Polymer Process Engineering
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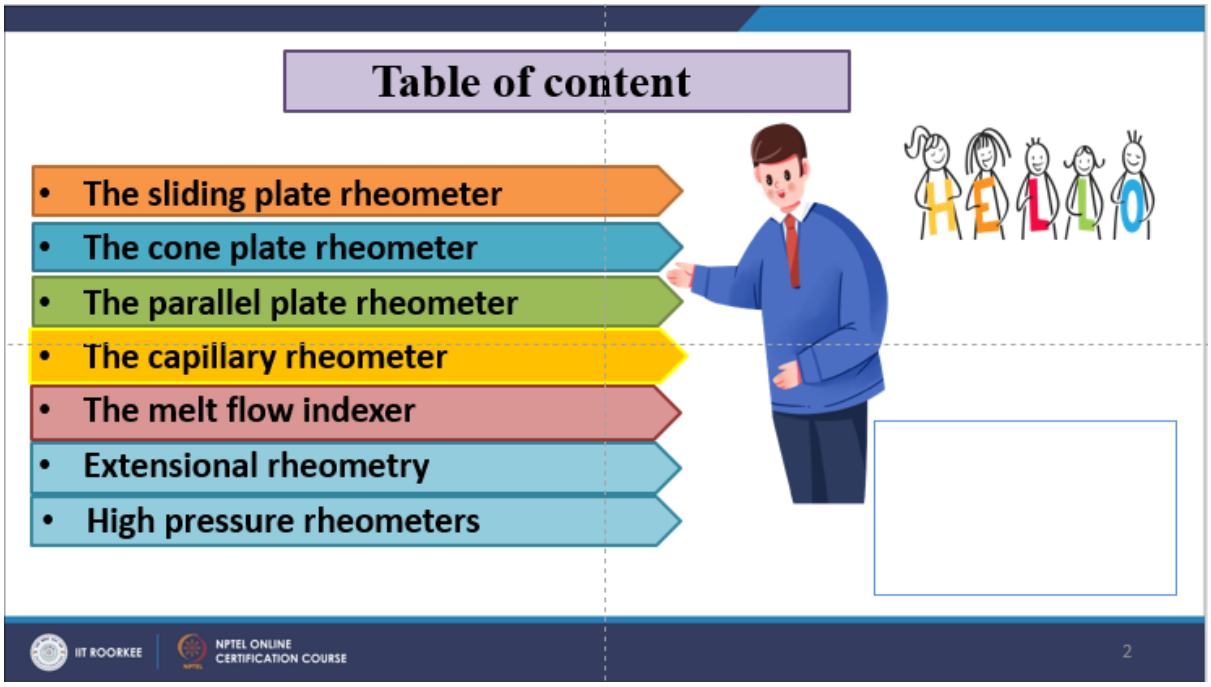
Lecture – 15
Applied polymer rheology: Rheometry

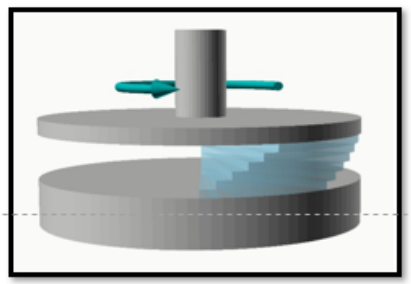
Hello friends, welcome to the applied polymer rheology under the ages of polymer process engineering. Here, we are going to discuss the rheometry. Now, let us have a look at that what we are going to discuss in this particular segment. We will discuss the sliding plate rheometer, then we will discuss the cone plate rheometer, the parallel plate rheometer, and the capillary and melt flow indexer. We will discuss the extensional rheometry and the high-pressure rheometer. Now, the rheometers are usually specialized tools or mechanisms that are needed for evaluating the fluid models and measuring the rheological parameters.



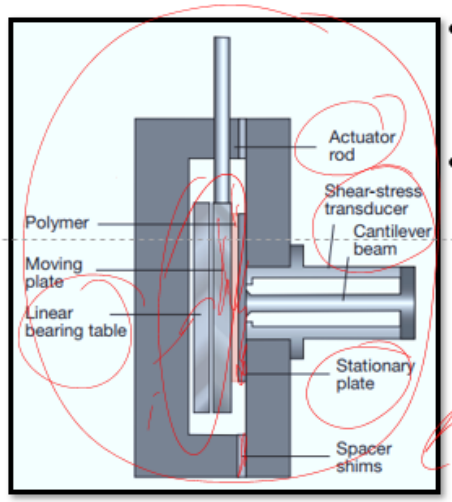
Usually, they are necessary for the complex measurement in the risk using the research, this including the development and assessment of various flow models as well as the analysis of a viscosity and a normal stress difference. Now, if we talk and there is different type of a rheometers available as on date based on the uses, based on the fluid use, based on the various kind of other approaches. Let us talk about the sliding plate rheometer. Now, using this is the typical figure of the sliding plate rheometer, where we are having the actuator rod, then we are having the transducers, cantilever beams, stationary plates, this one is a stationary plate and here this is the spacer shine and we subject the polymer over here and this plate is moving one and this is the linear bearing table.

Introduction

- Rheometers are specialized tools that are needed for evaluating fluid models and measuring rheological parameters.
- They are necessary for complex measurements in research, including the development and assessment of flow models as well as the analysis of viscosity and normal stress differences.



The Sliding Plate Rheometer



- Using a sliding plate rheometer, the simplest type of shear between two parallel plates can be detected.
- Two flat plates make up this sort of rheometer, with the upper plate moving back and forth at a consistent speed while the lower plate remains fixed.

Fig. Schematic of sliding plate rheometer with shear stress transducer

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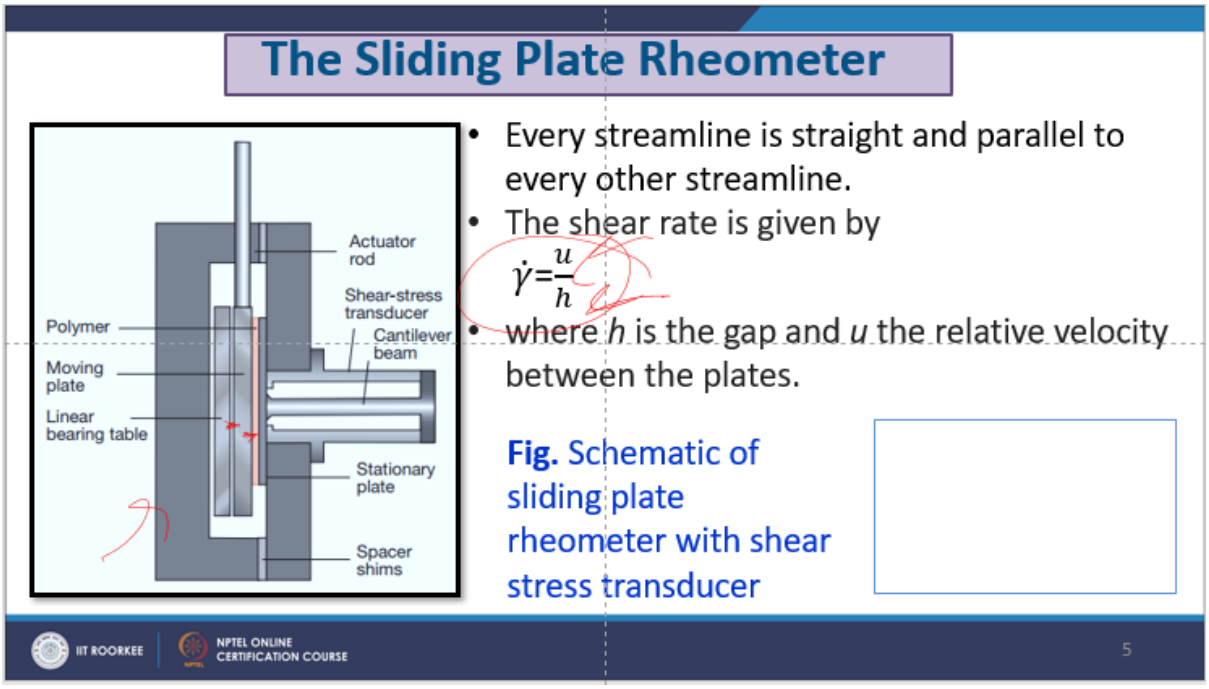
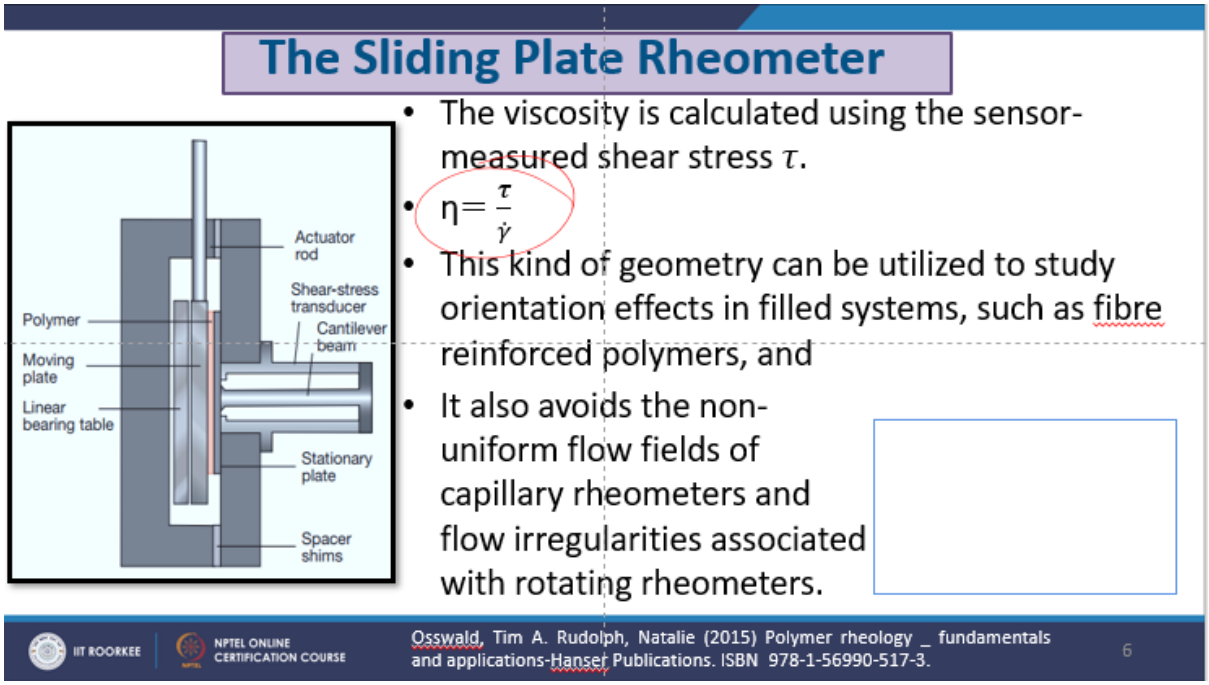
So, here this the sliding rheometer, this is the simplest type of shear between the two parallel plates. Now, the two flat plates make this top of this type of a sort of a rheometer with the upper plate moving back and forth at the constant consistent speed while the lower plate remains fixed. This is this like this. Now, every streamline is a straight and parallel to every other streamline. So, the shear rate can be given as gamma dot is equal to U over H, where H is the gap, the gap between the plates and U is the relative velocities between the plates.



