Rheology of Complex Materials
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Lecture - 56
Problems during rheometry – example of cone and plate

In this segment on the course, we will take a look at one example of a geometry and the rotational rheometer, in which this geometry is used. And we will try to discuss various possibilities in terms of problems that are encountered and the best way to do that is to look at how we do the theoretical analysis in terms of measurement of properties, and how many of the assumption that we make while doing that analysis will be violated when we might do experiments.

So, therefore, we will take an example of cone and plate, look at what kind of assumptions are done are made to analyze the fluid mechanics of flow within cone and plate. And parallely we will try to see what are the possibilities where these assumptions breakdown and then when we are doing experiments using cone and plate geometry, what are the possible ways in which we can either first know that maybe I will encounter a problem, or two, I will know what are the consequences of that problem being encountered or third how do I make sure that the problem is there or not.

So, therefore, we can try to assess our results in terms of the experimental artifacts which are possible in a geometry. So, therefore, we will look at the cone and plate geometry and the flow problem the way it is analyzed in cone and plate.

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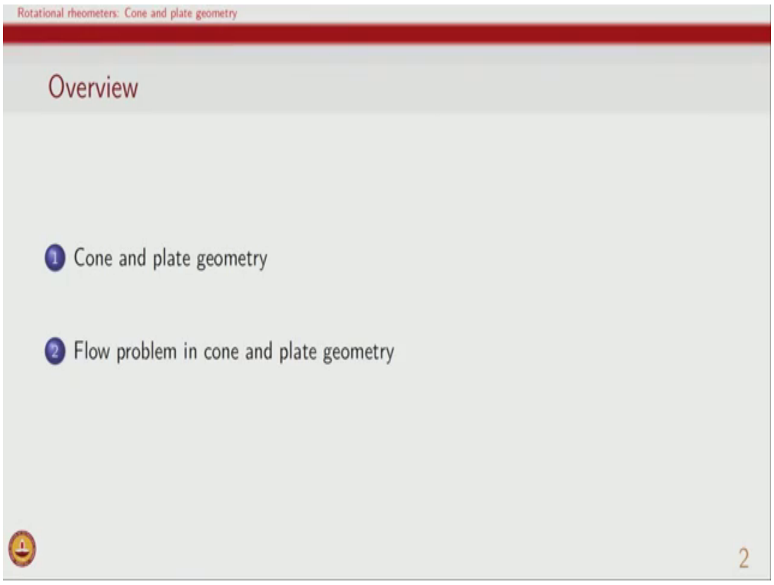

Overview

1. Cone and plate geometry
2. Flow problem in cone and plate geometry

And then we will try to see whether we can understand what some of the problems might be.

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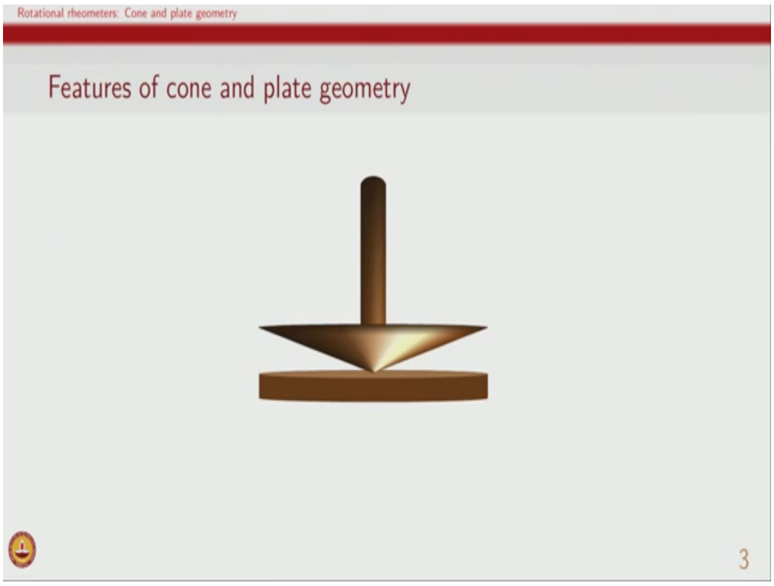

Features of cone and plate geometry

So, the cone and plate geometry is basically where we have cone rotating, and the plate is stationary, and the fluid is kept in between the two and what the main features of the geometry are as follows.

