Polymer Process Engineering

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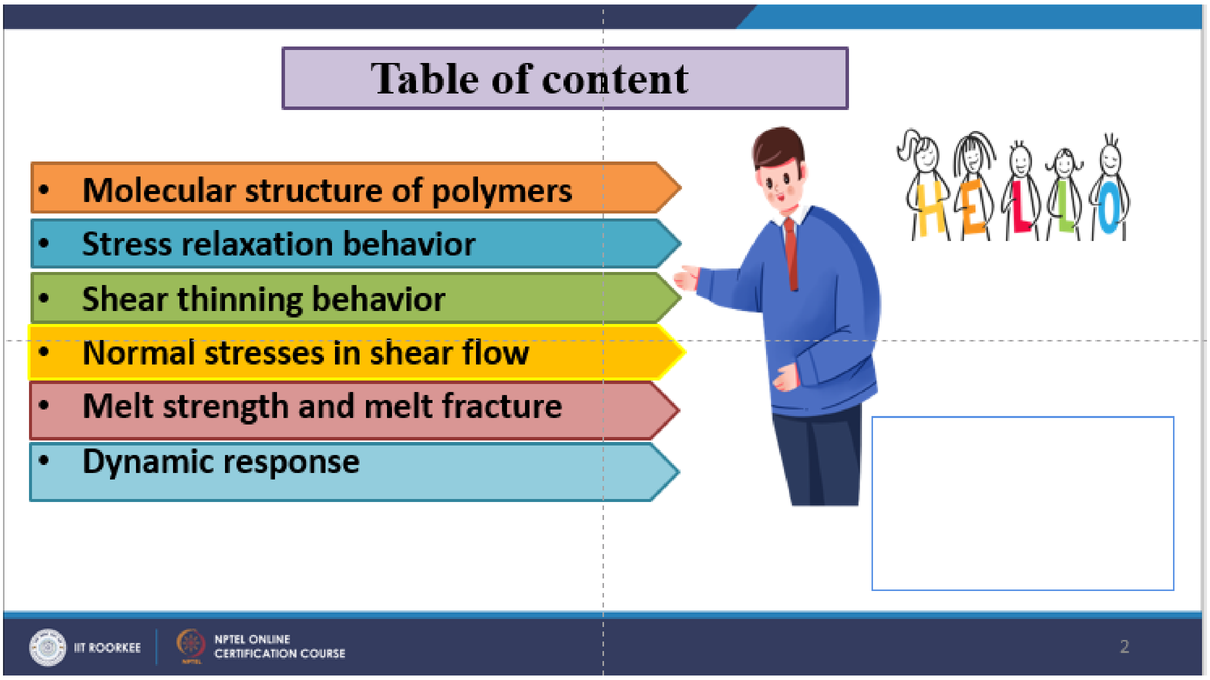
Lecture – 12

Applied polymer rheology: Structure and properties of deforming polymer

Hello friends, welcome to the applied polymer rheology under the edges of polymer process. Engineering, dear friends, here we are going to discuss about the structure and properties of deforming polymers. And this particular segment, we are going to discuss about the molecular structure of polymers. Then we will discuss about the stress relaxation behaviour, shear thinning behaviour and the normal stresses in shear flow, melt strength and a melt fracture and dynamic response. Let us talk about the molecular structure of polymers. The macromolecular structure known as polymer and they are frequently created synthetically, but can also occur naturally.



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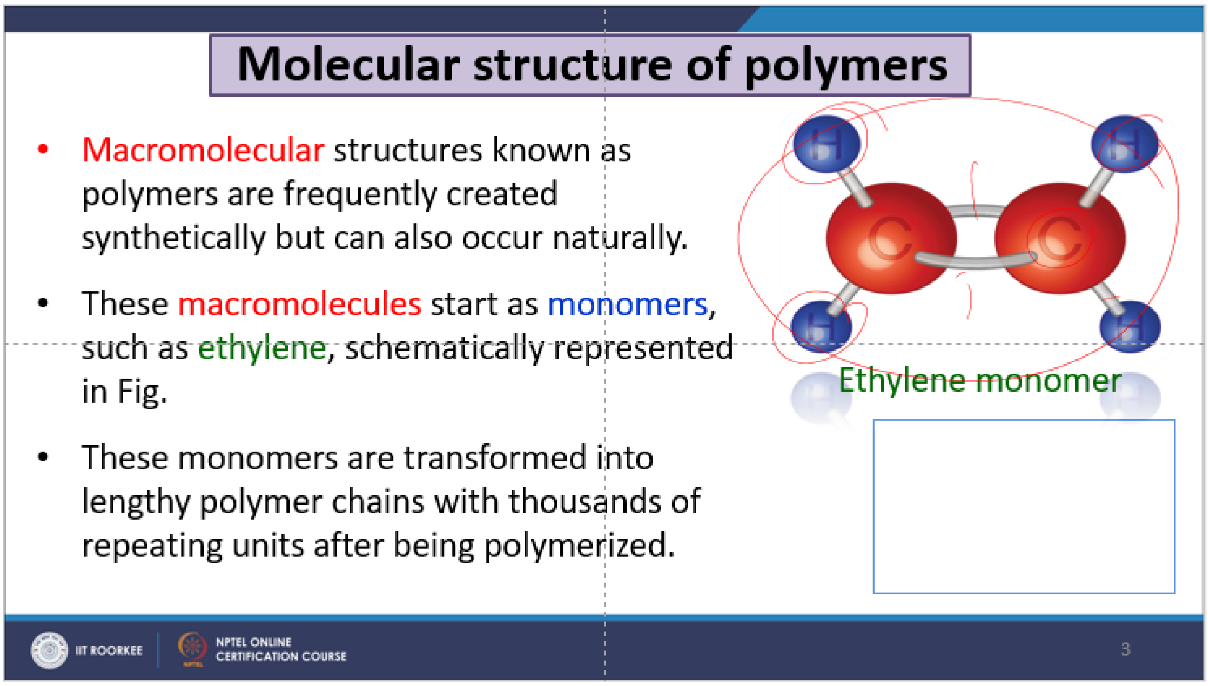
Now, these macromolecules, they start as a monomer like ethylene, which is represented over here like this, these are the carbon bonds and these are the hydrogen. So, this is ethylene. These monomers, they are transformed into blendy polymer chain with thousands of the repeating units after being polymerized. But they must possess some of the quality through which they can polymerize like here, the double bond or some sort of the functional group.

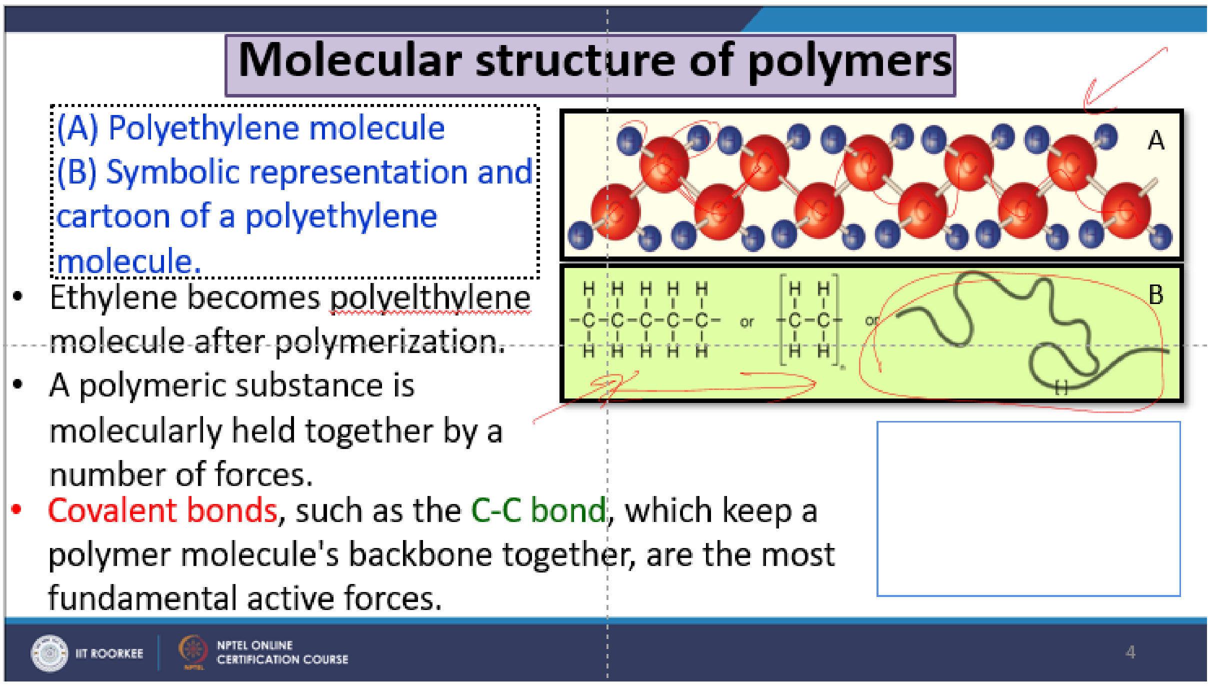


Molecular structure of polymers

- Macromolecular structures known as polymers are frequently created synthetically but can also occur naturally.
- These macromolecules start as monomers, such as ethylene, schematically represented in Fig.
- These monomers are transformed into lengthy polymer chains with thousands of repeating units after being polymerized.

Ethylene monomer

This makes them to polymerizable. Now, if we talk about the polyethylene molecule, now this represents the polyethylene molecule. Here you see the different carbon things and hydrogen, they are attached together to form that long polymer chain. Now, this is this particular figure, this represents a symbolic representation of the carbon of a polymer molecule. So, here this is the skeleton molecule and here this can be represented in the form of a chain.

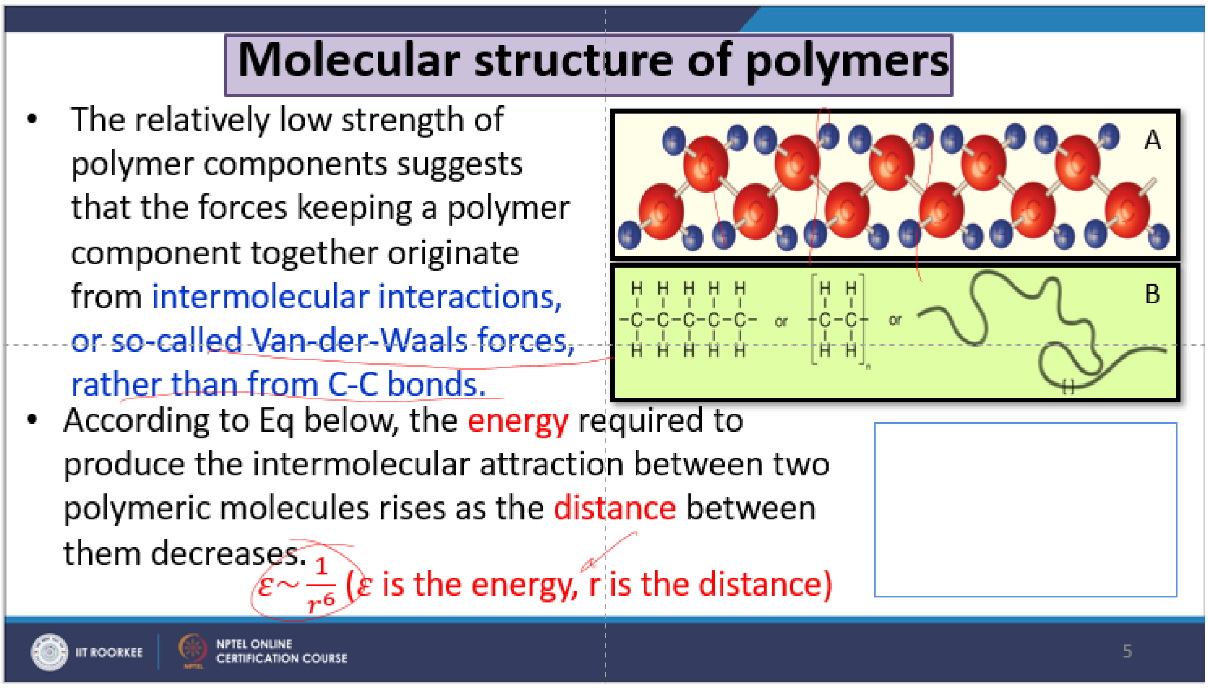


Molecular structure of polymers

(A) Polyethylene molecule
(B) Symbolic representation and cartoon of a polyethylene molecule.

- Ethylene becomes polyelthylene molecule after polymerization.
- A polymeric substance is molecularly held together by a number of forces.
- Covalent bonds, such as the C-C bond, which keep a polymer molecule's backbone together, are the most fundamental active forces.