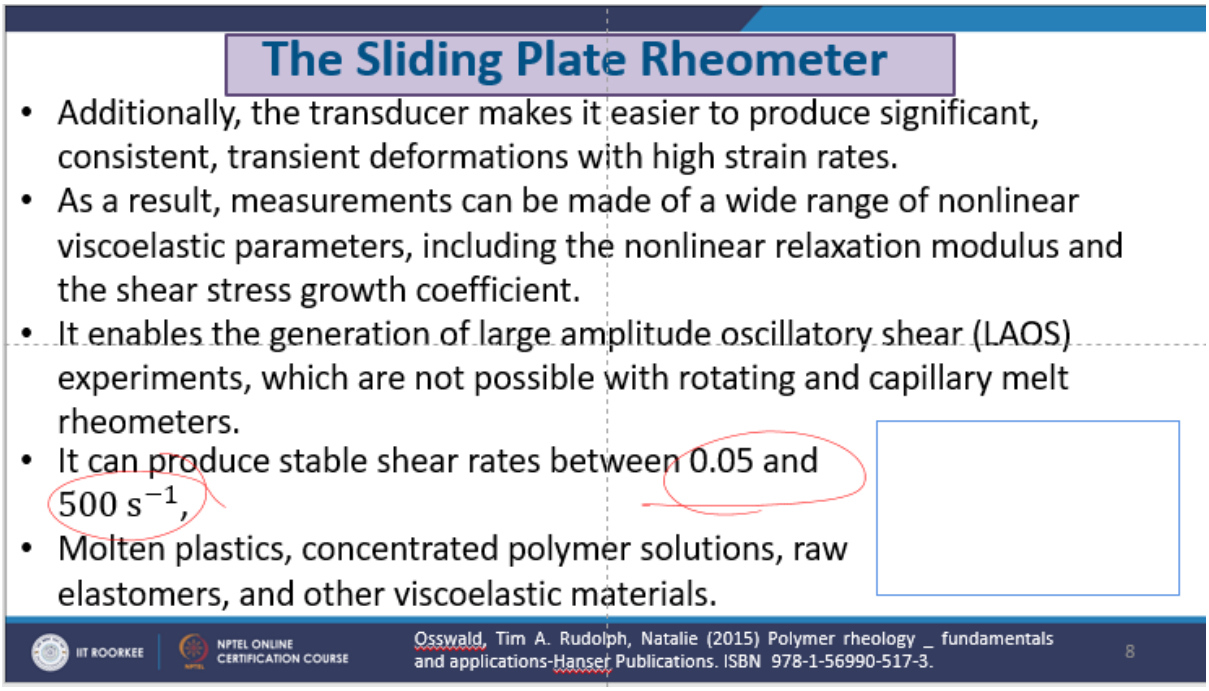
$\dot{\gamma}=\frac{u}{h}$

This is the schematic of the sliding plate rheometer with the shear transducer. Now, the viscosity is calculated using the sensor measured shear stress tau. So, neta is equal to tau over gamma. This kind of a geometry can be utilized to study the orientation effect in filled system which are very common in the polymetric system, can be used in the fiber reinforced polymers and can be used in different type of molding operation, injection molding all those things. Now, it is also a why it is the non-uniform flow field of capillary rheometers and flow irregularities associated with the rotating rheometers.

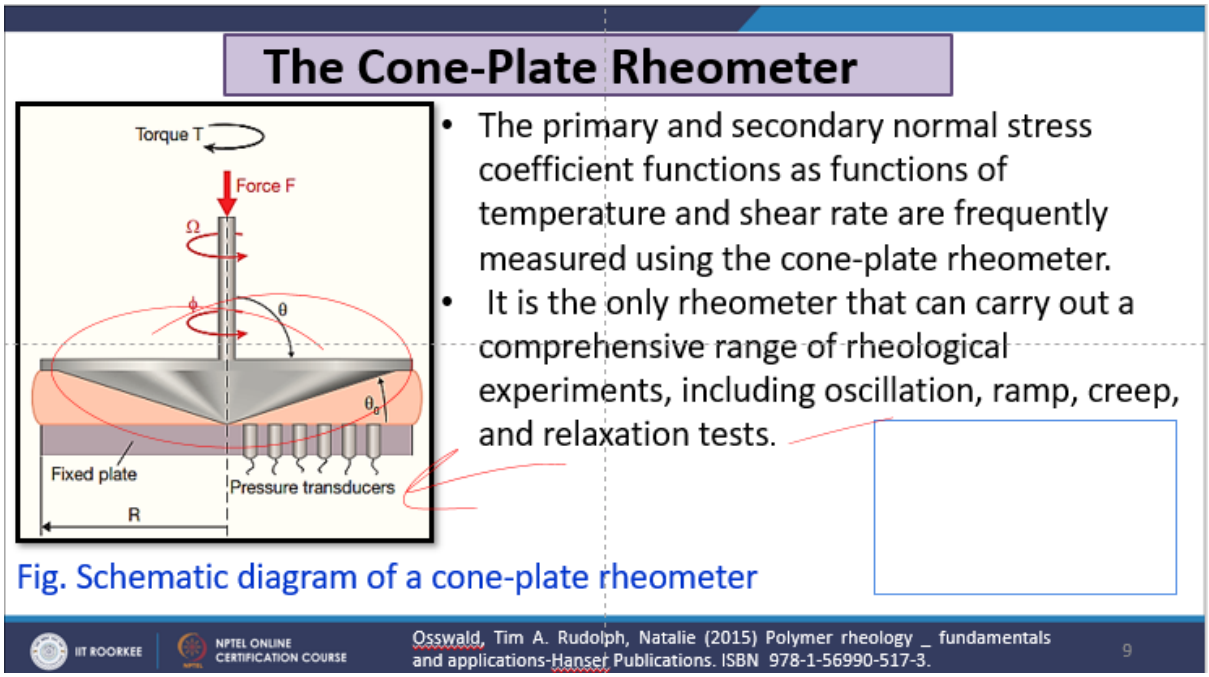


$\eta=\frac{\tau}{\dot{\gamma}}$

Now, the shear stress can be measured directly on a tiny section of one of the plates using the sliding plate rheometer and a shear stress transducer that is usually created to specify this particular purpose. It also enabled the measurement of linear and nonlinear viscoelastic properties over a broad range of shear rates by eliminating the source of inaccuracy in traditional sliding plate rheometers. Additionally, the transducers make it easier to produce significant, consistent transient deformation with a high strain rate. Now, as a result, measurements can be made to a wide range of nonlinear viscoelastic parameters including the nonlinear relaxation modulus and the shear stress growth coefficient. So, it enables the generation of a large amplitude oscillatory shear.



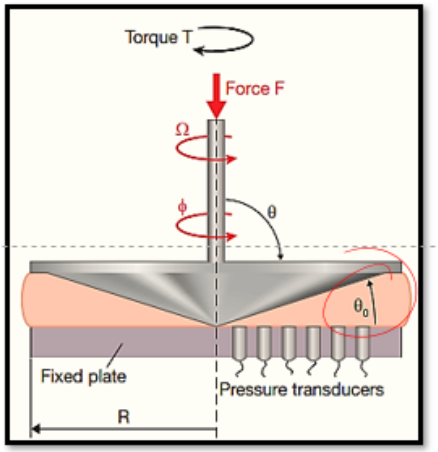
This experience which was not possible with the rotating and a capillary melt rheometer, it can produce stable shear rates between 0.05 and 500 per second. Molten plastic concentrated polymer solutions, royal osmosis and other viscoelastic materials they are the better candidate for this particular system. Let us talk about the cone plate rheometer. This is the schematic diagram of the cone plate rheometer, this cone type of a structure.



The primary and a secondary normal stress coefficient function as a function of temperature and shear rate are frequently measured using the cone plate rheometer. Now, it is only rheometer that can carry out a comprehensive range of rheological experiments including oscillations, RAM, creep and relaxation tests. Now, this particular figure depicts the cone plate rheometer geometry as well as the proper filling. Now, to ensure that the gap is entirely filled and to account for the of the resin Inflow caused by the Wissenberg effect, it is recommended to add extra melt between the cone and the plate. This is the fixed plate and this is the cone.

The polymer shear gap is the remaining space between the cone and the plate. The cone angle theta naught and the radius r establishes the geometry. Now, different parameters they are measured experiment like cone angular velocity, the torque required to turn the cone t tau, the total force normal to the fixed plate F and the pressure distribution on the fixed plate as a function of r. Now, angle theta naught is commonly between 1 degree and 4 degree. Therefore, the shear rate can be thought of as constant for theta naught is less than to 3 degree and it is given by gamma dot theta psi is equal to omega theta naught.

