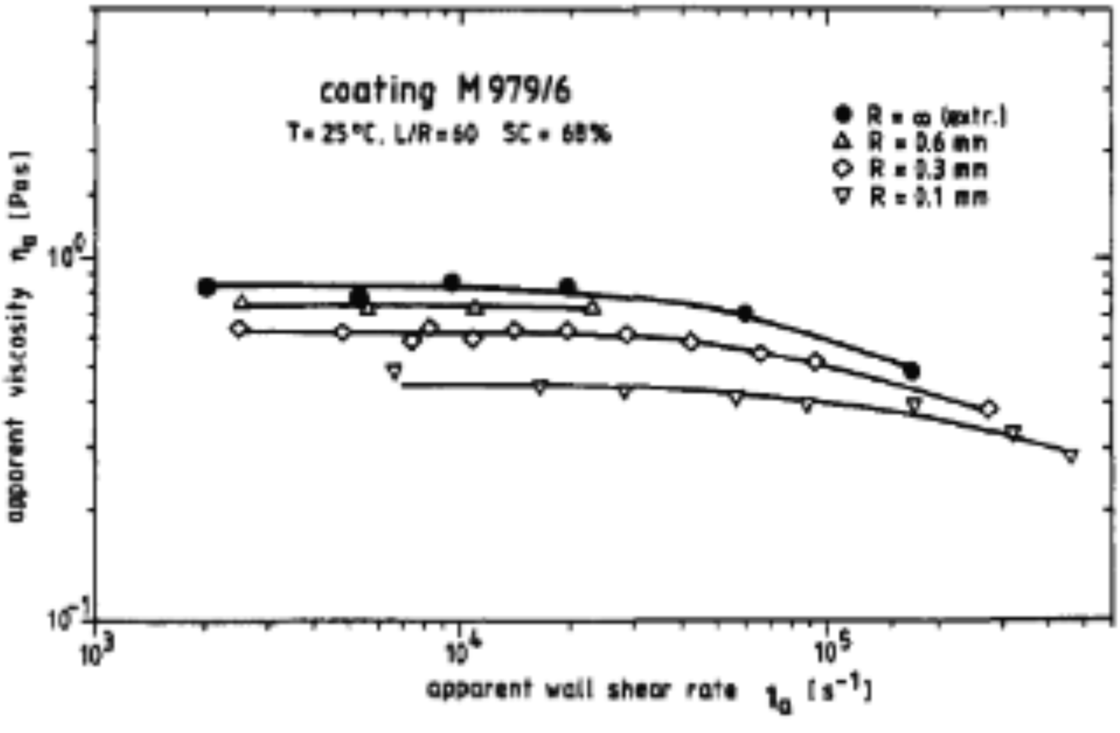
14. Data Interpretation Answers

These questions have been provided by affiliates at the University of Minnesota and the K U Leuven University, Belgium as well as Randy Ewoldt (University of Illinois Champaign-Urbana), Gerry Fuller (Stanford University) Gareth McKinley (Massachusetts Institute of Technology), Sarah Cotts (TA Instruments), Robert Prudhome (Princeton), Ali Mohraz (UC Irvine), Eric Furst (Delaware), and Kelly Schultz (Lehigh University).

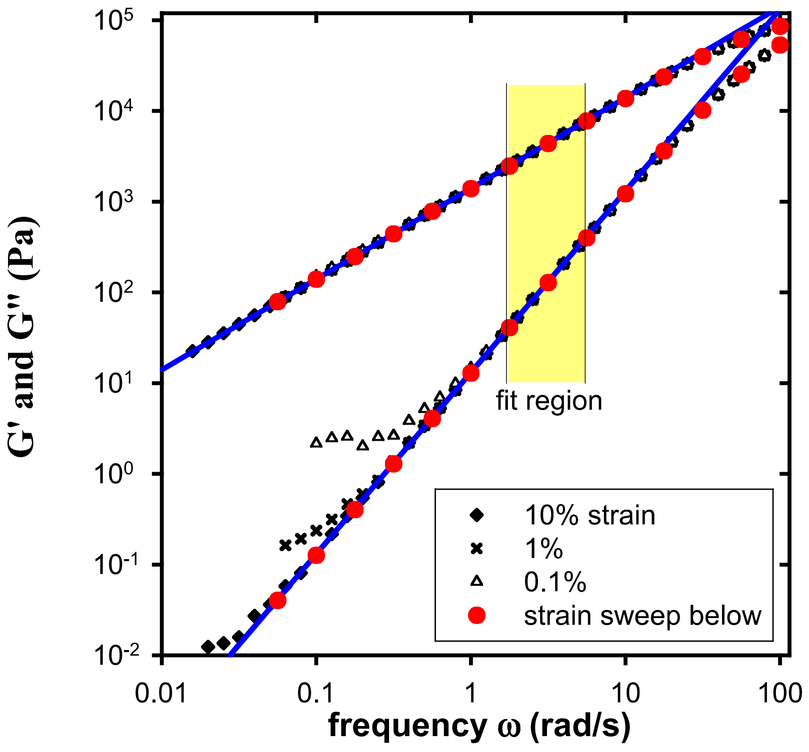
Answer 1. Capillary Rheometry of Clay Paper Coating

A slip layer forms at the wall in this clay filled suspension. The slip shows up as differences in the apparent viscosity at different gaps (diameters).



Answer 2. Low Frequency SAOS

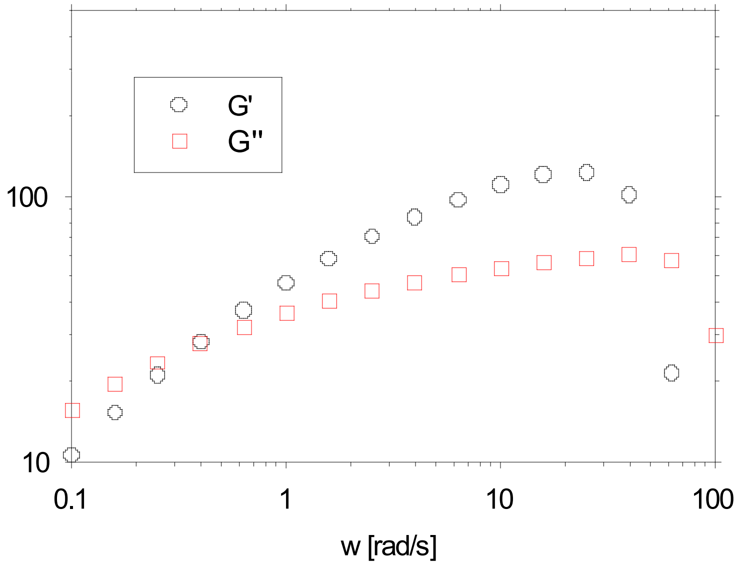
A rough consideration is that G' at low frequency is 25 times lower G", so the torque signal attributable to G' is only 1/25th of the total signal. The signal may be too low to resolve G' accurately, even though it is enough to resolve G". To verify that the sample really does show terminal behavior, a larger torque signal is necessary. One could try a higher strain, up to the linear region limit, or use larger diameter plates. Torque is proportional to the plate radius cubed, so that would be a powerful way to increase the signal. For more on the subject of terminal region data accuracy, see "How do I know if my phase angles are correct?" (*Rheology Bulletin*, 76(2) July 2007) The plot below from that article shows how G' can erroneously deviate from the terminal slope when the applied strain -- or resulting torque -- is too low.

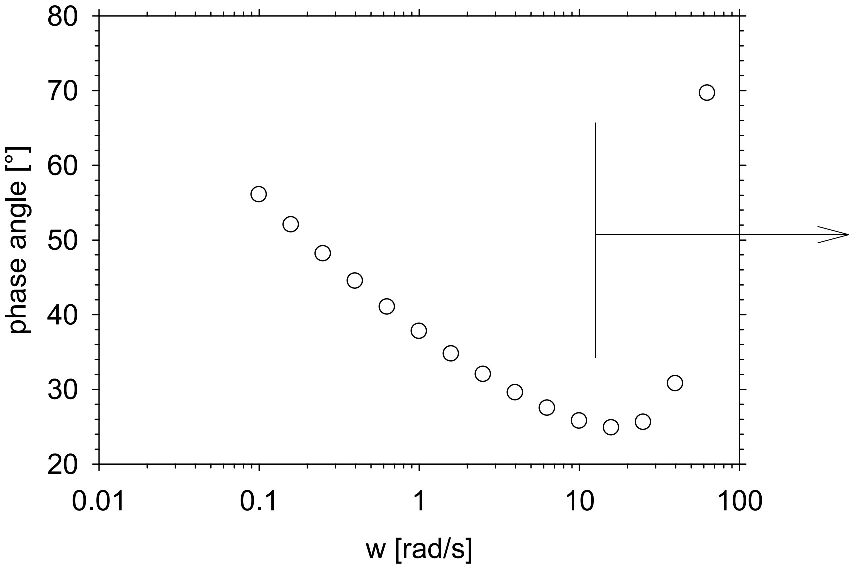


Answer 3. SAOS of PIB Solution

Instrument rotational inertia at high frequency will affect the torque and the phase angle. Recall that a purely elastic response should have raw phase difference=0° (torque ~ displacement), whereas a purely viscous response has raw phase = 90° (or torque ~ rate-of-displacement). The inertial response of the oscillation instrument mass, with no sample, would have torque ~ acceleration, i.e. raw phase difference = 180°.

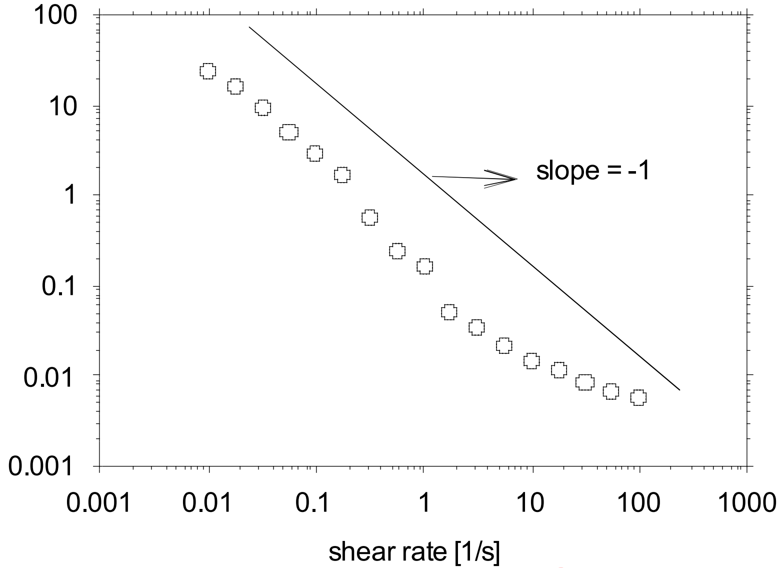
One can ask the software for the raw phase angle information, which can be seen to increase beyond about 10 rad/s. At high frequency for a polymer melt the phase angle should decrease. Here inertia is dominating the measurements.



