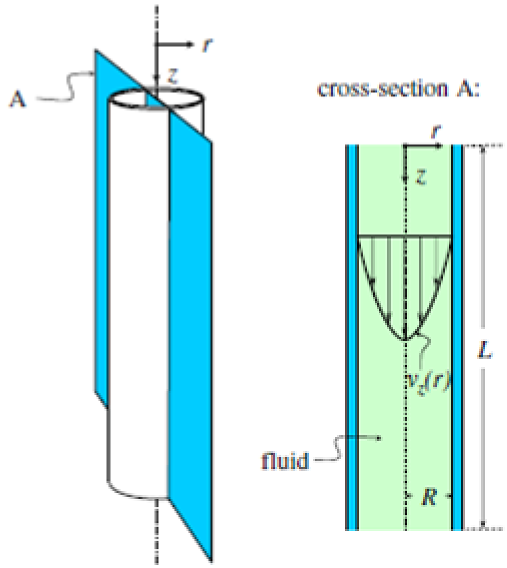
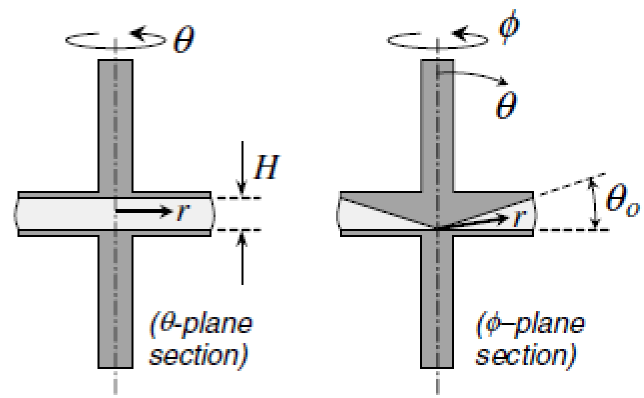
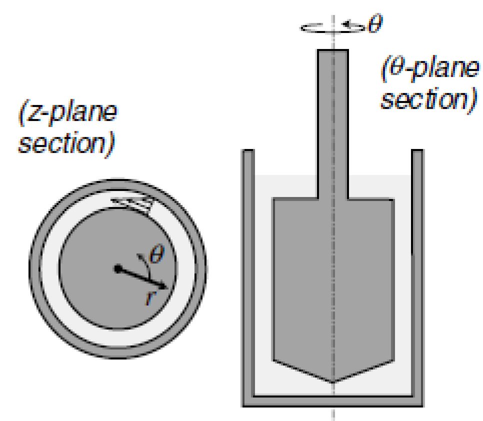
Rheometry

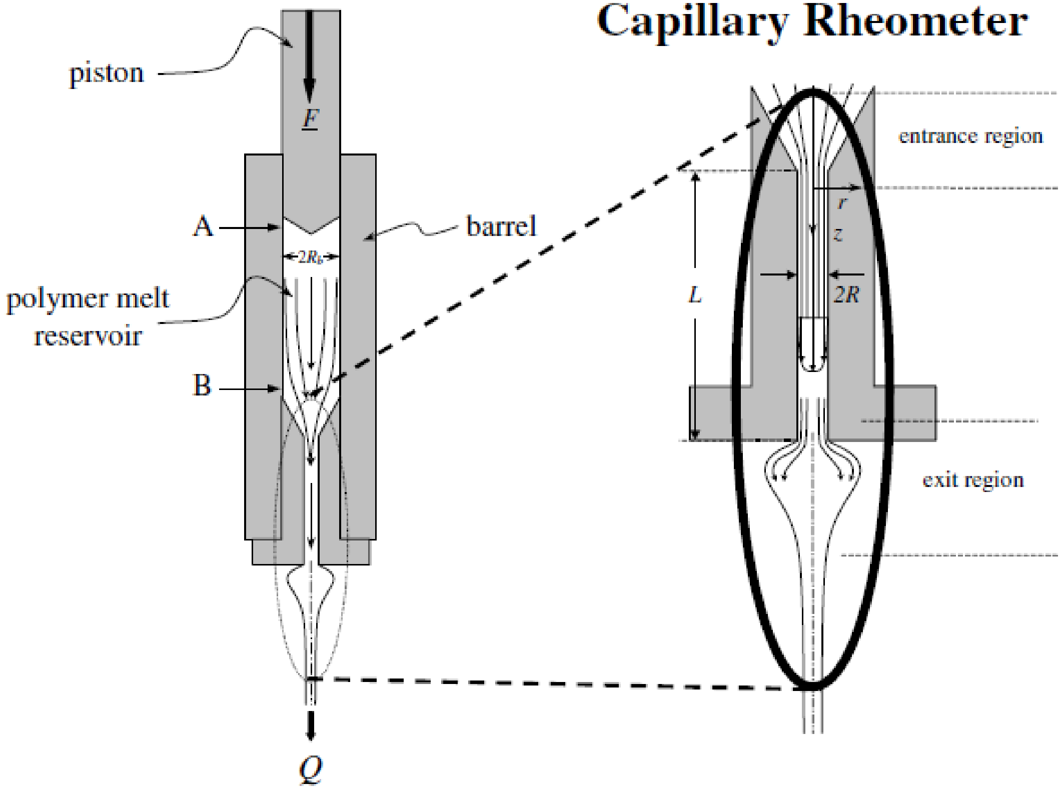
shear flow


A
z
r
cross-section A:
r
z
$v_z(r)$
L
fluid
R


θ
H
r
(θ-plane section)
ϕ
θ
θ_o
r
(ϕ–plane section)

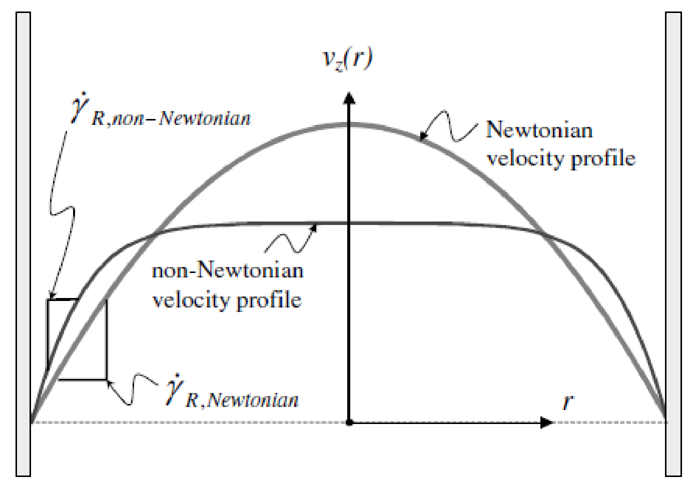

θ
(z-plane section)
θ
r
(θ-plane section)

Capillary Rheometer



Assumptions:

1. unidirectional flow
2. incompressible fluid
3. azimuthal symmetry
4. long capillary; z-variation is negligible
5. symmetric stress tensor
6. constant pressure gradient
7. finite stress at r=0



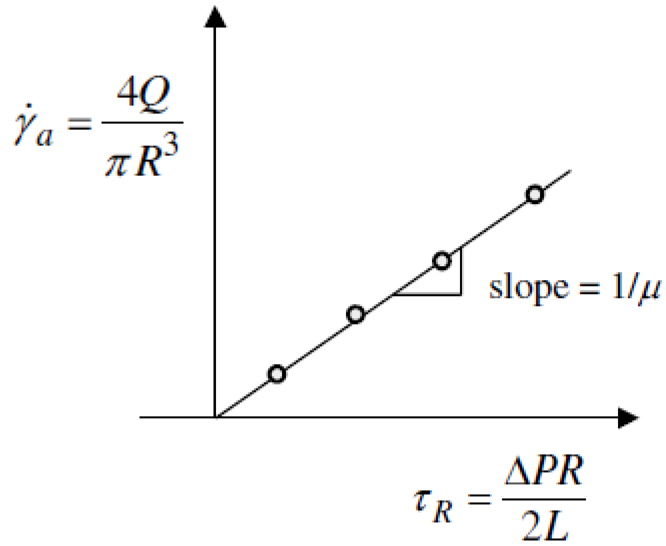
$$\eta = \frac{-\tau_{21}}{\dot{\gamma}_0} = \frac{\tau_R}{\dot{\gamma}_R}$$

