Mechanical Characterization of Bituminous Materials
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Module No # 05
Lecture No # 25
Dynamic Shear Rheometer – Part 1

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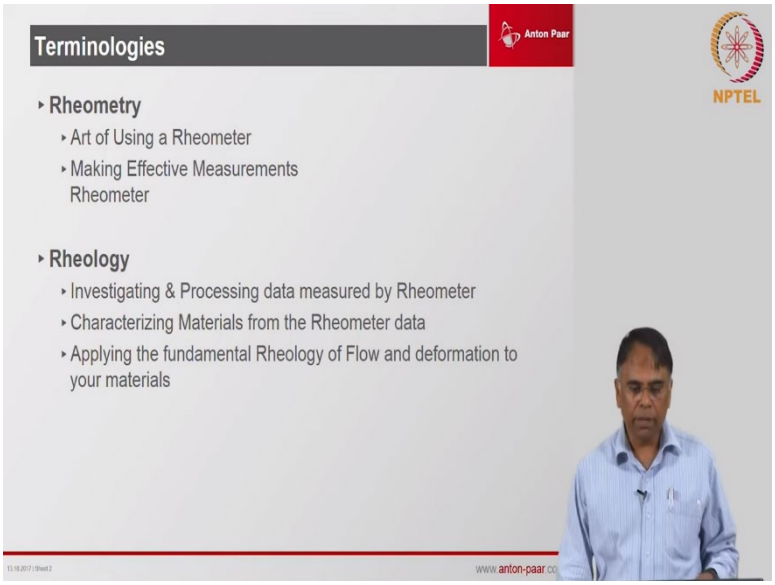

Hello everyone, will start with the basics of a rheometry. The word of course Rheometry is the slightly different from what we hear in terms of rheology. So, the terminology which is use generally for rheometry is basically how to use rheometer to generate you know good rheology data. And how we can really make effective measurements using a rheometer. So that is what I define Rheometry as and Rheology is generally investigating you know bulk properties of material by measuring viscosity, modulus and many such related parameters to investigate you know the properties of the material with respect to different parameters.

If the parameters can be based on the flow of that material or it can be also based on the deformation of the material or it can be combination of both these two. And we are basically going to characterize this material in terms of the flow and deformation behavior ok. So that is basically when we discuss about rheology it is a data which we are producing and then inferring the materials property you know based on this particular data that we have produced by measuring on a rheometer.

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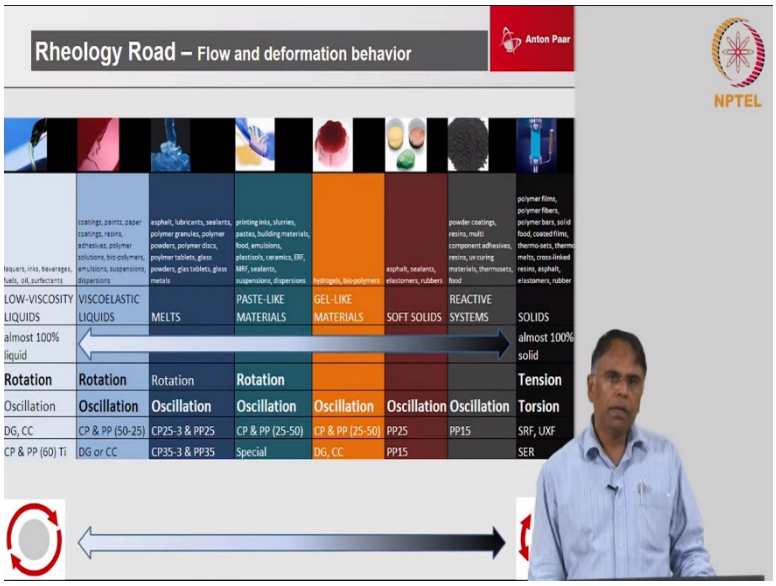

So, let us take a short look at the complete spectrum of material that we are going to you know measure using rheology. So, if you see the left side of the road you have very low viscosity materials, or I would say these are ideal Newtonian materials. So, these materials are very simple structures fluids like water solvents. And on the right side you can see materials which are absolutely solid like steel at room temperature for example would be a perfect solid.

But in between you see lot of different type of materials. These materials are actually a mixture of viscous as well as elastic part. As you move from the left side of the rheology road towards the right side you will see that the materials become more and more complex because if you take a pure viscous material it is easy to characterize at material by simply measuring viscosity. And we can say that viscosity of this materials will be constant irrespective of how we are deforming or flowing them.

And of course, the only thing that changes the viscosity of this Newtonian material are, is the basically the temperature ok and to a certain pressure but temperature will be the main criteria. But as we move towards the viscoelastic material as we have both the properties inside that material, then the materials are more complex to measure because they will have different viscosities at different deformations. And that is what we have to really measure.

