

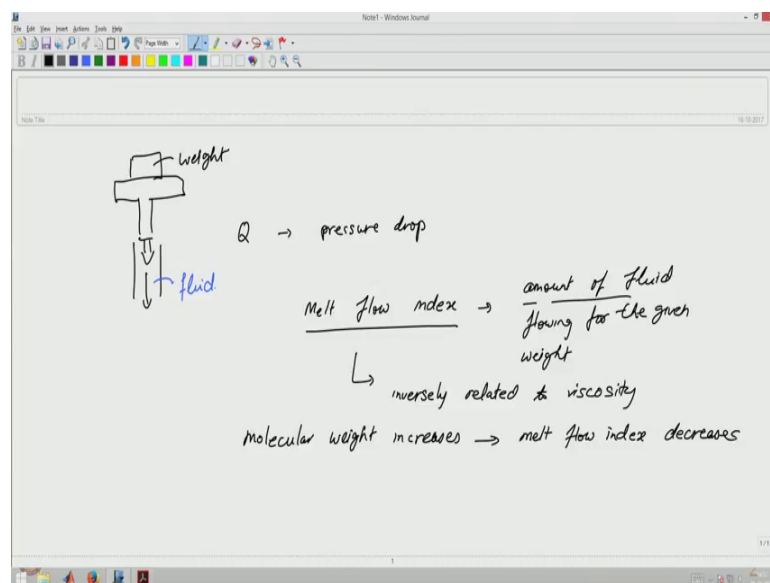
Rheology of Complex Materials
Dr. Abhijit P Deshpande
Department of Chemical Engineering
Indian Institute of Technology, Madras

Lecture – 23
Rheometers

These segments of lectures we will look at are rheometers, and how there are different principles based on which the rheometers are operated, and how different types of arrangements are there to get the rheological properties. And so, to begin with we will first also quickly look at some of the trade tests by trade test, we mean something which has been used for years in industry trade test more often than not are very complicated from the point of view of the type of flow they are imposing.

So, for example, in class we have already discussed that are the extensional flow, or shear flow, and we have a very controlled condition, but in case of trade tests they are far more optimized to work for one particular application, and one particular material they are usually far more complicated, but they give a number which is which has been found useful in that particular industry. For example melt flow indexer is nothing, but are again it is a flow through capillary. So, if we have a capillary.

(Refer Slide Time: 01:31)



And through which the material has to flow we know that the amount of material will depend on the pressure drop, and this is true whether it is a Newtonian fluid or a non Newtonian fluid. So, in case of the melt flow index what is done is there is device let us say a piston kind of an arrangement which is kept which will push the fluid. So, the fluid is kept in the capillary, and then it is pressed using some weight here. So, there is a weight placed.

So, you can see that this because of this weight there will be a pressure imposed on the fluid, and then we can measure the flow rate. So, this kind of a device therefore, it is a flow situation which we are quite familiar with in terms of poiseuille flow, but we do not really take into account what may be the velocity profile, how is the overall viscosity of the fluid, we only in the end get what is called a melt flow index. And this basically keeps track of the amount of amount of fluid flowing for the given weight.

So, you can see that in which way this is a very in some sense a crude test right, in the sense we are not measuring any finer aspects of flow, we are not measuring really pressure we are just employing a weight to push the fluid down, we are measuring how much fluid is being flown. And of course, the amount of fluid will be inversely proportional to viscosity. So, in general melt flow index will be inversely related to viscosity. And so, if you were to buy for example, plastic materials for making bags or CDs or buckets and so on, a many of the objects where molding is used quite often and the specification would come in terms of a melt flow index, and then there will be guidelines that first this melt flow index use these conditions and so, on.

So, it is a very specific to plastic processing and it is not really generic to any other material and of course, we know that melt flow index will increase with increasing temperature, because viscosity will decrease with increasing temperature, other way also a as a function of molecular weight, when molecular weight increases what would happen to melt flow index.

Student: decrease.

Melt flow index would.