So, ethylene, this becomes the polyethylene molecule after polymerization. So, a polymeric substance is molecularly held together by a number of forces. One force is called the covalent bond, such as CC bond, which keeps the polymer molecules backbone together like this, this is the backbone. And the most fundamental active force, this is the backbone. Now, the relatively low strength of a polymeric component suggests that the force keeping a polymer component together originates from intermolecular interaction and that is called Van der Waal force rather than from CC bonds.



Molecular structure of polymers

- The relatively low strength of polymer components suggests that the forces keeping a polymer component together originate from intermolecular interactions, or so-called Van-der-Waals forces, rather than from C-C bonds.
- According to Eq below, the energy required to produce the intermolecular attraction between two polymeric molecules rises as the distance between them decreases. $\varepsilon \sim \frac{1}{r^6}$ (ε is the energy, r is the distance)

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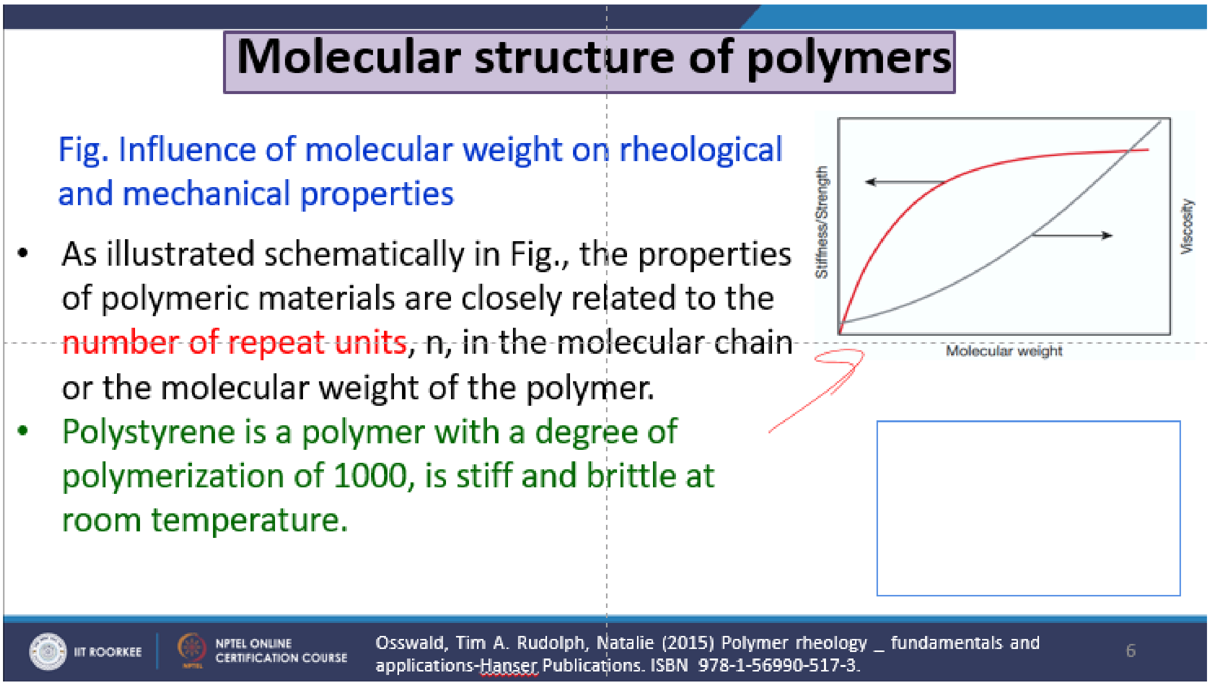
According to this particular equation, where epsilon is related to the 1 over r to the power 6, where epsilon is the energy and r is the distance, the energy required to produce the intermolecular attraction between two polymeric material rises as the distance between them increases, decreases. Now, this figure influence of a molecular weight on rheological and mechanical properties. Now, the properties of the polymeric material, they are closely related to the number of repeat units n, like here you see that these are the number of repeat units. Now, in the molecular chain or the molecular weight of the polymer, this is directly proportion to the number of repeat units or number of chains. So, polystyrene is a polymer with a degree of polymerization of a 1000 is stiff and brittle at room temperature.



Molecular structure of polymers

Fig. Influence of molecular weight on rheological and mechanical properties

- As illustrated schematically in Fig., the properties of polymeric materials are closely related to the number of repeat units, n, in the molecular chain or the molecular weight of the polymer.
- Polystyrene is a polymer with a degree of polymerization of 1000, is stiff and brittle at room temperature.

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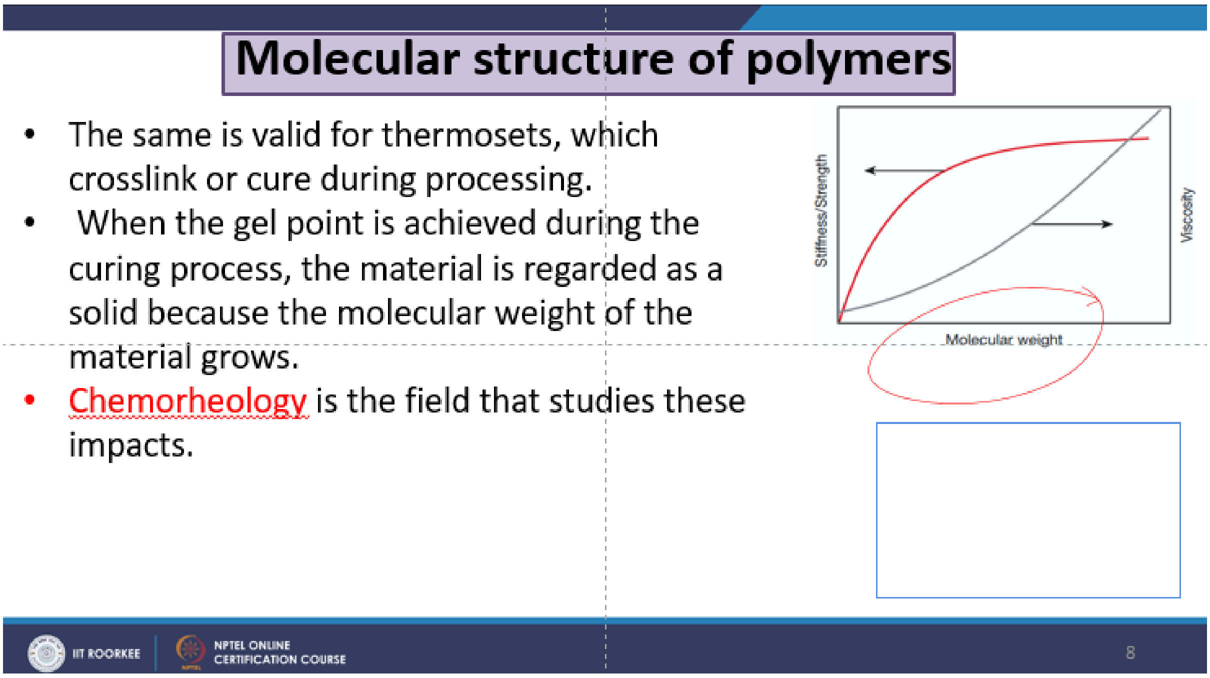
At room temperature, if we talk about the molecular structure of polymer at room temperature, the polystyrene is soft and sticky with a degree of polymerization of 10, while the viscosity grows continuously with the molecular weight, the stiffness qualities attain an asymptotic maximum. During the processing, several thermoplastic including some polymides, one one resin, this increases the molecular weight. This causes the viscosity to increase and could lessen sag as the polymer comes out of the extrusion dye. So, this need to be addressed. So, this particular figure reflects the molecular weight versus stiffness and strength and viscosity simultaneously.

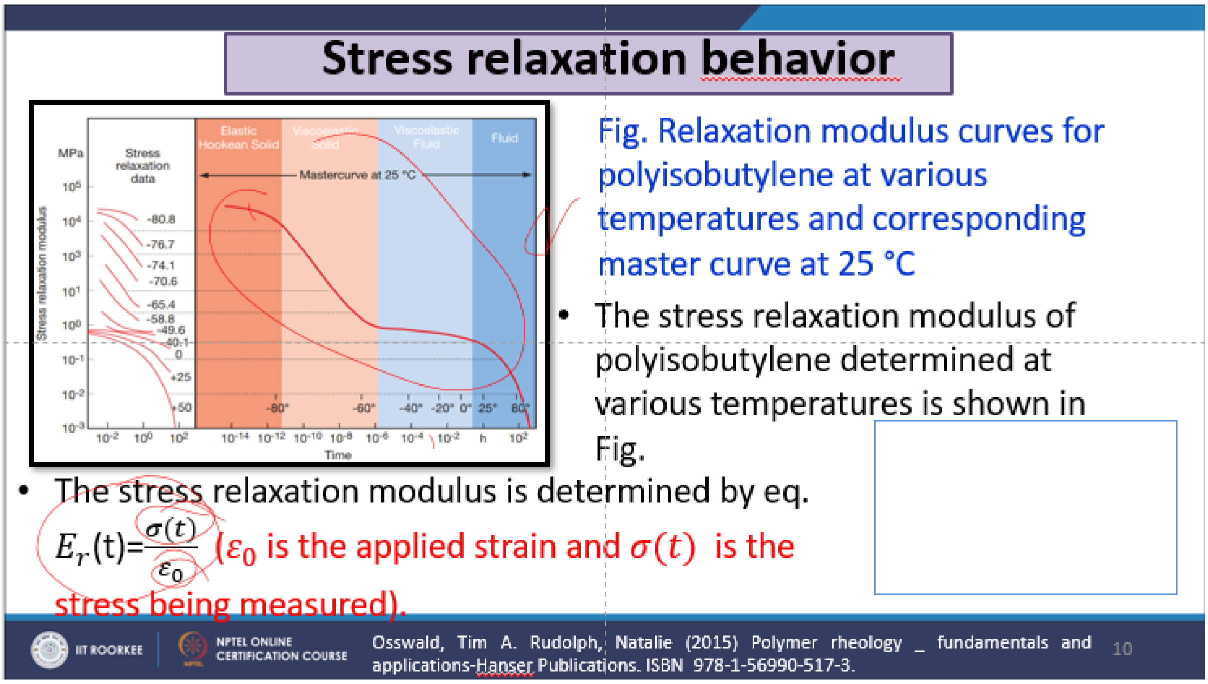


Molecular structure of polymers

- The same is valid for thermosets, which crosslink or cure during processing.
- When the gel point is achieved during the curing process, the material is regarded as a solid because the molecular weight of the material grows.
- Chemorheology is the field that studies these impacts.

Now, this particular thing is valid for thermosets, which cross link or cure during the processing. Now, when the gel point is achieved during the curing process, the material is regarded as the solid because the molecular weight of the material grows. Now, chemorheology is the field that studies these impacts. Let us talk about the stress relaxation behaviour. The stress relaxation is the most fundamental behaviour in polymer melts and solids.

Now, as a mass of polymer is subjected to stress, the molecules tend to move in an effort to relax those stresses. The initial stress brought on by this deformation relaxes in a specific amount of time and frequently, referred to as the relaxation time lambda, if these stresses are brought on by a constant strain. Now, this particular figure represents the relaxation modulus polyisobutylene at various temperatures and corresponding master curve at 25 degree Celsius. The stress relaxation modulus of polyisobutylene is determined the various temperature which is shown in this figure. Now, this stress relaxation modulus is determined by this equation ERP is equal to small sigma t over epsilon naught.



Stress relaxation behavior

Fig. Relaxation modulus curves for polyisobutylene at various temperatures and corresponding master curve at 25 °C

- The stress relaxation modulus of polyisobutylene determined at various temperatures is shown in Fig.
- The stress relaxation modulus is determined by eq.

E_r(t)=$\frac{\sigma(t)}{\varepsilon_0}$ (ε_0 is the applied strain and $\sigma(t)$ is the stress being measured).