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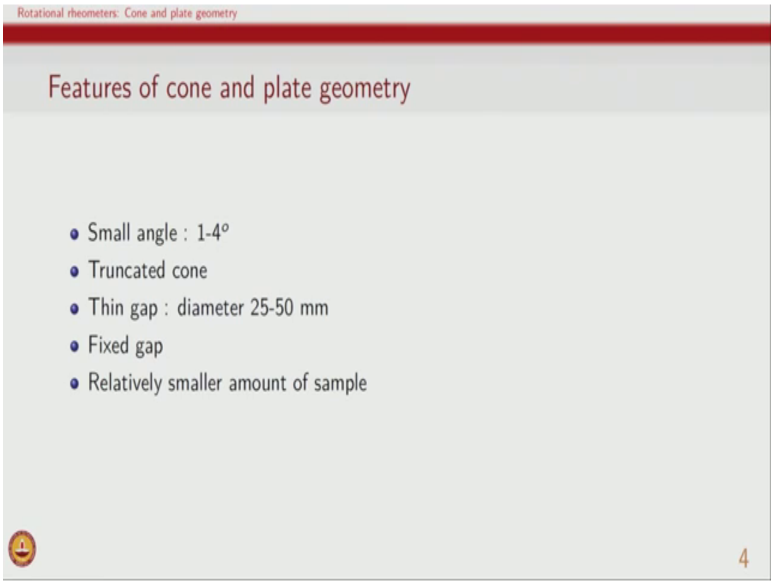

Rotational rheometers: Cone and plate geometry

Features of cone and plate geometry

- Small angle : 1-4°
- Truncated cone
- Thin gap : diameter 25-50 mm
- Fixed gap
- Relatively smaller amount of sample

That the cone angle is very small and so, this is one of the important features that if you have the overall the angle we are talking about is very small.

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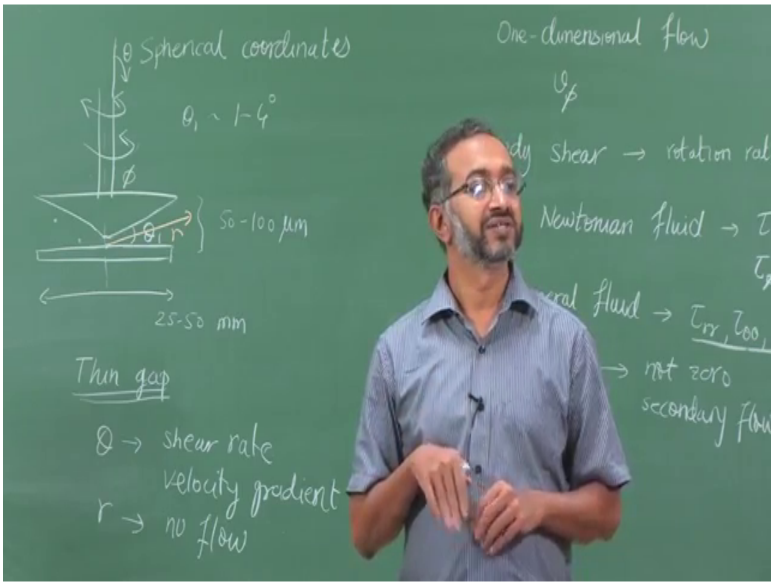

So, as we will see, this angle, theta 1 is very small. At the same time if you look at the size of the plate or the cone it is fairly large. So, generally, this gap is going to be let us say 50 or 100 micron, while this size may be 25 to 50 millimeter.

So, very thin gap in which fluid is placed, and also the angle is extremely small. It is almost like in fact, two parallel plates, but with very small angle with which the cone is made. And the idea of having cone and plate as a geometry is to ensure that there is uniform shear throughout; that is one of the most commonly sighted advantage of using cone and plate over any other geometry.

In most other geometries, the you shear let us say if you are imposing will not be uniform throughout. And so, what that implies is a for material is at this point or this point, will see different amount of deformation or deformation rate. And we have seen throughout the course that material properties depend on the extent of deformation as well as the rate of deformation and other derivatives.

So, therefore, since material properties depend on the extent of deformation and deformation rate, it is better for an unknown material to be subjected to the same deformation throughout the geometry which cannot be ensured in a parallel plate or concentric cylinder or other geometries. So, therefore, cone and plate is widely used as a geometry. The other thing we usually do is this cone is truncated; otherwise, it will go and touch the bottom plate.

So therefore, there is a small truncation, and what we do in general to analyze this problem is to assume certain features of the flow. So, since, it is a thin gap we have already mentioned that, and we also mentioned that the angle is very small and therefore, what you think are the possible features of flow that can be assumed in such a geometry. What are the possible features of flow? So, if given that the motion is being imposed, we generally use spherical coordinates to analyze this problem. And so, this direction is phi direction, the direction with respect to the shaft is theta, and the direction going away from the is the radial direction, right.