The Cone-Plate Rheometer



- The angle θ_0 is commonly between 1° and 4°, therefore the shear rate can be thought of as constant (for θ_0 < to 3°) and is given by

$$\dot{\gamma}_{\theta\varphi}=\frac{\Omega}{\theta_0}$$

- If the torque acting on the cone and the plate are equal, then the shear stress can also be thought of as constant and related to the observed torque.
- $\tau \cdot = \frac{3T}{2\pi R^3}$

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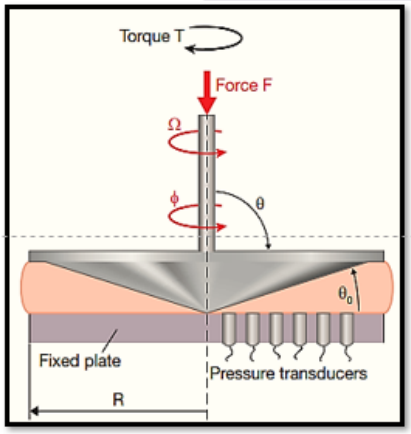
If the torque acting on the cone and the plate are equal, then the shear stress can also be thought as a as constant and related to the observed torque. So, torque can be given as tau dot is equal to 3 t over 2 pi r cube. Now, the viscosity function this can now be obtained from theta dot gamma theta psi is equal to tau dot theta psi. Over gamma dot theta psi is equal to omega theta naught 3 t over 2 pi r cube. Now, by measuring the force F normal to the fixed plate that is necessary to hold the cone plate, the primary normal stress coefficient function psi 1 is computed from the primary normal stress differential N 1.

The Cone-Plate Rheometer



- The viscosity function can now be obtained from

$$\eta\,(\dot{\gamma}_{\theta\varphi})=\frac{\dot{\tau}_{\theta\varphi}}{\dot{\gamma}_{\theta\varphi}}=\frac{\Omega}{\theta_0}\frac{3T}{2\pi R^3}$$

- By measuring the force, *F*, normal to the fixed plate that is necessary to hold the cone in place, the primary normal stress coefficient function, ψ_1, is computed from the primary normal stress differential, N_1

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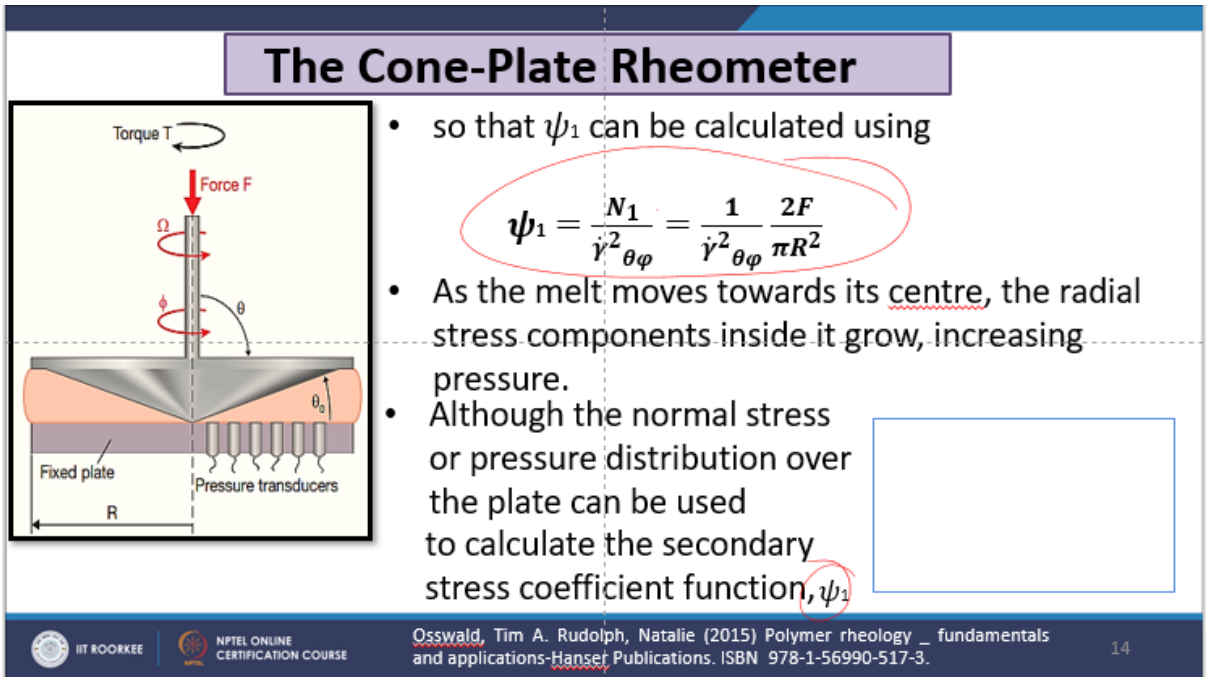
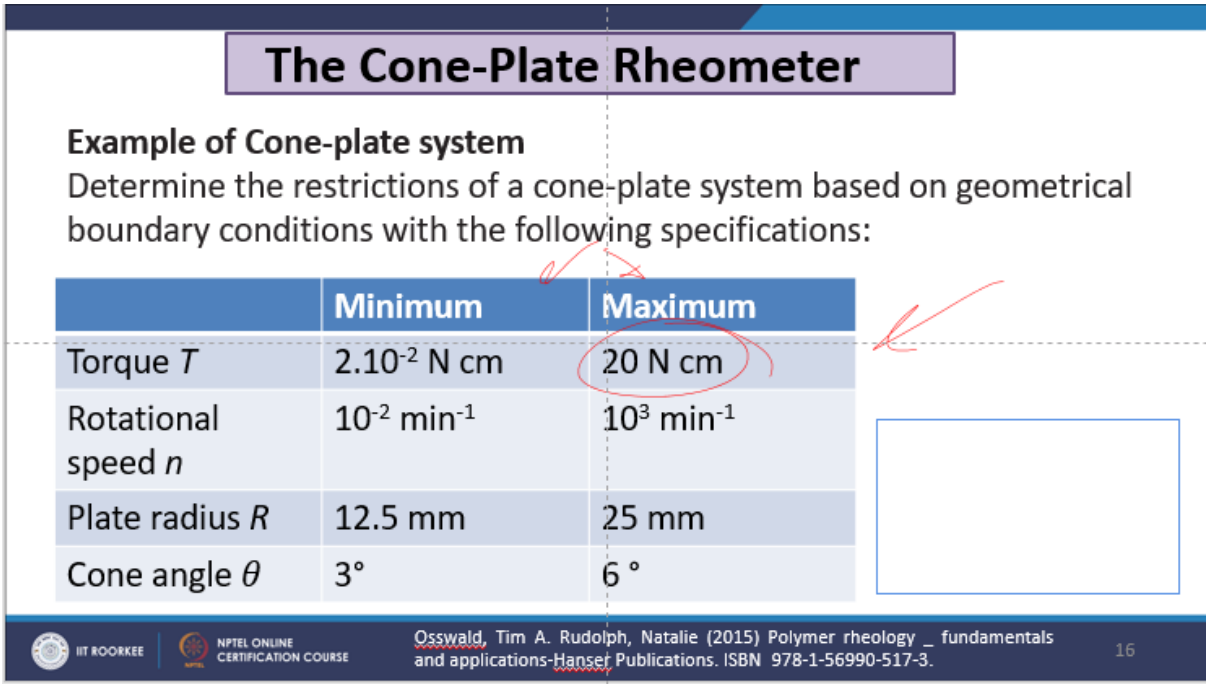
So, the psi 1 can be calculated using this particular formula that is psi 1 is equal to N 1 over gamma dot square theta psi is equal to 1 over gamma dot square theta psi 2 F over pi r square. As the melt moves towards the centre, the radial stress component inside it grow increasing pressure although the normal stress or pressure distribution over the plate can be used to calculate the secondary stress coefficient function psi 1. Now, the accurate data collection is exceedingly challenging. However, a rough estimate can be made using this particular general principle that is psi 1 is equal to 0.



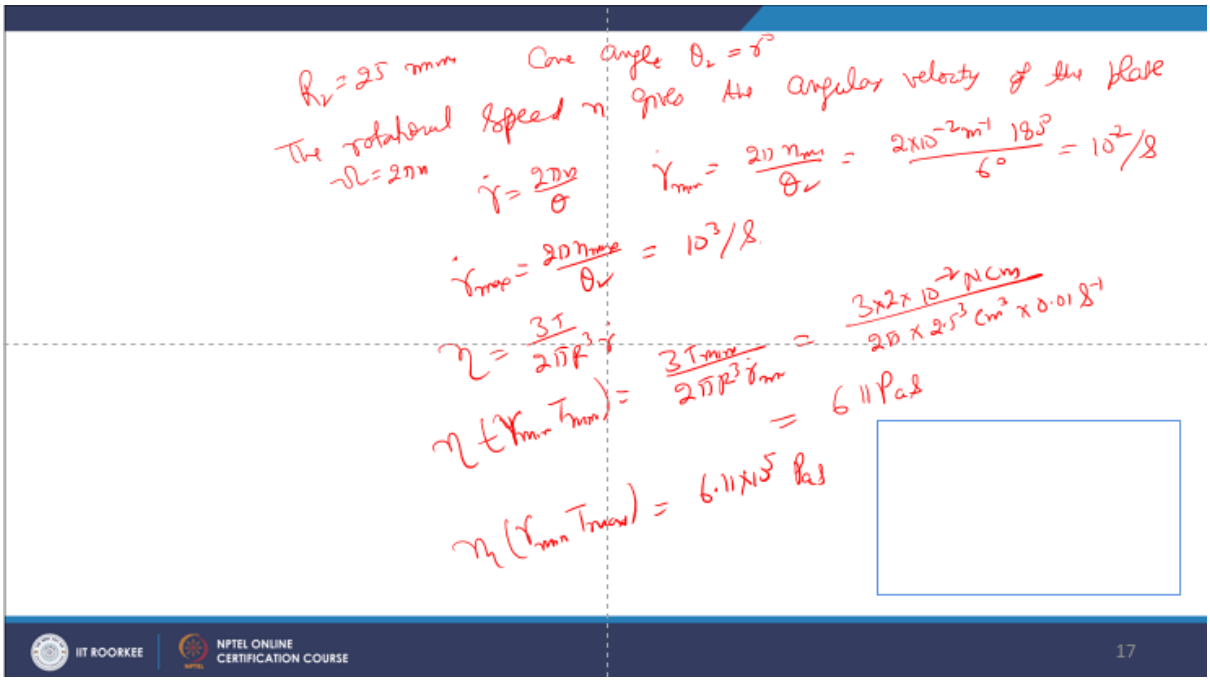
$$\eta\,(\dot{\gamma}_{\theta\varphi}) = \frac{\dot{\tau}_{\theta\varphi}}{\dot{\gamma}_{\theta\varphi}} = \frac{\Omega}{\theta_0} \frac{3T}{2\pi R^3}$$

