

Rheology of Complex Materials
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Lecture - 24
Rheometers

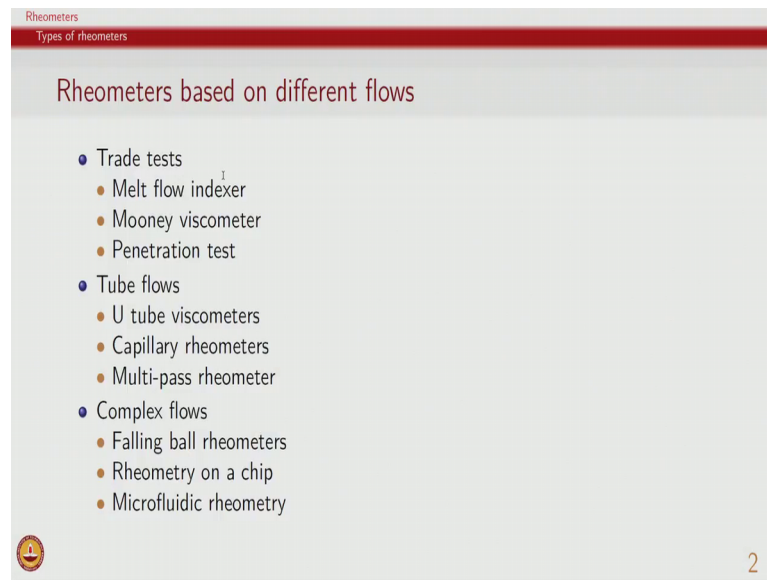
In this segment of lectures we have discussing Rheometers and different types of Rheometers which are used for analyzing the rheological properties. And we initially spend some time discussing the trade tests which are practice oriented tests which are designed to get us some numbers.

That design can be carried out; however, the flow is not controlled to the in the sense that there is no one type of flow shear or shear free flow also the deformation may not be constant deformation rate may not be constant stress may not be constant. Therefore, and the geometry which is chosen is also in some way it mimics the engineering application, but the flow that is that happens is actually very complicated. We can get a measure of material behaviour in terms of a number, but we cannot really call it a material property.

Because if I change the geometry slightly I will get a different number and therefore, it is not really a material characterization tool from the point of view of material properties.

Therefore, we look at in rheology those type of flows which can lead to controlled conditions and then the material properties themselves.

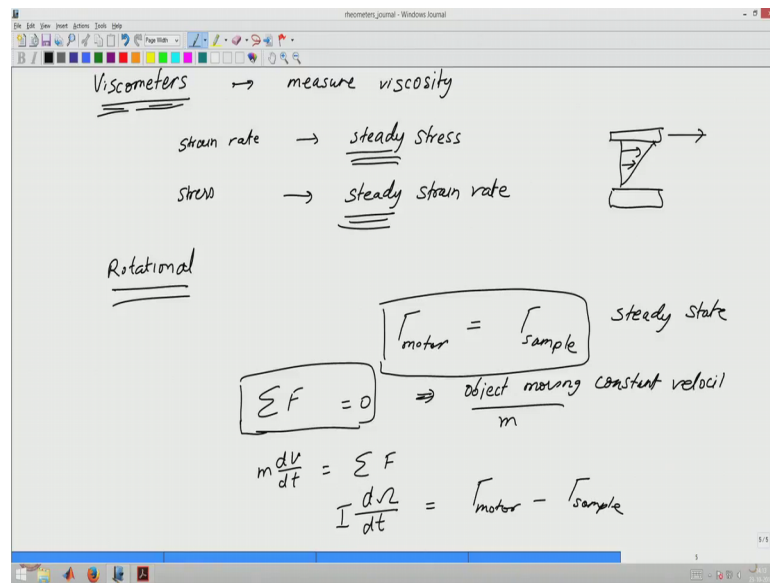
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Of course we have seen that we rather than measuring material constants directly like viscosity, we end up measuring material functions and, but these material functions are independent of the geometries in which the measurement was made or the type of flow which was used and all that. That once we get the material function using that we can try to predict the material behaviour in all types of flows under all types of conditions. That is the hope of doing rheology that I can do some selected experiments using that I characterize.