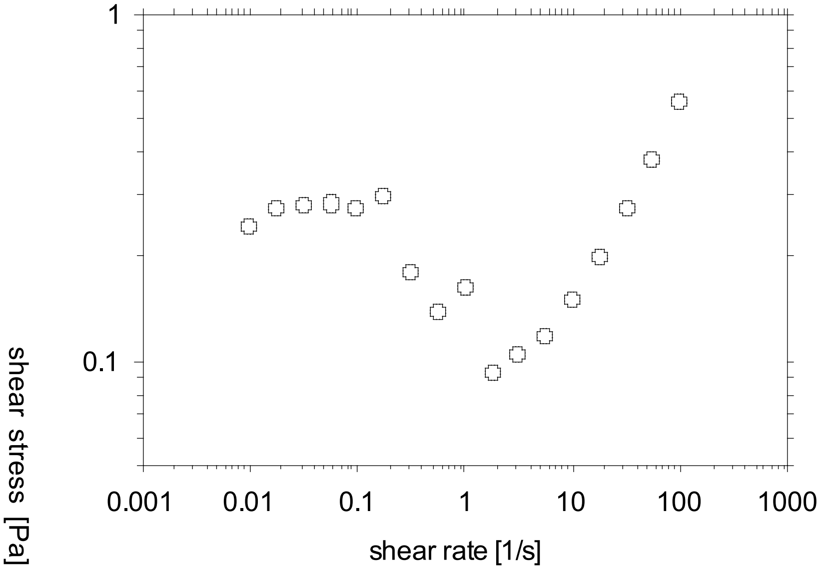


Answer 4. Silica Dispersion

Sedimentation is the problem with this suspension, which causes the apparent viscosity to decrease over time, and the shear stress response decreases so much that the stress vs. shear-rate curve is *non-monotonic*. Note that this issue is quickly revealed with the stress vs. shear-rate plot below…an encouragement to look at rheological data from many different perspectives! The non-monotonicity is a physically unstable and unlikely rheological response, since it implies that it is somehow easier (lower stress) to move faster (high shear-rates).



SEDIMENTATION !!

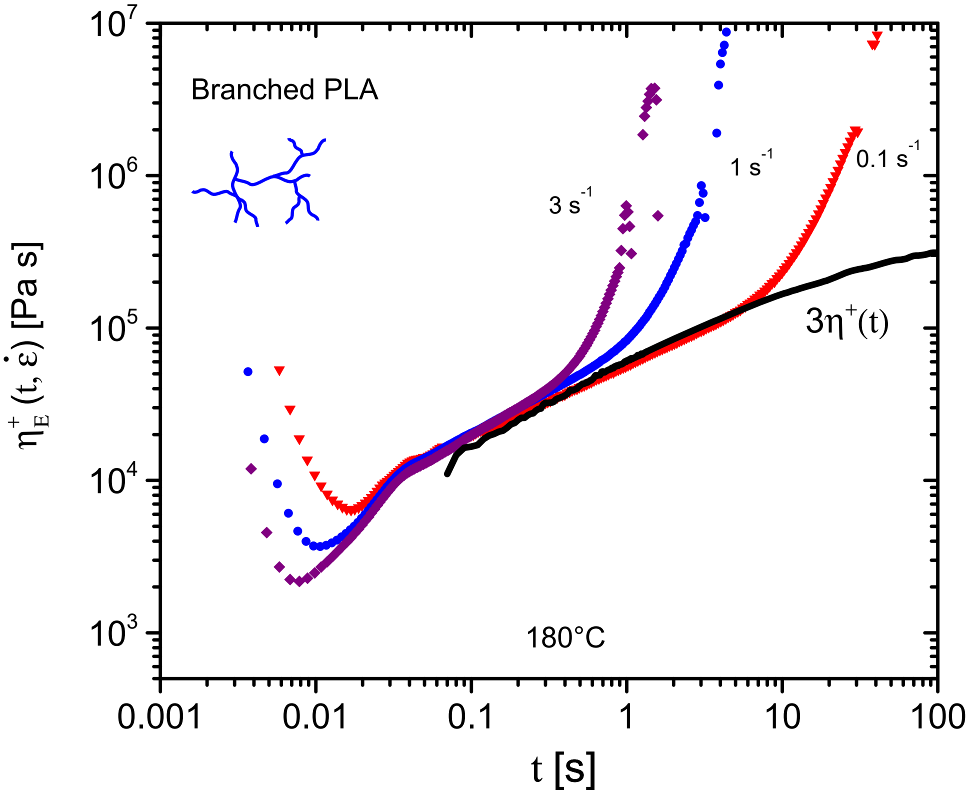


Answer 5. Extensional Viscosity

The data below 30 ms is suspect because it takes the motor some time to accelerate. The sample also may have some pretension or slack in it which alters the initial torque. Also remember this is a log-log plot. 30 ms represents a very small strain.

The break in the extensional viscosity data at extension rates of 1 and 3/s may be due to the sample wrapping around itself. Note that it occurs at about a strain of 3 for both and that at 0.1/s the extensional viscosity measurement was stopped at a strain of > 3.

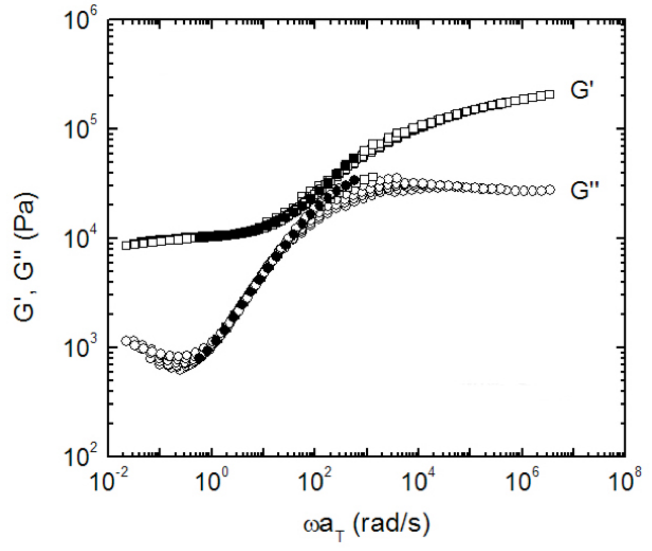
The strong increase in extensional viscosity at Hencky strain of ~1 at each strain rate is a clear indication of long chain branching. We have added to this plot data from cone and plate measurement of the start-up of steady shear viscosity. As expected for small strains the transient extensional viscosity should be 3η+. (A.T. Hedegaard, L. Gu, C.W. Macosko, Effect of extensional viscosity on cocontinuity of immiscible polymer blends, J. Rheol. (N. Y. N. Y). 59 (2015) 1397–1417. L. Gu, Y. Xu, G. Farnhorst, C.W. Macosko, Star vs. Long Chain Branching of PLA with Multifunctional Aziridine, J. Rheol, in press, 2017)



Answer 6. SAOS of a Gel

a. Does time-temperature superposition “work”, or not, in this system?

Not perfectly, but actually pretty well. For any polymer system that is not a homopolymer melt or solution, to achieve this kind of approximate master curve is unusual.



b. Is the reference temperature likely above, below, or equal to 90 ºC?

The data taken at 90 ºC extend up to almost 1000 rad/s, which is higher than most commercial rheometers can access. This suggests the reference temperature is above 90 ºC. (In fact, it is 120 ºC).

c. Were the data at the highest reduced frequency taken at low temperature or high temperature?

The higher frequency data correspond to faster motions, or motions on shorter lengthscales. To slow them down and bring them into the rheometer range, you need to cool the sample.

d. There are two plateaus in G’, one at high reduced frequency (above 10^4 rad/s) and one at low frequency (below 10 rad/s). This is unusual, but not unprecedented. What, if anything, does this tell you about whether this system is a chemical gel, a physical gel, or some combination?

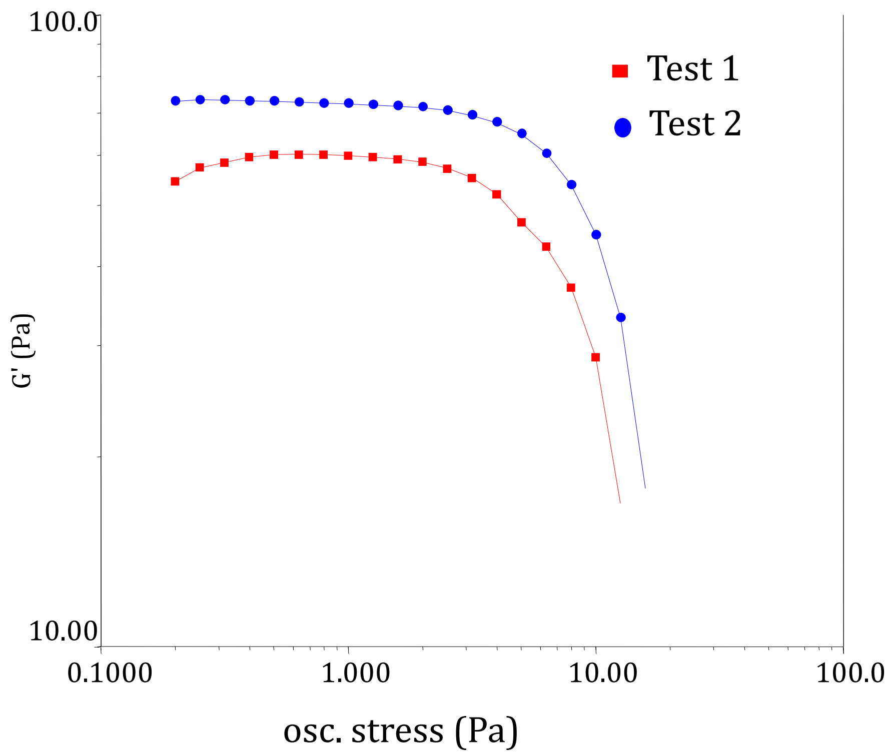
The high frequency plateau cannot be due to chemical crosslinks, because otherwise the plateau would continue to infinitely low frequency. The relaxation process evident near 100 rad/s tells us the higher plateau is due to physical crosslinks. For the lower frequency plateau the answer is not so obvious, but it too is due to physical interactions, see the following answer.

e. Is this sample a viscoelastic liquid or viscoelastic solid? What is the basis for your answer?

The upturn in G” at lowest frequencies hints at a relaxation process that is too slow to observe (if it were truly a solid, G” would not increase as frequency is lowered). Thus, the material is actually a viscoelastic liquid, even if solid on the timescales probed in the rheometer.

The system in question is actually an ABA triblock copolymer, which forms a network by self-assembly of the poorly solvated A blocks into micelles. The first relaxation process is due to pull-out of the A blocks from the micelles, allowing partial stress relaxation. However, the polymer concentration is so high (40% in this case) that the micelles are congested, which leads to the second plateau. If you wait long enough, the sample will flow at the reference temperature.

Answer 7. Latex Suspension: Linear Region



Test 1 is still recovering from the deformation during loading. Paint is typically thixotropic; its microstructure breaks down under shear and takes time to rebuild. Test 2 was given a few minutes to recover before the start of the test. A pre-shear followed by an oscillation measurement at low strain can be used to determine the time needed for the sample to recover.

