Lab 14: Torque Rheometer

The oscillatory torque rheometer is an instrument that can measure the complex viscosity or complex shear modulus for a material. The complex modulus is important for viscoelastic materials. The storage modulus is related to the loss viscosity and the loss modulus to the storage visocsity so that, for example, $\eta' = G''/\omega$. For a Newtonian fluid we expect $G'' \sim \omega$. For a Newtonian fluid we expect $G' \sim \omega^2$. This is a signature of flow. Other power-law relationships are detailed in the chapter by Ron Larson on polymer rheology. The plot below shows the flow regime at low frequency. At higher frequency a “plateau modulus” is observed that reflects the entanglement network of the polymer. The molecular weight between entanglements can be obtained in this regime. At highest frequency a power law of ½ is observed reflecting Rouse motion of the chain elements at sizes smaller than the entanglement network or mesh size. The loss modulus displays a non-monotonic behavior. This leads to the situation that the storage modulus is larger than the loss modulus at some frequencies then there is a crossover where the loss modulus is larger. At the point where the loss exceeds the storage we observe yield in the material, i.e. the yield point. The inverse of this frequency is the relaxation time. These characteristic relaxation times are associated with structural features (through theory and models) so rheologists refer to a structural feature where $G'' = G'$. The crossover seen in Figure 3.29 refers to the mesh size of the entangled chains. A second crossover at high frequency is related to the persistence length where Rouse relaxation is observed. It is important to keep in mind that rheology does not actually measure structure, “*morphology from rheology is theology*” R.S. Stein.

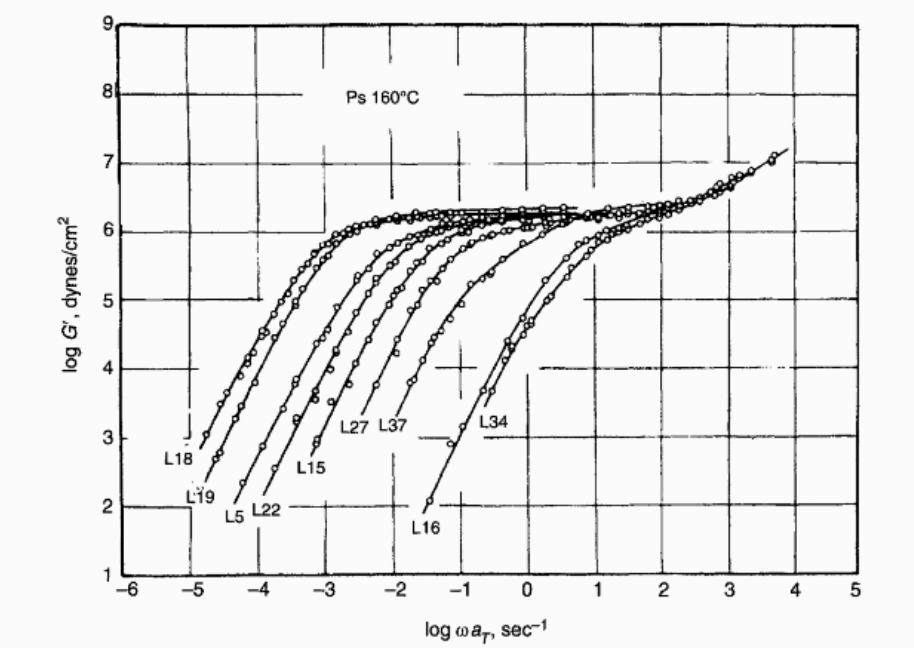

Figure 3.22 Storage modulus, G', as a function of frequency reduced to 160°C for nearly monodisperse polystyrenes of molecular weight ranging from 580,000 to 47,000, from left to right. (Reprinted with permission from Onogi et al., Macromolecules 3:109. Copyright 1970, American Chemical Society.)

From Ron Larson the Rheology of Complex Fluids (on UC electronic library chapter on entangled flow.)

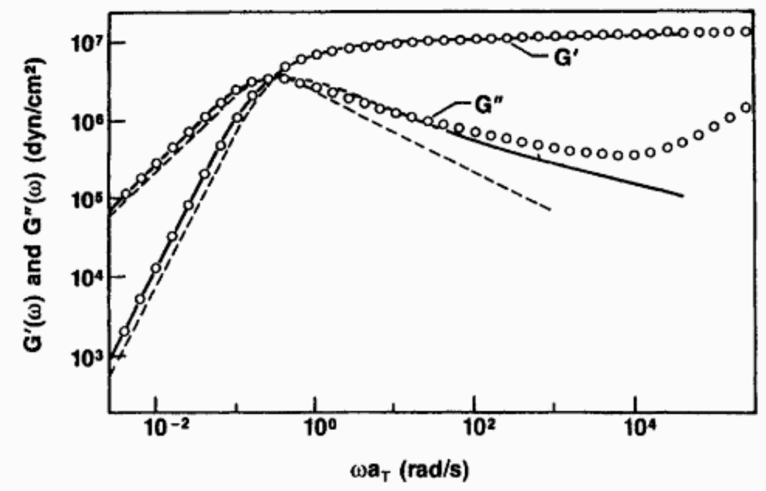

Figure 3.29 Linear moduli G' and G'' versus frequency shifted via time–temperature superposition to 27°C for a polybutadiene melt of molecular weight 360,000 and of low polydispersity. The dashed line is the prediction of reptation theory given by Eq. (3-67); the solid line includes effects of fluctuations in the length of the primitive path. (From Pearson 1987.)

From Ron Larson the Rheology of Complex Fluids (on UC electronic library chapter on entangled flow.)

We will measure several polybutadiene samples some with carbon black added. The samples will be measured at several different temperatures and the WLF equation will be used to create a master curve similar to figure 3.29. For the filled systems a change in the polymer viscoelastic behavior is expected.

Objective: Measure the dynamic viscosity of polybutadiene and G' G" as a function of frequency at several temperatures. Use time-temperature superposition (TTS) of data at various temperatures. Investigate the effects of filler on the data and TTS.

Materials: Polybutadiene rubber, filled PBD rubber.

Procedure: The Procedure will follow the operating manual for the rheometer

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Analysis:
1) Plot the storage modulus, loss modulus, and tan delta at different temperatures and frequencies.
2) Describe the curves.
3) Determine the plateau modulus and the entanglement molecular weight.
4) Plot a Cole-Cole Plot and determine if the material displays a single relaxation time and other information available from the Cole Cole Plot
5) Perform a time temperature superposition to show the master curve.
6) Compare the filled and unfilled samples.