So, we have r theta phi as the directions of the coordinate system. And so, one of the features that we could say is given that we are imposing flow only in the phi direction, the fluid will move only in the phi direction. So, that is one possible feature. What are the features can be thought of? What other features can we say; given the geometry given the specifications that you know it is a very thin gap small angle.

So, theta direction will be the where shear rate will be there right or in other words, we also know that velocity gradient will be there in theta direction and as he is suggested that most likely we will have no flow in r direction right, in r direction. So, what other features could be specify? What other features that we could think of? And then what we have to think is are any of these features being violated, when we are trying to do experiments.

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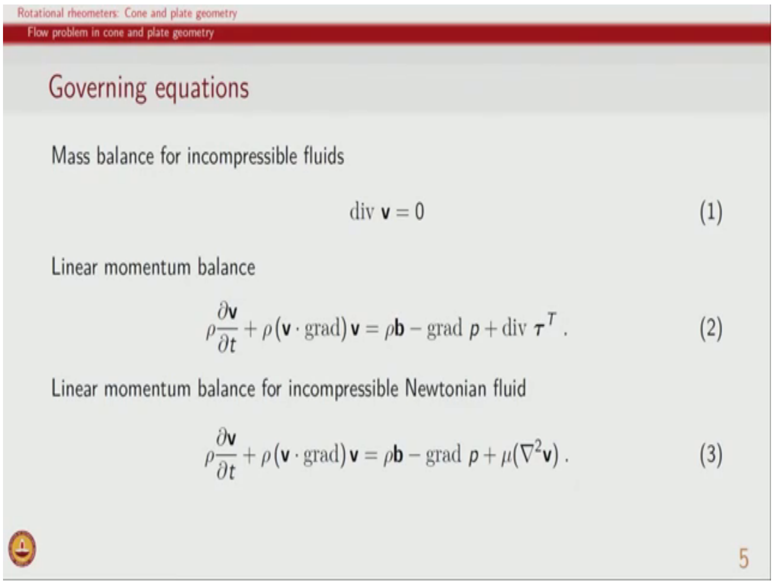

Governing equations

Mass balance for incompressible fluids

$$\operatorname{div} \mathbf{v} = 0 \qquad (1)$$

Linear momentum balance

$$\rho \frac{\partial \mathbf{v}}{\partial t} + \rho (\mathbf{v} \cdot \operatorname{grad}) \mathbf{v} = \rho \mathbf{b} - \operatorname{grad} p + \operatorname{div} \boldsymbol{\tau}^T . \qquad (2)$$

Linear momentum balance for incompressible Newtonian fluid

$$\rho \frac{\partial \mathbf{v}}{\partial t} + \rho (\mathbf{v} \cdot \operatorname{grad}) \mathbf{v} = \rho \mathbf{b} - \operatorname{grad} p + \mu (\nabla^2 \mathbf{v}) . \qquad (3)$$

So, the overall governing equations for this of course, for this flow are mass balance and linear momentum balance. We have seen that angular momentum balance leads to symmetry of stress tensor. So, therefore, stress tensor is symmetric, and they then the only two governing equations are mass balance and linear momentum balance. And for a general fluid, we can write the overall linear momentum balance in terms of stress tau, the deviatory stress because we do not really know how tau varies with deformation of deformation rate.

We do not yet know the constitutive relation of the material. If it is a Newtonian fluid, then of course, we can express it in terms of Navier stokes equation. So, if the fluid is Navier stokes, then we have the divergence of velocity is 0 and then the Navier stokes equation. Otherwise, we have divergence of velocity is 0, and then in terms of the stress tensor tau.

So, this is the overall governing equation, and the question is what happens to these governing equations for such a flow. In which case we have said that there is only seems to be one dimensional flow, right.