τ_R: wall shear stress

$\dot{\gamma}_R$: wall shear rate

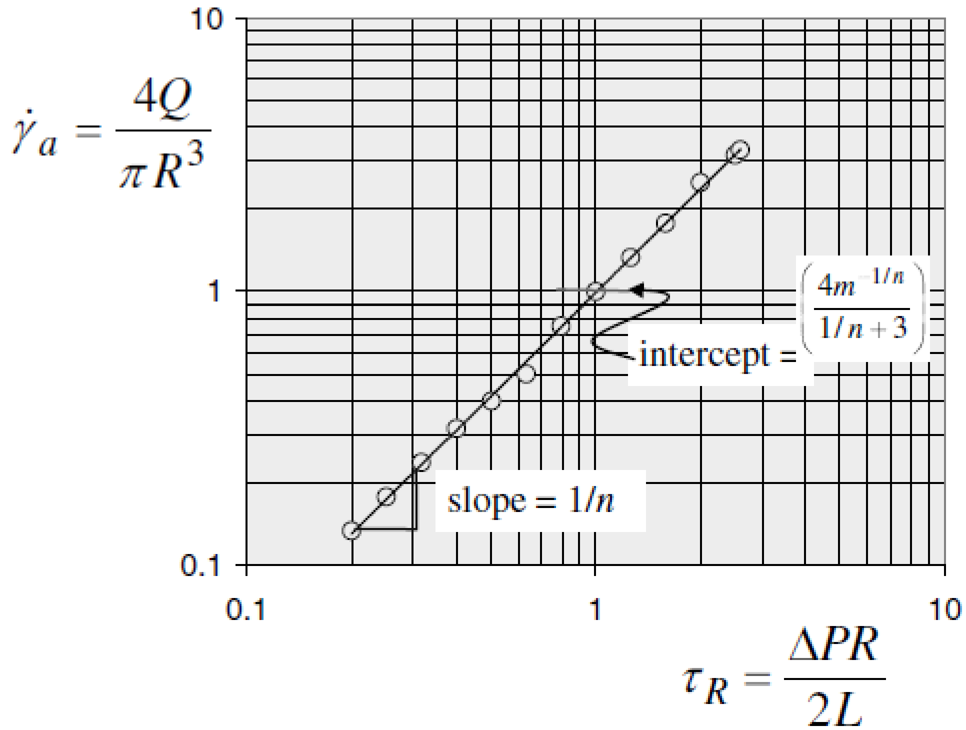
$$\tau_{rz} = \frac{(P_0 - P_L)r}{2L} = \tau_R \frac{r}{R}$$

Wall shear-rate for a Newtonian fluid



$$\dot{\gamma}_a = \frac{1}{\mu} \tau_R$$

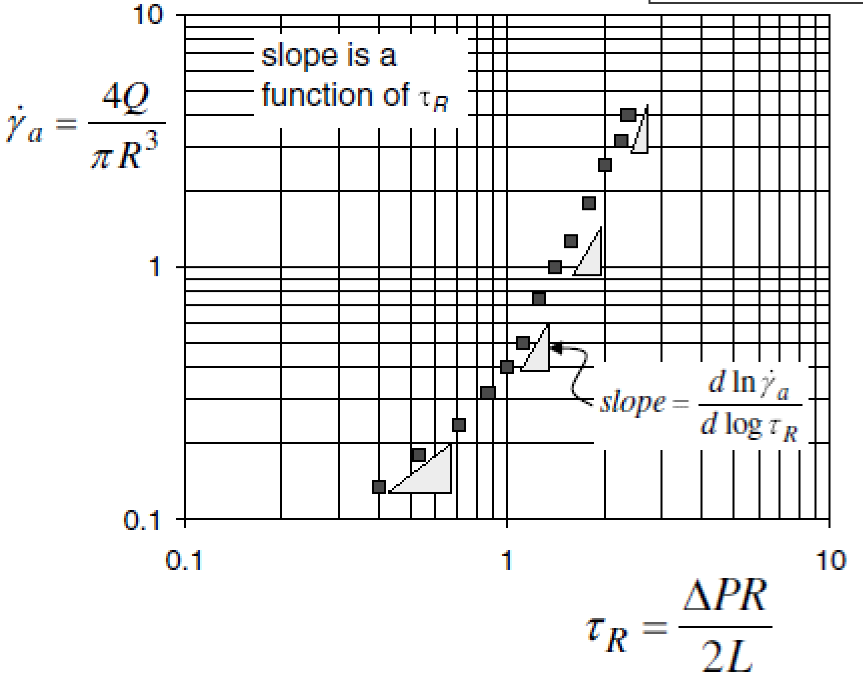
Wall shear-rate for a Power-law GNF



$$\log \dot{\gamma}_a = \frac{1}{n} \log \tau_R + \log\left(\frac{4m^{-1/n}}{1/n+3}\right)$$

Weissenberg-Rabinowitsch correction

$$\dot{\gamma}_R(\tau_R) = \frac{4Q}{\pi R^3}\left[\frac{1}{4}\left(3 + \frac{d\ln\dot{\gamma}_a}{d\ln\tau_R}\right)\right]$$



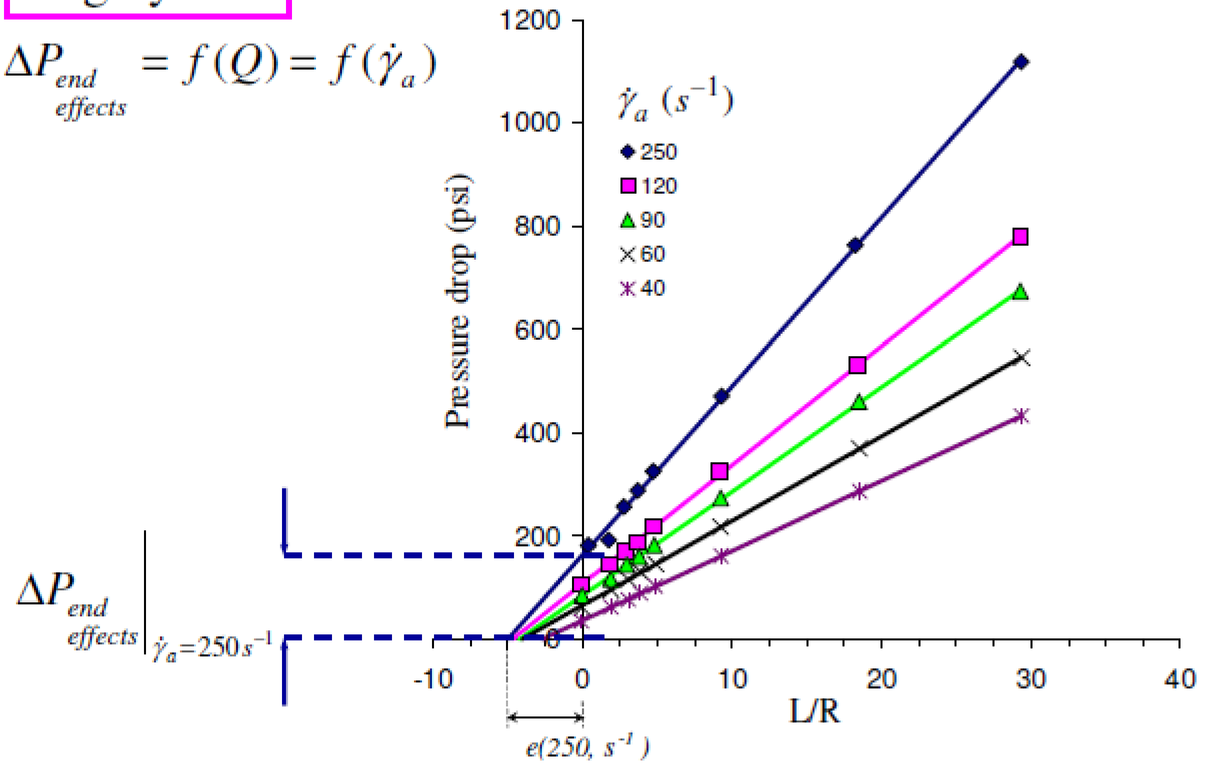
$$\eta(\dot{\gamma}_R) = \frac{4\tau_R}{\dot{\gamma}_a}\left(3 + \frac{d\ln\dot{\gamma}_a}{d\ln\tau_R}\right)^{-1}$$