$$\boldsymbol{\psi 1} = \frac{N_1}{\dot{\gamma}^2_{\theta\varphi}} = \frac{1}{\dot{\gamma}^2_{\theta\varphi}} \frac{2F}{\pi R^2}$$

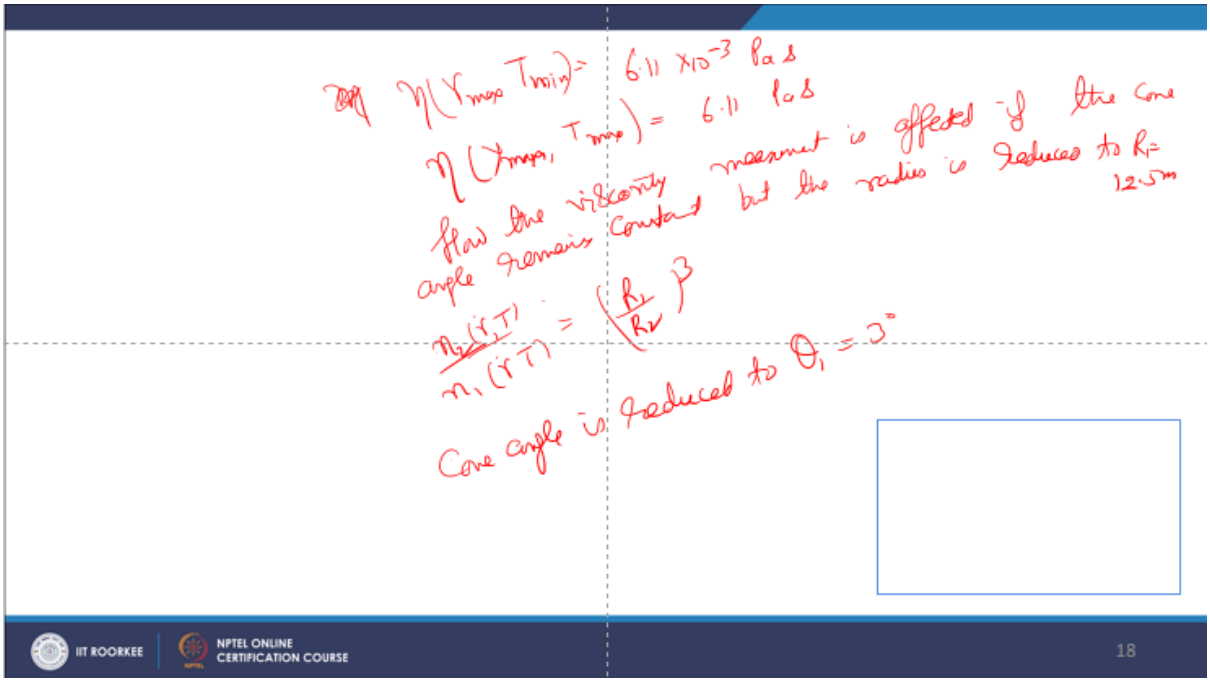
1 psi 2. Now, let us take example of a cone plate system. You need to determine the restriction of a cone plate system based on geometrical boundary condition. This can be the specification like torque T is minimum and maximum value is given 2 into 10 to the power minus 2 Newton centimetre and the maximum is 20 Newton centimeter. Rotational speed is equal to 10 to the power is 2 per minute and 10 to the power maximum is 10 to the power 3 per minute and the plate radius is given that is 12.5 mm and maximum is 25 mm and cone angle theta is 3 degree and minimum and 6 degree maximum.



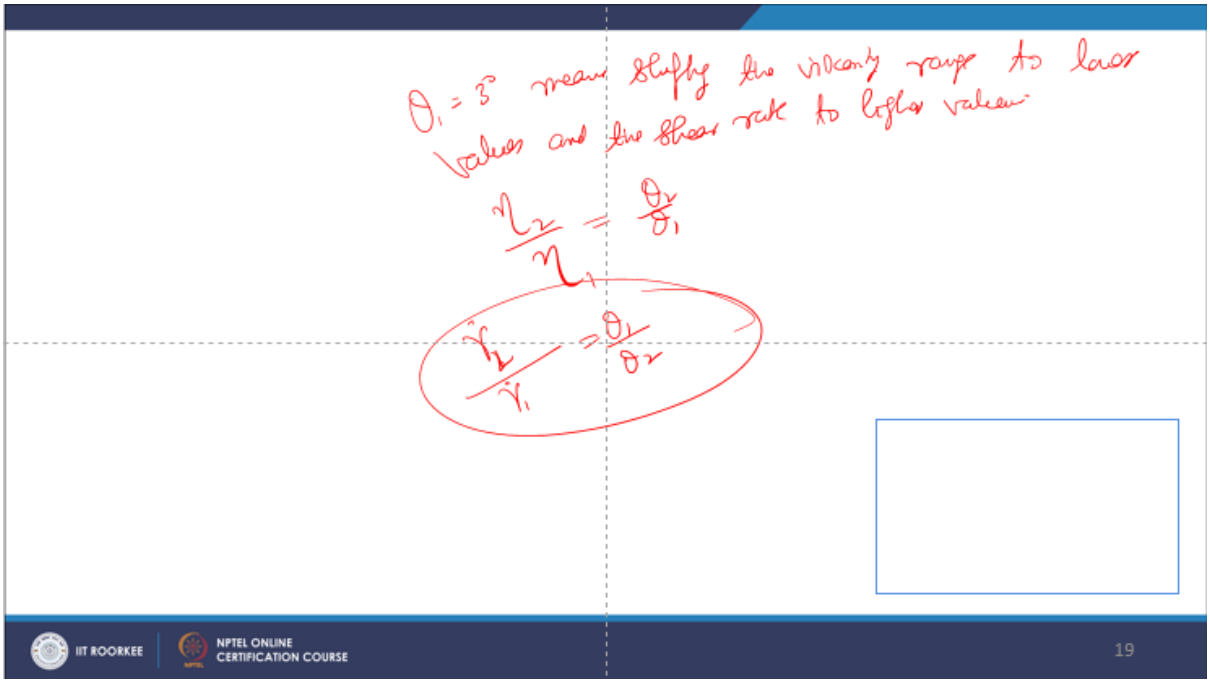
So, the first the viscosity and shear rate limits and this should be determined from the maximum plate radius r 2 is equal to 25 mm and cone angle theta is equal to 0.1 into theta 2 is equal to 6. So, the rotational speed n gives the angular velocity of the plate omega is equal to 2 pi n and the minimum and the maximum shear rate of the cone plate and the maximum shear rate of the cone plate geometry can be determined by gamma dot is equal to 2 pi n theta and gamma minimum 2 pi n minimum over theta 2 which if we substitute this thing into 10 to the power minus 2 180 degree 6 that is comes out to be minus 2 per second and gamma dot maximum 2 pi and maximum theta 2 is equal to 10 cube per second. So, the viscosity can be given as Neta is equal to 3 t over 2 pi r cube gamma dot, Neta r cube gamma dot, eta into gamma minimum t minimum this is equal to 3 T minimum 2 pi r cube gamma minimum, which is equal to 3.2, 3 into 2 into 10 to the power minus 2 over 2 pi into 2.



5 cubes into 0.01 this is equal to 611. So, Neta gamma minimum t maximum is equal to 6.11 into 10 to the power 5 Pascal second. Now, Neta gamma maximum t minimum is equal to 6.11 into 10 to the power minus 3 Pascal seconds.

