Mechanical Characterization of Bituminous Materials
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Module No # 06
Lecture No # 26
Dynamic Shear Rheometer – Part 1

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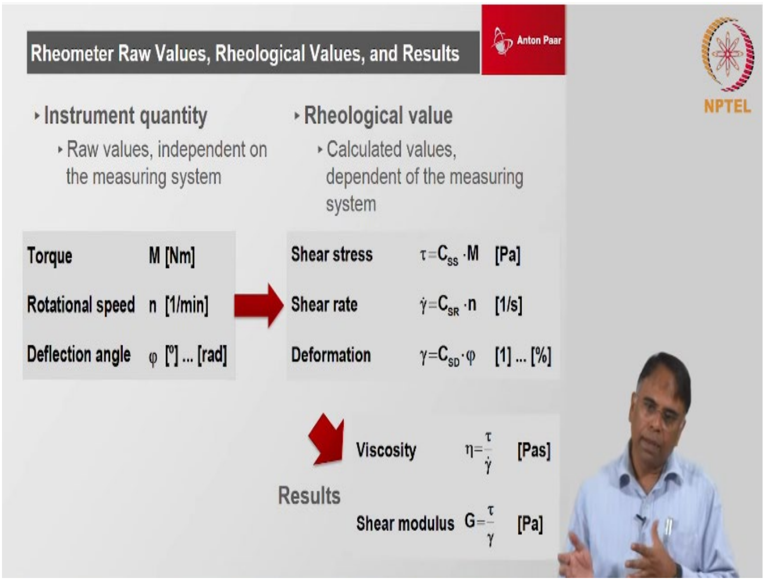

Detecting the problems. So when we are measuring the stress we are going to apply the you know certain parameters of the basic things that we are applying in the all the test is strain measuring the stress ok. And we have to see ok there is a noise data for example okay. So, we have to understand whether the you are measuring on a rheometer in that range of the rheometer ok that is the first thing.

So, you should understand the range of a rheometer. What is the minimum torque that rheometer can measure? What is the minimum displacement that rheometer really apply? Okay so these are important thing to notice a very basic thing I call those error cockpit errors. Because those can be easily identified by the person whose is measuring.

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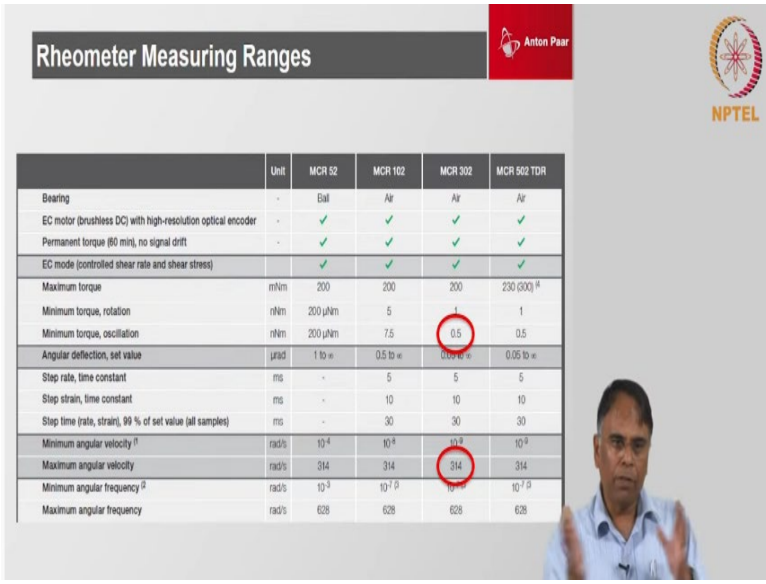

So when he the measure if we just looks at the specification of the instruments and he knows ok if I am measuring now the first data points of this measurements I am getting lot of noisy data. So first thing you should do this check the torque and see that torque is really in the range of the instrument many times the torque is so low and it is almost either lower than the capability of the instrument or very nearest to this lowest capability of the instrument ok.

So obviously you will not get a very good signal noise ratio that is you know edges of the measurement. If I am measuring at low torque or if I am going to measuring at high torque also okay. So, if I am measuring at high strains and if it is already taking to the maximum capability of the instrument then also of course the data will not be good. It will max out okay it will saturate okay.

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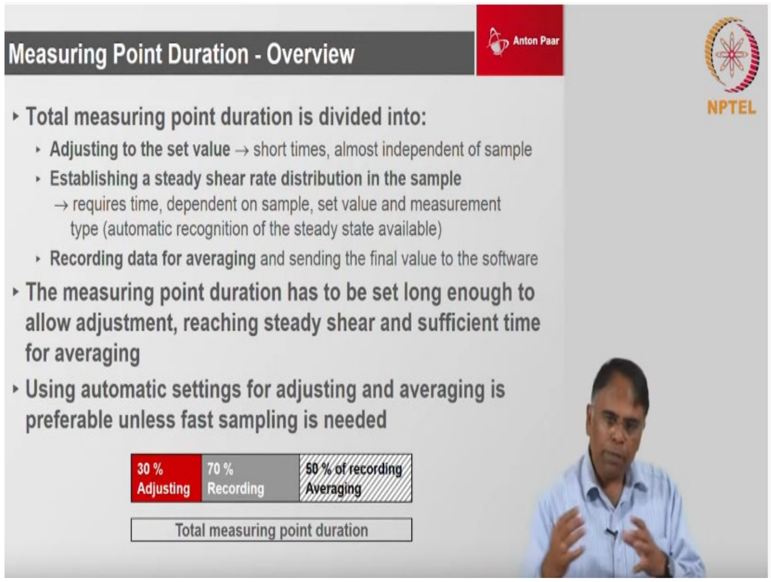

So, this is the first thing we should always observe you can see approach the low-end area okay specifically we can see the signal to noise ratio it is not bad and quality of the data becomes bad okay.

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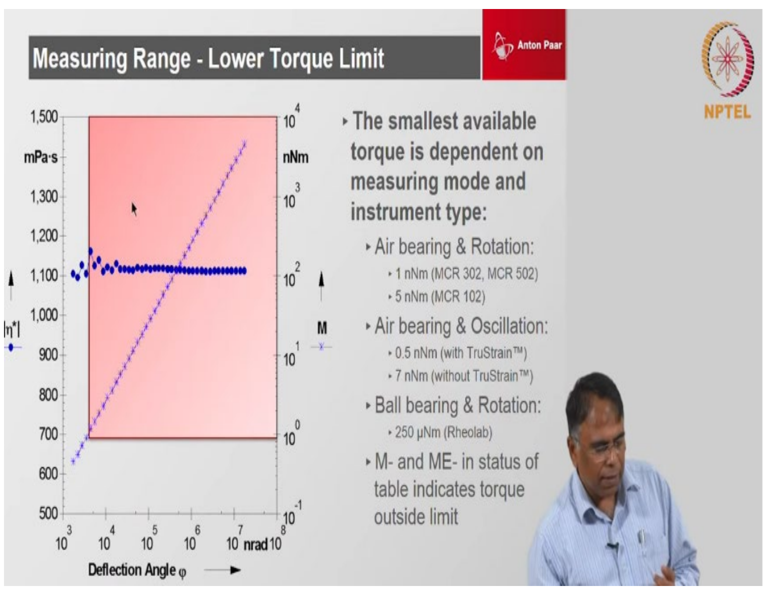

So always look at the first thing that we should look at you see the torque values at are the coming.

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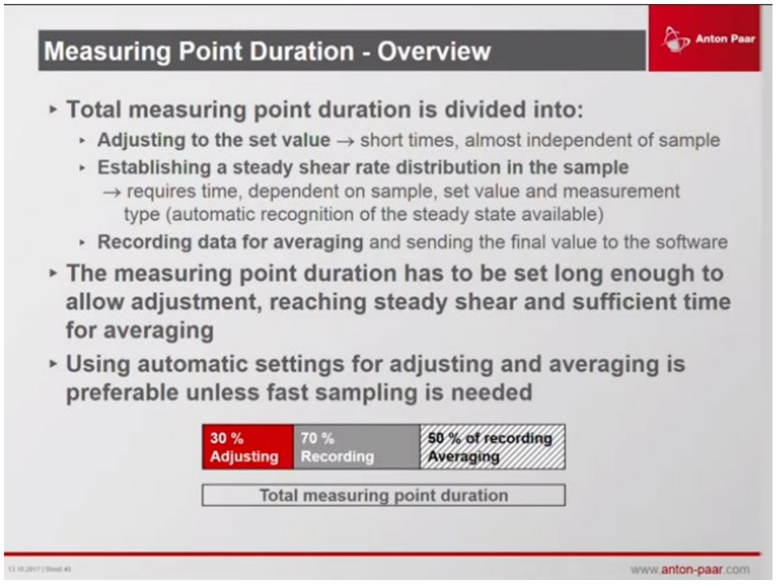

And all the rheometers basically they look into you know they will have the facility to plot even the torque so just can you place the torque the plot able and look at the values. And many of these you also tell you as certain status value like m- for example that is indicating that torque is too low okay. If you look at the measuring point duration specifically you should look at the studies prior experiment if I am doing a shear rate of 0.01 okay.

The unit of the shear rate is reciprocal of second. So, 0.01 shear rate actually will require that the event to complete will require the reciprocal of 0.01 which is 100 seconds. So physically that event will record 100 second to get over. So, if you major over these 100 seconds that would the minimum okay. For this event to get over plus you will also certain require additional time because physics is also there in the electronic.

Because the electronic of the instrument, the mechanical properties so that flow of data all these consuming time okay. And this when we are looking at microseconds and milliseconds timer also adding up okay. We should if you look at measuring point duration first 30% of time is used to apply and that you strain or stress that you have defined okay. Then the next 70% is actually used to record the torque over the complete event and then after that 50% of time is used for averaging out the data.

Because you get a lot of information the wave form is you know actually having 256 and 512 data points 512 data points of the strain and 512 data point of the stress they are arranged, they are vectored and then phase shift is measured between them. So, all these of course this is happening very faster in the electronic but still it is requiring few milliseconds to microseconds of time. So, these all basically has to be considering when we are measuring specifically experiments but of course many times you would like to measure transient formation.

