Rheology of Complex Materials
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Lecture – 53
Rheometer Demonstration

So, in the lecture so far, we have seen several rheological techniques, we have seen how different material functions are defined. We have also done survey of some classical data regarding rheological response of polymeric system as well as multiphase systems. in the next couple of lectures we would like to look at the experiments themselves, the idea is to get familiar with the instruments, and the procedures that are used to character characterize the rheological response of materials.

Ah for doing this with me is Ramya and Swaminath, both PhD students in our group. generally there are several instruments with various different types of capabilities, and generally each instrument will have certain strengths and features which are very good for certain applications. since we have 2 instruments in our lab, this 2 classes will be based on these 2 instruments. as I mentioned there are several possibilities and several other instruments which can also be used for such purpose. So, the way we will follow these 2 lectures is initially we will look at the overall instrument, what are the different components that are available in the instrument.

And then we will go on to look at some specific examples of steady shear oscillatory shears stress growth, normal stresses, and we will also try to look at how to attach something additional to a rheometer and then get additional information in addition to rheometer rheological data itself. So, we will begin by first looking at the overall instruments, and Ramya will explain some of the main components that are involved in a rheometer instruments.

Student: Thank you sir. So, like professor Deshpande had mentioned any a rheometer any make would basically have a body which looks like this.

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The rheometer has the head, in which you have the main components that help in the functioning of the rheometer. So, the rheometer has an encoder a motor and a air bearing. The purpose of an encoder is to monitor the extent of deformation that is being that is a rheometer that is what the rheometer has to do. what the motor does is, it basically converts the mechanical energy to the electrical energy so that the equipment can understand.

What the air bearing does as opposed to other other general-purpose bearing is it gives it basically takes the pressurized air from a compressor, and gives the friction free motion to the rotating parts of the motor. So, these 3 components together help in making the equipment work.

And if you recall we discussed rotational rheometer and this instrument is an example of rotational rheometer. And we have mentioned that motor and bearing are the 2 most important components as Ramya mentions.

Student: Then if you see carefully this part of the equipment is a couple fitting that is provided it to detect a geometry or measuring system.

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So, for example, let us consider a measuring system that we generally talk about in class. So, this is the cone and plate system with a very small cone angle.

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Cone this is the cone itself while the plate is here.

Student: So, this is the bottom plate, and when I detect this geometry. So, what you can essentially see is a cone and plate system, what you essentially see is the cone and plate system. Now there are certain other functions that are that are to be performed before you start an experiment. Though the motor knows, what the geometry what is a geometry that is detected it is essential for us to subtract the friction or the residual friction that is contributed by the geometry. Therefore, we need to do certain measurements called the inertia measurement and the motor motor adjustment. Now before going on to doing the test, I would also want to inform that the rheometer is communicating with the computer through an interphase.

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There is a software that comes with the equipment, the the software is based on is is based on comfort level of the person. It it is as user friendly as it can be. The software will communicate with the device, and it has all the functions, the it has it can do all the functions that have to ask the equipment to do.

For example, in class we discuss that we apply constant strain or we apply a oscillatory deformation at a certain frequency. To achieve this, what we will do in setting here is actually we enter the information on the computer and through the computer interphase the instrument will get controlled.

Student: So, before moving on to a doing any test, what we need to do is we look for the inertia and the motor adjustment. Therefore, the first thing is to to know if com the rheometer has communicated with the software. So, you need to switch on the rheometer.

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So, once we switch on the rheometer, we look we have to establish communication with the equipment communication between equipment and the software ok. So, once you open the software, the we need to initialize so as to establish the communication.

In one of the things you can see on the panel, is the fact that there is normal force and temperature position and the measuring system. So, these are the key things which one should be aware of while doing the experiments, measuring system since we have not yet put anything we do not have anything displayed there. Temperature of course, is whatever the system is the measuring. position and the normal force are very critical in for the instrument to make a decision about what to do.

