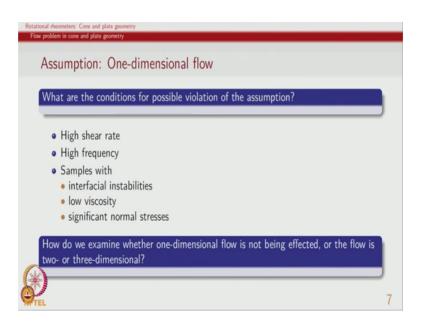
## Rheology of Complex Materials Prof. Abhijit P Deshpande Department of Chemical Engineering Indian Institute of Technology, Madras

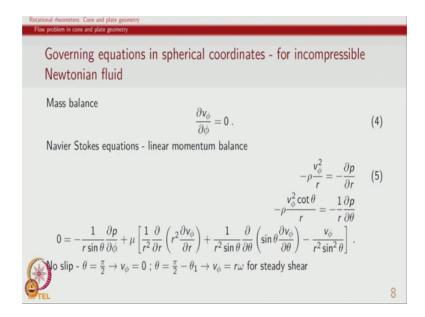
# Lecture – 57 Rotational rheometers: Cone and plate geometry

So we are looking at the rotational rheometers and through a cone and plate geometry example we are trying to look at various artifacts which might be present and how the analysis of fluid mechanics is affected based on violation of these assumptions. So, if you look at the overall governing equations for cone and plate flow and this is only for the Newtonian fluid the mass balance is basically saying that v phi will not be a function of phi itself and therefore, v phi will be a function of r and theta. It is a function of r because the velocity itself will be different at different radial positions and it is a function of theta because that is the direction of shear. So, velocity gradient does exist in theta direction.

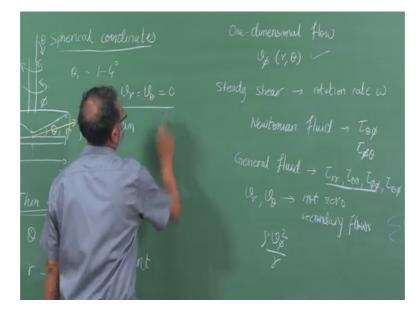
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So, if we now do the analysis and based on saying that it is a 1 dimensional flow and it is a Newtonian fluid. When we write down the overall governing equations with the assumptions we can see that all 3 components of equation of motion terms are there. So, we have the inertial term as we saw here rho phi square by r and that is balanced by the pressure gradient. Similarly there is another inertial term in the theta direction again balanced by the theta.

So, generally when we are saying that we are inertial terms are negligible we are assuming that we can ignore the terms such as v phi square by r and therefore, we need not worry about pressure variation also in r and theta direction and in fact, we will say that pressure remains uniform the flow is only because a solid surface is moving and it is an example of coquette flow, where flow is affected due to flow is being made possible because of motion of the cone. And even with these assumptions since v phi remains a function of r and theta we have basically partial derivatives with respect to r and theta in the governing equation right. So, this is the if we assume one dimensional flow and incompressible Newtonian fluid these are the set of governing equations.

So, we can ignore the first and second equation by saying that pressure is irrelevant inertial terms are 0. So, in the third equation we can ignore pressure and still we have a partial differential equation for v phi because it is function of both r and theta. We say that fluid element is of course, equal balance of mass also tells us that v phi is not a function of phi, but we could also use a symmetry argument to say that a material element moves and then comes back to the same phi position. So therefore things should

not be changing with phi because it cannot be changing and then suddenly it comes back to the same value. So, therefore, the symmetry argument can be used to say that nothing depends on phi.

