The Investigation of Extensional Rheology using the Sentmanat Extensional Rheometer(SER)

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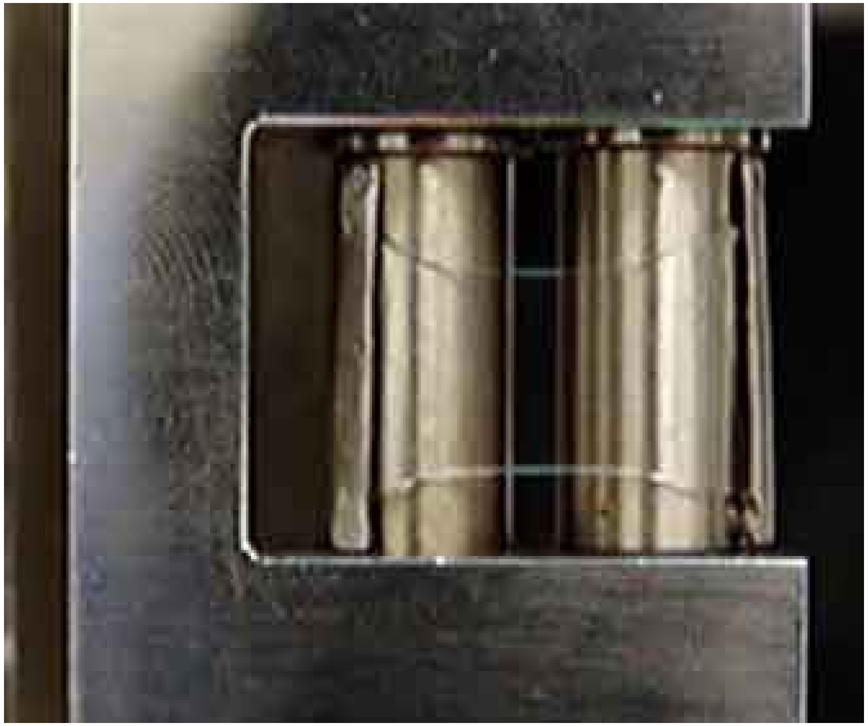
Goals

- Calculate extensional rheology properties of block copolymers, branched polymers, and ionomers
- Determine the relationship between the extensional properties and shear properties of the polymers.



Xpansion Instruments. (2007). http://www.xpansioninstruments.com/videos.htm#melt

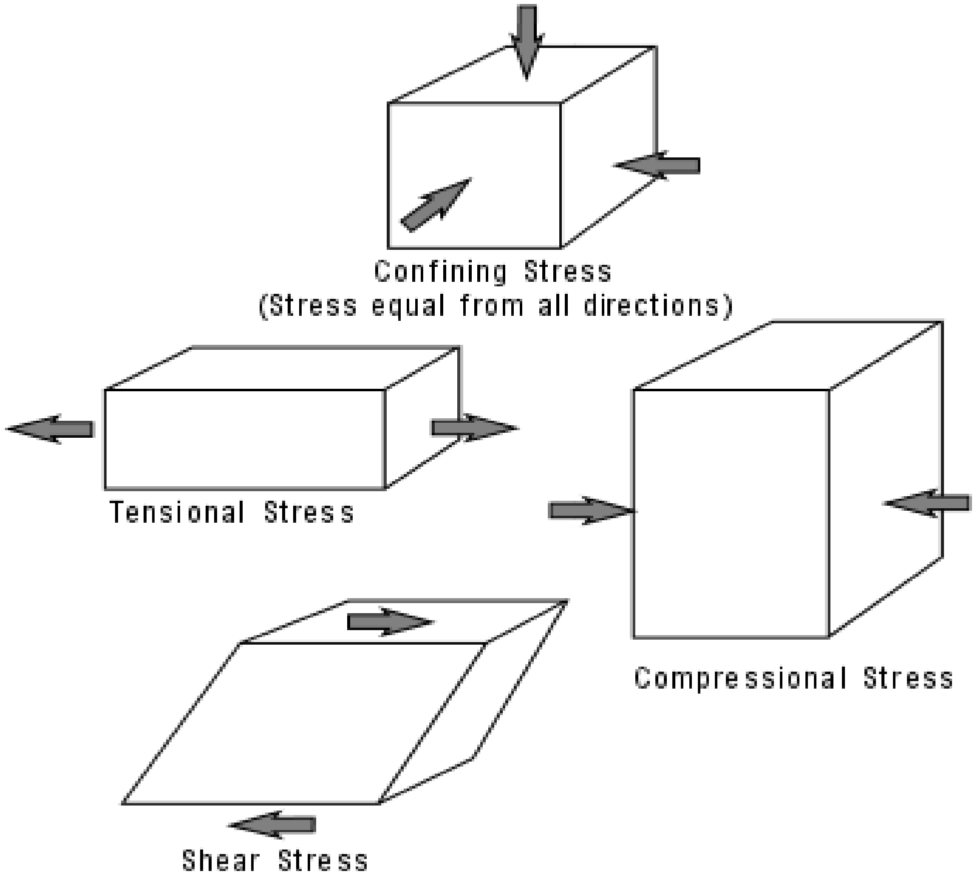
Background

Earth Science Australia. (2008). http://earthsci.org/education/teacher/basicgeol/deform/deform.html