For example, what we will do is we will put geometry here, and then we will ask the rheometer to actually go to the measuring position. So, for that these position and normal force are very closely related. The other components you can see here is this is an oven. So, because right now of course, it is room temperature in this at 26 degrees, but if we want to change the temperature then we can use this oven and depending again on the rheometer and the accessories which are available the such temperature controls can go all the way from minus 100 degree Celsius to about 500 degree Celsius. And in fact, there are specific rheometers which can even do measurements up to thousand degrees and so on.

Student: So, like professor had mentioned so, this is a convention temperature device and it is an oven. You can also have different Peltier systems. So, this is a Peltier systems that works with the help of temperature sensor.

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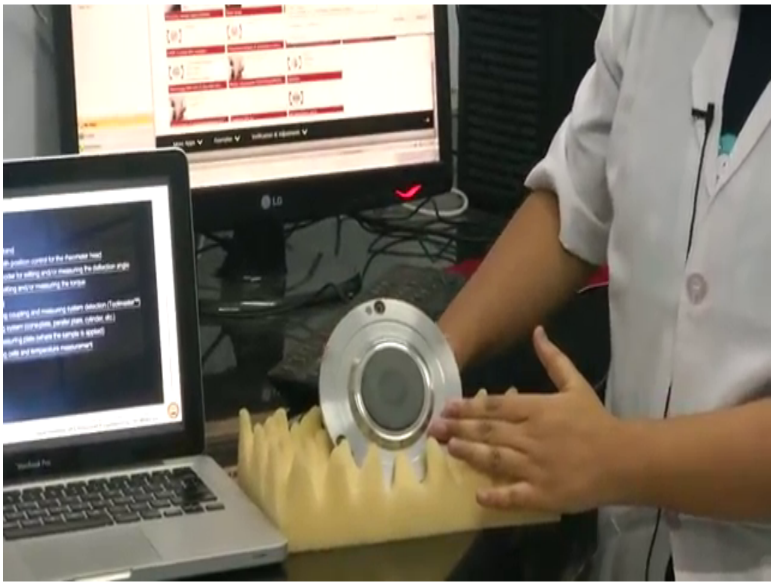

So, we can also have a system a bottom system wherein the temperature is controlled by a liquid controlled temperature device.

You can also see yeah, this is the other instrument that we have in our group as we have mentioned earlier, the overall body of instrument looks somewhat similar at the top we have the motor and the air bearing and the other controlled systems. the bottom part of the equipment in this case for example, is useful for the temperature control of the instrument. And again, we have a control panel in front which will display some key characteristics like temperature or position and so on.

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The back of the instrument is again with the communication cables and a communication card is connected with the computer so that we can effectively control the instrument using the computer as well as gather the data. I would acquire the data and do the data analysis. So, for instance we may do an oscillatory shear, in which case we will have to tell the instrument to apply a strain at a given frequency now of a given amplitude. And our acquisition will include taking readings regarding the talk which is required to make sure the deformation is reached at a given level. And then of course, we use data analysis methods to try to calculate G prime and G double prime from the acquired talk.

Student: So, once the communication is established between the device and the software, we need to choose the geometry that is required for testing conditions. Let us say we are going to do test with the cone and plate system. Now this is the cone and cone geometry or a cone measuring system with a very small cone angle, now this is how you detect the geometry. From the display or the interphase, you can see that the system is reading the measuring system that that has been put up.

So, based on the serial number of the geometry and the communication that I was talking about, equipment has detected the geometry. Now we will do these functions called inertia and motor adjustment. So, before going to that we need to do a set 0 gap. The geometry has to know how low or how high can it move in the window. So, we go for doing a set of measurements that are related to the geometry.

This is also part of the initialization of the instrument, given that we have seen that in parallel plate or cone and plate there is a gap which is adjusted between the top and the bottom geometry. And before we set a gap the instrument must know where is 0. So, that for example, in parallel plate one mm gap is needed, the instrument can then set the one mm gap. So, therefore, a initial setting up of the instrument is quite important for 2 reason. One is to make sure that inertia and all the other geometry calibration are done. Secondly, the instrument knows where the 0 is, 0 gap is.

