

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

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The slide is titled "Rheometer Raw Values, Rheological Values, and Results" and features the Anton Paar and NPTEL logos. It is divided into three main sections:

- Instrument quantity:** Raw values, independent on the measuring system.
 - Torque M [Nm]
 - Rotational speed n [1/min]
 - Deflection angle φ [°]... [rad]
- Rheological value:** Calculated values, dependent of the measuring system.
 - Shear stress $\tau = C_{SS} \cdot M$ [Pa]
 - Shear rate $\dot{\gamma} = C_{SR} \cdot n$ [1/s]
 - Deformation $\gamma = C_{SD} \cdot \varphi$ [1]... [%]
- Results:**
 - Viscosity $\eta = \frac{\tau}{\dot{\gamma}}$ [Pas]
 - Shear modulus $G = \frac{\tau}{\gamma}$ [Pa]

Red arrows indicate the flow of information: from Instrument quantity to Rheological value, and from Rheological value to Results.

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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Rheometer Measuring Ranges

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	Unit	MCR 52	MCR 102	MCR 302	MCR 502 TDR
Bearing	-	Ball	Air	Air	Air
EC motor (brushless DC) with high-resolution optical encoder	-	✓	✓	✓	✓
Permanent torque (60 min), no signal drift	-	✓	✓	✓	✓
EC mode (controlled shear rate and shear stress)	-	✓	✓	✓	✓
Maximum torque	mNm	200	200	200	230 (300) ¹⁾
Minimum torque, rotation	mNm	200 μNm	5	1	1
Minimum torque, oscillation	mNm	200 μNm	7.5	0.5	0.5
Angular deflection, set value	μrad	1 to ∞	0.5 to ∞	0.05 to ∞	0.05 to ∞
Step rate, time constant	ms	-	5	5	5
Step strain, time constant	ms	-	10	10	10
Step time (rate, strain), 99 % of set value (all samples)	ms	-	30	30	30
Minimum angular velocity ¹⁾	rad/s	10 ⁻⁴	10 ⁻⁶	10 ⁻⁷	10 ⁻⁷
Maximum angular velocity	rad/s	314	314	314	314
Minimum angular frequency ²⁾	rad/s	10 ⁻³	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷
Maximum angular frequency	rad/s	628	628	628	628



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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Measuring Point Duration - Overview

- ▶ Total measuring point duration is divided into:
 - ▶ Adjusting to the set value → short times, almost independent of sample
 - ▶ Establishing a steady shear rate distribution in the sample
 - requires time, dependent on sample, set value and measurement type (automatic recognition of the steady state available)
 - ▶ Recording data for averaging and sending the final value to the software
- ▶ The measuring point duration has to be set long enough to allow adjustment, reaching steady shear and sufficient time for averaging
- ▶ Using automatic settings for adjusting and averaging is preferable unless fast sampling is needed

30 %
Adjusting

70 %
Recording

50 % of recording
Averaging

Total measuring point duration

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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Measuring Range - Lower Torque Limit

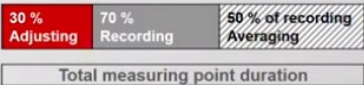
- ▶ The smallest available torque is dependent on measuring mode and instrument type:
 - ▶ Air bearing & Rotation:
 - 1 nNm (MCR 302, MCR 502)
 - 5 nNm (MCR 102)
 - ▶ Air bearing & Oscillation:
 - 0.5 nNm (with TruStrain™)
 - 7 nNm (without TruStrain™)
 - ▶ Ball bearing & Rotation:
 - 250 μNm (Rheolab)
 - ▶ M- and ME- in status of table indicates torque outside limit

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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Measuring Point Duration - Overview 

- **Total measuring point duration is divided into:**
 - **Adjusting to the set value** → short times, almost independent of sample
 - **Establishing a steady shear rate distribution in the sample**
→ requires time, dependent on sample, set value and measurement type (automatic recognition of the steady state available)
 - **Recording data for averaging** and sending the final value to the software
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- **Using automatic settings for adjusting and averaging is preferable unless fast sampling is needed**



Total measuring point duration

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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.