Some material functions and using these material functions, I will be able to predict material behaviour in any general situation. The trade tests and more complicated flows are generally more difficult to get material behaviour from and. We were discussing the 2 dominant type of rheometers which are controlled stress and control rate rheometers

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In this respect one important thing we can just try to make sure we understand the difference between viscometer and rheometer because there are many instruments which would measure viscosity alone and we know in the course that viscosity as a material function is defined only under steady conditions. We apply a constant strain rates and measure the steady stress and this of course, is the ratio of these 2 is the measure of viscosity we alternately also saw that we could do creep in which case.

We apply a stress and we wait for steady strain rate and both of these in one is control where you are actually controlling the position or the rate at which position changes, and the other one is you are controlling directly the force which is being applied, but in both cases we wait for the steady state.

Viscometers generally are good instruments from the point of view of steady operation only. Generally whenever you say we have a good viscometer, it will only do steady state operation and therefore, by definition it cannot do rheological measurements because in theology we apply step strains, we look at things changing as a function of time, we apply sinusoidal or oscillatory stress and strain and then we again look at time changing quantities.

Therefore, viscometers as instruments the way the motor is chosen the way the properties of motors is chosen is completely different. And therefore, let us just look at what happens when let us say you have a motor and you have a sample. Generally let us say

we are looking at if let us say rotational type of rheometer, we are applying stress or strain in a rotational motion. Generally what we have is the motor which is going to apply some torque.

So, let us say this is a torque which is applied by the motor. Let us say this γ capital γ motor is the torque of the motor now in steady state what will happen is, whatever the talk that the motor is generating is will be the torque which the sample will also feel right or the sample will.

Whenever we have let us said a solid plate and a fluid which is moving next to it and we have this we will say that at this point whatever is the force which is imposed by the solid on the fluid will be the same force fluid imposing on solid right it is just a continuity of a force and. Similarly here also whatever is the torque that is applied by the motor on the sample under steady condition whatever is a torque by the sample applied on the motor will be the same.

Let us say if there is a sample torque then these 2 will be same for steady state. The motor is generating a torque and the sample is experiencing that torque or sample is in turn imposing that torque on the material on the solid which is being used to move rotate. This is the same thing like saying that if I have force balance right. If I have a force balance then under steady conditions if velocity of the object that is moving is constant then what we have is the overall sum of forces which are acting on it will go to 0 right.

The sum of forces going to 0 implies that the object is either stationary or moving with constant velocity. This statement which is let us say for any arbitrary motion for rotating motion, it is the this corresponds to this. That at steady condition when the members are rotating at a constant rate the 2 torques will be the same now what happens if it is not a steady state to the force balance. Let us say this object has a mass of m , then we can say that it is $m \frac{dv}{dt}$ where the v is the velocity is equal to the sum of forces right and steady state of course, velocity goes to 0.

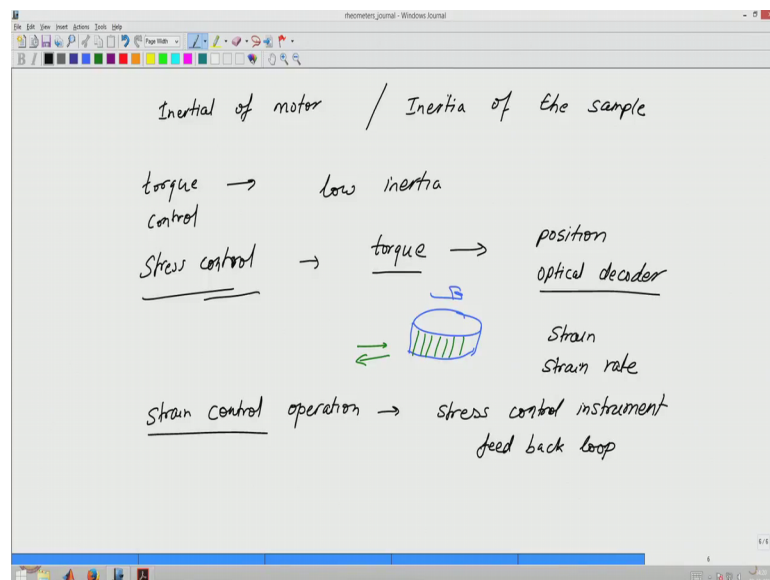
Now similarly for a motor also we will have a inertia of the motor and then great of change of the how the rotational velocity changes. Ω is the rotation rate and that will be the sum of torque. It will be this minus let us say this sample right. If you are looking at an unsteady situation and in theology we are always looking at unsteady situation it becomes very important as to how does motor with it is inertia respond in