So, right now you can see that instrument has in fact, both the top and the bottom geometries are touching each other so that 0 gap has being beside. And if you want to think about as to how the instrument might be trying to decide the 0 gap, you can think of what happens if the 2-surface come together in the directly there will be a force generated which in this case will be the normal force. So, therefore, by looking the normal force so, therefore, by looking at the normal force the instrument can decide, if the gap have been set to 0. If the gap becomes too less and normal force will suddenly be very high in the gap is not 0 at all the normal force will be 0. So, therefore, instrument does this and then make sure that the 0 gap is set.

Student: so, as we can see now that equipment is ready to use for whatever tests we want to do. So, we can several kinds of shears that that are available. So, one other friend swami will be showing this do specific set of tests and he will be talking hence forth.

Student: so, hi my name is Swaminath; so, am a PhD student here so, so now, we will seeing the oscillatory shear measurement of a thermo responsive polymer called (Refer Time: 12:52) or ordinary (Refer Time: 12:53) So, what happens here is that as you increase temperature we actually go from clear solution, such as what you see as this in this case, and as you go to high temperature, you see a turbid solutions as in this case.

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So, what you will observe is as you increase temperature, and we go from a clear solution to turbid solution, you will see increase in moduli and the viscosity. So, oscillatory shear is one way of characterizing this transition from what you say clear state to do a high viscosity state in this case ok.

So, in class we have discussed several times the oscillatory shear experiments, and we also looked at several example data. most often we either looked at G prime or G double prime as functions of frequency or as functions of strain amplitude. So, in this case since the interesting transition happens across temperature. When we change temperature from a low temperature to high temperature or high temperature to low temperature polymer solution goes from turbid to clear. So, therefore, since the transition is happening from one temperature to the other, we will keep the same frequency and same amplitude, but change the temperature. And the idea is to see whether rheological property can capture this transition which is either from turbid to clear or clear to turbid.

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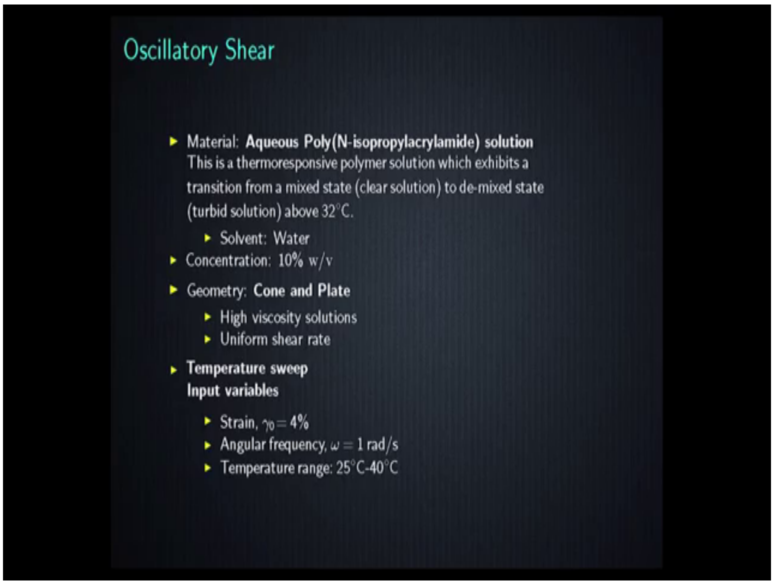

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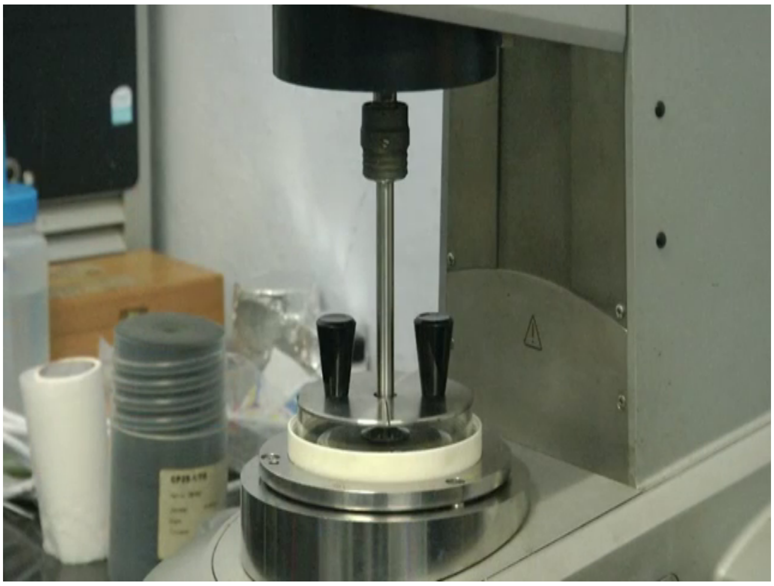