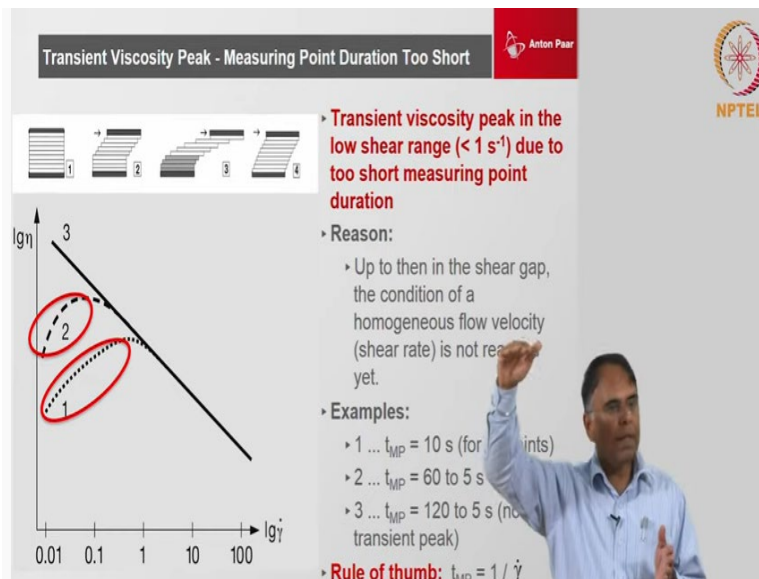
Because the electronic of the instrument, the mechanical properties so that flow of data all these consuming time okay. And this when we are looking at microseconds and milliseconds timer also adding up okay. We should if you look at measuring point duration first 30% of time is used to apply and that 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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Measuring Point Duration - Measurement of Stationary (Non-Transient) Properties

- Rotation - flow and viscosity curves, at constant shear stress or shear rate**
 - Choose measuring point duration according to shear rate ($\text{Time} = 1/\dot{\gamma}$ for low shear rates)
 - No time settings for automatic steady state recognition
 - Short measuring point duration at high shear rates to avoid shear fracture or shear heating
 - Automatic adjusting and averaging
- Oscillation - amplitude or frequency sweep**
 - No time settings
 - Automatic adjusting and averaging

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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Temperature Control - Temperature Distribution in a Sample

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Temperature Distribution without Hood

Temperature Distribution with Active Hood

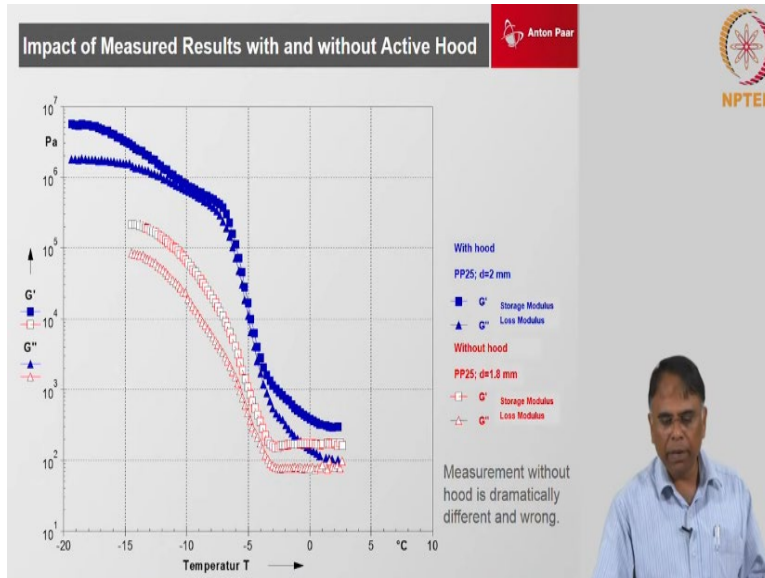
Cone-plate and parallel plates

- Significant influence of ambient temperature
- Gradients already at temperatures more than 5 to 10 K above or below ambient
- Temperature controlled hood required

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 chamber 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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Temperature Control – Equilibrium Time before a Measurement

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- Concentric cylinders / double gap system / stirrers
 - Large sample volume
 - Thermal equilibrium time at least 10 min
- Cone-plate systems / parallel plates
 - Small sample volume
 - Thermal equilibrium time 5 to 10 min for peltier and liquid systems
 - No time required for convection systems
- Thermal equilibrium time starts after target temperature is reached!**

Software interface showing: Variable: Temperature, Target value: 130 °C, Tolerance (σ): 0.25 °C, Timeout: 600 s.