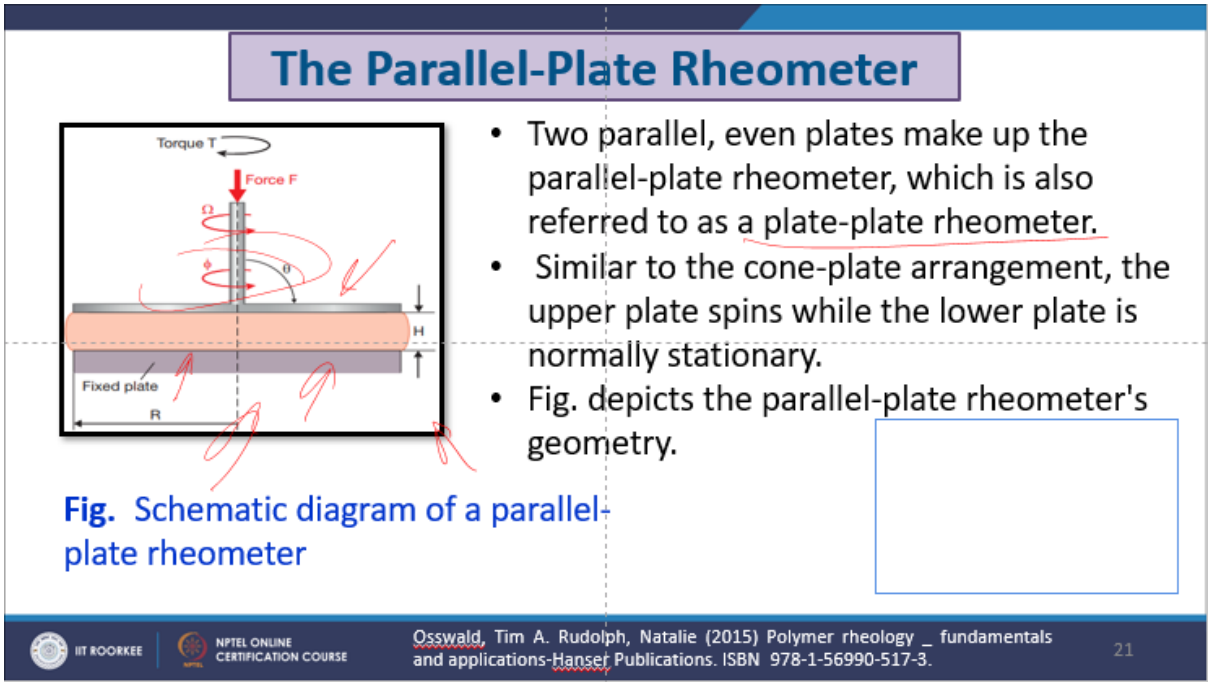


Neta gamma maximum t maximum is equal to 6.11 Pascal second. Now, we can evaluate how the viscosity measurement is affected if the cone angle remains constant, but the radius is reduced to r 1 is equal to R1 is equal to 12.5 mm. So, the reducing the radius by half to 12.



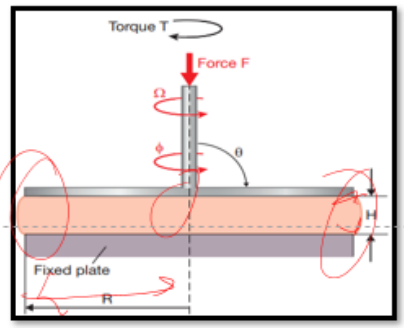
5 mm means shifting the viscosity to higher values according to eta 2 into gamma dot t over eta 1 gamma dot t is equal to r 1 over r 2 cube. The shear rates the shear rate range stays constant because it is unaffected by the radius. So, finally, we can evaluate how the viscosity measurement is affected when the radius remains constant, but the cone angle is reduced to theta 1 is equal to three degree.

So, Now, reducing the cone angle theta 1 is equal to 3 means shifting the viscosity range to lower values and the shear rate to higher values. So, if we take neta 2 over neta 1 theta 2 over theta 1 and gamma 2 dot over gamma 1 dot 2 theta 1 over theta 2.



The parallel plate rheometer now the two this is the schematic diagram of parallel plate rheometer that two parallel even plates make up the parallel plate rheometer which is also referred to as the plate plate rheometer. Now, similar to the cone plate arrangement the upper plate spins while the lower plate is the lower plate normally stationary. So, this is the stationary field and this plate rotates this is the typical geometry of the parallel plate rheometer. Now, to ensure the uniform flow throughout the whole gap the distance of the gap between the plate is maintained as h and this should be substantially smaller than the radius of the plate r. The shear rate increases with the distance from the rotational speed and that is small r is in between 0 and r which is drawback as compared to the cone plate arrangement.

The Parallel-Plate Rheometer



- To ensure uniform flow throughout the whole gap, the distance or gap between the plates, *H*, should be substantially smaller than the radius of the plates, *R*.
- The shear rate increases with distance from the rotational axis ($0 \leq r \leq R$), which is a drawback as compared to the cone-plate arrangement.
- The shear rate is $\gamma = 0$ in the centre ($r = 0$), and it reaches its maximum near the edge ($r = R$).

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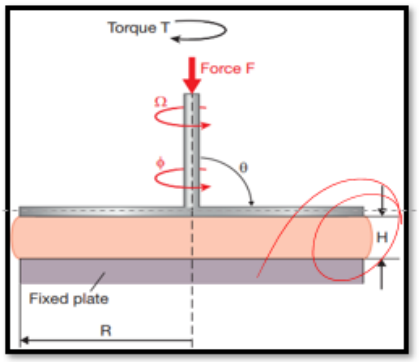
And the shear rate gamma is equal to 0 at the center r is equal to 0 and it reaches to the maximum near the edge that is r is equal to r. The maximum shear rate at the edge is utilized to analyze the measurements to analyze the measurements. And now as per this equation the gap height h also influences the shear rate. So, the gamma gamma dot r is equal to r omega over h the maximum shear rate at the edge of the plate is given by gamma dot r is equal to r omega over h. Now, these equation illustrate the importance of the gap height.

The Parallel-Plate Rheometer



- The maximum shear rate at the edge is utilised to analyse the measurements. As shown in eq, the gap height *H* also influences the shear rate.

$$\dot{\gamma}_r = \frac{r.\Omega}{H}$$

- The maximum shear rate at the edge of the plate is given by

$$\dot{\gamma}_R = \frac{R.\Omega}{H}$$

- These equations illustrate the importance of the gap height

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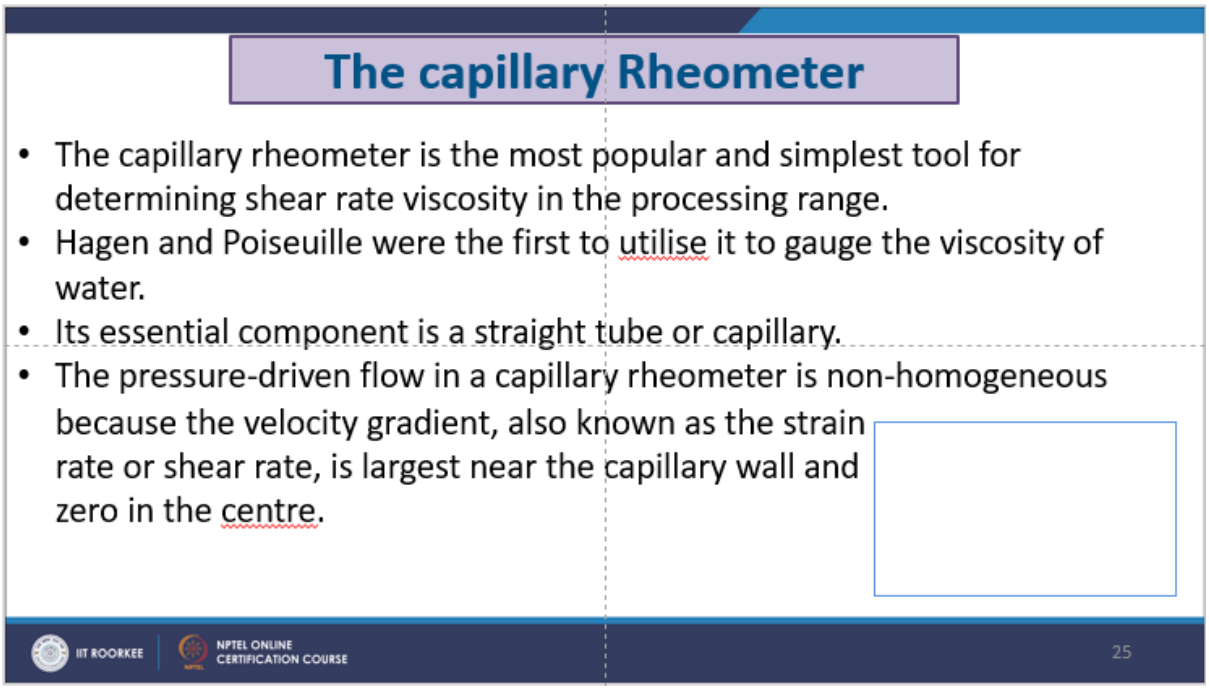
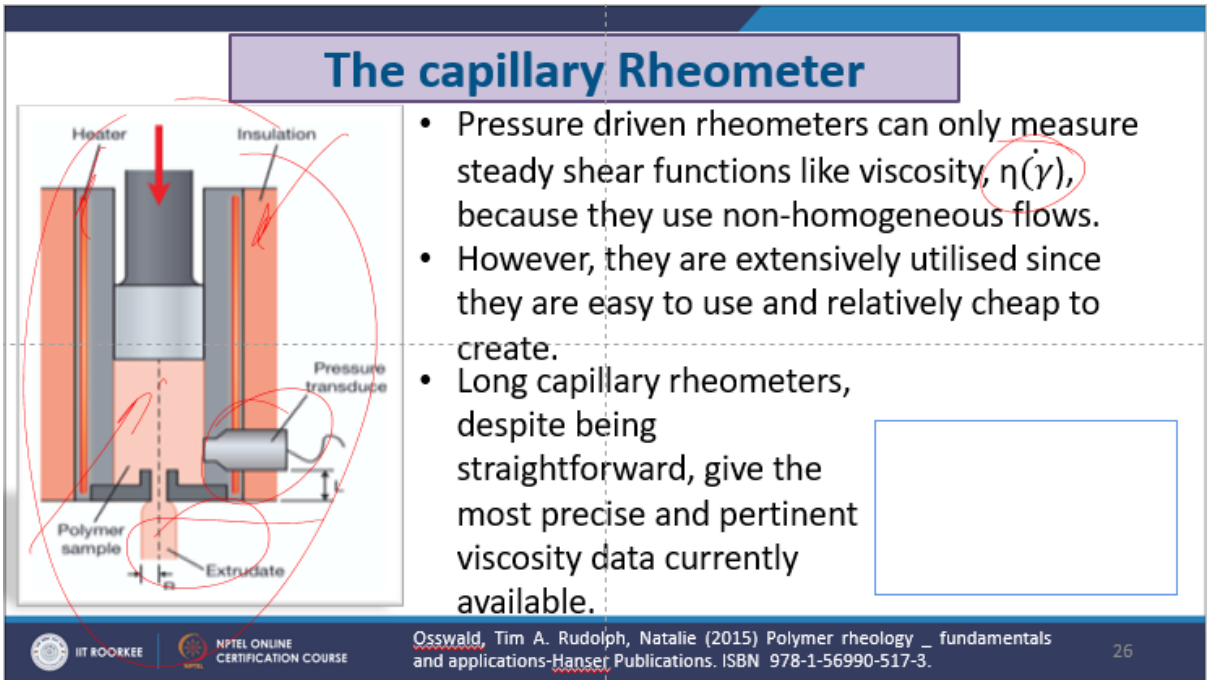
So, this is the gap height. Now, an increase in the gap causes a small shear rate at the same rotating speed and this device can therefore accommodate a wide range of shear rates. The cone plate rheometer is more popular than the plate the parallel plate rheometer for a variety of applications. However, the parallel plate system offers some benefits over the cone plate system. Although the

parallel plate system has several benefits the cone plate system is preferred because it offers a consistent shear rate in the conical gap. Let us talk about the capillary rheometer.



