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From lab to production,
providing a window into the process

Introduction to Capillary Rheometers

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Characteristics of Thermoplastic Polymers

- Complex material
- Has a softening and / or melt temperature
- Often processed in its melted form
  - Extrusion
  - Injection Molding

Effect of Increasing Polymer Molecular Weight on a Polymer Melt

MW

Increases ↓

Low Viscosity Liquid

Medium Viscosity Liquid

High Viscosity Liquid

Reasons to Test Rheology of Thermoplastic Polymers

- Polymers are very complex
- Polymer consistency is hard to control
- Rheology or flow of polymers is dependent on:
  - Molecular characteristics
  - Polymer / Filler Interaction
  - Time at temperature
- Rheology measurements are quick compared to alternatives

Definition of Different Types of Viscosity Measurements

- Viscosity – Measurement of the ability of a fluid to resist flow. Materials with higher viscosities require more energy / force to move them [LCR].
- Apparent Viscosity – Capillary rheometer measurement of viscosity without any corrections [LCR and LMI].
- Intrinsic Viscosity – The effect of PET on the viscosity of a solution of PET and a solvent. The result provides a measure of the Molecular Weight of the PET.
- Inherent Viscosity – The effect of Nylon on the viscosity of a solution of Nylon and a solvent. The result provides a measure of the Molecular Weight of the Nylon.

Some Common Thermoplastics [Test Temperature]

- Polyolefins
  - Polyethylene (PE) [190 C]
  - Polypropylene (PP) [230 C]
- Nylon (PA or Polyamide) [230 C or 265 C]
- Polycarbonate (PC) [300 C]
- Polyetherterephtalate (PET) [285 C]

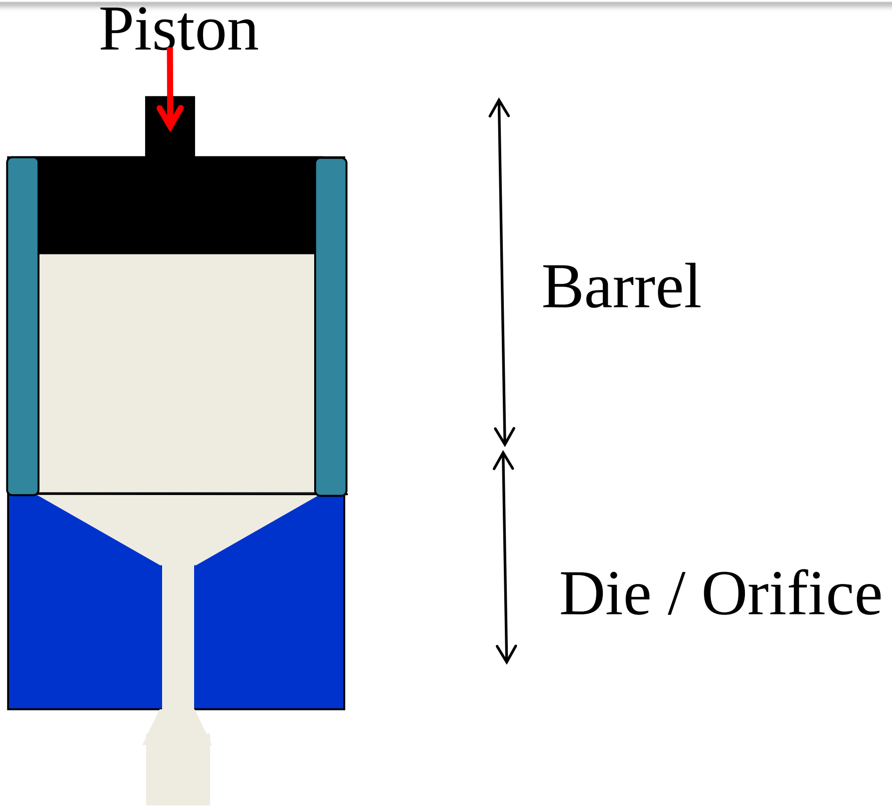
Ideal Requirements for Testing Rheological Properties of Polymer Melts

- Measure flow properties of polymers at process temperatures
- Determine the effect of flow speed on the polymer flow properties
- If possible, measure flow properties at the same speed as the process

Requirements are Met with a Capillary Rheometer

- Holds temperature within required range
- Measures change in properties with change in flow speed
- Can cover polymer flow speeds used in processing

Diagram of a Basic Capillary Rheometer



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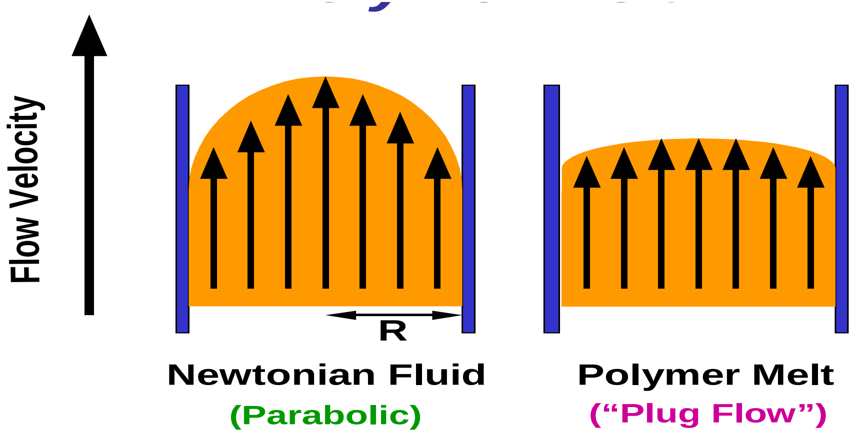
Melt Flow in an Orifice Die



Material in center flows faster than the material near the capillary wall.

Range of Samples

- Pellets
- Powders

Material must be melted in order to be tested on a capillary rheometer.