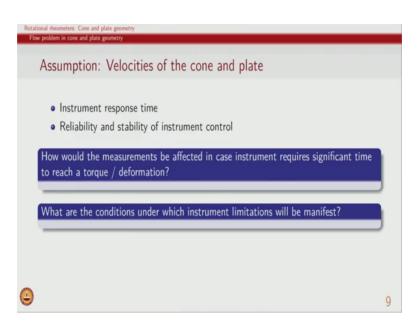
No that those viscous in the r and theta component the viscous terms will be v r and v theta based and we are assuming that v r and v theta are 0. We are saying only v phi is nonzero. We are saying that v r is equal to v theta is equal to 0. That is the assumption right? It is 1 dimensional flow. So therefore, equation of motion of r component and theta component will include Laplacian of v r and Laplacian of v theta and those components will be zero.

Student: This final equation comes from which equation?.

This is the y v phi component phi component.

Ok so and the other boundary conditions which are required to solve this flow given that we are solving for v phi are the fact that velocity at that cone is basically based on this r omega and velocity at the bottom plate is 0 and this is the no slip boundary condition. And so that is another major assumption that we are making that velocities of the cone and plate that are being imposed.

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So, we are imposing certain rotation on the cone we are making sure that plate is stationary and so, does this really happen. For example, we are saying that cone is rotating with some velocity omega right rotation rate, is the cone rotating or does the cone take some finite time before it comes to rotation rate of omega. Because when we are making a measurement we are saying that gamma dot theta phi is constant gamma dot theta phi depends on omega being constant and whether instrument has enough response time to quickly come to omega and then we can say whatever we are measuring is at constant omega and therefore, constant gamma dot theta phi.

So, this is one question that again some of these features may be there when you are operating the instrument at very high torques very close to the instrument capabilities that it may not be able to achieve whatever is being informed that it should achieve. Of course it also depends on the instrument control strategies that when we asked instrument to reach a constant value of omega how does the control happen? Usually depending on the control strategies being implemented there is an overshoot and then it settles down. So, whenever there is a control variable and a set vary of set point which is given to an instrument will try to meet that.

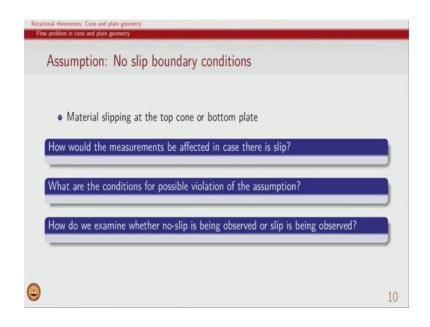
Now how fast does it do it? And is there an overshoot? And then settling of that value. If you are making a measurement during this period you will definitely get artifacts, because you are measuring in fact, the instrument capabilities and not really the material behaviour. Because material behaviour can be measured only when omega is become constant and gamma dot theta phi has become constant.

So, even though we are assuming that the fluid layer here is constant omega we have to make sure that instrument itself is able to rotate at omega. So, again in this case also one can think of the same question is how would the measurements be affected if instrument is not able to reach the set value right. So, if let us say we say 100 RPM it should rotate at, but it is rotating at 98 RPM. Then clearly the measurement will be off.

And again the conditions under which we would expect this is when we are either the sensitivities are too low. For example, if we ask the instrument to apply micro Newton's as a force. Let say we are doing a creep experiment and we ask it to apply very small stress then the motor will not be capable of producing such stress. So, instead of applying 1 micro Newton it might apply 1.5 and then it will adjust and it will come to 0.5 and so on. So, it will not be able to apply a constant stress, but it will wobble around that because of its control capabilities and the instrument operations. So, whenever we are

looking at limits of the instrument operation we should be careful that instrument response may be being measured as opposed to the material response.

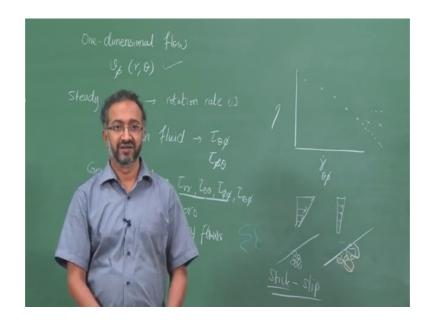
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And so, the next assumption which is a major assumption is again no slip boundary condition. We are saying that since cone is rotating at omega the fluid next to it also rotates at omega. Since this plate is stationary the fluid next to the plate remains stationary. So, this is this can be violated if material slips. So, the slip can be there at the bottom surface also slip can be there at the top surface also.

