Rheology and Processing of Paints, Plastic and Elastomer based Composites
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Lecture 22

Practical demonstration on PPR and DMA

Welcome back to NPTEL online certification courses on rheology and processing of paints, plastics and elastomer-based composites. And today we will give a practical demonstration on parallel plate rheometer or to be specific I will be talking about a whole deal of rheometers based on a model of AntonPaar 102 modular compact rheometer. So, first I will be dealing with the basics of the structure of the rheometer. So, if you look at the rheometer you will find a couple of things. This particular shaft is called the lower shaft or the lower geometry and I also have a couple of measuring systems. So, I will discuss about the measuring systems and when to use them.

If you go back to the machine we will find that it contains a upper portion which contains the motor and the transducer which will actually record the torque experienced by the motor. So, here I will open this cap and you can find this is the motor or the rotor and this is having air bearing. Now why to use a rheometer? Now in the subsequent classes and also the previous classes we have discussed about various rheometers like the ODR, MDR, also the mooney viscometer and we will be also discussing about the RPA. Although there are several drawbacks for processing or calculating or evaluating the rheological properties with the help of a rheometer in case of rubbery materials, but we cannot afford to have the sensitivity of the measurement like we do get in case of a rheometer.

Thus the transducer over here is very sensitive. So the torque it can measure can sometimes go to in a nano ranges. Secondly this rheometer is usually having not a mechanical bearing but an air bearing. So that is why the part which I showed just couple of minutes ago this one is very sensitive. So this is the heart of the machine.

I usually demonstrate the heart and the brain of the machine. So this is the heart of the machine. This is the main portion of the machine. That is why when the machine is turned on we have to ensure that the compressor connected with the machine should be on and the air flow is not restricted. Then only we will remove this cap and when we will close the machine before switching off the compressor we have to put the cap back.

So next you can see there is a chamber. You can see the chamber over here. This two side there is chamber. If you close this the chamber will be closed. So this is called CTD

450 controlled temperature device.

So the temperature inside the cabinet can be controlled. So there are heater and there are also you can see this black color ducts which can be connected with the liquid nitrogen supply system and thus it can be cooled also. Now if you don't have a liquid nitrogen supply system how to cool this? So there is also a mechanism for this. The air which comes to the machine can be divided into two parts. One will be supplied to the motor, the air bearings.

Another will be supplied to the shaft cooling as well as to the air cooling system in this particular assembly. If you look at the back of the machine you can see there are two flow meters. One is heating, another one is shaft cooling. So I will come back to this. I will switch on the air supply.

Now you can see I can control the air supply from the shaft cooling with the help of the knob. So where is this coming? And also for the heating also where is the air flow going? So again if you come back to the CTD, so here are perforations. So you can see if you can see over here some perforations are here, some perforations are here. So these perforations are actually connected with the air supply which will cool the lower shaft as well as the upper shaft. Also we have other perforations inside this which will help in cooling the cabinet.

Along with the air supply this particular assembly of CTD and the machine inside the machine wherever it gets heated up we also have a water cooling system. So we have a chiller connected with the machine and this particular pipe, this black color pipe on both the sides of the CTD contains the water line from the chiller. Now this lower shaft can be changed according to the experiment we want to conduct. So this can be opened using this particular knob and we can open this. Along with that we can also change the geometry.

Now what is geometry? Like this one is having a particular diameter. So the maximum diameter we can go over here is approximately like 40 mm. So this is how the things work. This is the upper geometry, this is the lower geometry. So the upper geometry comes in the proximity of the lower geometry according to the thickness of the measurement and it does the measurement either in an oscillatory motion or in the rotational motion.

Now if we want to change this lower geometry we have some options. Just like this geometry is having a plane geometry. As in the theory classes Professor Chattopadhyay has explained like, slippage is a major problem for the parallel plate. So in order to avoid the sleepage what we can do is we can have a profiled plates. So this is also a parallel plate like I will be putting this over here and I will put a upper plate.

So this is also a parallel plate but this is profiled. Like you can see it is having some profiles. So it is corrugated. That means this will help in gripping the sample and the slippage will be less. Apart from this if we are having a very small amount of sample and more specifically sometimes the sample can be very stiff so that time we have to go for a lower geometry, even lower.