You would like to understand how this you know let us find applying the shear rate of 0.01 so from 0 to point achieving 0.01 what is happening okay that also you can major okay. That is the transient information. So, in that cases when you are doing experiment of course you can take away this adjusting time that actually put adjusting time to zero. So, you can actually put the averaging time to zero okay and just use only the continuous recording okay so that is also possible.

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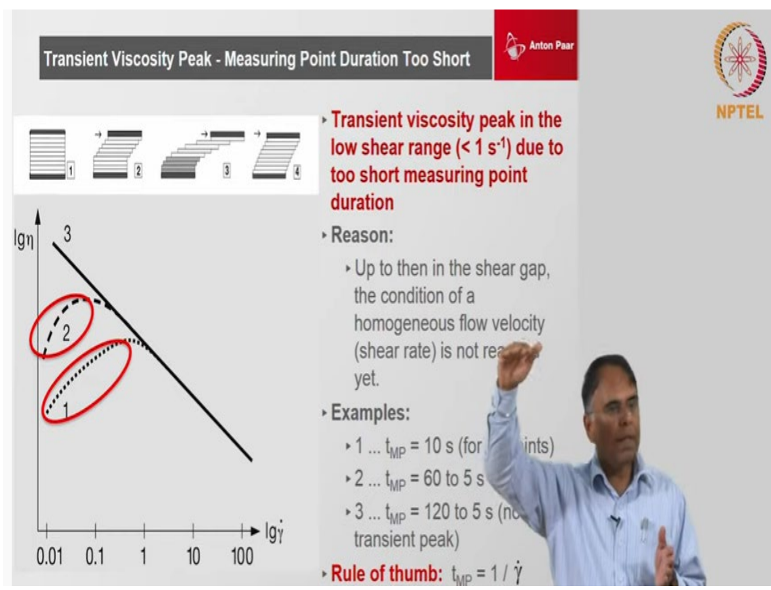

But if you want to do a non-transient experiment then of course you will require to get full you will have to give this sufficient time to make sure this complete aspect of adjusting recording and the application of the input parameter is done correctly over here. So, this is an example over here, see an example of a shear rate sweep ok if you have a shear rate sweep starting from 0.01 to 1 in the transient, you know with very short time you can see this banana curve ok which is happening okay.

So, you cannot really very get accurate low shear information at a lower side. So of course, you can if you want to major transient and only measure at 0.01 then, see how it is developing okay and then of course record the final developed version also okay.

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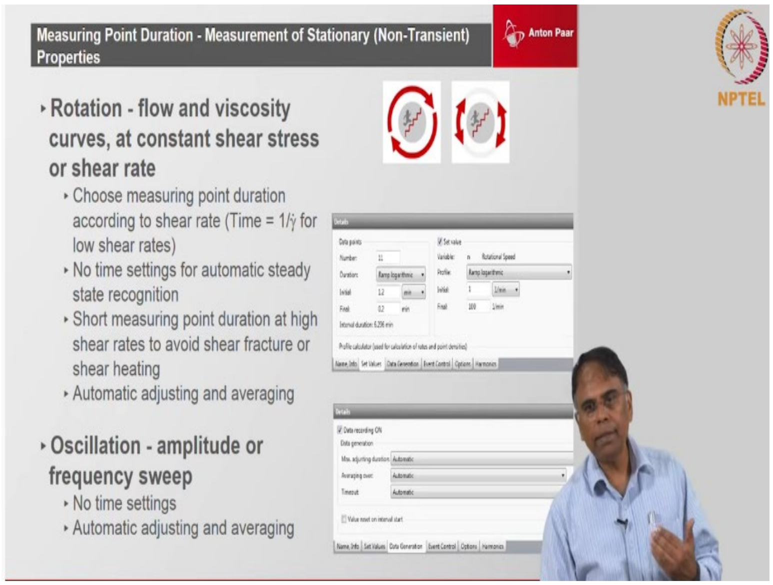

So, this is what you should really understand so when you are setting this parameters of shear rates okay take care of the timings that you are using if you are timing from low speed to high speed or low shear to high shear okay. So, you take a reciprocal of the time also let us am staring from 0.01 shear it to 100 shear it okay. Then I can start from the measuring time point see 120 seconds down to 5 seconds or 1 second we got 100 shear it overall in a fraction of second.

Whether there is 0.01 seconds require 100 seconds plus time okay. For oscillation and amplitude you know whether it is a amplitude frequency generally we give no time settings okay. But again, this is a non-transient experiments if you want to still collect transient data you can collect it at faster cycle time or very shorter cycle time.

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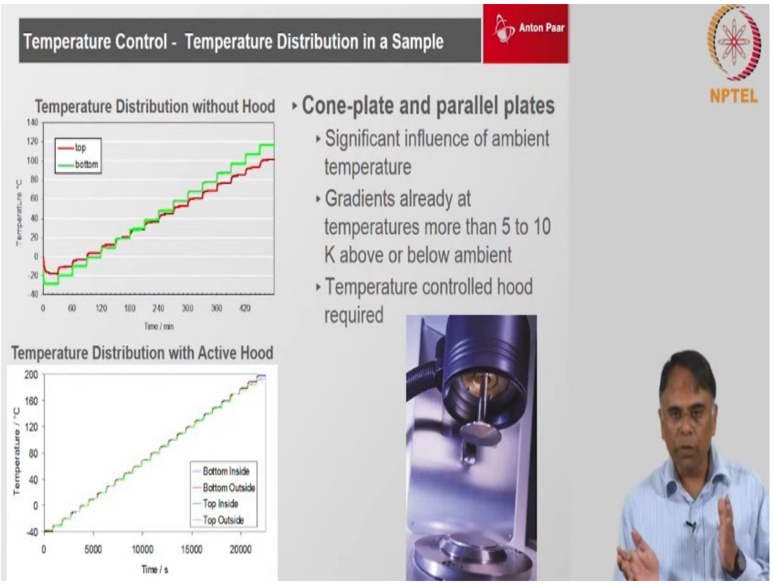

That case you can of course you can make these you know the adjusting time and averaging time to zero okay. You can still get very good measurements done using that parameters. Temperature control we have to also consider equilibrium times okay. Because the hardware's the geometry can heat up very fast, but the sample may not be heating up that fast. So always make sure that your sample is following the temperature steps that you are giving okay.

It is important to have the temperature control such that you do not have gradients in your sample. So, it is always better to have the heating, or the cooling done from all sides of the sample. So that is the reason only a flat plate may not be sufficient you can also have a sort of hood on the top or you have a nice enclosed convection camber which basically make sure that the heating is all around the sample and there are no gradient within the sample.

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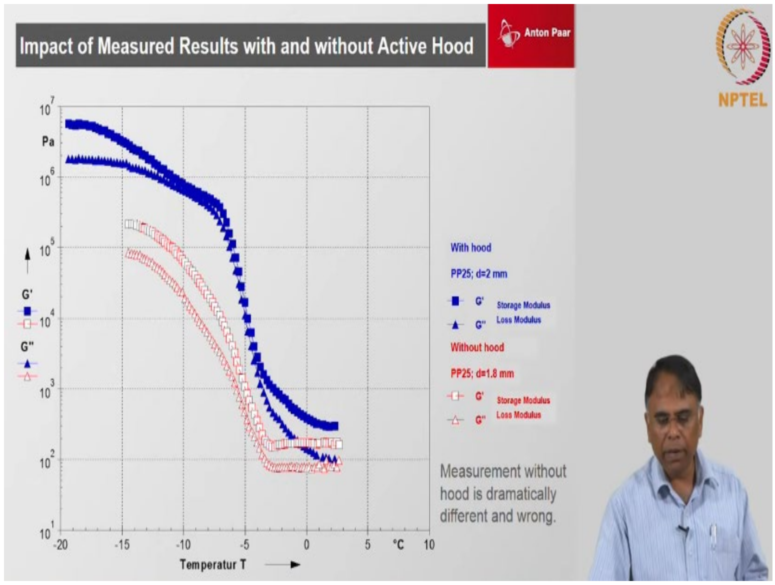

Example of the measurement between the same sample measure with the hood and without the hood you can see very big difference it is look like two different materials all together okay.

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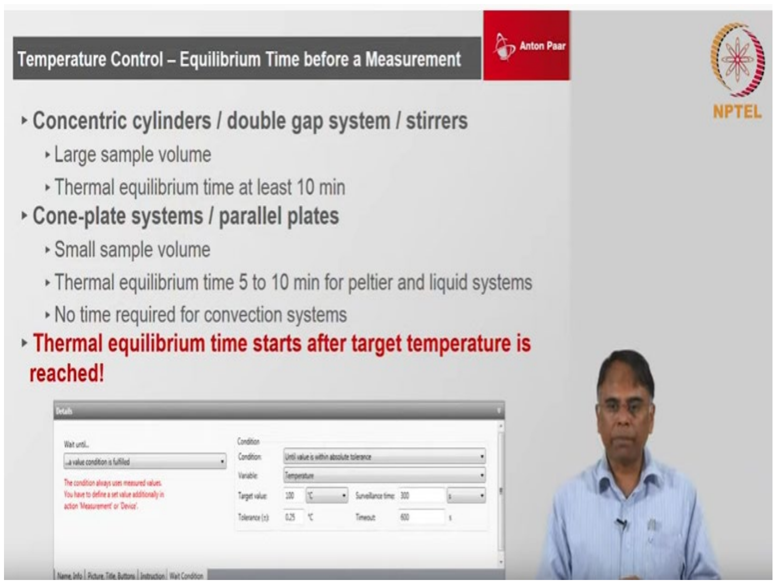

Also, if you look at equilibrium time from geometry to geometry, they are going to be different if you have larger geometries like coaxial cylinder systems you have to give much larger times because you have larger masses and the sample volumes are also bigger. But if you take a parallel plate and cone and plate you can marginally have smaller equilibrium times. Because the volumes of the sample are also the smaller and the geometry themselves are also not having that much high mass.

So the temperature to heat or the time to heat up the geometry than the sample will be much shorter than the parallel plate and cone and plate compared to that in a coaxial cylinder system or if you are doing a measurement with a solid fixture for example the samples are going to be quite big 50 mm length and 40 mm length of samples and those will take of course more times.