So, this test we will be using the cone and plate geometry which is just shown by Ramya, many a times the solvent evaporation can lead to changes in sample, and therefore, this apparatus on the Peltier which is covering geometry is a solvent trap. It basically traps the solvent and or avoids the escape of the solvent due to evaporation.

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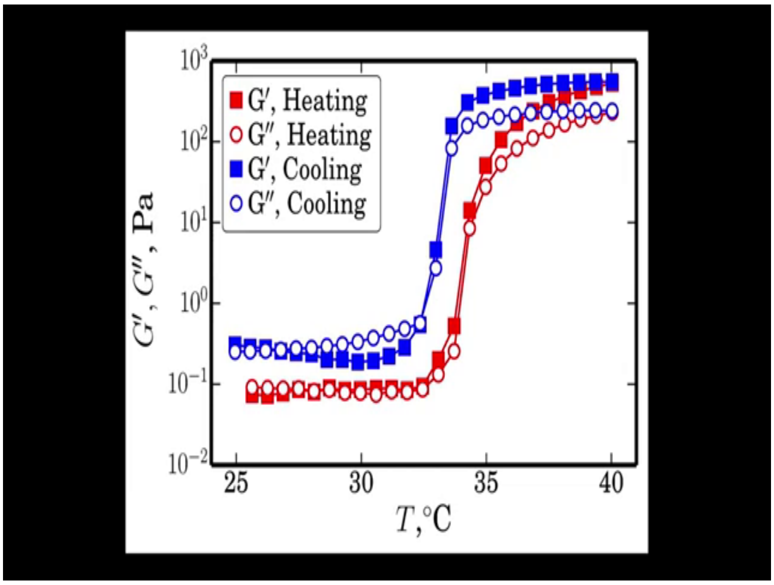

The transition temperature of this polymeric system is 32 degree Celsius. Below the temperature the solution is in the mixed state, polymer and solvents are mixed. due to which the G prime has as well as G double prime are less. As the temperature increase beyond 32 we observe a sudden increase in both moduli. I hope you read all that at high temperature this system actually becomes turbid. And therefore, it implies that it is the de mixing transition. In other words that the high temperature the solution undergoes micro phase separation leading to aggregation of the polymer and this aggregation in turn leads to high G prime and G double prime.

Student: so, here we have viscoelastic material wax. So, here we here we want to see when we apply a fixed shear rate how this stress grows in this material. So, to move out with this test, I will first show you how to put a different parameters in the rheometer interphase. as shown in the previous video, this how the interphase looks like. So now, we want to fix we want to keep fixed shear rate of 100 seconds and we want to see how the stress grows as function of time. So, for that if you see here we get we have the variable shear rate. So now, we can actually fix the shear rate in large number of waves, but for this test this test will fix it to constant value here and fix the value as 100 value of 100.

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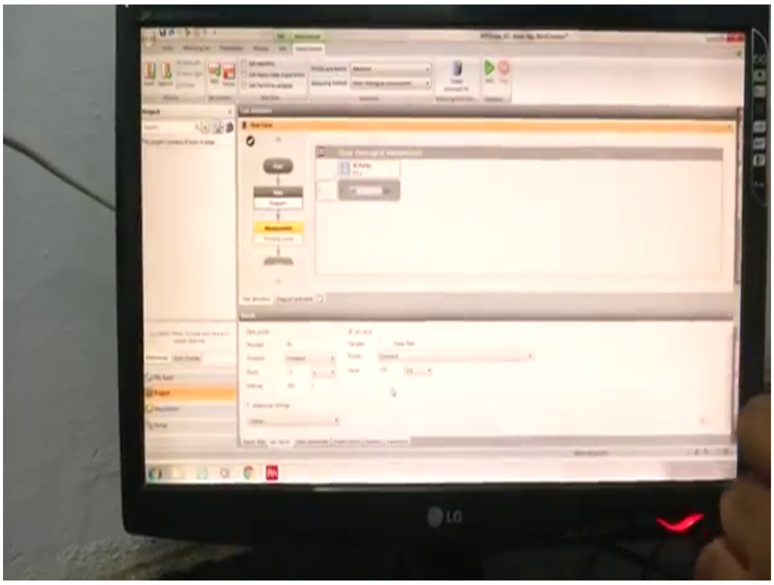