And we go when we move to what we call as viscoelastic liquids towards viscoelastic solid ok there of course we cannot characterize materials simply by you know steady shear measurements ok. So, from viscoelastic from viscos material to viscoelastic liquids ok you can easily do measurement by rotational measurement itself. So, it is steady shear measurement as we call it but we move from viscoelastic liquid to a viscoelastic solid we know another can really measure it with the simple steady shear.

In that case we have to apply different techniques which is basically called as dynamic oscillatory measurements ok. From that only we can really measure both this property you know the viscous properties and elastic properties. The reason is if you are trying to apply steady shear material to viscoelastic solid ok you generally will break the structure and this steady shear measurement would always go into what we call it as non-linear region of measurement of this materials ok.

But we want to have both the information the linear in the linear region what how these materials are behaving and what is happening of course also in the non-linear behavior. So, if you see on the bottom of this slide you have the rotation and oscillation. So, oscillation measurements can be done for any type of material ok. Whether it is a viscous material viscoelastic liquid, viscoelastic solid or even a solid.

But if you take a rotational measurement it is limited to, I would say viscous materials pure viscous materials and viscoelastic liquid and maybe very you know I would say very soft viscoelastic solids also. But very small part of that not the full range because if the elasticity more when I say elasticity more, I will explain you in more details how I can define and say that elasticity more. But for those material it is just impossible to do rotational measurement ok.

And at the bottom of that you see different types of geometry that are usually used for measuring this this on a rheometer. So, you have if you take very low viscous material and of course you require larger surface area of interactions. So, for that you will require coaxial cylinder geometry. And then assume move towards more stronger structures ok then you will require smaller surface areas. So, you move towards from you know coaxial cylinder system to plate plates or cone plates.

And this cone plates sizes also becomes smaller and smaller when you go towards viscoelastic liquid to viscoelastic solids. So maybe during the viscoelastic liquid measurements you will you may require 50 mm geometry. But for a viscoelastic solid you may require 25 mm geometry. And further down like if you go to very strong solids like you say thermosets you are doing a curing experiment. Then generally we use PP you know parallel page geometry like 12 mm, 8 mm types of geometries. So, they become smaller in size because the interaction required in this interaction that we measure in the small surface area are sufficient to characterize and get the required information.

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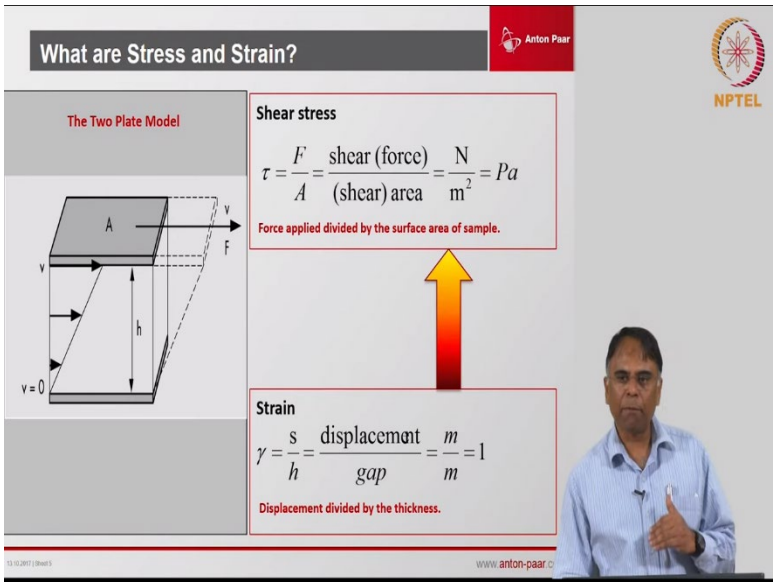

So, what we are doing is basically we are applying to in the measurement to the sample either a stress and measuring a strain or applying a strain and measuring stress ok. If you look at the two plate model what is important is in this measurement is you should have a defined gap between two surfaces. The sample is basically getting shared between these two surfaces. In general, these two surfaces are parallel ok and they are one of the surface is in a standard condition is stationary and they one of the other surface is moving ok.

So, when we are giving a deformation ok. We define this strain, which is the nothing, but the displacement divided by the gap between these two surfaces. And the velocity divide by the gap will be the shear rate. So, this strain is the deflection angle, or the displacement divided by the gap and the shear rate is nothing but the velocity divided by the gap ok. The stress induced due to

the application of the shear rate or the shear stress is nothing but the torque which is experienced by the sample ok.

So, when I am moving this upper plate with a certain displacement or with a certain speed. So, I will require certain amount of torque ok to get that define displacement or define speed. So that torque which is measured ok multiplied by the divided by the surface area sorry would give you the stress value. So, the torque divided by the surface area is your stress ok and your velocity by gap and displacement by gap will give you shear stress or the shear strain respectively.

So, this is what we see here stress divide by shear rate and here displacement by gap and of course this is how basically the measurements are going to take place on a rheometer. So, in the rheometer you can apply either the shear stress and measure the shear strain or you can apply the shear strain and measure the shear stress.

