Introduction to Soft Matter
Professor Dr. Aloke Kumar
Department of Mechanical Engineering
Indian Institute of Science, Bengaluru
Lecture 21
Lab Session

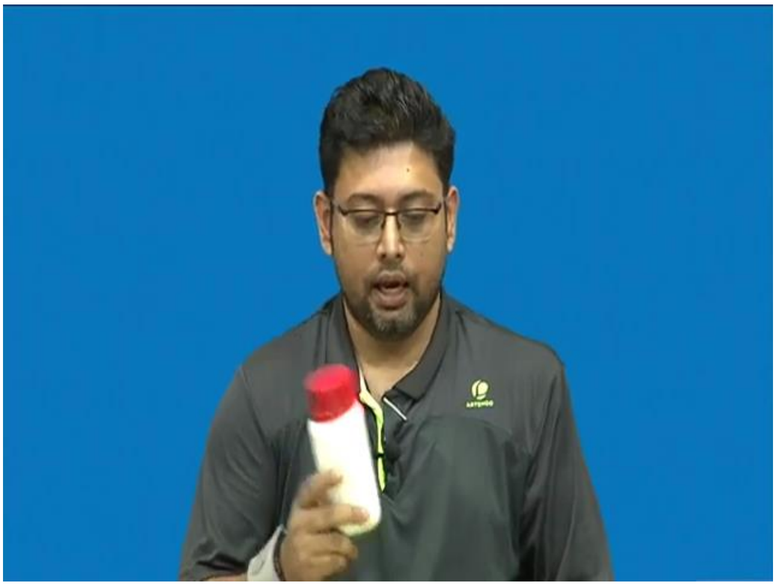
So, today is a very special class because we will be stepping out of the classroom and we will be heading into the lab and we are going to see in action some of the concepts that we have introduced and developed in the class here. We have discussed some of the examples of soft materials and we have said that polymers are one of the class of materials that demonstrate very interesting viscoelastic behaviour.

Today for the lab, what we have done is we have chosen a particular polymer and we have this material, which is a polyethylene oxide powder that comes in a dried state and this dissolves very nicely in water. And what we are going to do in the lab is we are going to make an aqueous solution of this polymer and then subject it to various rheometric tests.

Now, in the class we had discussed the stress relaxation and the creep phenomena. We discussed it in a theoretical setting, but what will a actual data look like? So, that is what we want to do in the lab where we will take our material and we will look at how an actual material behaves and what kind of graph would we get.

So, basically we are today interested in looking at the behaviour of a polymer from an experimental perspective.

(Refer Slide Time 2:03)



So, with us what we have is a box of polyethylene oxide. Now, this particular polyethylene oxide, you can get such materials from many different manufacturers. This one was sourced from Sigma Aldrich and here the manufacturer has created for us a document where some of the important information is provided to us.

So, for example, the viscosity average molecular weight is provided and the manufacturer tells us that this is 5 million grams per mole. The manufacturer also tells us that the PDI for this is between 1.14 and 1.37. This is a water soluble polymer and comes in a dried state. It is a powder state inside here. When we go to the lab we will open this and we will see how it looks.

Now, before we head into the lab, I must also mention one important thing that you should always keep in mind when performing experiments and that is the safety aspect. Whenever you use a chemical, please make sure that you read the material safety data sheet related to the chemical, which will specify the hazardous or the non-hazardous nature of that.

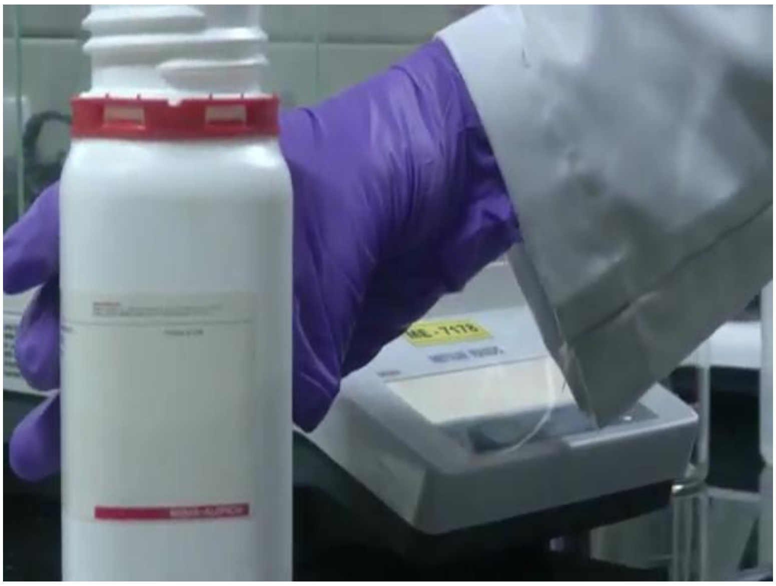
Now, this particular material, it has, you can find the material safety data sheet for this online and we have looked at it and the manufacturer clearly specifies that there is a section in the MSTS called hazard identification and the manufacturer clearly specifies that this is a non-hazardous chemical. The manufacturer also goes on to specify some first aid information.

And they also tell us that this material has no bio toxicity and bioaccumulation behaviour, but this is for this particular chemical. If you ever want to do an experiment in your lab, please make sure that the chemical you use, you are acquainted very well with all the safety related information before you perform any experiments. So, with this, let us go into the lab and let us see how the experiments turn out.

(Refer Slide Time 4:16)

In today's lab demonstration, we will be performing different rheological experiments using a solution of PEO or polyethylene oxide.

(Refer Slide Time 4:28)



Before we begin the experiment, we need to prepare a solution of PEO. The molecular weight of PEO we are using for this experiment is 5 million grams per mole. This is a viscosity averaged weight and is provided by the supplier. The poly dispersity index of this powder is 1.14 to 1.37. So first, we need to prepare a 1 percent solution of PEO.

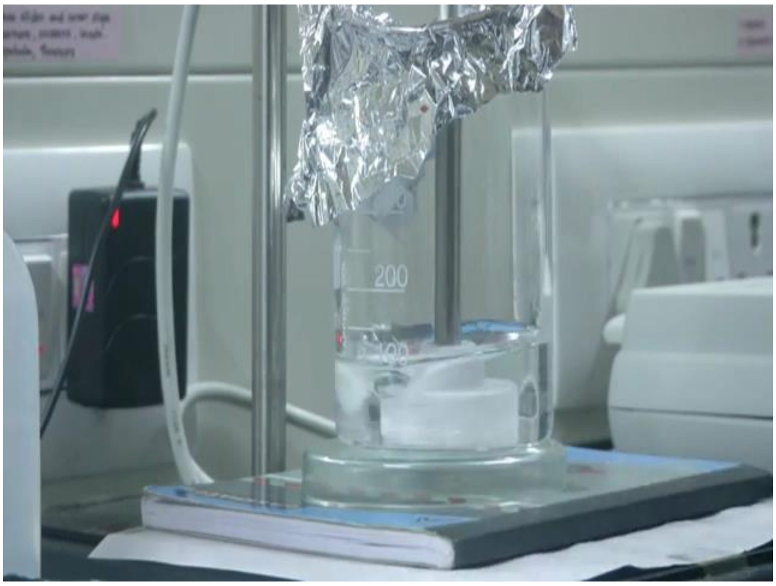
We do so by measuring 1 gram of PEO powder and then dissolving it in 100 ml of DEI water or de-ionized water. So, first we are taking the PEO powder and weighing it.