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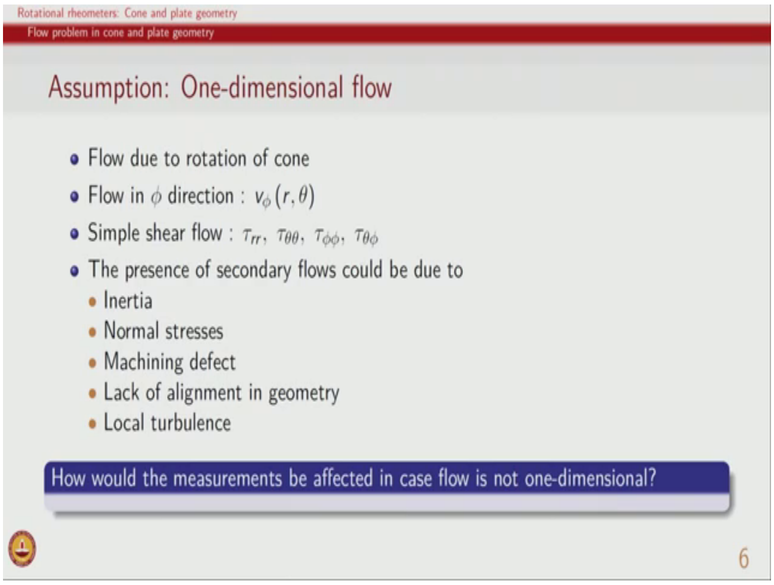

Rotational rheometers: Cone and plate geometry

Flow problem in cone and plate geometry

Assumption: One-dimensional flow

- Flow due to rotation of cone
- Flow in ϕ direction : $v_\phi(r,\theta)$
- Simple shear flow : τ_{rr}, $\tau_{\theta\theta}$, $\tau_{\phi\phi}$, $\tau_{\theta\phi}$
- The presence of secondary flows could be due to
  - Inertia
  - Normal stresses
  - Machining defect
  - Lack of alignment in geometry
  - Local turbulence

How would the measurements be affected in case flow is not one-dimensional?

So, what we are saying is it is a one-dimensional flow.

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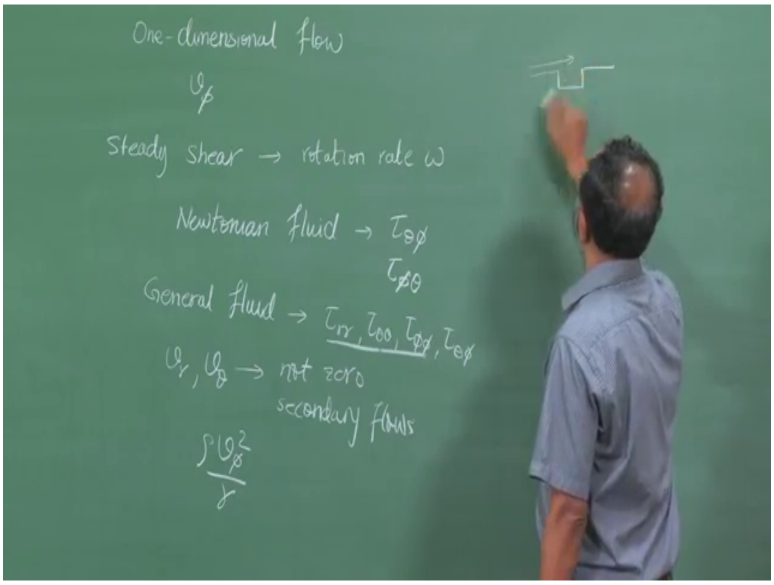

So, we are only assuming v phi, right? And the flow arises in the geometry because the cone is being rotated and this is an example of quite flow. Where a solid surface is moving and which is causing the flow. And we can say that let us say the rotation of this in case of study shear, we will have rotation rate omega, right. So, it is being rotated at some rate omega.

Student: Sir, v r will be 0 or neglected.

Student: (Refer Time: 09:27).

Yeah. So, the question is being asked is will radial velocity be 0 or are we neglecting it. So, what in which way should we try to answer this question?

So, in engineering and in analysis of such fluid mechanical problem, there will always be two possibilities. One the fact that there is a peach fluid mechanical phenomenon, but we will say it is a effect will be minimal and therefore, we can ignore it or second thing is to say that we are enforcing condition such that we will that radial component will not be there.

So, the question is if I am rotating the cone alone, can I expect the fluid to only move in the rotational motion and not have the radial component or no matter how careful I am there will always be radial component and therefore, I have to ignore it. So, these are the two questions right.

So, is it possible for me to be very careful, and then say that looks I do not really need to assume, that radial velocity will be 0. Under normal circumstances, I can assume that radial velocity will be 0. So, what do you feel among these two which one is more reasonable.

Student: Sir low c r rate, we can the r pattern the velocity at r distance will be almost 0.