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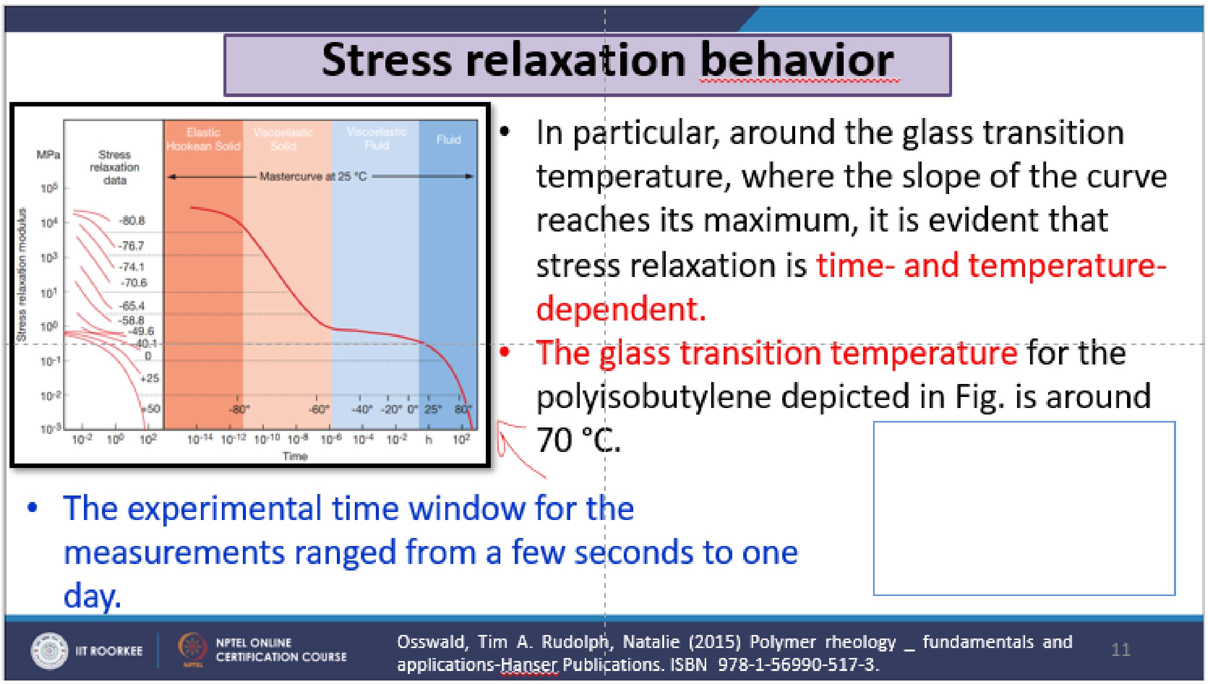
This epsilon naught is the applied stress, and this function is the stress being measured. In particular, around the glass transition temperature where the slope of the curve reaches its maxima, it is evident that the stress relaxation is a time and a temperature dependent and the glass transition temperature for the polyisobutylene depicted in this particular figure is around 70 degree Celsius. So, the experimental time window, this is the measurement ranged from the few second to one day. Now, the tests conducted at a lower temperature, these were utilised to capture the beginning of this relaxation of the rapidly decaying stresses while the tests conducted at higher temperature only recorded the conclusion of the relaxation. Now, this is the schematic stress relaxation on the molecule level at low top and high bottom temperature.



Stress relaxation behavior

- In particular, around the glass transition temperature, where the slope of the curve reaches its maximum, it is evident that stress relaxation is time- and temperature-dependent.
- The glass transition temperature for the polyisobutylene depicted in Fig. is around 70 °C.
- The experimental time window for the measurements ranged from a few seconds to one day.

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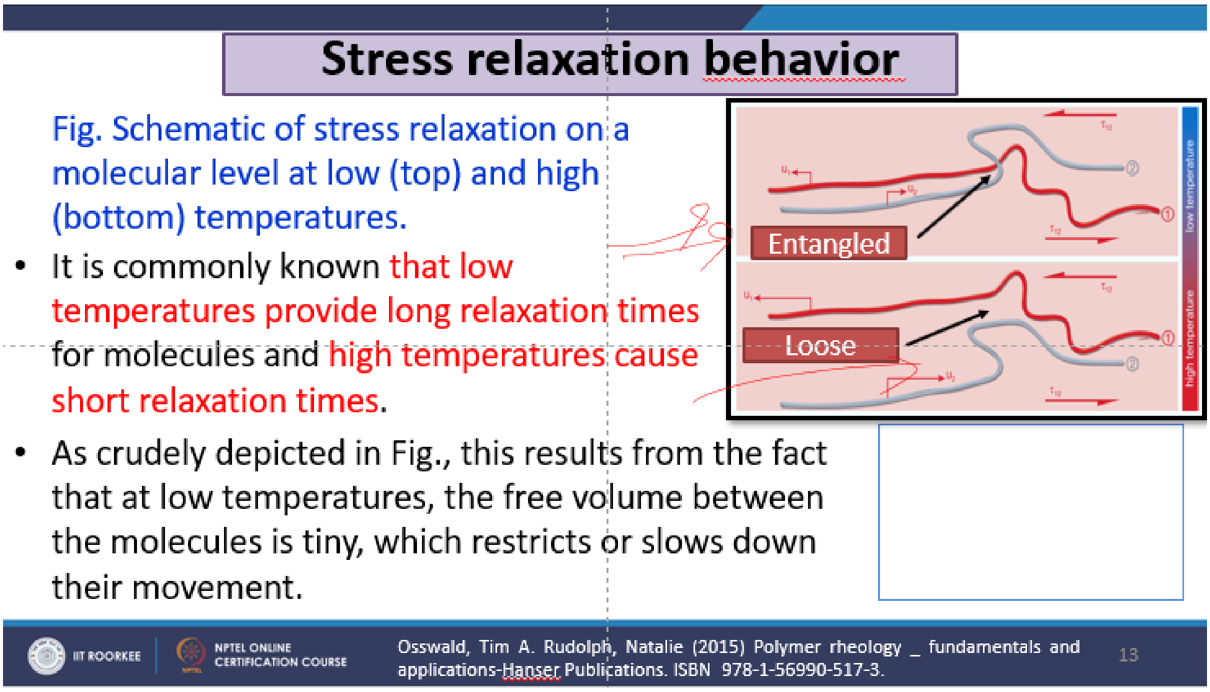
It is commonly known as the low temperature provide long relaxation time for molecules and high temperature causes the short relaxation time. As this crudely depicted in the form of entangled or a loose chain, this results from the fact that at a low temperature, the free volume between the molecule is tiny. So that all these chains are entangled all together and if we raise the term and which restricts and slow down their movement, but if we raise the temperature, then the free volume between the molecule is become larger and larger and they may become like loose like this. So, the form of the finding from the creep or a relaxation like here you see does not change as the temperature changes, but they are horizontally displaced to the left or right corresponding to the slower or faster response time. Now, here you see the molecular structure with increasing shear.



Stress relaxation behavior

Fig. Schematic of stress relaxation on a molecular level at low (top) and high (bottom) temperatures.

- It is commonly known that low temperatures provide long relaxation times for molecules and high temperatures cause short relaxation times.
- As crudely depicted in Fig., this results from the fact that at low temperatures, the free volume between the molecules is tiny, which restricts or slows down their movement.

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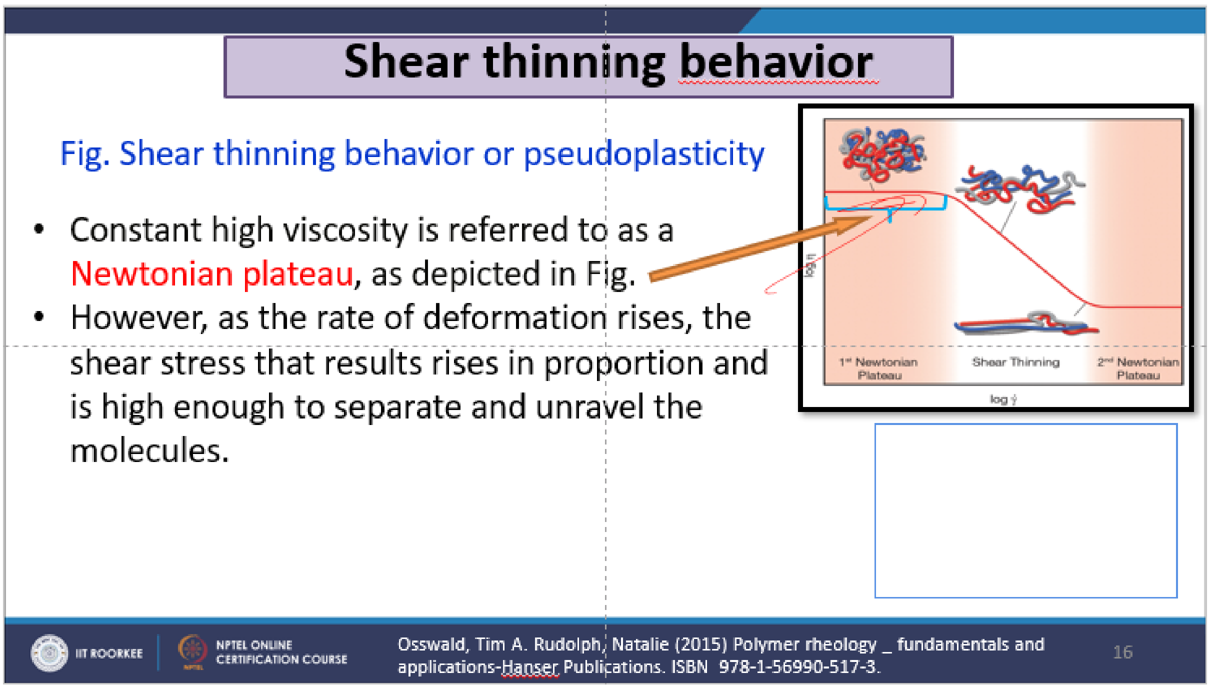
Now, this is the entangled molecules, they have the difficulty in slipping past one another at the modest rate of deformation leading to a relatively high viscosity. So, they are very much compact in nature. Now, this behaviour can be observed at a variety of small deformation rate of for the polymer melts. So, here you see they are trying to unentangle, and they here they are adopting the flow behaviour. Now, constant high viscosity is referred to as the Newtonian plateau, which is referred as this one.



Shear thinning behavior

Fig. Shear thinning behavior or pseudoplasticity

- Constant high viscosity is referred to as a Newtonian plateau, as depicted in Fig.
- However, as the rate of deformation rises, the shear stress that results rises in proportion and is high enough to separate and unravel the molecules.

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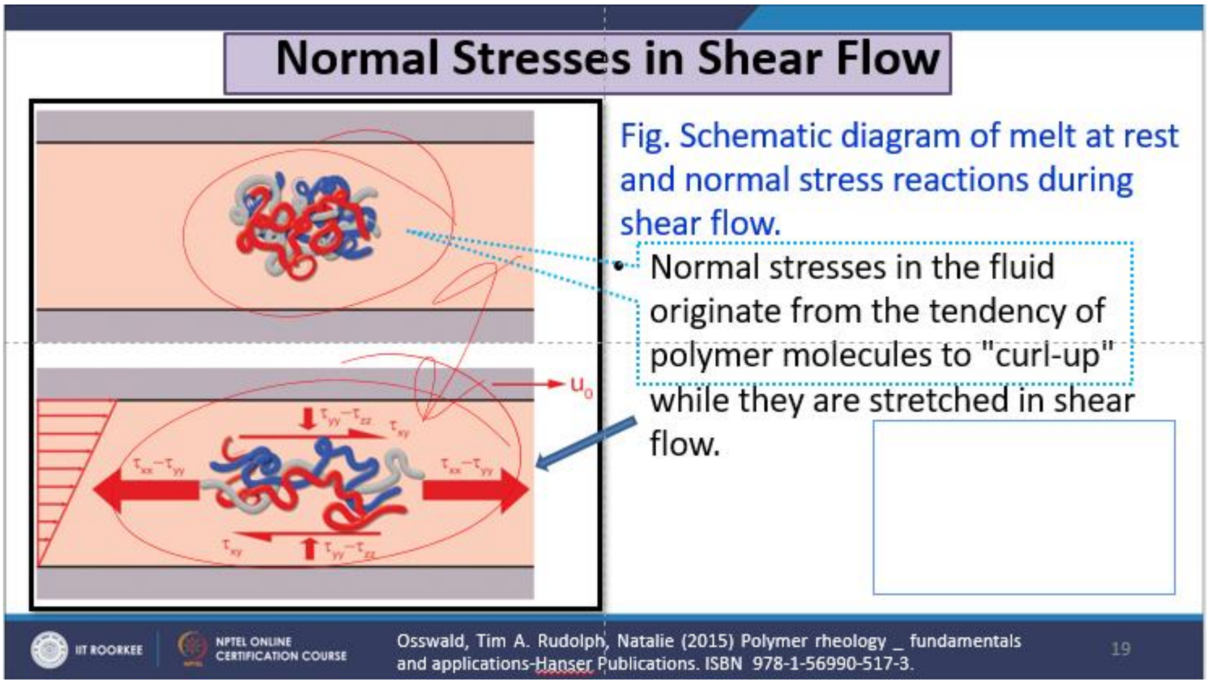
However, as the rate of deformation rises, the shear stress that results rise in the step of proportion and is high enough to separate and unravel the molecules, which is represented as like this. Now, because of disentangled molecule can pass their neighbours more easily and viscosity of the melt is generally reduced, the shear thinning behaviour, the pseudo plasticity or structural viscosity are term used to describe this particular phenomenon. The molecules eventually reach their limit of extension. Here you can see. Now, this particular point, which represents the lowest attainable viscosity, can be seen in the second Newtonian plateau at high deformation, which is represented over here.