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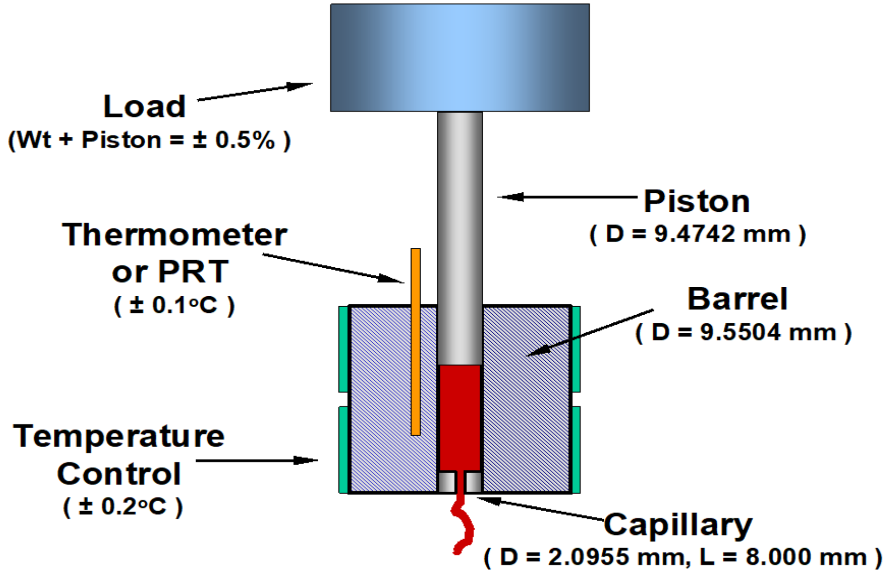
Capillary Rheometer Test

- Load Sample into Barrel and Tamp Material
- Ensure Correct Volume of Sample is Present
- Allow sample to warm up / melt to barrel temperature for a fixed time period
- Push sample out through orifice die
- Take measurement(s)

Controlled Stress (or Weight) Capillary Rheometer



Technical Names for Controlled Stress Test

- Melt Flow Rate (MFR)
  - When 20 kg weight used then MFR test is HLMI or High Load Melt Index
- Melt Flow Index (MFI) (flow rate of PE obtained under 190 C and 2.16 kg)
- Melt Index (MI)
- Melt Volumetric Rate (MVR)
  - Preference by European Companies
  - Simplified testing with digital encoder

Melt Flow Rate (MFR) Data

- Grams of Polymer Melt Flow in 10 minutes
- Requires a balance (3 decimal preferred, 2 decimal adequate)
- Viscosity
    - Can be calculated
    - Rarely used
- Melt flow rate is inverse of viscosity
    - Higher Melt Flow Rate means lower viscosity
    - Lower Melt Flow Rate means higher viscosity

Purpose for Melt Flow Rate Test

- Relative measure of viscosity
  - Higher value indicates lower viscosity
- Inexpensive method to measure viscosity
- Rapid test
- Well established in industry

MFR Varies With:

- Weight
- Temperature
- Viscosity (sample dependent)
- Die Diameter
- Die Length

- Need for Standardization

Melt Flow Rate Test

- ASTM D1238
  - Met by LMI 4000 Series
- Capillary Rheometer
  - Die Diameter: 2.095 mm
  - Die Length: 8 mm
  - Plunger Weight: Depends on Polymer / Temperature

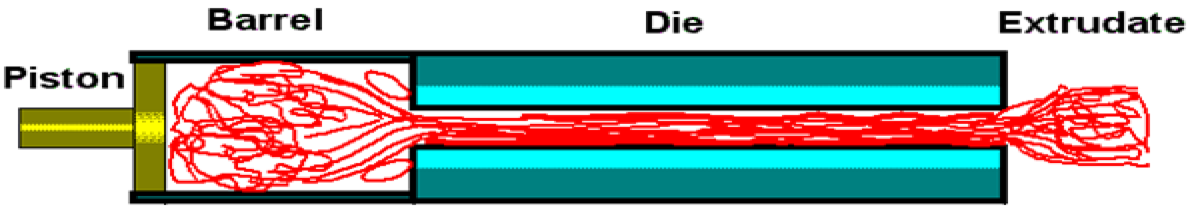
MFR Sensitive to:

- Viscosity of polymer melt
- Process aids
- Lubricants
- Molecular weight
- Molecular weight distribution
- Degradation
- Moisture in material
- Crosslinking agents

Disadvantages of Melt Flow Rate Test

- Labor Intensive
  - Someone must always watch the instrument
- Poor Repeatability
  - Test is subjective especially with "Fractional Melts" or low values.
- Single point measure
- Shear rate of test depends on viscosity
  - Cannot run a constant shear rate test