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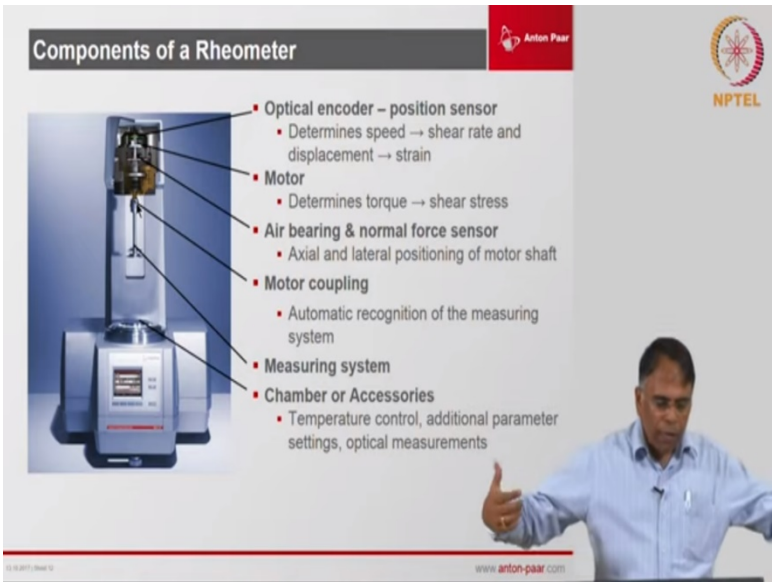

So how does the rheometer does this? Ok so if you look at the hardware of the rheometer, the rheometer will have a motor ok. And this motor is going to apply either the stress or the strain. So, in the motor basically you will have a coil which is basically given a current ok and because of the current basically the rotor which is generally a magnetic rotor will rotate ok. So, you will generate sort of drive inside the motor using the current ok. Now there can be different type of motors also ok but let me take an example of a very simple direct current motor ok.

Direct current motor will have multiple coils so it will have for example twelve coils and similarly in the rotor it will have twelve magnets ok. So, these twelve coils basically you can make these twelve coils in such a way that you can become north south north south north south north south. So, you keep on giving currents in such a way that you are actually in a way rotating making a rotating magnet. So, you are making these coils you know magnetic field rotate. Now since these coils magnetic field is rotating the magnets on the rotor which is on the center shaft ok. So, it is not connected mechanically to it.

So, this rotor is actually floating in air on what we called as an air bearing ok. The reason of course we would like to have a bearing like an air bearing because we want to have almost zero friction ok. So, we cannot have absolutely zero friction we will have some friction caused by the air, but this air friction is so low that we can easily you know get information of very small structures. The reason we want to have low friction is because we want to measure very small torques ok. And to measure this small torques we should have any friction in the air bearing.

So, this whole shaft with this rotor having these magnets is floating in the air and these outside coils are making this magnetic field move ok. And so, this rotor which also have the magnets so their rotors south will actually follow the north which will actually rotating ok. And similarly, the rotors south will actually you know the rotors north will actually align with the south of the moving coils ok.

So, it is actually synchronizing with this what we call as the rotating flux which is generated in this motor. And that is how we can control small positions ok. If I want to control position which are like into nano radians, I can apply only that much amount of current to generate a very small flux to have that small movement ok. So, you control position ok. so your deflection angle is controlled the deflection angle divide by the gap of the geometry is going to give us the strain.

So, you can control the strain or the speed at which you are changing the position can be also controlled. So, the speed is your velocity divide by the gap your shear rate can be controlled ok. So now since you know how much amount of current you are giving so that is the equivalent to the torque ok. So, this is also being measured ok. You basically measuredly give the current to

really control this movements and control this speed and you keep on measuring also this current ok. And that is equivalent to the torque which is very basically getting induced into the sample.

So, we measure the torque we measure the speed and the position and from that calculate the stress, the shear rate and the strain ok. And then use this into our equations to measure this. Of course, we are measuring each and every point of application. So, we can measure a steady shear or we can measure a oscillation ok and of course when we have the oscillation we are also going to use this vectors of oscillation to measure what is the phase angle between this you know the actual strain where we are applying. And the torque signal that we are getting which is also an oscillatory.

So, the torque vector is basically also used to measure the phase difference between the stress and the strains signal. So, when I say stress signal it is actually torque signal ok. And when I say strain signal it is actually a position signal ok. So, the position is converted to strain and your torque is converted to stress ok. Below so on this motor of course you will have a coupling and to this coupling we can have different types of geometries ok.

The geometries will define actually the surface areas and the gap ok and those are also actually used in the equation of converting the torque to stress and the position to strain ok. And at the bottom we can we will have different types of temperature controls ok. And this temperature controls will be used to control the temperature of the sample and the temperature controls can be of different types also.

You can have a very simple one what you see on the screen this is just of flat plate below that you have either heaters or you have electronic culture elements to you know either to heat or cool or you can have actually ovens which are actually going to come and close around this sample ok. Or you can have a flat plate and a hood on top of it.