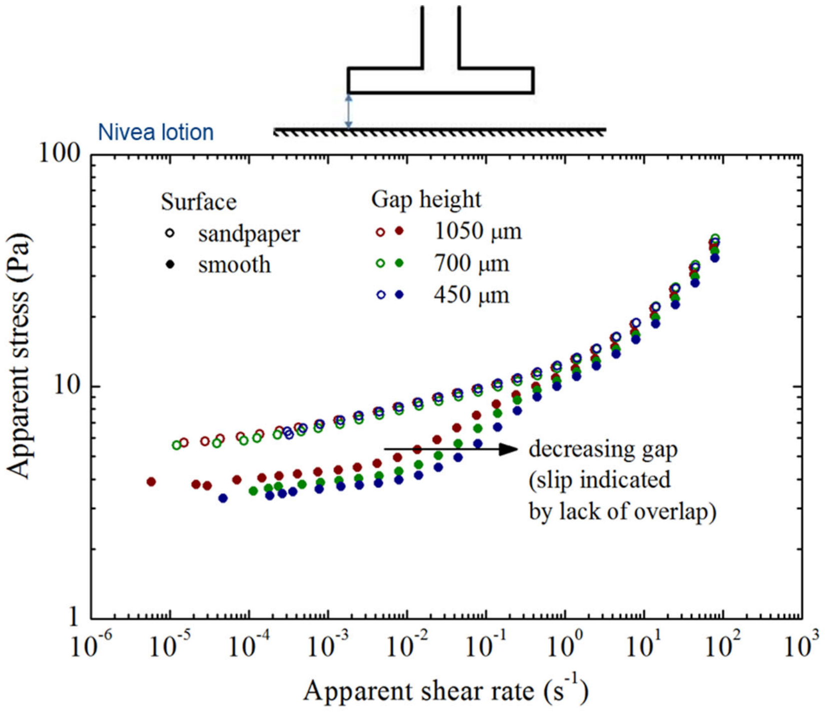
Answer 8. Parallel disk measurement and checking for slip.

a) Overlapping data is good, because it indicates that the properties do not change, even when tested with different geometries. This is true for "intrinsic" material properties.

b) The lack of overlap indicates a problem. Assumptions about stress and strain-rate may be wrong, there may be slip, or confinement effects. The non-overlapping data is bad.

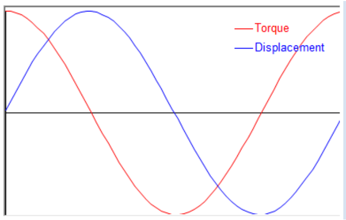
c) True material properties do not depend on the test geometry (type, size, etc.). Changing the geometry checks for slip but also other potential issues (confinement effects, edge fracture, etc.). For this material, we could also try a different diameter disk, a different diameter cone, or a concentric cylinder geometry. A concentric vane geometry may be particularly good with slippery materials

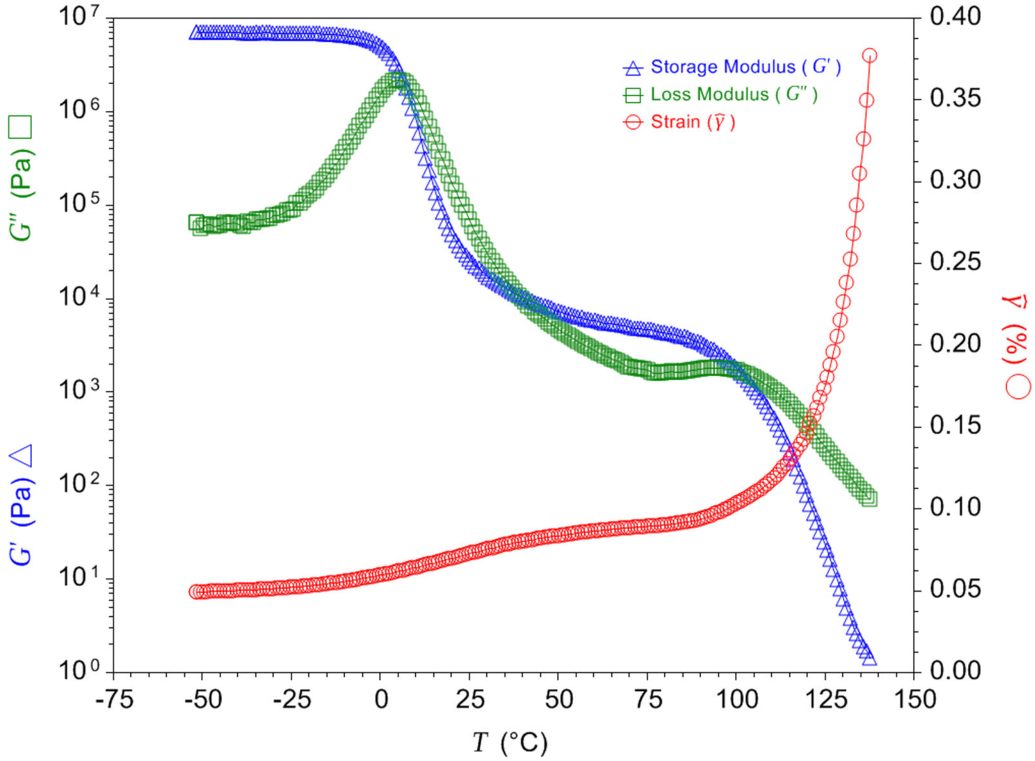
d) Bonus: slip and confinement effects shift data differently. Slip shifts data to the right with decreasing gap, because the apparent shear rate (what you think is happening to the material) is higher than the true shear rate. The problem is exaggerated with smaller gaps, since shear rate is calculated as velocity divided by the gap. Confinement usually increases the stress, shifting data up with decreasing gap (not observed here). Confinement (e.g. of the emulsion drops in the lotion) usually makes the material more difficult to flow, all other things being equal.



Answer 9. Temperature Ramp on a Hot Melt Adhesive

The raw signal of Stress and Strain (or Torque and Displacement) show sinusoidal stress response. Beyond this qualitative indicator, software can monitor 3rd and 5th harmonic of stress, which indicate non-linear response. This can be used as an indicator the increase in strain has gone beyond the linear viscoelastic region.





Storage Modulus (G')
Loss Modulus (G'')
Strain ($\tilde{\gamma}$)
G'' (Pa) □
G' (Pa) △
$\tilde{\gamma}$ (%) ○
10^7
10^6
10^5
10^4
10^3
10^2
10^1
10^0
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-75
-50
-25
0
25
50
75
100
125
150
T (°C)

Answer 10. Shear-thinning data: what to believe?

Which of these data do you believe? Why? (hint: proteins can behave like surfactants)

Original data from:
Sharma V, Jaishankar A, Wang Y-C, McKinley GH (2011) Rheology of globular proteins: apparent yield stress, high shear rate viscosity and interfacial viscoelasticity of bovine serum albumin solutions. Soft Matter 7:5150. doi: 10.1039/c0sm01312a

Figure below from:
Ewoldt RH, Johnston MT, Caretta LM (2015) Experimental challenges of shear rheology: how to avoid bad data. In: Spagnolie SE (ed) Complex Fluids in Biological Systems. Springer New York, pp 207–241. doi: 10.1007/978-1-4939-2065-5_6

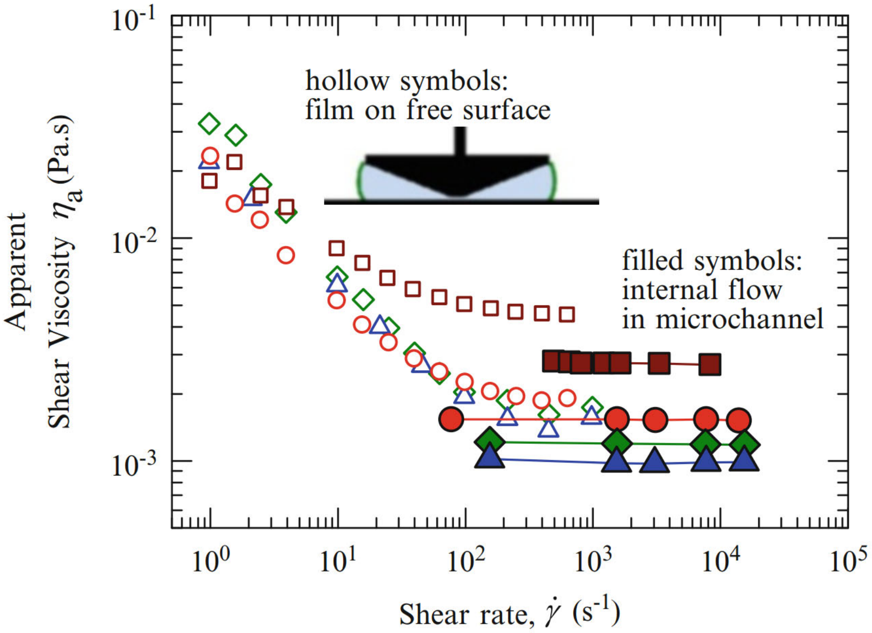
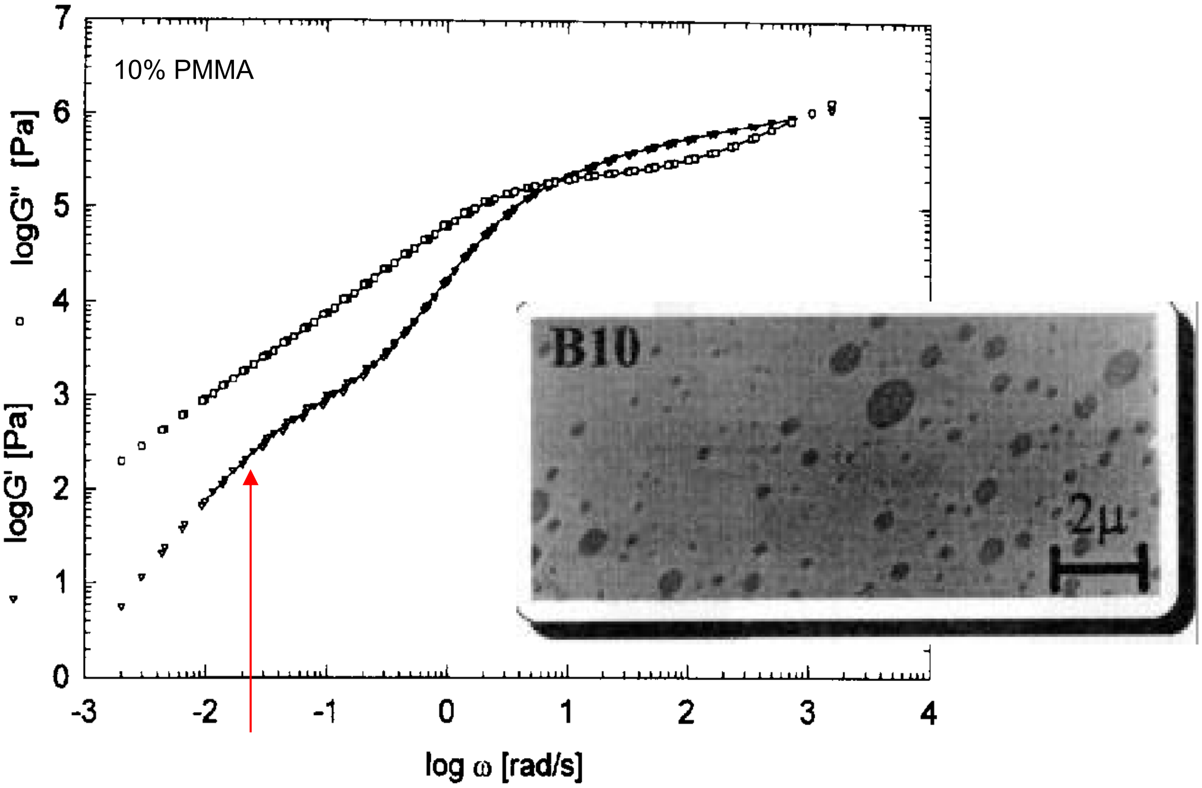
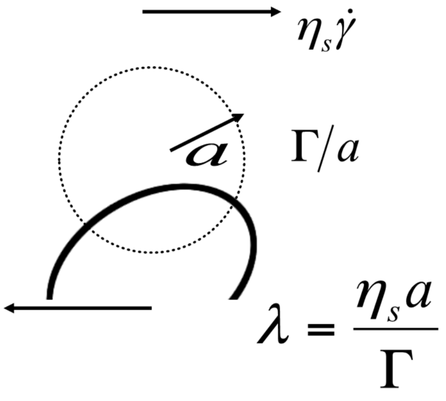


Fig. 6.12 Surface-active components in solution, such as the protein bovine serum albumin (BSA) shown here, may form a film at a free interface. Such films can show apparent yield stress and shear thinning of the apparent bulk viscosity when tested with cone-plate (*hollow symbols*). However, this is an interfacial rheological property and not a true bulk property as shown by the internal channel flow measurements (*filled symbols*). BSA at bulk concentrations of 10–200 mg/mL, *triangles-squares* (Data originally reported by Sharma et al. [51])

Answer 11. Polymer Blend

After chain entanglements relax all homogeneous polymers should show terminal behavior, i.e. G''~ ω and G' ~ ω^2. For a narrow molecular weight distribution the entanglement relaxation is typically very obvious as we can see at about 5 rad/s in this data. However, for this blend there is another relaxation at about 0.03 rad/s. This is due to relaxation of the droplets as illustrated in the image below. The time scale of this relaxation can be estimated by balancing surface tension stress which is interfacial tension divided by the radius of the drop, a, with viscous stress, viscosity times shear rate. The breadth of this transition can be used to estimate the drop size distribution just as the breadth of the entanglement transition can be used to estimate polymer molecular weight polydispersity.