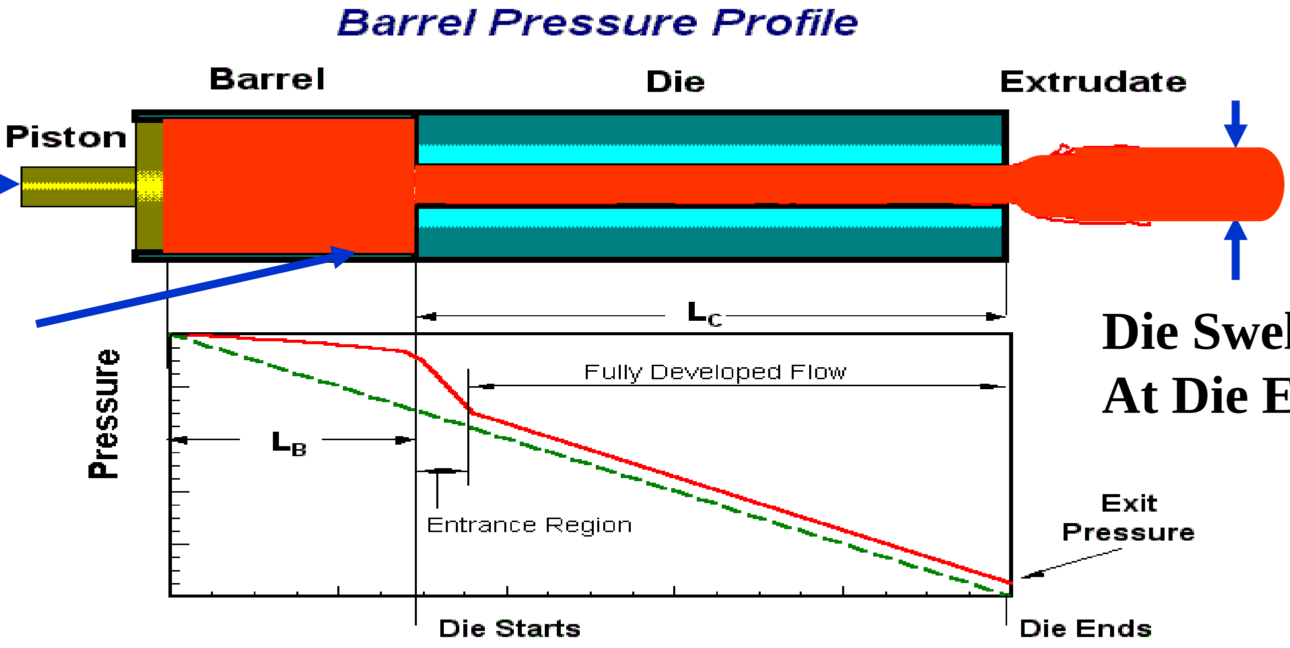
Schematic of Capillary Rheometer Is similar to Melt Flow Rate Device



However, the difference is in how the piston speed is controlled and the type of data collected.

Controlled Rate Capillary Rheometer

Force Measured At Piston



Pressure Measured In Barrel Near Die

Die Swell Measured At Die Exit

SPEED OF PISTON IS CONTROLLED DURING TEST

Controlled Rate Capillary Rheometer

- ASTM D3835
- Barrel and Die dimensions are not specified. Need to be determined before purchase.
- Specific polymers do not have defined test conditions as with LMI.

LCR 7001 General Specifications

- Barrel
  - Diameter: 9.55 mm
  - Available Length: 227 mm
  - Working Length: 125 mm
- Piston Speed
  - Mininum: 0.03 mm/min
  - Maximum: 650 mm/min
- Force Measurement
  - Load Cell (at piston): 10 kN
  - Pressure Transducer in Barrel: 140 Mpa
- Sample Volume: 10 cm^3

LCR Capillary Rheometer Data

- Viscosity (Viscous)
- Die Swell (Elastic)
- Extensional Viscosity (Viscoelastic)

Range of Samples

- Pellets
- Powders
- Elastomers cut into thin slices

Material must be melted in order to be tested on a capillary rheometer.

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LCR 7000 Data

- Measurements
  - Force (N, pounds force, kilograms force)
  - Ram Rate (mm/minute)
  - Pressure (kPa, psi)
  - Time (minutes, seconds)
  - Temperature (C, F)
  - Die Swell (mm)
- Calculated Data
  - Shear Rate (1/sec)
  - Shear Stress (Pa)
  - Viscosity (Pa-sec)
  - Die Swell (percent)

LCR 7001 Test Capabilities

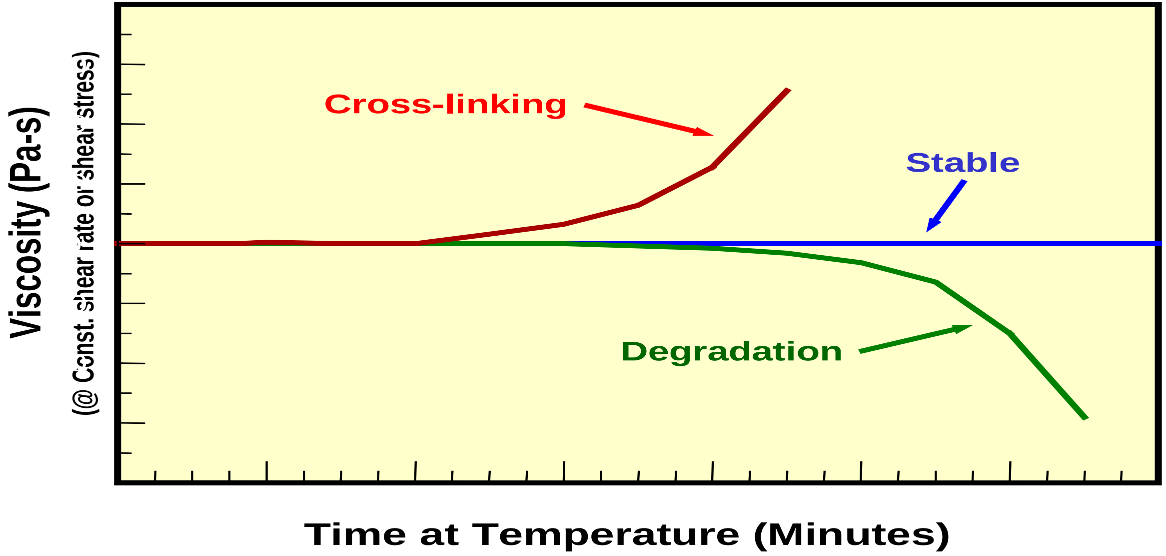
- **Stability Test**
  - **Sample must be stable at temperature in order to get good shear rate sweep data**
  - **Multiple data points at constant temperature and constant shear rate versus time**
- **Shear Rate Sweep**
  - **Four to 15 shear rates**
  - **Most plastic samples are tested from high to low shear rates**
  - **Most elastomeric samples are tested from low to high shear rates**