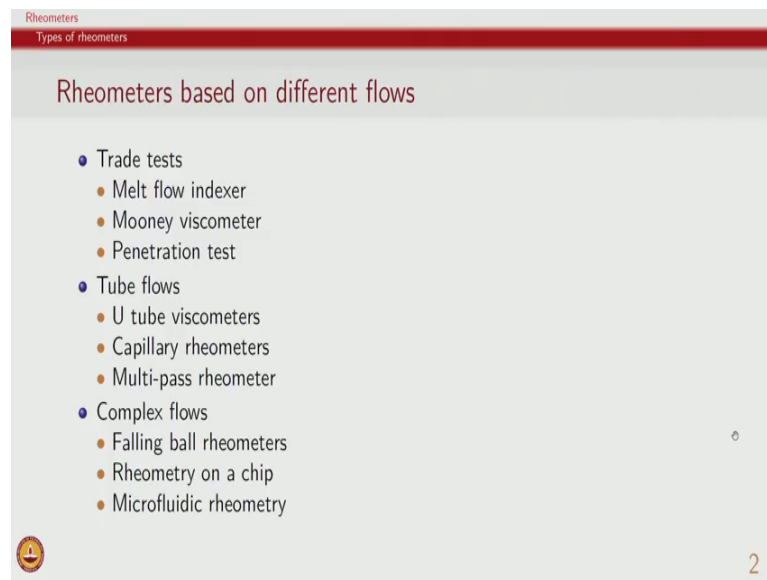
Student: decrease.

Decrease.

Student: decrease.

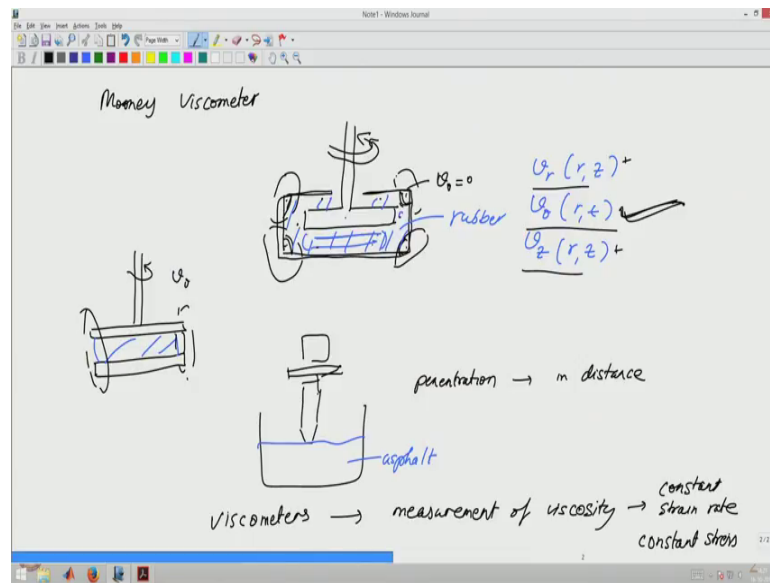
Because we know that viscosity generally increases, because of increase in entanglements and so on. So therefore, melt flow index will decrease. So, people who are in the trade, people who are knowing about this particular application will be having feeling about what kind of numbers are good for what kind of conditions, what kind of applications. So, that is why this is an example of a trade test.

(Refer Slide Time: 04:56)



Similarly, Mooney viscometer is used for rubber. It is again a very specific test which is only for testing the properties of rubber.

(Refer Slide Time: 05:09)



And if you look at Mooney viscometers geometry it is a shaft with rotating member which is a disc, and then in that there is a chamber. So, there is a chamber in which the fluid is kept. So, the in rubber in this case will be kept in this gap, and then the shaft is rotated ok.

So, it is somewhat similar to a parallel plate device that we have talked about, but compared to parallel plate device the flow in this is far more complicated, because in fact, flow is there everywhere. All the rubber everywhere is flowing, and so somewhere there is an annular region where there is flow there is between this flow here is only where which is about flow between parallel plates, but otherwise there is everywhere else also flow.

So, to if you were to analyze this problem using the fluid mechanics the way we have done for certain simple geometries, in this case we will have to say that V_r will be present, because in this case the flow will be there, V_θ will be there, and V_z may also be there. And in fact, all of them will also be function of r and z , r and z and r and z , even if we assume that theta dependence is not there. So, therefore, this kind of a flow is far more complicated to simulate and analysis of this will again be very difficult for us to get any material behavior.

