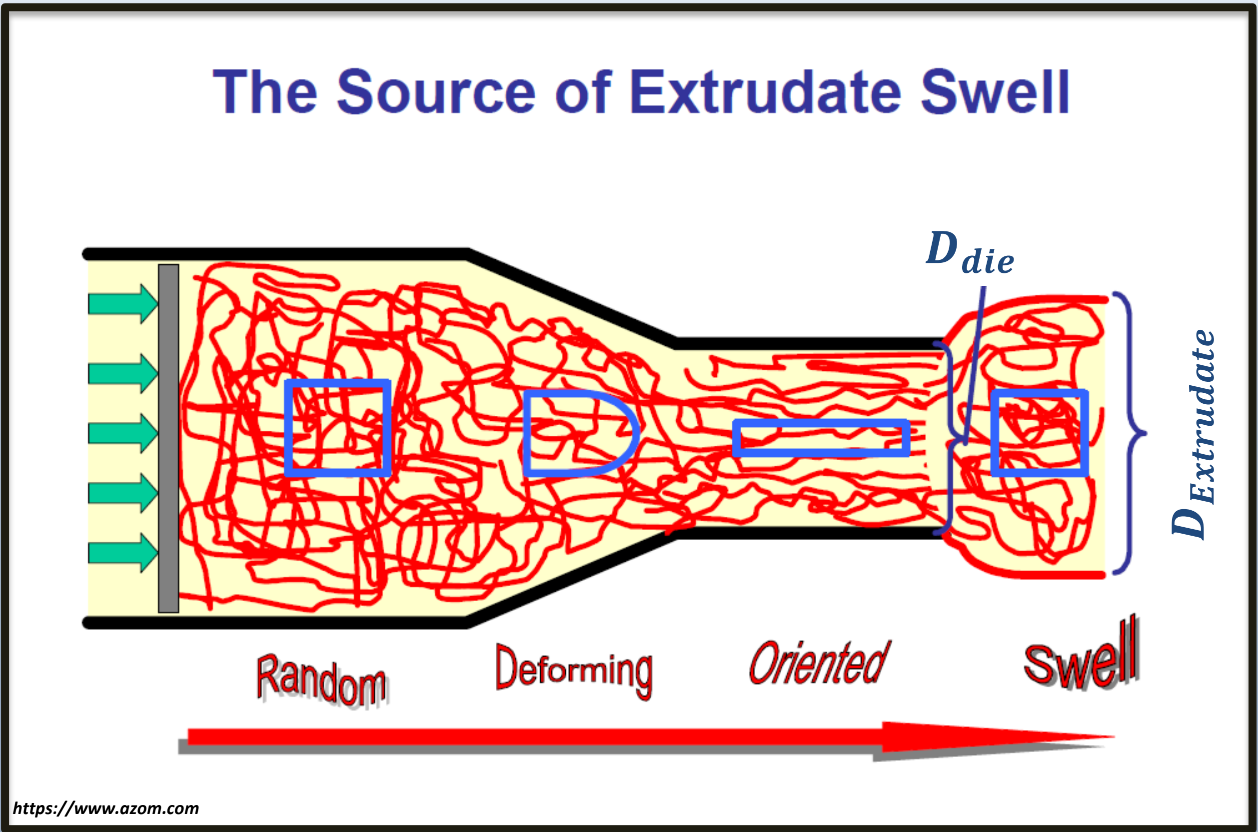


Plot of apparent viscosity versus residence time



Die Swell

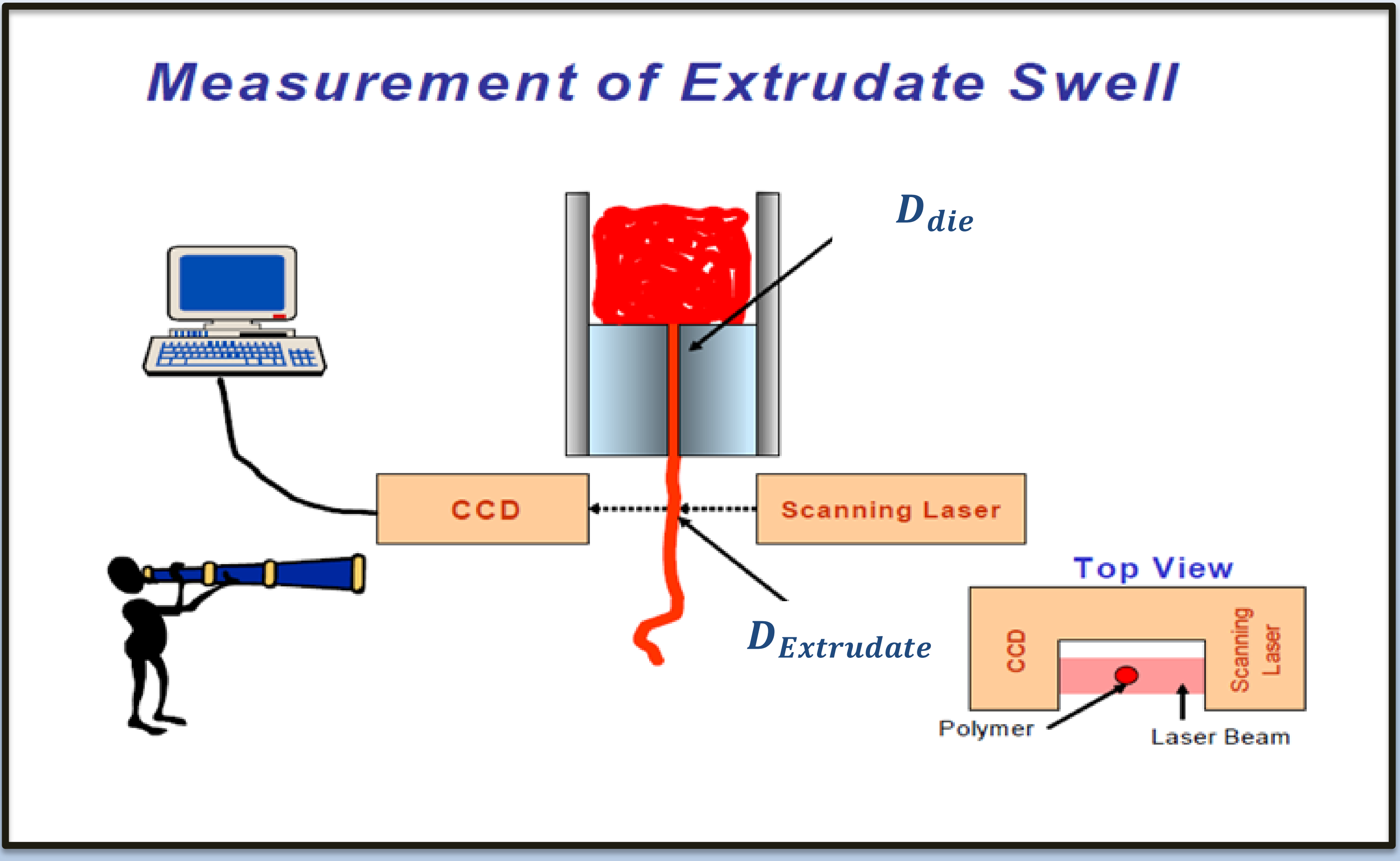
Die swell Ratio



$$Percent\ die\ swell = \frac{D_{Extrudarte} - D_{die}}{D_{die}} \times 100$$

- Expansion (re-coiling) of extrudate after exiting die.
- Qualitative measure of melt elasticity.
- Relaxed die swell is used to predict part dimension
- Running die swell is used to predict productivity of extrusion process.
- Analysis of extrudate smoothness.
- Trouble shooting by using larger die diameter, longer die, tapering the die, lower shear rate, or higher temperature.