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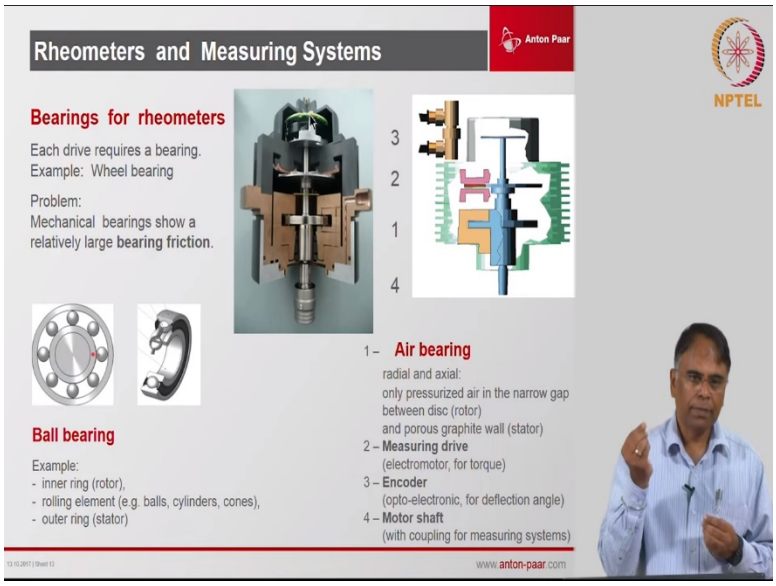

So that can be variety depending on what type of samples you are going to measure. If your samples are going to be very big then you will if your samples are very big then you would require bigger temperature devices. If your samples are very small like you measure between two parallel plates or cone and plate then you require very small simple flat plates that we saw in earlier screen would be good enough.

It is just a cross section of the motor that you see if you see this part over here is your air bearing. So that is where your air is flowing inside and making this whole shaft float into the air ok. And the bearing basically is in two parts. This bearing over here is your radial air bearing and the bearing below over here is your axial air bearing because we also want to make sure your shaft remains absolutely perpendicular does not wobble at all ok.

So that axial air bearing holds the shaft exactly straight ok and the radial air bearing makes it float and rotate on the axis without you know any or a I would say minimal friction over there. And this is your motor and on top of that you have your optical encoder and this encoder is actually measuring the position ok.

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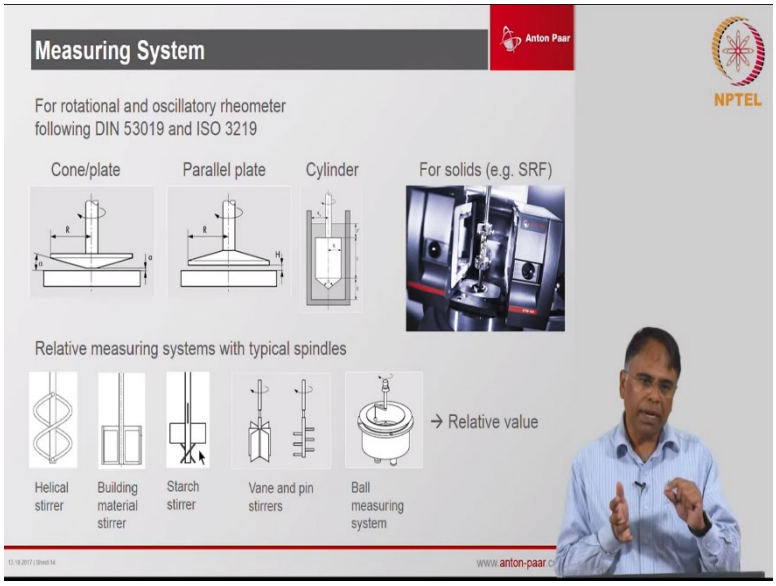

So, there is this is the basic hardware of the motor and these are the different measuring geometries. So, in general the parallel plate, cone and plate are the most common measuring geometry used because they are easy to load easy to clean and can measure a lot of spectrum of material. You know starting from I would say viscous material to viscoelastic solids can be taken care by just you know the parallel plates and cone and plates. For very low viscous material you may have to go to higher surface area like the concentrate cylinder as you see over here ok.

And then of course if you go to very stiff materials like solid bars ok in that case, of course we cannot use either the cone and plate, parallel plate or even the coaxial cylinder system then you will have to use different geometry like what you see on the right side over here. These are solid fixtures or solid clamps ok. So, this material would be then clamped between these two geometries and you will apply the oscillation across the cross section of either sample which is the cylindrical sample or the sample which is rectangular solid ok a pressure point.

There are other relative measuring geometries possible ok depending on again this samples if you have samples with very large particles for examples slurries, cements, motors, marmalade you know with big fruit species something like that then you cannot do it with small gap geometries like cone and plate or coaxial cylinder systems ok. Then you have to go to larger gaps ok.

So, these are the geometries which are going to be used like vanes or stirrer for such type of sample matrices. But when you go to such geometries these are not going to give us absolute values of viscosity and moduli. ok because the standard in equation for defining the proper shear stress and proper shear rate will not apply to very large gaps ok. So, you can get an approximation of that you can calibrate these geometries to get absolute values here.