So now, here we want to we want the test happen over time period of 306 where there is 40 points and a meter of 7.5 seconds ok. So now, will load the sample so, let us code the rheometer for that, here is the wax sample which will be used in this thing. So, we will be apply the wax sample on the surface here, and here we have parallel plate geometry. So, ya so, we have to put the sample in the middle spot.

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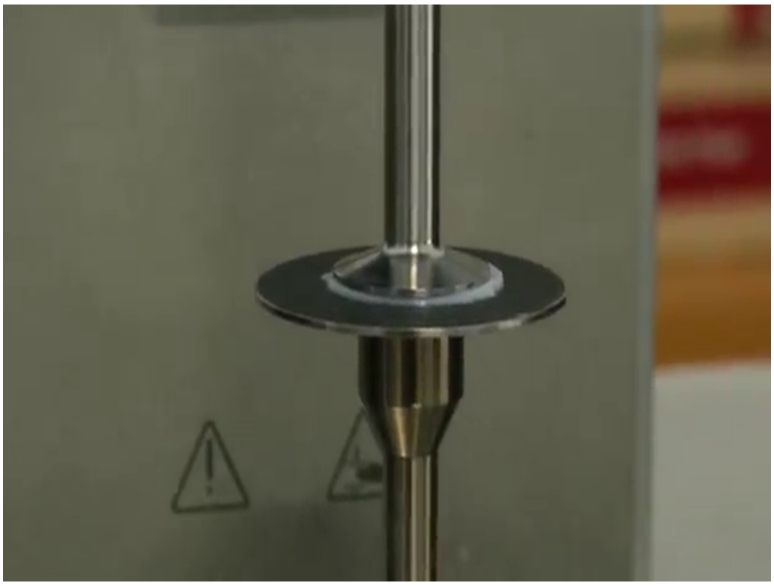

We have to make sure that the area under geometry's and fully covered by the sample, and there are no empty spaces under the geometry as well ok.

So now we will lower the geometry and trim the sample before starting the test. So now, we will go to the interphase again therefore, geometry and make the trimming of the sample, ya. So, here we see that this is the measuring position here in this case 1 mm. So now, we click this button and this is actually move the rheometer down and bring it to a trimming position. So now, will see the rheometer going down and the parallel trimming contact with the sample.

So now, we have trim the sample out so, this is as you can see around a geometry, we have some excess sample which is there which has to be trimmed out. So, which we will do it now, after trimming it out, we just remove the excess sample from the geometry, and we have to make sure. So now, we will move to the interphase and bring it to measuring position. Now it is at trim position we bring it to measuring position. As you can see in the interphase, it is says trim sample we have already trimmed a sample and now we can continue and allow the equipment to actually come to the measuring position.

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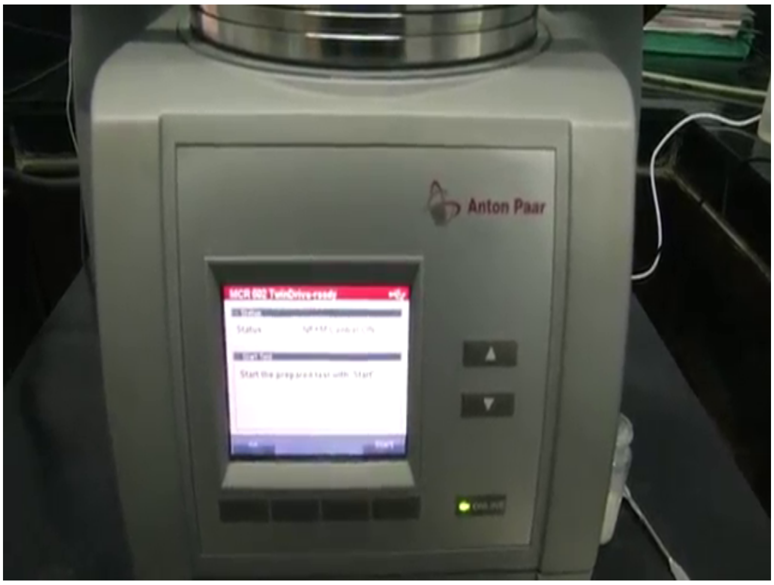