So this one particularly is a lower plate of 8 mm. So this one the diameter is 8 mm. So we can put this over here and we can use a 8 mm upper plate and we can do the measurement. This is valid for a viscous sample or a sample which is having moderate viscosity. For a higher like a viscosity elastic samples we have to go for the corrugated.

Now if the sample is very stiff then we can move from a rheometer to sort of a DMA. So then we have to change the lower geometry. Now this you can see this is the another part of the lower geometry. So we have to change this one, we have to lift this one and we have to put this inside and this helps in clamping the samples. So I will show you the samples can be clamped.

So here is a sample and we can clamp the sample inside and we can tighten the screw and eventually the sample will be tightened. So this tightening is done using a particular torque screw meter and this one is the temperature sensor. So in this case the temperature sensor is inbuilt but in this case we have a thermocouple which comes out of this shaft and it records the temperature. Along with this we can also have, so this one is mainly for the torsion. So we have a upper geometry which will catch hold of this.

I can show you like this, catch hold of this and it can do in a torsion mode just like this. If you want to do in a tension mode, so we cannot actually do in a tension mode because this particular motor can rotate. It cannot actuate something like in a tension. So it can give a shearing action. Now this shearing action can be transferred into a elongational action with the means of this device which is again the lower geometry, the change of the lower geometry.

Now this lower geometry if we insert over here and this one is the upper geometry. So this will, I am showing you like this but we have to clamp it over here for the ease of understanding. Now if we are pulling this like this that means the transducer or the motor is giving a shearing action. It is just oscillating. But actually the sample inside this, I will show you.

So if you can see over here, the sample over here if you are giving a oscillatory action on the upper shaft but it is actually giving a tensional force on the sample. So it is no more shear, it is tension. You can see the sample is being elongated. So this is actually converted into using various equations. The basic one is the E is equal to 3G and it can be converted into respective elongational modulus or tension modulus and so on.

So this completes the overall anatomy if we connect a CTD. Apart from this, we can also connect a Peltier device. So we have to open this whole assembly. We have to lift this up from here and we will be connecting a Peltier device. We can control the temperature directly using the electrical signals that will be completely monitored by the software.

So this is all about the lower plate. Now I will be discussing about the upper plates. So this one is a very basic one which is PP25. Why PP? It is parallel plate.

Why 25? It is 25 mm. So the diameter is 25 mm. Next is we also have PP08. That means same parallel plate but the diameter is 8 mm. So a smaller version if our sample is very stiff or something like that.

Next we have CP. That is CP means cone and plate. Why plate? Because the lower geometry is still parallel. So it is a plate but the upper geometry is cone. If you can focus on this particular plate, you can see it is actually a cone. So it is having a slanting over here and now see here it is written as CP25-3.

So 25 is the diameter and what is this 3? 3 is the angle. The angle of the cone that is 3 degree. Similarly we have CP08.

Same. So cone and plate. So this is actually cone. That is why it is cone and plate and 0e. Now this one as I discussed earlier, it is PP25 but P2. That means it is a profile version. So this is actually gross about the upper geometries or the measuring systems.

Why it is a measuring system? There is also a reason behind that. See if you focus over here, we have a tool. So this is actually an electronic circuit which fits into the upper motor and it connects with the motor and it has the coding encoded inside this and it will detect the measuring system and it will start measuring according to the measuring system. And this actually measures the torque from the sample. That is why this particular assemblies are called measuring system.

Now apart from this, we have torsion also as I showed the lower plates. Now this is the upper plate for the torsion and it is SRF, solid rectangular fixture. It is SRF. Another one is the UXF, universal fixture. Why universal? So as I mentioned earlier, it is mainly because of the tension.

So this is another fixture, the upper geometry which records the data in tension mode. So this grossly completes about the rheometer and this can also be extrapolated to a DMA. Why DMA? Because of the SRF and UXF attachments where we can measure the sample in the torsion and tension modes. And also in case of parallel plate, we can measure in the shear mode.