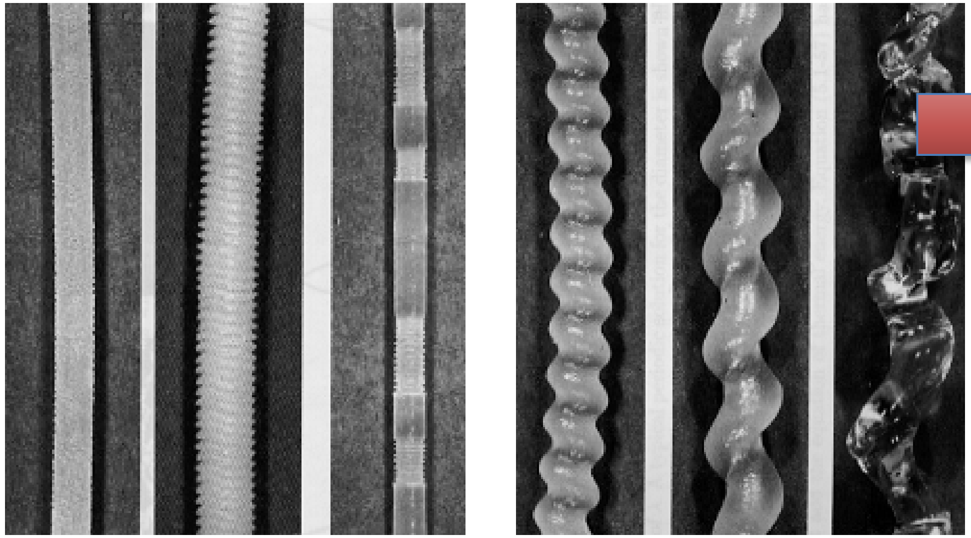
Die Swell Measurement in LCR



- Detection: CCD element
- Light source: 800 nm laser
- Resolution: 2.75 µm
- Measuring range: 0.13-23 mm
- Response time: 1.4 ms
- Accuracy: $\pm$0.003 mm