- Viscosity is a property of a material that involves resistance to extensional or shear stress.

$$\sigma = \frac{F}{A}$$

$$\eta = \frac{\sigma}{\dot{\varepsilon}}$$



- Shear stresses
- Extensional stresses
- Strain rate

$$\dot{\varepsilon} = \frac{d(\ln L)}{dt}$$

- Shear Thinning - viscosity decreases as strain increases
- Strain Hardening - viscosity increases as strain increases

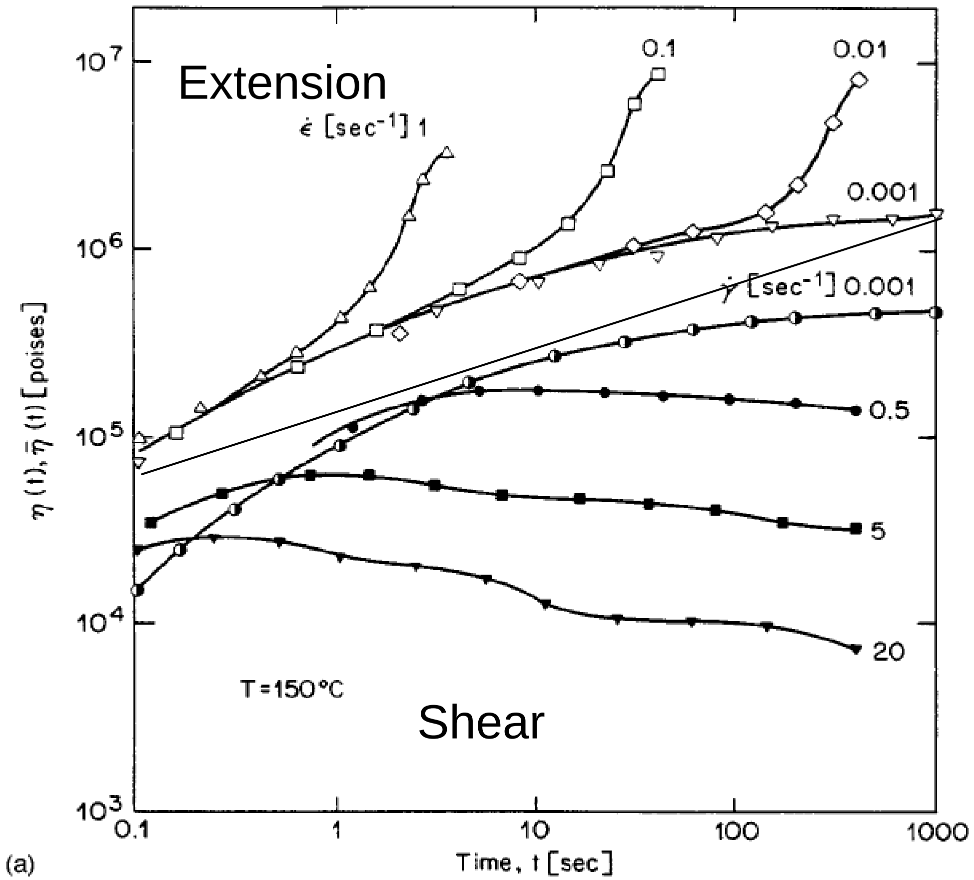
$$\eta = \frac{\sigma}{\dot{\gamma}} \qquad \eta_E(t) = \frac{\sigma_E(t)}{\dot{\varepsilon}}$$