So mainly two types of modes are there. One is rotational, another one is oscillation. So I will show you how these things are done. And along with that, as I have shown you, one is the cabinet based heating that is a control temperature device, 450 means it can go up to a temperature of 450. And there is also a Peltier device which we have is from minus 100 to plus 200. And we also have another attachment called the MRD that is magnetic device which records the rheology of a particular suspension or a system with respect to the magnetic fields.

So actually what happens is we have a device which will be connected with the magnetorheological device and it will give electrical or it will pass the current and it has coils. So from the very basics of the physics, we know that if there is a current and there is a, so there is also a magnetic field. So on increasing the current, the magnetic field increases and with the increase in magnetic field, the rheological properties changes. So that is again recorded by the transducer which is present at the top of the machine with the help of these measuring systems. Now as I told that there is again a problem for like there is an approximation when we are going for a tension in this particular machine.

And like Professor chattopadhyay has talked about the cantilever, single cantilever, single point, three point bending. So these are not possible in this machine. Why? This is having only one drive, that is single motor. For the samples to be experimented in those particular conditions, we need twin drive motors. So one motor will be up which can give torsional forces or the shear forces.

Another motor will be down which will give the reciprocative action. So if this combination is there, then only we can go for the tension, true tension and like the three point bending, single cantilever, all those things. So these options are available but in the higher version of this machine, so like this is 102 for 702 onwards, we have a hole in the downward portion of the machine and we can attach another motor which is a reciprocative motor. So that is why it is called twin drive. So now we will switch back to the software of the machine and we will show you simple experiments on shearing and that too with the parallel plate and cone and plate in rotation and oscillation modes.

So now we will focus on the software part. So we are using Anton paar RheoCompass. This window will come first. This window contains a whole lot of experiments with preconfigured tests and experiments. So we have to just search over here.

Suppose you want to do a amplitude sweep. So you have to write amplitude. Again in this we will get a whole lot of amplitude sweeps like, so this one is a amplitude sweep in parallel plate. This one is in tension. Now this one comes into frequency sweep in parallel plate.

This one is a temperature sweep. So it comes like this. Now if you give a frequency sweep, see again it comes like this. So you just have to write what you want to do and it will come. Suppose you want to do any magnetic sweep. So you just write as magneto sweep.

See it is coming over here. You want to do a creep test. See creep and recovery, creep and recovery in extension, in shear. And this compatibility is something suppose you have a MCR 102 or something like that which is a lower version machines. So we go for compatibility. So we also have a whole lot of experiments in compatibility. So you can see like in the viscosity region in case of frequency, in case of temperature sweep everywhere there is compatibility programs.

So this is something about the programs. Now the machine has to have a initialization. So it has to know what its highest and lowest points are. So we have to do a initialize. So it is reaching its highest position over here.

It has recorded its highest position. Now we have to use our measuring system. So now we will use PP 25 and we will wait till it says that it will come as reading MS and here it will come the name of the MS. See it is PP 25 with the serial number. Now what we have to do is the zero gap. Why? This gap is very much essential in order to calculate the rheological properties of the material because it depends on the thickness also.

So we will go for the zero gap. So it has got a zero gap and a 1 mm thickness. So now we are ready to go for the experimentation. Now we will use a simple amplitude sweep program. Any standard program will do because we are doing a shear mode.

So we are going for shear mode. So they will tell to give a name. We can give it as amplitude sweep and save this. So it has few test definitions. So this is the main program where we can alter the program from the details. So in case of strain sweep, the frequency is kept constant. So I will give a frequency of 1 hertz and I will go for a shear strain which is oscillating shear strain of 0.

01% to 100%. We also have a point density that means from 0.01 to 0.1. How many points do we want? Five is enough for me so I will apply. Now if you want to have a particular temperature on this test definition, you can just add. You can go for temperature and you put the particular temperature like you want to go for 25 or 30 degree centigrade.

So it is now 30. I will remove these things for the ease of understanding. I do not need, I will be calculating myself. So I will delete all those things. So the diagram is also like loss modulus, storage modulus, shear strain and shear stress. The table it will record shear strain, shear stress, storage, loss, tan delta.

So now we will load the sample. We will lift the machine. We can either lift it by pressing it up or if you want to lift it to a particular fixed position we can mention that position and lift it. I prefer lifting it by this method. This much is enough for me. Now I will slowly insert this sample over here.