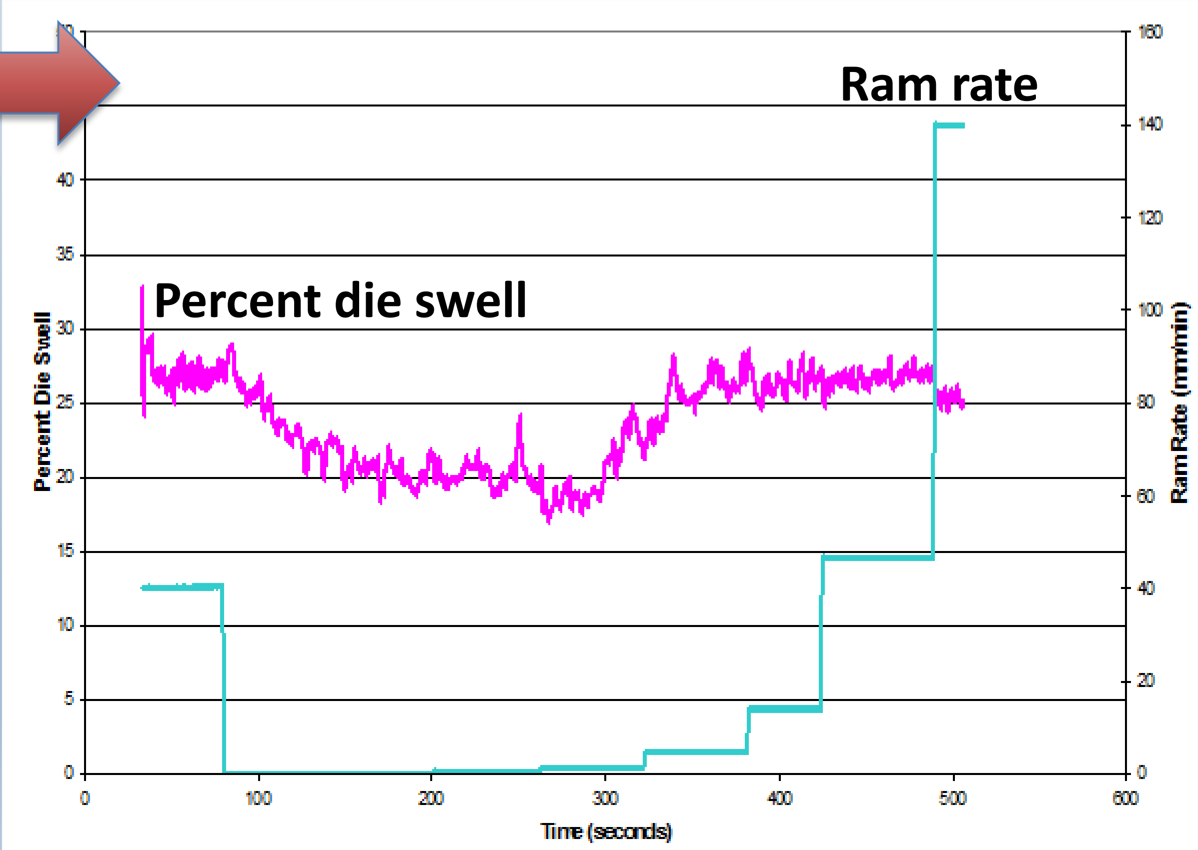
Die Swell Measurement in LCR

Melt Fracture

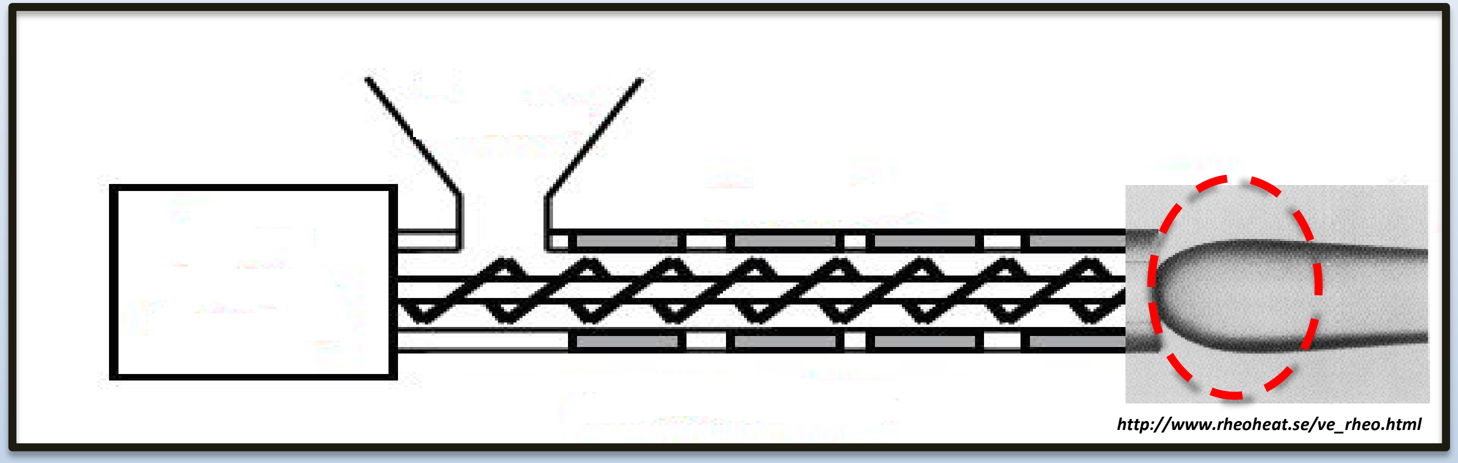


https://www.azom.com/article.aspx?ArticleID=13578

Melt fracture produce a noisy die swell measurements.



Die Swell Trouble Shooting in Extrusion Process



This phenomena happens due to elasticity of polymer melts. **Any procedure that reduces the melt elasticity will help to troubleshoot.** e.g.:

- ✓ Increasing temperature
- ✓ Reducing screw speed (shear rate)
- ✓ Increasing the die length or die diameter
- ✓ Tapering the die

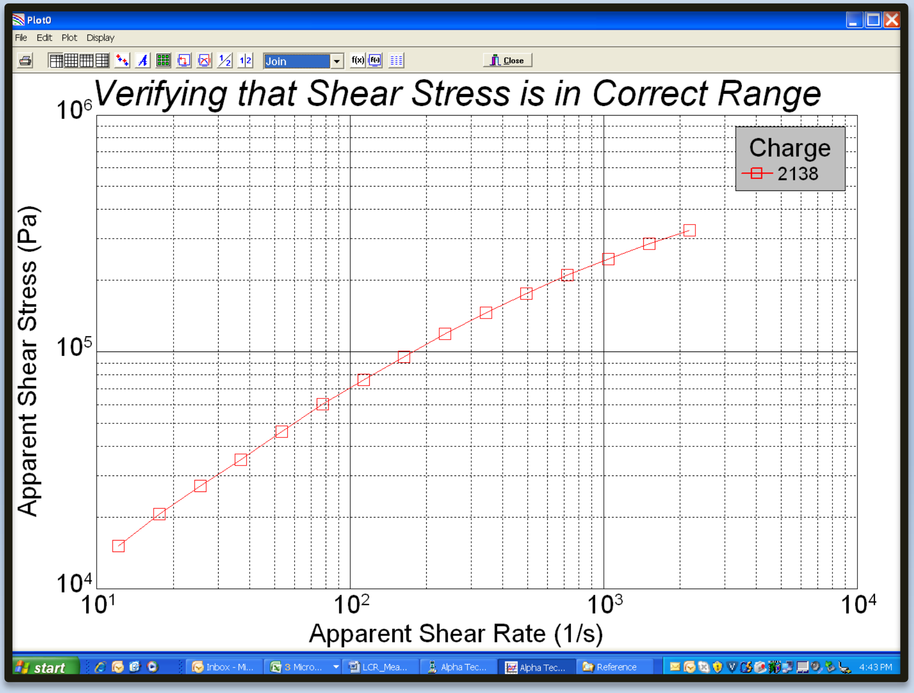
MFR Correlation

How to Calculate MFR from Capillary Rheometer?

1. Calculate the shear stress in the melt flow rate tester (using a standard die)

2. Determine the shear rate achieved at this shear stress from material flow curve

Weight in MFR test (kg)	Shear stress (Pa)
2.16	19350
5.00	44792
10.00	89584
21.60	193502



Dynisco

How to Calculate MFR from Capillary Rheometer?

3. Calculation of melt volume rate (MVR) as follow:

$$\boldsymbol{MVR = 600\dot{\gamma}\frac{\pi R^3}{4}}$$

where

- $MVR\ (cm^3/10\text{min})$: Melt volume rate
- $\dot{\gamma}\ (\frac{1}{s})$: Shear rate determined at "step 2"
- $R\ (cm)$: Standard melt flow rate tester die radius