So, which means the top surface which is contact with cone will not move at omega, but move less velocity or even though here plate is 0 velocity the fluid on top of it may slip. So, then the question is how would the measurements be affected if there is a slip. So, again I am trying to measure viscosity and the ideal case scenario and all the analysis is being done assuming that there is slip there is no slip, but if there is slip in the material what will I measure as viscosity. Will it be lower than the material viscosity or will it be higher? Any guesses on what it might be.

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So, the question is how might the velocity profile look like. So at any given point what we are saying is the velocity profile should look like this right? The cone the v phi at the top cone should be high and then it should reduce to 0, but if there is slip what we would expect is that velocity will be not as high and even at bottom there may be some velocity.

Student: Gradient will decrease.

Yeah. And so the torque rick. So, what would I measure as viscosity. So, the real fluid is actually only encountering a strain rate which is much lower therefore, the torque that I really required to influence this shear rate will also be lower.

Student: (Refer Time: 10:25).

And so, therefore generally, but the I am assuming that I am imposing this strain rate. So, when I do the calculation of viscosity I will say this is the strain rate being applied, but actually the strain rate being applied is this. So, therefore, in general the viscosity that I measure might be less than what is the real material viscosity. Now, again how does one detect slip and what are the possibilities, what are the possibilities that might lead to slip. So, when should I be more watchful about the fact that there may be slip in the experiment that I am doing.

Student: Suspensions.

Yeah. So, one easy answer is to say that you know if there is it suspensions is one possibility even polymer melts is also there. So, the question to answer is it possible that the material will adhere and then come off. So, in case of suspensions is easy to see that a set of particles, this whole cluster may move together instead of it. So, see the expectation is at the top surface the fluid and the particles which are there they will move along, but instead attraction force between clusters is so high, that this will break off here and suddenly this cluster will not move as fast and the fluid on top will be still moving at the same rate as the cone is moving.

So, in that case it is fall. Similarly in case of macromolecules also this is possible. That if you have an entangled melt, so let us say some the polymer will be adsorbed and will be interacting with the top surface and therefore it will move along with the cone, but this polymer is also entangled with some other molecules and given that there are other molecules.

Now, the entanglements will cause the polymer to come off from the cone. So, therefore, these are phenomena of stick slip. So, material remains stuck for some time with the top surface and then some other time it slips. So, therefore there will be a partial slip that is observed in such cases. So, that generally depends on the extent of interaction within the material itself and the extent of interaction between the material and the surface which is being used.

So, one easy way to modify slip would be to change the nature of this surface. For example, let say if you suspect that adhesion between the particle and the surface is not good, you can modify the surface to say that I will make the adhesion even poor and then see if I get even less viscosity right. Because what I can do is in this case I know that polymer does not absorb very well. Let me now go to a surface where adsorption is even poor. So, then I am saying that slipping will be even more.

So, if anytime I do let say measurements with different surfaces and I measure different viscosities I clearly know that something is wrong because material property should not depend on the surface of the geometry. So, quite often we modify the surfaces and then check the other way of course, is to enable more addition. One easy way of enabling more addition sometime is to induce little bit of roughness small amount of roughness, which will not induce enough secondary flow. So, that my flow 1 dimensional flow

assumption is violated, but at the same time roughness usually will increase addition between 2 materials. So, therefore, slipping of materials will be less if I increase roughness and again in all of these cases we must always make sure that qualitative features of the results remain the same with all these different types of geometries.

Student: You did not changed the material.

Yeah that is what. So, you can modify the material or you can change the material also. For example, if I let say I have a suspicion that the material is not this material is very hydrophilic and the surface is not hydrophilic enough and therefore, there is a partial slip I could say that I will use Teflon as the hydrophobic extremely hydrophobic material format the geometry. So, that I will ensure that more slip happens and then I will examine the result.