I will show you the difference. This is parallel plate without any grooving or something. I will slowly put this down and I will give a thickness of 4 mm. I can understand whether the measuring system has touched the sample with the value of sensing force from the transducer.

So it is already touched. So I am stopping the machine. So it is like 6 N. So I will just increase it a bit and try to have a value of around 2 N so that it can just press it. 2.5 is fine for me.

So I will give a test name as test pp25. Press I am not giving right now. We will continue this. We will wait for 1 minute for the recovery. So the experiment has started. Now if you can focus on the measuring system you might not be able to visualize the change in temperature. And because we gave a temperature of 30, so it is better that we close the cabinet and that should be done before the experiment.

So you have to now because the experiment has started, we have to slowly close it. For a particular temperature, if you want to do a experiment in a particular temperature, it is best that you give the temperature you want to do over here and set the value before starting the experiment. You wait for the temperature to come. You put the sample and you start the experiment. That will be easier. From here we can get the linear viscoelastic region, the linear zone as well as the nonlinear zone.

And from this experiment actually we will be calculating the strain and we will be using it for which we call a thixotropic test or the step test which is named over here as 3-ITT. So it is a 3-step test. You can go for 5-7 as you want. So I will show you later. I am not going into details of why Cone and plate or when Cone and plate is preferred over parallel plate or parallel plate is preferred over Cone and plate.

That has been taught by Professor Chattopadhyay. You can also get a stress versus strain curve from the data in the table which we are getting. So the linear zone is over and it is going towards the nonlinear zone. Now if I open the cabinet to show you about the slippage I was talking about, you can see it. You can see that the sample is actually sleeping.

There is slippage in the sample with the increase in strain. So still now it has not, it didn't went to that high strain but still there is a slippage. Also the sample which is out of this particular diameter is not experiencing the shear and is actually leading to erroneous results. You can see the sample has slipped out of the defined area for the experimentation and it will slip out. Then I will close this and wait for the experiment to complete.

So the experiment has completed. So we gave till 100%. So it is completed but you can see that the sample has slipped out. This much has slipped out of the experiment area. Now what we will do is we will leave this and we will change the geometry. We will repeat the experiment with the same sample like with the same composition of the sample, not with the same sample because it has already experienced this strain and the structures, internal structures have already broken.

So we will open this, we will put it back. We will now insert the cone and plate. We will wait for the measuring system to be detected by the machine. Again we have to do a set zero gap because it is a new measuring system.

So zero gap is completed. We will leave the measuring system. Now we have another sample. We will put it inside. In the same way, we will try to have the experiment.

So it is 4, then we will go for 3.75. So we have achieved a similar type of a sensor force. It is a normal force. Now again we will be starting the experiment. This time it is CP.

So the experiment has started. We will wait till the completion of the experiment. Now if you want to compare, so this was for the parallel plate, the black colour and the red colour is for the cone and plate. The modulus has decreased for cone and plate. Reasons you all know. The experiment is complete and here also slippage is there but not that much. So next what we will do is we will change both the upper and lower plates and we will go for the profiled plate.

So we will wait for the measuring system to be registered by the machine. Now we will do a zero gap. We have another sample. So we will put the sample.

We will go to the test. We will start the experiment. We will write it as PP25/P2 and we will continue. So the experiment has started. So for comparison purpose, I will select this tools. So you can see PP25 is the black colour, CP25 is the red colour and PP25 P2 is the grey colour.

So PP is in the same range but CP is down. So this answers the practical demonstration of whatever Prof. Chattopadhyay has taught you all regarding the angular contribution. As I was saying about the sensitivity of the instrument, in case of RPA you probably would have got a similar result. This much difference would not be there. But in case of a rheometer because of sensitive drives, like there is a difference in the sampling, like from the same composite the samples were cut but there is a slight difference.

Always negligible but because of the sensitivity of the instrument you can see the difference. Because of the grooves, the sample is moving with the measuring system or the measuring plates and there is hardly any slippage. So close the chamber. So you can see from the graph that the linear zone is almost same but the difference in the crossover point is there because of the slippage and because of the grip. So this came because of error during the experiment.