(Refer Slide Time 5:20)

Next we fill the beaker with 100 ml. We then add the powder to the beaker. The PEO powder is available in the form of water soluble powder, which is how we will be preparing the solution today. Once we add the powder, we need to mix the solution properly to get a uniform solution.

We can do so using several methods. In the current demonstration we will be doing so using two methods. One is using mechanical stirrer and the other using a magnetic stirrer. In the mechanical stirrer, we first place the beaker with the rod inside it and then start the stirrer. We set the stirrer to an RPM of 300 and then set it aside for about 3 hours.

(Refer Slide Time 6:36)

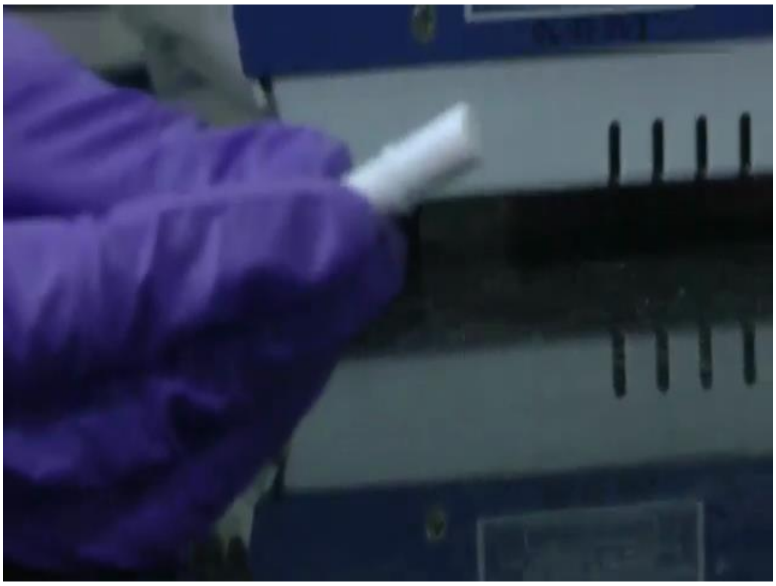


We will also be covering the top of the beaker using aluminium foil. This is done so to prevent any dust particles from falling into the solution. We will now set aside the mechanical stirrer.

(Refer Slide Time 7:12)

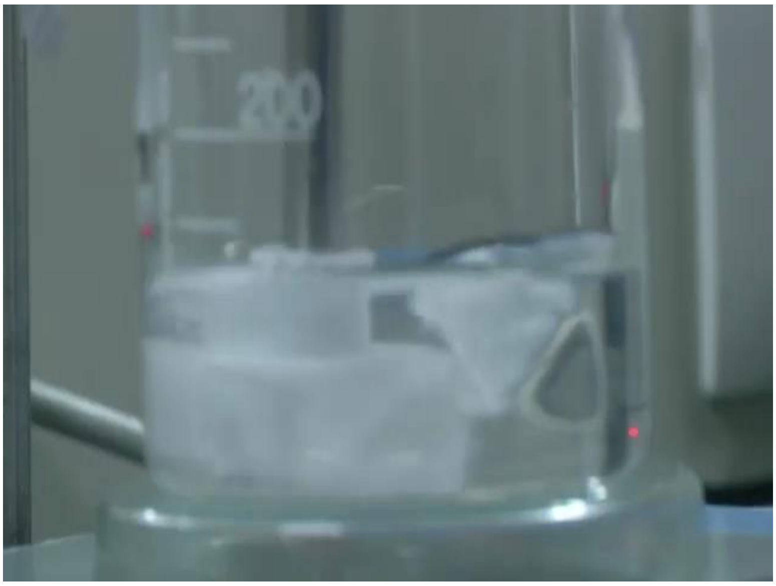
Next we move on to the magnetic stirrer. In the magnetic stirrer, we basically have a magnetic bead which is kept in the middle of the beaker which has the solution. Next we place it on the magnetic stirrer and set the RPM. In the current setup the RPM is set to 1000. It takes about 24 hours in the magnetic stirrer to prepare a uniform solution.

(Refer Slide Time 7:46)



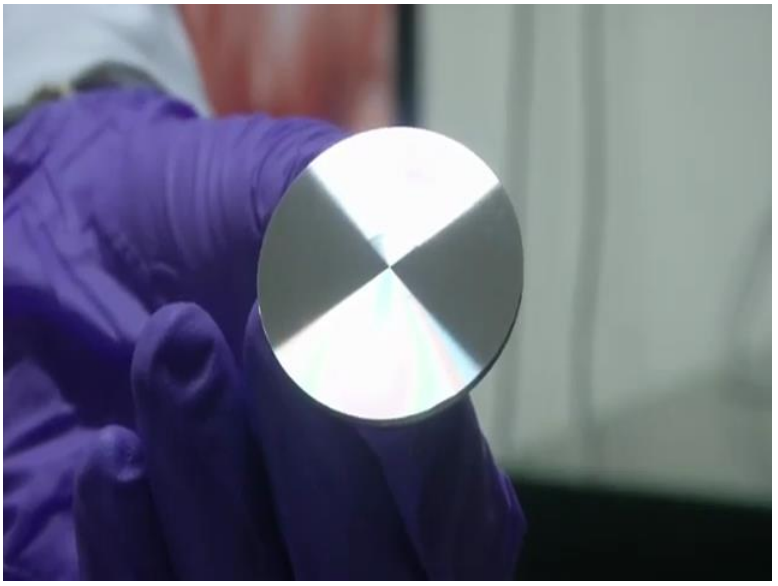
So, we now have the magnetic bead. It is basically a magnet and how this magnetic stirrer works is because of the rotating polarity of the equipment. Moreover in the magnetic stirrer setup, we can also set the temperature at which the solution needs to be prepared. At present it is set to the room temperature.

(Refer Slide Time 8:12)


200

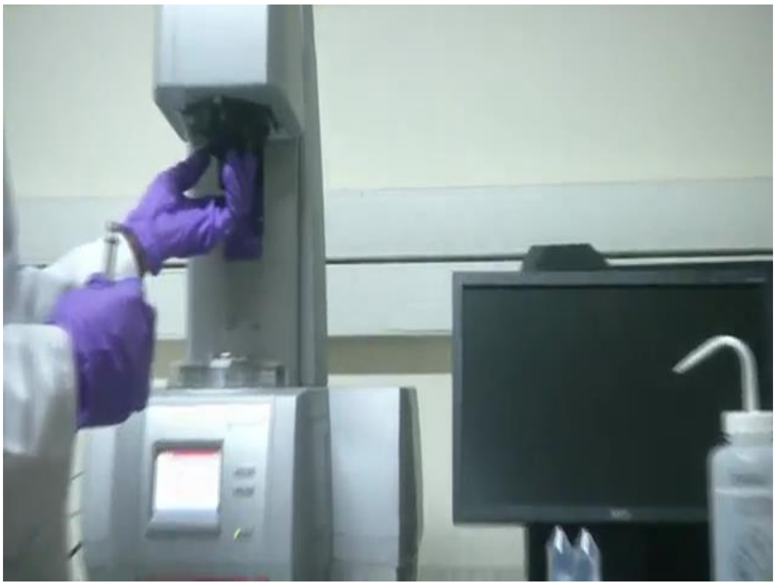
Now, coming back to the mechanical stirrer, it is useful to note that the mixing time is not fixed. It is not a fixed parameter, it depends on the solution that we are preparing and the method of mixing we are using. You can notice that using the mechanical stirrer, it only takes about three hours to prepare the solution, whereas the magnetic stirrer takes about 24 hours to prepare a uniform solution.