case of a viscometer you never have to really worry about the motor inertia because you will always wait for steady state and both stress and strain rate have become constant. Motor inertia is really irrelevant you can have 2 different motor inertias.

As long as you reach the same stress same material will give you same strain rate. It is not really relevant, but let us say if you have 2 different motors with different inertias and then how you are interested in how the torque changes as a function of time and then it becomes important to have a strong consideration for what is the inertia of the motor.

Now, what could we say should be the inertia of the motor ideally should it be a low value or should it be high value and why should it be low or why should it be high.

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Let us say inertia of motor and of course, we will also have inertia of the sample inertia of the sample unfortunately we cannot do anything about in the sense that that is the material we are going to measure. We cannot say that let us sort of let us change the inertia because it is the sample and it will have a certain density and mass and therefore, we will have a certain inertia associated with it, but from a motor point of view is it possible for us to state something what does the inertia really do.

Tendency to continue right that is what we know. Therefore, if we and if we are doing an oscillatory motion and if inertia is very high then what will happen is even though we think the motor is going to stop and turn it may actually go a little bit further and then

turn and the. Therefore, the we think we are applying is different compared to what they actually motor is doing

Therefore, the inertial contribution to the overall motion can be very significant and ideally then if inertia is small then what happens is these differences between what is being applied and what the motor does will be smaller. Therefore, for 1 set of motors which are being controlled with the torque mode inertia is required to be low now. If you control the torque directly, what you do is you generate you supply current or voltage to the motor and generate a torque and the motor will generate the torque it has to rotate at certain rate, but to reach the steady state or to reach some given value it will based on inertia.

As if it is low inertia then it will quickly come to whatever is the prescribed motion that we are describing. Therefore, in torque control low inertia motors are required even if you have low inertia there will be some finite inertia and therefore, there is a need to actually take into account what is the motor response itself.

Most of commercial rheometer instruments today on the firmware will include information about how does motor respond under all kinds of conditions and so. In fact, the inertial contributions of motor will have to be subtracted to give us the rate of change of torque which is the fluid sample torque itself or the rate of change of position which is only due to sample. Therefore, contribution due to motor inertia has to be subtracted and lower the motor the less correction you will have to be made if your motor inertia is higher then your correction better be very good and so. In fact, both types of rheometers are there are rheometers which inherently have a motor with very low inertia and therefore, they will say that we are doing a very good stress control then there will be other sets of instruments.

Which say that this motor inertia is higher, but we have a very good firmware which knows exactly how to subtract the motor inertial contributions and this is are basically what are called stress control instruments. In these kinds of cases our torque is applied and position is measured and generally position is measured these days by using an optical decoder optical decoder which is basically a principle that you know if I have a rotating member. If I let us say just if I make some disk let us say which is rotating and on this disk if I put some gradations and when this rotation happens the gradation will

also move and if I now with some laser and detector I can try to detect as to how fast this object is moving by counting.

Because if I put a laser and then reflection back I can then find out how fast this object is moving. Therefore, these kind of optical decoders or what are used. You can have apply a torque and let the motor move and then using an optical decoder you can try to measure as to how fast or slow the motion is.

When you are doing torque control you apply a torque you measure the position and therefore, you can measure strain as a function of time or a strain rate as a function of time or whatever frequency, whichever you can do the measurements of strain and strain rate now we saw that there are many types of experiments in which we want to control the strain itself for example, stress relaxation we apply a step strain or in case of oscillatory shear we would like to apply let us say oscillatory strain instead of oscillatory stress, then we need actually a strain control.