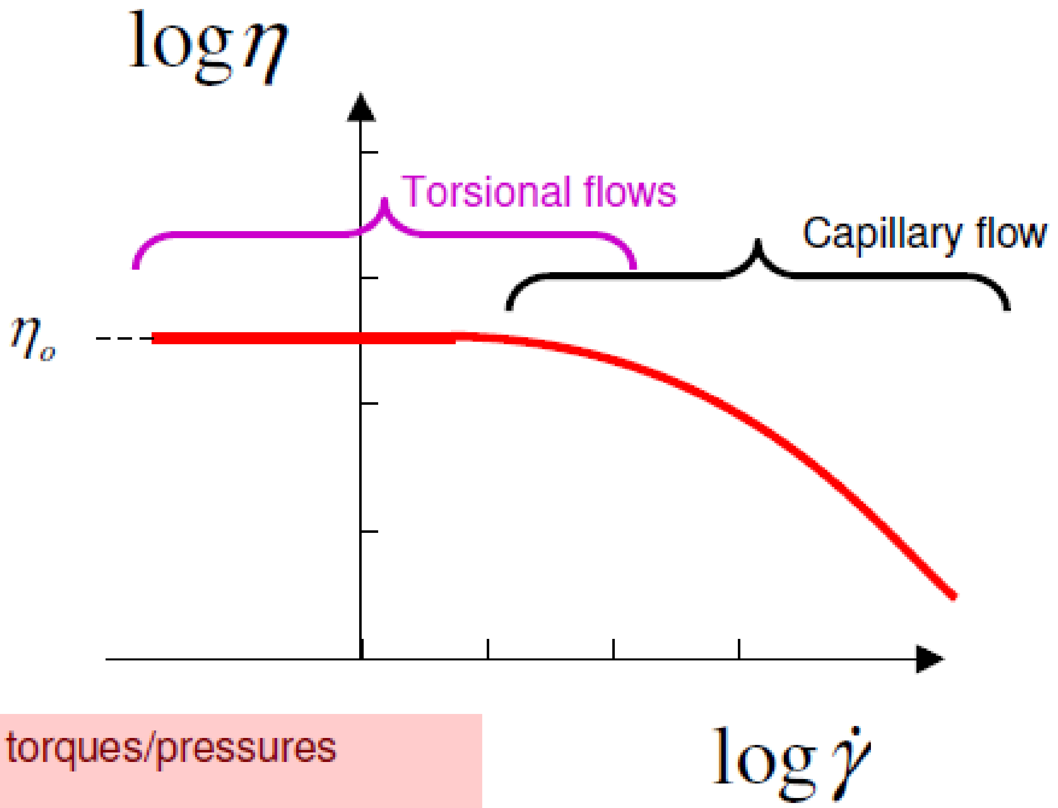
entrance effect
(Bagley correction)

Bagley Plot

$$\Delta P_{\substack{end\\effects}} = f(Q) = f(\dot{\gamma}_a)$$

$$\Delta p = 2\tau_R \frac{L}{R}$$

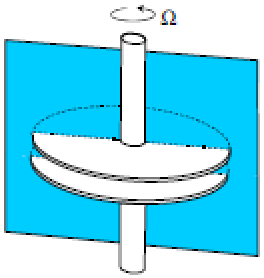


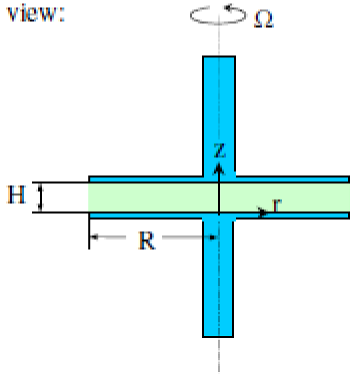


- At low rates, torques/pressures become low
- At high rates, torques/pressures become high; flow instabilities set in

Torsional Parallel-Plate Flow - Viscosity

Measureables:
Torque **T** to turn plate
Rate of angular rotation **Ω**





Note: shear rate experienced by fluid elements depends on their *r* position.

$$\dot{\gamma} = \frac{r\Omega}{H} = \dot{\gamma}_R \frac{r}{R}$$

By carrying out a Rabinowitsch-like calculation, we can obtain the stress at the rim (*r*=*R*).

$$\tau_{z\theta}\big|_{r=R} = -T/2\pi R^3 \left[3 + \frac{d\ln(T/2\pi R^3)}{d\ln\dot{\gamma}_R} \right]$$

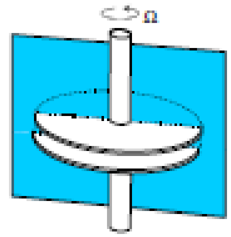
$$\eta(\dot{\gamma}_R) = \frac{-\tau_{z\theta}\big|_{r=R}}{\dot{\gamma}_R}$$

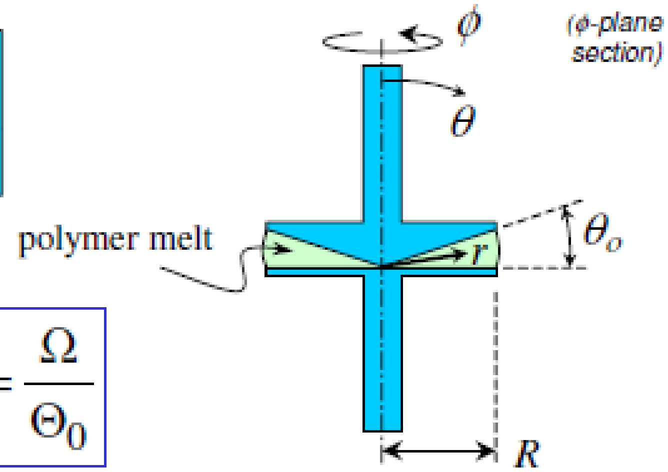
Correction required

$$\eta(\dot{\gamma}_R) = \frac{T/2\pi R^3}{\dot{\gamma}_R}\left[3 + \frac{d\ln(T/2\pi R^3)}{d\ln\dot{\gamma}_R} \right]$$

Torsional Cone-and-Plate Flow - Viscosity

Measureables:
Torque **T** to turn cone
Rate of angular rotation **Ω**





Note: the introduction of the cone means that shear rate is independent of *r*.

$$\dot{\gamma} = \frac{\Omega}{\Theta_0}$$

Since shear rate is constant everywhere, so is stress, and we can calculate stress from torque.

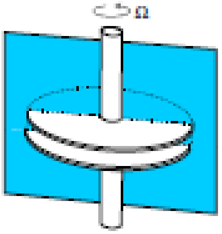
$$\tau_{\theta\phi} = \text{constant} = \frac{3T}{2\pi R^3}$$

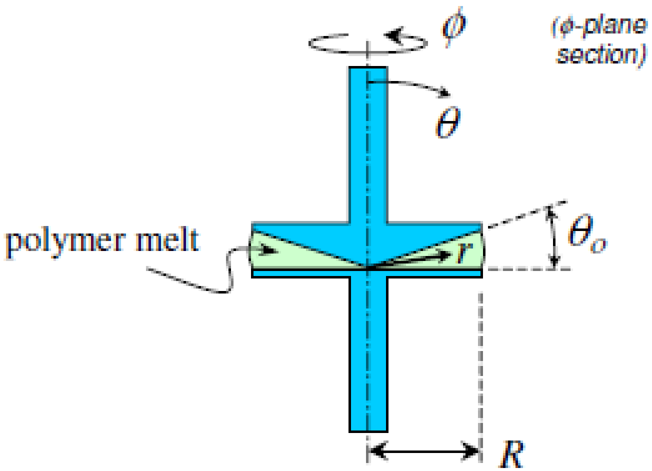
$$\eta(\dot{\gamma}) = \frac{3T\Theta_0}{2\pi R^3 \Omega}$$