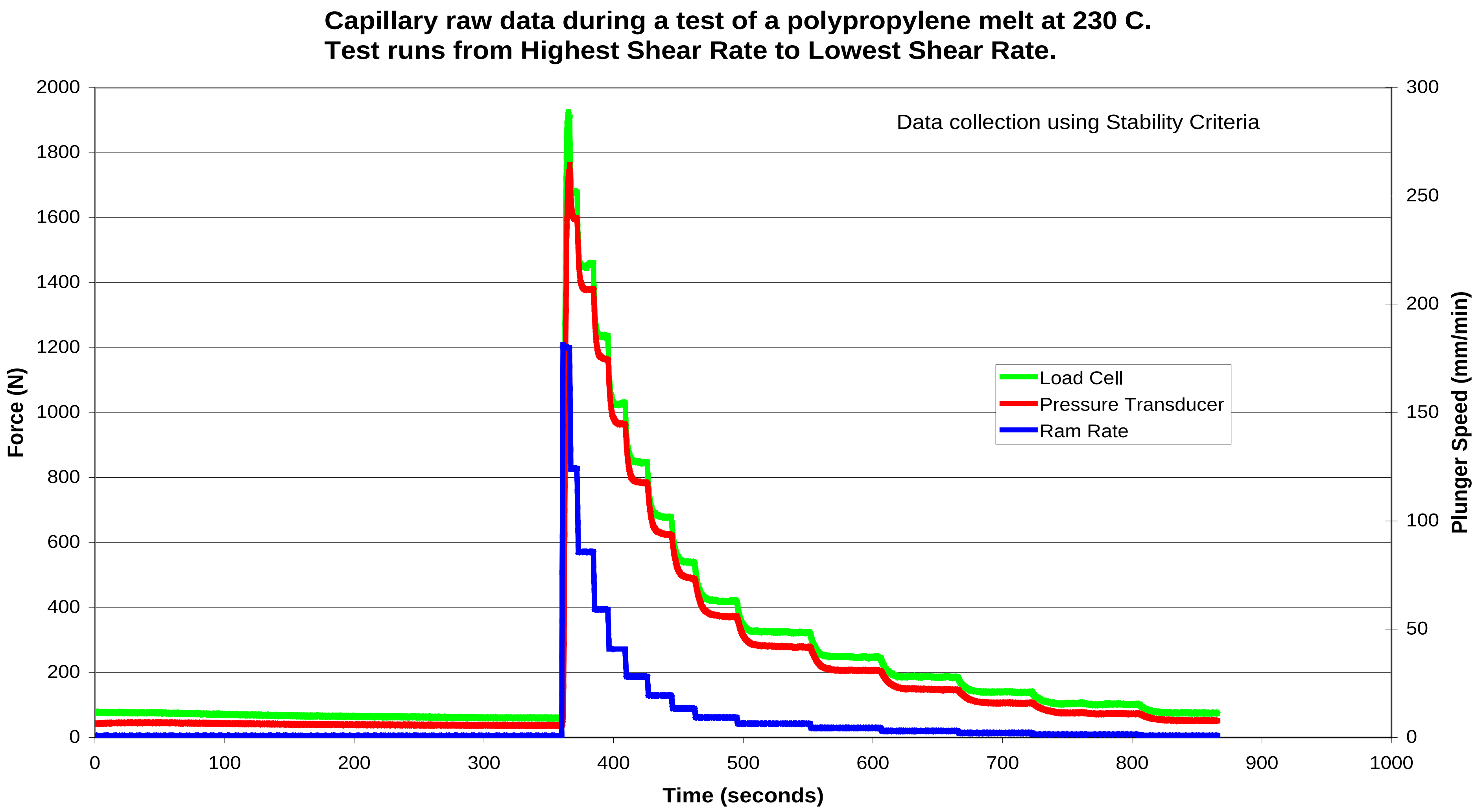
Creating a Test in the LCR 7001

- Select:
  - Preheat time (Typical 5 minutes)
  - Start position (Sample is compacted)
  - Plunger Speed / Shear Rates (enter one, the other is calculated automatically)
  - Capillary Die (diameter, length, entrance angle)
- Data Collection Options:
  - Stability of Pressure / Force readings
  - Position of Plunger (Manual)
  - Time Delay