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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Temperature Control – Heating and Cooling Rates

- Adjust heating or cooling rate to thermal properties of the sample
→ recommendation: max. 2°C/min
- Apply sample at room temperature for measurements at low temperatures avoids formation of ice
→ use closed accessory
- Melt and cool down samples for DMTA measurements with parallel plates
→ allow sufficient time for restructuring

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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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Temperature Control – Thermal Expansion of the System

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- ▶ Allow 10 min for thermal equilibrium before setting zerogap (→ thermal expansion)
- ▶ Set zerogap at measurement temperature or at medium temperature for a ramp
- ▶ Automatic compensation of thermal expansion for both measuring system and accessory (AGC – automatic gap compensation)
- ▶ TruGap™ allows gap measurement through the sample → correct values at all times

Principle TruGap™

measuring plate or cone
magnetic disc (closing the circuit)
primary coil (input current)
secondary coil (measured voltage)
protective layer
temperature probe
brazing solder
measuring plate
magnetic core

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.

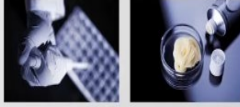
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Sample Handling, Loading, and Pre-test Considerations

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- ▶ Shaking, stirring, or syringing a sample prior to and during sample loading can impact the measured data.
- ▶ Wait for equilibrium to return after sample loading (structural and thermal equilibrium).
- ▶ Re-run a loaded sample only if certain it has suffered no unrecoverable damage in the prior test.
- ▶ Sample preparation, loading, and shear history must be controlled and be part of your test protocol.
- ▶ For structured fluids and gel samples, gap closure should be controlled, i.e. to fixed normal force or with fixed speed of closure.



Measuring Profiles Details: 1 Low viscosity

Mode:

Default measuring position: see measuring system settings

Minimum position: 0.001 mm

Maximum position: 125 mm


Normal force control hysteresis: 0.1 %

Normal force control timeout: 1000 s

Continue lift drive control after positioning

Position [mm]	Velocity [µm/s]	Normal Force [N]
0	25	5
1.5	50	5
25	200	5
5	2000	5

Navigation: Name Info | Measuring Position | Waiting Position | Loading Position | During Measurement | Zm





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 exact 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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Sample Filling – Cone Plate and Parallel Plate Systems





Ideal volume (with trim position) at rest	Correct volume at rest	Volume too small at rest
		
at shear rate of 100 s^{-1}	at shear rate of 100 s^{-1}	at shear rate of 100 s^{-1}
		
Measured viscosity value correct	Measured viscosity value too low	Measured viscosity value far too low

- For Single Gap Concentric Cylinder – add volume to fill line
- For Double Gap Concentric Cylinder and Disposable Cup Single Gap Systems – fill volumetrically

For all measuring systems the sample fill volume must be controlled for accurate and reproducible results

For CP and PP systems, the measuring system is locked during trimming.



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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Trim Position: Automatic Spindle Lock

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- Spindle automatically locked as geometry moves to trim position
- Software prompts user to trim sample and give a square edge
- Once trimmed, geometry moves to measuring position
- Geometry automatically unlocked for start of test

The diagram shows three stages of the process:

- Sample loaded:** A blue sample is placed between two metal plates.
- Trim Position:** A grey trimming tool is used to trim the sample. A dimension line indicates a gap of $60\ \mu\text{m}$.
- Measuring Position:** The sample is now between the plates with a gap of $50\ \mu\text{m}$.

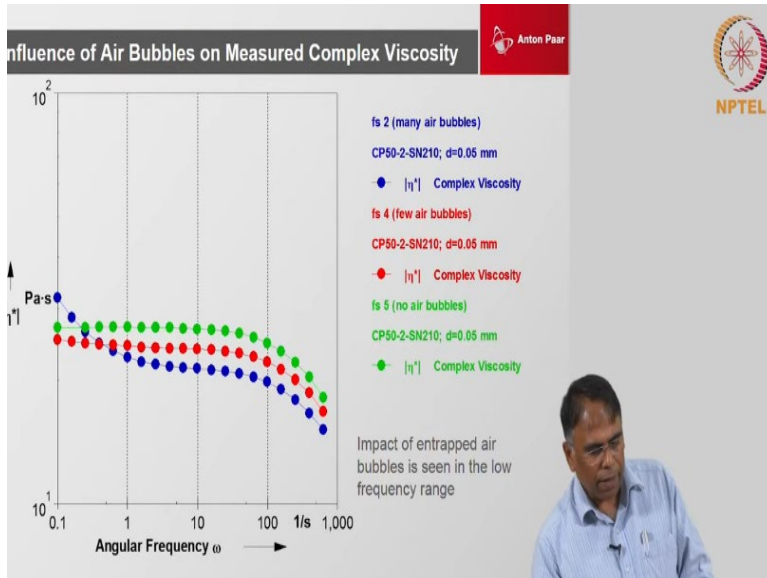
A man in a light blue shirt is visible in the bottom right corner of the slide.