No corrections needed in cone-and-plate

Torsional Cone-and-Plate Flow – 1st Normal Stress

Measureables:
Normal thrust **F**





The total upward thrust of the cone can be related directly to the first normal stress coefficient.

$$F = \left[2\pi \int_0^R \Pi_{\theta\theta} \Big|_{\theta=\frac{\pi}{2}} r dr \right] - \pi R^2 p_{atm}$$

$$\Psi_1(\dot{\gamma}) = \frac{2F\Theta_0^2}{\pi R^2 \Omega^2}$$

(see text pp404-5; also DPL pp522-523)

Torsional Cone-and-Plate Flow – 2nd Normal Stress

•Cone and Plate:

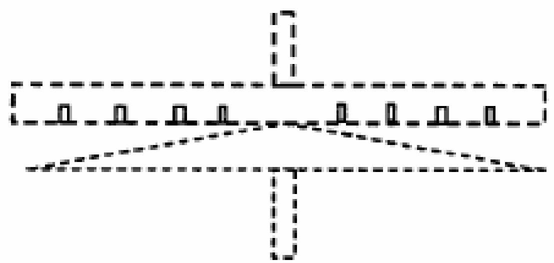
$$\Pi_{22} - p_0 = -(N_1 + 2N_2)\ln\left(\frac{r}{R}\right) - N_2$$

Need normal force as a function of *r* / R

(see Bird et al., DPL)

•MEMS used to manufacture sensors at different radial positions

S. G. Baek and J. J. Magda, J. Rheology, 47(5), 1249-1260 (2003)

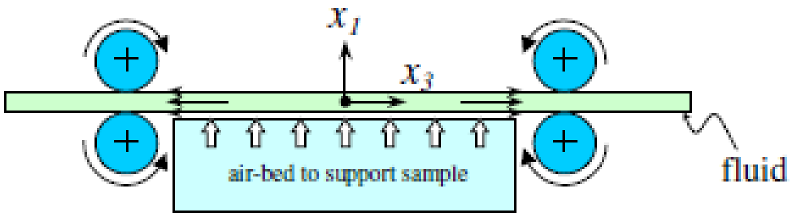


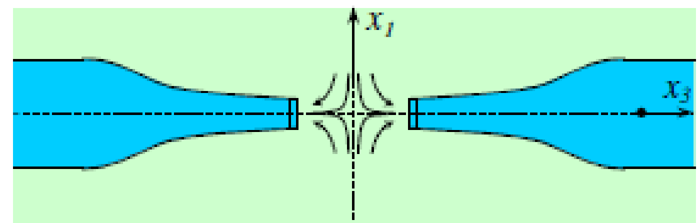
RheoSense Incorporated (www.rheosense.com)

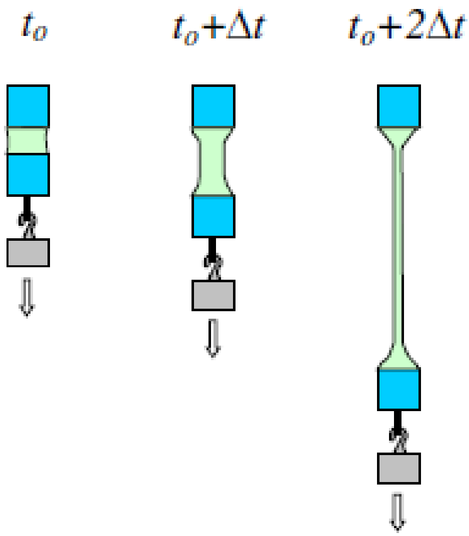
TABLE 10.3
Comparison of Experimental Features of Four Common Shear Geometries

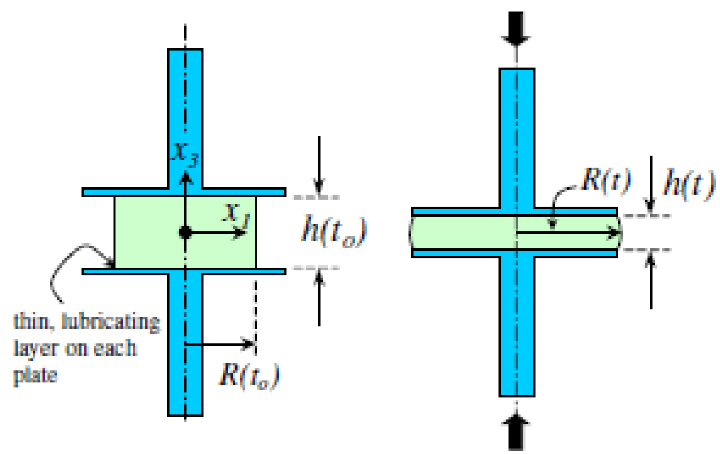
Feature	Parallel Disk	Cone and Plate	Capillary	Couette (Cup and Bob)
Stress range	Good for high viscosity	Good for high viscosity	Good for high viscosities	Good for low viscosities
Flow stability	Edge fracture at modest rates	Edge fracture at modest rates	Melt fracture at very high rates, i.e., distorted extrudates and pressure fluctuations are observed	Taylor cells are observed at high Re due to inertia; elastic cells are observed at high De
Sample size and sample loading	$<$ 1 g; easy to load	$<$ 1 g; highly viscous materials can be difficult to load	40 g minimum; easy to load	10–20 g; highly viscous materials can be difficult to load
Data handling	Correction on shear rate needs to be applied; this correction is ignored in most commercial software packages	Straightforward	Multiple corrections need to be applied	Straightforward
Homogeneous?	No; shear rate and shear stress vary with radius	Yes (small core angles)	No; shear rate and shear stress vary with radius	Yes (narrow gap)
Pressure effects	None	None	High pressures in reservoir cause problems with compressibility of melt	None
Shear rates	Maximum shear rate is limited by edge fracture; usually cannot obtain shear-thinning data	Maximum shear rate is limited by edge fracture; usually cannot obtain shear-thinning data	Very high rates accessible	Maximum shear rate is limited by sample leaving cup due to either inertia or elastic effects; also 3-D secondary flows develop (instability)
Special features	Good for stiff samples, even gels; wide range of temperatures possible	Ψ_1 measurable; wide range of temperatures possible	Constant-Q or constant-$\Delta\mathcal{P}$ modes available; wide range of temperatures possible	Narrow gap required; usually limited to modest temperatures (e.g., $0 < T < 60$°C)