- Zero-shear viscosity

$$\lim_{\dot{\gamma} \to 0} \eta = \eta_0$$

- Trouton’s Rule

$$\lim_{\dot{\varepsilon} \to 0} \eta_E = 3\eta_0$$

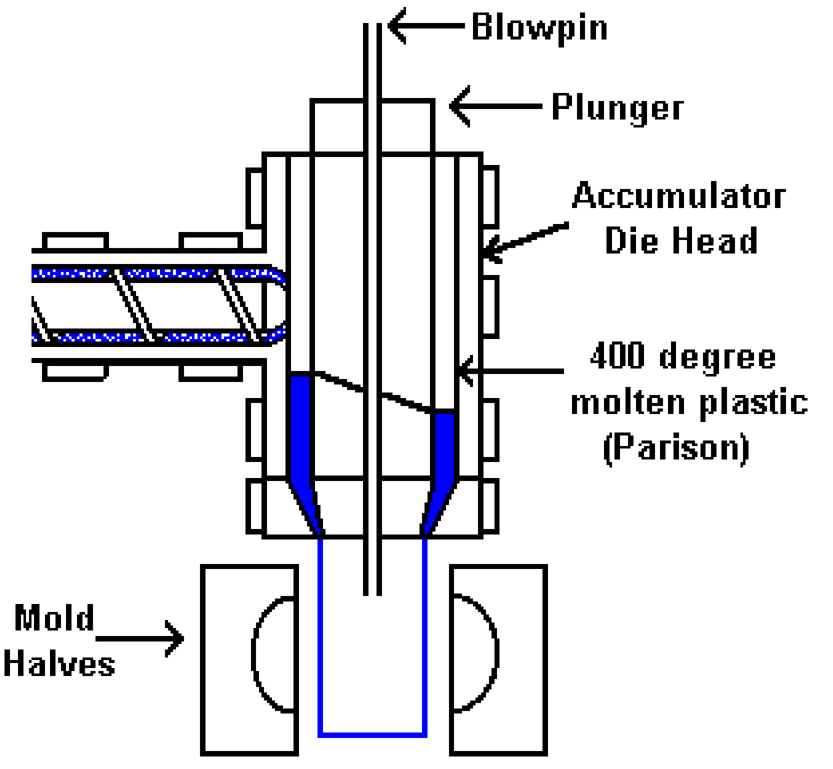


T.C.B. McLeish and R.G. Larson. *1997.*

J.M. Dealy and K.F. Wissbrum. *1999*

Industrial Applications

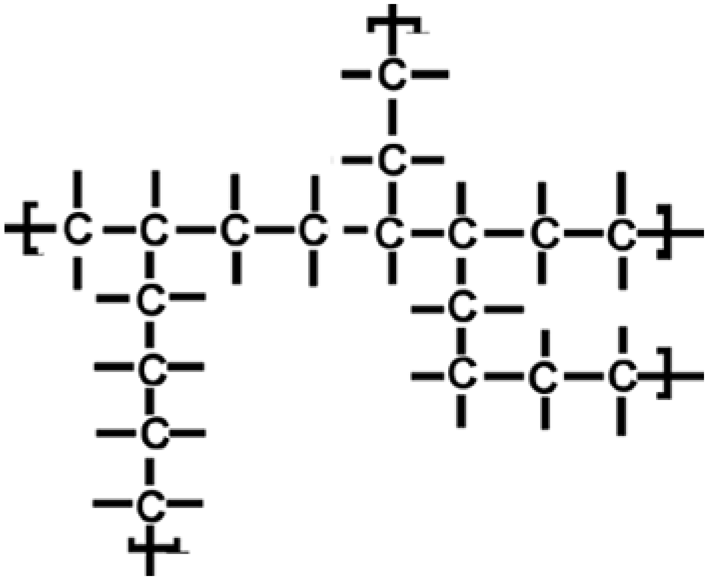
- In industrial applications, both shear and extensional stresses are applied. However, more commonly known are the shear properties of polymer materials.
- Fiber spinning
- Paint rolling
- Flow through porous media
- Blow molding
- Film blowing