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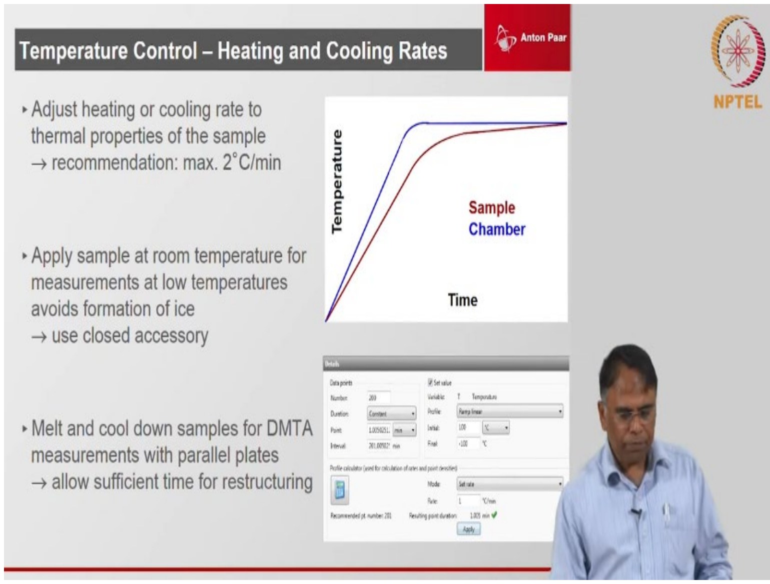

Even though the geometries in those conditions are not very big, the clamps are very small but the samples itself are quite big and of course they are mostly solid samples, so not flowing also so in a static condition so the heating time and cooling time of those geometry of those samples are going to be also very slow okay. So typically, you take coaxial cylinder systems or solid fixtures, temperature rates of 1 degree 2 degree C per minute and maximum of 3 degree C per minute are good enough okay.

Do not go higher than that but may be for a parallel plate like PP 08 or PP 25 you may, it may be possible to you go at a higher rate of five degree C per minute also, the sample can still follow that. You can see heating and cooling rates setting over here that can be done from the, here we can see how the sample is following, the chamber is going very fast, but sample is really not able to take. If you have sample with chamber having very low conductivity thermal conductivity definitely you will have to go to much lower heating rates you know than your sample.

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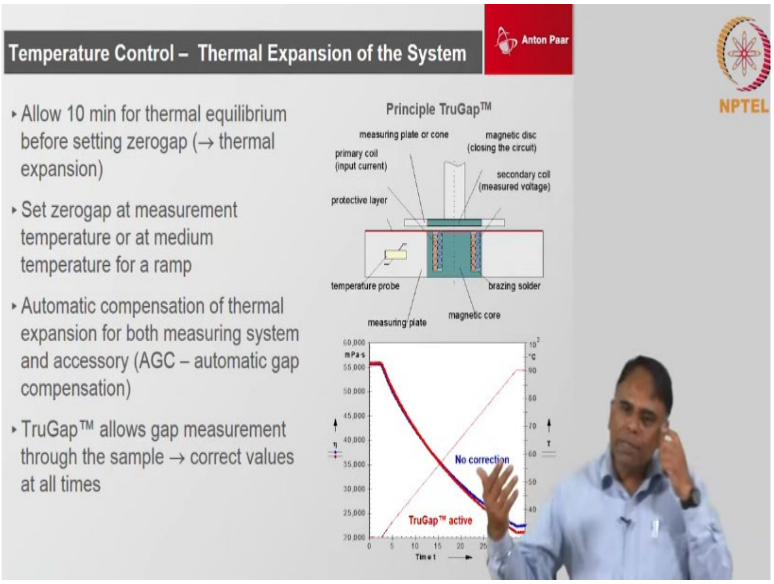

Also, the thermal expansion of this system have to be consider if you take cone and plate, then of course you cannot do a coaxial cylinder sorry you cannot do a temperature sweep with a cone and plate okay. There is a possibility of course which special geometry okay which will have senses in upper and lower geometries which can track the gap accurately then you can even do a temperature sweep on the cone and plate.

But these geometries are extremely costly, and they are only these sensors are only possible to be used in a limited temperature range. At very high temperature these sensors are magnetic in nature. So, they lose their magnetic properties, so these sensors are not useful. So, if I am going to use within say 230, 240 degree centigrade there is still possibility to do cone and plate measurement with temperature sweep using these types of geometries.

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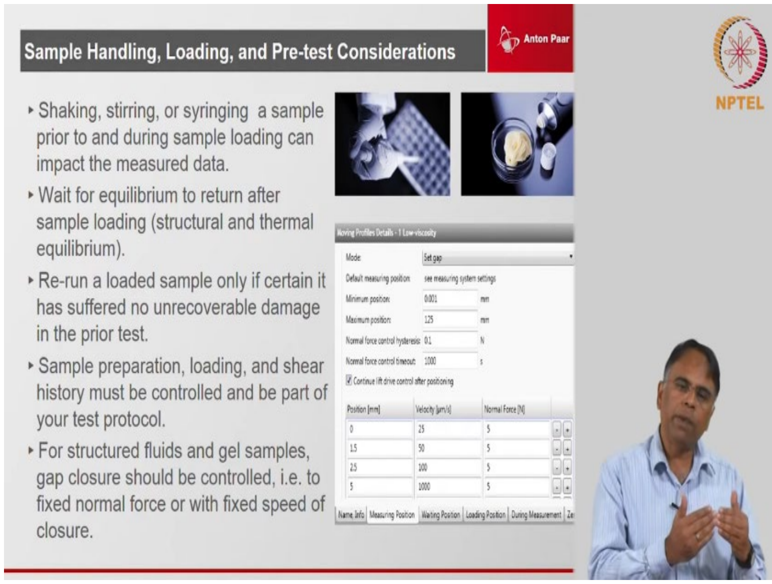

So, always when we have taken care of the right geometry for a right sample right testing parameter also understood what is really happening in that test? Looking at a torque you know setting right parameters to get the torque in the range over there at the same time you should also take care of the basic sample preparation.

So, when you are loading the sample, specially viscoelastic liquid samples to make sure that there are no macro bubbles in the samples. The easier way to load a sample is to load it by volume if you are able to do pipette that sample okay. Because if you have liquid samples which are pipettable you can load exacts volumes, you will not need to have any trimming every geometry will have a defined volume. So, you can easily take from any rheometer software that there software will be able to give you the sample volumes okay.

So, you can load that but of course those sample which are not easily pipettable and you cannot really take them by volume then you have to put those samples first with the certain extra volume, then the extra sample will have to be trimmed of okay.

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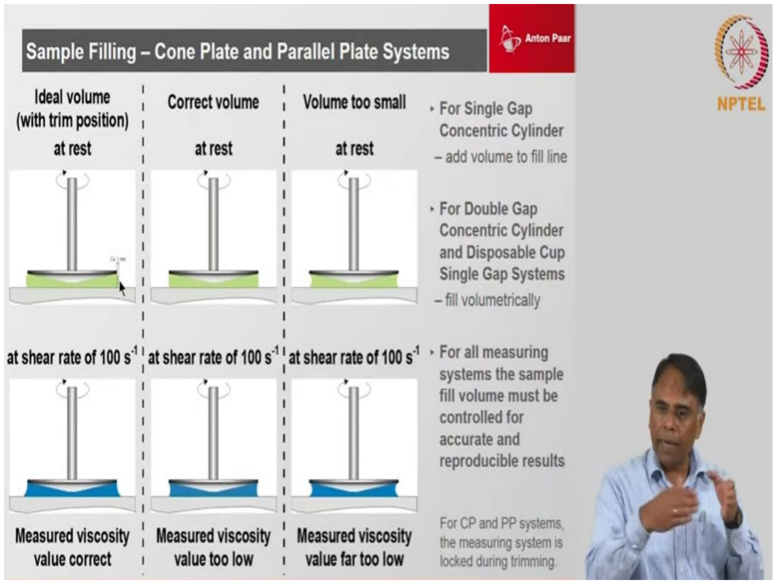

This is something which is always important so if you have a correct volume it is very easy to load and when you visually see the loaded sample make sure that you see a small bulge coming out of the sample specifically when you are loading in a parallel plate or a cone and plate okay. So, the sample should not have a bulge inward like this okay but should have a bulge something like this okay. The reason being specifically if you take geometric like parallel plate the stress in a parallel plate is concentrated at the edges okay.

So, if you have not properly loaded the sample, if there are the gaps, I would say unfilled even in the small amount of space the error that you get out of that would be very huge okay. So, it is very important to make sure that the sample loading in a parallel plate or cone or plate such that the sample actually bulges out at the measuring gap, okay at the measuring position over here.

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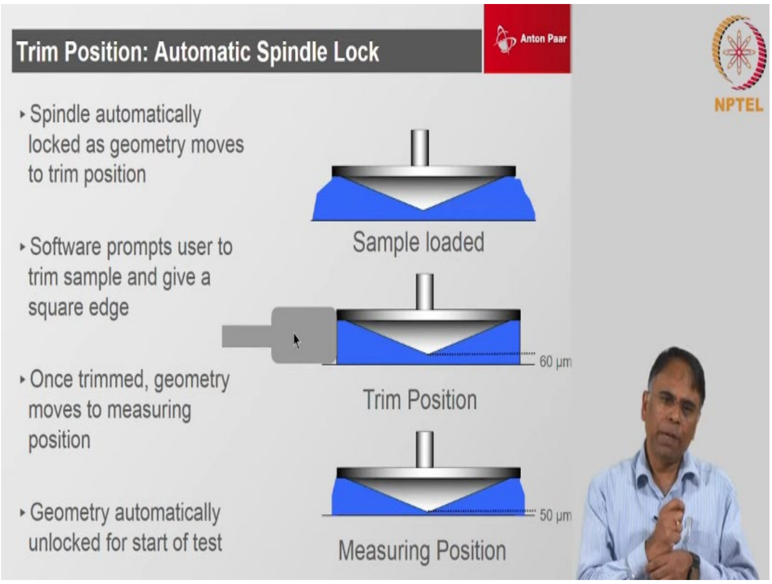

Of course, you will see that many times when you do a steady shear experiment specifically because of the centrifugal forces the sample actually can get contracted inside okay. So that is a reason the extra bulge will also help you out if you are going to measure, like if you have an ink sample if you want to measure at a shear rate of 1000 seconds inverse okay. It is very important to bulge so that when at this high shear rate of 1000 this you know ink is really going to go inside a little bit okay.