(Refer Slide Time 8:43)



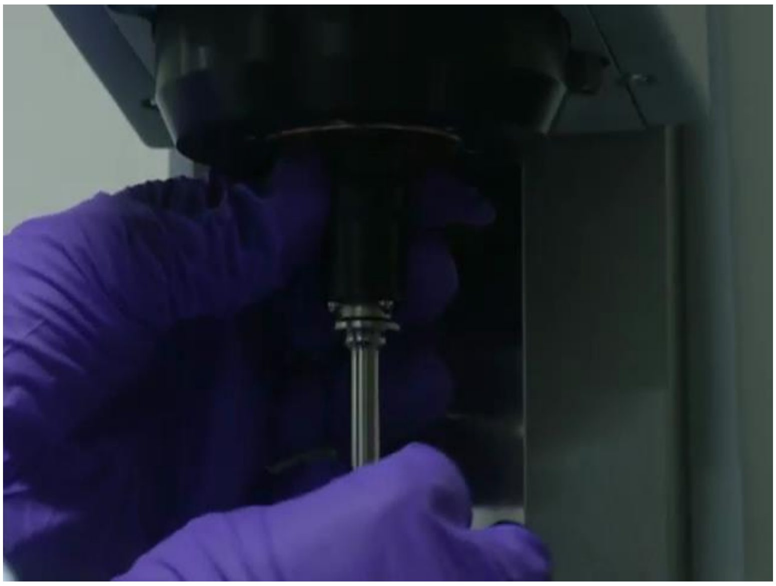
Now that we have the solutions prepared, let us move on to the experimental part of the demonstration. What you see is a conical plate, which has a 40 millimetre diameter and a 1 degree angle.

(Refer Slide Time 8:58)



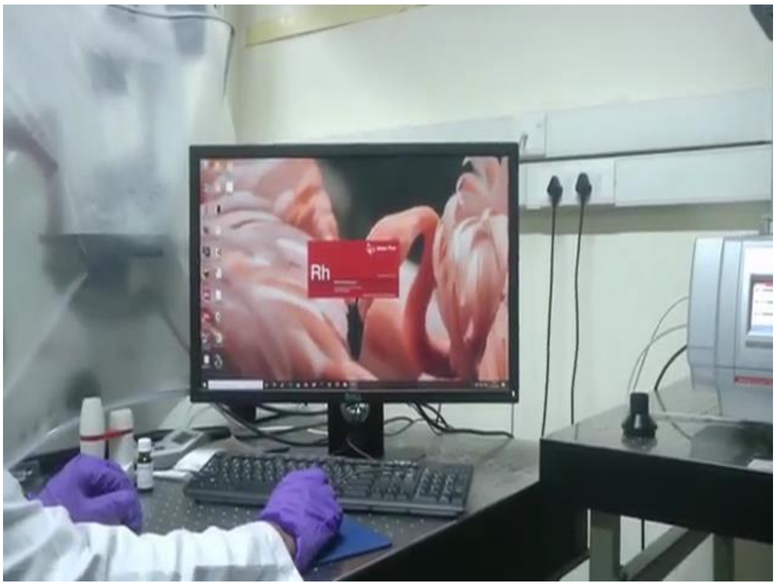
This is an Anton power rheometer with model number MCR 302. We will be performing today's experiments on this rheometer. So before we start our experiment, we will insert the conal plate into a cylindrical rod and then attach the cylindrical rod to the measuring system. Before we do that we need to remove the protective covering for the measuring system.

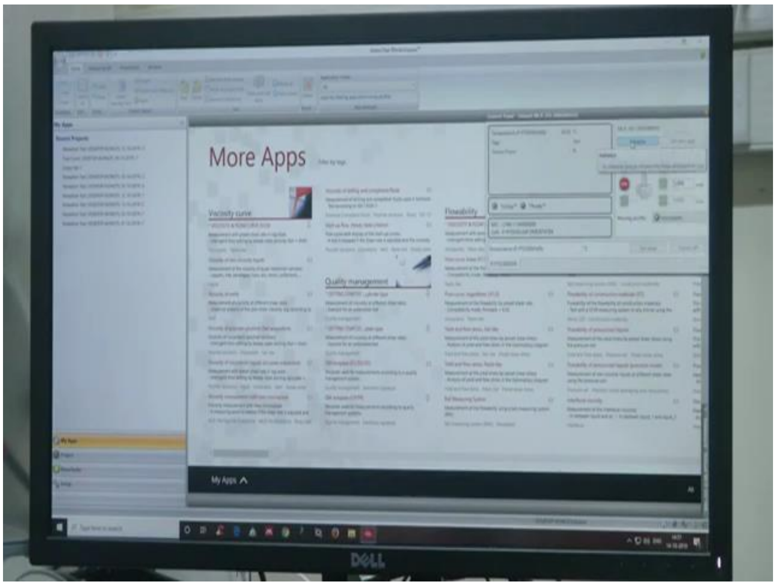
(Refer Slide Time 9:27)



We then insert the rod into the measuring system. The conal plate has already been attached to the bottom of the rod.

(Refer Slide Time 9:40)





After this we will be performing some calibration checks for the rheometer. We control the rheometer using a software custom designed for this equipment called the rheocompass. In the rheocompass, we can see several app modules. One of the app modules is for performing this calibration checks or the zero gap check.

(Refer Slide Time 10:17)

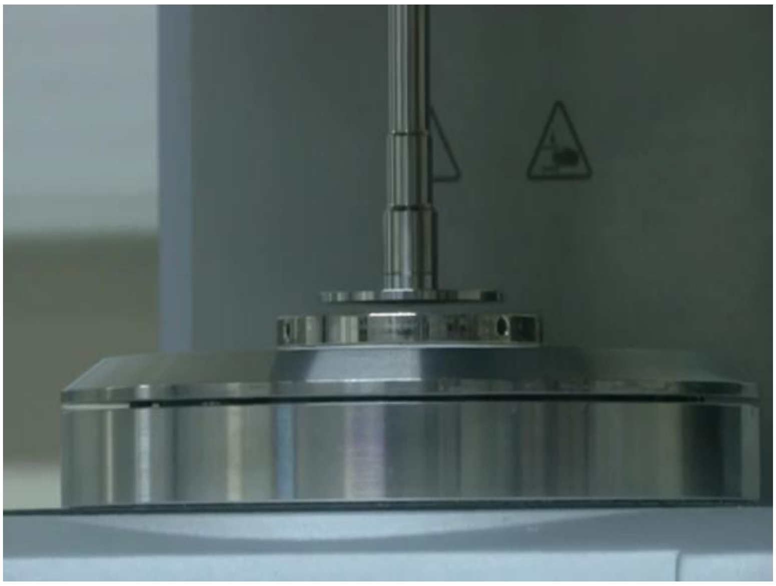
In the first part of the calibration check, the software tests the motors of the rheometer to ensure that it works properly. After this, it goes for the zero gap check. In the zero gap check the conal plate geometry is brought very close to the lower stationary plate. The actual value of the zero gap is pre-set.

So as you can see the equipment is bringing down the conal plate to the lower plate. It will go up to a gap of 0.08 millimetres.