http://www.engineershandbook.com/MfgMethods/blowmol.gif

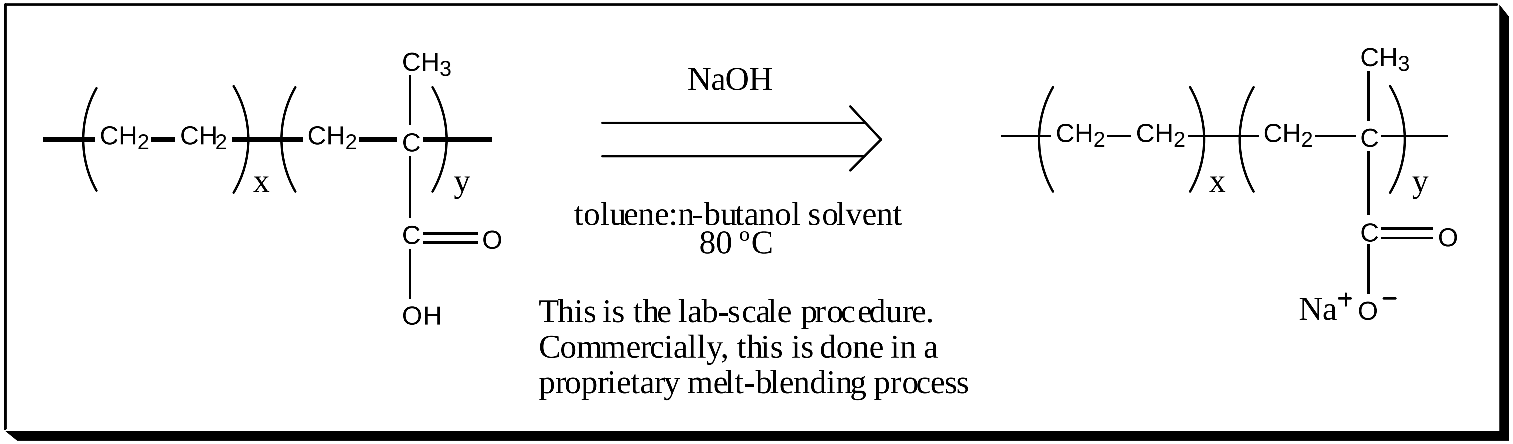
LDPE and Ionomers

- LDPE (Low Density Polyethylene)



http://www.chemistry.wustl.edu/~edudev/Designer/session2.html

- Ethylene-Methacrylic Acid Ionomers



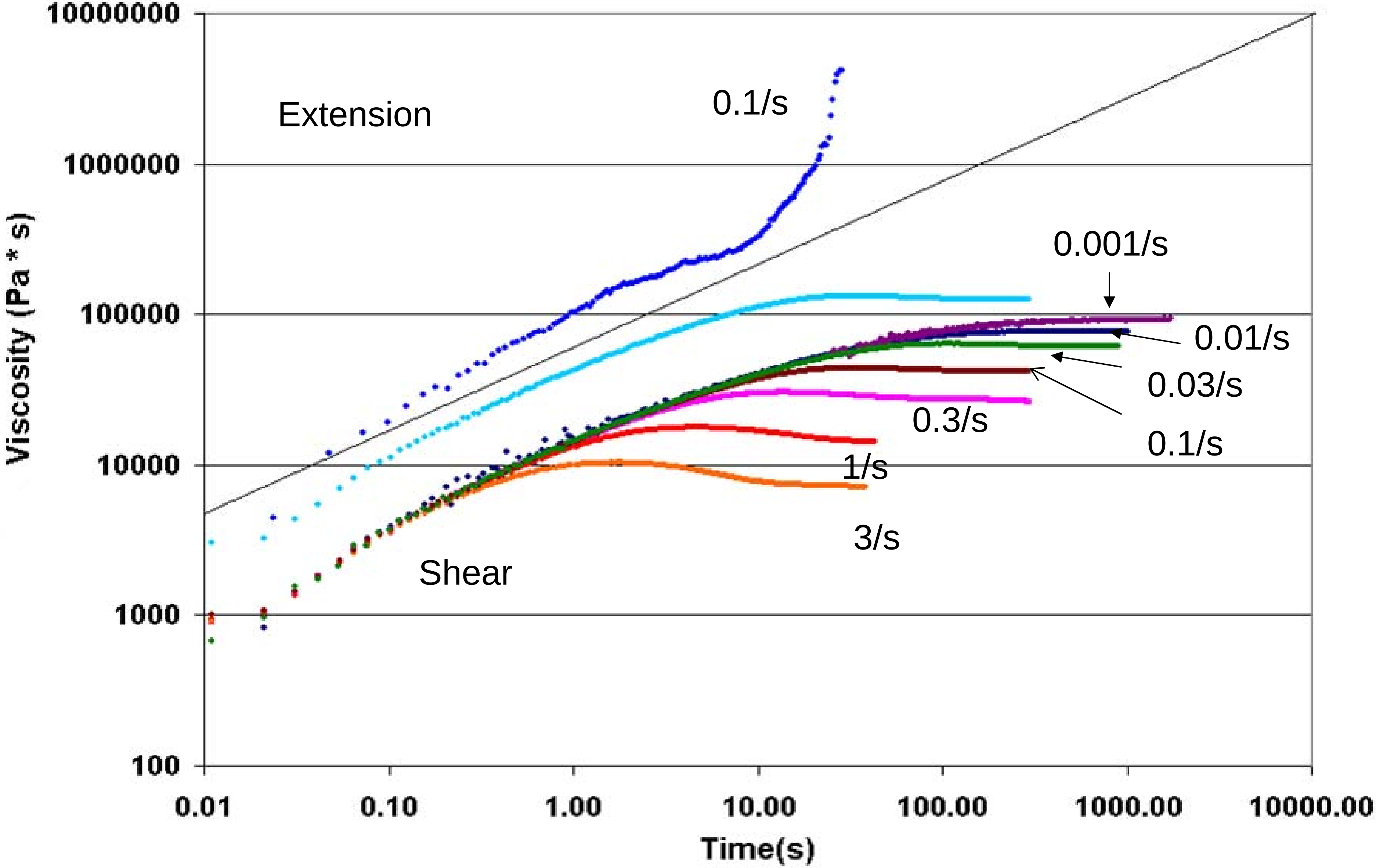
R.C. Scogna. 2006

Shear Experiments

- Shear and extensional experiments were done on the same polymer to verify the accuracy of the SER, using the well-known Trouton’s rule.
- A cone-and-plate fixture was used to perform a shear experiment on the polymer LDPE.
- LDPE was chosen because it’s properties are well-documented and would provide no expected difficulties.