But it will still maintain that full gap condition even at that shear range. Trimming the sample is important, if you take viscoelastic sample okay. Example asphalt, trimming is going to be one of the most important parameters and if you are going to trim samples which are hot okay make sure that your trimming tool that you are using is also warm enough okay. Do not take a cold tool try to trim that you are giving a very big thermal shock to the sample okay.

If you are taking a very cold you know trimming tool and just directly trimming a hot sample okay, you just imagine what is happening at the trimming edge over there okay. So, you have sudden you know quenching of the sample over there and it will also be difficult for you to trim with a cold trimming tool as compared with a warm trimming tool. Warm trimming tool will be easier to handle okay.

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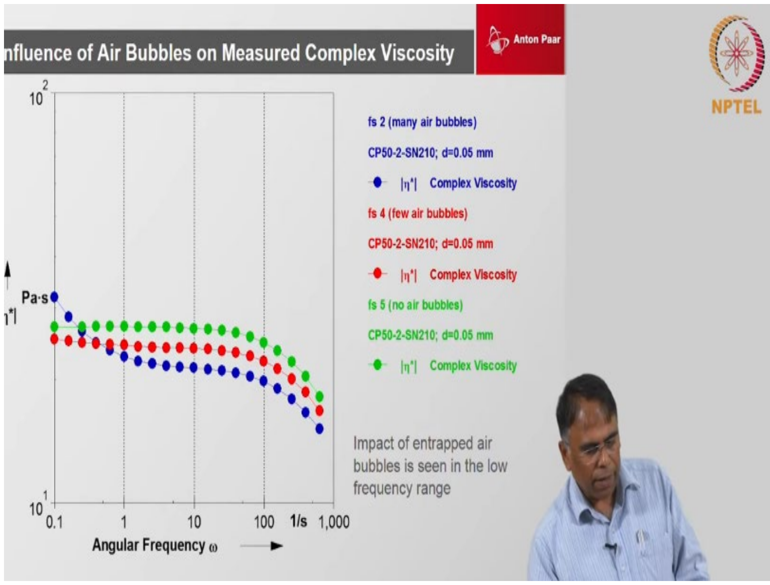

This is an example of an influence of a bubble if you have a bubble inside the sample you can see it will act like a particle inside a sample okay. It will give a same effect like a particle of the sample.

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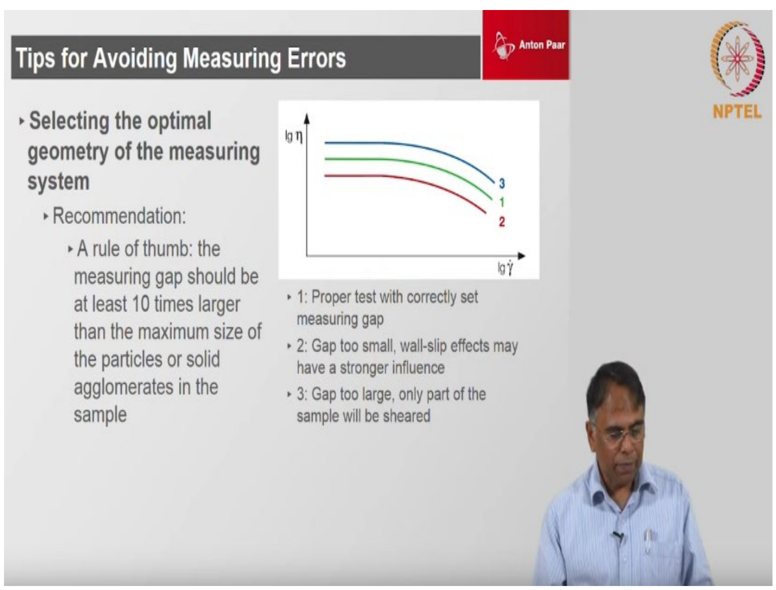

And then of course the particle size, as we were discussing if you have a particle size of 10 micron then the minimum gap you should is to be 10 times the largest particle size okay.

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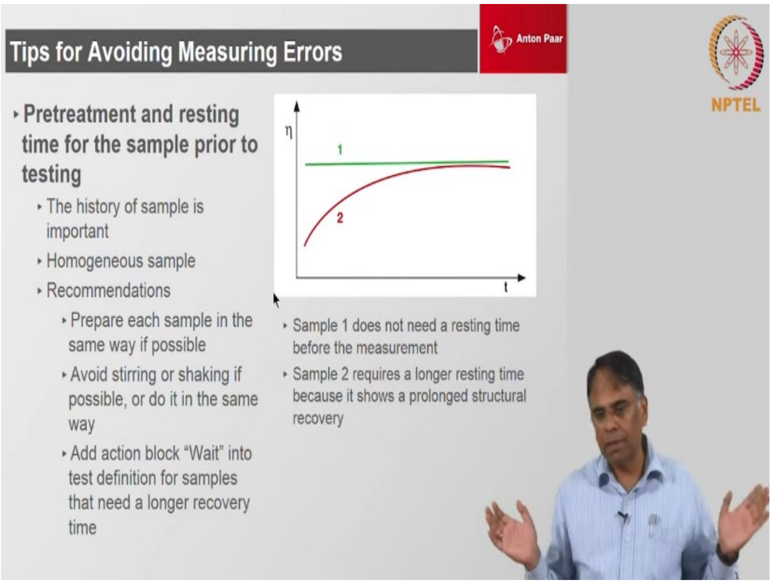

So always follow this gap, do not try to measure at very low gaps just you know without the information about the particle size okay because you will end up only grinding few particles and getting information of few particles that is not real bulk information that you are getting out of it okay. Sample which are with dispersions or slurries they would also require what is called as pre shear.

So the pre shear basically make sure that sample is well organized before testing and some time pre shear is also necessary if you take a coaxial cylinder system for example and you want to measure at say 135 degree centigrade it is better to do a soft pre shear to make sure that the temperature really well stabilized across the whole sample, the temperature is really homogenized and then you can do your viscosity measurements okay.

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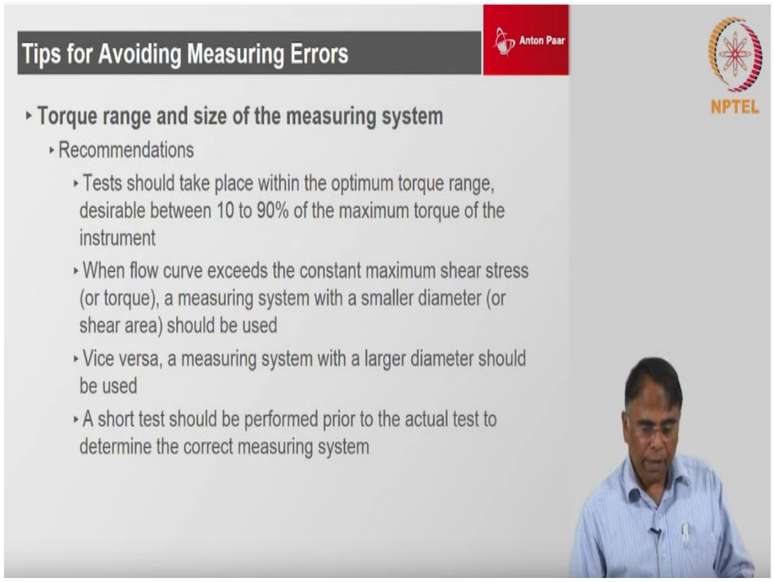

So, the pre shear helps, it is a very good pretreatment method over here okay and then of course the torque range and the size of measuring system. if you to find that the torque that you are getting of this especially the lower points is quite low okay at 25 mm geometry for example then you can immediately go for a 50 mm geometry that will automatically improve the torque at the small levels okay.

You can actually change the geometry size if you still want to measure at its lower shear conditions okay and you want to have a much better signal to noise ratio. So definitely the size of the plate can help you very easily to get a good data out of it. At high shear this one of the things that can happen specifically you are measuring very low viscosity material and you want to measure them at high shear you get what we call as a secondary shear effect.

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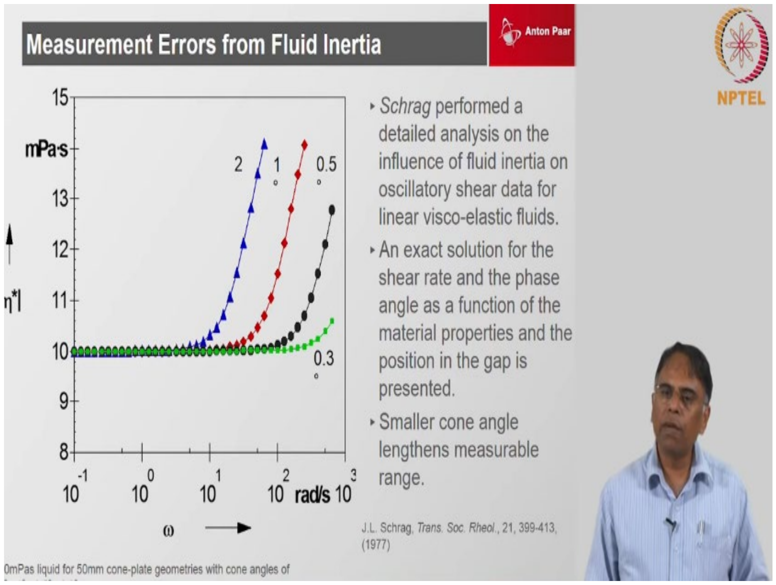

The secondary shear effect is nothing but you the material which was actually having a laminar flow it is goes into a turbulent flow okay. When it goes into a turbulent flow the layers which were actually flowing like nice laminar sheets, these laminar sheets actually are crossing each other now in a turbulent flow. And that is called crossing that extra shear and that is why you can see this viscosity increasing as a function of time.