Or I can choose a 3 4 different. So, there is copper, it is possible to use aluminium, steel, different metallic elements are possible different plastic glass. So, several such materials are possible and then roughen geometries and non rough geometries, smooth geometries roughness of different degree. So, with all these whenever we say slip is a possibility we have to make sure that experiments are done with different geometries and we conclude that qualitatively results remain the same regardless of which geometry we use then we can rely on the measurements. So, we have to try to make sure that slip is not there and the phenomenon that we are seeing is actually due to materials.

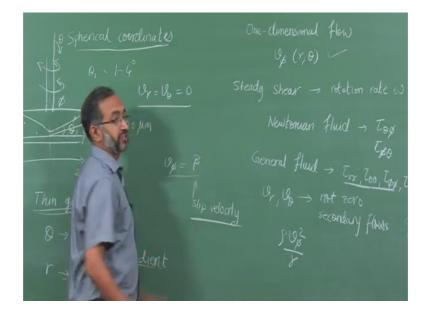
And of course, the same question you can also ask as to what are the possible conditions. Again high velocity of the cone or whenever the there are mechanisms in the material which will lead to strong adhesion right strong cohesive. So, this is basically strong cohesion versus addition. So, whenever we look at 2 dissimilar materials adhesive is between 2 dissimilar materials and cohesion is between this material itself. So, whenever cohesion exceeds addition then you might have stick slip phenomenon.

So, therefore, this is something we have to worry about and again how do we examine whether there is slip or no slip by doing systematic experiments with various geometries you could always of course, to flow visualization. And so, as we discussed during the course of the segment on rheology that by using NMR or using particle image velocimetry or using optical methods we can actually look at flow within the geometry. And in the last 10 15 years lots and lots of flow visualization has been done to visually

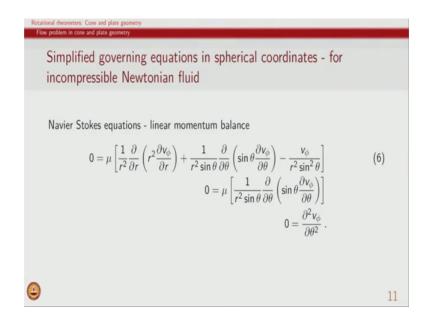
try to confirm that there is no slip being observed or if there is partial slip to what extent there is slip in the system so that, you can do your analysis again because sometimes let say if the material system is such that there is always some slip.

Then you need to your go back to the governing equations and say my boundary condition is no not that there is no slip I will say some partial slip. So, for example, what you can say at the bottom plate is v phi is equal to beta which is nonzero and this is the slip velocity. So, it is possible to then redo the analysis then calculate the overall torques that are required and then therefore, back calculate the viscosity we are of course, doing analysis by saying that v beta is 0 at the bottom plate similarly at the top plate velocities r omega instead of r omega by of some fraction of r omega this.

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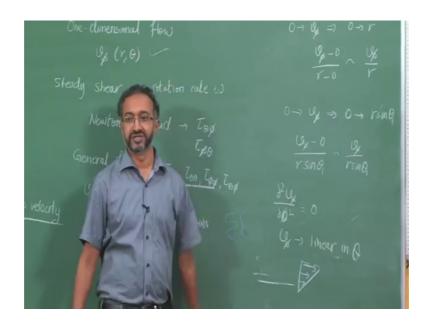


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So, that is as far as the other based on these assumptions given that we assume no slip and given that we assume that instrument is able to do what we are asking it to do right. So, then we can ignore now the r and theta components of equation of motion. So, we are only left with phi and then if you look at the phi component of motion we can assume that the variation in r is much less compared to variation in theta and that is what we have said here when we say that main shear rate will be in theta direction and that is again possible to do by doing them we can do order of magnitude analysis, v phi varies from 0 to r omega in distance r. Similarly v phi varies from 0 to r omega in the distance r theta. So, therefore by doing order of magnitude analysis we can see that the variation is much higher in theta direction and very low in r direction.