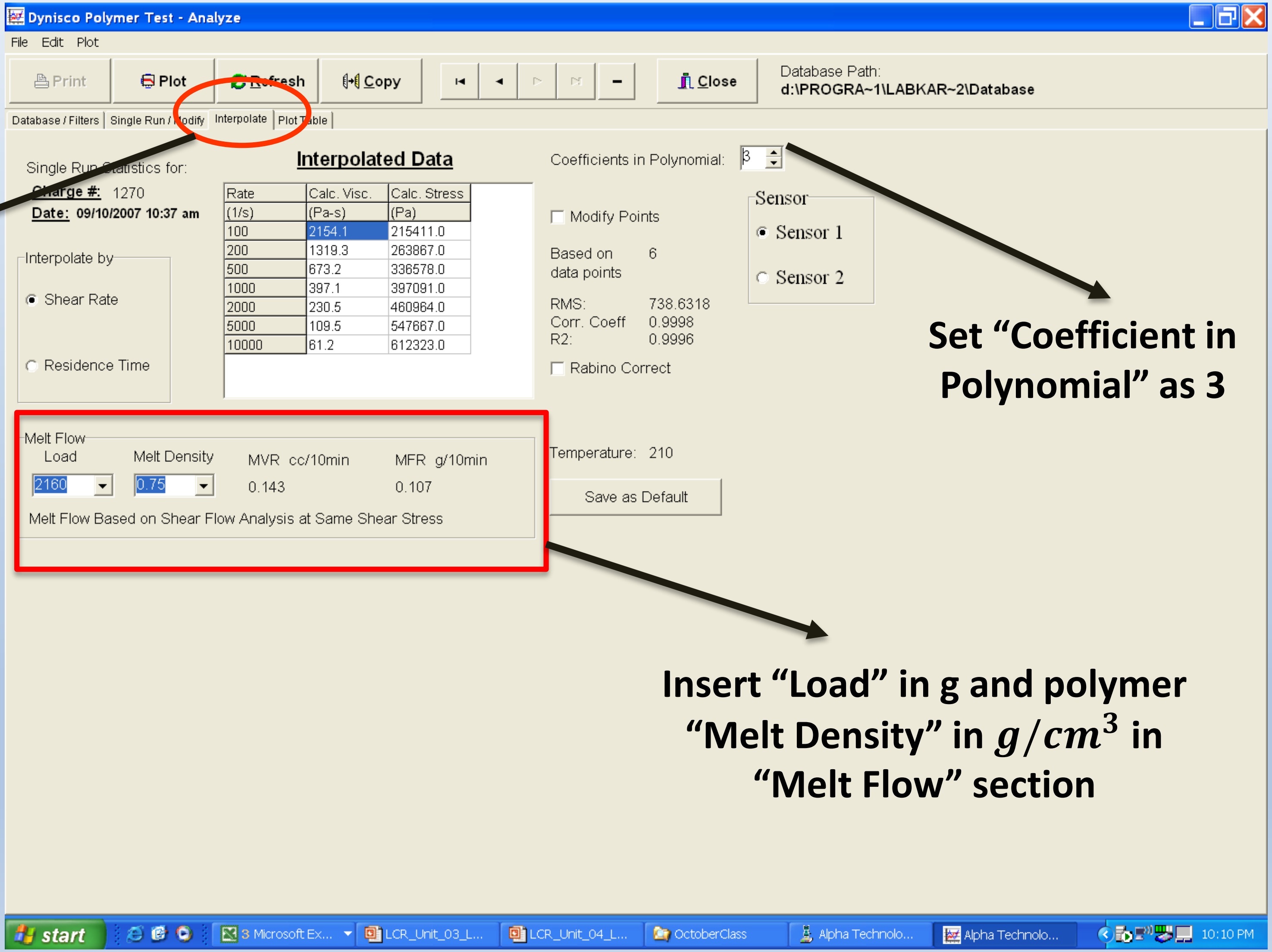
4. MFR calculation knowing polymer melt density as follow:

$$\boldsymbol{MFR = MVR \times \rho_m}$$

where

- $\rho_m(\frac{g}{cm^3})$: Polymer melt density

How to Measure MFR in LabKars?



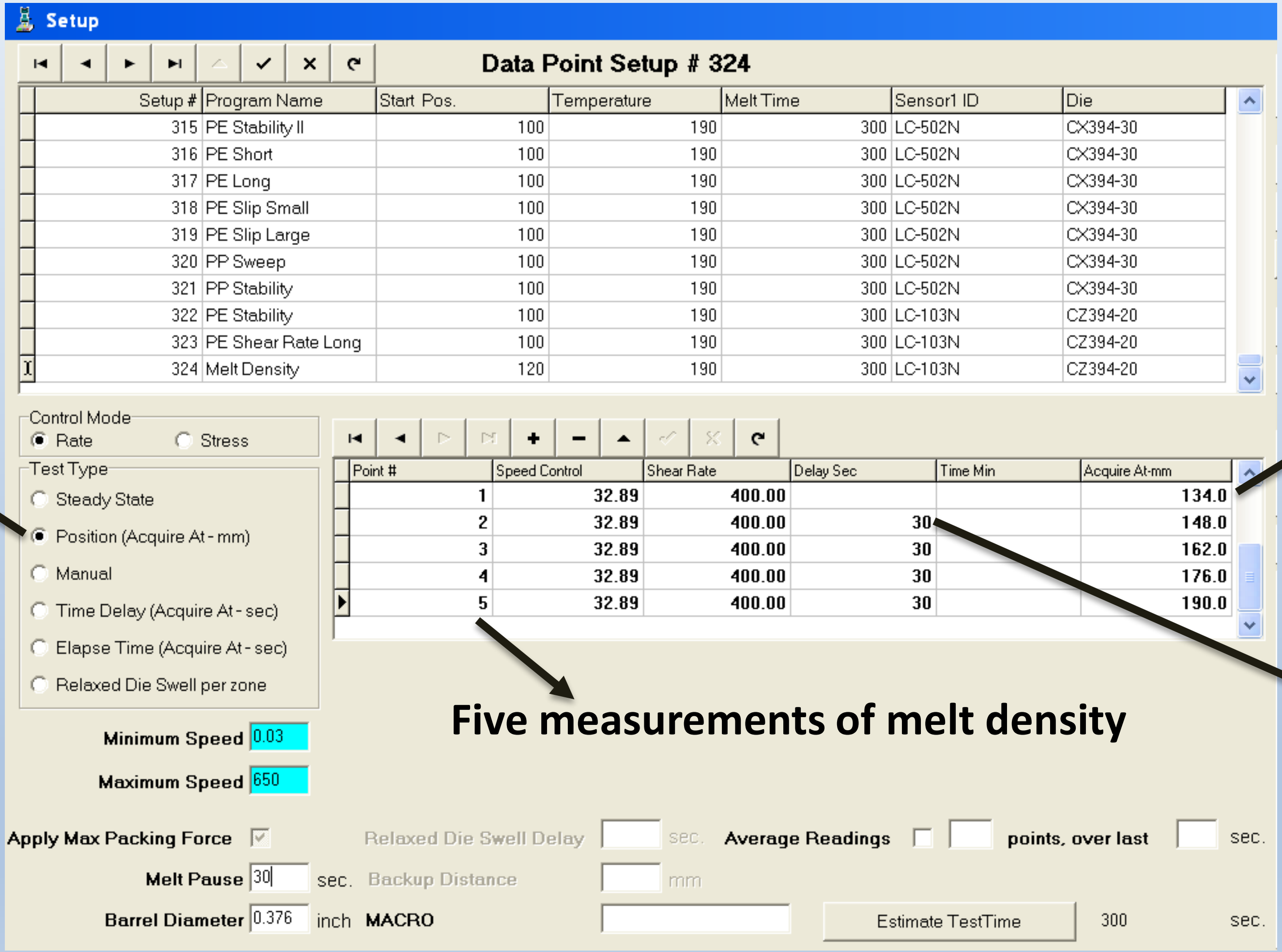
Select "Interpolate"

Set "Coefficient in Polynomial" as 3

Insert "Load" in g and polymer "Melt Density" in g/cm^3 in "Melt Flow" section

How to Measure Melt Density in LCR?