elongational flow



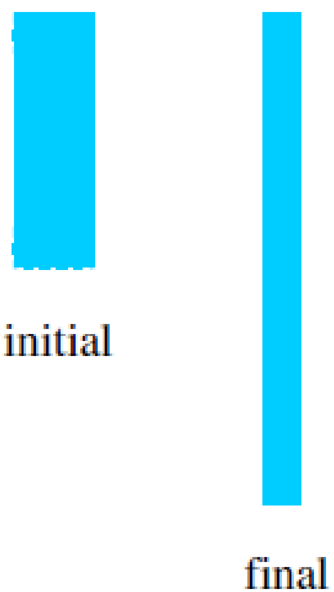




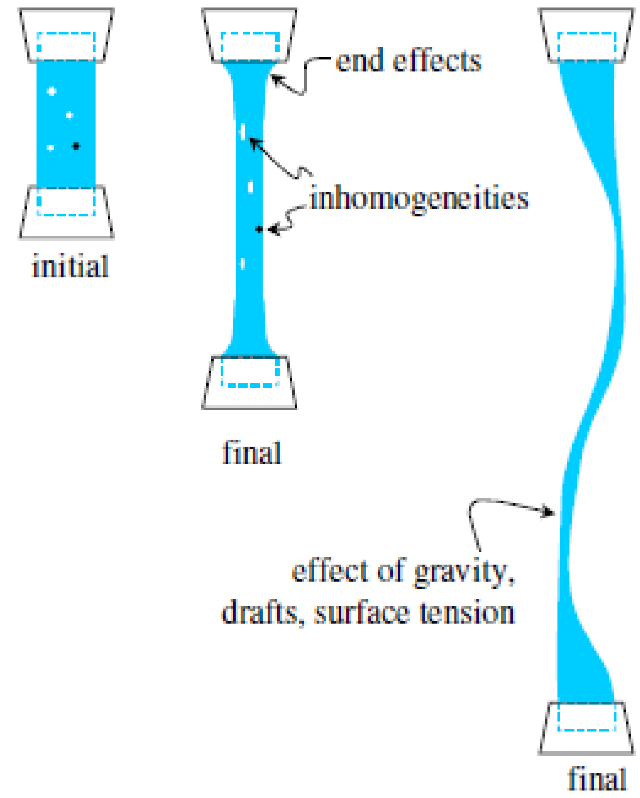


Experimental Difficulties in Elongational Flow

ideal elongational deformation



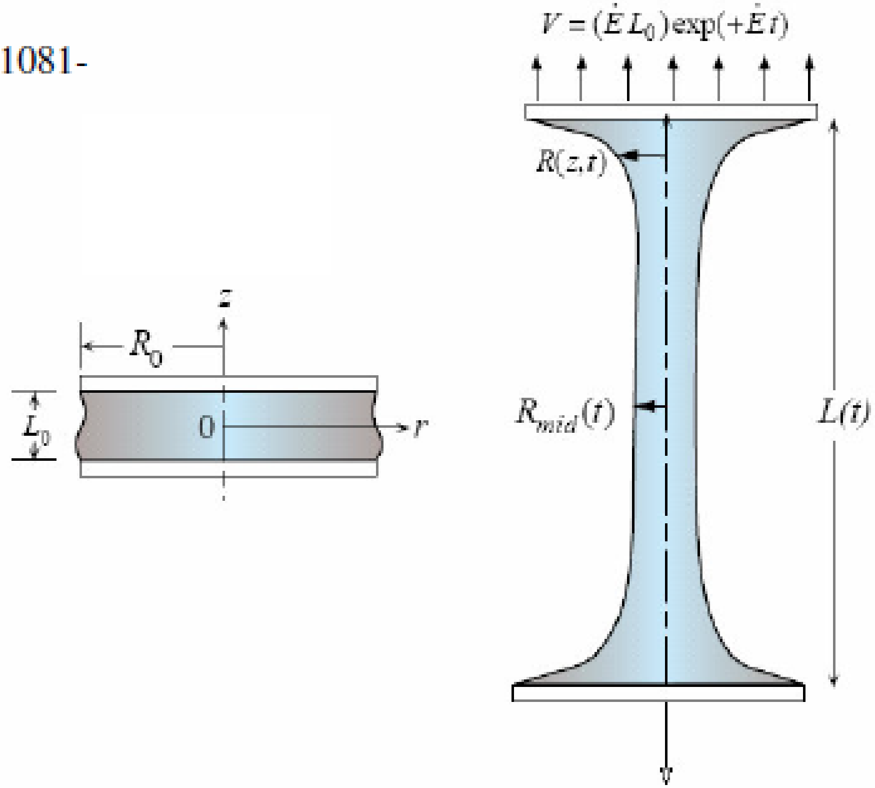
experimental challenges



Filament Stretching Rheometer (FiSER)

Tirtaatmadja and Sridhar, J. Rheol., 37, 1081-1102 (1993)

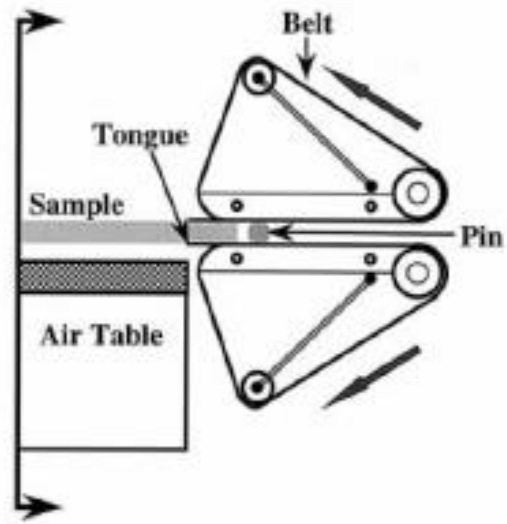
- Optically monitor the midpoint size
- Very susceptible to environment
- End Effects

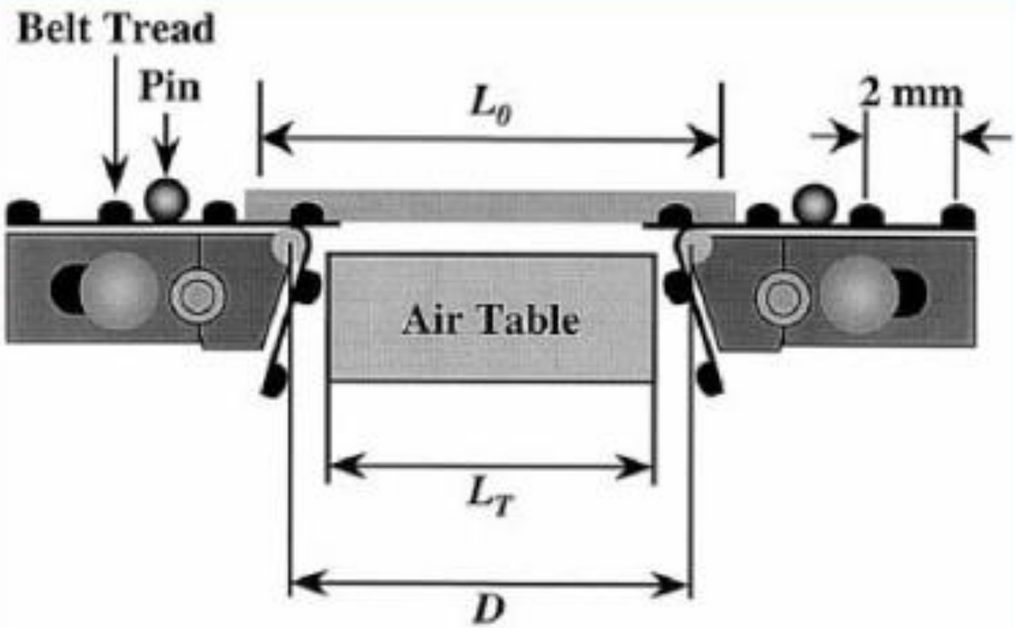


ORIGINAL CONTRIBUTION

A comparison of extensional viscosity measurements from various RME rheometers

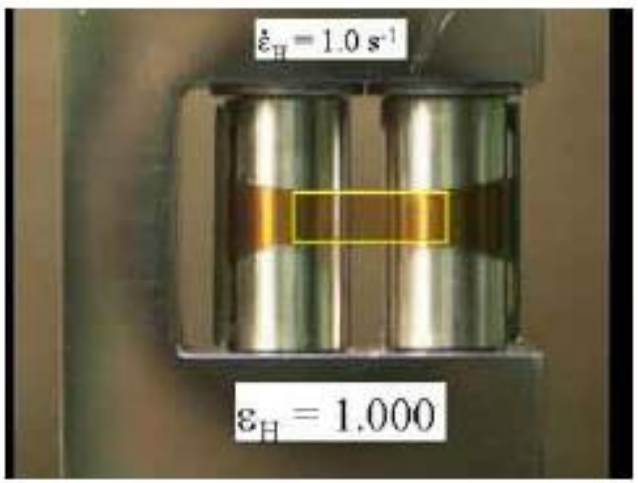
- Steady and startup flow
- Recovery
- Good for melts