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So, v phi goes from 0 to v phi when the distance goes from 0 to r. So, the gradient can be calculated by saying v phi minus 0 r minus 0 right. So, this is v phi by r. So, this is the order of magnitude of the variation in velocity with respect to distance travelled. When you go from the centre to the outer edge velocity will vary like this. Similarly velocity goes from 0 to v phi when distance goes from 0 to r theta or r sine theta right. Of course, if theta is small so, sine theta is same as theta.

So now, if you look at same way. So, v phi minus 0 divided by r sine theta and given that the sine theta is a small value, the velocity gradient in theta direction is much higher. So, velocity gradient in theta direction is much higher and so we can ignore the variation with respect to r and therefore, we can ignore the first term which is del y phi by dell del v phi by del r and only retain the velocity variation with respect to theta. Actually it will be theta 1. So, this value will be theta 1 because theta 1 is very small. Theta is being measured with respect to this.

So, therefore we can say that we can consider only v phi variation with theta. Now the next assumption that we can make is the value of sine theta will remain very close to 1 because we are looking at sine. Let say if this angle is 1 degree then we are looking at sine 89 which is cone and sine 90 which is the plate. Since theta is being measured with respect to this. So, the cone will be 89 and this will be 90 and sine 89 and 90 can be almost approximated equal to 1.

So, therefore, the overall governing equation basically reduces to. So, this will lead to the statement that v phi will be linear in theta, v phi will be linear in theta and that is the velocity profile that I had drawn that it will be a linear velocity profile, as we move in theta direction.

Student: This is for Newtonian (Refer Time: 22:40).

Yes. Yeah. So, what we will see is a consequence of this linear velocity profile is the fact that the stress is constant throughout the geometry, but if we do it for a general fluid we will again come up with the same condition that stress is constant. So, even though let say for an unknown fluid I cannot solve for the velocity profile. I can still solve the equation of motion to say that stress will remain constant. So, therefore, that value of stress divided by the strain rate can still give me a measure of viscosity without really confirming whether the velocity remains what is the velocity profile like.

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Rotational rheometers: Cone and plate geometry Flow problem in cone and plate geometry	
Assumption: Uniform strain $/$ strain rate throughout the geometry including the edges	
<ul> <li>Shape of air / fluid interface, instabilities, edge fracture</li> <li>How would the measurements be affected in case there are edge effects?</li> </ul>	
What are the conditions for possible role of edge effects?	
Examination of edges, for shape / flow	
12	2

So, the other thing that we have said therefore, a given that v phi only depends on theta in general whatever is the strain rate which is being seen in one location is seen throughout the geometry and that again can be justified based on the fact that when you go from here to here, the if you look at this height the height keeps on changing, but the top velocity also keeps on changing the height is proportional to r the top velocity is also proportional to r. So, since both are proportional to r the ratio of the velocity to the distance actually will remain independent of r. So, that is why we say that there is uniform shear rate throughout the geometry. In fact, it will depend on only omega and theta. So, the shear rate in this case can be shown to be equal to. So, the strain rate will be equal to, omega by theta naught a theta 1. So therefore, it is related to just the geometrical features and the instrument operating condition and this is same. It is not a function of position it is not a function of which location you are in the geometry.

So, the question now is whether this assumption of uniform strain rate will be violated when I am doing the experiment.

Student: This is valid only for theta 1 where range between 1 degree to 4 degree.

Small angle. So, the smaller the angle more better will be for this assumption. For example, I can make a cone with 6 degrees also, but then I should be careful in analyzing the result because maybe I will be closer to violating. If I make cone of 1 degree or point 5 degree I am better, but then some of the machining aspects and how to get the cone of that such a small angle with repeatability of tolerances may not be possible.

Student: As we are decreasing the angle.

hm

Student: Then we are going towards very close to parallel plate.

Parallel plate yes yeah.

Student: So, at that time this (Refer Time: 25:38).

So, as soon as we reach the parallel plate then the uniform sheer assumption will no longer be valid again yeah.

Student: It also will be (Refer Time: 25:45).