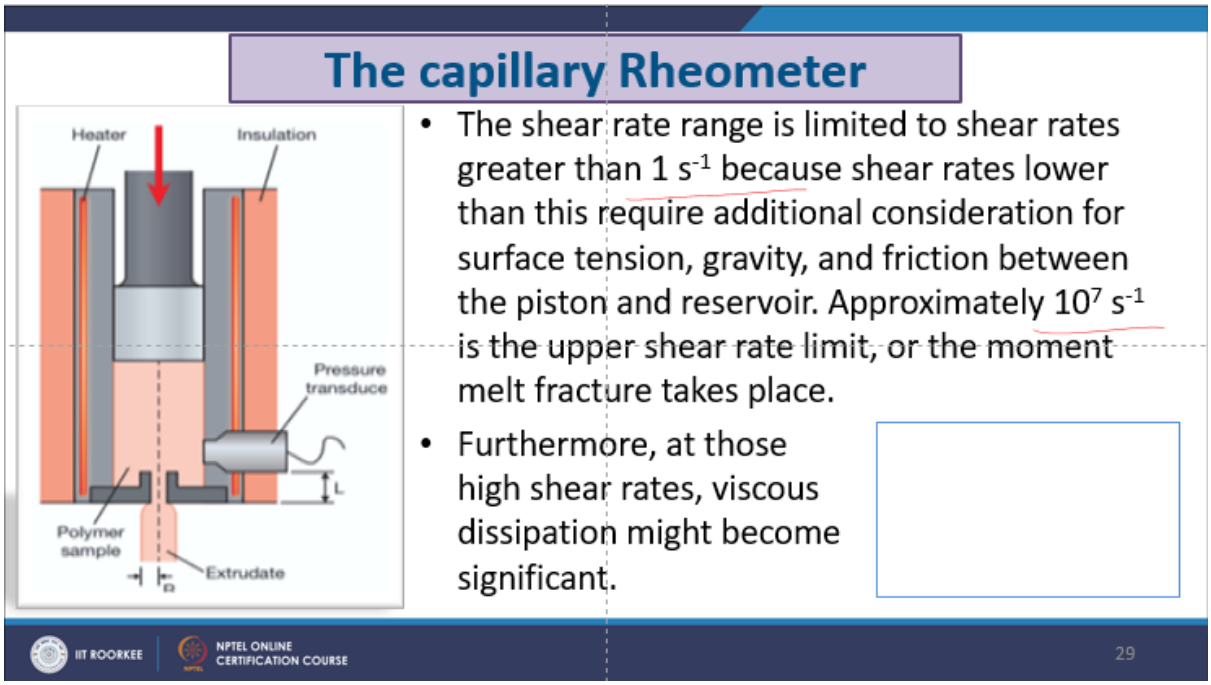
The capillary rheometer is the most popular and the simplest tool for determining the shear rate viscosity in the processing range. Hagen and Poiseuille they were the first to utilize to gauge the viscosity of water. Now, it is essential component and its essential component is a straight tube or capillary. The pressure driven flow in a capillary rheometer is non-homogeneous because the velocity gradient also known as a strain rate or shear rate is largest near the capillary wall and 0 at the center.



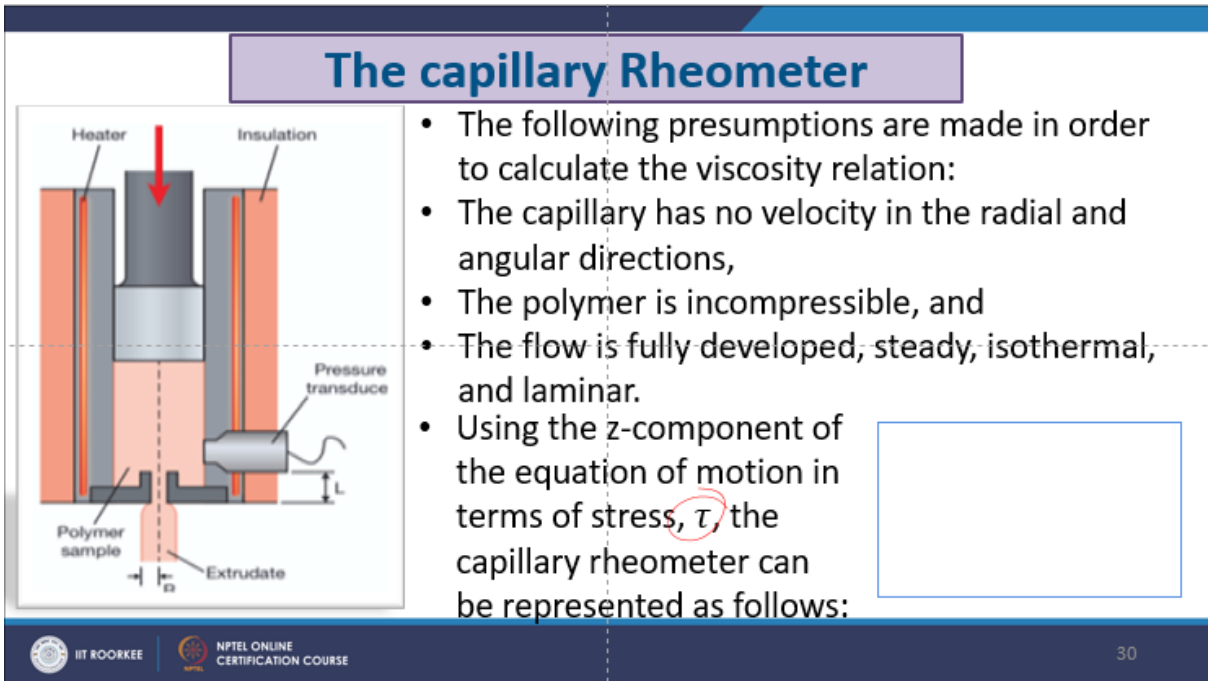
This is the typical figure. Now, the pressure driven rheometer can only measure the steady shear function like viscosity because they use the non-homogeneous flows. However, they are extensively utilized since they are easy to use and relatively cheap to create. Now, long capillary rheometer

despite being straightforward give the most precise and pertinent viscosity data which is currently available. Now, here you see the anatomy of the capillary rheometer. There is a heater and this well insulated.

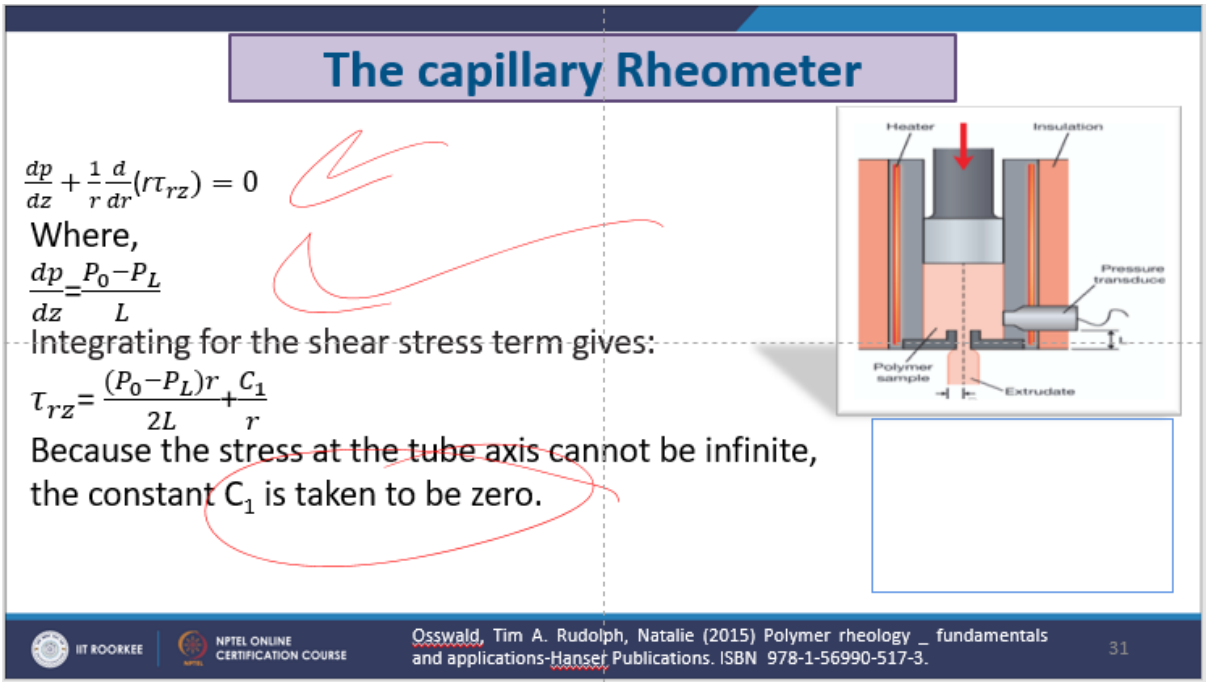
These are the pressure transducers subject to the polymer samples which extrudates. Another significant benefit is that in contrast to other kind of a rheometer like cone plate rheometer, capillary rheometer has no open surface in the test regime. Now, as shear rates greater than 100 per second, the capillary rheometer may be the sole viable option for detecting the strain rate dependent viscosity of the polymer melt. Now, this is crucial for the procedures like mixing extrusion injection molding that involve higher rates of deformation. The capillary rheometer can simply be mounted to the end of a screw or a ram type extruder for online measurement due to their simple design and requirement for only a pressure head at the entrance.