Normal Stresses in Shear Flow

Fig. Schematic diagram of melt at rest and normal stress reactions during shear flow.

- Normal stresses in the fluid originate from the tendency of polymer molecules to "curl-up" while they are stretched in shear flow.

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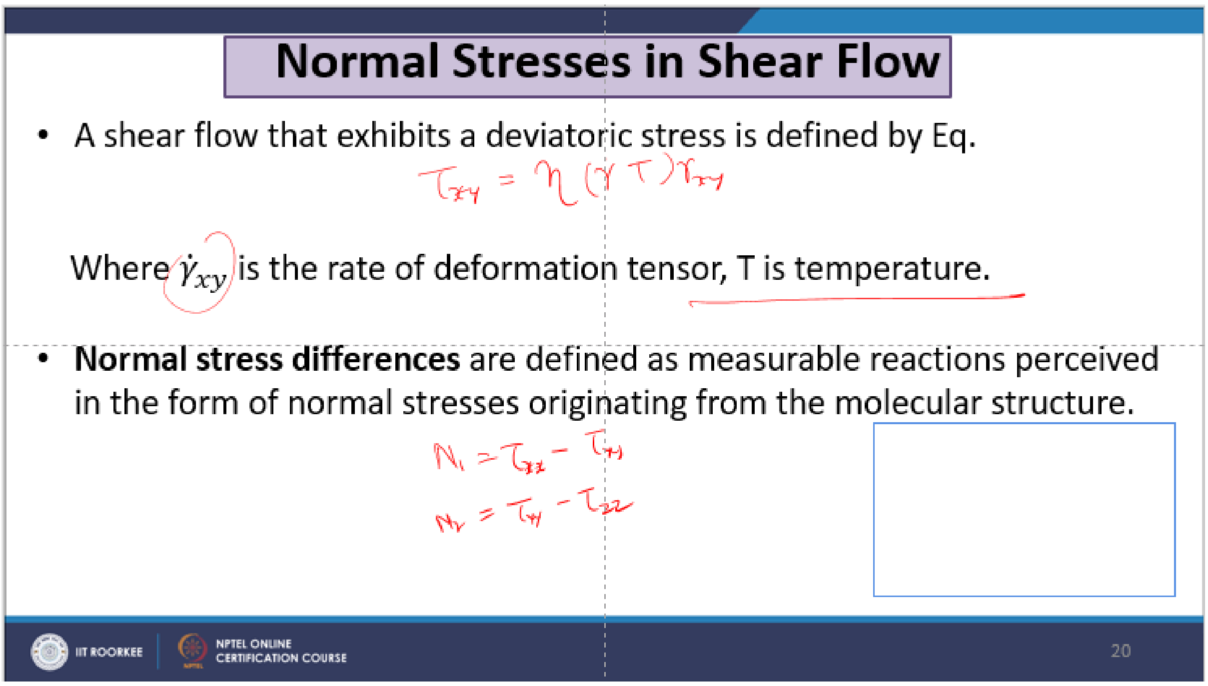
Now, as the temperature rises, the free volume between the molecule expands, reducing the intermolecular friction and resulting in a reduction in viscosity as per this particular figure. Now, this is the schematic diagram for met at rest and a normal stress reaction during the shear flow. This is the normal stresses in the fluid originate from the tendency of the polymer molecules to curl up while they are stretched in shear flow. Now, as the shear flow that exhibits a deviatoric stress, which is defined by the equation tau x y is equal to nita gamma for the flow behaviour t into gamma x y, where the gamma x y is the rate of deformation tensor and t is the temperature. Now, normal stress differences are defined as measurable relations, reactions perceived in the form of a normal stresses originating from the molecular structure.



Normal Stresses in Shear Flow

- A shear flow that exhibits a deviatoric stress is defined by Eq.

$$\tau_{xy} = \eta(\gamma\, T)\,\gamma_{xy}$$

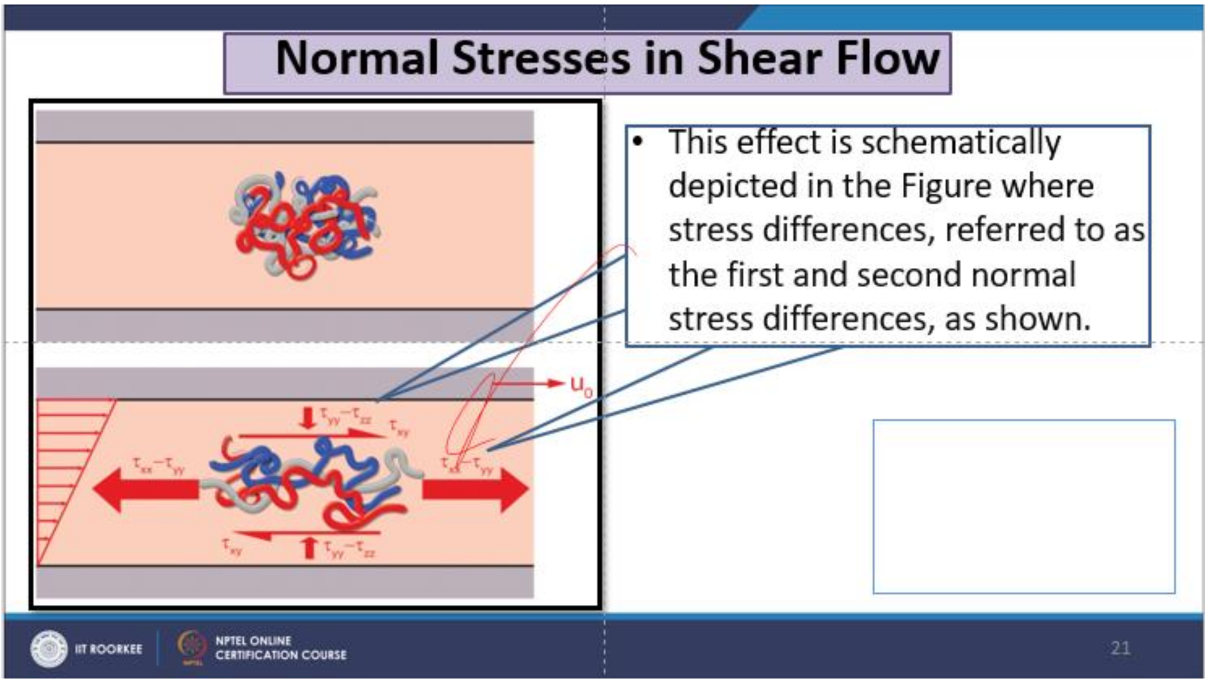
Where $\dot{\gamma}_{xy}$ is the rate of deformation tensor, T is temperature.

- **Normal stress differences** are defined as measurable reactions perceived in the form of normal stresses originating from the molecular structure.

$$N_1 = \tau_{xx} - \tau_{yy}$$

$$N_2 = \tau_{yy} - \tau_{zz}$$

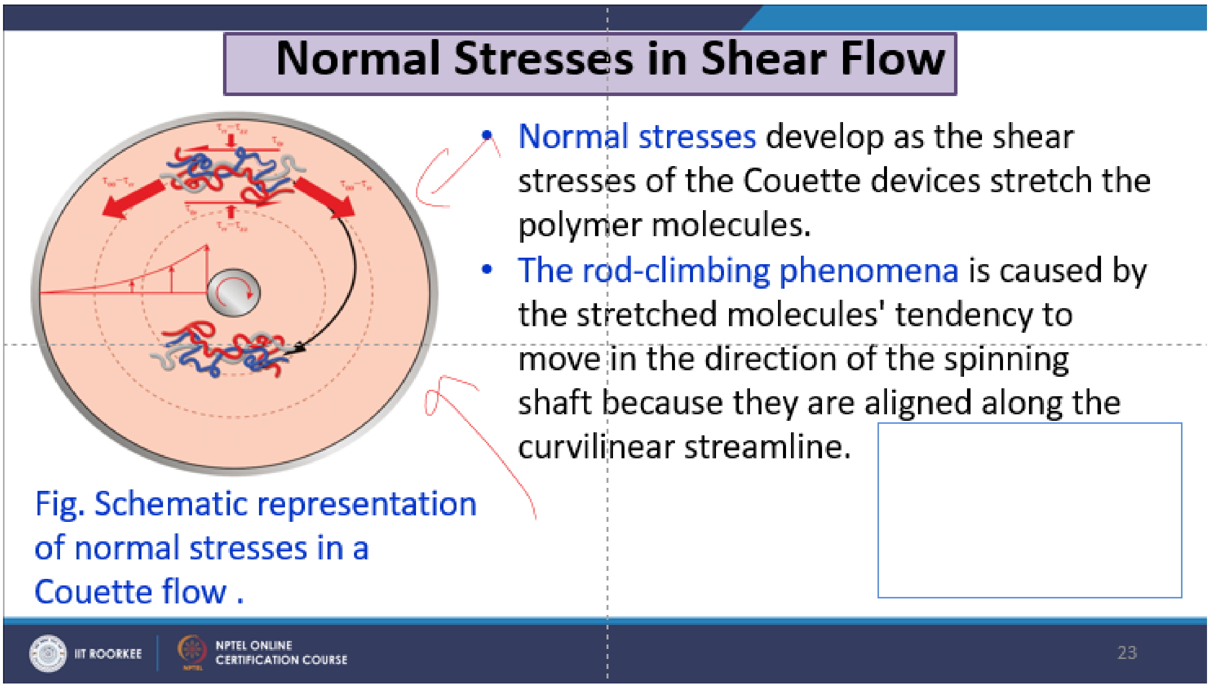
So, n 1 is equal to tau xx minus tau yy and n 2 is equal to tau yy minus tau zz. Now, this effect is schematically depicted in this figure where the stress differences referred to as the first and second normal stress differences as per this particular figure. Now, this is an experimental setup representing the normal stress differences effect is the so-called rod climbing experiment and also known as Wisenberg effect. The polymeric solution this moves towards the rotating shaft in this experiment instead of away from it as with the Newtonian fluid, the polymer consequently begins to ascend the revolving shaft. Now, this is the schematic representation of a normal stresses in the cuvette flow.



Normal Stresses in Shear Flow

- This effect is schematically depicted in the Figure where stress differences, referred to as the first and second normal stress differences, as shown.

The normal stresses develop as the shear stress of the cuvette devices stretched to the polymeric molecule. The rod climbing phenomena is caused by the stretched molecules tendency to move in the direction of the spinning shaft because they are aligned along the curvilinear streamline. The normal stresses just like viscosity, they are also the material dependent and can be defined as n 1 is equal to tau xx minus tau yy that is equal to psi gamma t gamma xy square and n 2 is equal to tau yy minus tau zz is equal to psi 2 gamma t gamma xy to the power 2. The primary and secondary normal stress coefficient denoted as psi 1 and psi 2 respectively are the material function. Now, these properties are dependent on both the magnitude of the strain rate tensor and the temperature.