Yeah yeah. So, finite angle has to be there for us to be making sure that the gap has to increase.

Student: Yes sir.

But if we make too much of a gap then other features may start playing a role. So, one of the reasons why the uniform strain and strain rate may not be observed is based on the interface. So, as we saw earlier that if there is an interface and the interface starts fluctuating then we may end up if there is an interface and it fluctuates then we may end up having non uniform shear rates.

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Because as we saw if the interface itself develops instabilities and there is motion of it. If we have something like this then we will lead to non uniform shear near there because there are secondary flows there. In fact, the extreme of such thing is called edge fracture. So, you can see cracks appearing on the in surface and again the question related to this is, how will the measurements be affected most often again you will see that viscosities may be larger because you have far more secondary flows around the edges which are actually leading to more dissipation.

So, this is of course easier to see right the interface effects and because you can visualize it. One again more systematic way of doing experiments is to say that you know can maybe I will try to load the sample and the way the geometry is lowered which eventually causes the interface I can do that in different ways. The way I load the sample I can follow 2 3 differ ways. I can use a scoop and then press it first and then press the geometry.

When I press the geometry also on to the fluid I can press it very fast or I can press it exponentially. So, there are various ways I can press the sample because all of this sample eventually will lead to the inter phase formation because when I start initially the fluid will be kept here. So, the fluid will be only kept on the bottom plate right and then this will be lowered. So, I will just do this and then I lower this. So, the depending on how I lower the interface will be formed. So, if let say again the results are dependent on how I lower then again I am not examining material behaviour, but I am examining something else. So, I need to make sure that I then I need to do some visualization to see if there are some edge effects. Other thing I can do is quite often to minimize the effect of edges and we can have little bit extra material loaded and then we can examine what is the result on our.

Student: No sir instrument will (Refer Time: 29:02).

Yeah.

Student: (Refer Time: 29:04).

So, there are various things we need to do. For example, whenever we lower the geometry a lot of material will be there. So, then we have to remove it because we do not want that material to be flowing and contributing to the motor torque. So, depending on the regime we are in and depending on what we are trying to do we will have to do several again systematic trials. So, when you are working with a new material it is always better to first lower load appropriate amount and then do some tests.

Student: Yes sir.

Then just for trial load a little bit extra and then see what happens to the results or load little bit less and then see and qualitatively the differences should not be there. Quantitatively results will be different, but suddenly if you see a qualitative difference then you know that under some conditions there are some artifacts which are being there in the material.

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Rotational rheometers: Cone and plate geometry Flow problem in cone and plate geometry	
Assumption: Uniform strain / strain rate throughout the geometry including the edges	
<ul> <li>Regions of high and low shear, shear banding</li> <li>How would the measurements be affected in case there is shear banding?</li> </ul>	
What are the conditions for possibility of shear banding? <sup>1</sup>	
How do we examine whether shear banding exists?	
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So, one last effect which is also quite common is related to non uniform shear where the material instead of having a linear velocity profile as we have said we are imposing the top plate. So, that velocity will remain imposing the bottom plate. So, that velocity will remain, but you may have some feature like this . So, the material breaks into 2 different shear rates part of the fluid even though you are imposing from outside only one rate omega and therefore, you assume that the strain rate in the material is this, but the material undergoes what is called shear bending. So, 2 bands appear one band in which shear rate is very less and one band in which shear rate is extremely high and again this is a very interesting phenomenon which has been observed for many materials and again visualization is of course, always the easiest way to characterize, but not all materials you can do visualization.

For example, if you have cement paste you may not be able to visualize. Only with transparent materials you can do visualization or you may have to have another x ray or neutron beam some other way of trying to examine. So, therefore, the examination of material structure and velocity profiles using other probes is very useful in determining whether you have shear banding or not and again lots of systematic experimentation is required for us to try to answer this question is the system shear banding or is the system non shear banding.