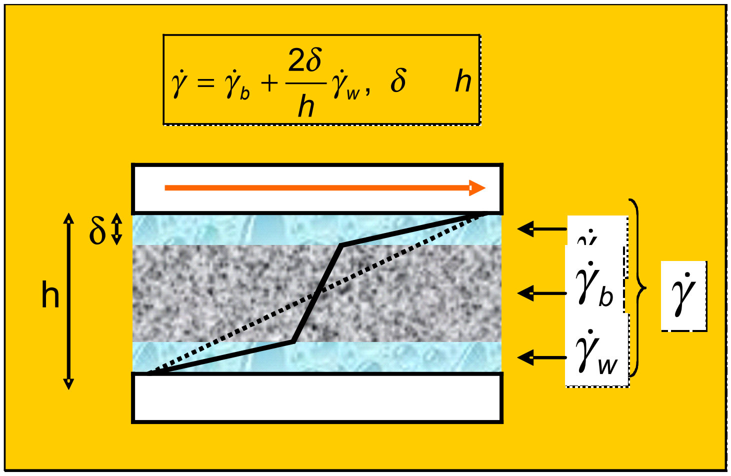


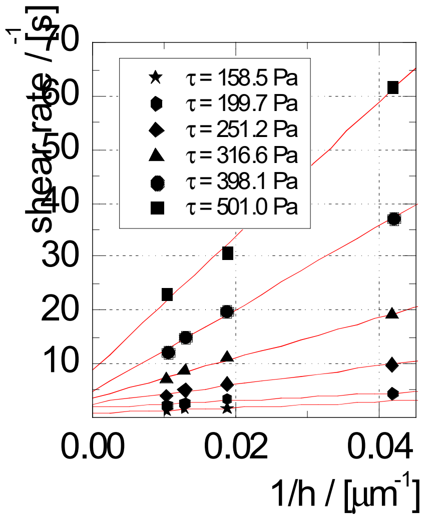
$$\lambda = \frac{\eta_s a}{\Gamma}$$

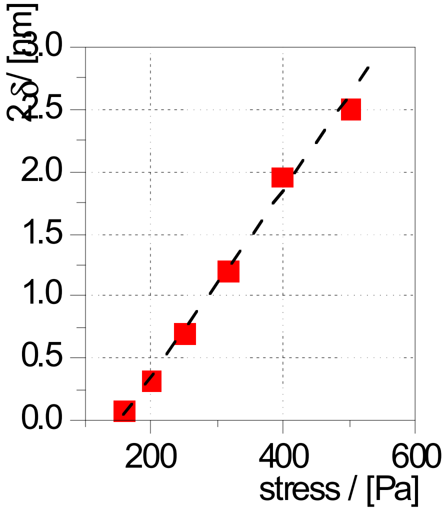
Answer 12. Mayonaise

Wall Slip is occurring during testing of the mayonnaise. It is seen that for equivalent stress, the *apparent* shear-rate is higher for smaller gaps. Recall that apparent shear-rate is the ratio of the local plate velocity to the gap height. In the extreme limit that the mayonnaise behaves as a solid, a finite plate velocity may originate entirely from slip, and be constant as the gap is decreased. In this extreme case the *apparent* shear rate (velocity over height) will increase, but not because of the material property of the mayonnaise.

To be more precise, one may hypothesize that the mayo does have a viscosity, and that a viscous slip layer with thickness □ exists at the walls, where □ is constant for the same stress. Then a lower gap *h* would also produce different (higher) apparent shear rates. Data at multiple gaps could be used to fit a model for slip layer thickness □, as shown in the following figures.



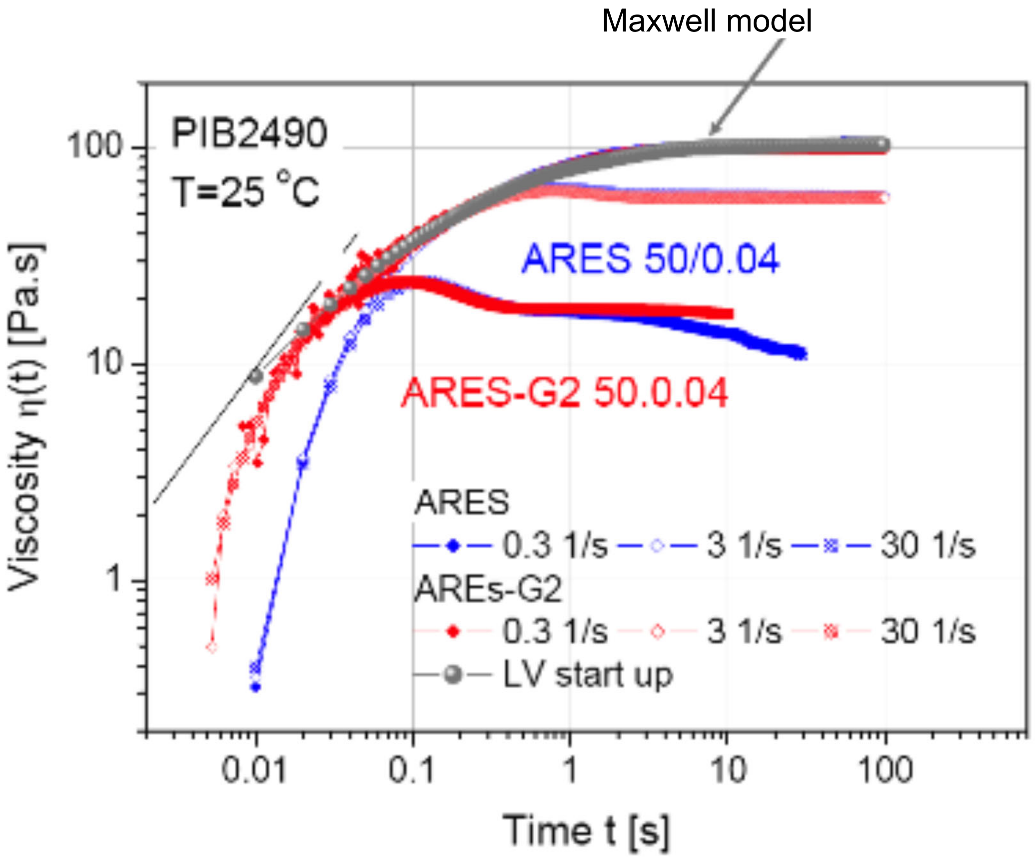




Answer 13. Start-up of Shear

a) The ARES-G2 has faster time response so it is more accurate at short time. It can reach the commanded strain in about 10 ms.

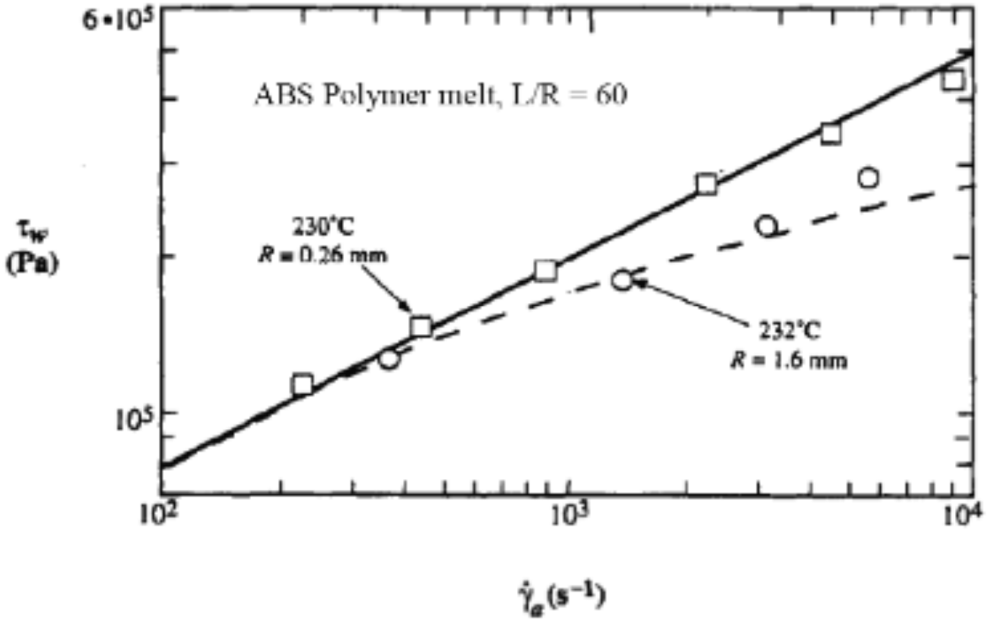
b) There is only one line for the Maxwell model because it has no shear rate dependence. It gives the time dependence of viscosity in the limit of zero shear rate. The Maxwell model is higher for viscosity < 10 ms because it uses G(t) to calculate η(t). G(t) is calculated from G' and G" from data up to 500 rad/s or 2 ms.



Answer 14. Capillary Rheometry of ABS Polymer Melt

Shear heating is a bigger problem with the larger diameter die – there is more volume which generates heat and a longer conduction path (larger radius R) for heat to leave the sample.