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.

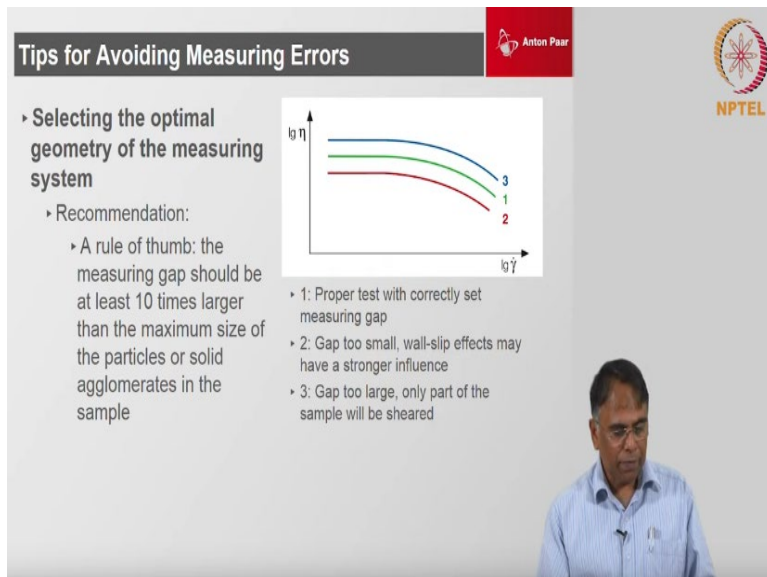
If you are taking a very cold you know trimming tool and just directly trimming a hot sample okay, you just imagine what is happening at the trimming edge over there okay. So, you have sudden you know quenching of the sample over there and it will also be difficult for you to trim with a cold trimming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

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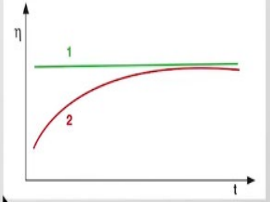
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▸ **Pretreatment and resting time for the sample prior to testing**

- The history of sample is important
- Homogeneous sample

▸ **Recommendations**

- Prepare each sample in the same way if possible
- Avoid stirring or shaking if possible, or do it in the same way
- Add action block "Wait" into test definition for samples that need a longer recovery time



- Sample 1 does not need a resting time before the measurement
- Sample 2 requires a longer resting time because it shows a prolonged structural recovery

(A presenter is visible in the bottom right corner of the slide.)

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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Tips for Avoiding Measuring Errors

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► **Torque range and size of the measuring system**

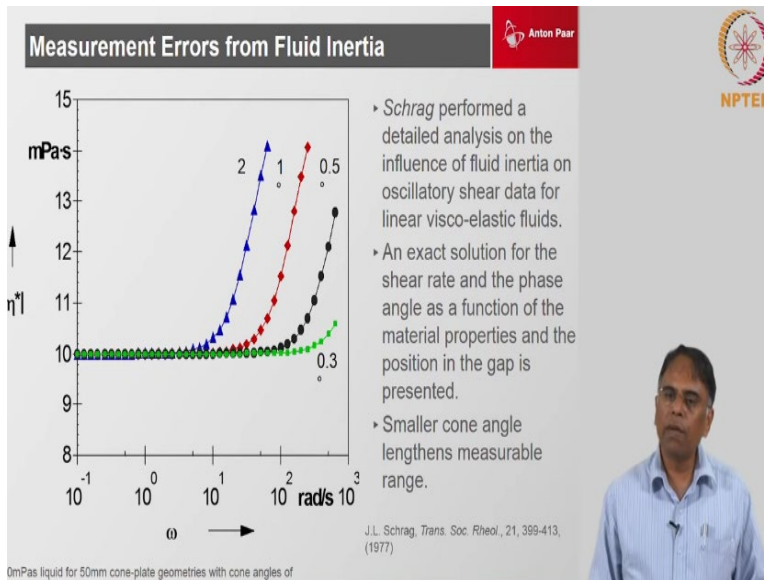
- Recommendations
 - Tests should take place within the optimum torque range, desirable between 10 to 90% of the maximum torque of the instrument
 - When flow curve exceeds the constant maximum shear stress (or torque), a measuring system with a smaller diameter (or shear area) should be used
 - Vice versa, a measuring system with a larger diameter should be used
 - A short test should be performed prior to the actual test to determine the correct measuring system

Video inset: A man in a blue shirt speaking.