Yeah. So, the therefore, one of the responses will be is to first try to think of as to under what condition radial velocities might arise in the fluid. So, for example, if I ensure that I am only moving in theta direction, it is safe for me to say that fluid will also moving theta direction; however, radial velocity may arise if there is a centrifugal force.

In that case, and as was pointed out that if we are doing the experiment at higher shear rate with more degree of more rate of rotation, it is possible that fluid will get thrown out. And in fact, this quite how happens quite a few times in rheometry, when you are trying to measure a sample and you go to high shear rates you can see fluid coming out of the geometry. So, clearly you know that you have exceeded and the one dimensional flow assumption is clearly being violated.

So, in that case, radial velocity is present and cannot be ignored, but in general cases we will say that we were there is no need to say that we are ignoring the present radial velocity. We will say that we since fluid is being only forced to rotate, and go have a v phi motion there will not be any radial motion. And so, we are looking at only simple shear flow. So, we know that there are only four components of stresses which are non-0 at general most general. For a Newtonian fluid we will only have tau theta phi as the non-zero component.

But for any general fluid so, given we are doing shear flow for a Newtonian fluid, we will only have tau theta phi, right. And of course, this is a symmetric sensor so, we have tau phi theta. But for any general fluid which we do not know the behavior of, we have seen that only tau r r tau theta theta tau phi phi and tau theta phi will be non-zero.

So, at most these are the stress components will have will have to be analyzed and so now, the question is what are the conditions which; might lead to the violation of the one-dimensional assumption. And the fact that flow is no longer one-dimensional flow; we call it by saying that there are secondary flows in the geometry.

So, even though we are imposing only a circular motion and we expect each and every fluid element to go around in circles, secondary flows might arise. And these secondary flows will lead to velocity other components v r and v theta also being there. So, if these are not 0, then we say that there is secondary flows. So, what are the reasons for which due to which secondary flow forces can be there.

So, one reason we already mentioned is inertia. So, centrifugal acceleration is an example of inertia where if the velocity is very high in the phi direction, the fluid will have an acceleration in the radial direction. So, that is one possibility, if you look at the equation of motion for a general fluid, we will see that the presence of these components can also lead to v r and v theta ok. So, both inertia and normal stresses will be present in the governing equation for v r and v theta.

Because generally what we will see is, basically we have 3-dimensional situation so, there are three governing equations of Navier stokes, three components, v r 1 for v r 1, for v theta or r direction Navier stokes, theta direction Navier stokes, phi direction Navier stokes. So, what we will see is, in the r and theta direction also, these stresses will appear and similarly centrifugal acceleration rho phi squared by r, right terms like this. This is nothing but inertia, right? This will also appear in the r component of Navier stokes equations or in equation of motion.

So, clearly whenever there will be inertia or normal stresses, there is a possibility of radial stresses, what we have to make sure is that we are operating in the regime where inertial forces are not very significant and one of the ways to think about that in terms of looking at Reynolds number. So, if Reynolds number is low, then we will be safely ignoring inertial terms. And given that we are operating with a very thin gap given that quite a few times we are working with viscosities of higher magnitude many of the non-Newtonian fluids, or many of the complex materials have reasonably high nominal viscosity.

So, given that dimension is small and viscosity is large, generally the Reynolds number can be kept fairly low. But if fluid elasticity itself is also significant, then we need to make sure that those do not cause any secondary flows in the material. What do you think is meant by machining defect? If for example.

Student: The given specification is not met.

Yeah, the tolerances of the geometry are not very good, and the angle for example, this is supposed to be let us say a 2-degree cone; it will be 2 degree all throughout, right. It is the solid angle of 2 degrees, but if there is a slight wobble in the machining. Then what happens is, fluid will itself also develop a radial or theta direction flow.

So, any defect which is there in terms of the machining of the geometry is very important in terms of causing such secondary flows. And therefore, maintenance of geometry is very crucial in case of rheometry. When we use let us say a material to scrape off, it is easy that we will put a scratch there and that scratch will lead to some secondary flows locally; because any scratch is basically if you look at microscopic scale is something like this.

