

Rheometry

ROTATIONAL AND SLIDING SURFACE RHEOMETERS (COUETTE DEVICES) GAP LOADING vs. SURFACE LOADING

Must compare rheometer gap h to the shear wavelength λ_s .

$$\text{Gap Loading Limit: } \frac{h}{\lambda_s} \ll 1 \quad (7-9)$$

In the gap loading limit, the shear wave propagates across the gap without damping.

$$\text{Shear Wavelength } \lambda_s = \frac{2\pi}{\omega \sqrt{\rho/G_d} \cos(\delta/2)} \quad (7-10)$$

$$\text{Surface Loading Limit: } \frac{h}{\lambda_s} \gg 1$$

In the surface loading limit, the shear wave is completely damped before it can travel across the gap. For polymer melts and concentrated solutions, $G_d \equiv \sigma_0/\gamma_0$ is large and we always operate in the gap loading limit.

TWO CLASSES OF GAP LOADING INSTRUMENTS:

1. Impose Strain and Measure Stress
2. Impose Stress and Measure Strain

GEOMETRIES OF GAP LOADING INSTRUMENTS:

1. Cone and Plate
2. Parallel Disks
3. Eccentric Rotating Disks
4. Concentric Cylinder
5. Sliding Plate

Rheometry INSTRUMENT AND TRANSDUCER COMPLIANCES

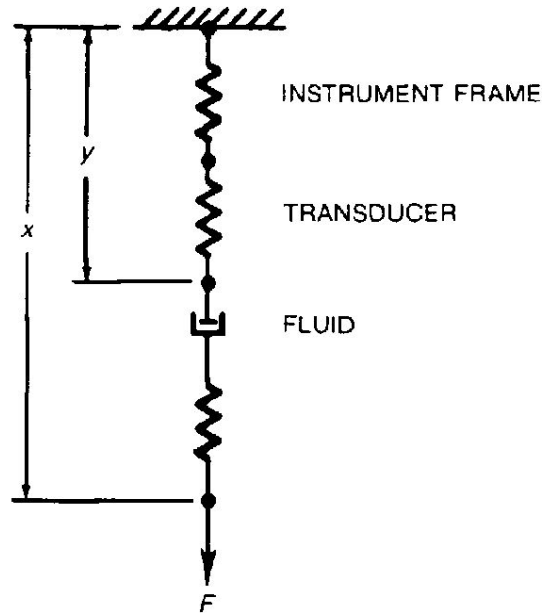


Figure 1: Simple Model for Instrument and Transducer Compliance: A Spring (the instrument and transducer) in Series with a Maxwell Element (the sample).

Actuator moves Δx
 Instrument plus transducer moves Δy
 Sample actually deforms by $\Delta x - \Delta y$

$$\frac{\Delta x - \Delta y}{\Delta x} < 1$$

and depends on time and material

Rheometry INSTRUMENT AND TRANSDUCER COMPLIANCES

Series combination of Maxwell Model and Linear Spring.

$$\text{Dashpot} \quad F = \eta \frac{dx_3}{dt}$$

$$\text{Material Spring} \quad F = Gx_2$$

$$\text{Instrument/Transducer Spring} \quad F = \frac{K}{c}x_1$$

K is a geometric constant with units of cm^{-3} .

c is the instrument/transducer compliance $(\text{dyne cm})^{-1}$.

$$\frac{K}{c}x_1 = Gx_2 = \eta \frac{dx_3}{dt}$$

$$x_1 + x_2 + x_3 = x_0 \text{ (a constant)}$$

Solution is the same as the Maxwell model, but with time scale $(\frac{c\eta}{K} + \lambda)$, where $\lambda = \eta/G$ is the material's relaxation time.

Solve to get apparent oscillatory shear moduli:

$$G'_a = \frac{\eta \left(\frac{c\eta}{K} + \lambda\right) \omega^2}{\left(\frac{c\eta}{K} + \lambda\right)^2 \omega^2 + 1} \quad \text{text has typo!} \quad (7-1)$$

$$G''_a = \frac{\eta\omega}{\left(\frac{c\eta}{K} + \lambda\right)^2 \omega^2 + 1} \quad \text{text has typo!} \quad (7-2)$$

For a known instrument/transducer compliance, one may calculate the true moduli of the material from the apparent values.

$$G' + iG'' = \frac{G'_a + iG''_a}{1 - \frac{G'_a}{k} - \frac{iG''_a}{k}} \quad (7-3)$$

Rheometry

VISCOUS HEATING

All mechanical energy input to the sample must either be stored (and hence recoverable) or dissipated as heat.