Sentmanat Extension Rheometer

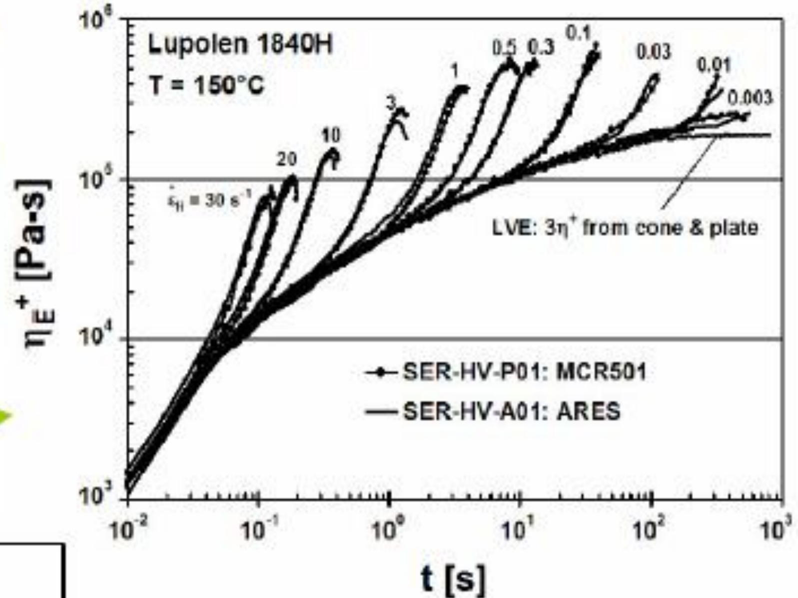
- Originally developed for rubbers, good for melts
- Measures elongational viscosity, startup, other material functions
- Two counter-rotating drums
- Easy to load; reproducible

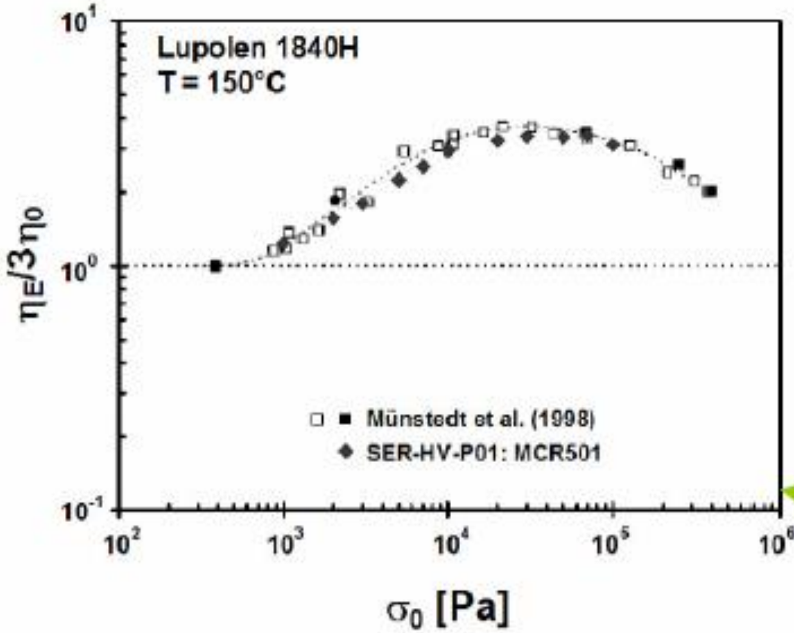


www.xpansioninstruments.com

Sentmanat et al., J. Rheol., 49(3) 585 (2005)

Comparison on different host instruments





Comparison with other instruments (literature)

CaBER Extensional Rheometer

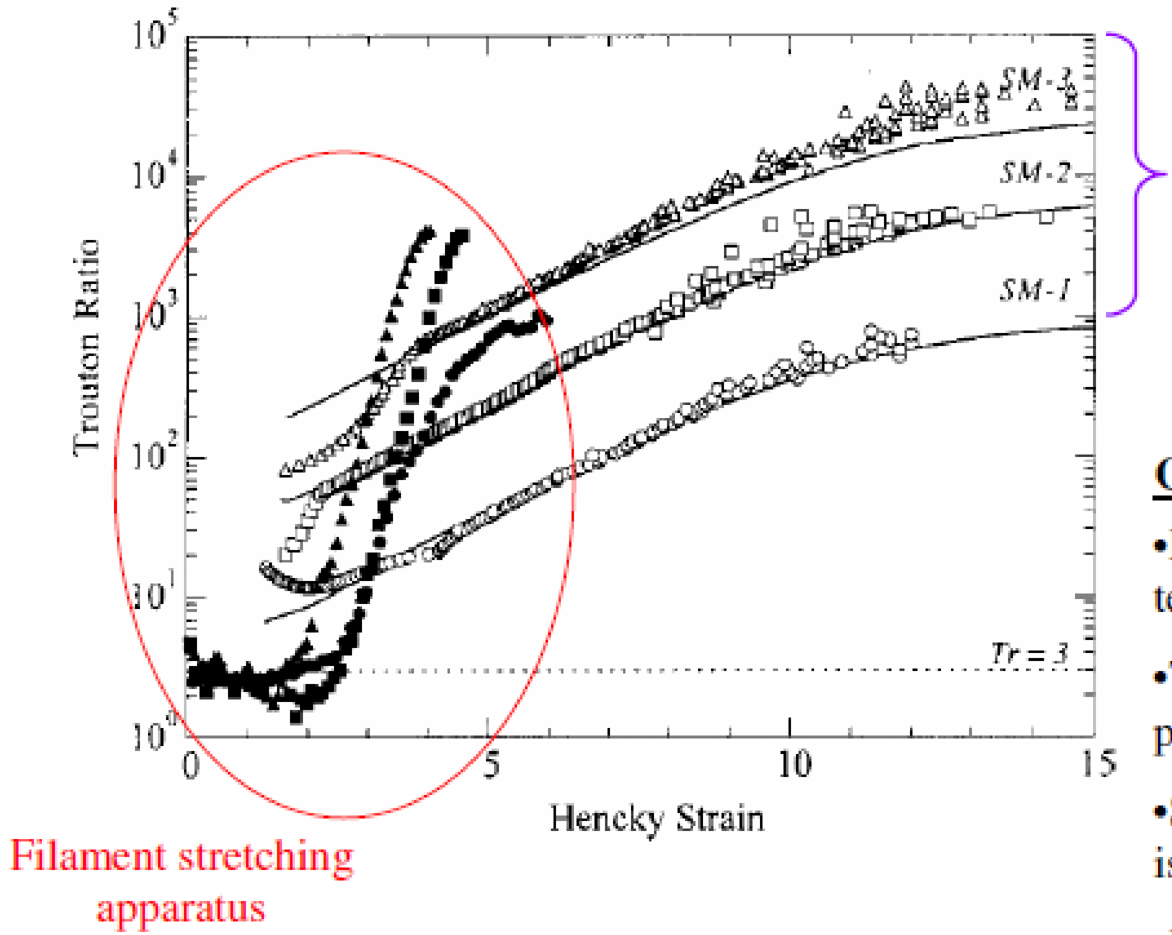
- •Polymer solutions
- •Works on the principle of capillary filament break up
- •Cambridge Polymer Group and HAAKE