(Refer Slide Time 10:55)

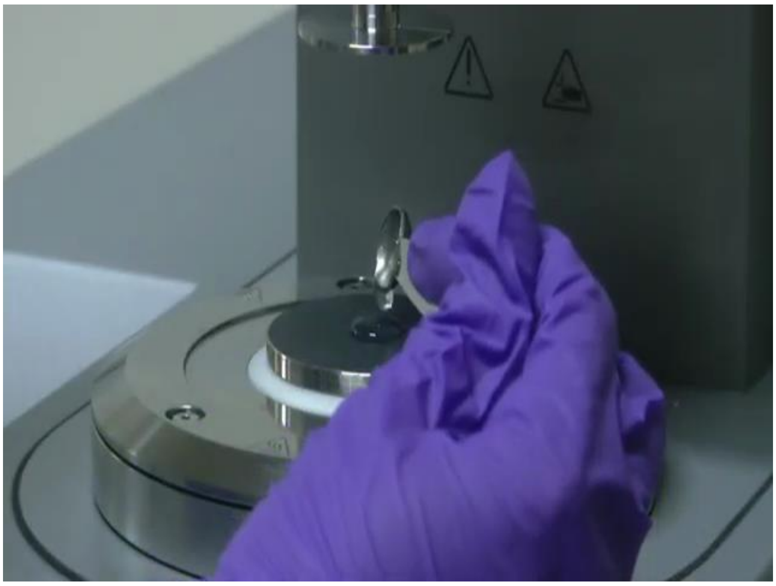
So as you can see on the screen, the gap is currently 0.08.

(Refer Slide Time 11:06)



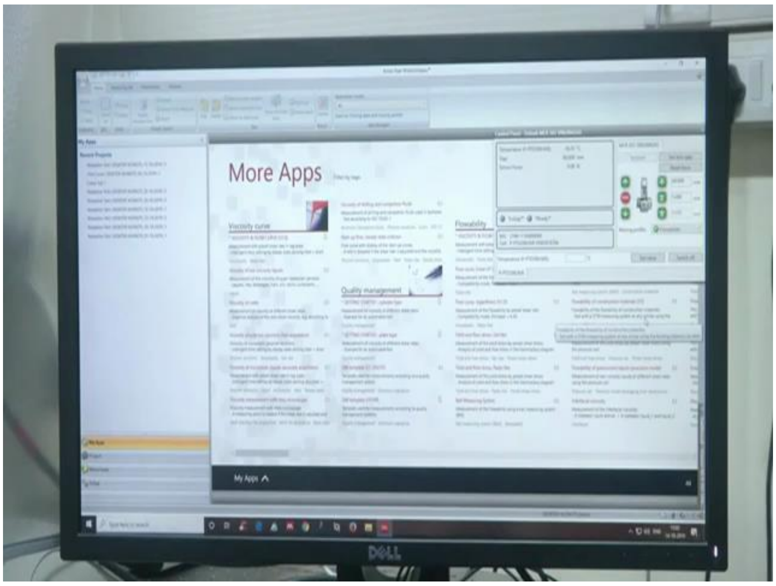
Once this calibration is done, the conal plate is taken back to another place at height of 60 millimetres.

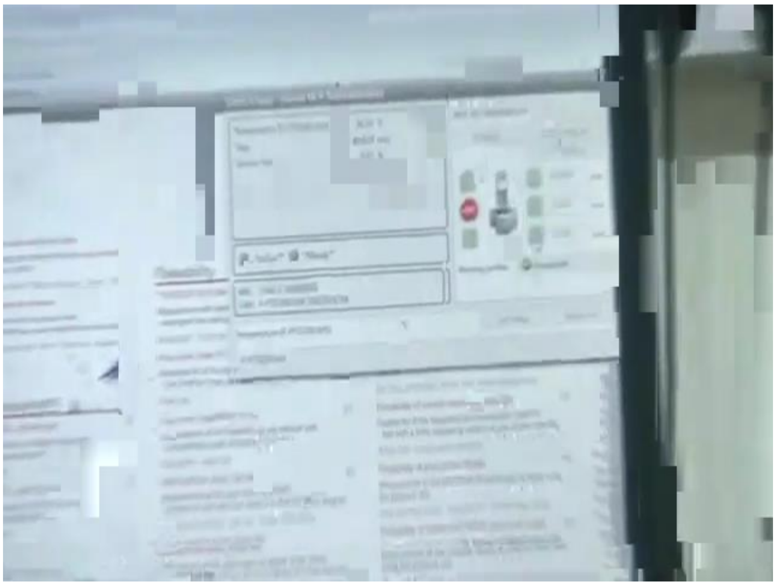
(Refer Slide Time 11:15)



Once the plate is fully retracted, we place the sample fluid on the stationary plate. While placing the sample, we must be careful to place it roughly in the centre so that the conal plate can cover it completely.

(Refer Slide Time 12:06)



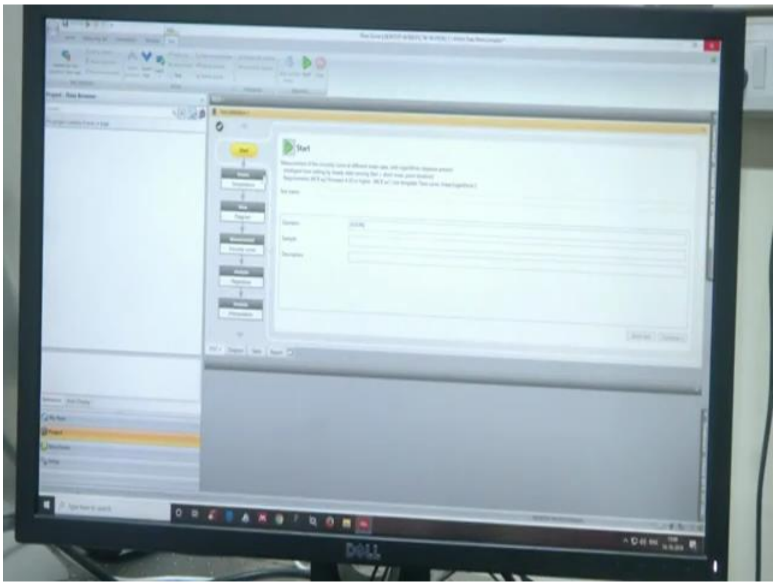


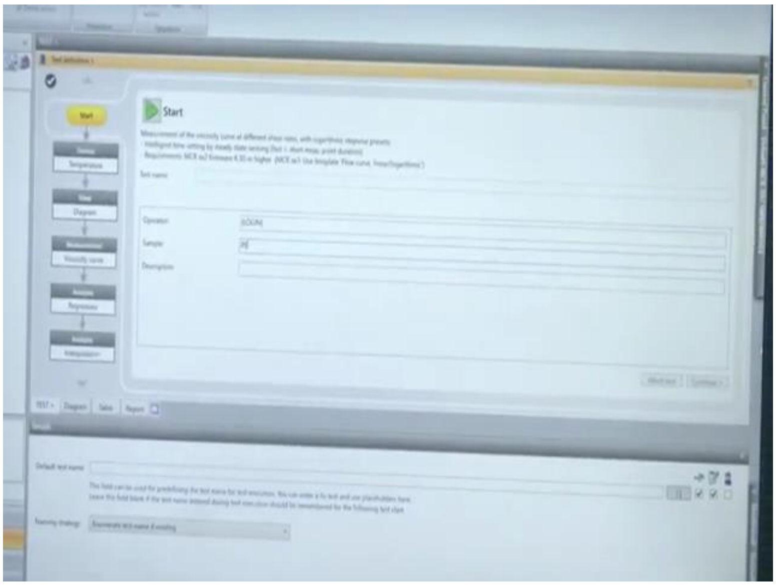
After the sample is placed, we give the command to bring the conal plate down to preset height of 0.08 millimetres. This is the height at which we will be performing our experiments.