On the other hand based on the rotation rate and the torque that is required to rotate we can get a number, and that number again which is Mooney viscosity is useful depending

on the torque which is required and the rotation rate, we can calculate what is called a Mooney viscosity, which is applicable only again to rubbers in this situation. And again people who are in rubber industry we will know how to use it yeah.

Student: sir

Yeah.

Student: theta dependency (Refer Time: 07:23) .

Theta dependence yeah generally because this is a problem in which fluid will be flowing in theta direction right the rotation is there of the shaft. So, generally fluid will be moving in theta direction, and generally I mean most dominant component is going to be theta right, because that is what has been imposed, but because there are walls which are stationary and there are corners here, where we are not really maintaining it is possibility that V_z and V_r will also be there, in the parallel plate what we do is in the parallel plate case we strictly have only a parallel plate right, and it is a thin gap and then we even, we put a fluid here, we are saying that there will only be V_θ in this case. And In fact, we are assuming that at the corners and all wherever there may be possibilities of additional flows their contribution is very negligible.

But in this case by design itself we put in a chamber in which fluid is filled in which this disc will go, and then rotate V_r and V_θ will only be there in the corners, because basically there can be secondary flow patterns and so on. It is not a possible that there will be sharp corners here and this we have not put a stirrer right, in the end the design of this member is to rotate at a certain velocity and it may not ensure that there is a velocity uniform everywhere, which is certainly going to be the case because V_θ will be 0 all over the wall. And since V_θ is 0 there is a possibility of secondary vortices the only difference between this and the fact that in this case the end effects, how significant are they and in this case, how significant are end effects right.

So, that is the other thing which is different in this case, and this case there are there is an annular region where there is flow there is upper region where there is flow there is a lower region where there is flow.

Student: So, this means within the plate it is still would be eighty percent.

Yeah everywhere it will be v_{θ} which will be the more dominant yeah, but given that it is a closed chamber it is there is a far more possibility of V_r and V_{θ} just being present all around the corners. The other example which is there of a trade test is a penetration test, which is used for asphalt and it is again asphalt or bitumen it is similar in which case there will be a penetrating needle, and then this is again with certain weight or a certain load which is given on it, and asphalt is kept and given under the action of whatever load, and whatever the penetrating member how much is the penetration happens in length.

So, how much is the penetration is measured, and penetration high means that the fluid is softer or it is more it has less viscosity. If penetration is very less then it is a very stiff material so, therefore, penetration in distance is a measure of the overall properties of the material. Now in each of these cases because such trade tests have been done, and they have been used in case of a specific application the numbers which are generated by these tests form design guidelines.

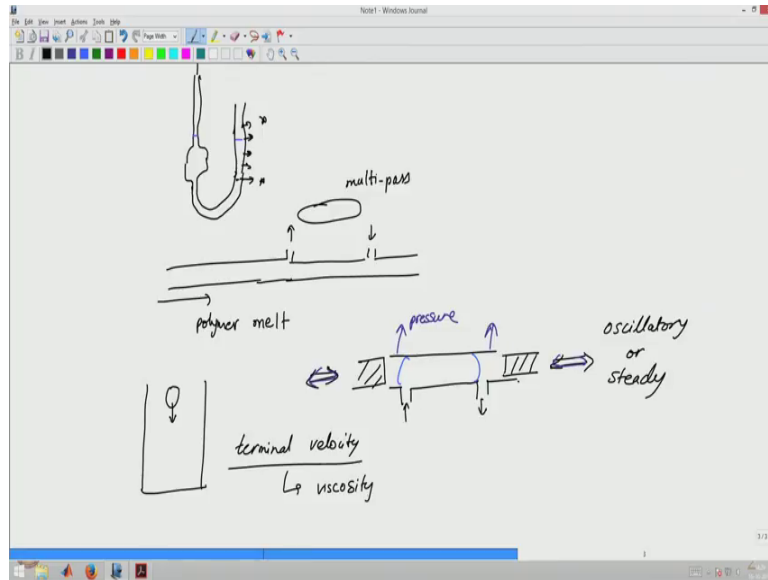
For example in case of asphalt unders in Chennai one kind of penetration index it will be useful, in some other location other penetration index may be useful. Of course, many of these trade tests have also become not so, useful in current scenario because our designs are far more precise, and we want to push the envelope and not have factor of safety very large. So, therefore, in addition to taking tests of these kind of ah measurements we also actually will use a rheometer and try to measure few other properties, and then incorporate then them in our design as opposed to just maintaining these.