Xpansion Instruments. (2007). http://www.xpansioninstruments.com/videos.htm#melt

LDPE (Shear and Extension)

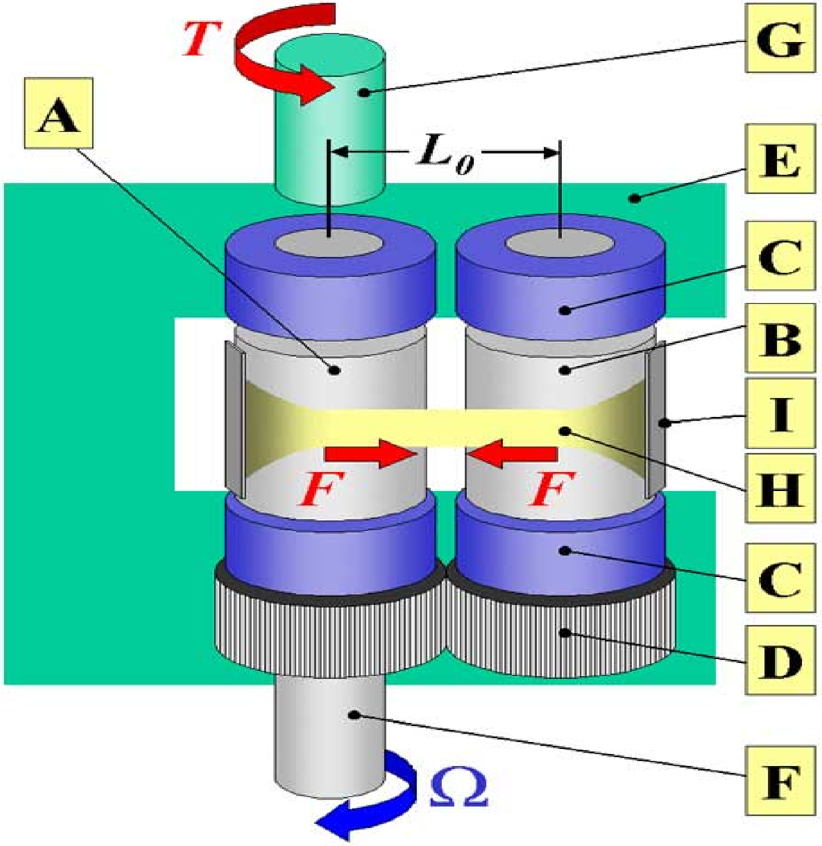


Usefulness of the SER

- Strain rates are supported up to 30/s and as low as 0.005/s.
- Small size makes for easy cleanup.
- Temperature Control
- Cylindrical scheme allows for polymers to be stretched very long.

Methods and Techniques

- T = Torque applied in order to rotate the cylinders.
- B = Wind-up drums onto which the sample is placed
- F = Drive Shaft
- H = Sample (a polymer material in this case)
- I = Clamps to secure the sample



http://www.xpansioninstruments.com/products_principle.htm

Mechanics of the SER

M.L. Sentmanat. *SER Universal Testing Platform.* 2004

- The ends of the sample are fixed to the wind-up drums, behind the clamps. The strain rate at which the sample is being stretched is:

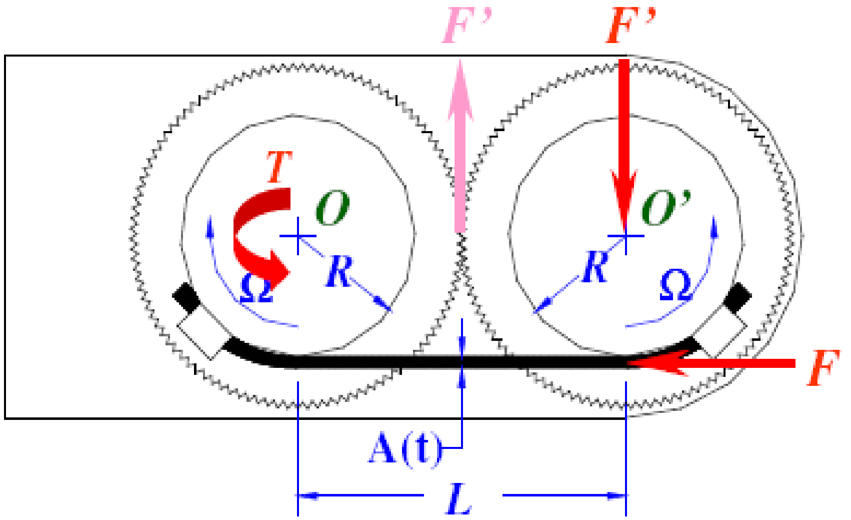
$$\dot{\varepsilon}_H = \frac{2\Omega R}{L_0}$$