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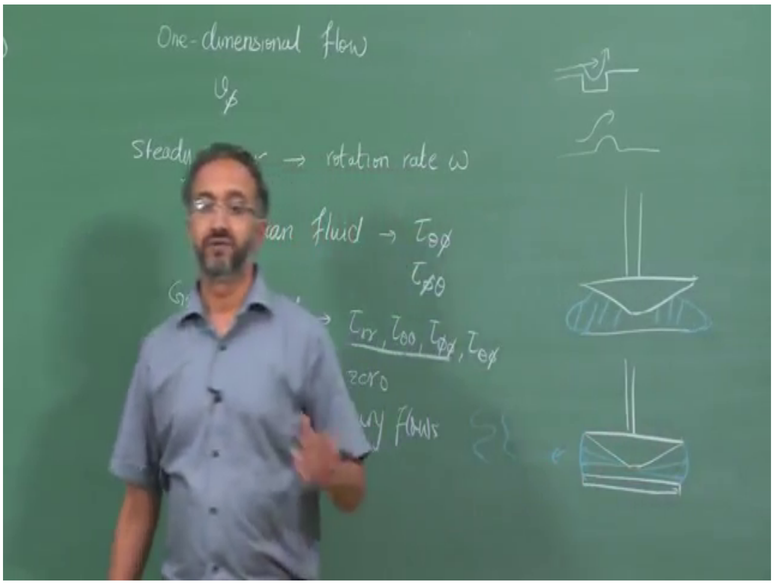

And then if we have a fluid flowing like this, it is going to develop other velocity components or if some material is left behind in the geometry itself.

So, if you have a geometry and then some material is left behind. Again, this will lead to secondary flows. So, any defects in terms of the material not the geometry is not being clean or when they were machined itself there are tolerances. So, that could also lead to secondary flows. The other aspect is alignment, because we are assuming that this is a perfectly 90 degrees. The cone and plate are the access of the perpendicular to this plate, and the access of the shaft which is connecting the cone are exactly aligned.

If there is a small tilt in the cone, then again, we will have a wobble and that wobble will lead to again v theta v r v phi. And so, again so, this depends on the construction of the instrument and it is stability and of course, the ability of the instrument may also be different depending on what conditions it is being operated upon. For example, if we take the instrument to very high torques which are limits of the instrument, or we take the motor to very high rotation rates, which are again limits we might expect that some of the non-rigidity is associated with the overall instrument might come into play and we may get the small misalignment.

So, therefore, some of these features are more often than not experience when we are operating the instrument that extremes in terms of the instrument capabilities. And then given that we have many different types of fluids, and depending on what is the nature of flow here, there can be local pockets of turbulence. For example, we know that in micro-fluidic channels, even though Reynolds number is low, it is possible to have turbulence at a lower Reynolds number much lower compared to 2000 which we know generally learn that in industrial case, in pipeline flow Reynolds number about 2000 or 3000 we assume turbulence.

But in case of micro channels, even at lower Reynolds number turbulence can develop. So, depending on the disturbances that are developed due to the rotation or oscillation in the geometry, you can have local pockets of turbulence.

So, therefore, in all these cases secondary flows are possible. So, the one question that comes us how would the measurements be affected, if we know that this assumption of one dimensional flow is being violated in which way? For example, if now one is doing study shear and trying to measure viscosity using cone and plate, and there are these secondary flows. So, what would happen to the measure of viscosity?

Student: Here other component of the c r light tau other components also will be coming in to the picture.

Right.

Student: Viscosity will decrease. So, viscosity will be (Refer Time: 20:43).

So, what you are measuring will generally will that be higher or will that be lower compared to.

Student: It will be lower.

So, the options are that viscosity may remain the same or viscosity will generally be found to increase or viscosity will be found to decrease ok. From a material point of you the viscosity is one value, but because in our assumptions and in our design, we are saying that we will only look at one dimensional flow, but the actual condition is that there are secondary flows.

So, then the torque required will it be more or less right, that is the question. So, one answer is that possibly the torque required will be less. In that case, I will measure the viscosity to be less and so, therefore, there will be error in terms of measurement, the actual viscosity is eta while I might measure 0.9 eta. So, will I measure 0.9 eta or will I measure 1.1 eta, that is the question and why. So, if we expect let us say that the viscosity to be lower, why would it be lower? So, one way to think about is again to go back to very basic ideas that we have discussed related to energy storage and dissipation, right. And viscosity is a measure of dissipation, right? And then so, if you have secondary flows do we expect dissipation to be more or less.

Student: More.

More, right? So, therefore, what will end up happening is generally you will require higher torques. Because you are not just moving the more fluid in the circular your whatever you are imposing is also responsible for several other flow. So, therefore, in general the dissipation that will happen will be higher, then if it were just rotating based on whatever you are imposing. So, in general with the secondary flows, it is commonly seen that the measure viscosity would increase. In fact, if you are let us say interested in analyzing the instability of fluid flow.