So, in case of asphalt for example, or case of polymer melt it is quite often possible to actually use a full fledged rheometer, and then measure rheological properties, and then incorporate them in design. So this is first set of tests which indirectly measure rheology of materials the second broad category is of that of tube flows. So, the capillary flow or flow through pipe or flow through tube is used as a standard principle in terms of measurement, just as a quick note about viscometer versus rheometers. So, viscometer is just measurement of viscosity. So, which means it is a basically steady shear property. So that implies we are looking at constant strain rate and constant stress.

So, only these properties are usually measured using a viscometer, and generally whenever we say rheometer we have seen that there are number of ways in a which

rheometer can be controlled number of parameters can be controlled in as a function of time or they can be kept constant and different types of material functions can be measured. So, YouTube viscometer is a simple device in which case we take the fluid and there are reservoirs and chambers also.

(Refer Slide Time: 13:28)



So, there may be for example, reservoir and then a fluid like the, and then what we can do is we can pull we if we fill fluid in this it will occupy let us say certain level.

Now, what we can do is we can pull suction apply suction, and then what is usually done is let us say on this there will be marks. So, from one point to the other point how much time does the fluid take to travel, so what we can do is we can let us say apply suction so that this fluid level goes up, and then release the suction. So, that fluid will start again flowing back, and we can note the time required to go from here to here.

So, this is a measure of viscosity, and if you want to make precise measurements near let us say where viscosity of water and oil then, these are the best instruments. Because many of the rheometers are designed in general to measure properties of pastes and asphalt and many other solid like materials also. So, in general they are talk sensitivities can be a problem when you are trying to measure viscosities of very dilute solutions of water.

So, in that case Ubbelohde viscometer this is one example of this YouTube viscometers called a Ubbelohde viscometer. So, that is also very useful, and so these measurements can be very precise though the method appears to be crude compared to other as far as one is only interested in viscosity, then this can serve its purpose and if viscosity is very low this is far more accurate compared to many other rheometers, because the strength of rheometers is in terms of the wide ranging properties they can measure by controlling different conditions, and most of the fluids where we are interested in viscoelastic response happen to have nominal viscosity which is very high. It is at least 100 times more than water. So, therefore, in that case stock sensitivities are not very low. So, therefore, it is not easy always to measure viscosity of water and dilute solutions using the rheometers.

So, in YouTube viscometer again one can measure non Newtonian fluid flow also and, but it. So, happens that many of the time since we are measuring very dilute systems the shear thinning or shear thickening nature is very minimal. So therefore, but one should usually say if you expect the fluid to be shear thinning or shear thickening then we should say that we are measuring 0 shear viscosity, because the flow rates which are involved are small and the Reynolds number is of course, always small in case of YouTube flow. So, that flow is laminar region and also we are doing measurements under conditions of shear rate which are reasonably low.

And multi pass rheometer on the other hand is a modern instrument which came about in 19s which was associated with again a specific requirement of measurement of polymer melt property in line, there is an extrusion line or there is any molding line where polymer melt is flowing. So, if polymer melt is flowing through some die or some device is it possible for us to tap into it takes some amount of melt out pass it through some other device, and then measure viscosity. So, it is for in line measurement online measurement of viscosity.

So, what is done in this case is so it is possible to again put the fluid back. So, that it is basically an online device. So, the multi pass rheometer will be somewhere included in this and multi pass device is nothing, but again a capillary and in that there will be piston, and it is possible to have multiple pistons and this piston can move up and down and so, depending on so the fluid can come in fluid can go out. So, the polymer melt can be and then what you can do is you can measure pressure at different points. So, you can

say you can measure pressure at multiple points and therefore, you can get an idea about what's happening what the melt flow is.

All of these are related to their all shear measurements because in all the tubular flows the predominant mode of deformation is shear, in therefore and most often we have seen also so far whatever we have discussed we are discussing steady properties. Generally in Mooney rheometer, Mooney viscometer you can wait for the torque value to remain become constant, and then you can measure. In this case it is possible to do a viscoelastic measurement because the 2 pistons can also do an oscillatory motion. So, therefore, it is possible to do some oscillatory or steady motion of pistons.