In a stress control instrument to get strain control is not possible. What has to be done is a feedback. You apply a torque you measure the position and see whether it is what you want because you by the way you are doing this in an unknown sample and if whatever the strain is the.

You take few quick measurements and then see what is the strain or strain rate going to be and then quickly correct the torque value that is being applied. Therefore, the stress control operation in a stress controlled instrument is being done through a feedback loop and again the effectiveness of feedback loop and everything will be more effective if you know exactly your motor inertia and what it is doing because then the feedback loop will be far more effective because you will be able to.

Quickly change torque going. That you know how motor is going to move because if it has large inertia then you will need to predict as to how much the motor is going to move therefore, the feedback is an inherent component of strain control operation if you are using a stress control instrument yeah.

That how much stress and how much strain or strain rate is required will vary from material to material and quite often most of these instruments will come with a lower limit and upper limit of any of these quantities or the lowest torque that they can measure

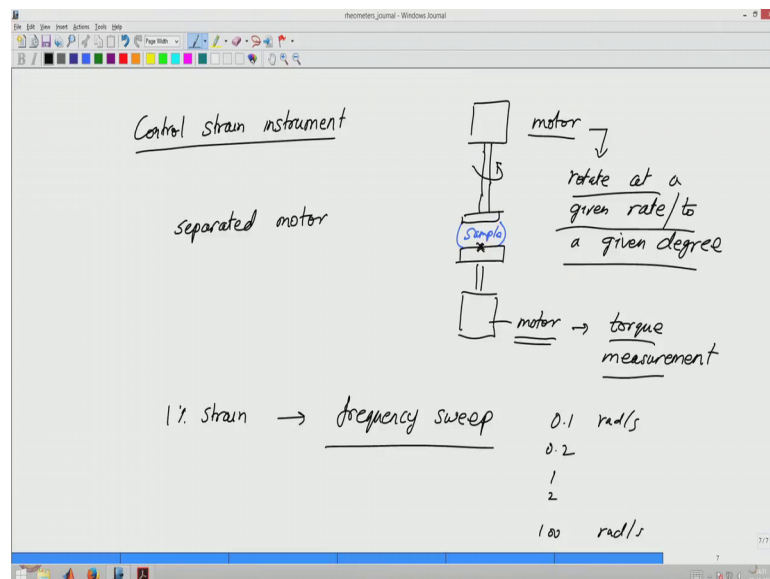
or lowest strain they can measure and generally what we do is we start with a reasonable range. For example, in when we do oscillatory shear most often we say that you know let us start with point one frequency to maybe about 100 radians per second as a frequency range when we do let us say steady shear we again say that.

Let us start from 0.1 to about 100 if it is in terms of stress then it depends on how soft or hard the sample is for example, shampoos and all we may just have about 100 or thousand Pascal as the loads, but if it is let us say asphalt then it may even go to 10 lakh or those kind of values of. Megapascal range of stresses, generally depends on what sample we have and these are those stress control instruments and.

In fact, the viscometers can be anything stress or strain control it does not really matter because in the end you only wait for steady state, but given that in rheology we are necessarily interested in time dependent properties it matters.

Whether you have a stress control instrument or a strain control instrument because we need to know what is being done by the instrument because we are going to give a time dependent parameter for the material to experience the other type of rheometries.

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Which are there are control strain rheometers and they also are rheometers where there are actually 2 motors used quite often in what is called a control strain instrument in this case what you can do is. In fact, you use the one motor to in a and this type of motor

when you supply a particular current it will rotate at a particular rate therefore, it is a strain controlled measurement.

You apply user motor which can rotate at a given rate or to a degree to a given degree and once we apply the torque for example, let us say if I want to rotate it by 10 degree. I know how much torque to give. Therefore, now you need to also measure sorry how much current to give. Then it will rotate by 10 degrees, but now I also need to know how much torque is being applied and what is done is. Therefore, this let us say is connected and the sample is in between. This is the sample between 2 plates what is done is the other plate you attach another motor and what happens is since this motor is rotating this will cause.