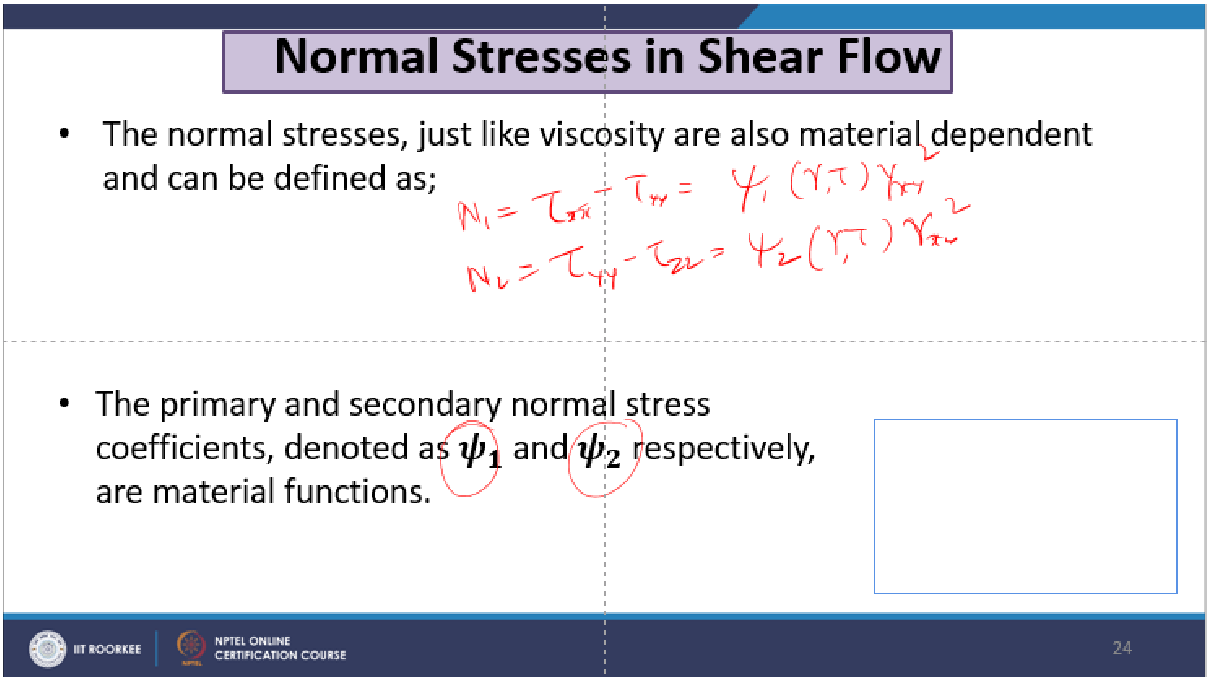


Normal Stresses in Shear Flow

- **Normal stresses** develop as the shear stresses of the Couette devices stretch the polymer molecules.
- **The rod-climbing phenomena** is caused by the stretched molecules' tendency to move in the direction of the spinning shaft because they are aligned along the curvilinear streamline.

Fig. Schematic representation of normal stresses in a Couette flow .

Similar to viscosity, the first and second normal stress differences remain unchanged in sign even when the direction of the strain rate is altered. The second normal stress differences, they are more difficult to measure and therefore, the data they are often approximated by psi 2 gamma is equal almost equal to minus 0.1 psi 1 gamma. Now, let us talk about the melt strength or melt fracture. The melt fracture, they are also known as the plastic deformation occurs when a polymer is forced to deform and move at rates that cause processing time that are less than the polymers relaxation time.



Normal Stresses in Shear Flow

- The normal stresses, just like viscosity are also material dependent and can be defined as;

$$N_1 = \tau_{xx} - \tau_{yy} = \psi_1(\gamma, T)\gamma_{xy}^2$$

$$N_2 = \tau_{yy} - \tau_{zz} = \psi_2(\gamma, T)\gamma_{xy}^2$$

- The primary and secondary normal stress coefficients, denoted as ψ_1 and ψ_2 respectively, are material functions.

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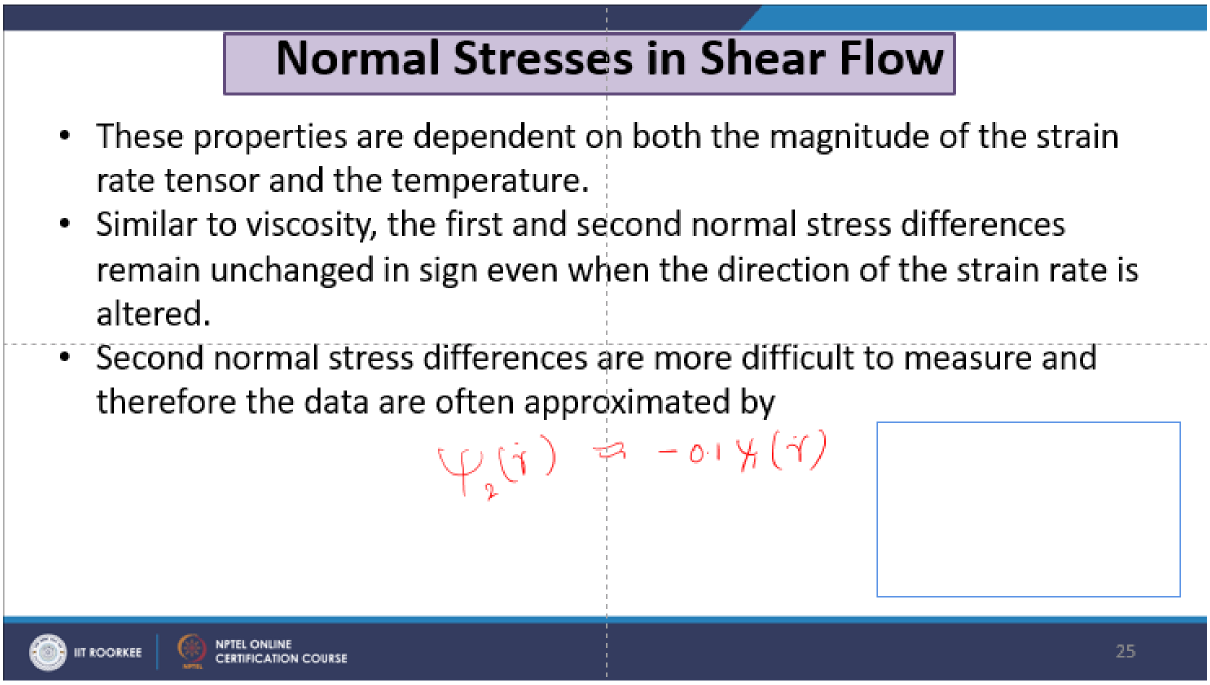

Normal Stresses in Shear Flow

- These properties are dependent on both the magnitude of the strain rate tensor and the temperature.
- Similar to viscosity, the first and second normal stress differences remain unchanged in sign even when the direction of the strain rate is altered.
- Second normal stress differences are more difficult to measure and therefore the data are often approximated by

$$\psi_2(\dot{\gamma}) \approx -0.1\,\psi_1(\dot{\gamma})$$

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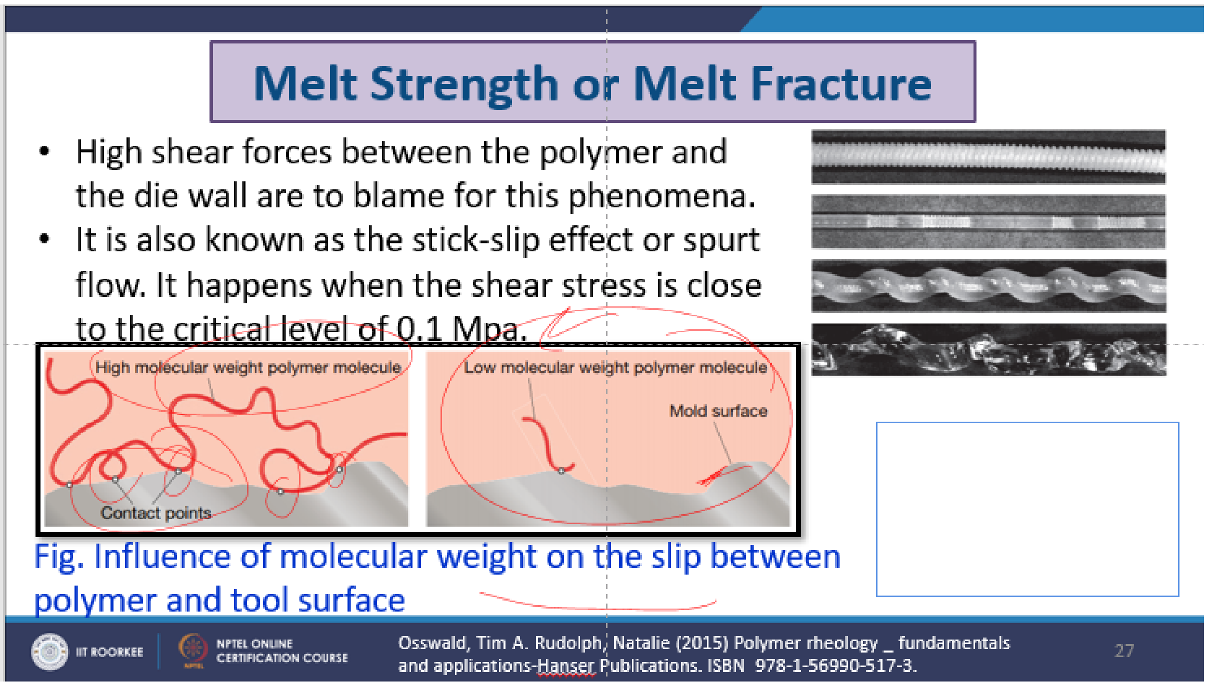
Now, this particular phenomenon might appear in several ways depending on the method. For example, high extrusion speeds that prevents the polymer from relaxing inside the extrusion dye may cause waves to emerge in the extruded. Here you see the different type of the things. The high shear forces between the polymer and the dye wall are to blame for this particular phenomenon. It is also known as the stick slip effect or spurt flow.

It happens when the shear stress is close to the critical level of 0.1 mega Pascal. Here you see the influence of the molecular weight on the slip between the polymer and tool surface. This is the low molecular weight polymer molecules and this is the mold surface you can see the effect and similarly, you see that these are the context points. So, this is the high molecular weight polymer molecule and the contact points are more compared to the low molecular weight polymer molecules.



Melt Strength or Melt Fracture

- High shear forces between the polymer and the die wall are to blame for this phenomena.
- It is also known as the stick-slip effect or spurt flow. It happens when the shear stress is close to the critical level of 0.1 Mpa.

Fig. Influence of molecular weight on the slip between polymer and tool surface

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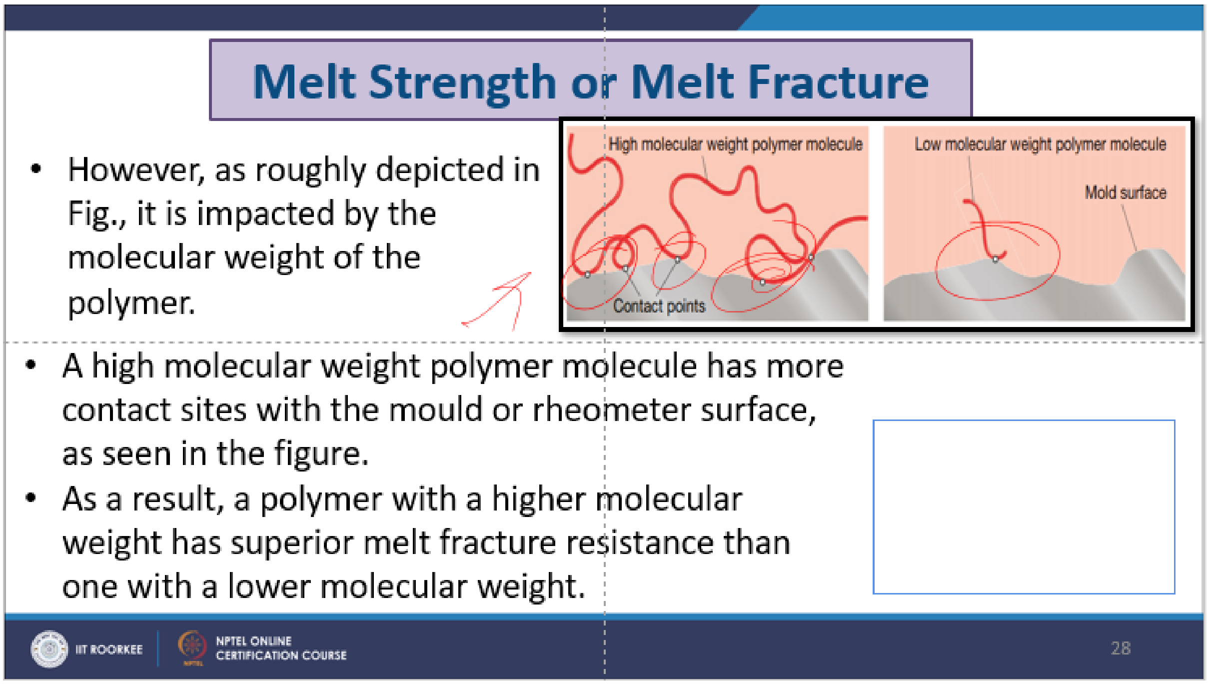
However, if we talk about as a roughly depicted as per this particular figure impacted by the molecular weight of the polymer. A high molecular weight polymer molecule has more contact sites in the surface. As a result, a polymer with the high molecular weight has superior melt fracture resistance than one with a lower molecular weight because the number of contact points they play a very vital role. According to the source, the weight average molecular weight has an inverse relationship with the critical shear stress of 0.