In the sense, when does a secondary flow develop, right? Under what conditions under what Reynolds number or under what flow conditions does the secondary flow developed? You could take a fluid of constant viscosity in this geometry and start let us say increasing the rotation rate. Suddenly, at some point you might see that now the viscosity that is being measured is higher than what you what is the viscosity of the fluid. Then clearly that is the transition point at which secondary flows have developed.

So, therefore, the secondary flows will most often lead to higher viscosity measurement. There is possibility of it leading to lower viscosity, if the secondary flow leads to let us say loss of material between the geometry, right. So, if you have a material here, right? And the secondary flows leads to some of the fluid going out so that after sometime you are in fact, doing experiments with only a limited material. And so, in that case, let us say if you later on due to if this is the fluid, right? It is gone out and it is no longer in fact, contacting the overall surface. So, in that case then it is certainly possible that you might measure viscosity lower then what is the correct value.

Now, the main question in many of the cases is that you do not know what you are the viscosity of the fluid that you are trying to measure, right? In many cases if you know the viscosity then you will not be measuring. So, these questions you have to answer when you know that you are looking at an unknown material and then in that case you have to try to estimate as to whether it is possible to for you to think of when will this assumption be violated.

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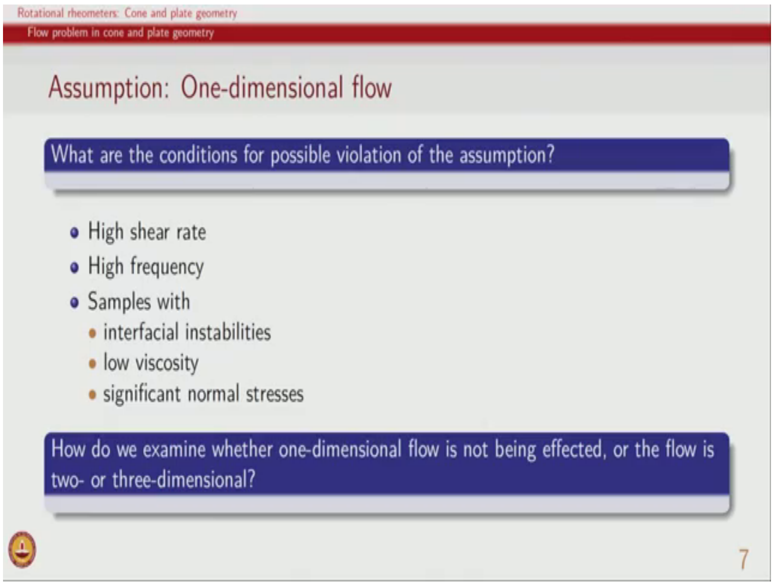

Assumption: One-dimensional flow

What are the conditions for possible violation of the assumption?

- High shear rate
- High frequency
- Samples with
  - interfacial instabilities
  - low viscosity
  - significant normal stresses

How do we examine whether one-dimensional flow is not being effected, or the flow is two- or three-dimensional?

What are the likely scenarios when I am trying to do rheometry on an unknown material, that this assumption of one dimensional flow may not be valid. So, one of the reasons we already said, right? For example, high shear rate, because high shear rate implies high rotation rate, high rotation rate implies generally high value of the velocity and therefore, high value of centrifugal acceleration inertial terms. And therefore, that will lead to radial flow.

So, in general, these are some of the features which will lead to high shear rate and high frequency usually will imply that there is higher velocity. And because there are higher velocities, it is possible that we will have issues related to secondary flows. The other aspect is what is the interface like. Given that we are using the spherical coordinate system for analysis, and if we normally plays of fluid in air and on a solid, what is the shape of the interface if we make a bubble generally? What is the shape of the bubble? If you make a one drop in another drop, what is the shape of that interface.

So, generally we expected to be spherical, and that is based on minimization of surface interfacial energy, right? That we know that spherical surface per unit volume is lowest.

So, therefore, interfacial energies are lowered. And so, similarly if you see here also, we have air fluid and solid, right. So, there is this air fluid interface, and generally this will also be spherical cap. So, the most often when we form the interface would be radial. So, we can see that the radius of curvature is constant, but depending on the surface tension, depending on the working conditions, it is possible that this interface will deform. Just the way when I jet comes out of tap, it will be a nice cylindrical jet when the flow rate is low, but later on it will you will see instabilities on the surface and eventually it will breakup into droplets and like a spray.

So, therefore, depending on the shear rate, you may also have interface deformation, and that will also lead to secondary flows because interface deforming implies that there is velocity in the radial direction. Because what will happen is this interface will become maybe it will become shape like this and then it may even have a higher and higher fluctuation.