Of course, you can control these by the geometric condition to a certain level so if you minimize the gap you can push these non you know non laminar region little bit further, but you cannot eliminate it completely. You can probably get to a little bit more high shear condition without going to this turbulent region by taking smaller gap size okay for the same viscosity.

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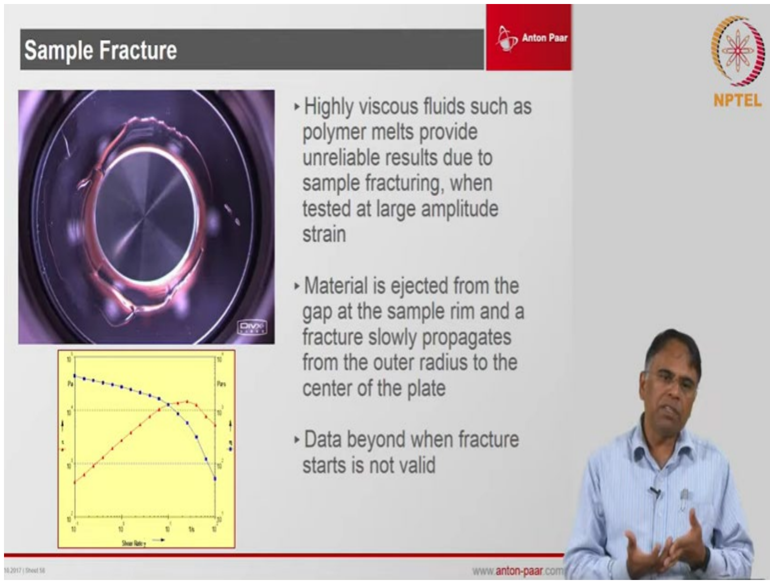

The other factor that is comes is the edge fracture, we can call it as a sample fracture. In simple terms if you have viscoelastic samples the when you are applying a shear on the samples the sample also develop what we call as a normal stress okay. And this normal stress is coming due to the you know Weissenberg effect. This normal stress will push the you know sample up but if you have a plate there is nowhere to go, so the only way the place to go is outside okay.

So, the sample actually starts living the gap and when it leaves the gap it actually fractures okay. And these fractures travel from this edge towards the center okay. So this is a typical thing that happen in viscoelastic solids okay because elasticity is high over there, the Weissenberg effects are more stronger over there and when you go specifically into the non liner region you will see this normal forces strength increases more and more, so the edge fracture effects will happen at this high strain non linear regions.

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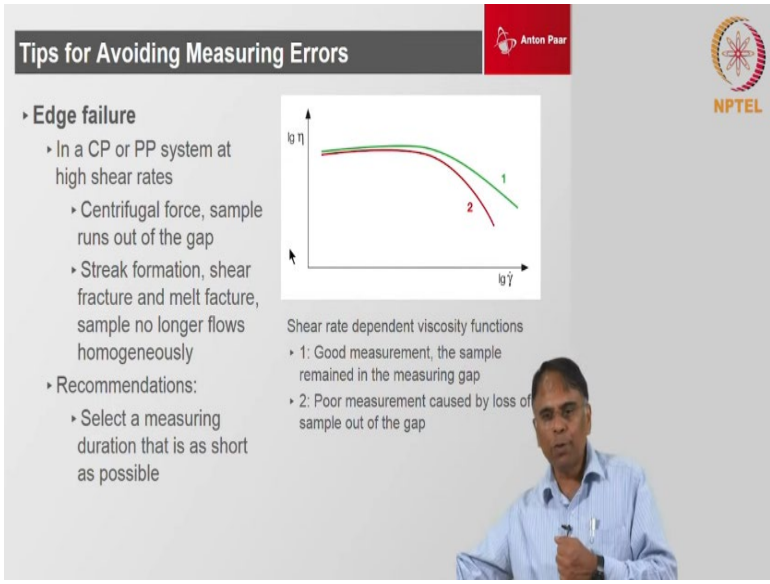

And we really need to know these where they are happening because otherwise, we start understanding that this is due to really the degeneration of the structure, but it can be due to the sample not getting hold inside the gap okay. So, the sample is not filling the gap at all and that is actually also crossing the fall of the modulus okay.

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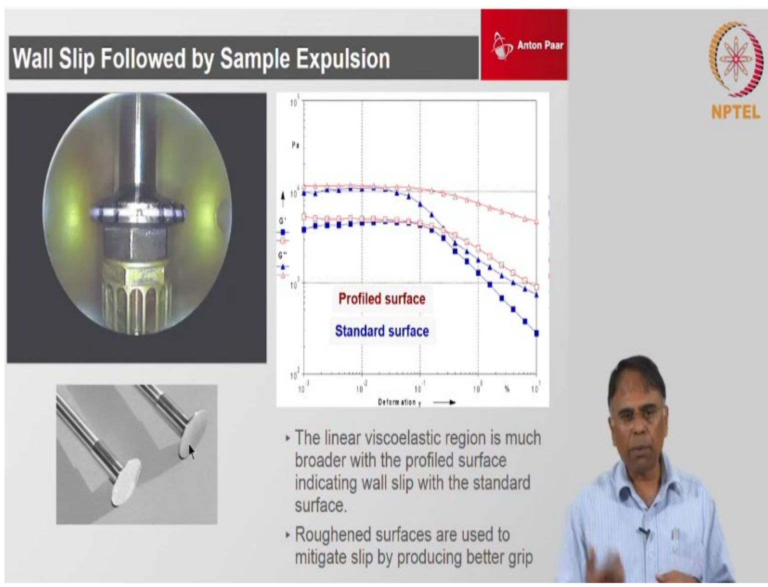

There are certain geometries that can be used for you know I would say not eliminating this but try to minimize effect of this or pushing against this effects too much higher strains okay. So, there is a geometry which we have you know cone partition plate which is generally used for this. Then the other you know artefact that get is the slippage between the samples. If you generally the geometries that we use very simple smooth surface is okay.

And if you are measuring viscoelastic solids at the very stiff conditions, they have the tendency to slip you know this geometry when I say slip the geometry is moving much faster than the sample and they are not moving as a unit okay. The geometry slips move faster than the sample itself. The sample is really not getting the same strain that the geometry is applying okay. So that slip is applying you know this artefact you will get different torque out of it and because of that different stress and because of that of course of the different moduli or different viscosity.

So, to counter that you can use geometry with surfaces which are roughened in a certain way the roughening can be done by doing sand blasting and you can do profiling okay. So, if you have very slippery gel and profiling is good but if you have very tough viscoelastic solid like rubber or asphalt and low temperature, we can use you know sand blasted plates.

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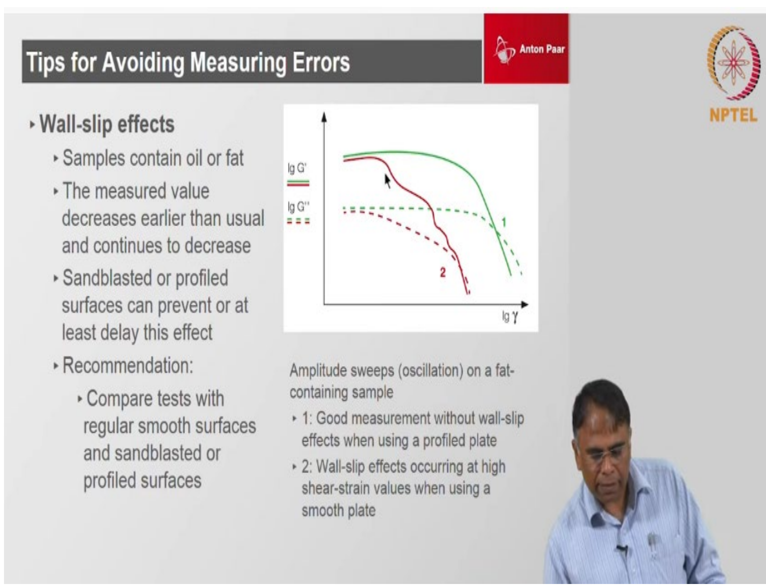

So, these are the examples typical examples of all slip you can see when you do a simple amplitude sweep, from these you can see these data you know if you see these steps like these, this generally according due to the slippage. You can also if you have a facility to also record waveforms and if you see the waveforms also the strains you know in these when the slippage is happening the strains are actually non sinusoidal, you are actually introducing more harmonics into the waveforms okay.

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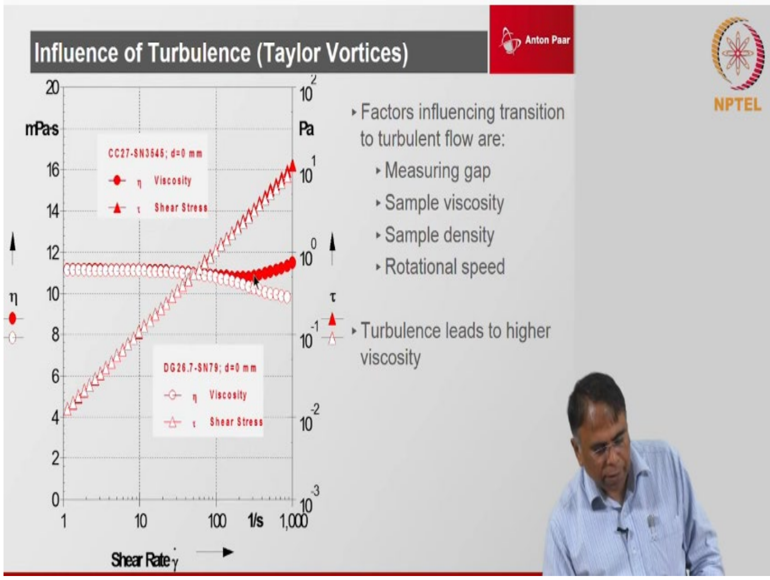

This is the influence of the Taylor vortices or the turbulence, we have already discussed that and we can actually reduce these or I would say push this forward by adjusting the gap or lowering the gap.