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Rotational rheometers: Cone and plate geometry Flow problem in cone and plate geometry	
Assumption: Uniform strain $/$ strain rate throughout the geometry including the edges	
• Transients in flow due to material response How would the measurements be affected in case there is transient behaviour?	
What are the conditions for possibility of transients?	)
How do we examine whether transients exist?	
9	14

And so, one last feature is also the transients. We said that omega is become constant therefore, we also say the linear velocity profile has reached. But it may take some amount of time before the material actually reaches the steady velocity profile of linear. So, therefore that also needs to be ensured and again many times what we therefore, do is we do experiments in 3 4 different modes. If I can explain oscillatory shear data for a material I will do steady shear and try to explain. In all of the features we should always examine whether the material microstructure has any features which will lead to some of these artifacts. So, you always need to always carefully look at the material microstructure and the possibilities also.

Student: Sir we can do that for some time we are doing the experiment at constants here.

Right.

Student: Initial value of eta will be higher due to (Refer Time: 32:48).

Right.

Student: It will not take that one where we are.

Yeah.

Student: Getting the (Refer Time: 00:00) value.

Right. But if let say you are examining non linear visco elasticity you would want to take the data of eta going increasing and decreasing right. We said stress growth.

Student: Yes.

So therefore under those cases you want changing value of viscosity as a function of time to analyze material behaviour. Now whether that eta as a function of time is it due to material? Or is it due to transients? That question needs to be answered for your.

Student: (Refer Time: 33:20).

Benefit. When we are dealing with steady state yes we wait for values to become constant we wait long enough.

Student: yes.

Yeah.

Student: Sir how can I remove the shear banding.

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It is not possible to remove it. It is a material response. So, actually the shear banding material if you from a theoretical point of view if you look at stress versus strain rate, if we have this kind of a behaviour right if a fluid if a material is able to have this kind of a behaviour what we have is at one particular stress it is possible that there are two strain

rates that the material itself. In such materials there is a micro structural reason. So, in this region there is a orientation and therefore, material viscosity goes down suddenly and higher strain rate is possible. So, so the material it under some conditions you cannot prevent shear banding. So, it is a material feature therefore.

Student: That will be some exceptional (Refer Time: 34:30).

Yes yeah yeah. So, not all them not all material show sheer banding some class of material show sheer banding.

Student: That is you have gave question here.

ah

Student: In the assignment you gave a question.

Yeah which is related to shear banding.

Student: Yes sir.

Yeah yeah.

Student: Sir how we know if shear banding is (Refer Time: 34:46).

You that is what there is no one easy way to answer that one the easiest way is of course a flow visualization, but it is not always possible especially you have yield stress fluids.

stduent: Ah.

And shear banding and all these features are similar. So, it is not always a trivial way you can answer this question whether there is shear binding present in the material or not.

Student: (Refer Time: 35:09) this is expected one (Refer Time: 35:11) some.

Yeah no there is some materials may not be yield stress and then still they will for shear binding. In case of yield stress what you will see is no flow end flow you will generally not see 2 different strain rates. So, therefore, the feature in experiments are you seeing because of yield stress yielding phenomena or is it because of shear banding many of these are all mixed up and unfortunately or fortunately many of the colloidal systems

will show both. They may show shear bending and they may show yield stress also. So, it is not always easy to say which one is the predominant feature.

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Flow problem in cone and pla	nd plate geometry sie geometry
Assumption	n: isothermal
<ul> <li>Viscous h</li> </ul>	neating
	orm heating / cooling
sample?	
What are the	conditions for possibility of temperature differences?
How do we ex	xamine whether temperature gradients exist?
How do we ex	amine whether temperature gradients exist?

Ok so, with this we come to the last example which is the isothermal case right. We have always assumed temperature is uniform. We are saying we are dealing with high viscosity materials. So, that can lead to viscous heating and so therefore, it is possible that the temperature may not be uniform throughout the sample and this can easily lead to errors in measurements. So, with that we have finished review of one geometry and all possible artifacts and it is very clear that there is no one recipe for saying that I have avoided all these artifacts. We must always examine our data carefully to ensure that there is minimum influence of these artifacts on the experimental results