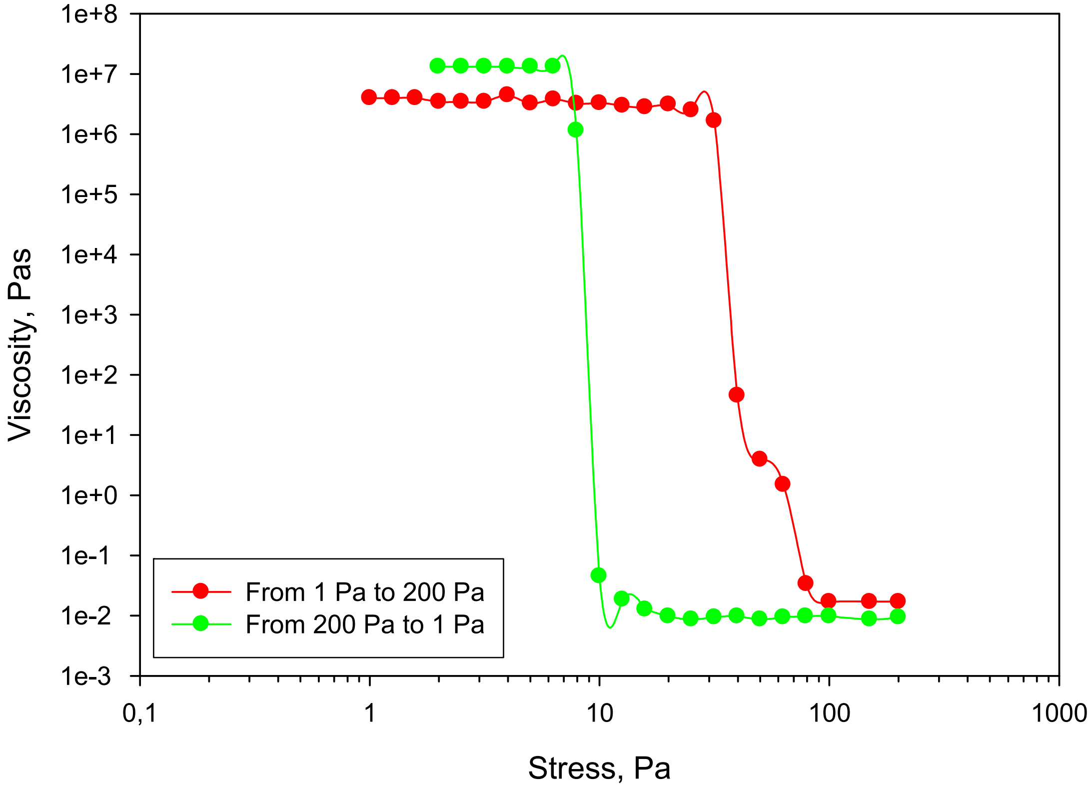
Slip is not a problem with this polymer melt until melt fracture which must be > 500 kPa. Note that in Question 1, it was observed that viscosity decreased as radius R *decreased* (we associated this with slip), while here in Question 14 the viscosity decreased as R *increased* (which we associated with shear heating).



Answer 15. Yield Stress of a Flocculated Suspension

Thixotropy, or time-dependent microstructural breakdown, can explain these two apparent yield stresses. For the ramp up in stress, the microstructure is *slowly breaking down* when approached from a low stress. In contrast for the ramp down in stress, the microstructure is quickly broken down at high stress and then *slowly reforms* as the stress decreases (therefore the apparent yield stress is lower for this curve). Running the ramp down more slowly might reduce the difference between the curves. Note that the viscosity of the suspending media is low and the change in viscosity on yielding is enormous.

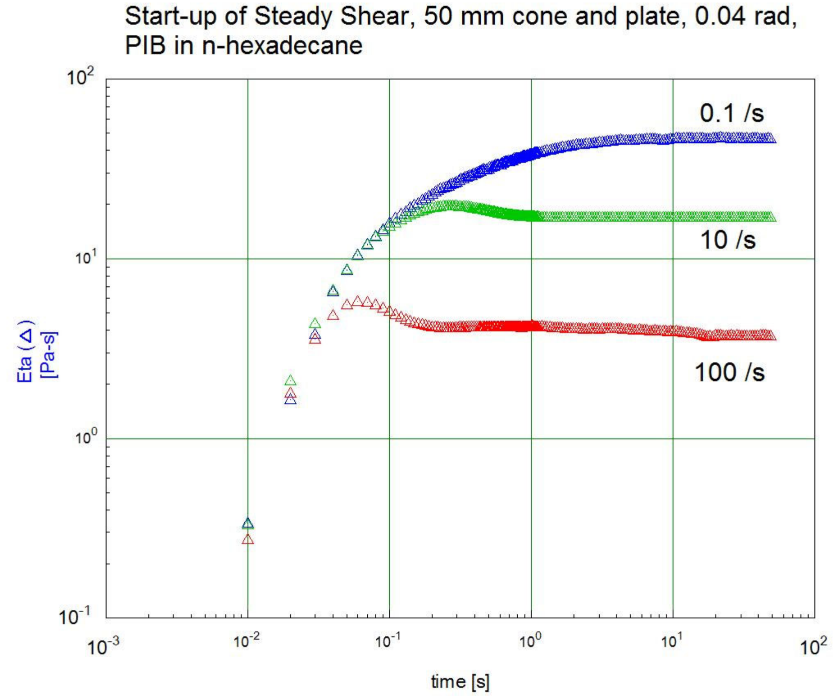
One should be careful about reaching “steady state flow” when testing for a flow curve – it may be possible to get a single flow curve if sufficient time is allowed to collect each data point. As a practical matter, one may want to consider the timescales involved in the actual problem of interest, and consider if the fluid is naturally used with stress ramping up or ramping down.



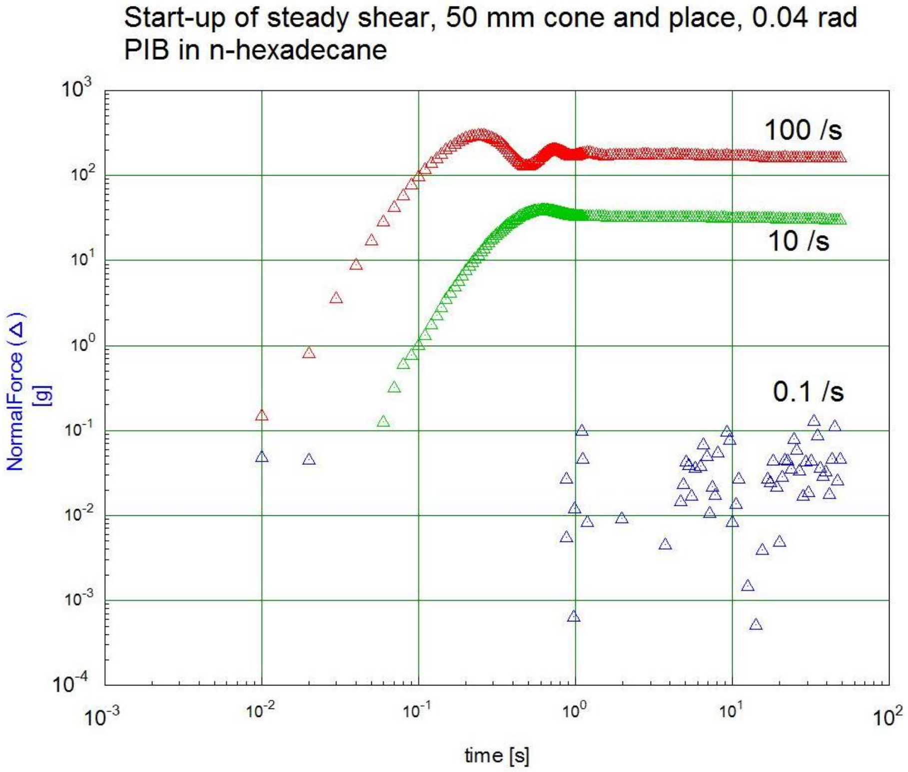
Answer 16. Start-up of Normal Stress

For the wiggles think about the response time for the normal force rebalance transducer.

For the scatter think about the magnitude of the normal force being measured.



Start-up of Steady Shear, 50 mm cone and plate, 0.04 rad, PIB in n-hexadecane

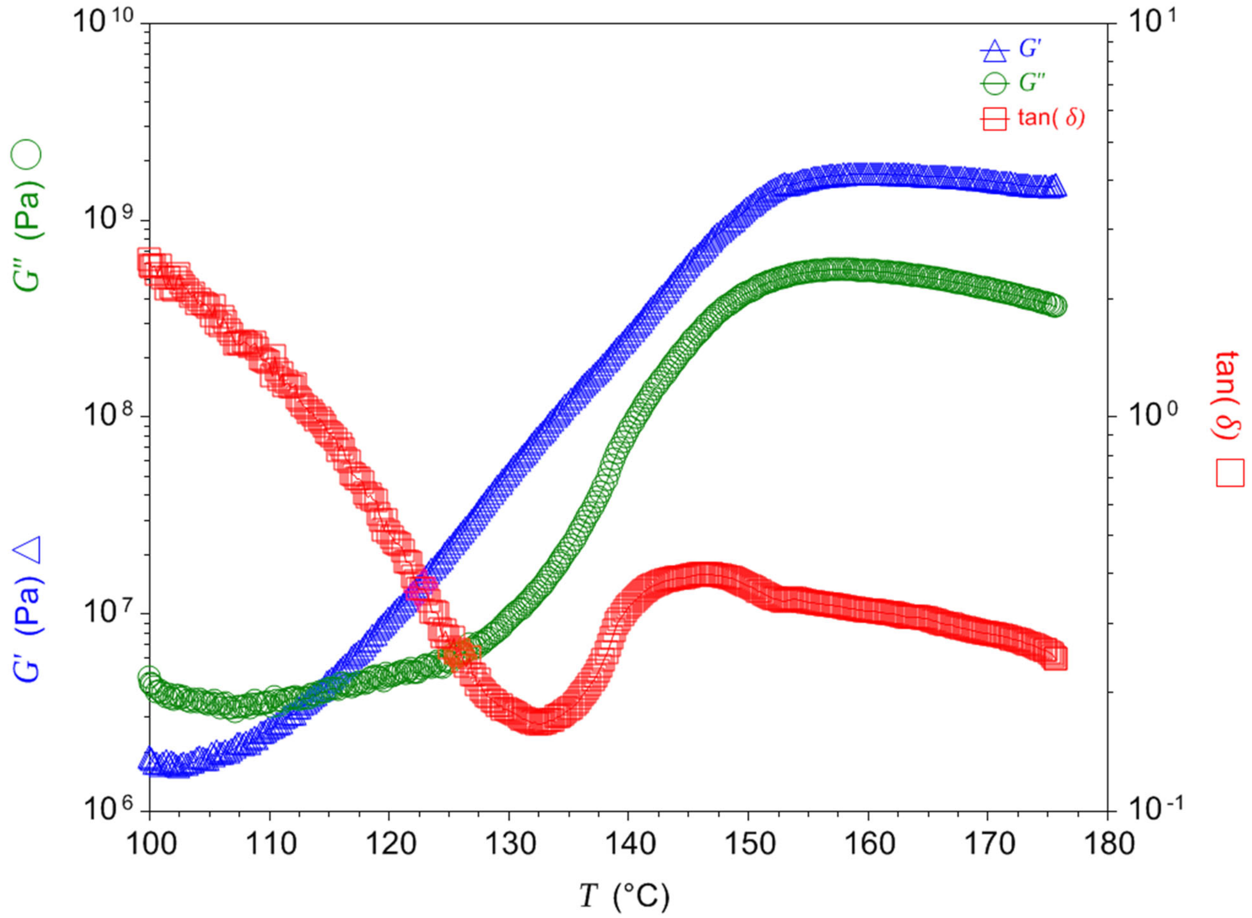


Start-up of steady shear, 50 mm cone and place, 0.04 rad PIB in n-hexadecane

Answer 17. Gel Point of a Thermoset

This sample is not a pure resin; it has a large amount of solid particles. The measurement represents the elastic contributions of the filler, so it shows a higher G' than the resin alone.

Modulus crossover point is not a true indicator of a gel point. A better approach is to find the point at which Tan Delta is independent of frequency. Testing could be done by scanning multiple frequencies, using multi-wave strain input, or making separate measurements at different frequencies.