EXAMPLE: Steady-State Temperature Distribution in the Sliding Plate Rheometer

$$T(x_2) = T_0 + \frac{\eta\dot{\gamma}^2 h^2}{2k} \left[\frac{x_2}{h} - \left(\frac{x_2}{h} \right)^2 \right] \quad (7-4)$$

k is the thermal conductivity

T_0 is the temperature of the two plates.

The maximum temperature occurs at the midpoint between the plates ($x_2 = h/2$).

$$T_{max} = T_0 + \frac{\eta\dot{\gamma}^2 h^2}{8k} \quad (7-5)$$

Typical Numbers: $\eta = 10^4$ poise, $\dot{\gamma} = 10^2$ s⁻¹, $h = 0.1$ cm,
 $k = 10^4$ ergs/(s cm K)

$$\frac{\eta\dot{\gamma}^2 h^2}{8k} \cong 10\text{K}$$

Since viscosity changes with temperature, viscous heating can be very important!

END AND EDGE EFFECTS

Rheometers with moving surfaces usually have free sample surfaces (in contact with air, nitrogen or vacuum).

Cone and plate or parallel disk rheometers develop surface irregularities.

Concentric cylinder rheometers exhibit rod-climbing (the Weissenberg effect).

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CONE AND PLATE RHEOMETERS

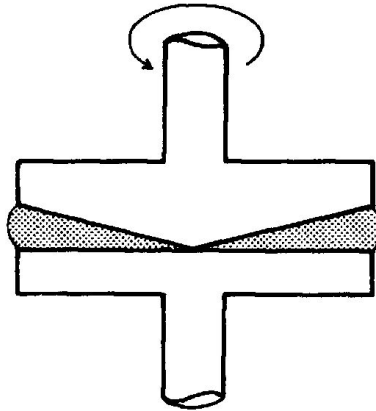


Figure 2: The Cone and Plate Rheometer.

Advantages:

- (1) Small sample size (roughly 1 gram)
- (2) Uniform shear rate
- (3) Easy to load and clean

$$\text{Shear Rate} \quad \dot{\gamma} = \frac{\Omega}{\Theta_0} \quad (7-11)$$

Ω is the angular velocity (rad/s).

Θ_0 is the cone angle.

$$\text{Shear Stress} \quad \sigma = \frac{3M}{2\pi R^3} \quad (7-12)$$

M is the torque (dyne cm).

R is the radius (cm).

$$\text{Normal Stress Difference} \quad N_1 = \frac{2F}{\pi R^2} \quad (7-13)$$

F is the normal force (dynes).

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PARALLEL DISK RHEOMETERS

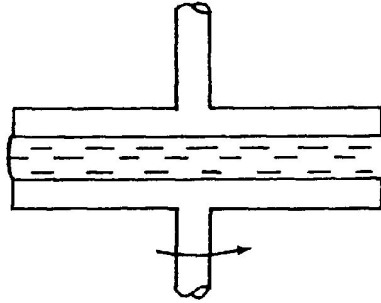


Figure 3: The Parallel Disk Rheometer.

Advantages:

- (1) Small sample size (roughly 1 gram)
- (2) Can change temperatures without reloading sample
- (3) Easy to load and clean

Disadvantage: Shear rate is not uniform (OK for linear viscoelasticity, but parallel disks are no good for nonlinear studies).

$$\text{Shear Rate} \quad \dot{\gamma} = \frac{\Omega r}{h}$$

Ω is the angular velocity (rad/s).

h is the sample gap height (cm).

The shear rate in the parallel disk rheometer varies from **zero** at the center to a maximum at the outer edge ($r = R$) $\dot{\gamma}_{max} = \Omega R/h$.

Total torque on either disk in steady shear is

$$M = \int_0^R 2\pi r^2 \sigma dr = \frac{\pi \eta \Omega R^4}{2h}$$

for a Newtonian fluid ($\sigma = \eta \dot{\gamma}$).

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ECCENTRIC ROTATING DISKS

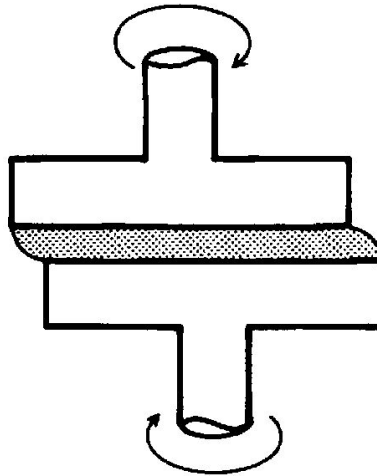


Figure 4: The Eccentric Rotating Disk Rheometer.