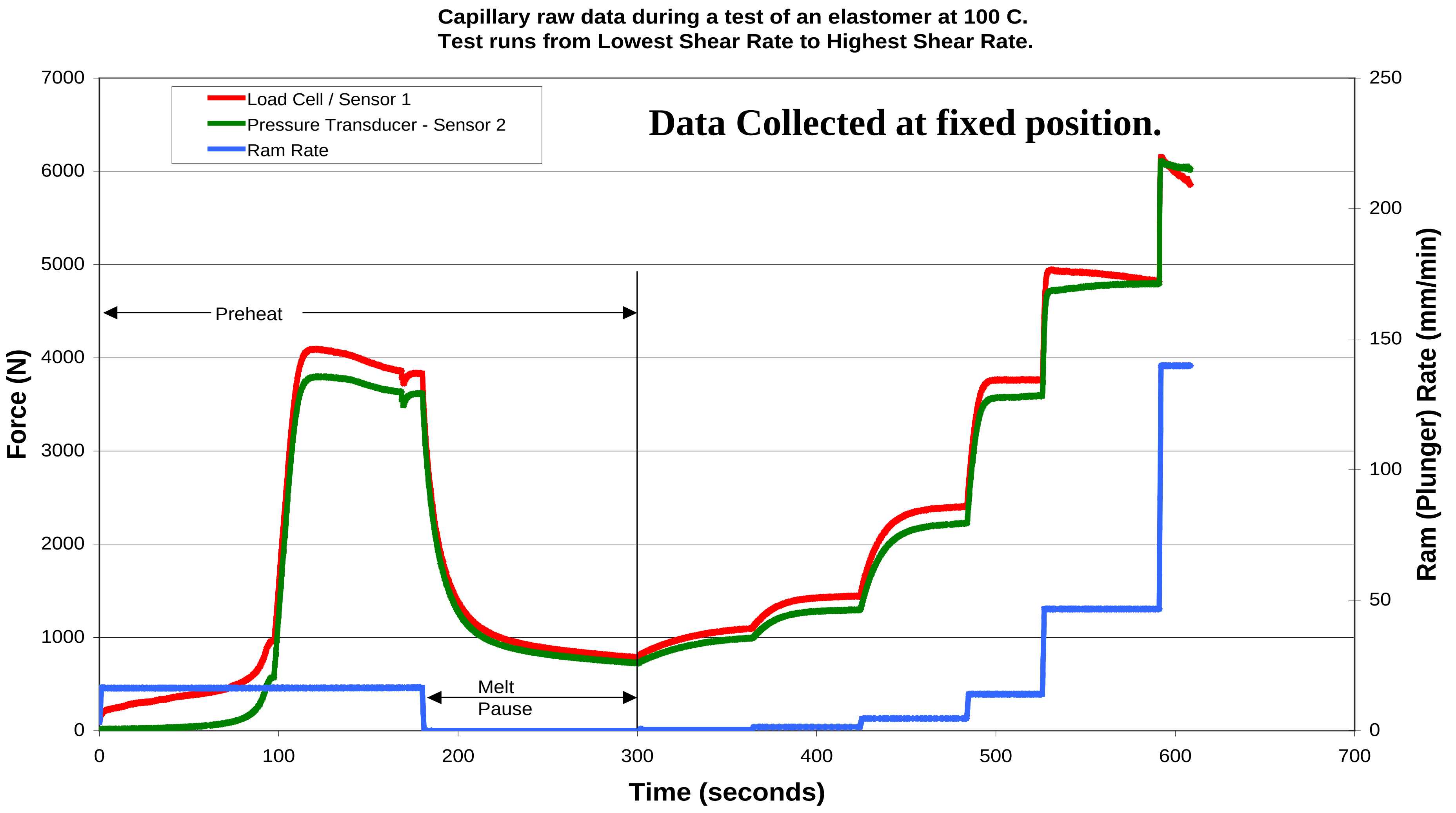
Thermal Stability of Polymer Melt

(Constant temperature and shear rate or stress)

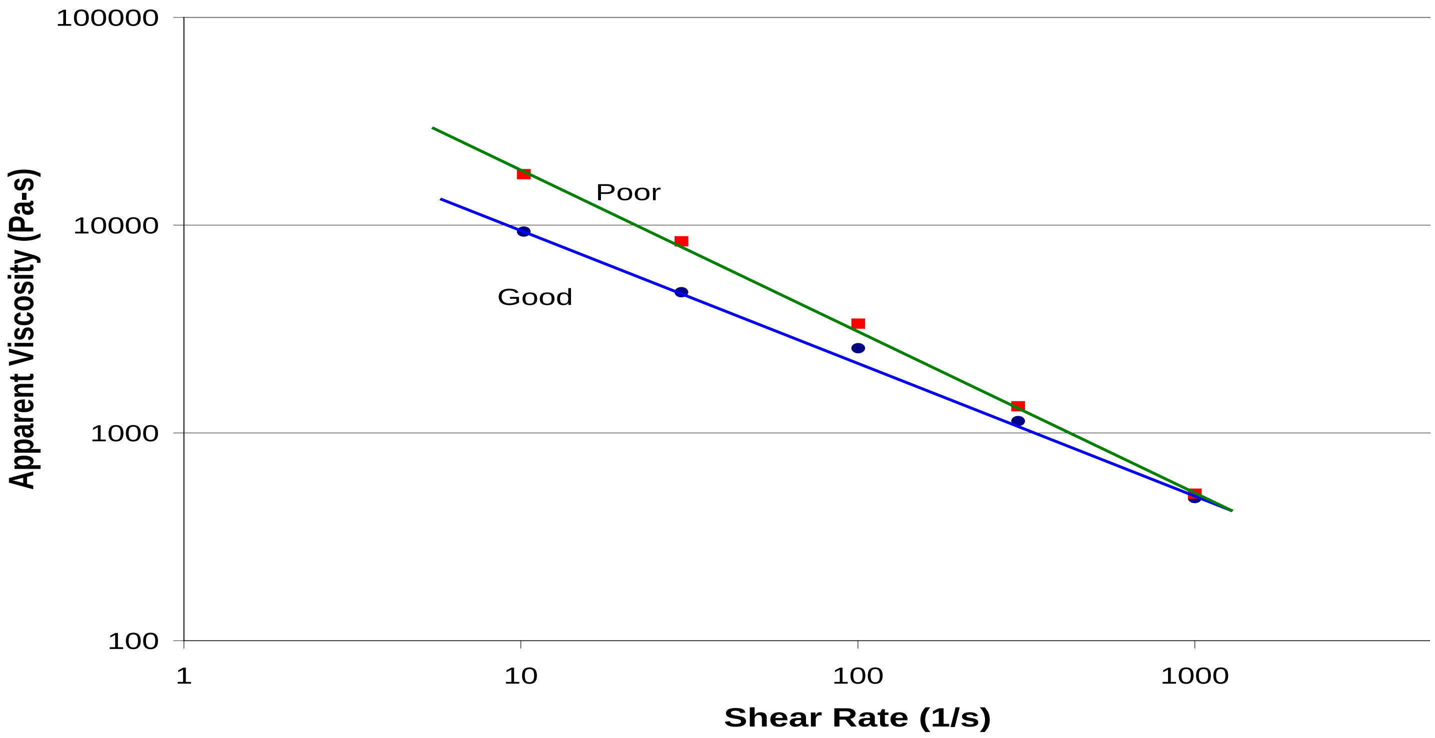




Capillary raw data during a test of a polypropylene melt at 230 C.
Test runs from Highest Shear Rate to Lowest Shear Rate.
Data collection using Stability Criteria
Load Cell
Pressure Transducer
Ram Rate
Force (N)
Plunger Speed (mm/min)
Time (seconds)
0
200
400
600
800
1000
1200
1400
1600
1800
2000
0
50
100
150
200
250
300
0
100
200
300
400
500
600
700
800
900
1000