For more on theory see: campoly.com/notes/007.pdf

Brochure: www.thermo.com/com/cda/product/detail/1,,17848,00.html

Operation

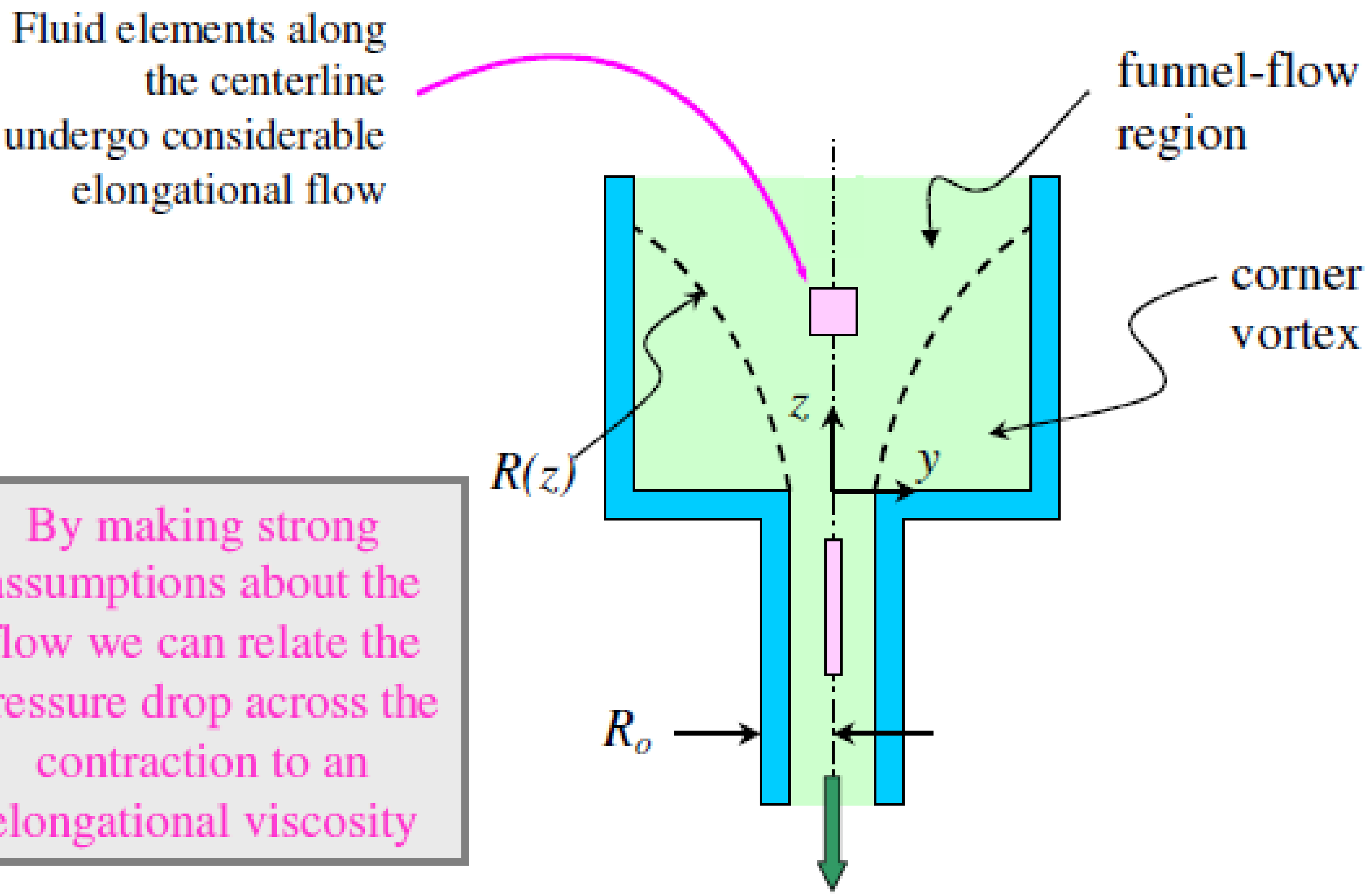
- •Impose a rapid step elongation
- •form a fluid filament, which continues to deform
- •flow driven by surface tension
- •also affected by viscosity, elasticity, and mass transfer
- •measure midpoint diameter as a function of time
- •Use force balance on filament to back out an apparent elongational viscosity



Comments

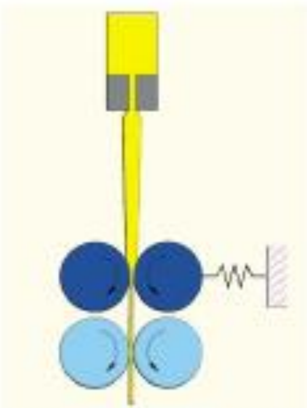
- Must know surface tension
- Transient agreement is poor
- Steady state agreement is acceptable
- Be aware of effect modeling assumptions on reported results

Elongational Viscosity via Contraction Flow: Cogswell/Binding Analysis



By making strong assumptions about the flow we can relate the pressure drop across the contraction to an elongational viscosity

Rheotens (Goettfert)



•Does not measure material functions without constitutive model
•small changes in material properties are reflected in curves
•easy to use
•excellent reproducibility
•models fiber spinning, film casting
•widespread application

from their brochure:

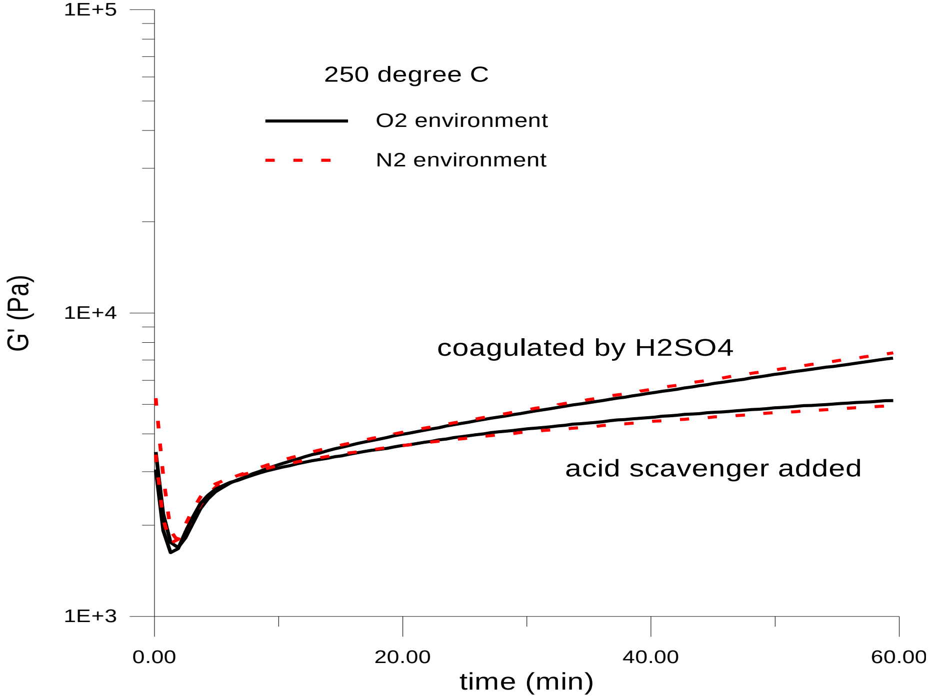
"Rheotens test is a rather complicated function of the characteristics of the polymer, dimensions of the capillary, length of the spin line and of the extrusion history"

www.goettfert.com/downloads/Rheotens_eng.pdf

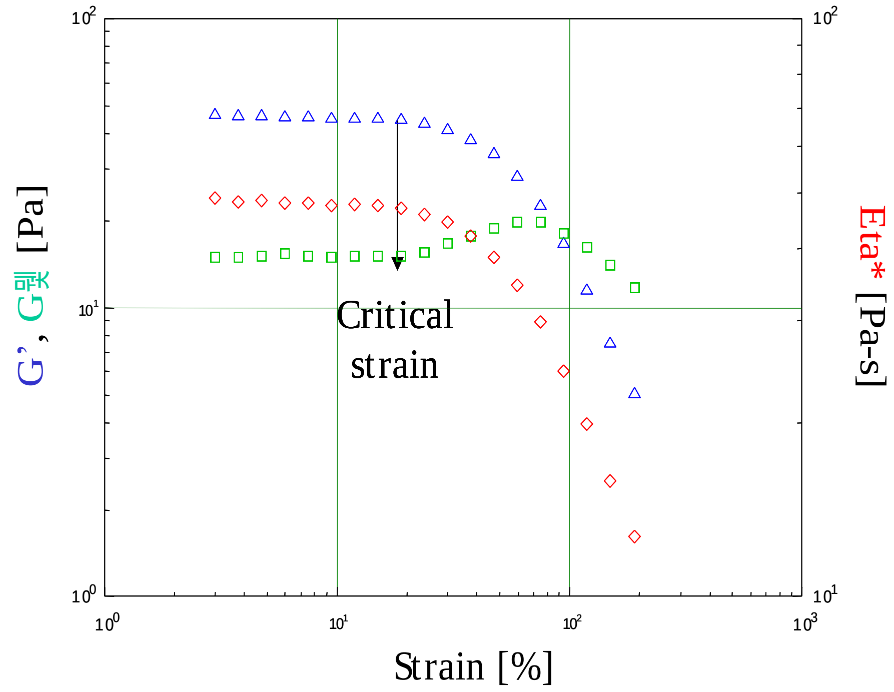
레오미터 사용시 주의점

- 시편 제조 상의 유의할 점 (pre-history)
- 열안정성
- Strain sweep
- 관성 (inertia)의 영향
- 증발의 영향
- 시료의 안정성
- Slip의 영향
- 기계적 영향 (측정가능범위)
- 기타