- As the sample is stretched by the rotating cylinders, it begins to exert a force F in the opposite direction on the fixture. This allows for a torque to be calculated as

$$T = 2FR$$

- As the width begins to decrease with stretching, the cross-sectional area also does.

$$A(t) = A_0 \exp[-\dot{\varepsilon}_H * t]$$



- Using these values the extensional viscosity can be calculated as a function of time in the following manner.

$$\eta_E = \frac{\sigma_E}{\dot{\varepsilon}_H} = \frac{T}{A(t) * 2R * \dot{\varepsilon}_H}$$

Procedure

- Each polymer was pressed at 20° above their melt temperature.
- The polymer sheets were then handcut to be of the following dimensions.

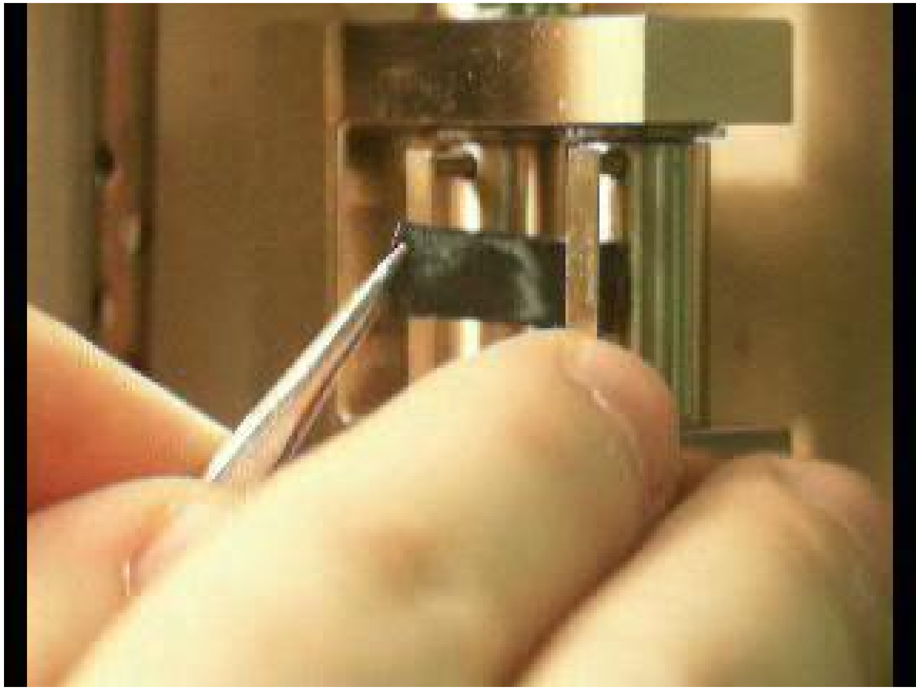
$$L = 19 \ mm$$

$$W = 12 \ mm$$

$$D = 0.4 \ mm$$

- The oven was be pre-heated to the desired temperature and allowed to sit for at least 20 minutes. After this time the sample is placed onto the fixture and allowed to reach the experimental temperature for 2-3 minutes.