The sample also to rotate and the overall stress torque will be generated in the fluid and even the bottom surface of the fluid will impose a stress on the bottom plate even though the bottom plate is stationary what will happen is the fluid will impose a force. It what you can do is you can force this motor to remain stationary and if you force the motor to retain stationary then you can try to measure the amount of torque that is required to keep it stationary. That way now your rotation of N strain control is independent of the torque measurement. The bottom motor is being used for torque measurement. In this case therefore, you are applying a strain through a motor and then you are measuring the torque in the other case.

We were doing exactly opposite we were applying a torque and using optical decoder we are trying to measure the strain. Therefore, this is for example, twin motor or a separated motor technology. Those are the types of terms which are used to describe such measurements now why do you think it is important to have control strain or control stress measurements and why is it.

Important for us to know about such fine details about the instrument why would it be of interest I have an unknown sample for I am doing theology and this unknown sample I put it in the rheometer do I really need to care that oh is the rheometer stress control or is the rheometer strain control should I care and should I do my analysis accordingly or it does not matter because anyway in the software I will give that you know you apply this much strain or you apply this much stress and I will get the results while looking at that analysis of the result.

Do I really need to worry about the fact that oh maybe it is a stress control or a strain controlled instrument under what situation do you think it might be useful to know this difference now the first instrument where I apply stress control what I can do is I can use a feedback loop right that is what I mentioned in the previous thing. if I let us say I have to use a stress controlled instrument in a strain control operation then what I do is I apply a stress see what is the strain rate generated.

Then modify the stress value that torque and that I get a requisite strain rate make the measurement go to the next stress value and see if I get the next strain rate which is my target strain rate so, but it will be done through a feedback loop similarly in this case if I were to do a creep experiment I cannot do a creep experiment directly I will apply I will.

Instruct the top motor to rotate at certain rate or given to a given degree and then I will measure how much is a torque and if the torque is less or more then I quickly need to do a feedback and I will need to modify the rotation. Is it important for me to know whether the 2 instruments are different?

The one easy thing to realize is if I am doing let us say a stress relaxation experiment it better be done on a strain control device because if I am doing strain control it is better to do it because there is no.

Feedback loop involved because otherwise the control loop determines how effectively the strain is being applied. If you are doing let us say a constant strain or a constant stress X. If you are doing a creep experiment it is much better to do it on a stress control right of course, though we will rarely have enough luxury to have all the instruments at our disposal right. It may happen that the lab in which we are working has only a stress control rheometer, but we are also wanted to do a stress relaxation experiment.

In that case we should know under what conditions do I expect the instrument to not perform as well as a strain control experiment or if I change go from one material to the other are there do I expect under some conditions the motor inertia to play a much bigger role. These are the questions which are important and.

Therefore we must know whether we are using a strain control instrument or a stress control instrument for example, if I have a very low viscosity fluid right if I have a fluid let us say which is our polymer solution a dilute polymer or semi dilute solution which

has very low viscosity or I have a micelle solution of surfactant again with a low viscosity remember low viscosity implies the inertial contributions are little more significant sample inertia itself.

Now, if I am trying to use a stress control with a feedback stress control instrument with a feedback given that the inertial forces are important the feedback may not be able to correctly predict what might be the role of sample inertia. If I am doing oscillatory shear and if I am doing what in oscillatory shear quite often what I do is I will.

Specify a constant strain amplitude I will say let us say I am using one percent strain and then I usually do a frequency sweep assuming that this is in the linear regime I will do a frequency sweep and what frequency sweep will imply is initial measurement I will do let us say at point 1 radian per second then 0.2 radian per second and on and then 1 2 and on up to maybe hundred radians per second.

As I go to higher and higher frequency the inertial contribution start to play a bigger and bigger role because the sample is sort of doing now applying the strain at very high rates and if there is inertia of the fluid important it may not may be doing something independent compared to what I am assuming the sample is doing .

Because generally when we are applying the strain we assume that whatever is the top plate doing the rest of the sample is completely conforming to that, but if inertia is important then even though the plate has reached that right the one end fluid may still continue to move to the right the plate has already started moving to the left. The fluid inertia is actually going to give very different the contributions compared to what we are assuming in our analysis.