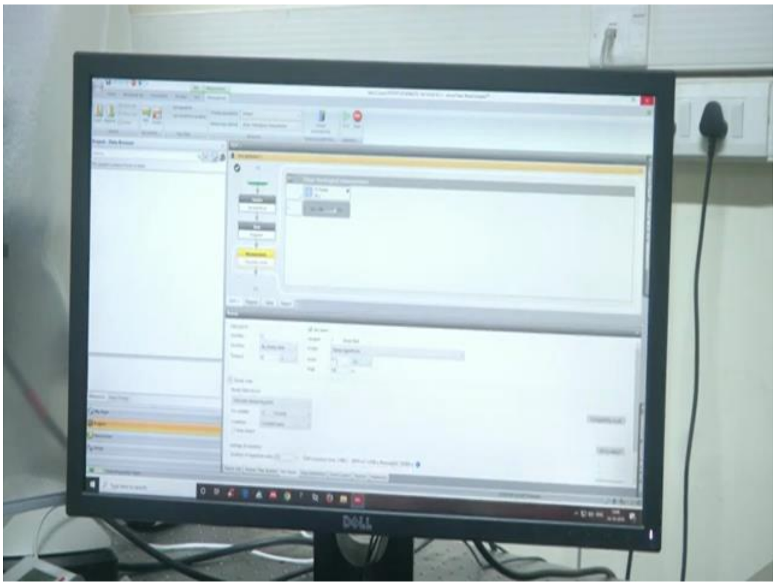
(Refer Slide Time 12:21)

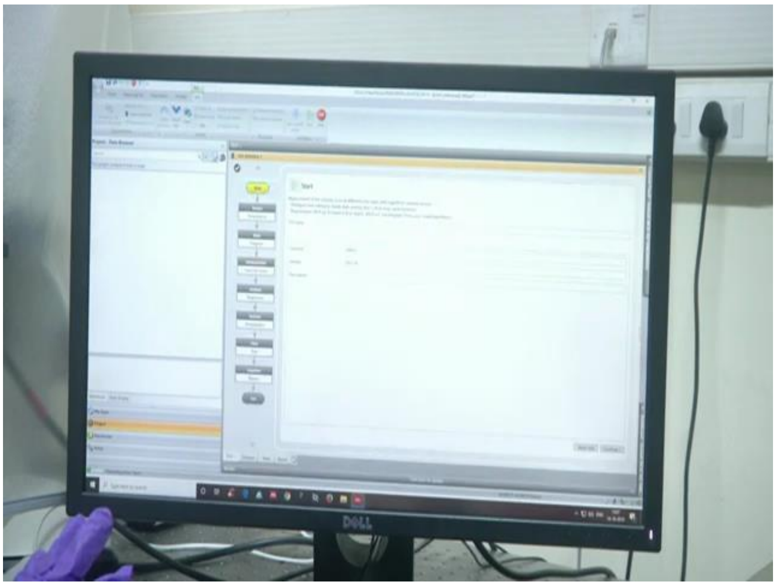
After the plate is lowered completely, make sure that it subsumes the fluid. If there is still some excess fluid around the outer edge, then it must be trimmed off before we start the experiment. Now we can start the actual experiment. We first begin with the flow curve. The flow curve measures the viscosity of the sample with respect to the shear rate.

(Refer Slide Time 13:03)




Start



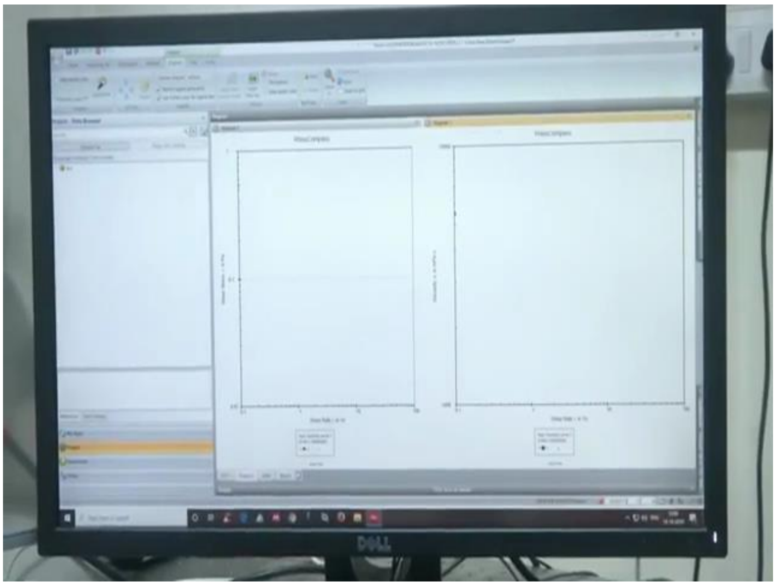


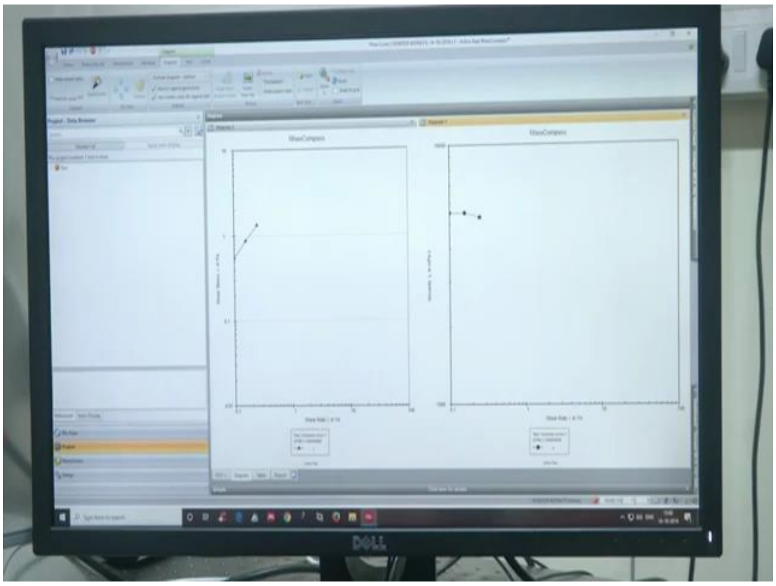
To run this experiment, we first open the required test module within the rheocompass software. This gives us the following user interface. We first enter the details of the experiment such as the sample name, the percentage concentration of PEO in the solution that we are using, etc. Next, we give our input commands. For the present experiment, it is the range of the shear rate.

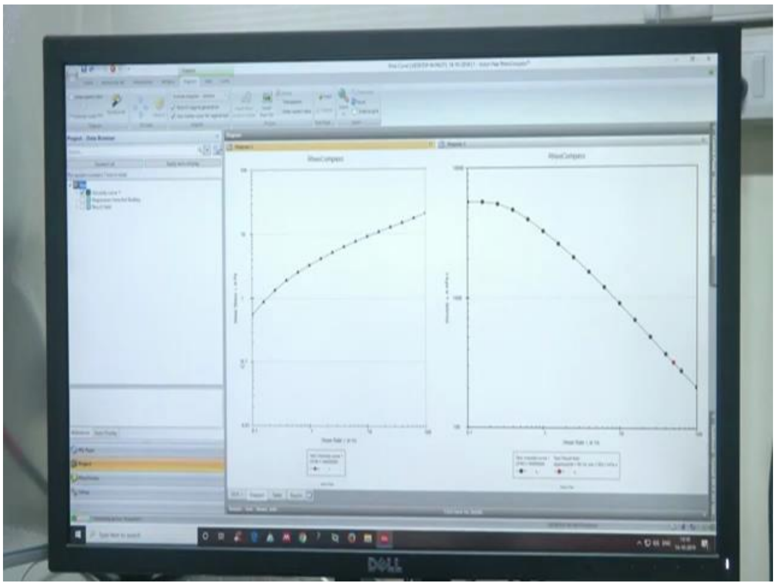
We are considering this range to be from 0.01 to 100 per second. The shear rate variation is set to lamp linear mode and then we go ahead and give the start command for the experiment.