Now, this makes the capillary rheometer an efficient tool for industry. The shear rate range is limited to the shear rates greater than 1 per second because shear rate is lower than this requires additional consideration for surface tension, gravity and friction between the piston and reservoir. Approximately 10 to the power 7 per second is the upper shear rate limit or the moment melt fracture takes place. Furthermore, at those high shear rate viscous dissipation might become significant. There are certain presumptions that are made in order to calculate the viscosity relation.



The capacity the capillary has no velocity in the radial and angular direction. The polymer is incompressible and the flow is fully developed steady isothermal and laminar. So, using the z component of the equation of motion in terms of stress tau, the capillary rheometer can be represented as per this particular mathematical representation where d p over d z plus 1 over r d r d over d r r tau r z is equal to 0 where d p over d z is equal to p naught minus p l over l. Now, if we integrate the shear stress term then tau r z is equal to p naught minus p l into r over 2 l plus c 1 over r because of this the because of the stress at the tube axis cannot be infinite and the constant c 1 is taken to be 0.

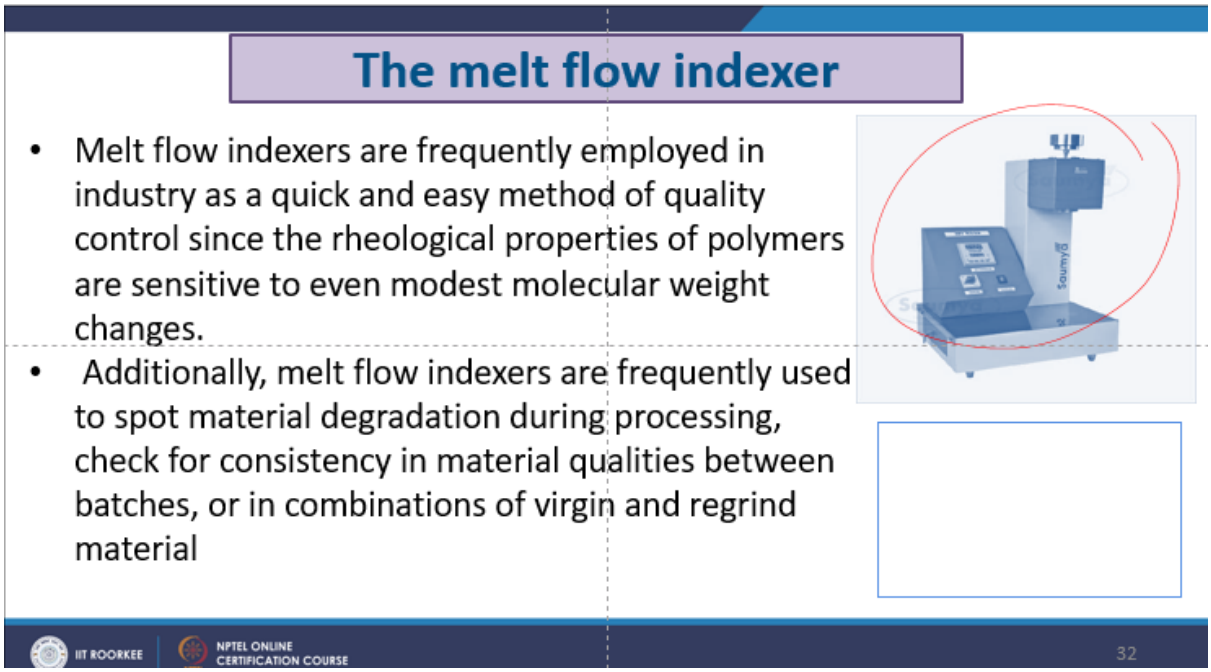


$$\frac{dp}{dz} + \frac{1}{r}\frac{d}{dr}(r\tau_{rz}) = 0$$

$$\frac{dp}{dz} = \frac{P_0 - P_L}{L}$$

$$\tau_{rz} = \frac{(P_0 - P_L)r}{2L} + \frac{C_1}{r}$$

Let us talk about the melt flow indexer. The melt flow indexers are frequently employed in the industry as a quick and easy method of quality control since the rheological properties of polymer are sensitive to even modest molecular weight changes. Additionally, the melt flow indexers are frequently used to spot the material degradation during the processing check for consistency in material qualities between the batches or in combination of virgin and regrained material. This is the typical anatomy of melt flow indexer where we are having the capillary, the polymer then the thermometer and all these things are very common in nature. This is a single point measurement is taken out on a ram type extruder or extrusion plastometer under the standard testing setting conditions unique to each polymer class. ASTM D1238 outlined the recommended process for determining the flow rate of thermoplastic using an extrusion plastometer.



Let us talk about the extensional rheometry. The polymer processes like film blowing, blow molding, thermoforming, fiber spinning, foam generation they are described by the elongational deformation rather than shear. Large extensional component they are present in the flow near the flow front during mold filling as well as in the converging or diverging regions of dyes and molds. Because of their high molecular orientation or the orientation of filler with the large aspect ratios, the regions of extensional flow have a significant impact on the final parts qualities. Now, this is the explored extensional flow geometries like simple extension, this is the squeezing, this is the sheet stretching.

Extensional rheometry

- Polymer processes like film blowing, blow moulding, thermoforming, fibre spinning, and foam generation are described by elongational deformation rather than shear.
- Large extensional components are present in the flow near the flow front during mould filling as well as in the converging or diverging regions of dies and moulds.
- Because of their high molecular orientation or the orientation of fillers with large aspect ratios, regions of extensional flow have a significant impact on the final part's qualities.

The extensional behavior of the polymer melt cannot be determined using the apparatus which you discussed in the previous section for measuring the shear behavior of polymer. Although practically every manufacturing process exposes processors to extensional flow, extensional rheometry is the least studied area of rheology. The most common modes of deformation that generate extensional flow they are shown in this particular figure. Now, stretching a polymer rod at a high temperature is the easiest method to determine the extensional viscosity. Now, due to the rods decrease cross sectional area, when we stretch then the cross-sectional area will decrease.

Extensional rheometry

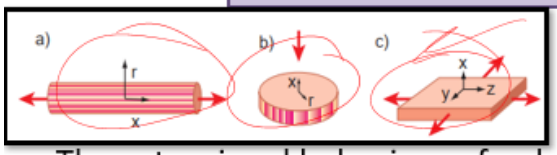
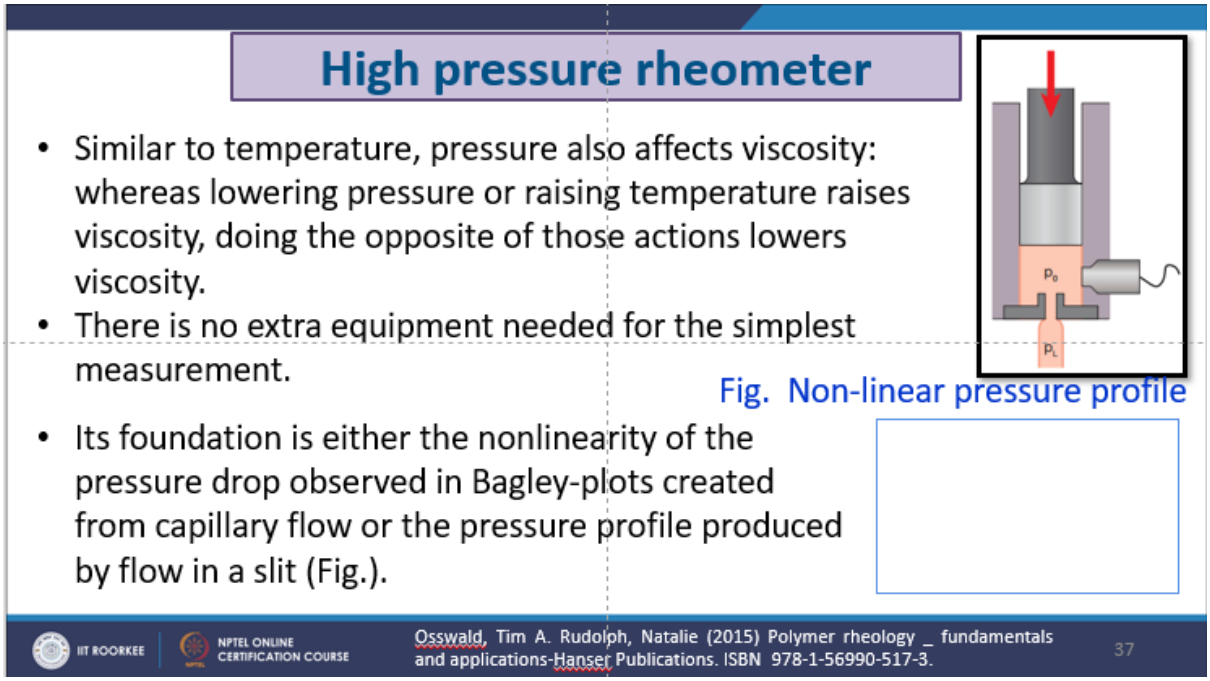