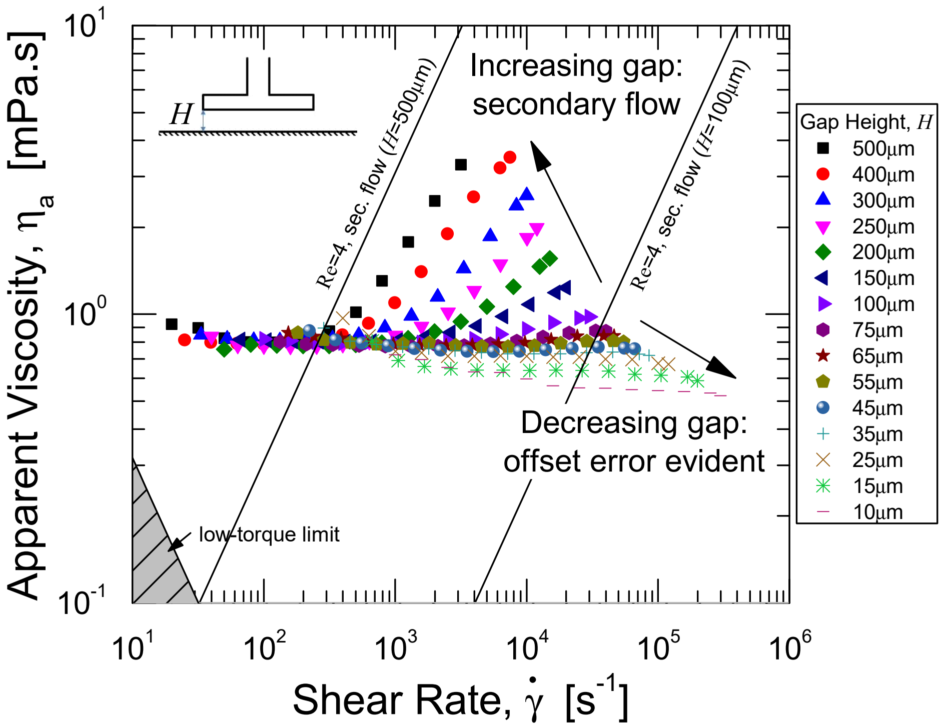


Answer 18 High Shear Viscosity of Water

High shear rate measurements with variable gaps using parallel plates

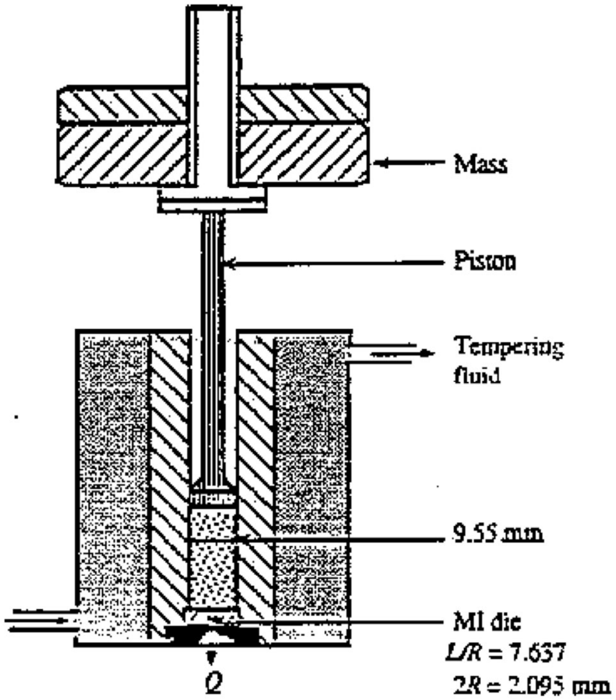
a) For larger gaps at high rates, the inertia of the liquid may cause **secondary flows**. It is common to assume that the liquids will travel in circular stream lines, but centrifugal effects will tend to push fluid outward near a rotating boundary. This secondary flow causes increased dissipation, and a larger measured torque, and hence a larger apparent viscosity. The effect increases as a function of Reynolds number, defined as $\mathrm{Re} = \rho \Omega H^2 / \eta$, so the effect is evident for higher velocity Ω, larger gaps H, and low viscosity fluids. Lines for $\mathrm{Re}=4$ are shown in the figure below for two representative gap heights.

b) For small gaps, the main error is caused by **gap offset error**, for which the actual average gap is larger than the calibrated value. The zero gap position is calibrated based on contact force at the first point of contact. Two issues arise. (i) A finite force is often observed before solid-solid contact due to viscous resistance of air flow in the squeezing gap. (ii) the parallelism is not perfect, and the average gap will often be larger than the "first point of contact" gap. Both of these effects contribute to gap offset error, so that the actual gap is larger than what you think. Viscous resistance to flow is lower with a larger gap, hence the lower apparent viscosity as seen in the figures, but only for small gaps where the offset would be non-trivial.



Answer 19. Melt Index

The melt Index is based on a single die and it is short, L/D=3.8. This means that a significant part of the pressure may be used in the extensional flow which occurs when the polymer goes from the 9.55 mm barrel to the 2 mm die. Some pressure may also be used to overcome friction between the piston and the barrel walls. Thus the measured flow rate is a combination of extensional and shear viscosity and the melt index is not a true rheological measurement. Also it only measures flow at one stress level. Typically a rheometer measures a material function like shear viscosity over a range of stresses or shear rates.



Answer 20. Interfacial Viscosity

Crude oils are notorious for containing surface active constituents, such as asphaltenes. These will cause the interfacial viscosity to be much larger than one would otherwise expect. It is not simple to verify this with just interfacial viscosity measurements or to correct the data. The presence of such constituents can be revealed by solvent extraction methods.

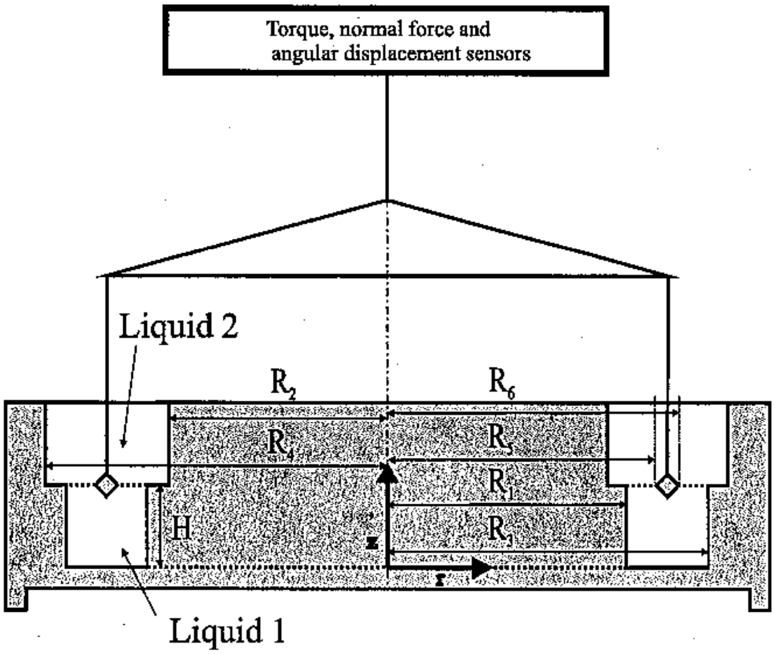
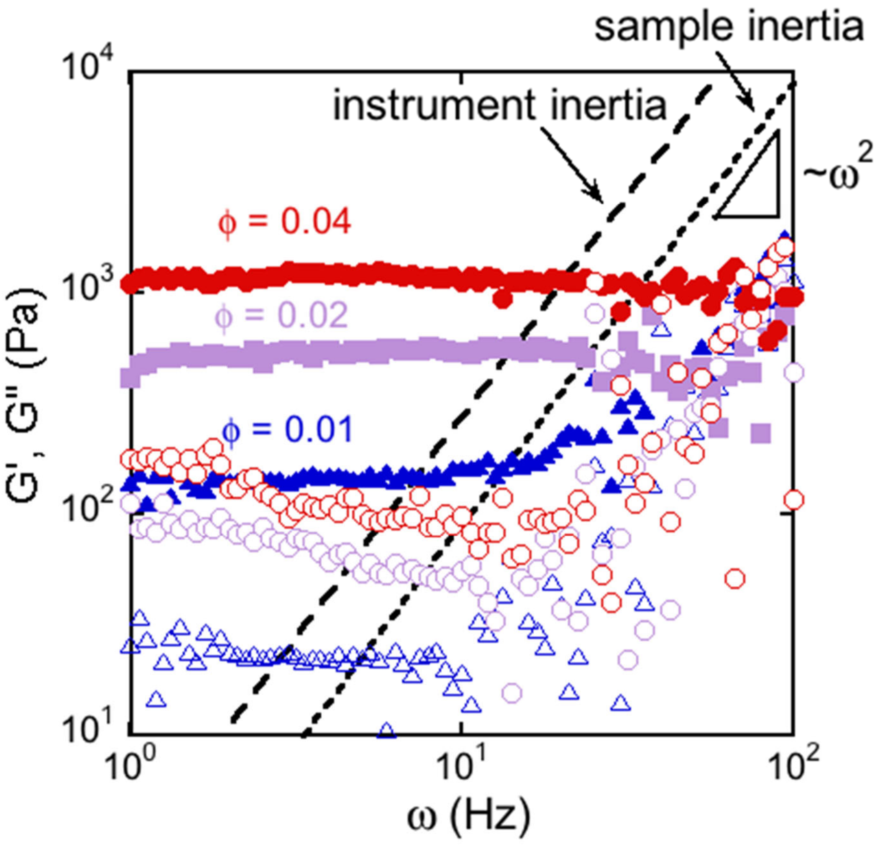


Fig. 1 Cross section of the DWR setup

Answer 21. Bijels: Soiid-Stabilzed Emulsions

The upturn is due to instrument inertia, and has nothing to do with the material itself. This can be confirmed by plotting the material's inertia to get a sense of the window for reliable data (for more, see "*Experimental challenges of shear rheology: how to avoid bad data*" by R.H Ewoldt *et al*, in Complex Fluids in Biological Systems, Chapter 6, Springer Science and Business Media New York). The upturn roughly coincides with the boundary for reliable data, and also has the expected ω^2 functionality for data contaminated with instrument inertia. The reason it does not happen for the sample at $\phi = 0.04$ is that G' for this sample is just large enough to overcome this issue for the entire range of frequencies tested (see graph below).



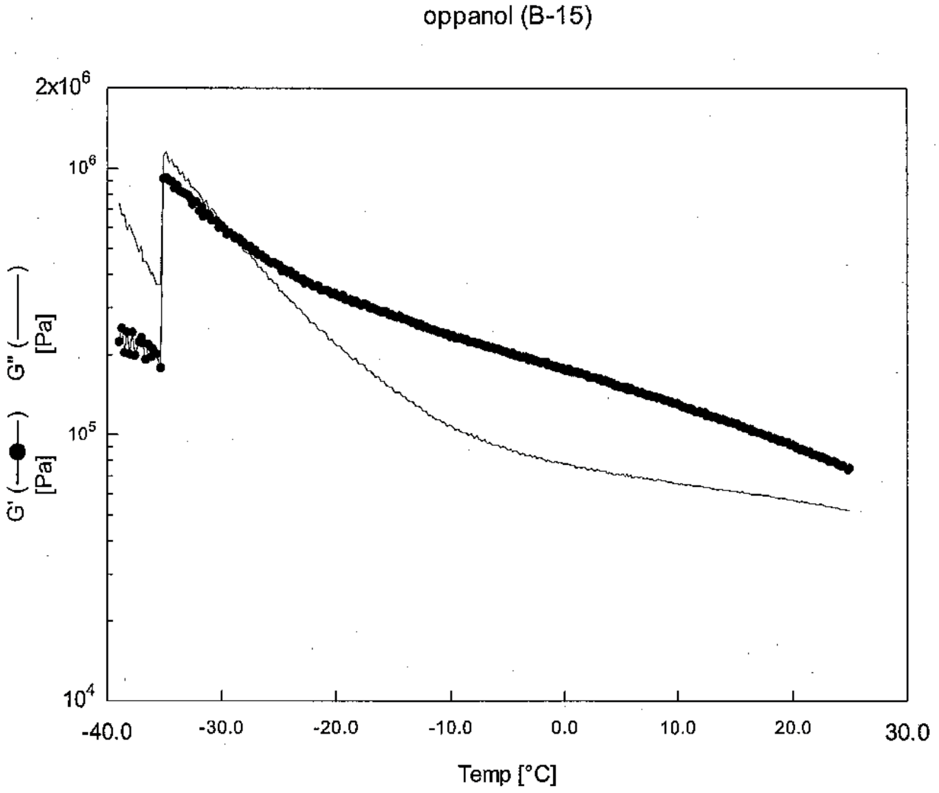
Answer 22. SAOS during Cooling

The ARES measures torque with a Force Rebalance Transducer (FRT). FRT's have a wide range of operation from minimum torque to maximum torque (four decades!), but if the coupling between the fixtures – the sample itself – is too stiff, they will go into resonance. The stiffness of the coupling needs to be reduced, either by reducing the diameter or increasing the gap. The diameter dependence goes as R^3 so that is the most effective adjustment to make.