Melt Strength or Melt Fracture

- However, as roughly depicted in Fig., it is impacted by the molecular weight of the polymer.
- A high molecular weight polymer molecule has more contact sites with the mould or rheometer surface, as seen in the figure.
- As a result, a polymer with a higher molecular weight has superior melt fracture resistance than one with a lower molecular weight.

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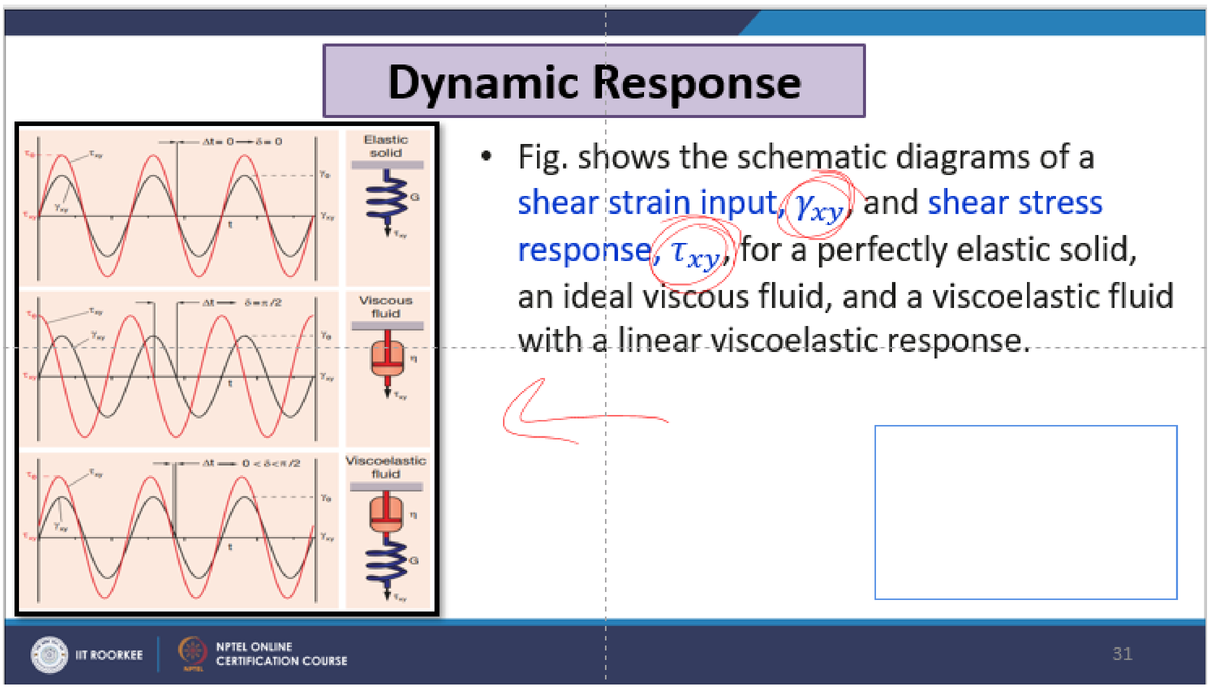
1 mega Pascal. A different type of melt fracture may happen during the elongational deformation such as during the spinning of fibre. The excessive drawing speed causes tensions to build up before they have a chance to dissipate resulting in a threat tear or fracture. Let us talk about the dynamic response. Oscillatory test, a sample is aroused by a low frequency stress or strain input and both the stress or strain responses and input are recorded. In this particular figure, you see that the oscillatory shear experience experiments of a for an elastic solid, a viscous fluid and a viscoelastic fluid.

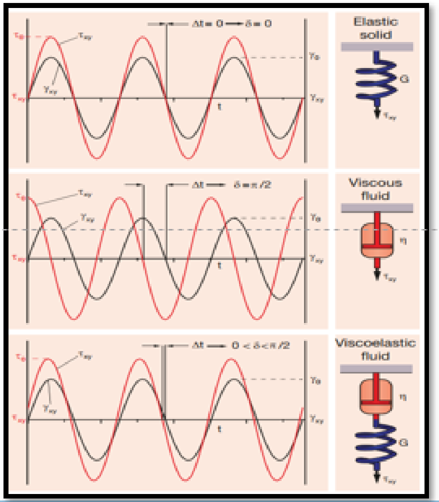
This figure shows the schematic diagram of shear strain input gamma xy and shear stress response tau xy for a perfectly elastic. This particular figure shows the schematic diagram of a shear strain input by gamma xy and shear stress response tau xy for a perfectly elastic solid and ideal viscous fluid and a viscoelastic fluid with a linear viscoelastic response. The strain input and stress response in oscillatory test, this can be explained if the sample is a Hohian solid or a perfectly elastic and that is gamma xy t is equal to gamma naught sine omega t and tau xy is equal to tau naught sine omega t, where t is the time and omega pi t, omega is equal to 2 pi f with f being the frequency of strain oscillation. The stress response and the strain input in phase as would be predicted as a result when the computing the shear modulus, the transient effects are cancelled out leaving the resultant elastic modulus G and which can be represented as G is equal to tau naught sine omega t over gamma naught sine omega t. The stress response on the other hand is proportional to the strain rate gamma naught omega cos omega t which may be alternatively be understood as a lag of pi by 2 radii after the strain input when an ideal viscous fluid is subjected to an oscillatory strain.



Dynamic Response

- Fig. shows the schematic diagrams of a shear strain input, γ_{xy}, and shear stress response, τ_{xy}, for a perfectly elastic solid, an ideal viscous fluid, and a viscoelastic fluid with a linear viscoelastic response.

Dynamic Response



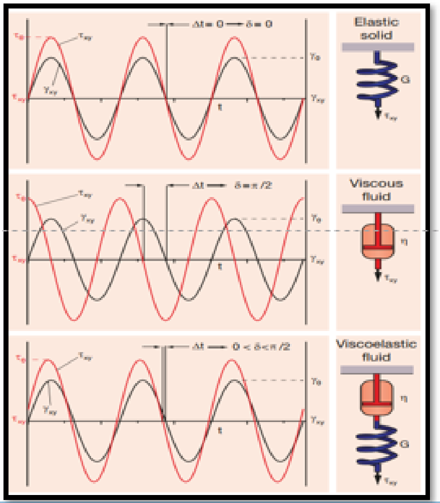
- The strain input and stress response in an oscillatory test can be explained as follows if the sample is a Hookean solid or perfectly elastic.

$$\gamma_{xy}(t) = \gamma_0 \sin\omega t$$

$$\tau_{xy} = \tau_0 \sin\omega t$$

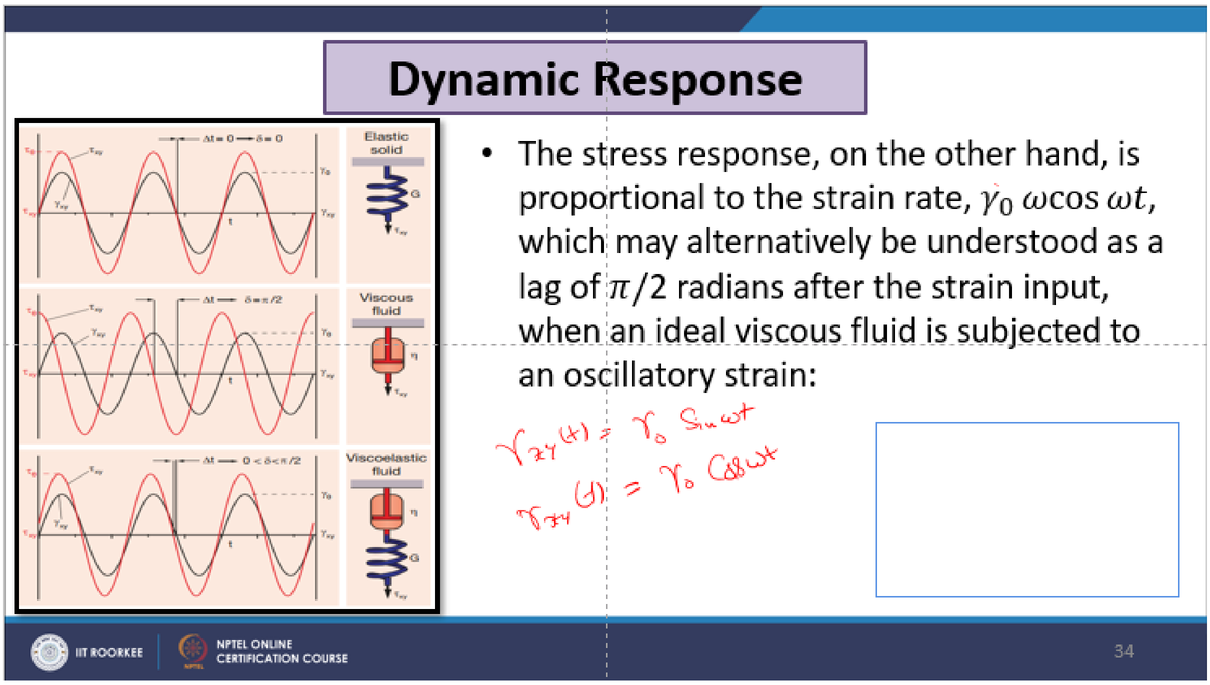
$$\omega = 2\pi f$$

Dynamic Response



- The stress response and the strain input are in phase, as would be predicted.
- As a result, when computing the shear modulus, the transient effects are cancelled out, leaving a resultant elastic modulus, *G*.

$$G = \frac{\tau_0 \sin\omega t}{\gamma_0 \sin\omega t}$$



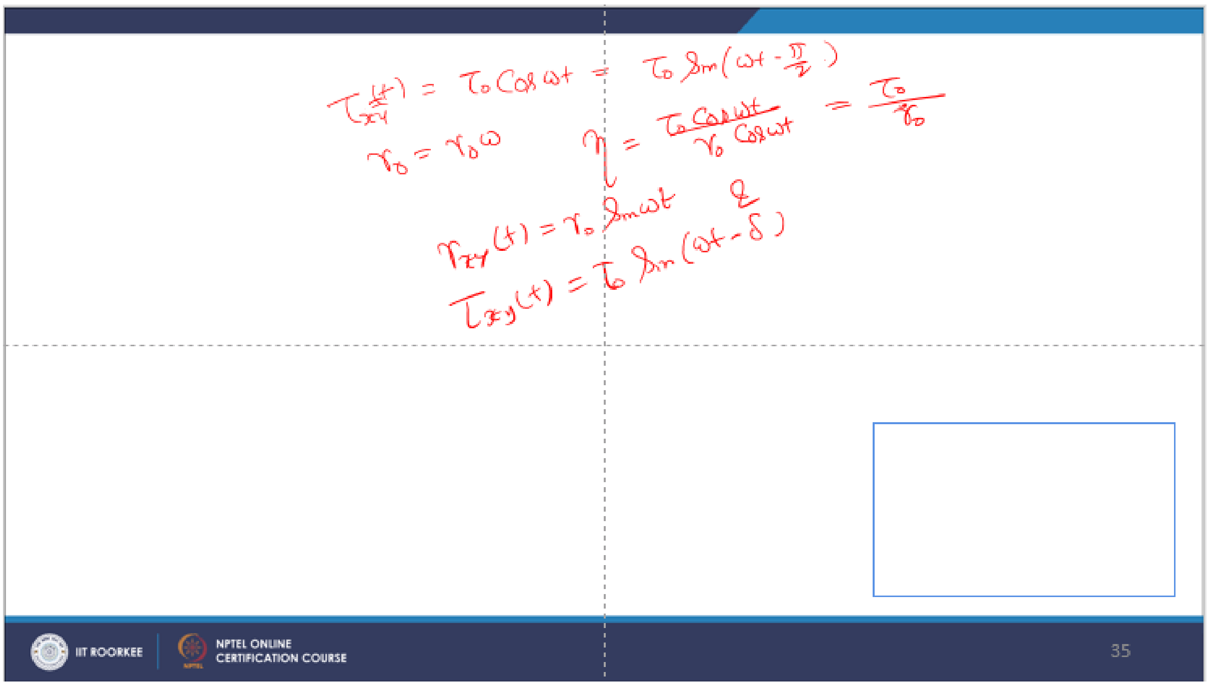
Dynamic Response

- The stress response, on the other hand, is proportional to the strain rate, $\dot{\gamma}_0\, \omega \cos \omega t$, which may alternatively be understood as a lag of $\pi/2$ radians after the strain input, when an ideal viscous fluid is subjected to an oscillatory strain:

$\gamma_{xy}(t) = \gamma_0 \sin \omega t$

$\gamma_{xy}(t) = \gamma_0 \cos \omega t$

So, this is gamma xy t gamma naught sine omega t and gamma xy is equal to gamma naught cos omega t. So, here tau xy t is equal to tau naught cos omega t is equal to tau naught sine omega t minus pi by 2. Now, where omega gamma naught is equal to gamma naught omega using this particular equation, we can compute the viscosity using the definition of a Newtonian fluid which says that eta is equal to tau cos omega t over gamma naught cos omega t is equal to tau naught gamma naught dot. So, we see in here that to the transient effect, so cancel each other out and resulting the constant viscosity. However, the viscous viscoelastic material such as polymers they behave somewhere in between the perfectly elastic and the perfectly viscous material depending on the time scale.



