Rheology and Processing of Paints, Plastic and Elastomer based Composites Prof. Santanu Chattopadhyay Rubber Technology Centre Indian Institute of Technology Kharagpur Lecture 23

Practical demonstration on Capillary Rheometer

Welcome back to NPTEL online certification courses on Rheology and Processing of Paints, Plastics and Elastomer-based Composites. Today we will be covering on the Capillary Rheometer. So now we will demonstrate about the capillary rheometer. So why a capillary rheometer is necessary? You all know that in case of parallel plate rheometers or in case of RPA also, the actual scenario which happens in case of injection molding or especially in case of extrusion is not possible. So in order to simulate that kind of a shear rate phenomena in a extrusion, we go for a capillary rheometer. So this machine is mainly having well (hollow cylinder) which contains the sample. one

It has a piston and it also has a load cell. So when I will open this chamber, you can find that the load cell, the piston, all these things you can see. Apart from that a die is being placed at the end of this well and we have kept three dies over here. As per the discussion, theoretical discussion while Prof.

Chattopadhyay has given you all, the L by D ratio plays a vital role. So these three dies have different L by D ratio. So particularly this die which I am having right now is having L40 D1. So if I rotate the die, you can find here is the die head or the die inlet and this much is the 40 mm. So 40, this length is the 40 and this one is the diameter.

Similarly, if I take another die, so this is having 40 and 2. So 40 remains the same and the diameter is 2. So if I keep it side by side, you can understand that the difference in diameter can be easily observed. So this is 1 and the second one is 2. So 40 is to 1, 40 is to 2, another one is 10 is to 1.

So you can see 10, you can see it is a hollow cylinder. It is a hollow cylinder. So this much is the 10, 10 mm and 1 mm is, you can see the die as a 1 mm gap. Now the die which we have fixed over here is 20 is to 1. Now for extrusion purpose, we will use various shear rate and we will also check whether how the profile is coming out.

So the rubber which will be extruded from the capillary will be cut and preserved and we will check the change in the dimension in a optical microscope. We have also online survey of the die swell that is the die diameter is known that is 1 mm or 2 mm. So that is fed into

the computer and we have a laser which detects the immediate swell which is at the down portion of the machine. So this one is the laser and this one is the detector. So if I put my hand, you can find the laser light.

I do not know whether you can see or not. This is the laser light, this is red colour light. So whenever object comes in between the laser light, it measures its diameter and we can get the swell. So that is the instantaneous swell and we will keep the sample. We will wait for 24 hours or 48 hours that is the equilibrium and we will measure the swell using a optical microscope and that is called a equilibrium swell.

So we will start the experiment. So you can see we have a 20 kN load cell. So it senses the pressure of the piston. This is the piston. It goes inside, it fits inside the load cell and we can push this inside the well and this is the plunger or the piston which goes inside and pushes the rubber and the rubber extrudes through the die which I have shown you earlier.

Now we usually take at different temperature, we take using different dies and we go for different types of corrections like the Bragley corrections and all other corrections which has been discussed by Professor chattopadhyay in his theoretical lecture classes. So as you can see that the cavity, the opening of the cavity is very small. So we cannot just insert the rubber into it. So we have cut the rubber into small pieces. As you can see I have made the rubbers ready into small pieces which can be easily fed inside the cavity.

Now make sure before starting any experiment the cavity should be cleaned using suitable solvent. So we will put one by one these samples inside the cavity. So we will put the samples. Initially the sample will slip inside. Then we have to use a plunger to push the samples inside.

So this takes a bit time. Another thing we must take care is usually for capillary we use a master batch sample where or a sample, compound sample where sulphur or the vulcanizing agents are not added. Otherwise in this chamber or the cavity itself the sample might cure. So as you can see that the barrel is full or the cavity is full with the samples but it may so happen that the air is entrapped inside because we have manually fed the sample inside. So now what we have to do is we have to put the plunger.

So we have to put the plunger. We will close the lid. And now we will go towards the software. Now you can see that we have already set a program which will have this following shear rate at a very low shear rate that is 50, then 100, 500, 1000, 2000 and 5000. So this includes the whole range of extrusion which is usually practiced in various industries.

And we have also set the barrel temperature or the cavity temperature at 100 degree centigrade that we have to set before starting of the experiment. And now as I told because we have manually fed the sample inside so we need to pack the sample first in order to get rid of any air cavities or voids inside the cavity. So we will do a piston down at a very slow rate, 120 is fine, mm per minute. So you can see that the piston is going down. This is done to avoid the damage to the load cell because it will slowly enter the cavity, the barrel.

So the piston has entered the barrel and now you can see that the sample will come from beneath. See the sample has started coming. So we will stop the piston and we will take out the samples. We will go for a piston up and we have to again fill up the cavity. So now the machine is ready with the sample and we will start the experiment.

So we will click on start. Then we will click on continue. So you have seen that a preheat time of 300 seconds was given and it started. So preheating is very essential for any rubber compound. So now the experiment will start, the preheating has ended.

So now you can see that we are getting a pressure versus time curve and the material is being extruded at the rate of 50 s⁻¹ which is very less. That is why you might not feel like something is being extruded. If I go for the viscosity graph, you can see it in the inset. Because the rate is very less, that is why the stability time which is given to the software.

So the swell has started. Now we have to click on swell and it will record the swell. You can see that the material is being extruded very slowly. So this portion which is coming out right now is the 50 s^{-1} shear rate extruded profile. So we will cut a piece of the material. As I told that we will be using it for equilibrium swell method.

We will keep it aside. We will show you at the end of the experiment how the profile looks like. You can take multiple sections to validate the die swell statistically. So now it has came to 100 s^{-1} . You can see that the speed of the extrusion has increased. So now it is taking a stability.

It is waiting for the stability. Then the swell button will be activated. See now the swell button has been activated. And we will take a section of the profile.

Now 500 s⁻¹ has been started. You can see that the speed has increased. It is waiting for its stability. Now the swell has started. It has started recording its swell. Again we will take a section of the material of the profile.

Now 1000 s-1 speed has started. Here you can see that how much material is left inside

the barrel. So we must be sure enough to fill the barrel so that with this various shear rate the material does not end otherwise the record the program will not be recording its data. So the swell has started. Now we will click on swell. And again we will take a section of the material.

Now it is 2000 s^{-1} . So now we will click on swell and we will cut a section of the material. It is 5000 s^{-1} . So you can see the speed has increased. Click on swell.

We will take a section of the same. Take multiple sections as the speed is high. And this completes the experiment. So the experiment has been completed. You can see the viscosity versus shear rate curve over here which is actually the curve which is usually seen in case of polymeric samples.

So here is the data. And we have all the details of the material, the shear rate versus viscosity of the sample. And here you can find that we have also the swell ratio across the various shear rate. So in the particular shear rate we have the swell ratios. So here is the shear rate, here is the shear rate and here is the swell ratio. So you can see clearly these two different graphs.

And also we can have the viscosity versus shear rate curve using these two columns. So using different temperature and different dye you can do various corrections. So now it is time to see the profile which we have cut while doing the experiment. As you have seen while doing the experiment I have cut the samples. So as I have mentioned earlier we will use these samples, we will keep the samples aside for maybe 48 hours and then we will go for a microscopic study to study about the die swell and also the surface finish of the extruded profiles.