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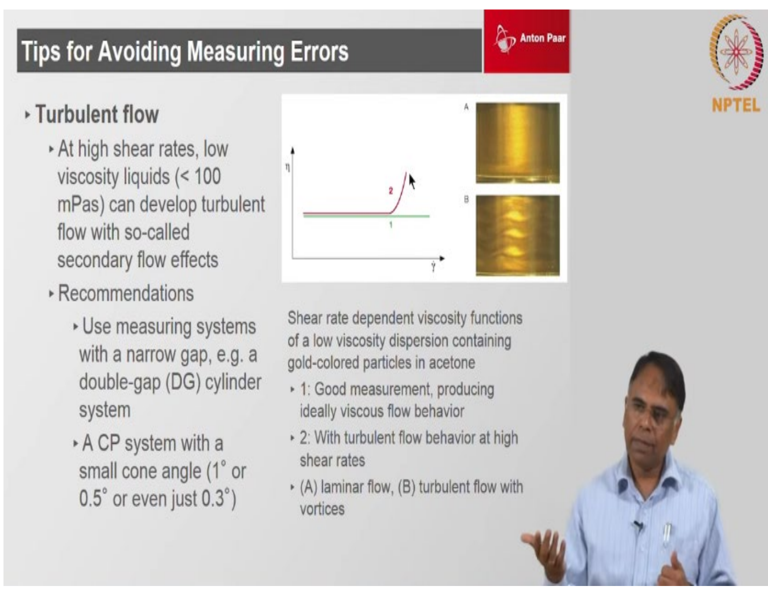

If of course the sample allows that. So, it depends on the particle sides inside that, so you can see the turbulent flow will generally cause an increase in viscosity. Many people say that this is shear thickening no this not shear thickening this is simply the physics which is giving us effect of high viscosity because of turbulent flow okay. So, the viscosity of the metal has not changed okay. It is simply because of the non-laminar flow the secondary shears are giving higher torques and those are actually giving us the effect of high or increase in viscosity.

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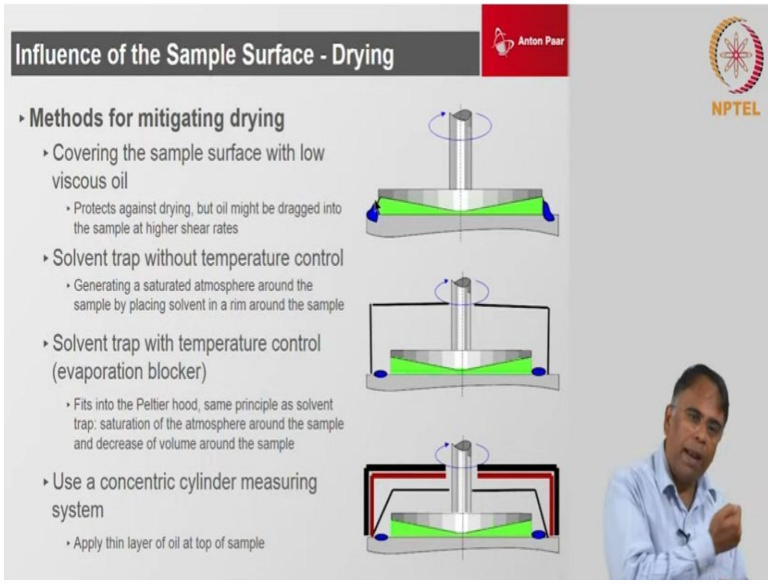

Then you have the drying effects, if you are measuring emuslions or dispersion or suspensions you can see the edge drying also gives us problems. So, if you have test which are long or you are doing at a little bit elevate temperature where there is a evaporation of the you know solvent phases inside there. So that can lead to that again you know wrong interpretation of the data you can see a moduli or you know a viscosity increases.

Because of the edge drawing but it is not because of the sample actually it is because of its drying effects forming a very fine film and you know parallel specifically what happening this influence greatly. Because your maximum measurements is being done actually at the edge okay. So, it is important that if you are using a geometry like parallel plate and cone and plate for some samples with some volatile which can dry, then it is better to use what we call as solvent trap.

Or simply I can also let us your sample for water based for example then I can actually load the sample with a dropper at a very lower viscosity, oil covering at the edge. So that also can you know almost minimize or make sure that the evaporation is really arrested at the edge.

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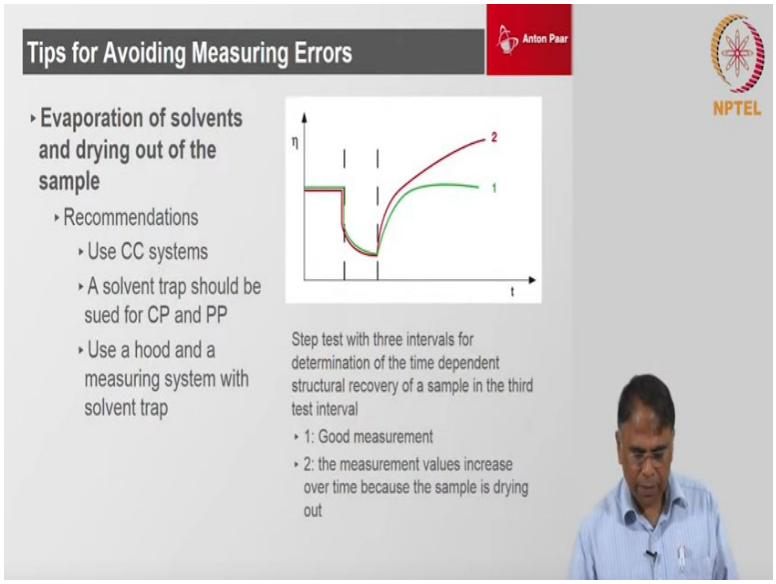

So certain tips which are there so the solvent trap is one very good you know condition which can be used or the you know covering the sample edge with the oil that can help us.

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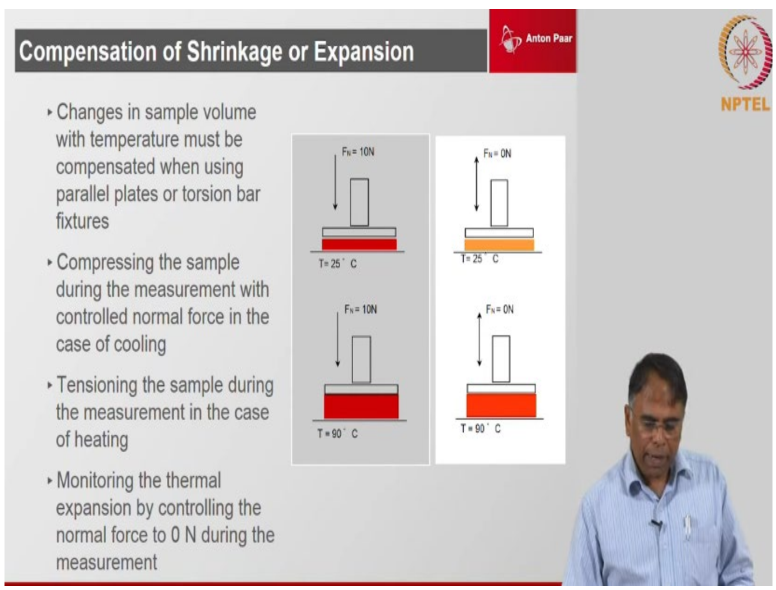

There are other possibilities like if you are doing samples which are going to crosslinking for example as a function of temperature or time cross linking generally reduces the volume okay free volume. So, it contracts so you can actually use normal force to control and counter that okay. So, the normal force if the contract happening it will start pulling this geometry down okay. You can set a normal force of zero for example and it will as an geometry gets pull down the rheometer will lower the gap to make sure that the force again becomes zero okay.

So that way we can control this you know because what will happen if you have contraction of the sample then your gap is unfilled okay. So, you are again now measuring with the unfilled gap and this modulus that you are getting out of fitting is not correct. So, to make sure that sample gap is completely fill at all times you can use this normal force control to maintain the gap completely full by adjusting the gap during the test using this normal force control.

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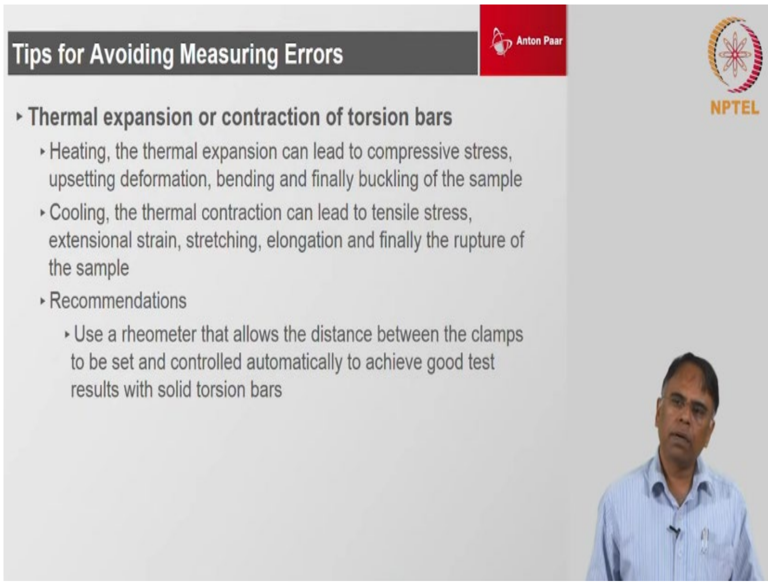

So, this is very good way to make sure that any temperature expansion or contraction is compensated by using this normal force control so of course this can be also used for bars.