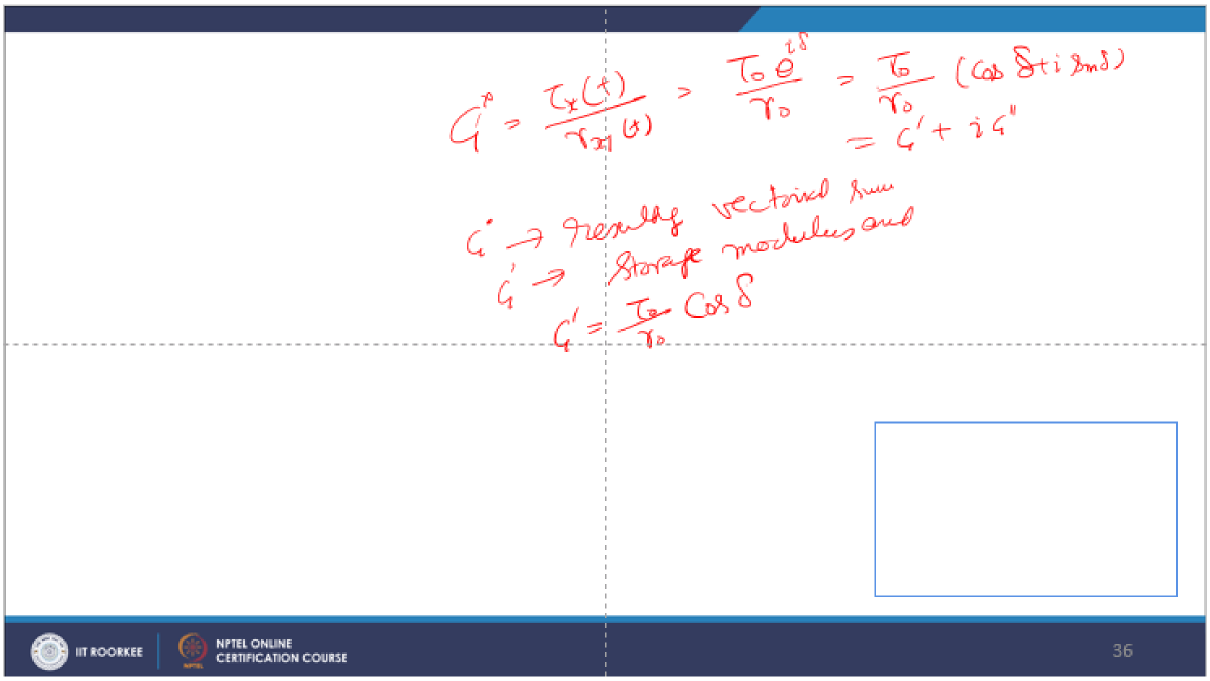
$\tau_{xy}(t) = \tau_0 \cos \omega t = \tau_0 \sin(\omega t - \frac{\pi}{2})$

$\gamma_0 = \gamma_0 \omega$

$\eta = \frac{\tau_0 \cos \omega t}{\gamma_0 \cos \omega t} = \frac{\tau_0}{\gamma_0}$

$\gamma_{xy}(t) = \gamma_0 \sin \omega t$ &

$\tau_{xy}(t) = \tau_0 \sin(\omega t - \delta)$



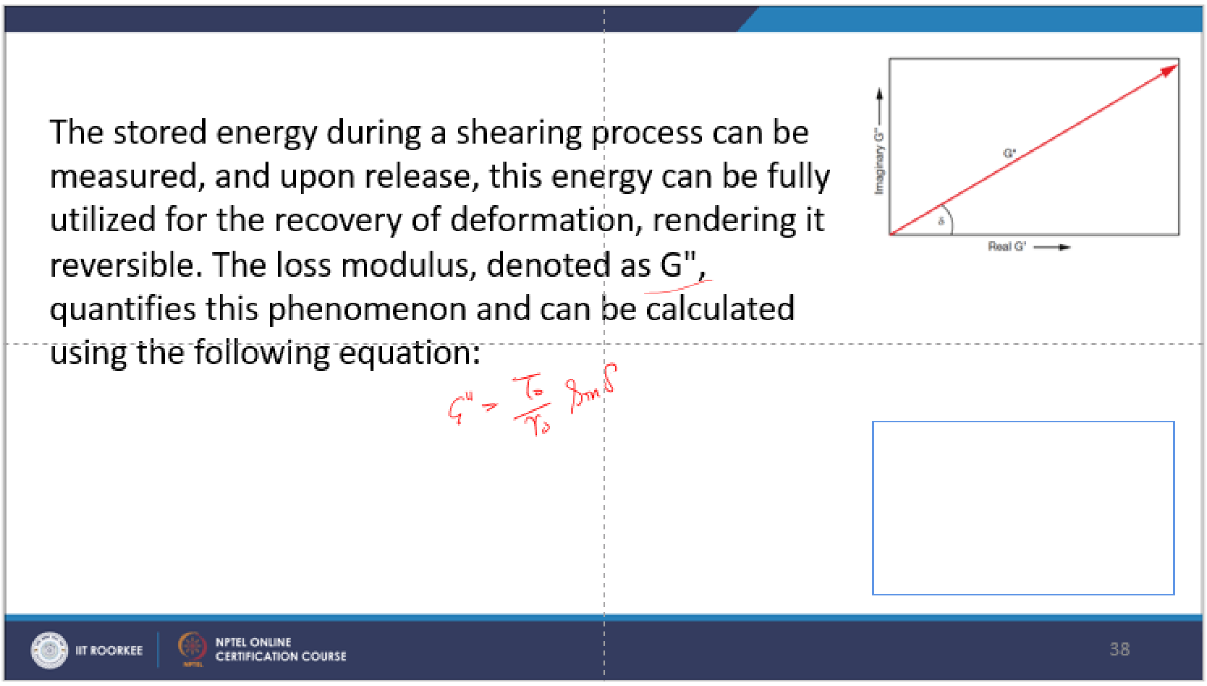
$$G^* = \frac{\tau_x(t)}{\gamma_{xy}(t)} = \frac{\tau_0 e^{i\delta}}{\gamma_0} = \frac{\tau_0}{\gamma_0}(\cos\delta + i\sin\delta)$$
$$= G' + iG''$$

$G^* \rightarrow$ resulting vectorial sum

$G' \rightarrow$ Storage modulus and

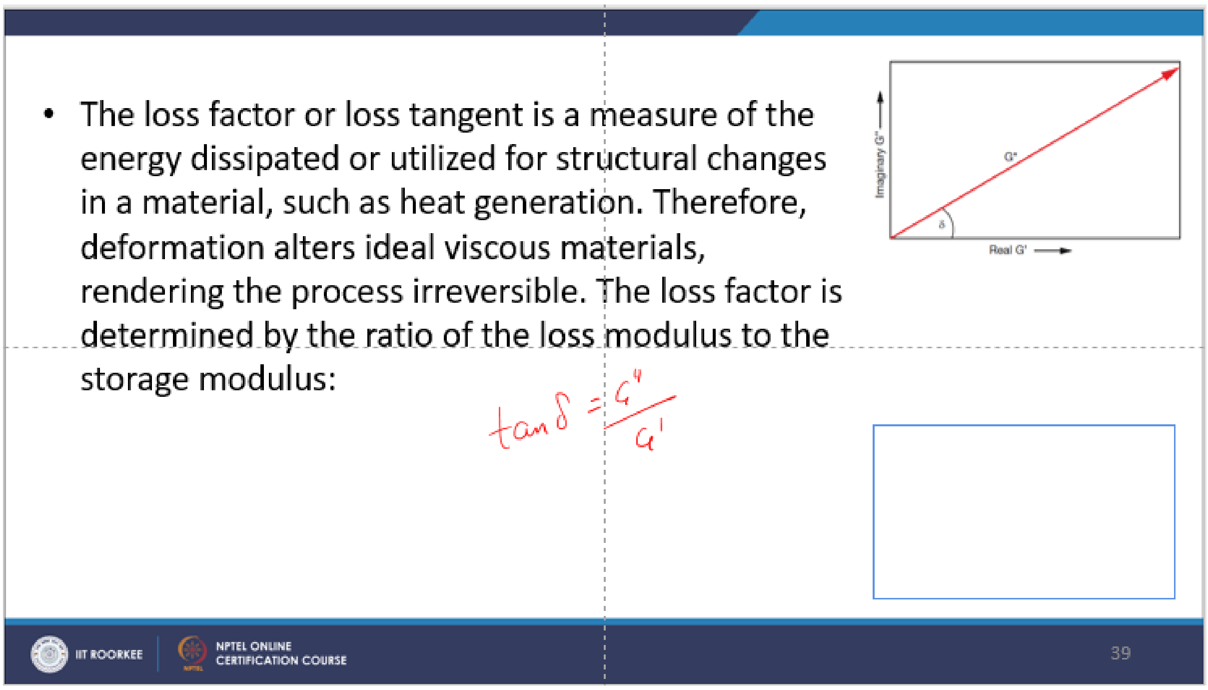
$$G' = \frac{\tau_0}{\gamma_0}\cos\delta$$

Now, their representation can be described by gamma xy t is equal to gamma naught sine omega t and tau xy t is equal to tau naught sine omega t minus delta. Now, this showing the stress response of lag of delta radius behind the strain input. Now, the shear modulus computed using that particular ratio of stress to strain, this take a complex form with real and imaginary term and can be written as G star is equal to tau x t over gamma xy t, this is equal to tau naught e to the power i delta over gamma naught and tau naught over gamma naught cos delta plus i sine delta and this is equal to G prime plus i G double prime. Now, this is shown in the figure and referred so to as a complex modulus and the length of each vector represents the absolute value of the parameter. Therefore, G prime is the resulting vectorial sum and G prime is referred usually referred as a storage modulus and represented by G prime is equal to tau naught over gamma naught cos delta.



The stored energy during a shearing process can be measured, and upon release, this energy can be fully utilized for the recovery of deformation, rendering it reversible. The loss modulus, denoted as G", quantifies this phenomenon and can be calculated using the following equation:

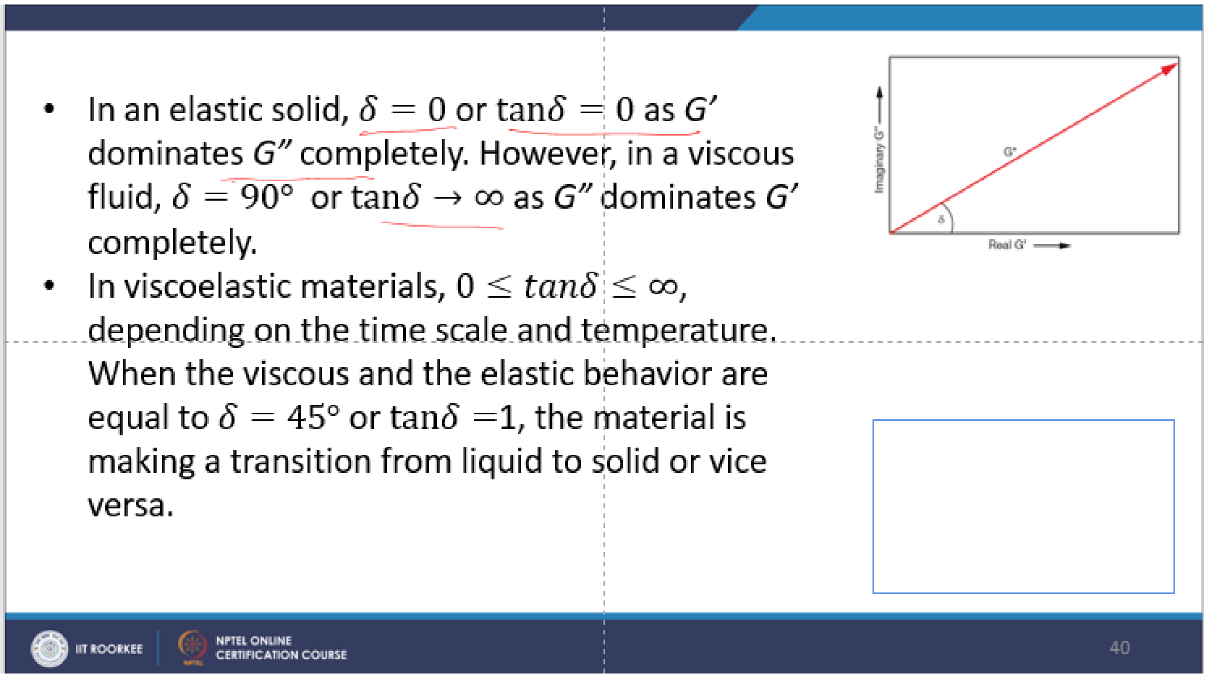
$$G'' = \frac{\tau_0}{\gamma_0}\sin\delta$$