Capillary raw data during a test of an elastomer at 100 C.
Test runs from Lowest Shear Rate to Highest Shear Rate.
Load Cell / Sensor 1
Pressure Transducer - Sensor 2
Ram Rate
Data Collected at fixed position.
Preheat
Melt
Pause
Force (N)
Ram (Plunger) Rate (mm/min)
Time (seconds)
0
1000
2000
3000
4000
5000
6000
7000
0
50
100
150
200
250
0
100
200
300
400
500
600
700

Apparent Viscosity versus Shear Rate



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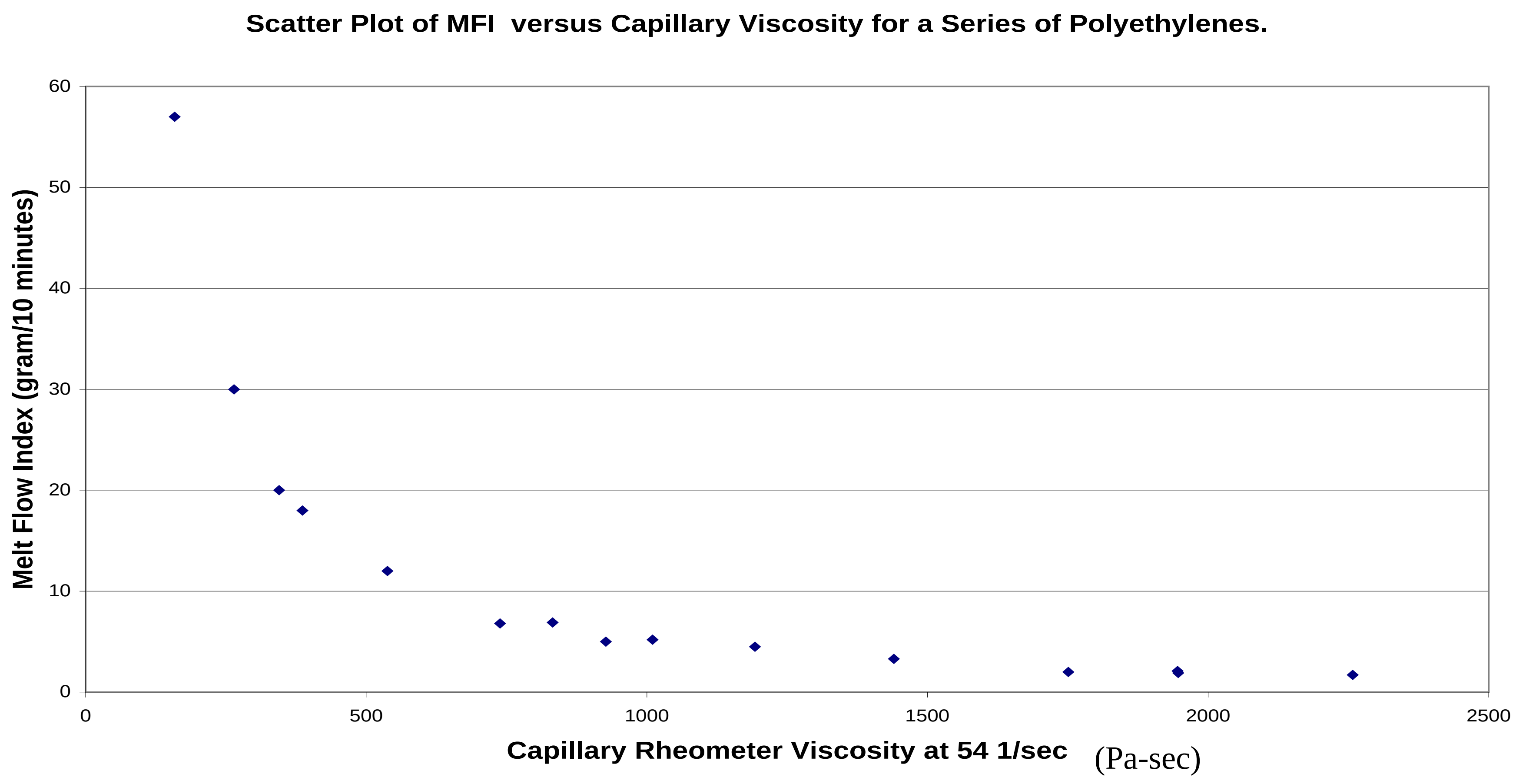
LCR 7001 Summary

- Tool for Plastics Extrusion and Injection Molding Application
  - Rheological Measurements at High Shear Rates
    - 0.1 to 30,000 s^{-1}
  - Viscosity
  - Die Swell
  - Extensional Viscosity
  - Room Temperature to 425 C

Comparison of Controlled Stress and Controlled Rate Capillary Rheometers

Item	LMI (Stress)	LCR (Rate)
Barrel Dia.	9.55 mm	9.55 mm
Orifice Dia.	2.095 mm	0.5 to 3 mm
Orifice L/D	3.82	0 to ~60
Weights	0.1 to 20 kg	N/A
Rate	N/A	0.03 to 650 mm/min
Shear Rate Range	1 to 500 sec^{-1}	1 to 30,000 sec^{-1}
Main Data	Grams/10 minutes	Apparent Viscosity vs. Shear Rate
Other Data	Cm^3 /10 min	True Viscosity Die Swell Extensional Viscosity
Other Tests	Ratio Test (2 wts.)	Stability Test