So this completes the difference between PP, CP and a profile geometry. Now I am talking about rheometer, rheometer, so something which deals with the rheology and the viscosity but I am not talking about something on the viscosity, so nothing viscosity over here. So we can add viscosity but we can get a complex viscosity because it is in oscillation mode. Now how to get a absolute viscosity? So we are actually going the other way round.

So for that we need to have a rotational test. So I am coming to that. So again we will go to the test type. We will give viscosity. So here is the viscosity curve. Temperature we do not need right now.

So this will give you the viscosity curve from 0 to 100 second inverse. We do not need these things now. This is the rough program which we have. See viscosity versus shear rate and the table also we will get this values. Anyways, so now we will change the mode like it was profile geometry. We will go for plane geometry. So this is the test where actually people test for the various liquids like paints, adhesive, various coatings, specifically their flow curves.

So again we have changed the measuring system. So we have to wait for the reading the measuring system by the instrument and we will do a set zero gap. So after the zero gap is done, we will lift off the measuring system. So in the oscillation you can also like we did amplitude sweep.

You can also go for frequency sweep. You can also go for temperature sweep. And in case of rotation we are going for a viscosity versus shear rate. So it is a shear rate sweep. But you can also go for a temperature sweep at a fixed shear rate.

And we will give it as a 1 mm gap. After this gap comes we have to trim the sample from this side. So sample trimming is very important. Otherwise the contribution will come as I have mentioned earlier.

And then we will give it as a continue. So it has came to 1. And then we will start our experiment. So we will give a name as viscosity versus shear rate. So it is like V versus S and we are doing it in pp 25. So we will continue.

So the experiment has started. You can see the viscosity versus shear rate and this is in the rotation. So you can see that the shaft will be rotating. You can also see a pseudo plastic behaviour.

So test has been completed. We will lift off the measuring system. Make sure we have to clean this. And after this we will be doing the same experiment with a CP geometry. That is this was pp plate and plate parallel plate.

And now we will do it with the cone and plate. Again we will do the set zero gap. So the zero gap has been set. Now we will go for loading the sample. We will lift off the shaft.

Make sure there should be no bubbles. Otherwise the data will be erroneous. Now we will go for the minimum one trim position. Or you can go for 1 mm because last time also we did for 1 mm.

So we will go for 1 mm. Then we can compare the results. So it has reached the trim position. We will just trim the samples off. And we will start the experiment. We will name it as CP. So you can understand a similar type of result decrease in viscosity. Because the contribution of the diameter is taken care of in case of CP geometries.

This completes the experiment where you have a table of shear rate, shear stress, viscosity at a particular temperature. You can do many such experiments in rheometer. One of the best experiment which we find that a step test where you give a linear strain. You monitor it for few seconds or few minutes. Again you give a strain at a higher nonlinear zone and you get a curve from where you can predict about the recovery of the material.

You can also comment on the thixotropy behaviour of the material. I am not doing the experiment but I will just show you experiment which I did. So like this is one of the samples where I did the thixotropy experiment. Now from here you can compare and you can tell that suppose this one. So this particular the first step this is for the low strain that is the LV strain.

Now next for the next 50 seconds it was at a non LV strain, higher strain. Again we came back to the LV strain, again at a higher strain, again came back to the LV strain. Now while we are coming back to the LV strain within how much time it is again coming back to its original modulus, the recovery, the rate of recovery. It is actually we can comment on the about the sealing property of various materials.

The recovery, the rate of recovery can be calculated from this experiment. So this grossly covers whatever we can do in a rheometer. Although many more things can be done like creep test and various other tests. So many tests can be also done like you can make some test like you I have shown in the software itself. There are columns where you can add various conditions, where you can add various intervals like for the step test there are intervals. So in a nutshell I tried to cover maximum of the rheometer what you can do mainly in the rotational and oscillation modes. And one more thing which I should mention that this machine where we were talking about the CTD, controlled temperature device is connected with the liquid nitrogen unit.

It is not directly connected, it is connected with the EVU, it is the evaporation unit and that controls the flow of the liquid nitrogen. Anyways, so this completes this hopefully this gives you a overall depiction about the rheometer and to somewhat a DMA its implications. And rest of the theory portions and other nitty-gritties of this are being covered by Professor Chattopadhyay in his theory classes.