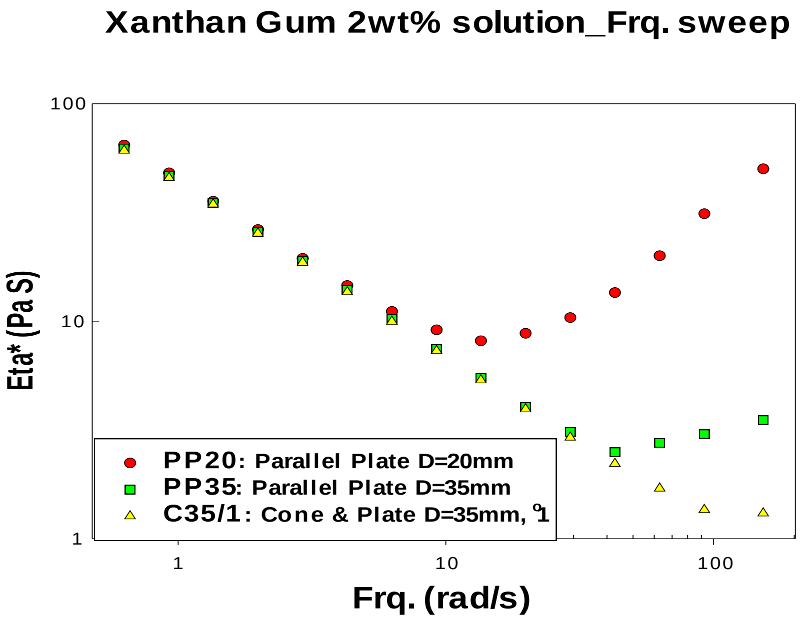
Thermal stability



Strain sweep

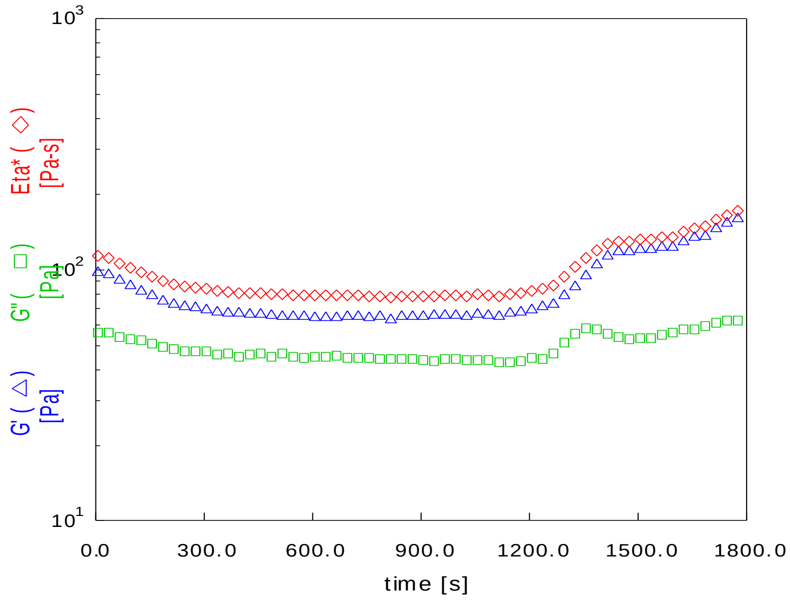


Inertia effect



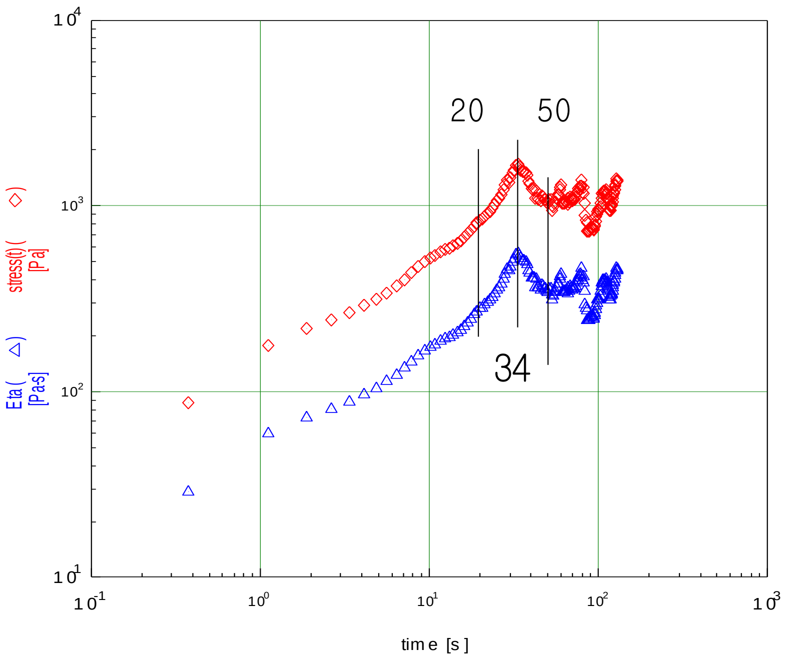
Evaporation

W/O silicone oil

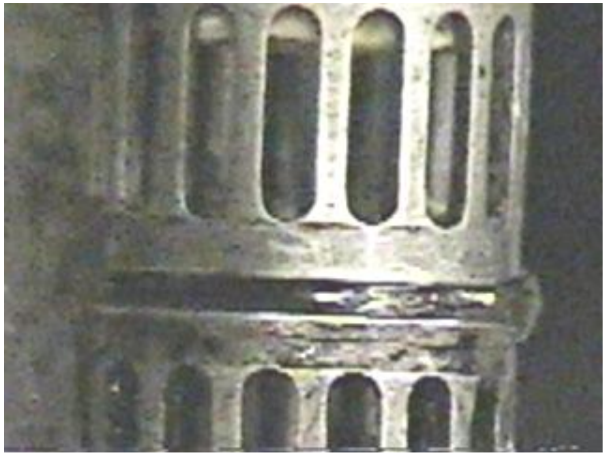


Stability

Gap 2_step rate_3__pva2%borax1%_ noE

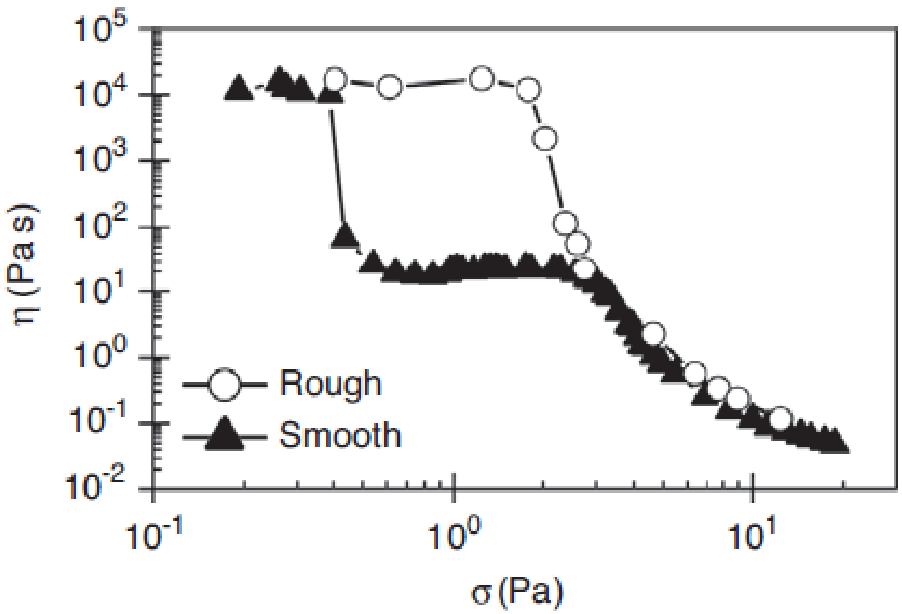


At t=15s



At t=25s

Slip



10^5
10^4
10^3
10^2
10^1
10^0
10^{-1}
10^{-2}
η (Pa s)
Rough
Smooth
10^{-1}
10^0
10^1
σ (Pa)