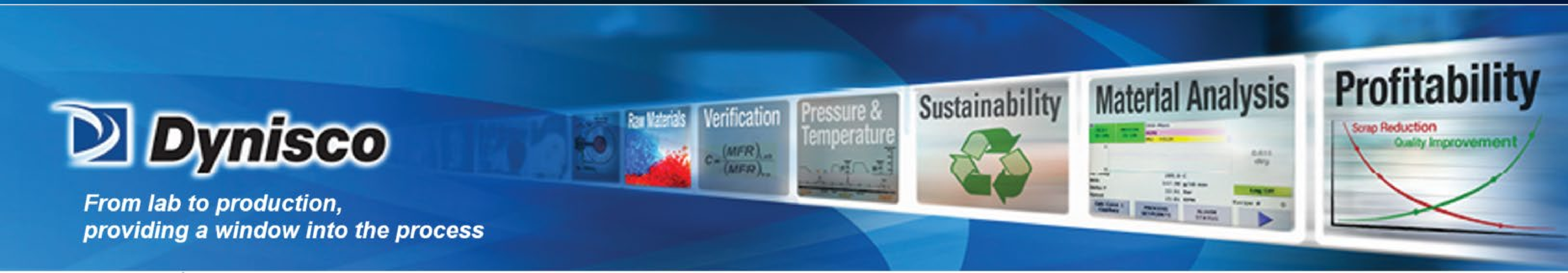
Select "Position" Test Type



Five measurements of melt density

Manage the positions to have 14 mm of piston travel distance between points

Whenever the plunger is in its delay time, cut the extrudate and weigh it



Intrinsic Viscosity of PET

What is Intrinsic/Solution Viscosity (IV)?

http://alokrj.weebly.com/dpt-3rd-sem.html

- Creating a dilute solution of the polymer and comparing the flow rate of the solution to the flow rate of the pure solvent

- <u>**Advantages**</u>
  - Performing at room temperature
  - No need to melt the polymer
  - No need to dry hygroscopic polymers (e.g. PET, PA)
  - Common flowability specification (rather than MFR) among the manufactures of hygroscopic and filled polymers

- <u>**Disadvantages**</u>
  - Delicate apparatus
  - Using of noxious chemicals as solvent
  - Not environmental friendly

Dynisco

Relationship between Melt Viscosity and Intrinsic Viscosity

Melt viscosity $\eta_{melt} \, \alpha \, M_W$

Fox-Flory Equation

$$\eta_0 = K_1 (M_W)^{3.4}$$

$$ln[\eta_0] = lnK_1 + 3.4ln[M_W]$$

Intrinsic viscosity $\eta_{Intrinsic} \, \alpha \, M_W$

Mark-Houwink Equation

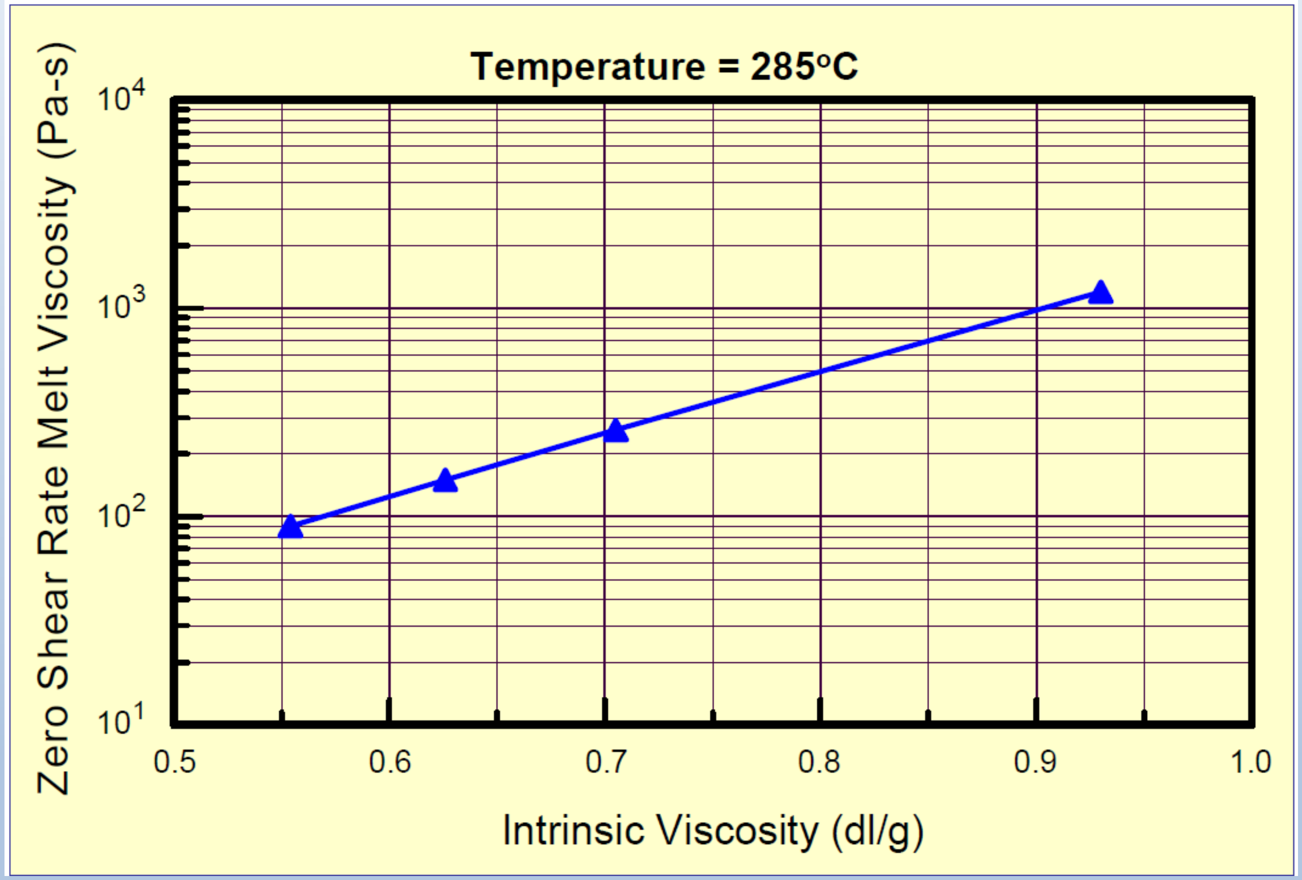
$$\eta_{Intrinsic} = K_2 (M_W)^a$$

$$ln[\eta_{Intrinsic}] = lnK_2 + aln[M_W]$$

L.H. Sperling, *Introduction to Physical Polymer Science*, John Wiley & Sons, 4th Edition.