So, that necessarily implies that there is flow in the r direction and possibly theta direction and of course, we are imposing phi direction flow and the generally when we are working with low viscosity fluid, we would expect some of these effects to be more because low. Viscosity implies inertial forces to be more significant.

So, generally when we are trying to measure low viscosity materials, we should be very careful in terms of whether there are artifacts due to the secondary flows in the material and then one last aspect is of course, normal stresses. Since, we know that the secondary flows can be caused due to either inertia or due to normal stresses whenever we have fluids with high normal stresses, or low viscosity we can expect the overall flow to not remain one dimensional.

Now, the question is how do we examine, whether the flow is indeed one dimension or whether it has, I mean, this is all gases in terms of fine it is good to us to know that higher shear rate might cause, one dimensional flow assumption to break down. But shall I measure at 100 per second, 200 per second; shall I rely on the data that I get till 500 per second or 50 per second? So, it is not clear.

So, then how do we examine? I mean, how what can I try? To answer this question that I have done measurement, I know I am working with let us say a low viscosity fluid, and I have done measurements at 50000, 200, 500, and now I have to convince myself and of course, others also that the data is reliable. So, how do I examine? How do I think about whether the one-dimensional flow assumption was indeed valid during the operation?

Student: As I various speed if will do and we will see a how the viscosity it is a constant in a particular geometry and then we assume that secondary flow is less but.

Where you.

Student: (Refer Time: 30:29) a suppose we are increasing from very small velocity to higher velocity, definitely if will plot the viscosity, when the turbulence of secondary flow will come into the picture there.

Right.

Student: We can see the deviation of viscosity.

Here, but we do not generally know the viscosity right in the sense what might happen is if you let us say plot viscosity as a function of strain rate. So, since we are look discussing mostly the example of we are discussing viscosity as a function of strain rate.

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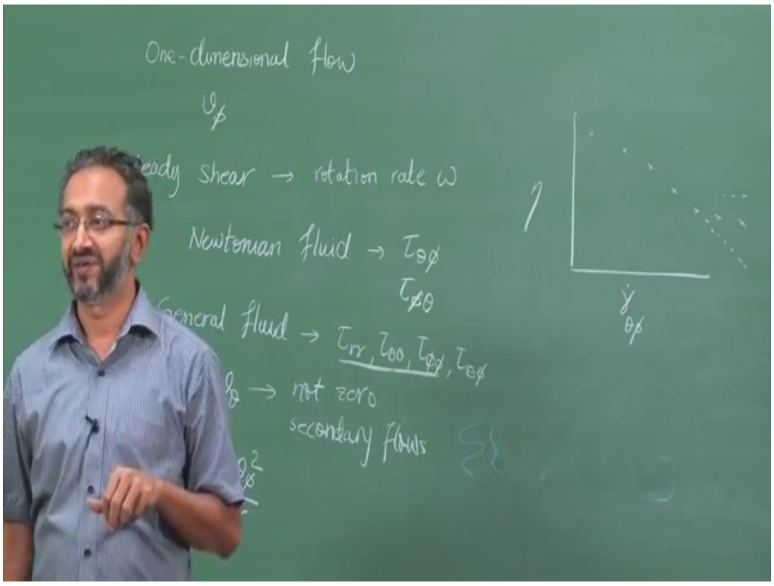

So, gamma dot theta phi, let us say, right which depends on omega we may get data points like this, right?

So now the question is, this viscosity seems to be flattening out, right? Is this real? Am I measuring this viscosity to be higher though actually it viscosity should have actually decrease, but because I am getting secondary flows now I am measuring high viscosity. The other possibilities in fact, the real viscosity is in fact, should become a constant, but because the flow it is getting thrown out I am somehow getting lower viscosity so, how do we examine?

So, therefore, you need to do careful examination of the working condition. This is little easier to predict because you can see, right you can observe whether the fluid is coming out. This phenomena is far more difficult, because you may not have an access to see whether secondary flows are caused or not. So, quite often we will have to rely on flow visualization, to try to see whether indeed the one-dimensional flow is valid. The other thing you can do is to try to do experiments with different geometries because any material behavior should qualitatively remain the same.

So, for example, if this flattening out is a real phenomenon, it should not get completely different in two different geometries quantitatively it may be different. So, any feature that you want to claim as material phenomenon will remain the same in different geometries ok.

So, with next part of the segment we will continue and ask some of the questions related to other artifacts which are possible during the instrument operation.