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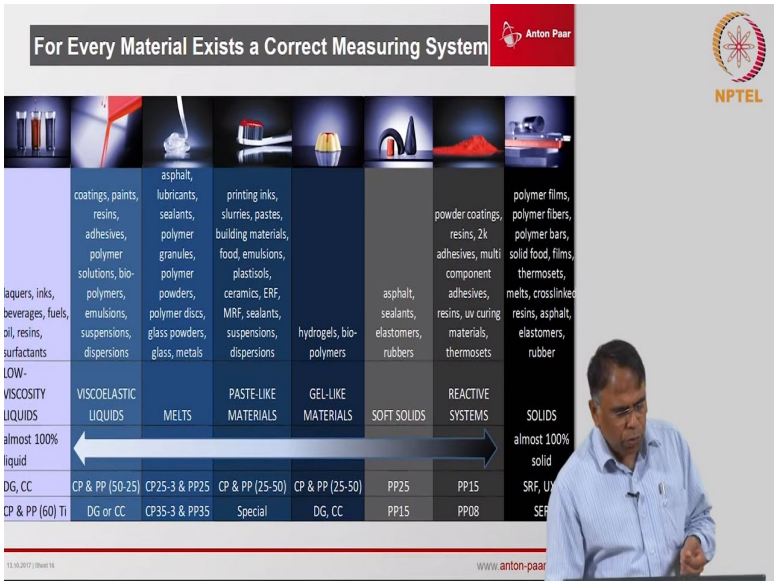

So, to choose you know proper measuring geometry for your samples you can just look at this surface area of geometry as a main parameter ok. If you have samples which are very weak very thin very structures like you know foams for example ok very so they are, the structural strength of this material is very weak.

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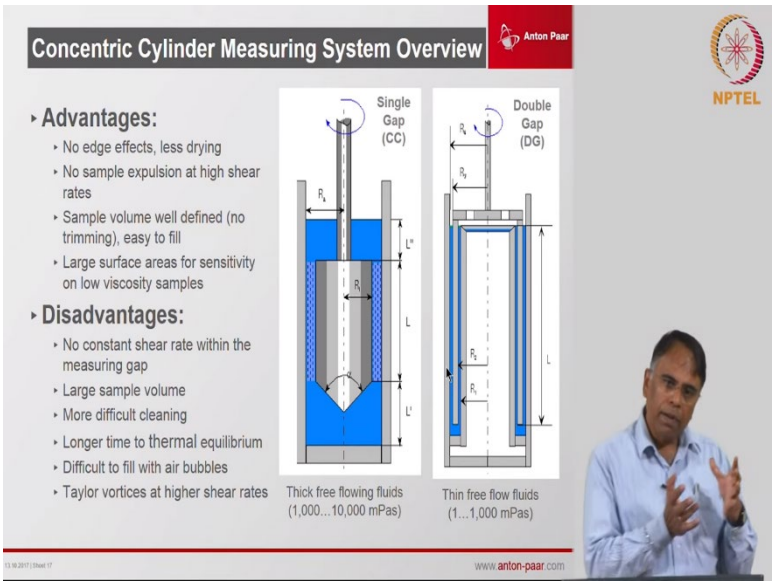

So, for that of course to get a response for this material you require larger surface area of interaction. So, in that case you can use coaxial cylinder systems. The coaxial cylinder systems will give us the largest surface area of interactions. Again, the coaxial cylinder system can be a single gap or a double gap over here. Double gap will increase this surface area even further. So, if you were measure if you measure very small viscosities and you want to measure at very small strains ok then you would definitely require a double gap.

But if you are going to measure lower viscosities at minimum shears or good enough strains values then you can also do it with a single gap. For example, if I want to measure water at a shear rate of 1 or 0.1 it would be impossible to measure with the single gap coaxial cylinder system then you would require to have a double gap. If I want to measure with water at a shear rate of 50 or 30 ok no problem that can be done with a single gap ok. So, it is just a matter of the structural strength and of course matter of what is the deformation if you want to measure at very low deformation then of course you require even large surface areas.

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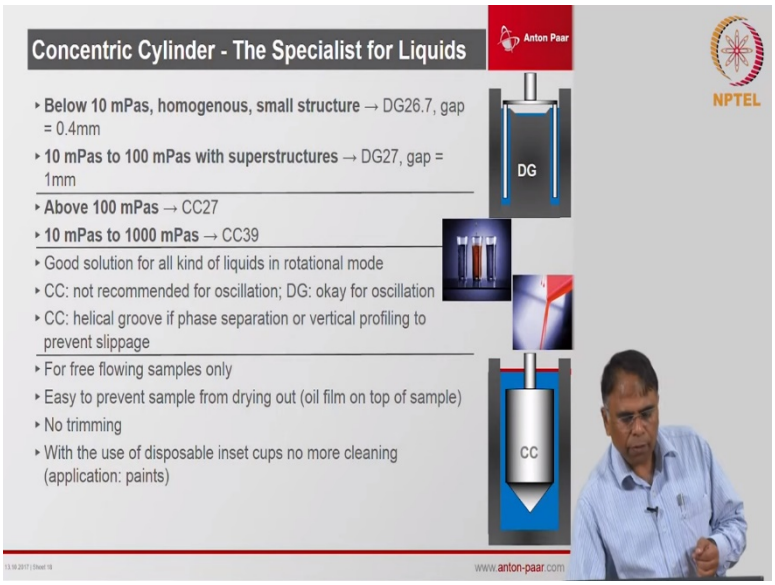

So it is a we call it coaxial cylinder system as a specialist for liquids.