$$ln[\eta_{Intrinsic}] = \frac{a}{3.4} \ln[\eta_0] + K''$$

PET Melt Viscosity Versus Intrinsic Viscosity

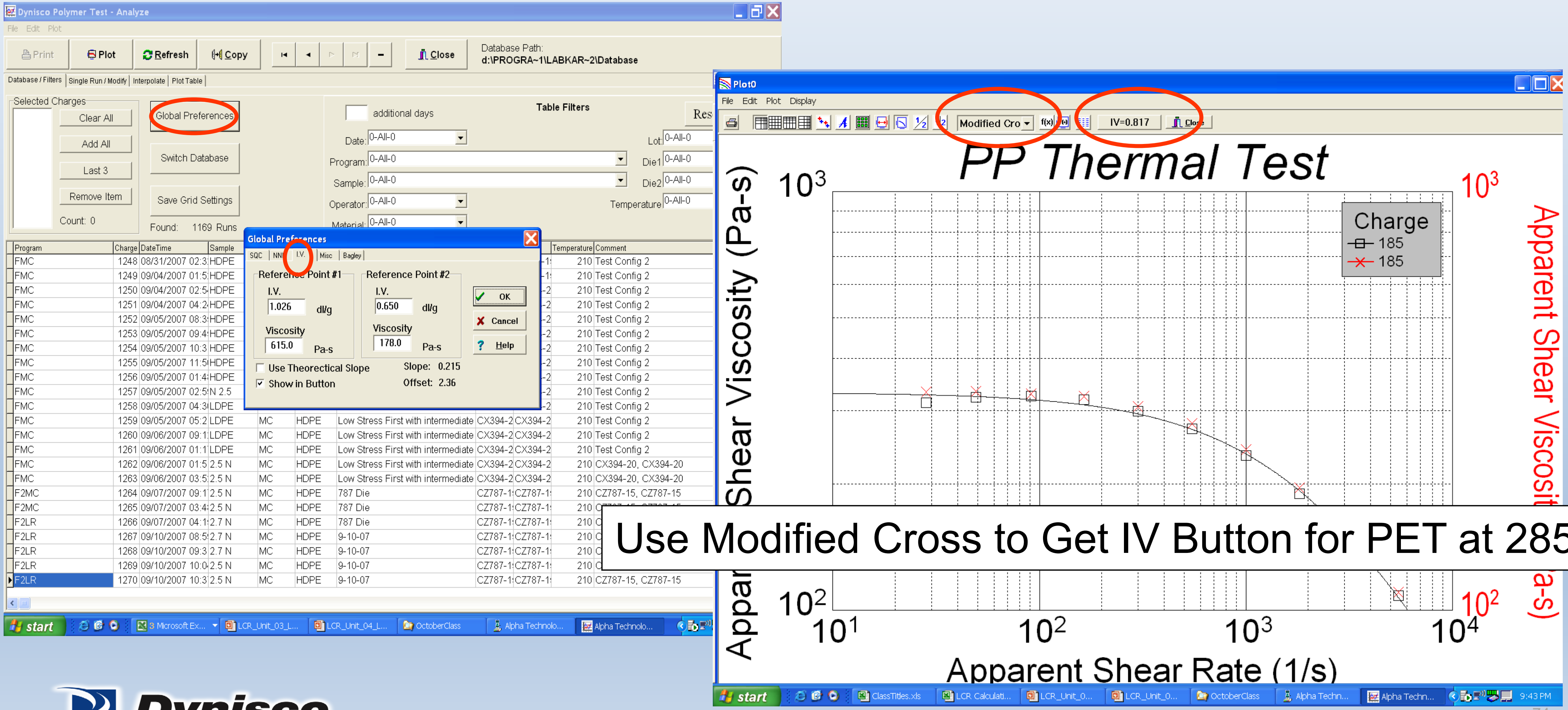


$$\boldsymbol{ln[\eta_{Intrinsic}] = \frac{a}{3.4}\ln[\eta_0] + K"}$$

Slope: $\frac{a}{3.4}$ → For PET: a=0.75

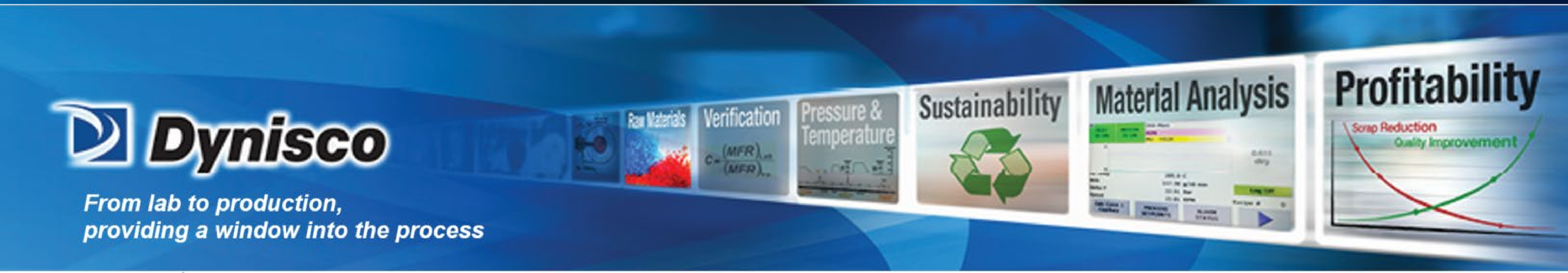
Intercept: $K"$

How to Determine IV of PET in LCR Capillary Rheometer?



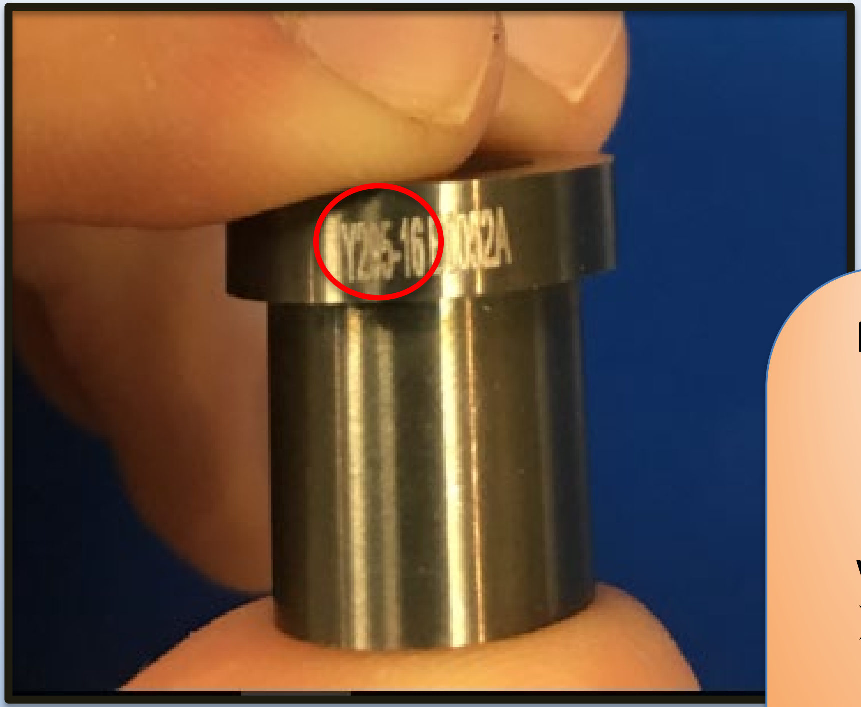
Use Modified Cross to Get IV Button for PET at 285 C