(Refer Slide Time 14:24)


Anton Paar





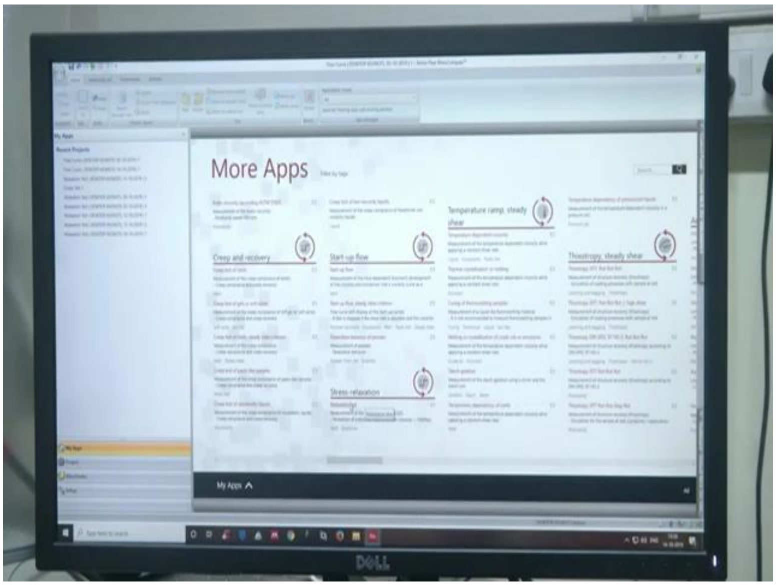


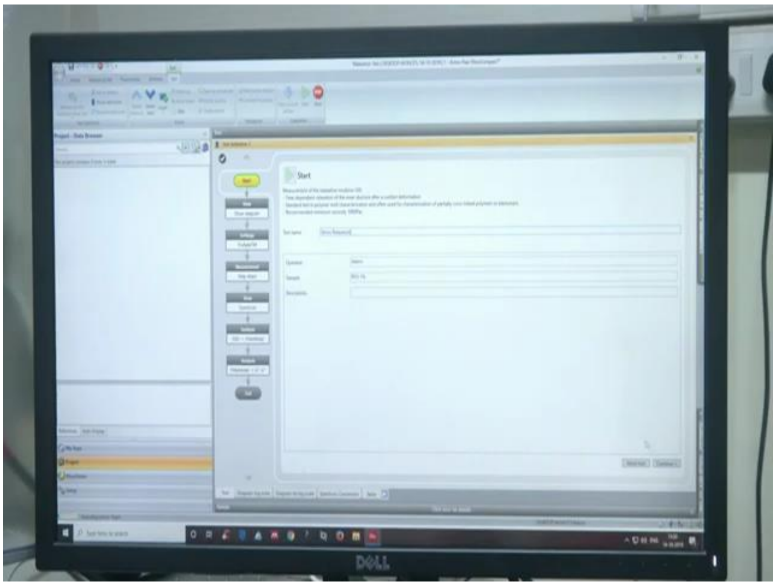
Moreover, in the rheometer, we also have the option to set the temperature conditions for the experiment. In this case, however we have set it to the room temperature. The data from the experiment can be observed in real time on the software screen in the form of an output graph. The graph on the left side shows the shear stress vs shear rate plot and the graph on the right shows the viscosity versus shear rate plot.

Now skipping ahead to the final result, we can see the shear stress increasing with shear However, on a more important note, we can see the decrease in the viscosity of a fluid sample.

This implies that our fluid is a shear thinning fluid. Moreover, if we let the stress run for a longer duration, I mean, if we let the experiment run for a longer duration, we can also get the value of the infinite shear viscosity. However, for the sake of time, we have ended it at 100 per second shear rate.

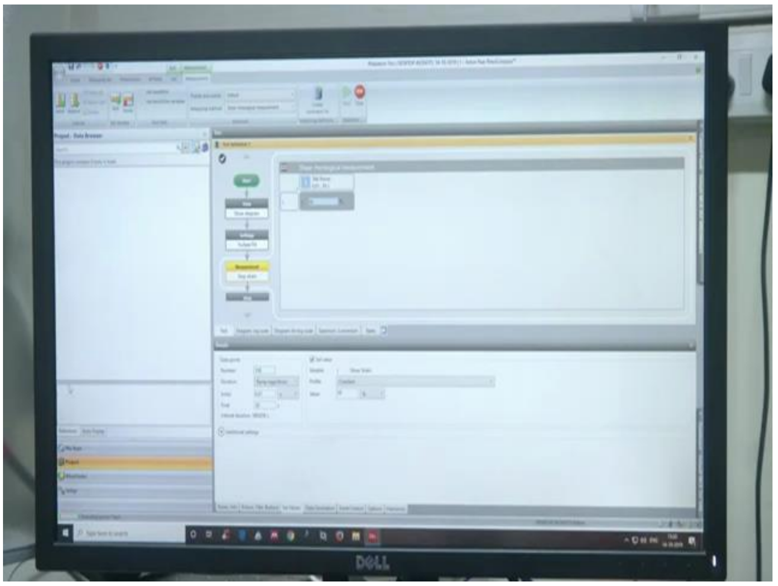
(Refer Slide Time 15:37)





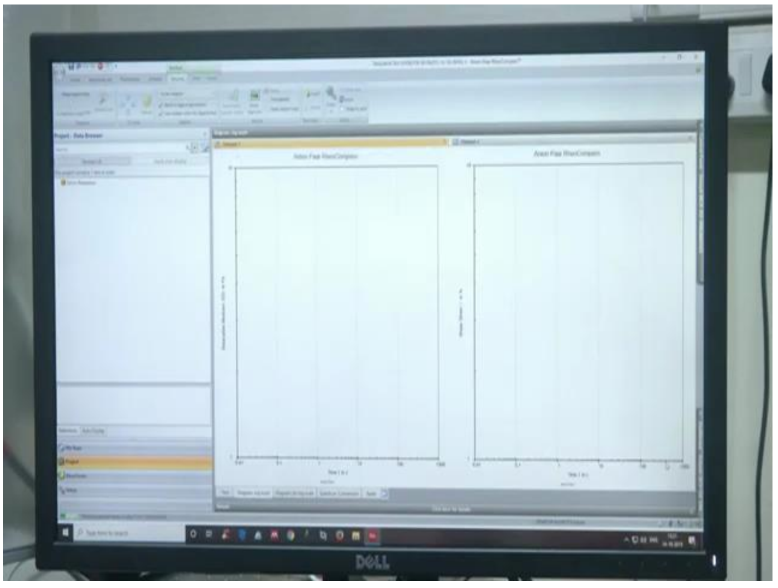
Next, we carry out a stress relaxation test. For the stress relaxation test, we give a constant strain rate and observe the changes in the values of the fluid stress. Similar to the previous experiment, we first note down the sample concentration data and name. Next, we give a strain rate as the input parameter and then start the experiment.