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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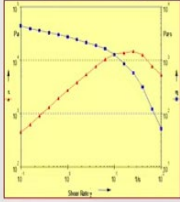


The secondary shear effect is nothing but you the material which was actually having a laminar flow it is goes into a turbulent flow okay. When it goes into a turbulent flow the layers which were actually flowing like nice laminar sheets, these laminar sheets actually are crossing each other now in a 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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Sample Fracture

- Highly viscous fluids such as polymer melts provide unreliable results due to sample fracturing, when tested at large amplitude strain
- Material is ejected from the gap at the sample rim and a fracture slowly propagates from the outer radius to the center of the plate
- Data beyond when fracture starts is not valid



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The other factor that 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.

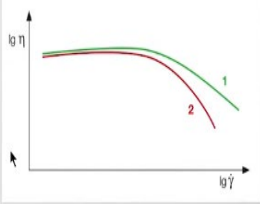
So, the sample actually starts leaving the gap and when it leaves the gap it actually fractures okay. And these fractures travel from this edge towards the center okay. So this is a typical thing that happen in viscoelastic solids okay because elasticity is high over there, the Weissenberg effects are more stronger over there and when you go specifically into the non linear 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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Tips for Avoiding Measuring Errors

Edge failure

- ▶ In a CP or PP system at high shear rates
 - ▶ Centrifugal force, sample runs out of the gap
 - ▶ Streak formation, shear fracture and melt fracture, sample no longer flows homogeneously
- ▶ Recommendations:
 - ▶ Select a measuring duration that is as short as possible




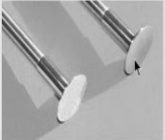
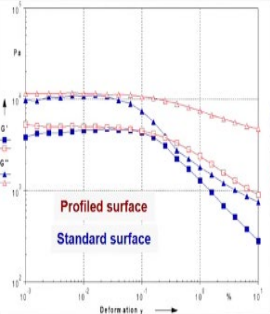
Shear rate dependent viscosity functions

- ▶ 1: Good measurement, the sample remained in the measuring gap
- ▶ 2: Poor measurement caused by loss of sample out of the gap

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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Wall Slip Followed by Sample Expulsion

The linear viscoelastic region is much broader with the profiled surface indicating wall slip with the standard surface.

Roughened surfaces are used to mitigate slip by producing better grip

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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The slide is titled "Tips for Avoiding Measuring Errors" and features the Anton Paar and NPTEL logos. It contains the following text:

- Wall-slip effects**
 - Samples contain oil or fat
 - The measured value decreases earlier than usual and continues to decrease
 - Sandblasted or profiled surfaces can prevent or at least delay this effect
 - Recommendation:
 - Compare tests with regular smooth surfaces and sandblasted or profiled surfaces

The graph shows $\log G'$ on the y-axis and $\log \gamma$ on the x-axis. It displays two sets of curves: a solid line (1) representing a good measurement without wall-slip effects using a profiled plate, and a dashed line (2) representing wall-slip effects occurring at high shear-strain values when using a smooth plate. The dashed line shows a significant drop in G' at higher strain values compared to the solid line.

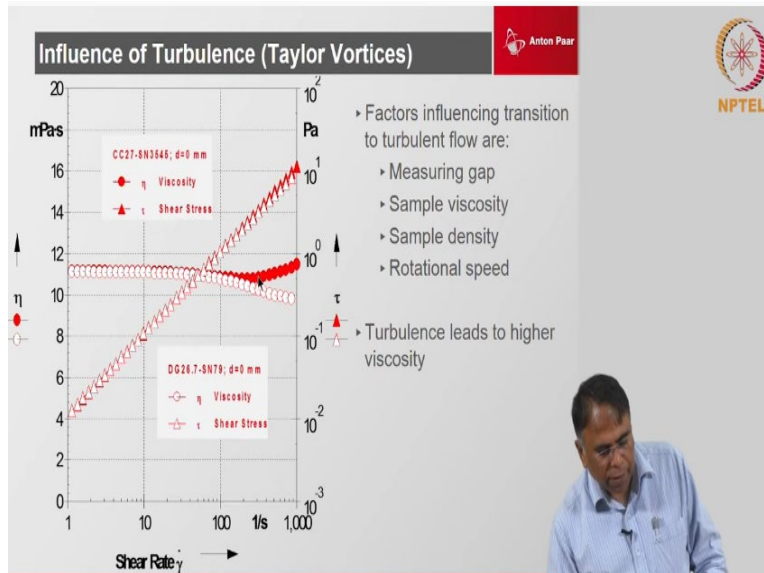
Amplitude sweeps (oscillation) on a fat-containing sample

- 1: Good measurement without wall-slip effects when using a profiled plate
- 2: Wall-slip effects occurring at high shear-strain values when using a smooth plate

A presenter is visible in the bottom right corner of the slide.