Dynisco



LCR Dies Information

LCR Die Part Number Code



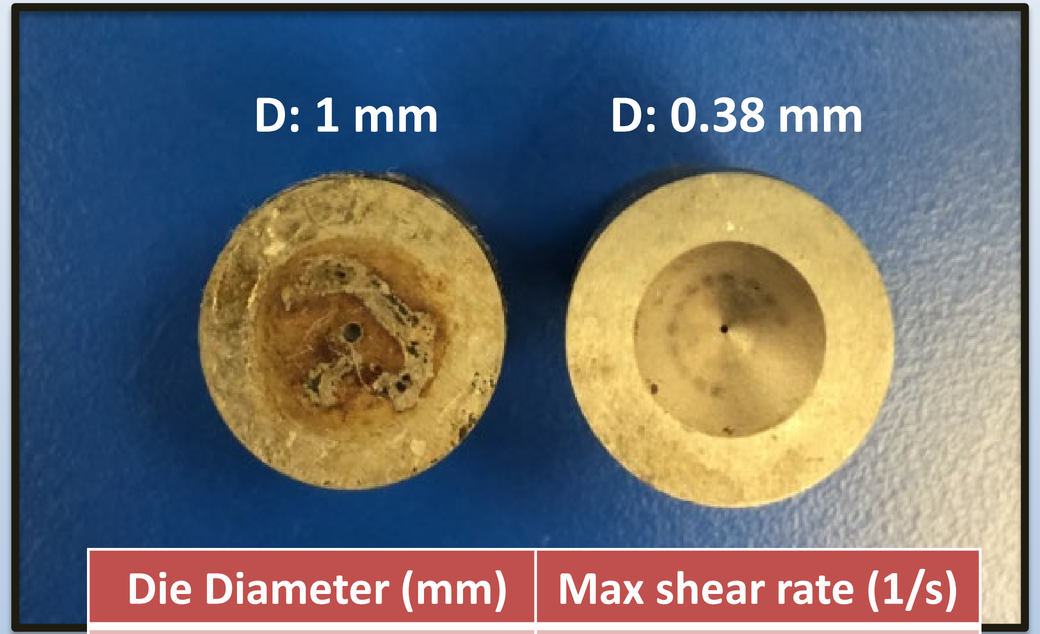
Formula for die part number:

ADDD-LL

where

- A: entry angle
  (W=60°, Y=90°, X=120°, Z=180°)
- DDD: diameter in inches×10000
- LL: length to diameter ratio

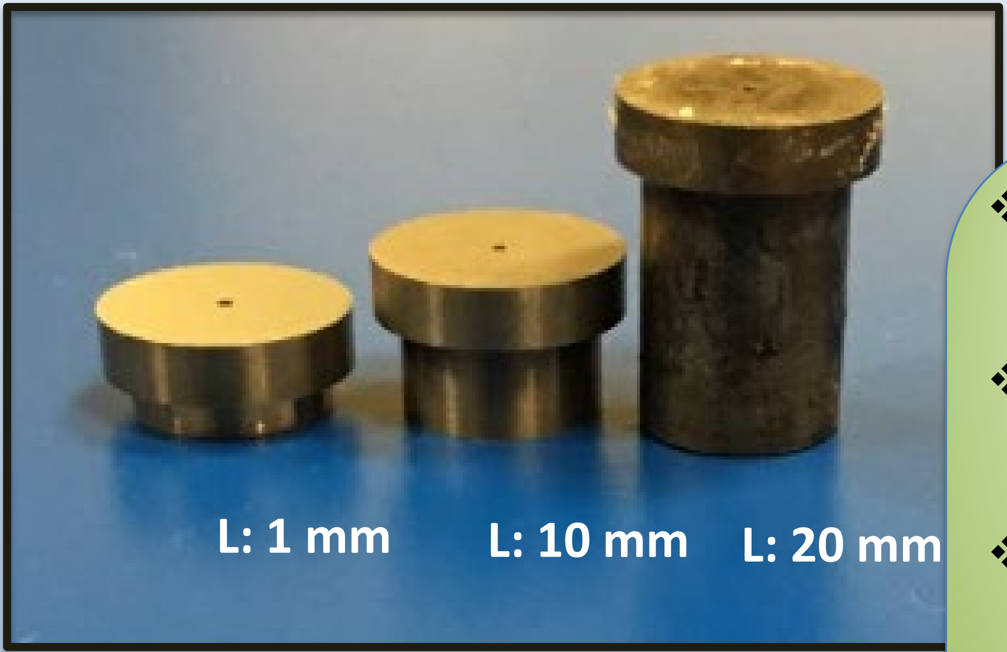
Die Diameter



Die Diameter (mm)	Max shear rate (1/s)
0.38	142000
0.75	18700
1.00	7900
1.50	2300
2.00	980

- Smaller diameter produces higher shear rates.
- Larger diameter causes less elastic deformation applied at the entrance of the die.
- larger diameter cause less entrance pressure drop, less die swell, less extensional deformation, and less slippage.
- For calculation of wall slip velocity at least 2 dies with different diameters (same L/D) are required.