(Refer Slide Time 16:24)



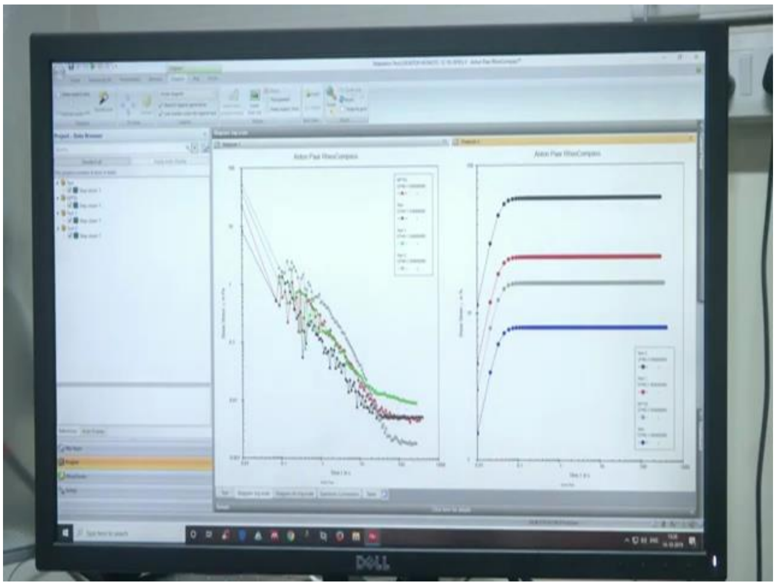
As you can see on the screen, the right side is the input. So currently we are setting the value of the strain to be 20 percent and we are collecting 100 data points.

(Refer Slide Time 16:54)



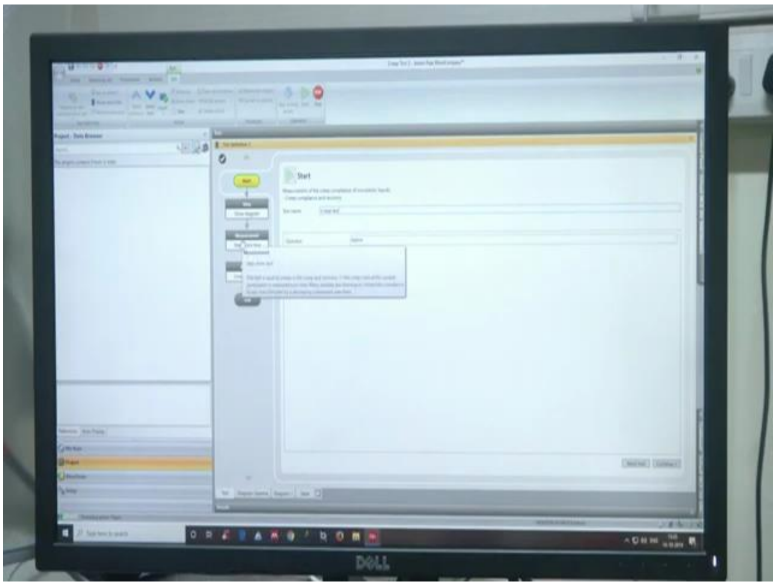
As you can see on the screen, the right side is the input strain and the left side is the variation in the fluid stress. The strain takes a finite amount of time to reach a steady value. So, while analysing the experimental data, we must be careful to remove the output stress in the region where the strain is not constant. Now, we have already done experiments for various other strain rates also. So, as you can see, the fluid stress is decreasing with time.

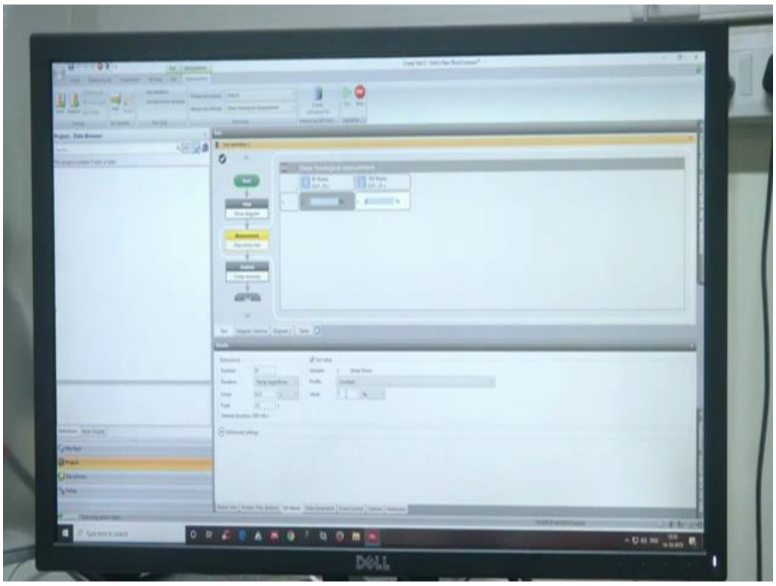
(Refer Slide Time 17:44)



Now, we have already done experiment for other strain rates also and you can see the results here. An interesting question that comes up here is whether we can say from the stress relaxation plot whether the fluid shows linear behaviour or not?

(Refer Slide Time 18:19)