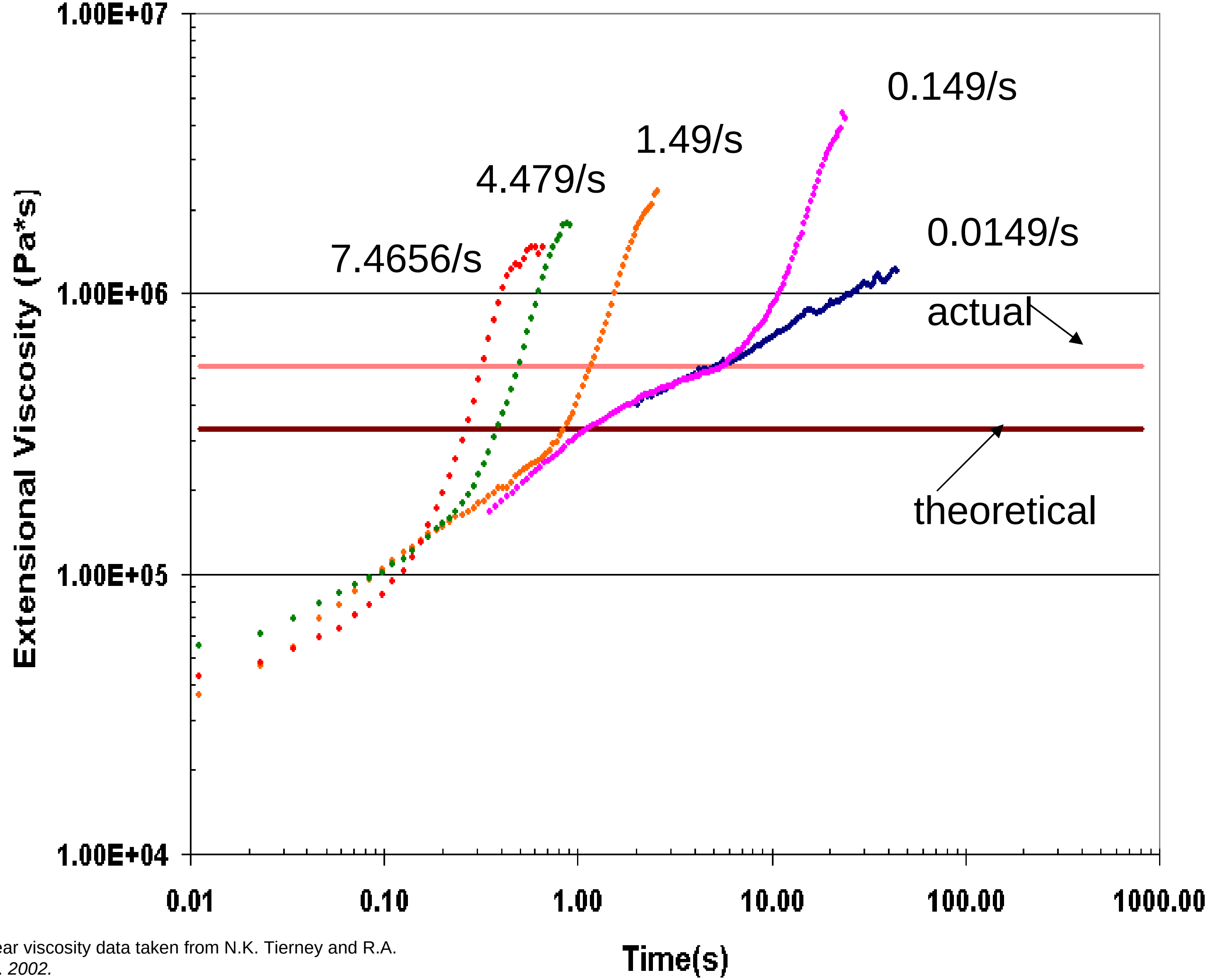
- Samples were run until breakage was confirmed.



Xpansion Instruments. (2007).
http://www.xpansioninstruments.com/videos.htm#melt

SER with Ionomers

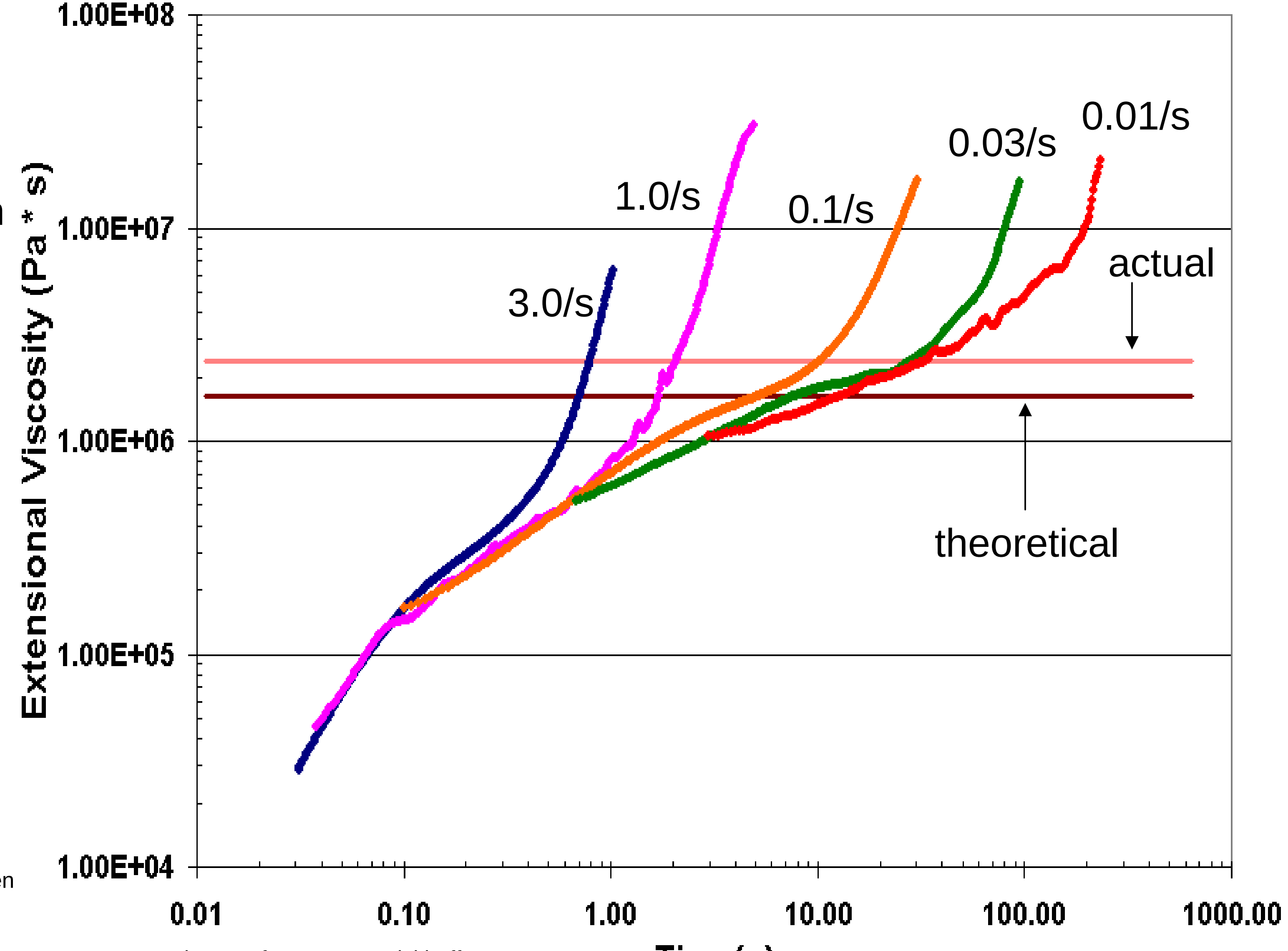
- 15 wt % methacrylic-acid.
- 51% of that acid is neutralized by Na.
- All runs were performed at 135°C
- Five strain rates provide “master curve”
- Trouton’s rule and factor of 1.6