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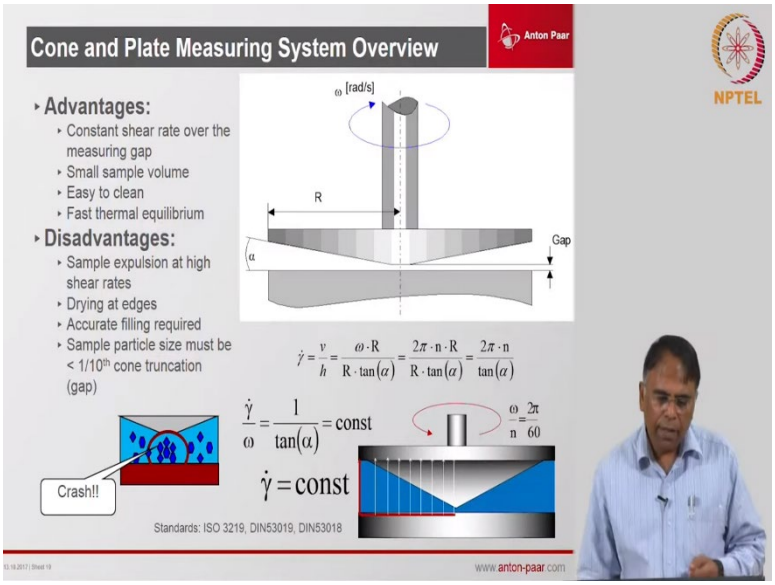

We have the cone and plate which is I would say one of the best geometries which can measure from low viscosities to medium high viscosities most versatile geometry I would say. It has lot of advantages because it has shear distribution across the surface which is constant from center to the edge ok. So, it is very good for it is good accurate measurement of viscosity using a cone and plate ok.

So, it is like a it is very similar to the coaxial cylinder system but coaxial cylinder system will require larger amount of samples and they are also difficult to clean ok. But cone and plate the

sample volumes are smaller and they are very easy to clean the sample loading is also very easy ok. The only disadvantage in cone and plate is if you have samples with big particles then it is not possible to measure.

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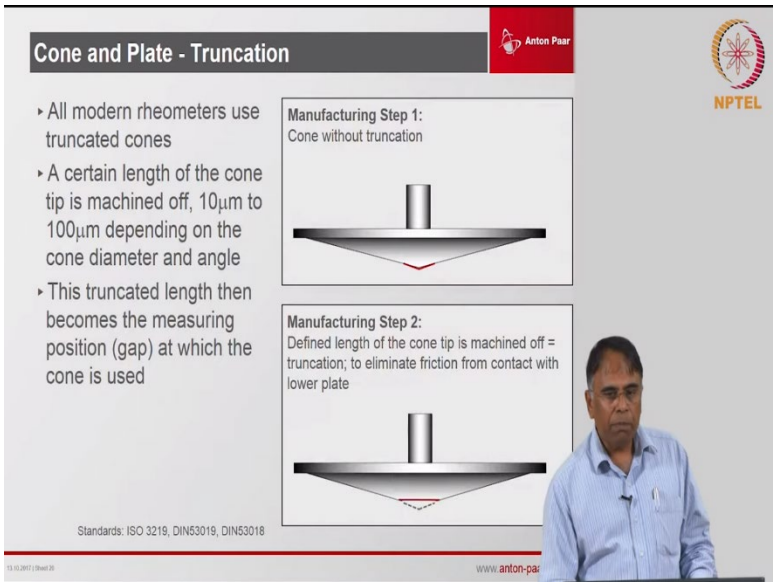

The reason being if you see a cone ideally a cone would have a very sharp tip at the center and the tip would actually touch the bottom plate ok. But if you if you manufacture that cone in that way the tip is touching then we will start measuring the friction between this you know touching point of the cone to the plate ok. So what we do is we trim of this tip ok and whatever we trim of that is measured ok and that will become our gap ok.

And now this is very important that we maintain this gap ok. If this gap let us say I have trimmed off 50 micron or 100 microns ok then I have to maintain that gap. So that virtually that cone when it gets completely still touching ok so that is why the maintenance of the gap is very important. Now but if I am going to do a temperature sweep for example that case my gap is actually changing physically because there is going to be expansion this is this are made of metals.