One disk is forced to rotate at fixed angular velocity Ω (rad/s). The second disk rotates freely at the same angular frequency. ERD is a simple way to do oscillatory shear with a steady rotation motor.

Advantages are identical to the parallel disk rheometer.

Disadvantage: Only performs oscillatory shear measurement.

$$\eta' = \frac{G''}{\omega} = \frac{F_x h}{\pi \Omega d R^2} \quad (7-17)$$

$$G' = \frac{F_y h}{\pi d R^2} \quad (7-18)$$

F_x is the force parallel to plates.

F_y is the force normal to plates.

h is the gap height, d is the eccentricity offset,

R is the disk radius.

The ERD device was originally developed by Bryce Maxwell in the 1960's, and is now virtually extinct.

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CONCENTRIC CYLINDER RHEOMETER

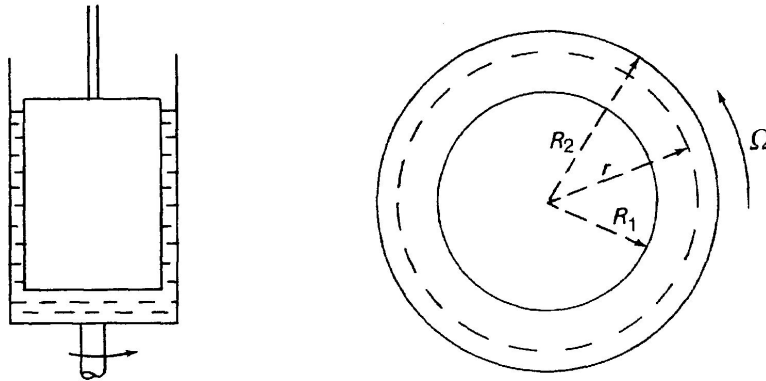


Figure 5: The Concentric Cylinder Rheometer (side view on left and top view on right).

This rheometer was invented by Maurice Couette in 1890.

Advantages:

- (1) For large radii the shear rate is nearly constant.
- (2) Ideally suited for pourable liquids.

Disadvantages:

- (1) No way to load high viscosity polymer melts.
- (2) Large sample volume (typically 3 – 30 grams).

Ω = angular velocity of the outer cylinder

$$v_\theta = v_\theta(r)$$

$$v_r = v_z = 0$$

$$\text{steady state } \partial \vec{v} / \partial t = 0$$

constant pressure

negligible gravity

$$\text{Navier-Stokes Equation} \quad \eta \left[\frac{\partial}{\partial r} \left(\frac{1}{r} \frac{\partial}{\partial r} (r v_\theta) \right) \right] = 0$$

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CONCENTRIC CYLINDER RHEOMETER

Outer cylinder rotates at Ω , thus $v_\theta = \Omega R_2$ at $r = R_2$

Inner cylinder is stationary, so $v_\theta = 0$ at $r = R_1$

$$\left[\frac{\partial}{\partial r} \left(\frac{1}{r} \frac{\partial}{\partial r} (rv_\theta) \right) \right] = 0$$

Integrate once: $\frac{\partial}{\partial r} (rv_\theta) = Cr$

Integrate again: $rv_\theta = C_1 r^2 + C_2$

$$v_\theta = C_1 r + \frac{C_2}{r}$$

at $r = R_1$, $v_\theta = 0$, $C_1 = -\frac{C_2}{R_1^2}$, so

$$v_\theta = C_2 \left(\frac{1}{r} - \frac{r}{R_1^2} \right)$$

at $r = R_2$, $v_\theta = \Omega R_2 = C_2 \left(\frac{1}{R_2} - \frac{R_2}{R_1^2} \right)$, so

$$C_2 = \frac{\Omega}{\left(\frac{1}{R_2} - \frac{1}{R_1^2} \right)}$$

$$v_\theta = \Omega \frac{\left(\frac{1}{r} - \frac{r}{R_1^2} \right)}{\left(\frac{1}{R_2} - \frac{1}{R_1^2} \right)}$$

Angular Velocity $\frac{v_\theta}{r} = \Omega \frac{\left(\frac{1}{r^2} - \frac{1}{R_1^2} \right)}{\left(\frac{1}{R_2^2} - \frac{1}{R_1^2} \right)}$