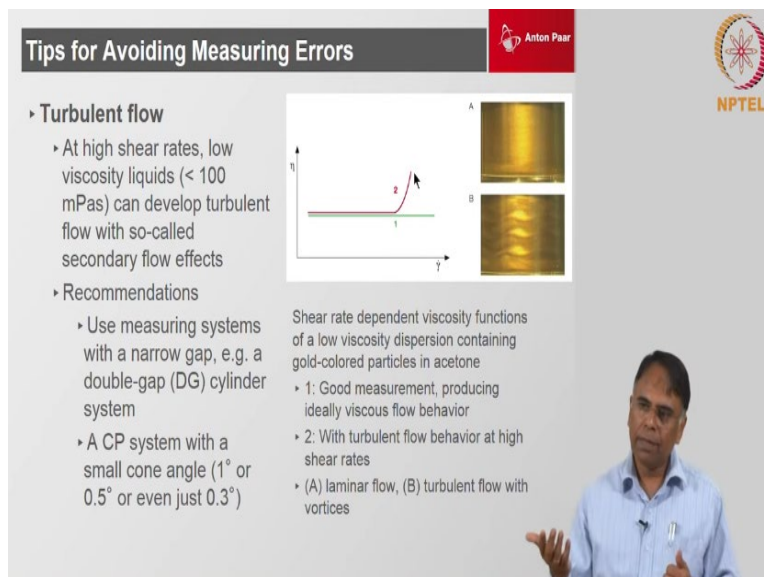
So, these are the examples typical examples of all slip you can see when you do a simple amplitude sweep, from these you can see these data you know if you see these steps like these, this generally according due to the slippage. You can also if you have a facility to also record waveforms and if you 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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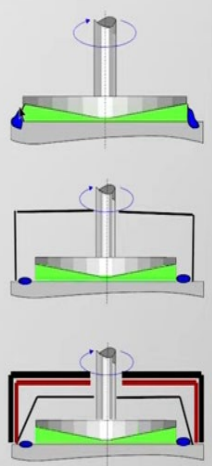


If of course the sample allows that. So, it depends on the particle sides inside that, so you can see the turbulent flow will generally cause an increase in viscosity. Many people say that this is shear thickening no this not shear thickening this is simply the physics which is giving us effect of high 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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Influence of the Sample Surface - Drying

- **Methods for mitigating drying**
 - Covering the sample surface with low viscous oil
 - Protects against drying, but oil might be dragged into the sample at higher shear rates
 - Solvent trap without temperature control
 - Generating a saturated atmosphere around the sample by placing solvent in a rim around the sample
 - Solvent trap with temperature control (evaporation blocker)
 - Fits into the Peltier hood, same principle as solvent trap: saturation of the atmosphere around the sample and decrease of volume around the sample
 - Use a concentric cylinder measuring system
 - Apply thin layer of oil at top of sample



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NPTEL



Then you have the drying effects, if you are measuring emulsions 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 elevated temperature where there is an 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 modulus or you know a viscosity increases.

Because of the edge drying 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 is happening this influence greatly. Because your maximum measurement 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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Tips for Avoiding Measuring Errors

Evaporation of solvents and drying out of the sample

- Recommendations
 - Use CC systems
 - A solvent trap should be used for CP and PP
 - Use a hood and a measuring system with solvent trap

Step test with three intervals for determination of the time dependent structural recovery of a sample in the third test interval

- 1: Good measurement
- 2: the measurement values increase over time because the sample is drying out

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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Compensation of Shrinkage or Expansion

- Changes in sample volume with temperature must be compensated when using parallel plates or torsion bar fixtures
- Compressing the sample during the measurement with controlled normal force in the case of cooling
- Tensioning the sample during the measurement in the case of heating
- Monitoring the thermal expansion by controlling the normal force to 0 N during the measurement

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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Tips for Avoiding Measuring Errors

- ▶ Thermal expansion or contraction of torsion bars
 - ▶ Heating, the thermal expansion can lead to compressive stress, upsetting deformation, bending and finally buckling of the sample
 - ▶ Cooling, the thermal contraction can lead to tensile stress, extensional strain, stretching, elongation and finally the rupture of the sample
 - ▶ Recommendations
 - ▶ Use a rheometer that allows the distance between the clamps to be set and controlled automatically to achieve good test results with solid torsion bars