- The loss factor or loss tangent is a measure of the energy dissipated or utilized for structural changes in a material, such as heat generation. Therefore, deformation alters ideal viscous materials, rendering the process irreversible. The loss factor is determined by the ratio of the loss modulus to the storage modulus:

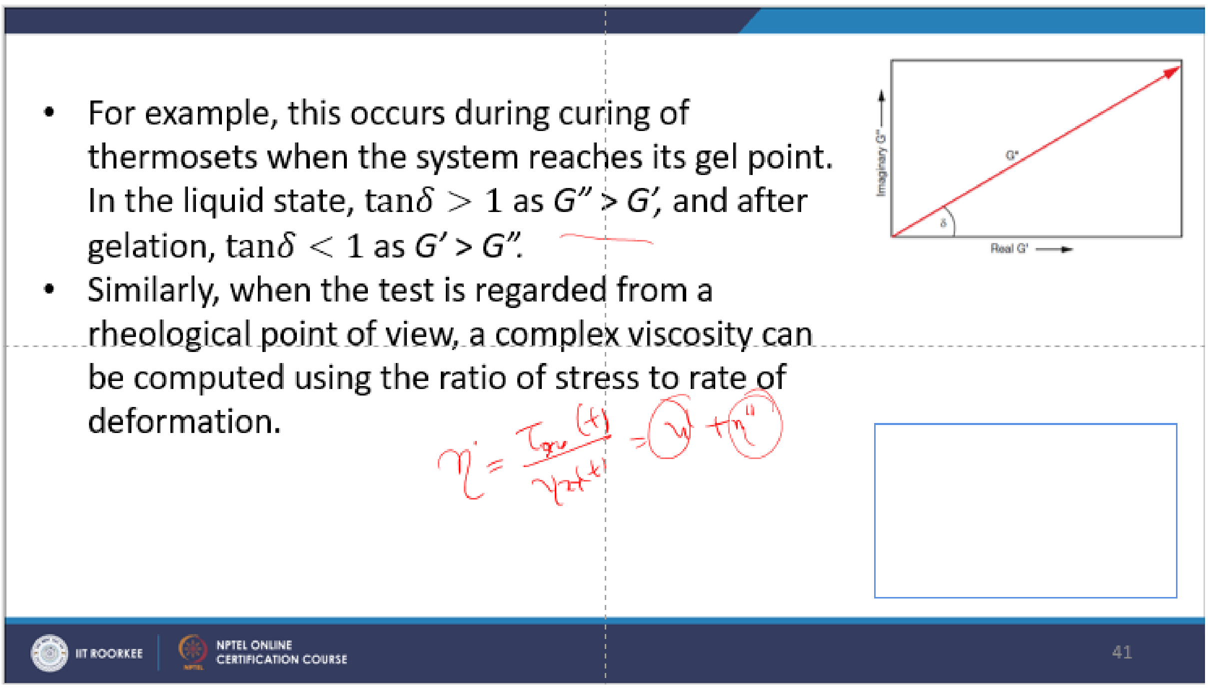
$\tan\delta = \frac{G''}{G'}$

Now, it is the measure of during a shearing process can be measured upon release and this energy can be fully utilised for the recovery of deformation rendering it reversible and the loss modulus this is denoted by G double prime quantifies this particular phenomenon and can be calculated using the different equations. Now, G double prime is equal to tau naught gamma naught sine delta. Now, the loss factor, the loss factor or the loss tangent is measure of the energy dissipated or utilised for structural changes in the in a material such as heat generation and therefore, the deformation alters ideal viscous material rendering the process irreversible. The loss factor is determined by the ratio of the loss modulus to the storage modulus and referred as tan delta is equal to G double prime over G prime. Now, in an elastic solid delta is equal to 0 and tan delta is equal to 0 and G prime dominates G double prime completely however, in a viscous fluid delta is equal to 90 degree and tan delta is equal to infinity or tends to infinity and G double prime dominates G prime completely.



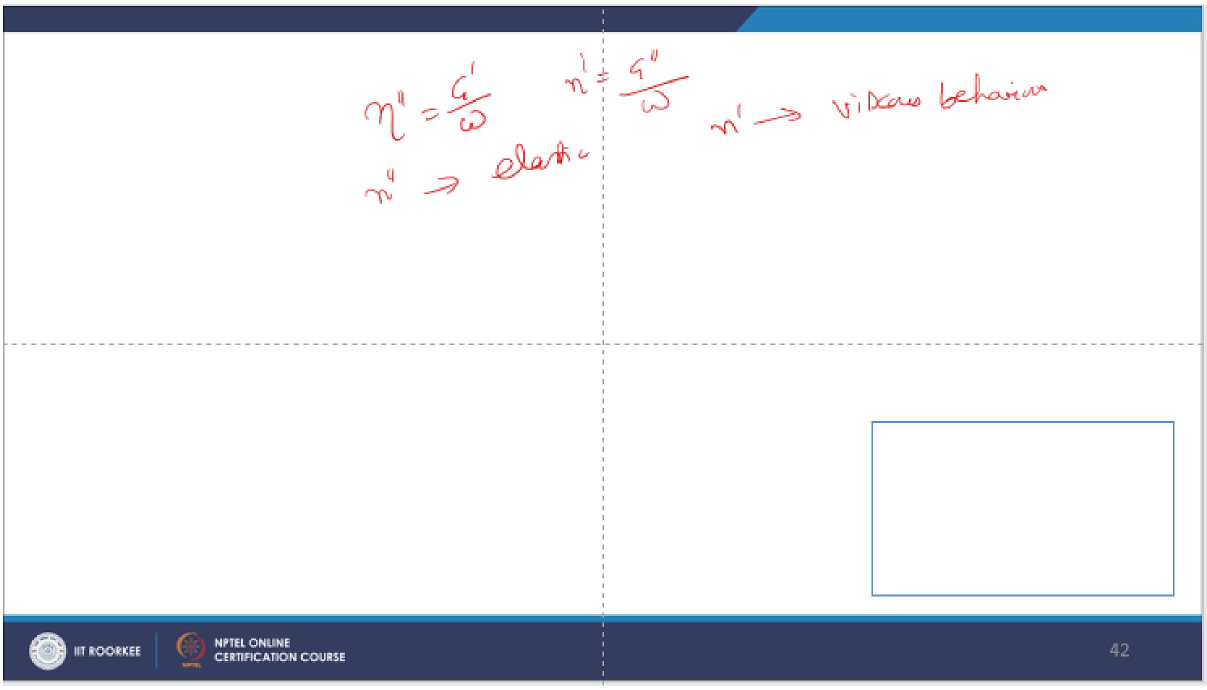
- In an elastic solid, $\delta = 0$ or $\tan\delta = 0$ as G' dominates G'' completely. However, in a viscous fluid, $\delta = 90°$ or $\tan\delta \to \infty$ as G'' dominates G' completely.
- In viscoelastic materials, $0 \le tan\delta \le \infty$, depending on the time scale and temperature. When the viscous and the elastic behavior are equal to $\delta = 45°$ or $\tan\delta = 1$, the material is making a transition from liquid to solid or vice versa.

Now, in viscoelastic material, the tan delta is equal to or greater than 0 and less than equal to infinity depending on the time scale and temperature when the viscous and elastic behaviour are equal to delta is equal to 45 degree or tan delta is equal to 1 the material is making a transition from liquid to solid or vice versa. For example, this occurs during curing of a thermoset when the system reaches its gel point and in the liquid state tan delta is greater than 1 as a G double prime or G prime and after gelation tan delta is less than 1 as the G prime is greater than G double prime. Similarly, when the test is regarded from a rheological point of view, a complex viscosity can be computed using the rate of stress to rate of deformation. Now, this can be represented as the eta is equal to tau xy over gamma xyt is equal to eta prime plus eta double prime where this one is the viscous component of the complex viscosity. This is also referred as the dynamic viscosity and eta double prime is the elastic component also referred as out of phase component of a complex viscosity.



- For example, this occurs during curing of thermosets when the system reaches its gel point. In the liquid state, $\tan\delta > 1$ as $G'' > G'$, and after gelation, $\tan\delta < 1$ as $G' > G''$.
- Similarly, when the test is regarded from a rheological point of view, a complex viscosity can be computed using the ratio of stress to rate of deformation.

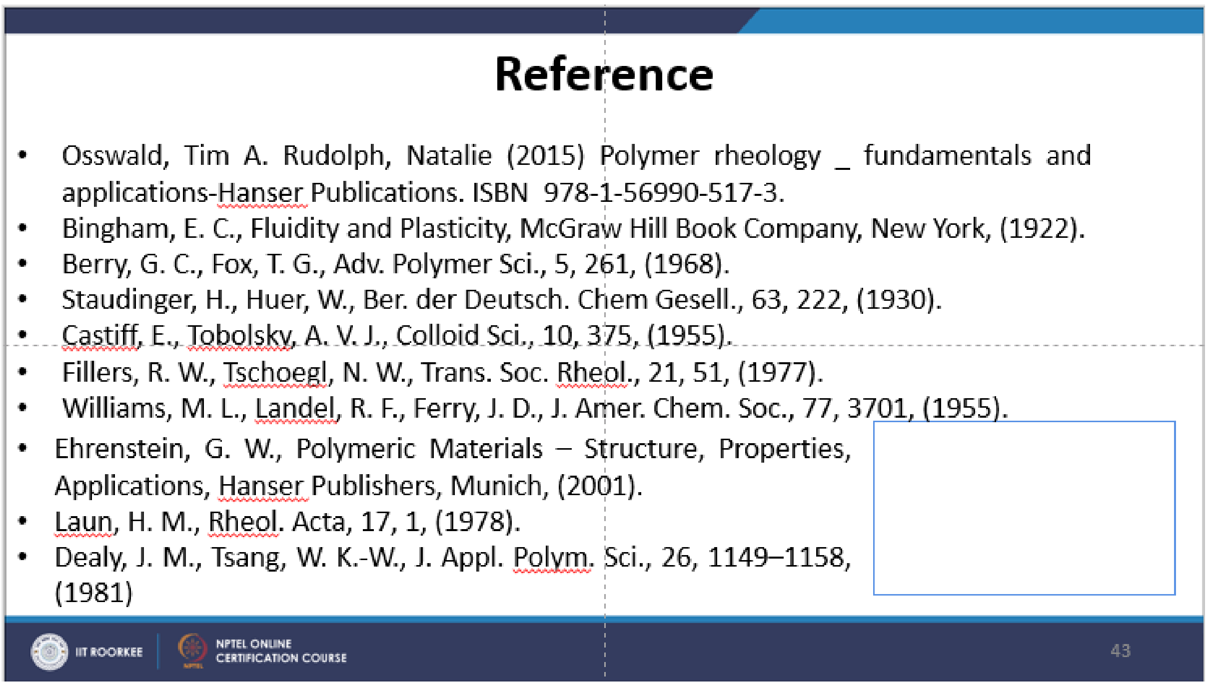
$$\eta^{*} = \frac{\tau_{xy}(t)}{\gamma_{xy}(t)} = \eta' + \eta''$$



$\eta'' = \frac{G'}{\omega}$ $\eta' = \frac{G''}{\omega}$ $\eta' \rightarrow$ viscous behaviour

$\eta'' \rightarrow$ elastic

The relaxation between the complex modulus and complex viscosity component, this can be described by eta double prime is equal to G prime over omega and eta prime is equal to G double prime over omega where this eta double prime this represents the elastic and this eta prime represents the viscous behaviour.



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This complex modulus representation is often used in industrial testing. So, dear friends in this particular segment we discussed the different type of a rheological behaviour with a grass root polymer chain phenomenon and for your convenience we have interested several references which you can utilise over the period of time if required. Thank you very much.