Scatter Plot of MFI versus Capillary Viscosity for a Series of Polyethylenes.
Melt Flow Index (gram/10 minutes)
60
50
40
30
20
10
0
0
500
1000
1500
2000
2500
Capillary Rheometer Viscosity at 54 1/sec (Pa-sec)

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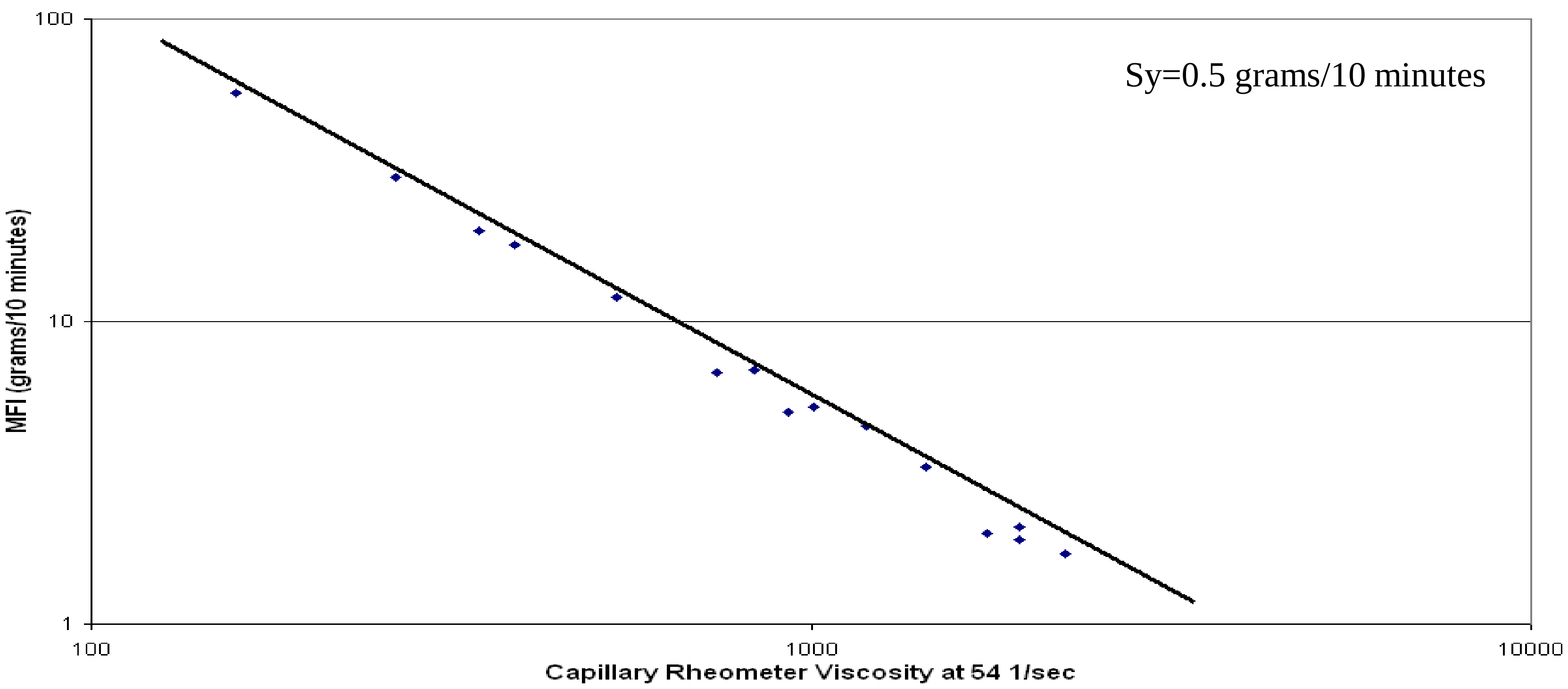
Observations about the Scatter Plot of MFI versus Capillary Rheometer

- Non-Linear Relationship
- Represents difference in Controlled Stress versus Controlled Rate

Figure 3. Correlation of MFI to Capillary Viscosity at 190 C for a Series of Polyethylenes using a Log-Log plot.



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Correlation Equations Based on the Plot of Melt Index and Capillary Viscosity.

Log-Log Plot

$$Log(Melt\_Index) = (A)Log(\eta_{\dot{\gamma}}) + B$$

Or

Scatter Plot

$$Melt\_Index = 10^{B}\eta^{A}$$

Equations are identical (A=-1.3403, B=4.7173)

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Figure 2. Melt Flow Index versus Capillary Viscosity at 190 C for a Series of Polyethylenes.