So, in this case it is possible to do actually a viscoelastic measurement. So, that is these are all examples of what are called paisley flow, in which case a pressure is imposed on the material and material flows in predominantly shear mode inside a tube. Most often all of these the tube is a circular cross section there are some examples of complex flows which can be used as a measurement of rheological properties. And the first example here is falling ball rheometer which since we know the solution to stokes law we can measure basically so, since we know using stokes law what is the terminal settling velocity of a marble or any other spherical object, we can measure this terminal settling velocity and then related to viscosity. And again if let us say the terminal velocity is very small and if ah shear rate or the overall strain rate which is there in the fluid is very low then we can measure the 0 shear viscosity.

But if let us say the particle velocities are little larger we have 2 issues one is in terms of the applicability of stokes law because, if the Reynolds number is high the stokes law will not be applicable secondly, if strain rates are large and if a fluid is non Newtonian then again stokes solution is only for a Newtonian fluid. So, therefore, many times it may not be easy to analyze and actually get the material properties if you want to do this kind of an experiment.

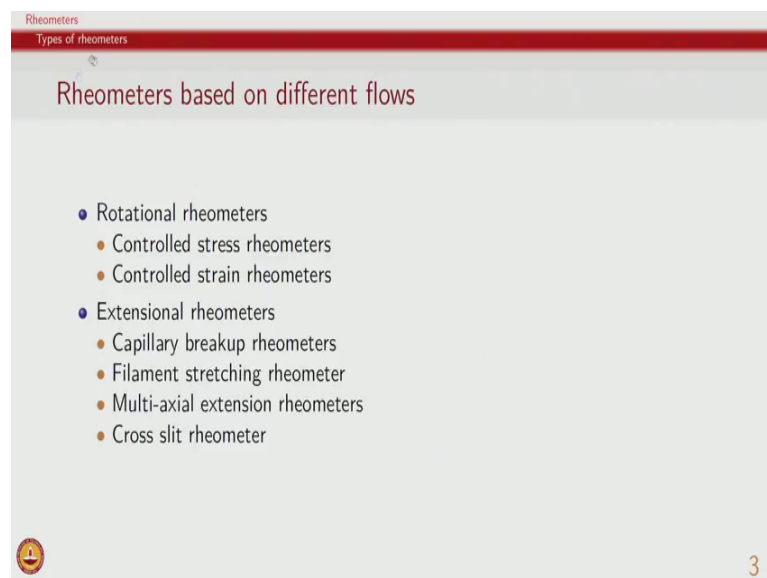
For a Newtonian fluid and for terminal velocities which are small this will work very well, and of late in the last 10 years or so, people are coming up with ideas that we can use flow in a in a chip, we can we can have in fact electro wetting of a fluid I can apply a small electric field and then try to manipulate a drop of fluid, and based on how it responds to electric field I can try to back calculate the rheological properties of that

material. So, those kind of things therefore rheometry on a chip very small droplets can be manipulated electric fields can be applied and so on. So, that is what is meant by rheometry on a chip.

In all of these cases we will have to apply a flow which is necessarily not viscometric or rheometric flows, then what we have discussed in the course so far, but it is a more complicated flow in which case surface tension may be involved interaction between the solid and fluid may be involved, electric field response may be there, but at least in some specific example cases we may be able to back calculate viscosity or other rheological properties.

Similarly, microfluidic rheometry is since we are researching where a variety of microfluidic devices can be used the flow in a microfluidic channel to back calculate the rheological properties. So, these are all the rheometry on a chip as well as microfluidic rheometry, would still I think belong to the class of research flow problems though there have been, claims in the literature in the last 10 years or so, that such and microfluidic device can be used effectively or using this kind of a geometry on a chip we can measure properties, but they still remain in the realm of concepts and proof of concepts, so the next set of rheometers are really the bulk of the rheometers that are used.