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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Tips for Avoiding Measuring Errors

- ▶ Shear waves in low-viscosity liquids, e.g. measured with a CP or PP system at high frequencies (oscillation)
 - ▶ Recommendation
 - ▶ For frequency sweeps on low viscosity liquids, select a CP or PP system with a diameter that is as large as possible and a measuring gap that is as small as possible

Frequency sweep (oscillation) on an uncrosslinked polymer

- ▶ 1: good measurement
- ▶ 2: interfering effects occurring at high (angular) frequencies when using too small a measuring system or too wide a measuring gap

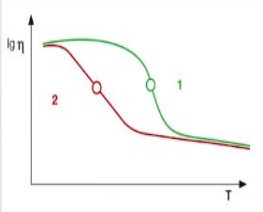
For bars also you will have the same the bars so the bars itself will also expand or contract okay based of the temperature and we can actually use very small normal force to control this effect.

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Tips for Avoiding Measuring Errors



► Sufficient temperature equilibration of sample and measuring system


- The temperature is the most important influencing factor for all rheological values
- Recommendations:
 - Temperature equilibration time at least 5 min or even 10 min
 - Heating and cooling rates of 1°C/min and maximal 2°C/min



Temperature-dependent viscosity functions of a mineral oil during cooling for determination of the pour point

- 1: Good measurement at a cooling rate of 1°C/min
- 2: The value determined for the pour point was too low because the cooling rate was too high to achieve a uniform temperature within the entire oil sample



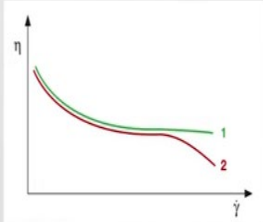
And change in length also can be measure as a function of this effects, so you can actually as the normal force control you see this you know the gap changing as a function of the force okay to maintain that force you can actually measure your gap as a function of the temperature also.

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Tips for Avoiding Measuring Errors



► Viscous-shear heating at very high shear rates


- Maintaining a constant measuring temperature at very high shear rates is a challenge
- Recommendation:
 - Preset a measuring duration that is as short as possible, e.g. a small number of measuring points with one second measuring duration



Shear rate dependent viscosity functions of a polymer solution

- 1: Good measurement
- 2: too long a measuring-point duration at high shear rates. Here, due to viscous-shear heating, the viscosity decreases continuously



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Tips for Avoiding Measuring Errors

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► **Rod climbing (Weissenberg effect)**

- At increased rotational speed (or shear rate), viscoelastic liquids climb up the stirrer shaft which is called Weissenberg effect leading to decreased measured values in CC system and edge failure in CP and PP systems
- Recommendation:
 - Observe the behavior of a sample to limit the maximum shear rate if necessary



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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Tips for Avoiding Measuring Errors

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► **Start-up effects and transient effects**

- In the range of low shear rates (e.g. below 1 s^{-1}), transient effects (also called start-up effects) can occur if the measuring-point duration is too short.
- Recommendations
 - A rule of thumb for tests in the low shear range ($\dot{\gamma} < 1 \text{ s}^{-1}$): the duration for each measuring point (t_{MP}) should correspond at least to the reciprocal shear rate value ($t_{MP} \geq 1 / \dot{\gamma}$)
 - It is better to select fewer measuring points for each shear rate decade and provide a sufficient period of time for each individual measuring point

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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Rheometer Fine Tuning - Motor Adjustment






- ▶ **What does it do?**
 - ▶ Measures residual friction in the bearing
 - ▶ Stores values measured at each position like a map and uses the information during measurements
 - ▶ Specific fine tuning for each measuring system
- ▶ **Why and when is it important?**
 - ▶ Rotational tests of low viscous materials
 - ▶ Rotational tests at low shear rates
 - ▶ Significant influence of residual friction on low torque values (<10 μNm)
- ▶ **How to conduct?**
 - ▶ After MCR on air and power for 1 hr
 - ▶ At measuring position (CC and DG)
 - ▶ Remove cup first for double gap systems
 - ▶ At 1 mm gap (CP and PP)
 - ▶ Use the default adjustment unless advised by service
- ▶ **Follow with air check to confirm performance**

Verification and adjustment



Verification and adjustment, MCR (air check)






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.