So, these small expansions also are going to contribute to a very large error in a cone and plate. So that is also the reason why in a cone and plate we cannot do a temperature sweep. You can only measure at isothermal conditions. So, if you are measuring at 50 degrees so you do this zero

gap and you set the gap at 50 degrees and measure at 50 degree that is it. You cannot then change from 50 degrees to 40 degrees without doing a zero gap again ok.

So, it is very important in a cone and plate to measure at isothermal conditions only ok. And you cannot measure with larger particles because these particles can come at the tip can get you know trapped at small gap and then you are actually measuring only those small amounts of particle which are trapped over there and you get problem. So, it is I would say not a best geometry for measuring slurries or dispersion with large particles. For homogenous fluids it is best ok no problem at all ok.

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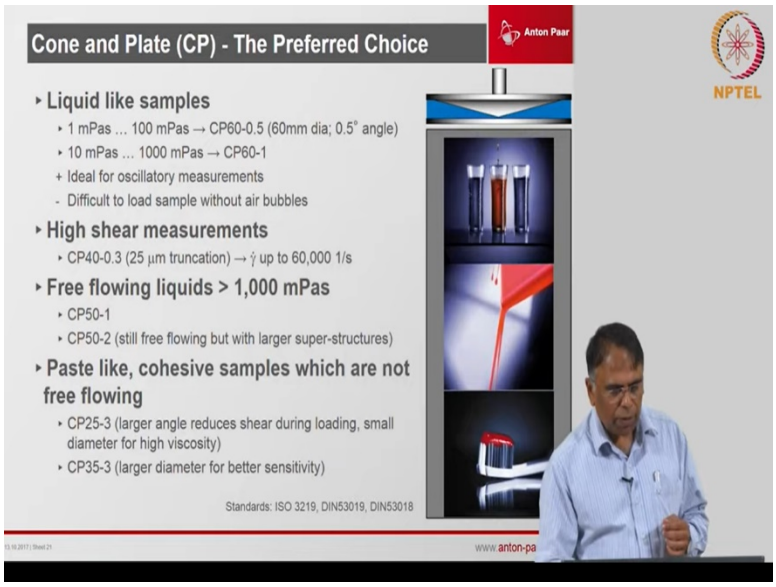

So, it is one of the most preferred choice of measurement and you can have so many varieties of geometries. If you want to measure very thin fluids you can have a small angle, if you have very strong structures you can measure a cone with large angles you can have different diameter different angles. So, variety of combination of cone and plates can be possible to suit any type of you know very wide variety of material I would say.

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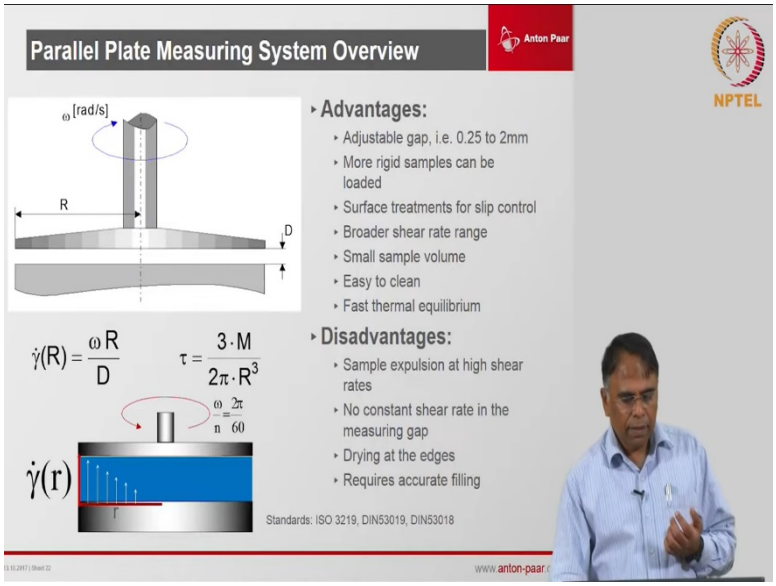

The other geometry besides the parallel plate ok it is one of the most adaptable geometry after cone and plate and the main advantage I would say for the parallel plate is easy sample loading and of course you can do temperature sweeps ok. Here we can do temperature sweeps because even if the gap is changing ok we can measure at any gap we can actually use that gap calculate.

Let us say start with 1 mm gap ok now as a temperature is changing let us say the temperature is increasing then the expansion is happening so the gap is actually becoming smaller. We already can track that by knowing the material we know what the coefficient expansion of that material is and based on that we can know that how much amount of gap is getting you know lowered and lowered ok.

And so that is actually used for calculations ok and that correction is made very easily with that. That correction cannot be done with the cone and plate ok. But with the parallel plate it is very easy to do a temperature sweep.