Rheometry CONCENTRIC CYLINDER RHEOMETER

$$\text{Shear Rate} \quad \dot{\gamma} = \left| r \frac{\partial(v_\theta/r)}{\partial r} \right| = \frac{2\Omega}{r^2 \left(\frac{1}{R_1^2} - \frac{1}{R_2^2} \right)}$$

$$\text{Shear Stress} \quad \sigma = \eta \dot{\gamma} = \frac{2\eta\Omega}{r^2 \left(\frac{1}{R_1^2} - \frac{1}{R_2^2} \right)}$$

$$\text{Torque on the inner cylinder} \quad T = \int_A R_1 \sigma dA = \int_0^L \int_0^{2\pi} (R_1 \sigma) R_1 d\theta dz = 2\pi R_1^2 L \sigma$$

$$\text{Evaluate Shear Rate at } r = \bar{R} = \frac{R_2 + R_1}{2}$$

$$\dot{\gamma} = \frac{2\Omega}{\bar{R}^2 \left(\frac{1}{R_1^2} - \frac{1}{R_2^2} \right)} = \frac{2\Omega R_1^2 R_2^2}{\bar{R}^2 (R_2^2 - R_1^2)} = \frac{2\Omega R_1^2 R_2^2}{\bar{R}^2 (R_2 + R_1)(R_2 - R_1)} = \frac{\Omega R_1^2 R_2^2}{\bar{R}^3 (R_2 - R_1)}$$

Small gap approximation:

$$R_1 \cong R_2 \cong \bar{R}$$

$$\dot{\gamma} \cong \frac{\Omega \bar{R}}{(R_2 - R_1)}$$

When the gap is small, the shear rate is nearly uniform everywhere and is essentially the same as simple shear between two flat plates.

$$\dot{\gamma} = \frac{v}{h} \quad \text{where } v = \Omega \bar{R} \text{ and } h = R_2 - R_1$$

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SLIDING PLATE RHEOMETER

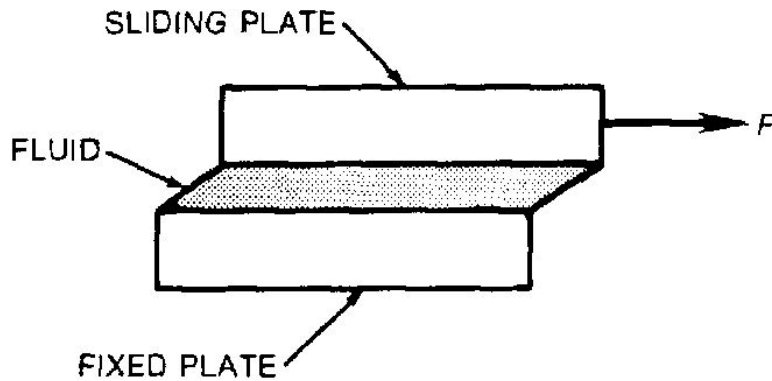


Figure 6: The Sliding Plate Rheometer.

Advantages:

- (1) Shear rate is uniform
- (2) Simple shear \Rightarrow Simple equations

Disadvantages:

- (1) Difficult to load and clean
- (2) Requires fairly large sample size (5 – 10grams)

$$\text{Shear Stress} \quad \sigma = \frac{F}{A} \quad (7-19)$$

$$\text{Shear Strain} \quad \gamma = \frac{X}{h} \quad (7-20)$$

X is the displacement of the moving plate.

h is the gap height between plates.

$$\text{Shear Rate} \quad \dot{\gamma} = \frac{V}{h} \quad (7-21)$$

$V = dX/dt$ is the velocity of the moving plate.

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SHEAR-SANDWICH RHEOMETER

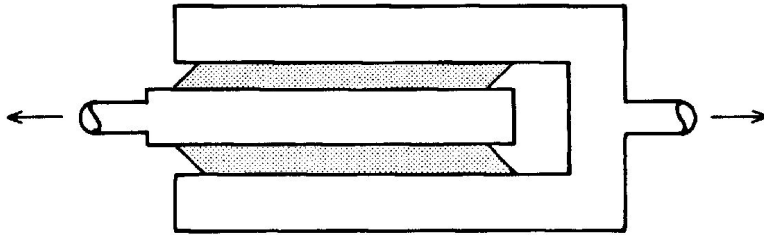


Figure 7: The Shear-Sandwich Rheometer.

The shear sandwich is simply two sliding plate arrangements. The advantages and disadvantages are the same as the sliding plate rheometer. One additional advantage of this design is the **symmetry** that keeps each slice of sample in simple shear.

This geometry is popular for rheo-optical measurements because shear rate is uniform and there are no curved surfaces.