Zero-shear viscosity data taken from N.K. Tierney and R.A. Register. *2002.*

SER with Ionomers

- 15 wt% methacrylic-acid
- 67% neutralization by Na
- 5 different strain rates were used to construct the following “master curve”.
- Trouton’s rule and factor of 1.5



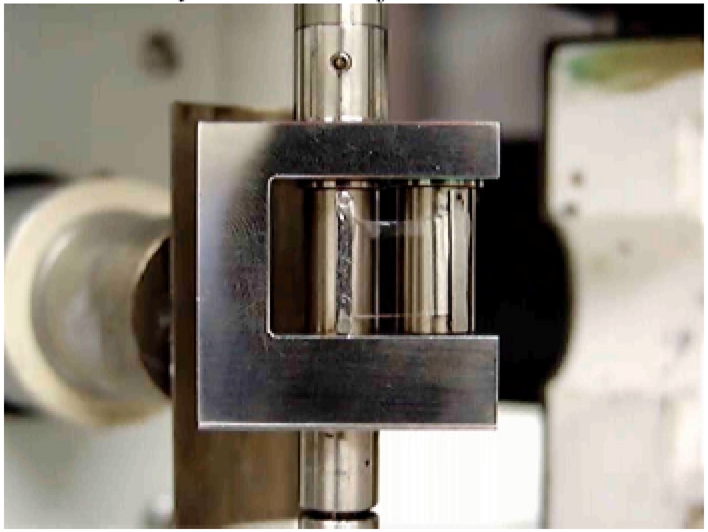
Zero-shear viscosity data taken from N.K. Tierney and R.A. Register. *2002.*

Assistance from James Eickhoff

Conclusion

- Greater deviation from the expected viscosity was found when using LDPE than the ionomers.
- Measuring the extensional viscosity at very low rates is difficult, due to the imprecision in torque measurements.
- Thus making it very difficult to verify the relationship between shear and extensional data at such low rates, where Trouton's rule holds true.

Polyethylene melt at 150°C and a Hencky Strain Rate of 1.0 s^{-1}



M.L. Sentmanat. *Melt Fracture of Polyethylene and the Role of Extensional Flow Behavior*. 2005

Acknowledgements

- Professor Rick Register
- Register Group
- Robert C. Scogna
- Andy Marencic
- DuPont Packaging and Industrial Polymers

Full Equation for Extension

- Torque Offset and “pre-load” conditions
- Time Offset and “slack”

$$\eta_E = \frac{\sigma_E}{\dot{\varepsilon}_H} = \frac{T - T_{offset}}{A_0 * \left(\frac{\rho_S}{\rho_M}\right)^{2/3} * \exp[-\dot{\varepsilon} * (t - t_{offset})] 2R * \dot{\varepsilon}_H}$$