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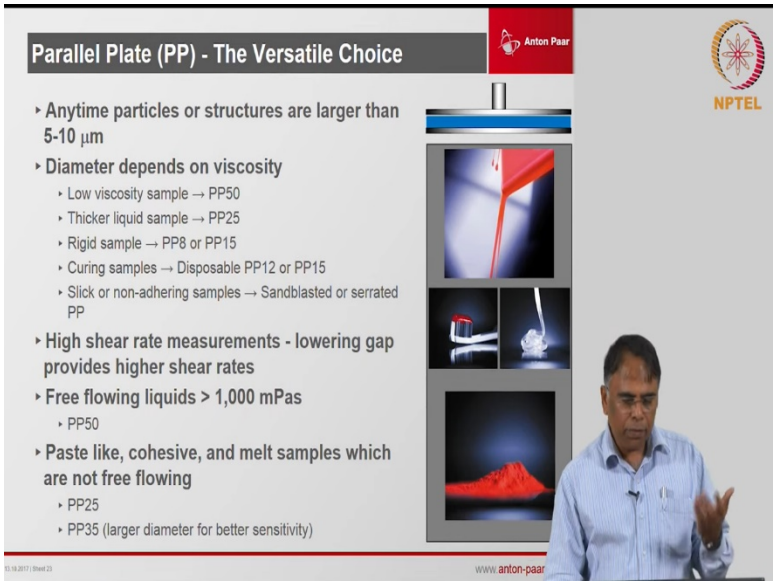

And also, since the parallel plate can be done at variety of gaps you can use 1 mm gap you can use 0.5 mm gap you can use 0.25 mm gap ok. So, the changeable gap basically also allows you to accommodate different type of samples. If you have samples of say with have particles as big as 10 microns then I can easily go to 10 times of that gap which is 100 micron and so on so. I can actually adjust the gap based on the maximum particle sizes which have present in the sample.

In a cone I do not have that option. I have to work at a fix gap which is equal to the truncation that I have done on that cone ok. So, but in a parallel plate because of the variable gap ok you are able to also accommodate samples like dispersion slurries with large particles and of course it has the biggest advantage of doing a temperature sweep. So, for a temperature dependent experiment the parallel plate is really best, of course you can do a temperature dependent experiment also on a coaxial cylinder system because that is also free of you know any artifacts which are cost due to that temperature.

But the mass of the coaxial cylinder system is so big because the volumes are so big over there it is not the preferred because you know it takes long time for the sample to get the temperature that you are giving over there. But parallel plate sample amount are very small typically 0.5 to 1 ml is the sample volume of most of the parallel plate geometries. And to heat or cool such type of sample mass is very simple specially if you are working at a gap of 1 mm and below then even a bad insulator of heat can be easily you know have a very good temperature control. So you can

go you can really do a very decent temperature sweep temperature dependent experiment using the parallel plates.

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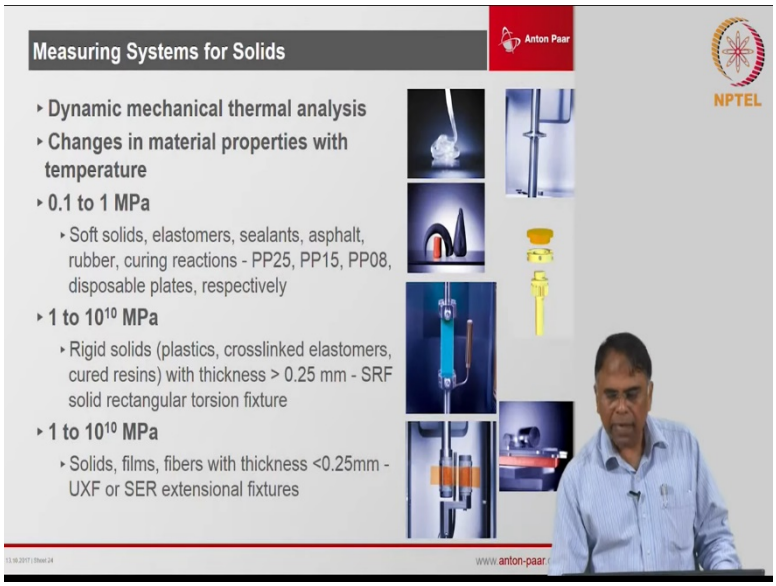

Then of course there are measuring systems for solid which I had told you that they are simple clamps ok. And these clamps can be of variety of type you can have solid rectangular fixtures for rectangular samples. You can have circular fixtures for cylindrical samples, and you can also have a holders for films and fibers also. So, this are something like this over here.

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In the most popular one are the solid rectangular fixtures ok which are used for variety of solid samples polymer composite or you know asphalts reinforced materials, laminates and so on. And

then of course you have this whole gambit of relative measuring system which are used for measuring very difficult samples you know like large particles slurries, cement, motors, concretes ok. So, you have cylinders which are profiled or you have vane geometry you have helical geometries or you can have ball measuring systems many of this use the typical DIN equation or modified DIN equation for calculations.

And many of them use a different set of calculation like there is ball measuring system which you see over here with basically uses the Mueller direct equation ok for doing the measurement. So, it has to be back calculated in terms of the CSS and the CSR factors for measuring the viscosity and modulus.