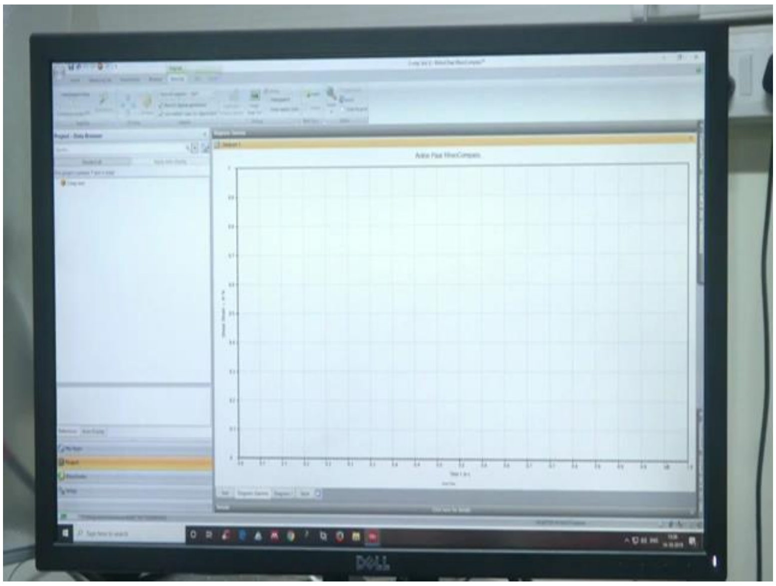


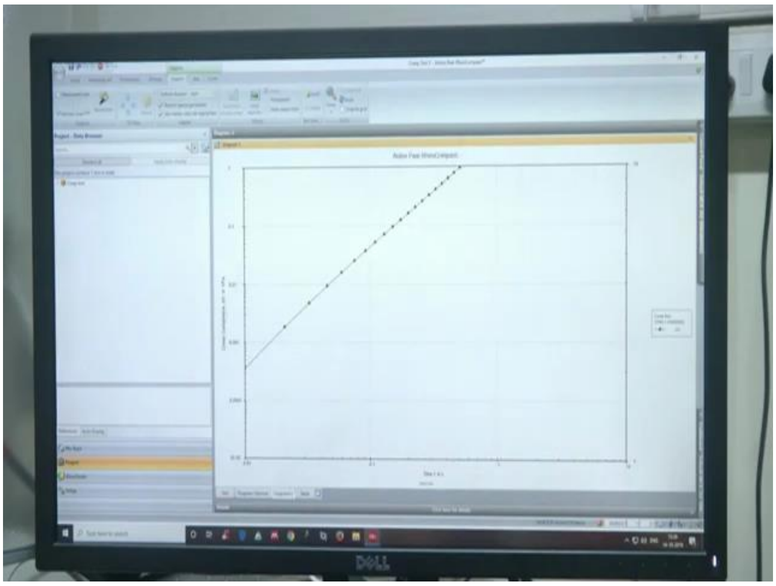


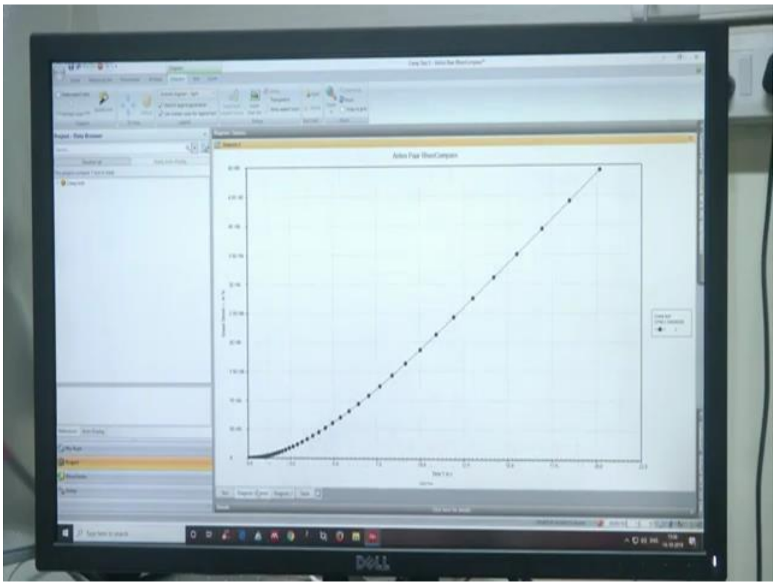
We now move on to the creep test. In a creep test, we give a constant stress input. In this case we will be giving a 100 Pascal and then observe the changes in the value of the strain. Similar to the previous cases, we first give the test name details. Then we set the value of the stress input and the number of data points.

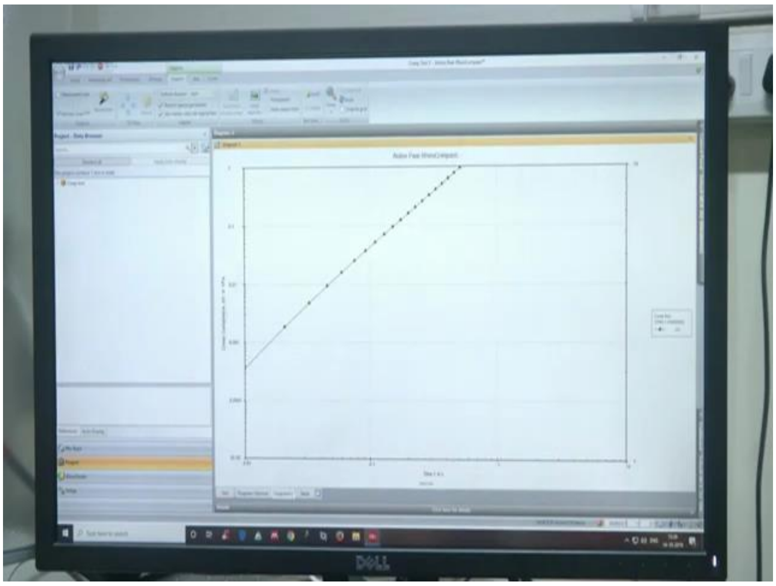
So the stress is set to 100 Pascal's. We now start the experiment.

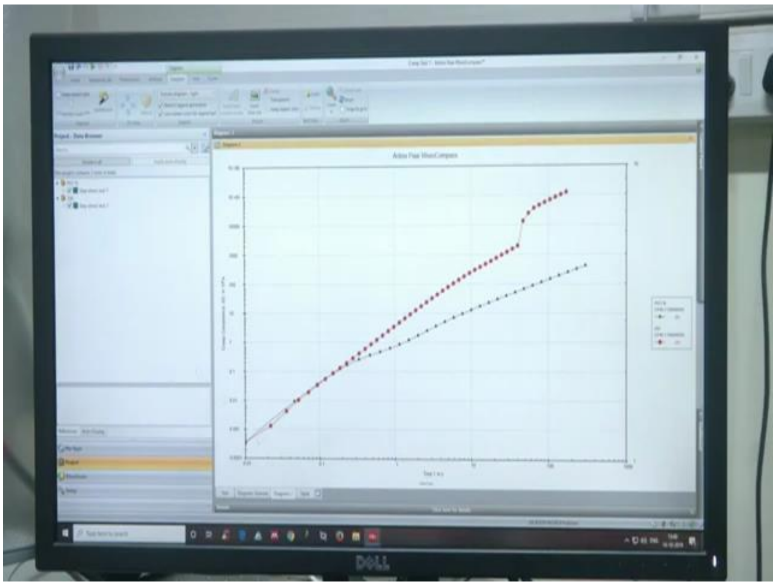
(Refer Slide Time 19:51)











So, you can see the changes in the value of the strain for the given stress. That is you see the creep compliance varying with respect to time. In the creep experiment, it is the variation of the creep compliance that is of interest to us. For this test, we have also performed the experiment under stress of 200 Pascals.

And you can see the results here. The one in the red is for 200 Pascals and the one in black is for 100 Pascals. Here again, an interesting question comes up of under what conditions are on in which range is the creep compliance showing a linear behaviour?

So, we are back from the lab and we have done some interesting experiments. And we investigated, first we looked at the viscosity of the polymeric solution as a function of shear rate. We also looked at the stress relaxation behaviour of that for four different step strains. And we also looked at the creep compliance behaviour.

(Refer Slide Time 21:51)

So, that was an interesting experiment and it was very interesting that this polymer now because it was dissolved in water, it is almost colourless, and it almost looks like water. So if you work with it, you will see that it is very much like water, but it is just a little bit viscous. So, you can see that I can actually pull this out and you have this thin films, then strings that are developing.

It gives you an insight that this material has now, the behaviour of this is very different from that of liquid water. So, even though you have dissolved a very small quantity of powder, the final material has a very strong viscoelastic characteristic. We saw that the stress relaxation phenomena seems to indicate that the linear scaling may not apply in this particular case.

But I must warn you that we have only done one experiment and before you come to any conclusions, you must repeat your experiments a few times. Repeatability and robustness. have to be ensured. So, and then there are different geometries, we did not look at all the possible geometries, this is not an exhaustive course on geometry, we only took us one simple geometry that we had in the lab to investigate all these behaviours.

In a realistic case, you will probably have a set of different geometries from which you will be able to choose and make an appropriate choice based on that. The shear thinning behaviour was also clearly seen and this corresponds nicely to for example, the Carreau model that we have discussed.

So, I think I hope you had an enjoyable time in the lab and you enjoyed taking a look at the actual experiments. And from the next class we will be back in the classroom setting, discussing a few more theoretical ideas.