To measure very soft samples, larger diameter geometries are required to give enough torque.

To measure very stiff samples, smaller diameter geometries are required!

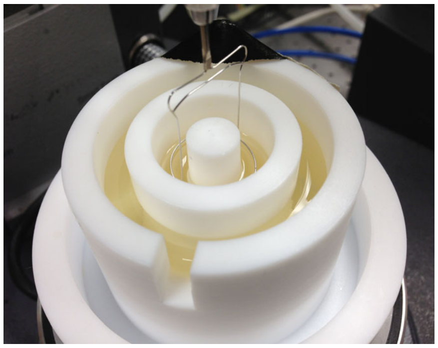
If the resonance was allowed to go on for a long time, the transducer could be damaged, so unmonitored or overnight tests should only be conducted if we know that the modulus will not become too large.



Answer 23. Biofilms

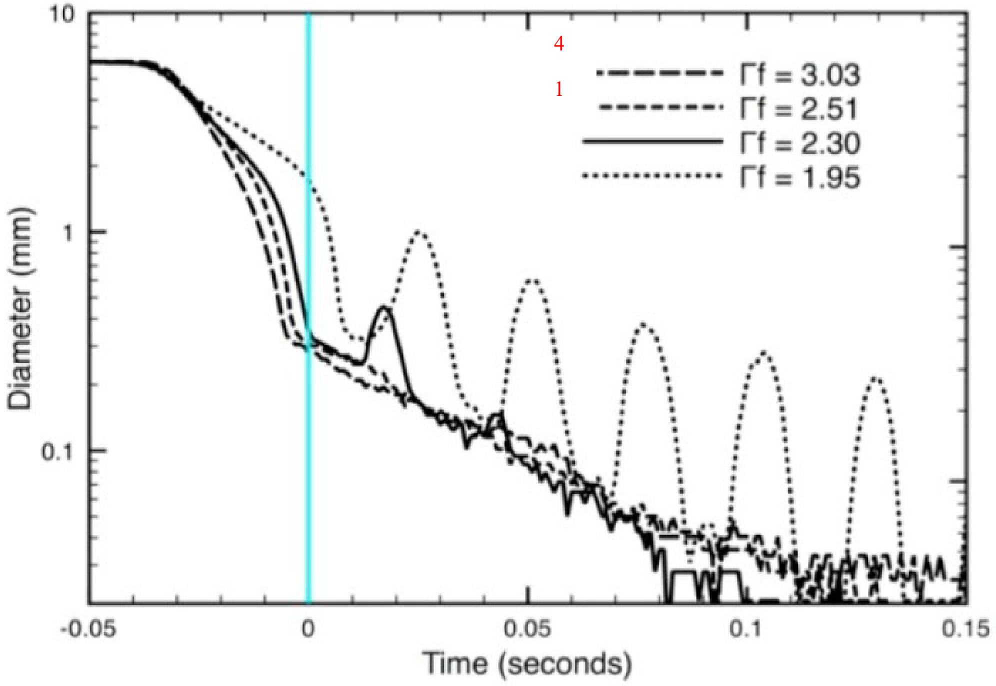
The maximum in the elastic modulus is a result of desiccation. This method requires that the ring be properly located at the colony/air interface. If sufficient desiccation occurs, which evidently has occurred after more than a day of measurement, the interface can be lowered to the point that the ring detaches and the torque signal will drop. This problem will not occur precipitously since capillary forces will “pin” the interface onto the ring. However, at some point, these forces will not be sufficient to retain contact and the signal will diminish.

There are several ways to avoid this problem. For example, one could continuously replenish the subphase by pumping in this liquid at a volumetric flow rate that is equivalent to the evaporation rate. This can solve the problem but very often this rate is difficult to ascertain. In the Fuller lab, this was solved by designing the double wall ring cell shown below.



In this modified cell, the bacteria colony is grown within the most inner gap, into which the ring is also placed. External to that inner gap is another concentric gap into which cell media is fed. Not shown is a small orifice connecting the inner and outer gaps so that the liquid/air interface in the two gaps is identical. The cell media fed into the outer gap overflows that gap and flows out through the notch shown in the outer wall. In this way, the interface is maintained indefinitely at the level of the notch.

Answer 24. Capillary Thinning



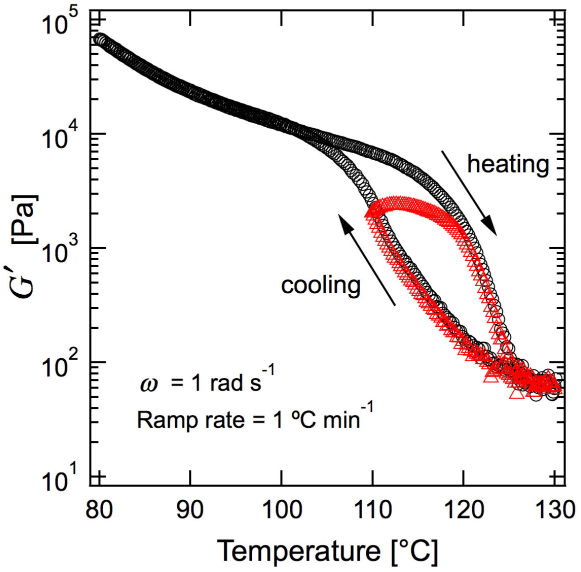
A) See ranking in plot above.

B) The “bumps” in the plot indicate bead formation. Remember how we talked about the importance of final aspect ratio in the CaBER experiment. If your final top plate height is too low (hence your aspect ratio is too low), then you will more than likely observe bead formation. If your final top plate height is just right for the sample in question, then we don’t observe bead formation.

C) Curves 1 and 2 are considered “bad/unreliable” because of bead formation. We cannot use this data to fit to a constitutive model to extract the extensional flow parameters that we want. Curves 3 and 4 are acceptable to fit to a constitutive model to extract parameters such as the extensional relaxation time and apparent extensional viscosity.

Answer 25. Block Copolymer

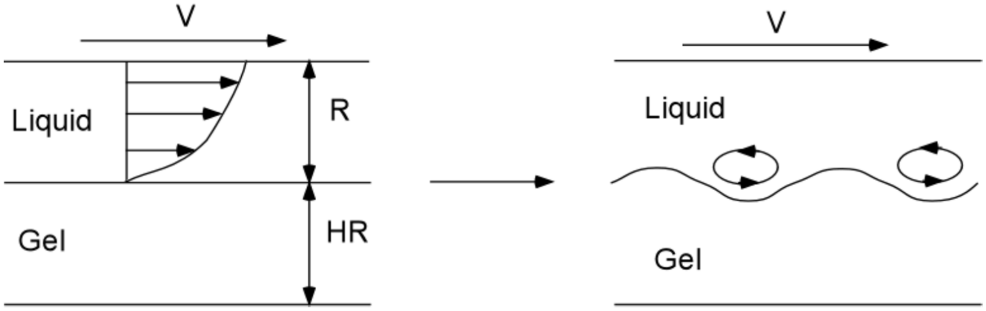
1. The user is looking for some sort of transition (ODT, OOT) of the block polymer morphology. Due to the values of the moduli, it's safe to assume the user is looking for the T_{ODT} (order-disorder transition temperature).
2. Hysteresis exists because it takes time for the triblocks to form long-range order from the disordered state (kinetic effect). The modulus subsequently increases as this long-range order forms.
3. Typically, if there are no transitions, the modulus would decrease monotonically with temperature. The slight increase in the modulus is because the triblock is still ordering. The increase in modulus from the formation of long-range order is greater than the decrease in modulus from the increase in temperature. Below is data shown for an isochronal temperature ramp undergoing exactly the same conditions, except the sample was cooled to 80 °C. From this, it is apparent that the triblock had not achieved its equilibrium morphology at 110 °C of the original isochronal temperature ramp test. One could also do time sweeps at a given temperature and wait until the modulus reaches a steady-state value to ensure equilibrium has been achieved.



4. Yes, the frequency was appropriate. At these temperatures, the frequency of 1 rad s^{-1} is low enough to access the time scales in which terminal scaling can be observed. This is apparent from the terminal scaling that is readily apparent at 130 °C at ω > 1 rad s^{-1}.

Answer 26. Soft Plate

The polymer gel is deforming under the action of the applied shear stress. If the applied shear stress is small enough, the gel behaves like a rigid plate and the apparent viscosity is the same as that which would be measured if the gel was absent and the liquid was contained between two rigid plates. When the applied shear stress becomes sufficiently large, the gel starts to deform significantly and no longer behaves like a rigid plate. Waves begin to form at the fluid-gel interface and the flow is no longer parallel:



A simple estimate for when this happens can be obtained by balancing viscous and elastic stresses: □V/R ~ E/H, where □ is the true fluid viscosity, V is the linear velocity of the top plate, R is the fluid thickness, and HR is the gel thickness. The critical shear rate at which the apparent viscosity increases is then V/R ~ E/(□H). The critical shear rate is thus smaller for softer gels (low E), thicker gels (high H), and higher fluid viscosities (higher □). For additional discussion, see M. D. Eggert and S. Kumar, “Observations of Instability, Hysteresis, and Oscillation in Low-Reynolds number Flow past Polymer Gels,” J. Colloid Interface Sci. 278, 234-242 (2004).

Answer 27. Surface Rheology of Asphaltenes

1. Can the Young-Laplace equation be used to extract measurements of the surface tension?

No, it cannot. The Y-L equation was developed for a “simple”interface where there surface stress is equal to the surface energy. The asphaltenes render the interface viscoelastic and this simple relationship is no longer true.

2. Wrinkles appear on the droplet surface. What do they tell you?

The development of wrinkles are a direct indication of the elastic nature of the interface and tells us that the interface can support shear stresses, which is not true of a simple interface.

3. Does the contraction cause a pure dilatation of the droplet surface?

The contraction of this large droplet will produce a mixed deformation of the surface - a mixture of pure dilation, pure shear, and pure extension.

4. These images were taken 45s after the droplet was introduced into the water. What would occur if the experiment was performed immediately after introduction of the droplet?

Very early on in the absorption process, the interfacial mechanics will have the surface energy dominate over viscoelasticity and the Y-L equation should apply.