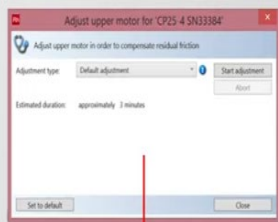
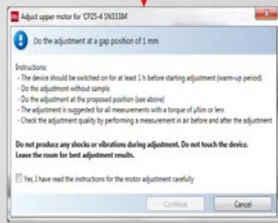
So that you get accurate values which are actually coming out only from the sample not from the geometry okay. So, the motor adjustment has to been done at least every 90 days in general you can do every week or every day also not a problem. It is a short measurement you waste only 5 minutes out of it 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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Adjustments - Motor Adjust

- ▶ Found in measuring set ribbon
- ▶ Minimize residual torques
- ▶ Running procedure
 - ▶ At measuring position
 - ▶ Without sample
 - ▶ Accept terms to Continue
- ▶ **Do Not Disturb!!!**








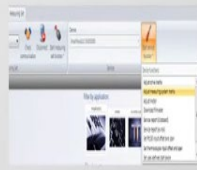

There are geometrics where if you are going to measure even the normal force then you can do a motor adjustment for a longer duration of time at very low shear condition so you can map not only the torque but also the normal force at all 360 degrees okay. If you do a long adjust it is actually both torque 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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MCR Fine Tuning - Moment of Inertia

- ▶ Required for all measurements
- ▶ Affects oscillatory measurements at high frequencies, especially on low viscous materials
- ▶ Drive Inertia: determined before delivery → do not change
- ▶ Measuring system inertia → adjustment for every new system, parameters can be adjusted for optimum results
- ▶ Determines the thrust required to accelerate and decelerate the drive and measuring system

And always keep on tracking and recording the right inertia of all the geometrics 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 geometrics okay .

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Adjustments - Moment of Inertia

- ▶ Found in measuring set ribbon
- ▶ Performed at measuring position
 - ▶ Plate-Plate: 1 mm
 - ▶ Cone-Plate: 1 mm
 - ▶ Cylinder: without cup
- ▶ Default parameters recommended
- ▶ Previous vs. New

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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Verifying Calibration of Your Rheometer

- ▶ Only use certified viscosity standard (rec. Cannon S600)
- ▶ Use concentric cylinder system CC27, if possible
- ▶ Recommended viscosity range 1500 – 2000 mPas (for CC27)
- ▶ Use oil with appropriate viscosity range for measuring system used
- ▶ Ensure accurate temperature and avoid bubbles during filling
- ▶ Allowed errors (use envelope curve for measurement)

Torque [µNm]	Max. error [%]	Torque [µNm]	Max. error [%]
> 40	0.5	5	4
30	0.67	1	20
20	1	0.5	40
10	2		

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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Care and Maintenance for Your Rheometer

Anton Paar

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- ▶ **Three most critical aspects for a healthy MCR**
 - ▶ Air
 - ▶ Water (Peltier counter-cooling)
 - ▶ Power (good UPS with surge protection recommended)
- ▶ **Three most critical aspects for reliable data**
 - ▶ MCR location free from vibrations and drafts
 - ▶ Adjustments and calibration check conducted routinely; level the MCR before adjustments
 - ▶ Working within the MCR's measuring range
- ▶ **Install the protection tube during periods of disuse and always pack the MCR with is foam and in its original box for shipping**






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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Keeping Your MCR Healthy - Air

- ▶ Clean, dry and oil free
- ▶ Constant supply pressure of 80-90 psi
- ▶ Consumption of 2.8 SCFM
- ▶ Recommend replacement of filter cartridges annually or any time auto drain discharges
- ▶ Do not move measuring system or motor shaft unless air is supplied to the MCR
- ▶ Check MCR display for **No Air Pressure** warning before using.
- ▶ Install the protection tube during periods of disuse
- ▶ Always properly pack the MCR before relocation or shipping (see manual)








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Keeping Your MCR Healthy - Water

- ▶ **MCR requires water for:**
 - ▶ Peltier counter-cooling
 - ▶ Liquid controlled chambers
 - ▶ Oven shell cooling
- ▶ **In all cases, the water must be:**
 - ▶ Clean (suggest use of algacide)
 - ▶ Full
 - ▶ Flowing
- ▶ **If Peltier counter-cooling, choose correct set point:**
 - ▶ Bath must be able to maintain set point
 - ▶ Liquid temperature should be between -20°C or higher than +70°C
 - ▶ 50:50 glycol/water for sub-zero temperatures

Measuring temperature	Counter-cooling temperature
-40°C	-15°C
-30°C	-5°C
-20°C	0°C
-10°C to 150°C	25°C
160°C	30°C
170°C	40°C
180°C	50°C
190°C	60°C
200°C	65°C

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Thank you for your attention



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