(Refer Slide Time: 22:53)

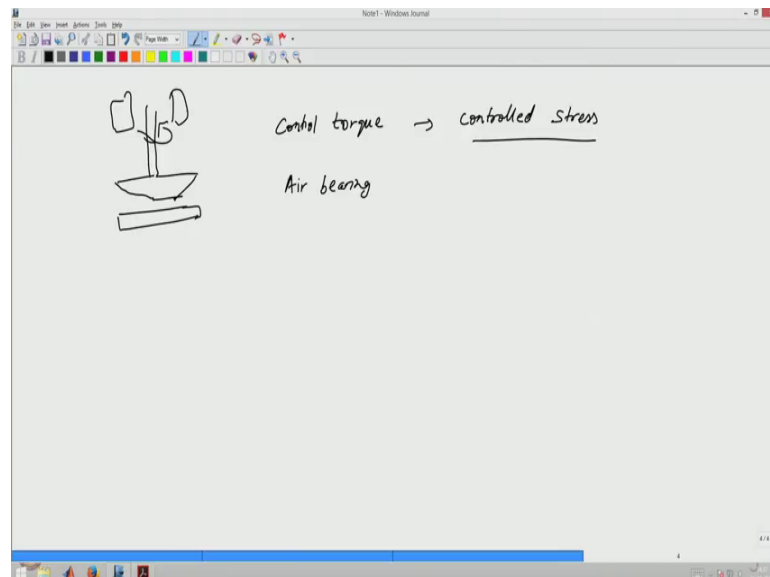


In that also the rotational rheometers really are the rheometers so, in general when we say rheology more often than not it is the measurement using rotational rheometers. The

ease with which rotational rheometry can be done the reliability of the results that you get from rotational rheometry have sort of made them very widely acceptable for research as well as industrial applications. So, generally when we say rheology it is rotational rheology and therefore, it is not surprising that in this course many of the examples of flow that we have discussed all belong to this class of rheometers.

In this there are two different types of rheometers which are possible controlled stress rheometer, and control strain rheometer, there is also the main problem associated with a rheometer instrument design is the fact that we are applying some torque or some force.

(Refer Slide Time: 23:48)



So let us say the cone which we are which is being used to rotate, and the plate which is there. This cone is being rotated at a certain rate and the motor has to be used to do this rotation, and whatever is happening to the cone has to be in a precise manner. So, the motor should be able to apply the torque, because motor will require certain torque for it is own rotation. And then it requires torque over and above that for this rotation, there is inertia of the motor itself you will have plate cone has to be rotated back and forth.

Now, motor also therefore, has to go back and forth. And if there is inertia then what happens is the motor will go and by the time the reverse direction goes, motor will would have gone little bit further then when we actually stopped gave the instruction for motor to stop and that is what is called inertia. So, in all of these cases the inertia of a motor is a very important characteristic. Also whether the torque that is being given to motor how

much does it require motor itself, and how much of it is being given to the cone itself, because it is the cone which is transferring the torque to the sample. So the sample torque is related to what is being given to cone, but generally for the motor and the whole assembly to rotate itself another torque will be required.

So, in general we have this crucial aspect of how to do management of rotation of the motors. And so one could have two different modes of doing this control it is one way let us say if we apply certain current to the instrument the motor, then maybe reply generate a certain torque. So, then this will be called a controlled stress instrument, because by controlling the voltage or current signal to the motor we can control the amount of torque that is being generated. Of course, we will still need to make sure that we know how much of that torque is actually going to the geometry. So, that calibration will have to be done.

But so, in general when you control the torque basically what you are doing is controlled stress, and then since you are now generating this torque and you would want basically the in all of these cases there will be stationary parts and mobile parts. So, there has to be bearing between the stationary mobile part there has to be frictionless movement, because the overall geometry will be housed in a stationary. So, there will be a stationary set of overall instrument control, and then this when you want to rotate this cone this cone should rotate with as less friction as possible.

So, the manipulation of again friction in case of rheometers is very crucial, because lot of the torque otherwise will go in overcoming friction. And more importantly this torque may depend on the rotation rate right. So, therefore, there will be many uncertainties associated with managing friction in such rotating geometries.

So, in that case generally what is done is air bearings are used, which means air is used as a fluid, which is using being used for lubrication instead of ball bearing or other such bearings. And so pressurized air is a must for many of the rheometer operations and so, with these rheometers you can directly control the torque therefore, if you want to operate them in control strain mode you will actually have to use a feedback loop. So, this and other aspects we will see in the next segment of the lecture.