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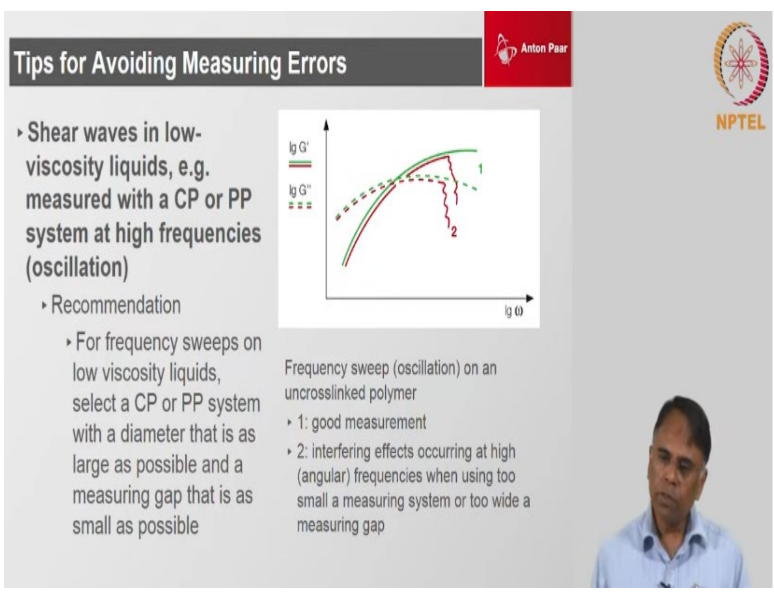

For bars also you will have the same the bars so the bars itself will also expand or contract okay based of the temperature and we can actually use very small normal force to control this effect.

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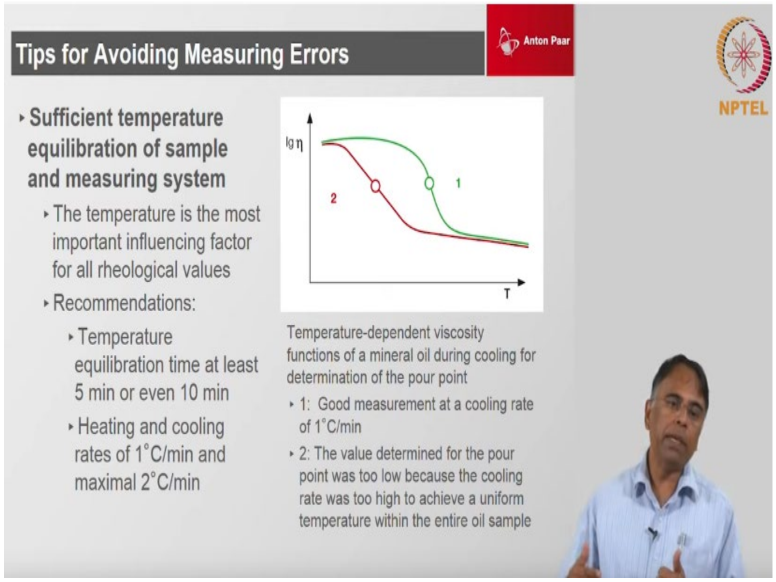

And change in length also can be measure as a function of this effects, so you can actually as the normal force control you see this you know the gap changing as a function of the force okay to maintain that force you can actually measure your gap as a function of the temperature also.

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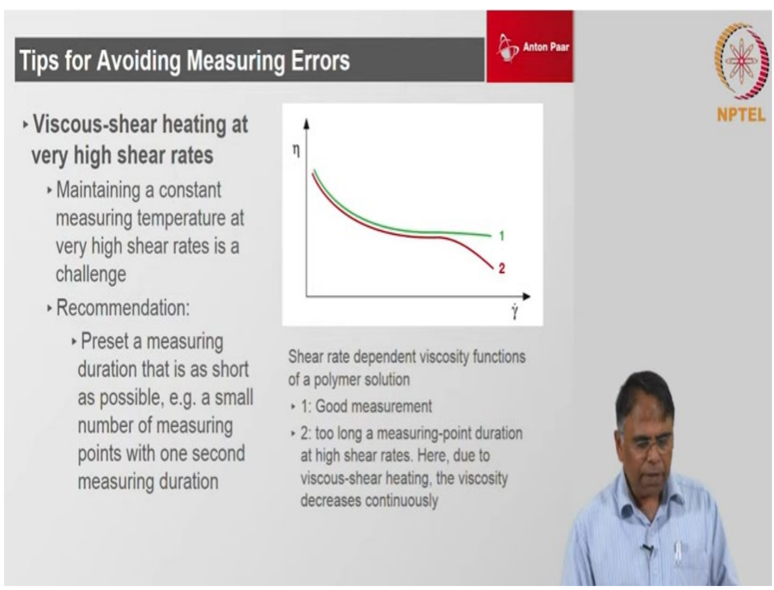

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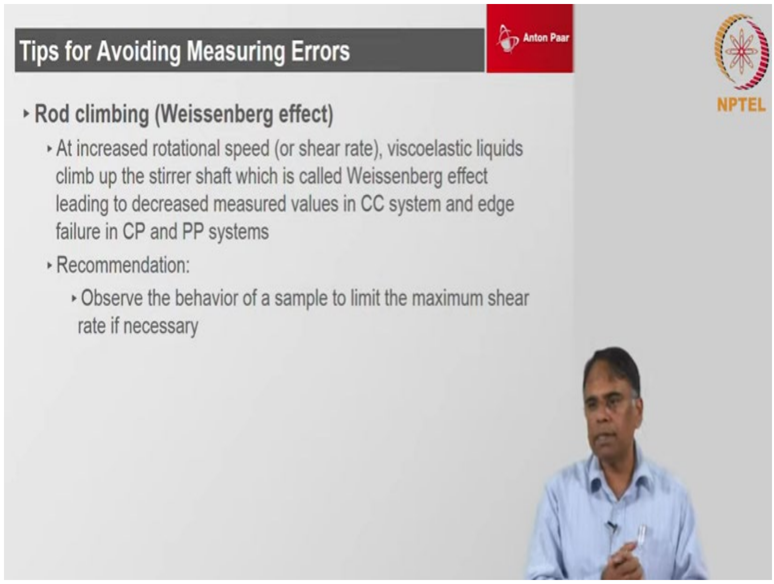

So other simple effects viscous-shear heating effects at very high shear rate, the Weissenberg effect at if you are using coaxial cylinder system, we can see sometimes the sample climbing up the rod this coaxial cylinder system okay. It can happen only in coaxial cylinder system not a parallel plate and cone plate. In the parallel plate and cone and plate it leads to edge fracture effect but in a coaxial cylinder system has the place to climb up it will climb up the rod.

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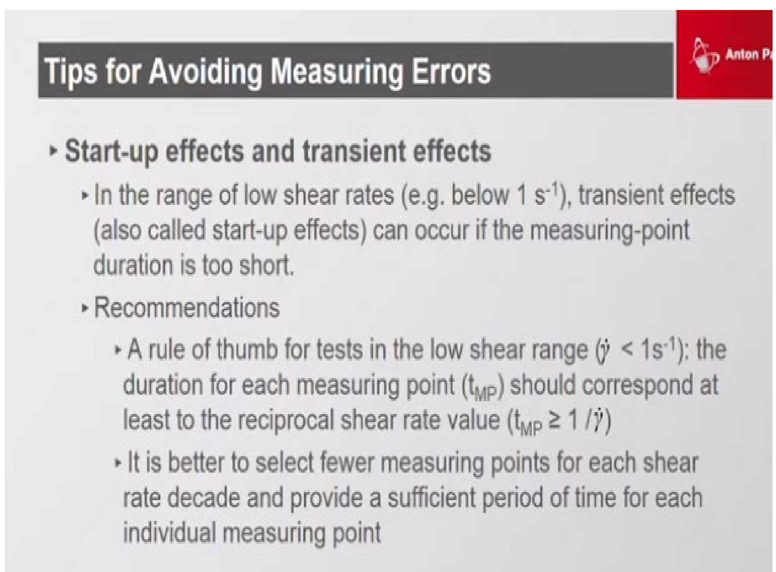

Okay so this is typical for viscoelastic liquids to happen at, again it will happen in the nonlinear region where you will start getting the Weissenberg effect.

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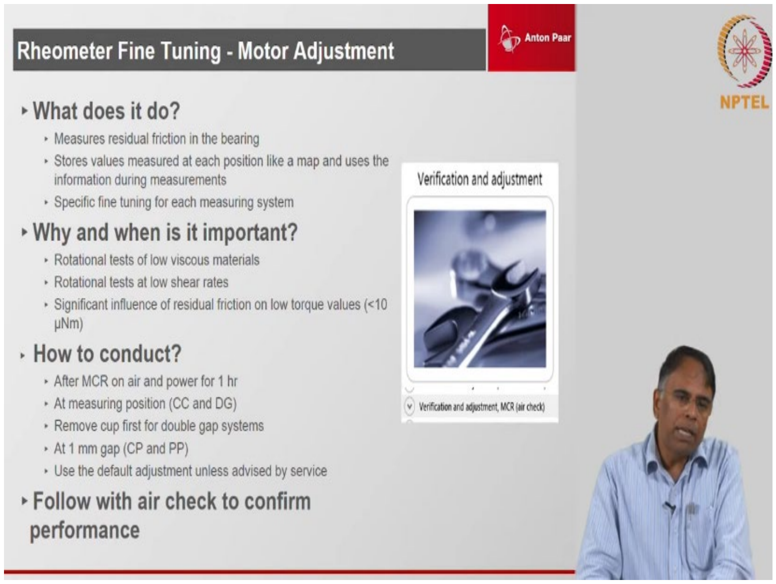

You can have other startup effects, transient effects when we are trying to measure at very short duration time always make sure that your rheometer is always upkept well. Every manufacture will have its own small way of making sure that the torque is very well calibrated for the geometries. So, these are certain tools one is basically the motor adjustment in a motor adjustment what is done for every geometry? What is the residual torque across the whole 360 degrees is recorded and kept in the memory? And that is used also to actually when you are doing sample measurement, so this residual torque is actually subtracted from the sample measurement.

So that you get accurate values which are actually coming out only from the sample not from the geometry okay. So, the motor adjustment has to been done at least every 90 days in general you can do every week or every day also not a problem. It is a short measurement you waste only 5 minutes out of it but if you do it also every day it is not a problem. You can do the measurements of the motor adjustments for all types of geometries every geometry that you are using.