Now, it is at a measuring position and we can start the test now, and we see the variation of the viscosity and the stress shear ok.

In all the cases there is a pre-shear of point one per second to make sure that all the tests have the same shear history.

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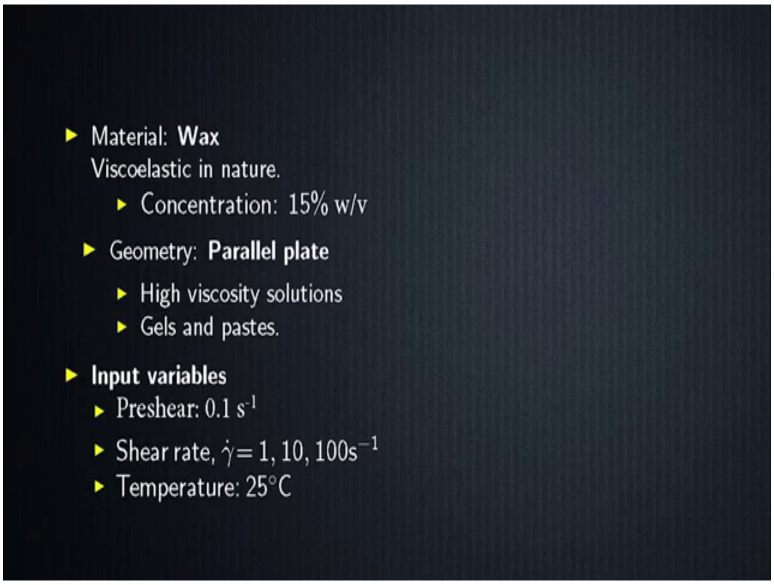

By the way a pre-shearing is the very important aspect of rheological characterization. The idea of pre-shearing is to remove subjective condition of the sample due to several deformations, which are encountered during the processes prior to rheological testing. For example, when we prepare the sample or when we store the sample or when we transfer the sample to the geometry, the sample undergoes deformation. So, therefore, by doing pre-shearing we ensure that all the samples are conditioned identically. Regardless of what was they are handling before.

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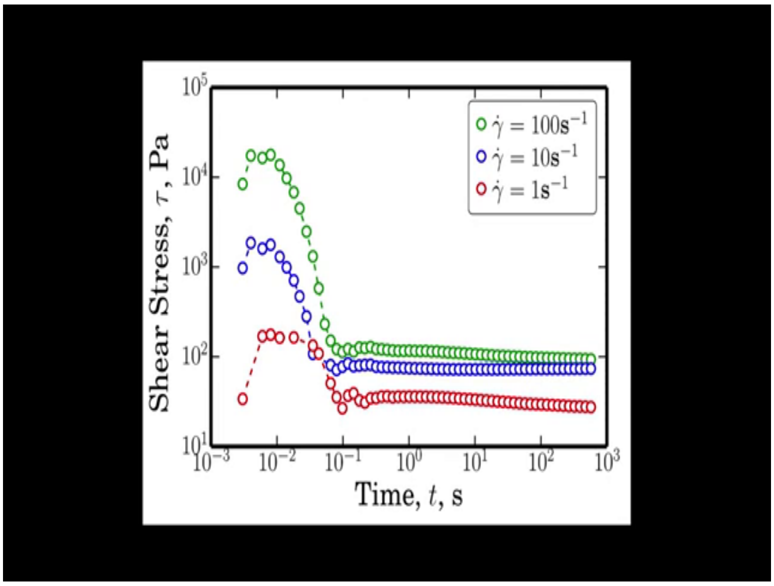