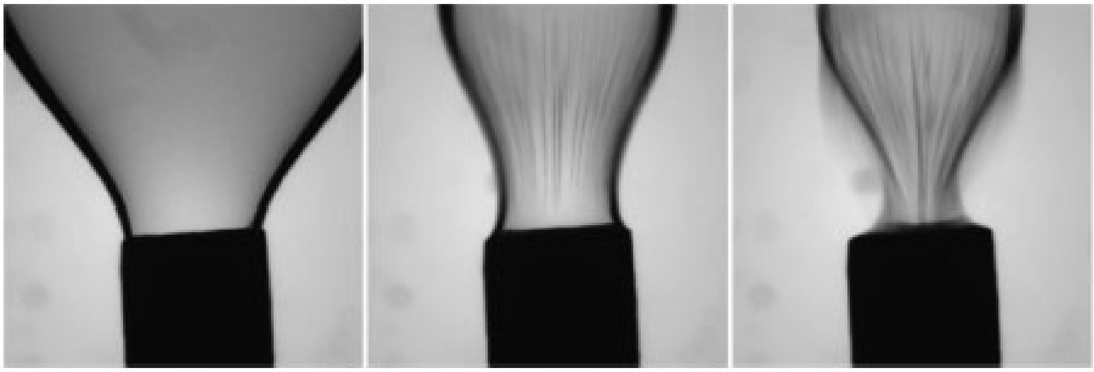


Figure 4. Droplet contracted by a factor 10 in volume (45 s adsorption, 100 ppm in 85−15% heptane−toluene).

Pauchard, Vincent, Jayant P. Rane, and Sanjoy Banerjee. "Asphaltene-laden interfaces form soft glassy layers in contraction experiments: A mechanism for coalescence blocking." *Langmuir* 30.43 (2014): 12795-12803.

Answer 28. Oilfield Drilling

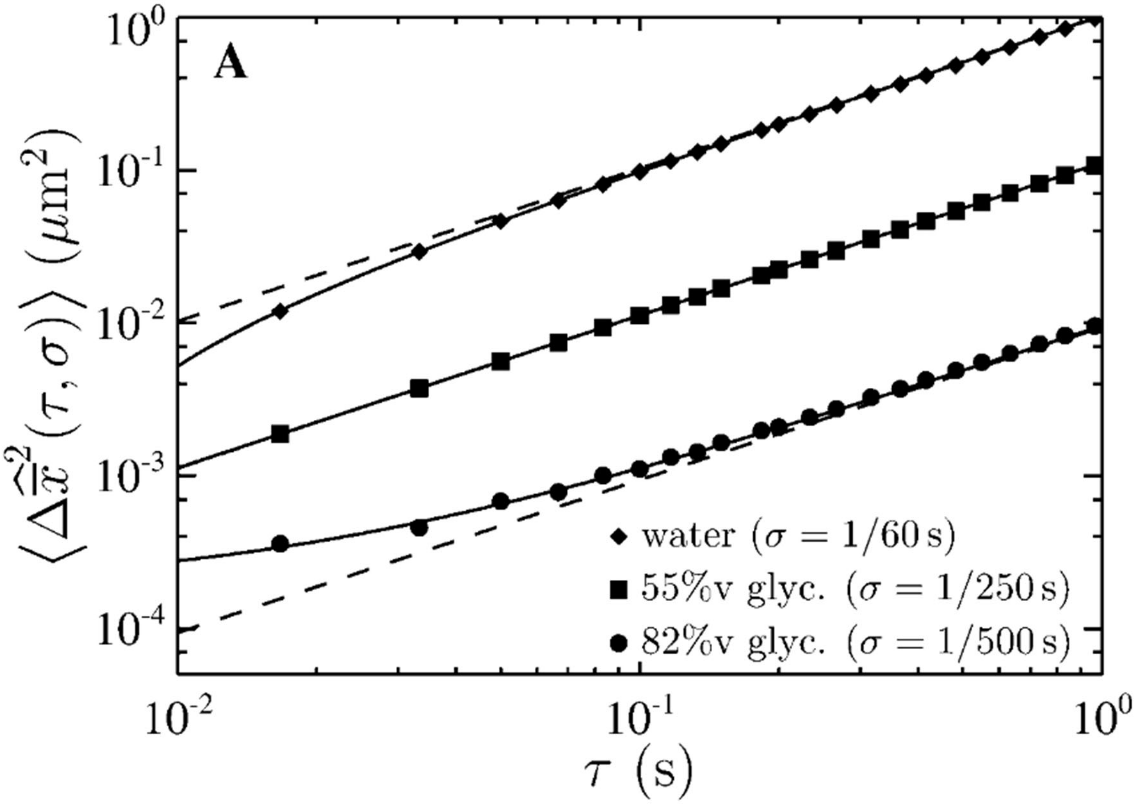
In oilfield drilling and cementing an oil-based drilling mud is used to drill the well, then a surfactant-based spacer fluid is injected to dissolve/remove the mud. Finally the aqueous cement slurry is injected to seal the annual space between the steel casing and the rock. The data above are on mixtures of the mud (M), spacer fluid (S) and cement (C). The pumping requirements for the fluids are determined from computer simulations of the flow processes. What rheological model or models would you use to represent the data? Consider what information is required, what the data will be used for, how parameters should be evaluated from this laboratory data, and how it would determined on field samples using less sophisticated rheological instruments (Fann 35). From this data what might you say about chemical interactions that might occur during the sequential pumping?

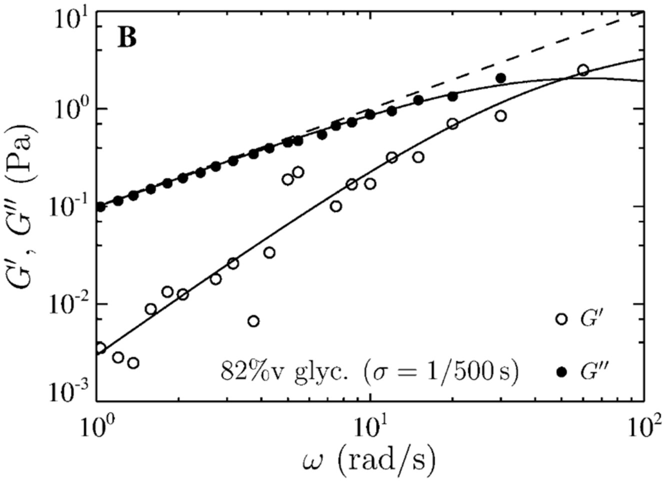
RK Prud'homme comments on Oilfield Drilling Example.

1) The drilling mud is essentially Newtonian.
2) The spacer fluid is shear thinning with a yield stress.
3) The cement is essentially Newtonian with a yield stress (ie a Bingham fluid)
4) The information required for pumping is a stress at a relatively high shear rate. The key question the students should ask is what is the shear rate of the pumping process? It turns out to be 200-500 s^{-1}.
5) The static yield stress, or lack thereof, is not important in this problem. The whole process of displacing mud and placing cement occurs in less than an hour. Settling is not a significant problem because the vertical distances are hundreds of meters.
6) While a Hershel-Bulkley model can be fit to the data with high precision from this high quality data, in the field the operators will have much cruder measurement tools. What should be prescribed is a 2-3 point viscosity measurement at 200, 375, and 500 s^{-1} and then fit the data to a Bingham model. This will provide accurate results to predict pumping power requirements and flow rates. More elaborate fitting is not warranted.
7) The students might note that the lower viscosity mud is displaced by the higher viscosity spacer and then finally by the highest viscosity cement. This ordering to to minimize viscous fingering and to improve displacement.

Answer 29. Microrheology of Glycerol

The high viscosity of the glycerol solution means that particles are moving small distances at short times. A significant number of displacements fall below the detection limit of the particle tracking (roughly 10nm in this experiment.) The resulting curvature of the *apparent* mean-squared displacement makes the fluid look as if it has a high-frequency elastic response.





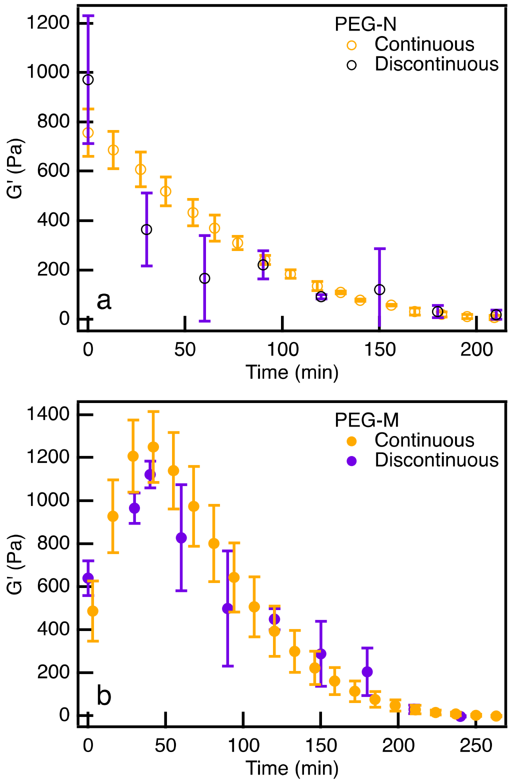
Submitted by: Eric Furst (Jul 2021)

Answer 30. Degrading a hydrogel for molecular release

There are 2 hypotheses made that can cause this:

1. shear imparted during the experiment can cause structural rearrangement (such as polymeric alignment) and increase the modulus of the material.

2. the cross-linking reaction is causing this change.



To test the first hypothesis and determine whether shear is the factor that causes the increase in modulus after initiation of degradation, we measure scaffolds during degradation when we impart continuous and minimal shear. For continuous shear, we use the time sweeps described above. For minimal shear, we make a series of gels and incubate them in enzymes for a specified time and then measure each gel with a single frequency sweep. We measure no difference in the modulus during degradation when the amount of shear added to the gel is changed.

The second hypothesis is that the cross-linking reaction is the reason the modulus increases. This is tested by measuring 2 gels with different cross-linking reactions and different methods of initiating the reaction. The PEG-N gel can only cross-link further when expose to additional light, so no additional cross-linking can form when the scaffold is incubated in enzymes. This is why we measure that the modulus only decreases indicating that the scaffold only degrades. For the PEG-M gel, the functional groups can continue to react when they are in a basic solution, which includes the enzyme solution used for degradation (pH 7.4). Therefore, <u>unreacted functional groups can react as the material swells and starts to degrade, resulting in an increase in the modulus.</u>

Submitted by: Kelly Schultz (Aug 2021)

Answer 31: LAOS for 3D printing on the moon

The qualitative features look like a yield-stress fluid: there is an elastic modulus at low amplitudes, which decreases dramatically as a function of stress.

However, some things are peculiar.

1. At low amplitudes, there is not a constant plateau (which would be expected from a linear regime). Instead, the elastic moduli increase with amplitude.

There may be something changing in time with the sample, e.g. drying or sedimentation, which changes the sample in time. This could be assessed with a time sweep. The rheologist further reports that samples formed an outer crust before completion of tests. This could be assessed by taking and reporting photos of the test.

2. The apparent yield stress seems to depend on the gap in a peculiar and non-monotonic fashion. The largest gap (3mm) has the lowest yield stress, and the smallest gap (1.84 mm) has a much higher yield stress. No yield stress matches the expected value.

For the large gap and lower yield stress, this is likely poor sample preparation: the granular particles may have settled, or the material did not completely fill gap and failed to maintain contact with top plate.

For the higher yield stress at the smaller gaps:
a. Samples may be over compacted when lowering top plate, thus increasing the yield stress
b. Fixed gaps do not allow room for the dilatancy effect (sample volume increase) caused by particles re-arranging, resulting in increased normal stress and confinement effects which may increase the yield stress.

Submitted by: Gerry Fuller (Aug 2021)
With contributions from: Randy Ewoldt