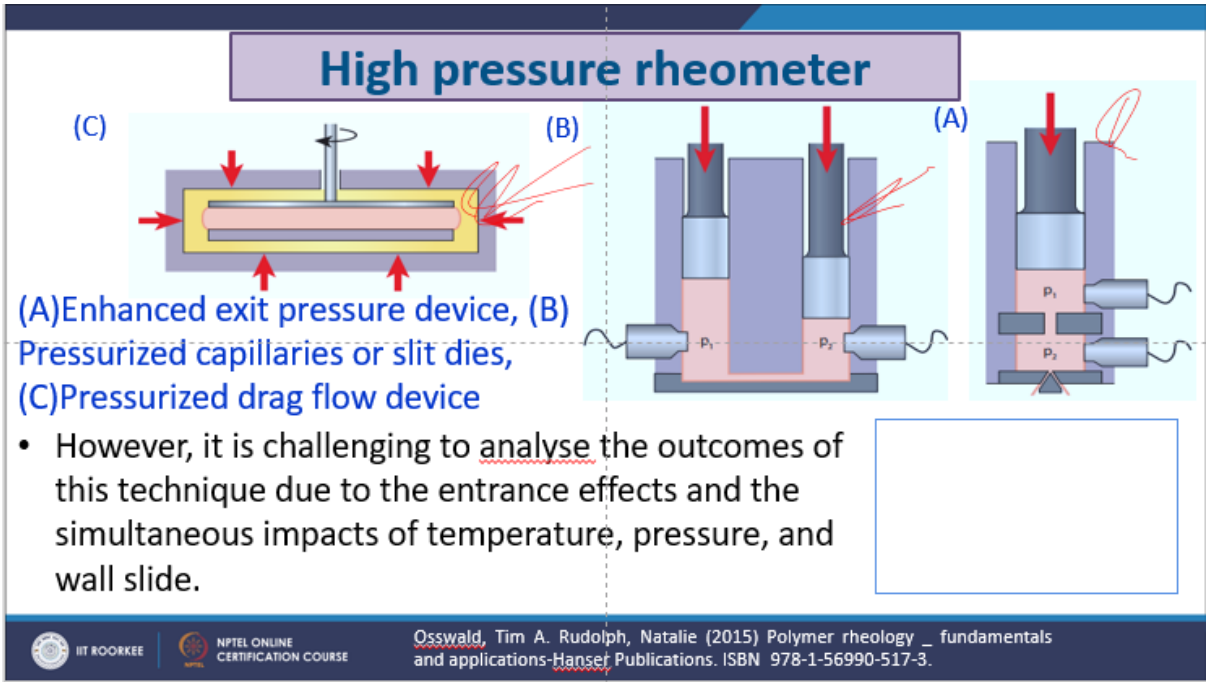
Fig. Purely extensional flow geometries; a: simple extension, b: squeezing, c: sheet stretching

- The extensional behaviour of polymer melts cannot be determined using the apparatus described in the previous sections for measuring the shear behaviour of polymers.
- Although practically every manufacturing process exposes processors to extensional flow, extensional rheometry is
- the least studied area of rheology.
- The most common modes of deformation that generate extensional flows are shown in Figure

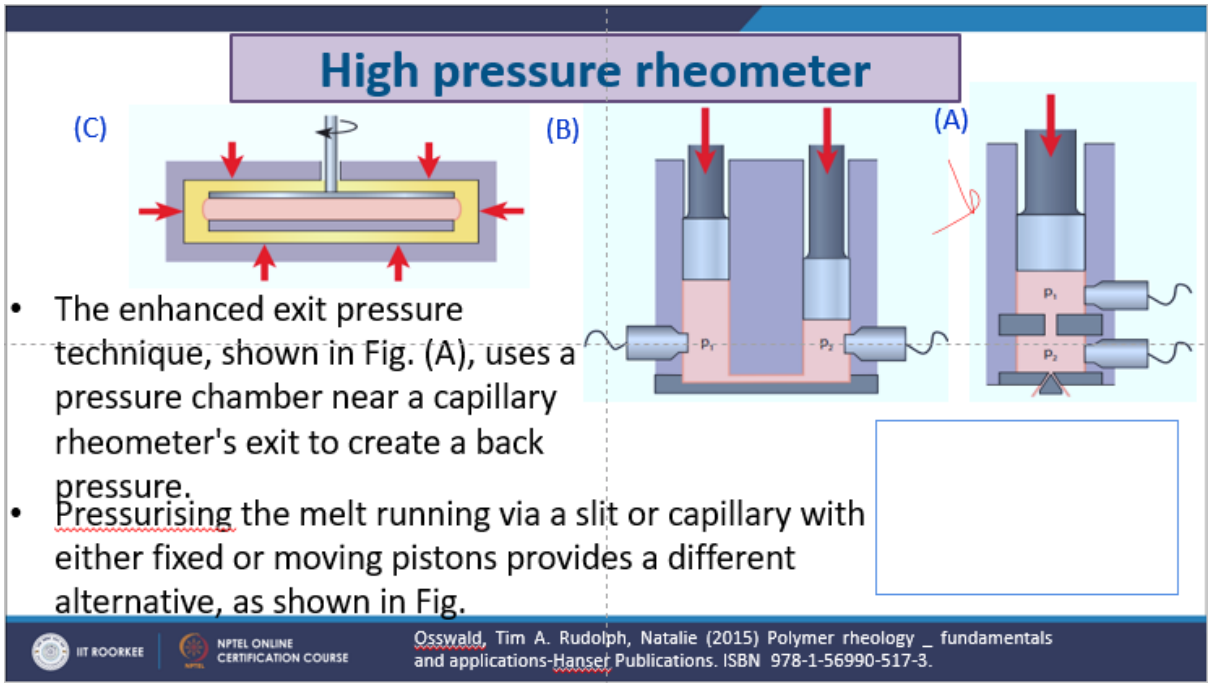
The stretch rate must constantly rise in order to keep the strain rate constant. This uniaxial deformation is crucial for processes like fiber spinning and those requiring converging flows the least understood area of rheology and depends on the materials macromolecular structure. Let us talk about the high pressure rheometry. Now, similar to the temperature, pressure also affects the

viscosity whereas, lowering the pressure or raising the temperature raises the viscosity doing the opposite of those sections lower the viscosity. There is no extra equipment needed for the simplest measurement.



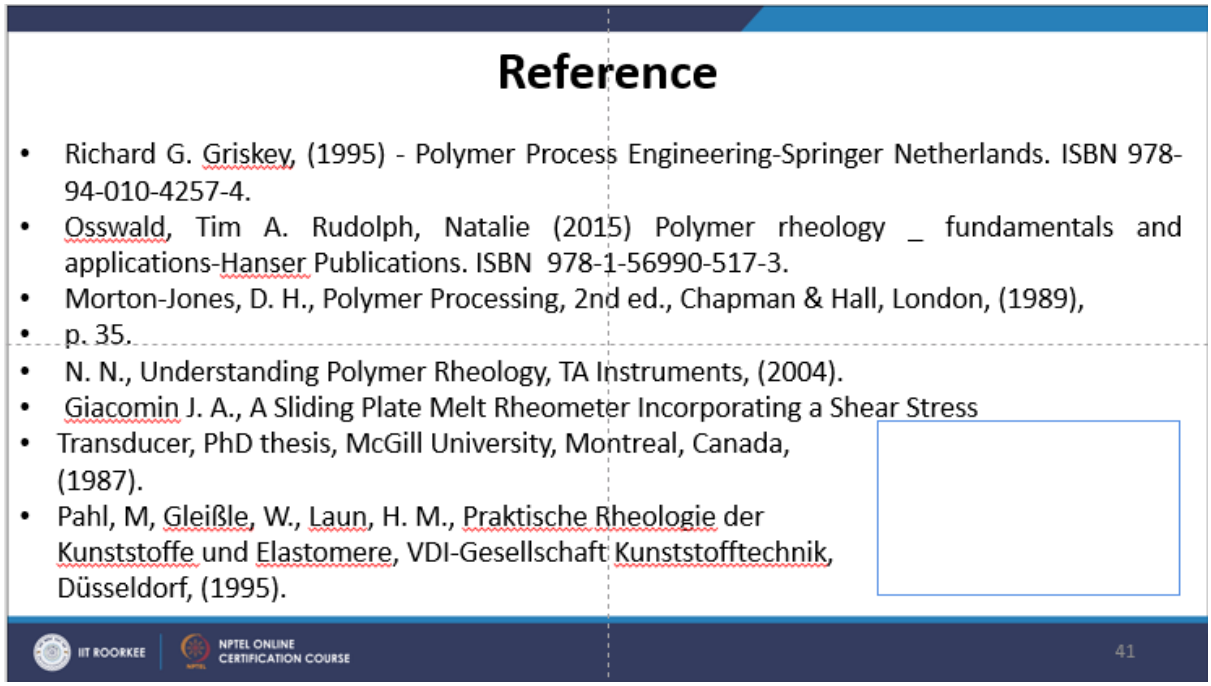


Its foundation is either a nonlinearity of the pressure drops observed in a Bagley plots created from the capillary flow or the pressure profile produced as per the flow in a slit.



Now, here you see that different type of high-pressure rheometers like enhanced exit, the enhanced exit, the pressure device then pressurized capillary or slit dies, then pressurized drag flow device. However, it is challenging to analyze the outcome of this particular technique due to the intrinsic effect and the simulation impacts of the temperature, pressure and wall slides. Now, the direct approaches which are shown in these three figures need specialized equipment to test viscosity at high pressure. The enhanced exit pressure technique shows in this particular figure, this uses a pressure chamber near a capillary rheometer to create a back pressure.

Now, pressurizing the melt running via a slit or a capillary with either fixed or moving piston provides a different alternative.



So, dear friends in this particular segment, we discussed different type of a rheometer for measuring the viscosity as we discussed that the rheological behavior plays a very vital role to achieve the final shape of any polymer, whether it is injection molding, compression molding and all kind of polymeric operations. So, for your convenience, we have enlisted several references, especially pertaining to the polymer rheology, you can utilize those references as per your requirement. Thank you very much.