So, in the data that is generated here we can observe an initial rise in the shear stress followed by a decay to a steady state value. This response is signature of viscoelastic behavior and especially non-linear response. If you remember a linear model like Maxwell predicts only monotonic increase in stress. So, the stress overshoot at initial times, that we see here which is a non-linear viscoelastic feature this actually larger for higher shear rates. As we know that at higher shear rates the elastic contributions are longer. In addition, the final steady state value of the stress also is higher for higher sheer rate; however, the steady viscosity value would be lower with higher strain rate because of shear thinning nature of the sample.

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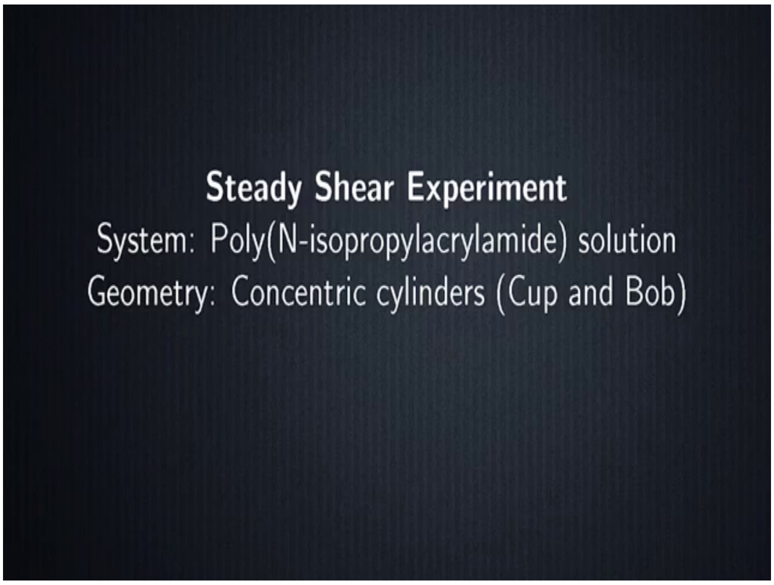

Student: so now, we will look at a variation of viscosity of thermo responsive polymer (Refer Time: 21:09) volume in it as the function of the shear rate it is as a function of shear rate. So, if we now look at the clear solution it is more like water the viscosity is actually low as a very low. Viscosity in this case it is more like water should actually measure the modulus we cannot use pp or cp, because they require high viscosity samples. So, one problem we have call this in a geometry. So, where we have more amount of sample so, the amount of signal available to the rheometer is more due to which it is able to characterize these materials which are low modulus and low viscosity.

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So now, we will see inside the rheometric geometry where we can see that the mixture is de mixed state and we will see a turbid solution towards a side in annular region of the concentric cylinders. So, first we have to filter the samples in a concentric cylinders in a geometry, and now it is a de mixed it.

So now we will set up parameters for steady shear test. So, let us go to the interphase and see it. So, here we actually fix the strain rate to actually vary from point one to 100, in this case, and then along this shear rate we will see how the viscosity of sample varies as function of as we increase shear rate from point 1 to 100.

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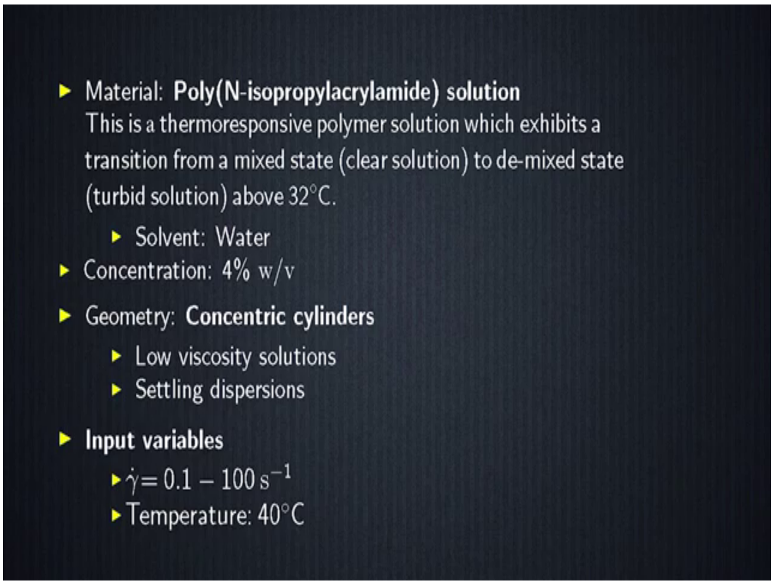