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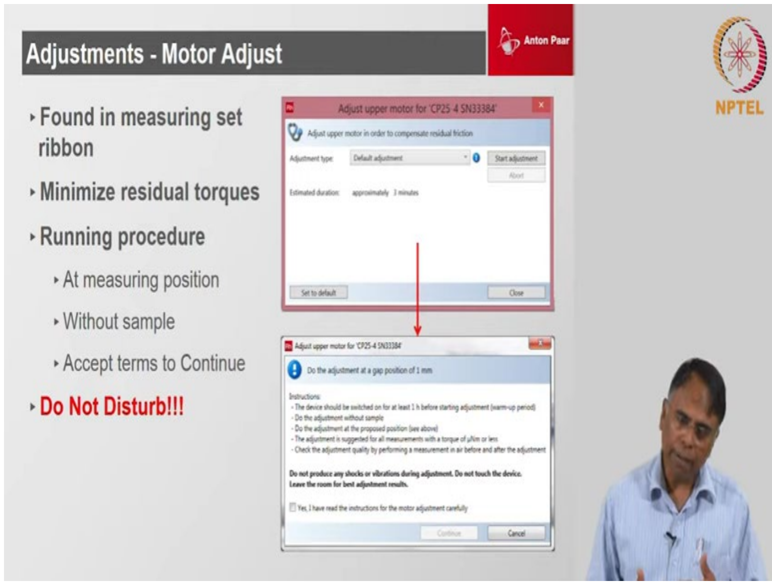

There are geometrics where if you are going to measure even the normal force then you can do a motor adjustment for a longer duration of time at very low shear condition so you can map not only the torque but also the normal force at all 360 degrees okay. If you do a long adjust it is actually both torque and the normal force and if you are doing short adjust generally only the torque will be mapped so this is what is done.

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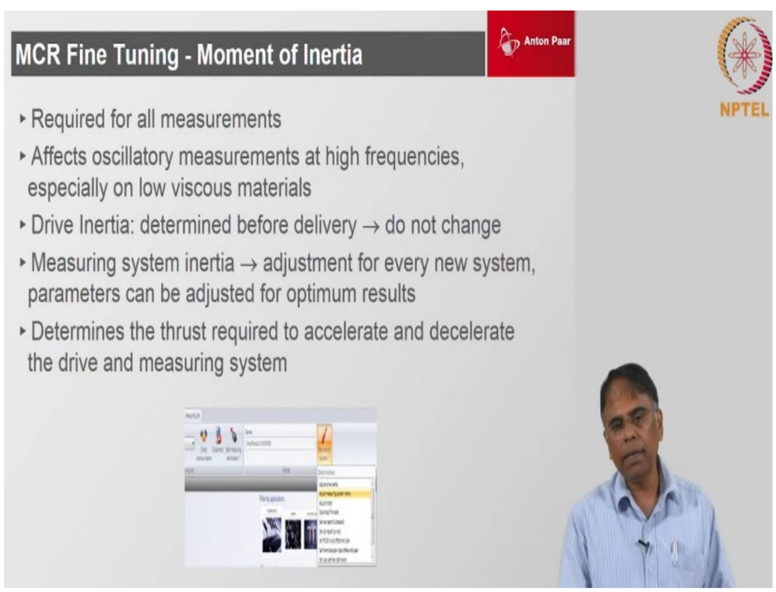

And always keep on tracking and recording the right inertia of all the geometries okay. So these are the I think so two very necessary things that you need to do in terms of regular you know upkeep of the instrument the motor adjustment and the inertia of the geometries okay .

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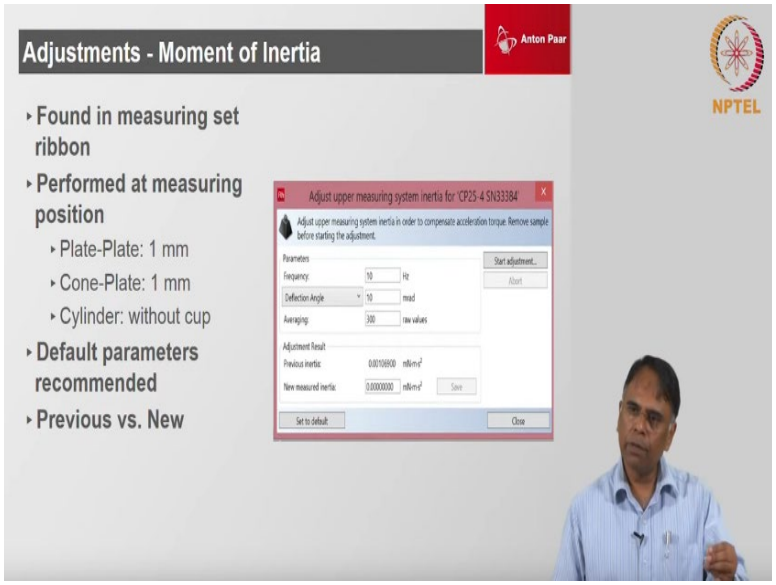

Both these things together will take altogether five minutes okay. So you required to put your tool without any sample to do a zero gap and do an inertia and motor adjustment either way you do a motor inertia or inertia and motor adjustment not a issue over here.

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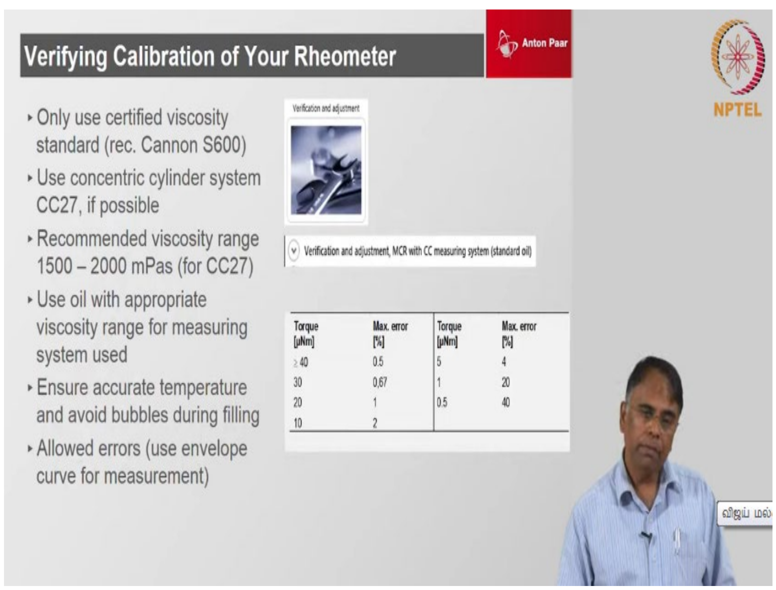

Temperature calibration is important, it has to be done once in a year at least and always cross check with either standard oil or standard reference material like a PDMS which has certain known values okay. Just to check that your measurements are really in the right place okay so this is something which also needs to be done on a regular base.

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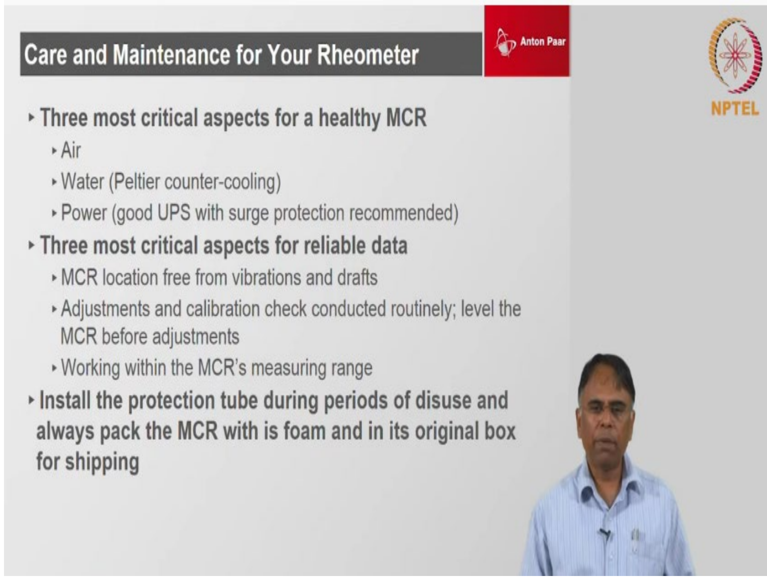

I would recommend at least once in 3 months or once in 6 months you should do the measurements. And then of course other things which are important to look at you are supplying air to the air bearing of this rheometer, so the dryers of this air bearing are important so keep on watching these dryers. They will be usually most of the dryers will have a color change as they get deteriorated.

So you should replace them you know this filters which are these dryers can be replaced every once in a year or once in two years you can look at the color changes and also if you are using circulators or thermostats for counter cooling the temperature devices then of course the liquid inside the counter cooling those should be replaced very frequently and cleaning of these whole path once in a year once in a 6 month would be always necessary.

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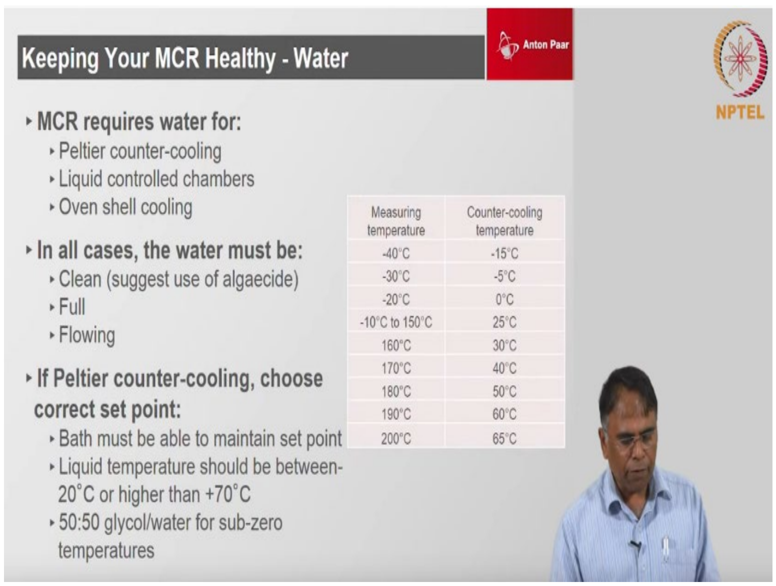

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So that actually is about maintaining the instrument maintaining the healthy condition of your rheometer. So, with this I would like to end the basics of Rheometry. Thank you very much