In all of these cases by the way we can always take into account the fluid inertia also and then solve a problem, but it is far more complicated problem to solve. That is why when we do rheumatic analysis we will say that we will be in those regimes of operation where fluid inertia is negligible. That our analysis becomes easy we can do the characterization easily.

Ha exactly then what you say is if I have a low viscosity sample and I am using a stress control instrument then I better be careful about my fifty radians per second or 60 radians per second that data. There if I let us say see that G' is increasing with frequency or

G or there is a G double prime is decrease I should not immediately conclude that that is the real result I need to check double check and then look at and how will this.

Double check be done what kind of things can be done to let us say research or a development engineer who is trying to work with these materials what are the tools available at my disposal that I read something in the book or I heard this in a lecture that at high frequency low viscosity please be careful. So, I will be careful I want to know whether the data I have got is correct or not.

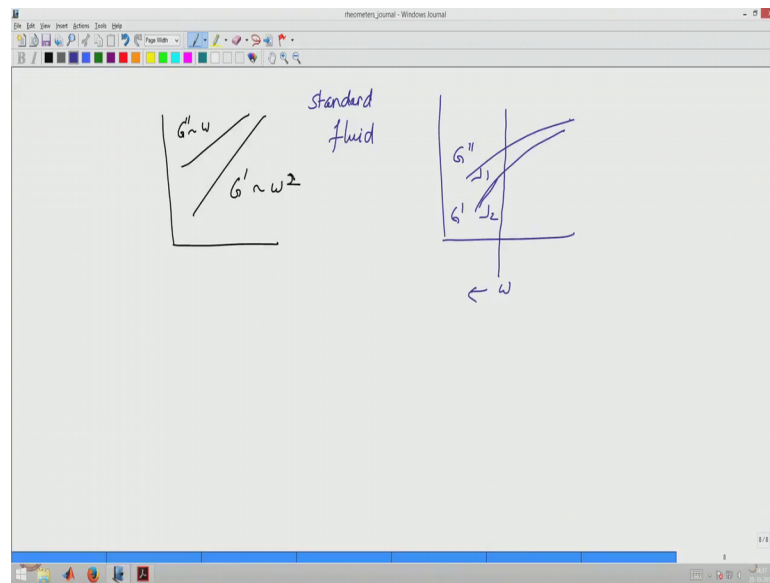
What should I do what can I do yeah. The steady shear viscosity will be correctly measured because the inertia of the motor or inertia of the sample is irrelevant once it comes to steady state. What you are talking about this talk sensitivity of the instrument. That will that will be important if we are looking at the steady state properties that is one usually one parameter at our disposal is to say look at systematic of the data.

In the sense that if we vary a composition we have certain expectations as to what might be happening that is one way of doing it, but there again all the samples that you have are unknown the first composition itself you do not know the properties therefore, you are studying it now you make and yes there is some checks and balances possible the other sets of things you can do is for example, if in the governing equations with inertia are being there is the gap important for example, the gap between parallel plate.

If I am using a parallel plate. I can try to measure properties at 2 different gaps the lower the gap the lower will be the Reynolds number at the same velocity. Lower will be the contribution of inertia. Then do I see a systematic trend again with different gaps. That is another way of doing it of course; in all these cases we always also have some standard fluids.

Which have a much defined viscosity, I can take a standard fluid and I know that standard fluid let us say is a Newtonian fluid and its response is known. I can measure it is η' η'' at high frequency also and let us say it is not corresponding to what I know it should be then I clearly know that there is some problem for example, if it is a Newtonian fluid then we know that η' is generally.

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What we will get is G' will increase as ω^2 and G'' will increase as ω . This is the result that I will get, but let us say if I collect some data in a rheometer and I get results like this where things are changing and also they seem to. I can possibly say that you know up to this frequency maybe if I look at the slope it is 2 and this slope is one and therefore, maybe I can use the instrument only up to this frequency beyond that there seem to be results which are not corresponding to a standard fluid measurement.

This is also another way of doing it, but in all of these cases what is clear is there is no prescription standard prescription that what do this do this and you will get the good results all of these cases one has to do trial and error and try out different things and then. We should always examine whether we are able to explain the results based on the mechanisms that are there in the material.