Here we can observe that the data is noisy in nature this may be due to the evaporation of the solvents as the sample has been kept at a higher temperature for long time. Therefore, one must be careful about these aspects while performing a rheological tests; however, we can see that even though the data is noisy, it does show an overall shear thinning behavior as expected in polymer solutions.

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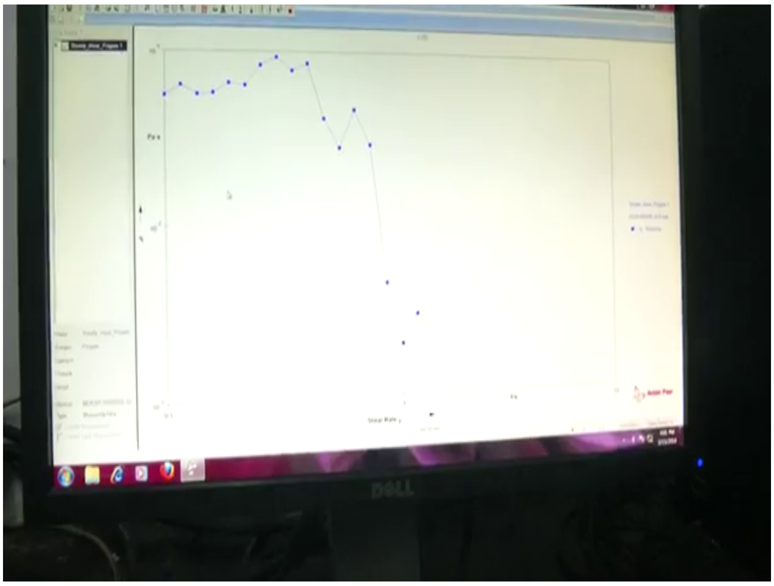

Now in the next graph, we have taken into account the evaporation of the solvent and ensure that there is no loss of it by using a solvent trap.

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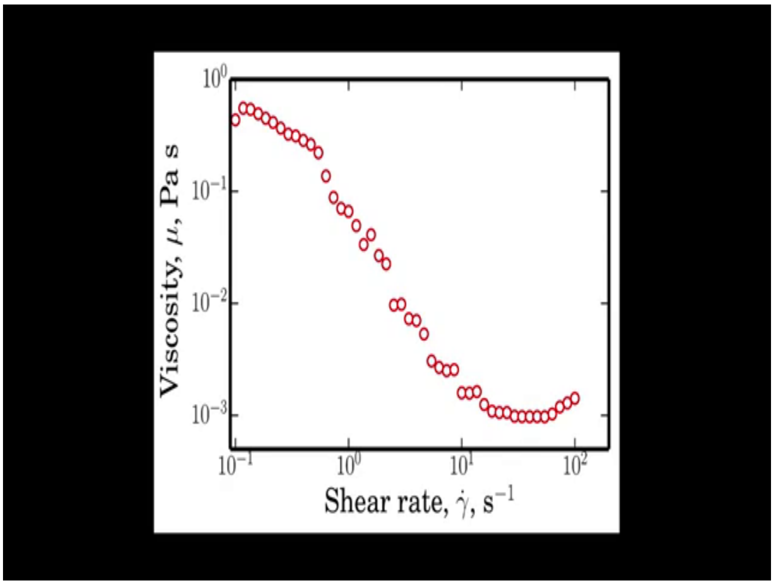

And there we see a very smooth decrease in the viscosity with the shear rate. Now what happens here is that, at high shear rates the polymer on an average orients in the flow direction, which causes the overall viscosity to decrease.

Thank you.