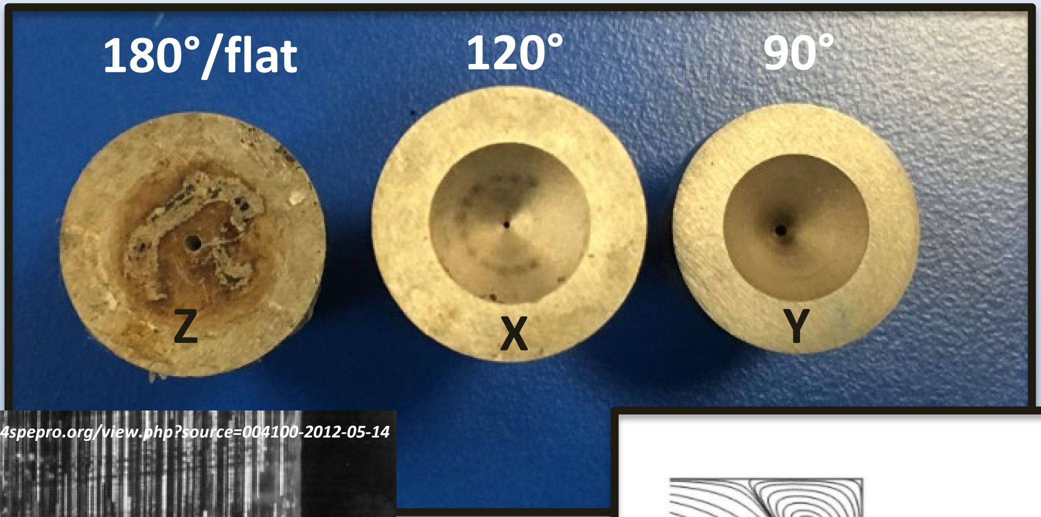
Die Length

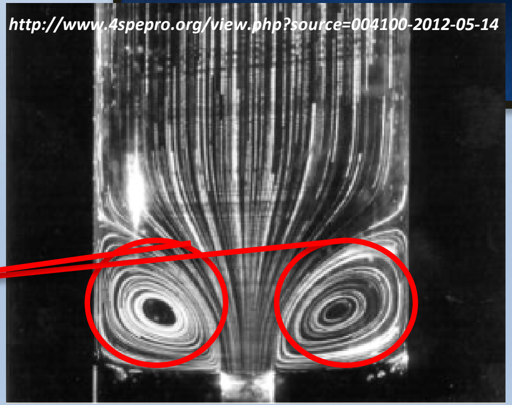


- The portion of fully developed flow in compare with entrance region increases with increasing the die length
- The percent error produced by entrance region is less with longer die.
- Short die for measuring elastic deformation (e.g. die swell, slippage) and long die for measuring shear viscosity.
- Noisy reading from short die at very low shear rates
- For calculation of entrance pressure (Bagley correction) and extensional viscosity, at least 2 dies with different L/D ratio (same diameter) are required.

Die Entrance Angle

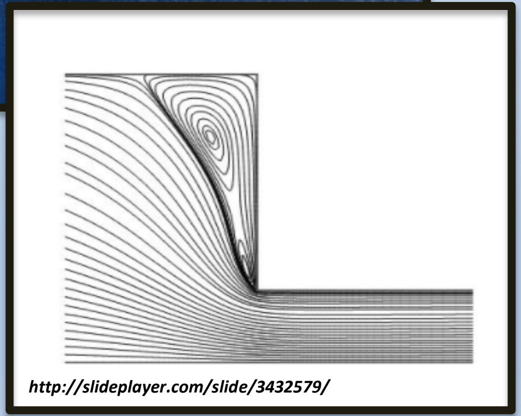
- Entrance angle has effect on flow patterns at the entrance to the capillary die.
- Lower angle causes smoother flow, less vortex flow, and less energy consumption at the die entrance.
- Lower angle reduces the elastic deformations (e.g. less entrance pressure drop, extensional deformation, die swell, melt fracture and wall slippage).
- Lower angle favors shear flow.

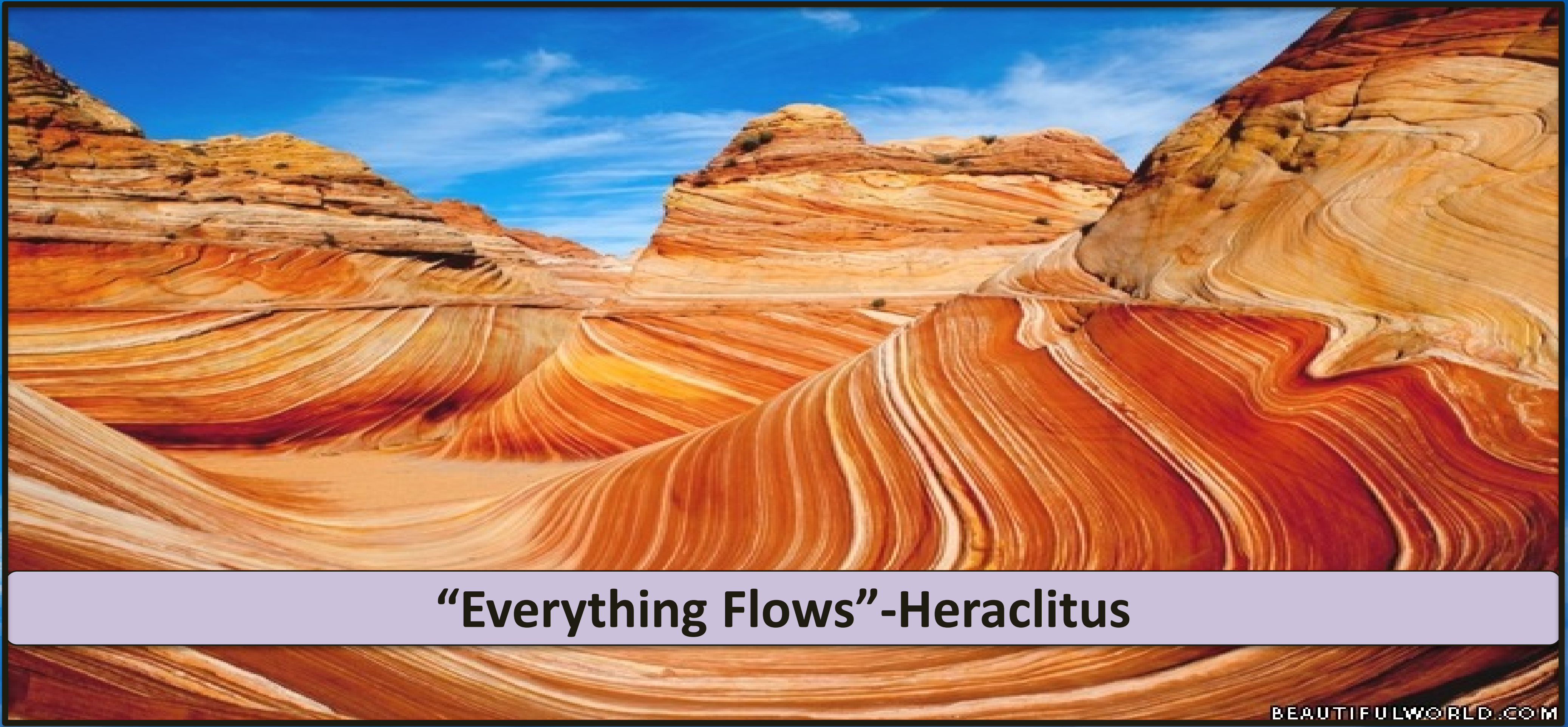




Stationary vortex flows in the corner region

Flow pattern at the entrance to a die with a flat entrance (180°)





"Everything Flows"-Heraclitus

QUESTIONS ?