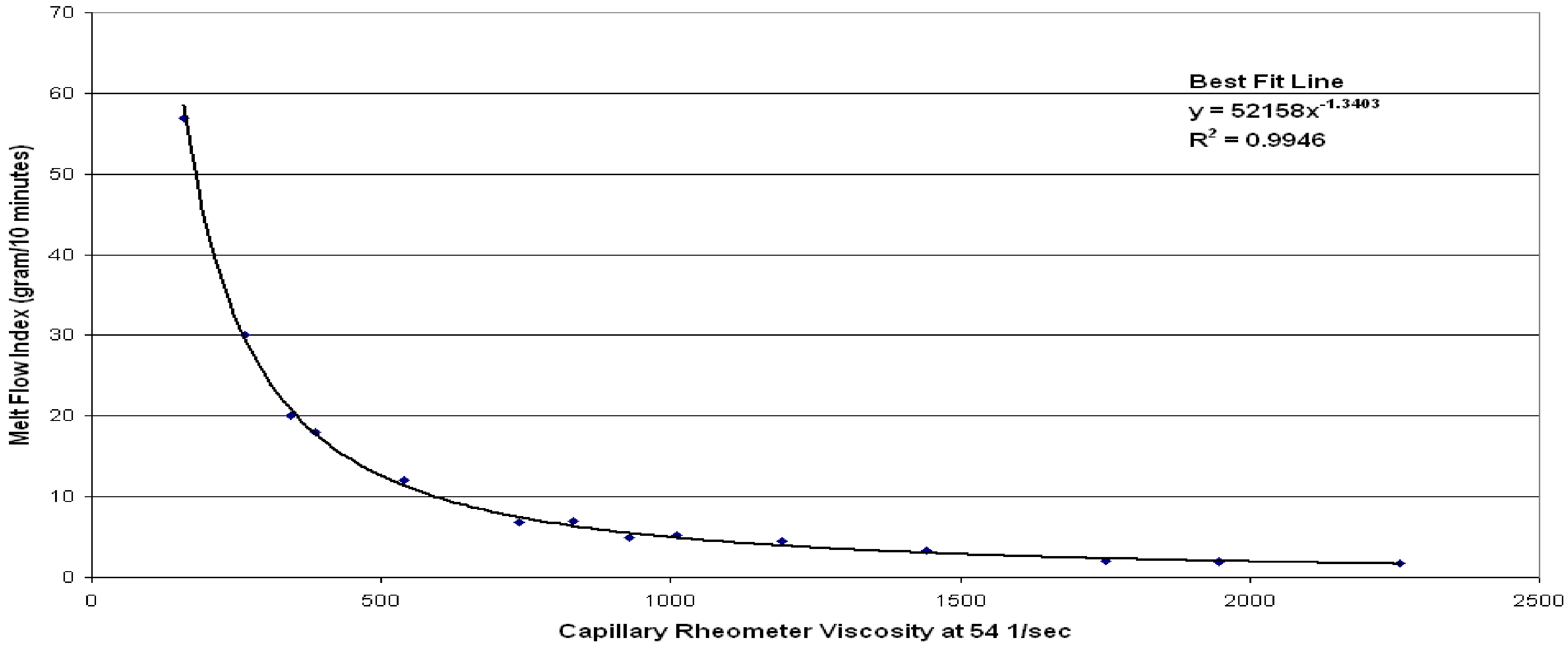

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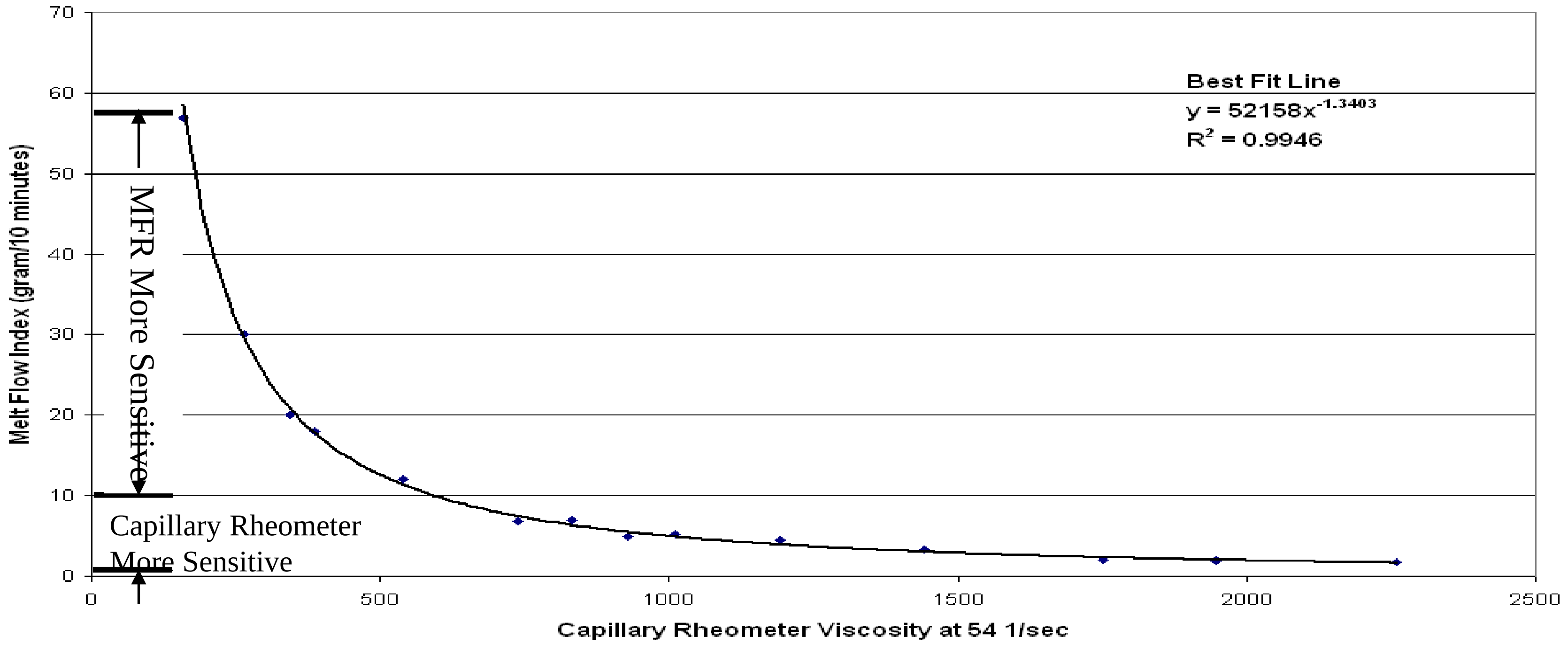
Figure 2. Melt Flow Index versus Capillary Viscosity at 190 C for a Series of Polyethylenes.



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Correlation of MFR and Capillary Rheometer Viscosity

- Follows non-linear equation
- Capillary rheometer can estimate MFR
- Capillary rheometer requires less time from operator (load and go)
- Different Correlation Required for Different Dies and LMI Weights