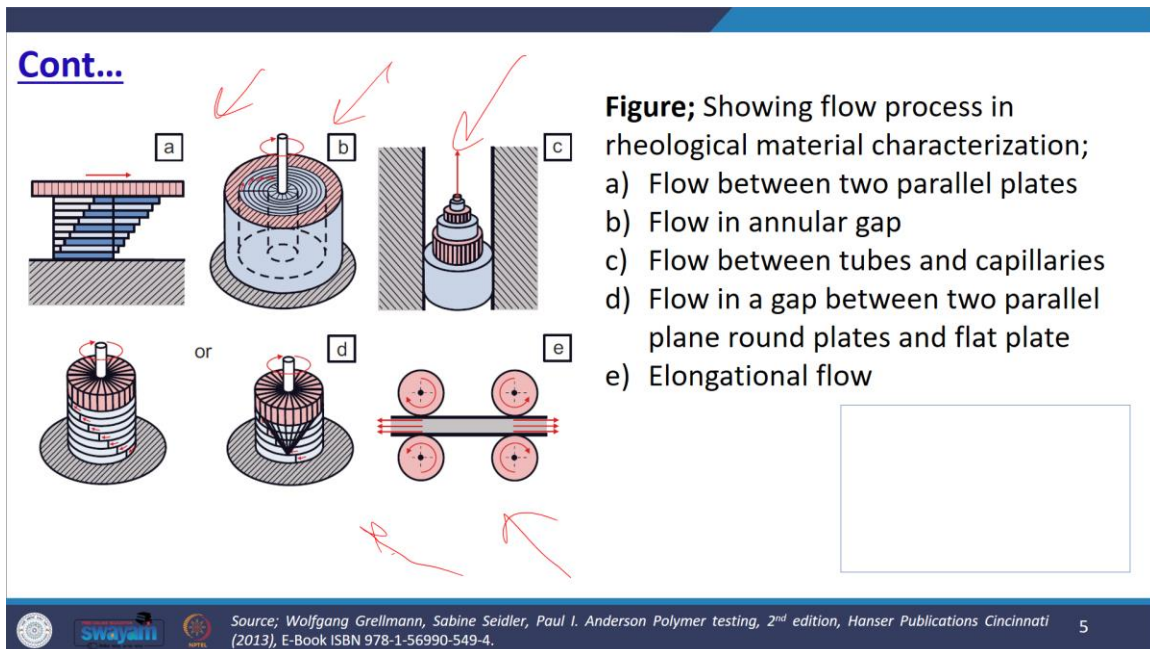


Lecture 39 - Polymer testing-04 (Measuring of rheological properties)

Hello friends, welcome to the polymer testing the next segment. Here we are going to discuss how to measure the rheological properties. In the previous lecture we discussed about the conditioning, we discussed about the apparatus for conditioning, air conditioning, rooms, enclosures, hygrometers, thermometers all these things we have discussed. Apart from this we discussed about the bulk material properties like mass density and measurement. We started the rheological properties aspect. In this particular segment we are going to discuss about the measuring aspect of these rheological properties.

We will discuss about different type of rheometers, how we can classify them. We will discuss about the rotational rheometers, capillary rheometers, extensional rheometers. So the first thing is that rheometry or viscometry. It is possible to experimentally demonstrate the link between the shear stress and the shear rate for defining the flow behaviour of a polymer metal utilizing a variety of setups that simulate certain real load conditions or particularly of the flow to be monitored.



the characteristics flow they are seen in the various rheometers and must be taken into account when assessing the experiments. So rheometers they are instrument used to measure the viscoelastic properties of solid, liquid and fluid between the ideal solid and fluids and viscometers they are the instrument used only to measure the flow behaviour of any kind of a viscous fluid. this particular figure shows the flow process in the rheological material characterization like this is the flow between the two parallel plates. This represents the flow in annular gap, this represents the flow between the tubes and

capillaries and then this represents the elongational flow and this particular figure represents the flow in the gap between two parallel plane or round plates and flat plates. So Let us talk about the classification of a rheometer as per their design and a working principle they are classified into the rotational rheometer, capillary rheometer, extensional rheometer.

Let us talk about the rotational rheometer, the fluid to be characterized they can typically be placed between the two rotationally symmetric component and that are mounted on a common axis in rotational rheometer. The spinning segment, the angular velocity determines the shear rate and the torque determines the shear stress and usually the rotating rheometers measuring methodology is standardized by the ISO 3219. for the determination of the flow characteristics there are two ways on which the rotational rheometer is based. One is the shear rate is specified in CS-rheometer, CS stands for the controlled stress and the velocity gradient is calculated proportionally to viscosity and second one is the shear rate is selected and resulting shear stress is calculated using CR rheometer and CR is the controlled rate. So based on the working principle it can be further classified into two different categories cuette measurement system and cell measurement system.

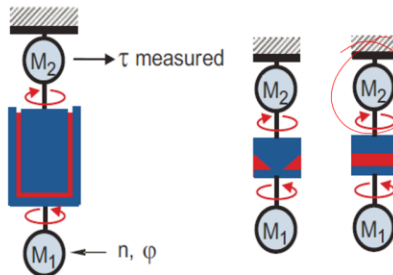
The cuette measurement system an electrical motor power in the external cylinder and lower plate being used. The fluid must be characterized and usually it is made to flow in the measurement gap where the shear resistance causes an internal cylinder upper spinning body to experience a torque that varies with viscosity. The internal cylinder usually is connected to a second motor which can be represented as M2 first motor can be represented as M1 this can produce the torque opposite to the motor M1 the previous first motor. The fact that the torque motor the second torque motor continuously readjust until the internal cylinder comes into a stop despite the flow of the test substance in the measurement gap determines the viscosity proportional torque transferred from the external cylinder or a lower plate via fluid. here it can be visualized easily in this type of a system the rotating measuring cup lower plate the shear stress measured on inside cylinder or a cone or upper plate therefore the magnitude of torque is the compensatory power needed by the motor M2 to operate and the stated number of rotation of the cylinder external cylinder or a lower plate leads to the velocity gradient.

Cont...

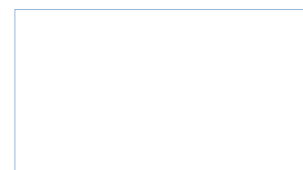
Couette type system; Rotating measuring cup/lower plate shear stress measured on inside cylinder/cone/upper plate.

✓ Thus, the magnitude of torque is the compensatory power needed by motor M2 to operate.

✓ The stated number of rotations of the external cylinder or lower plate leads to the velocity gradient.



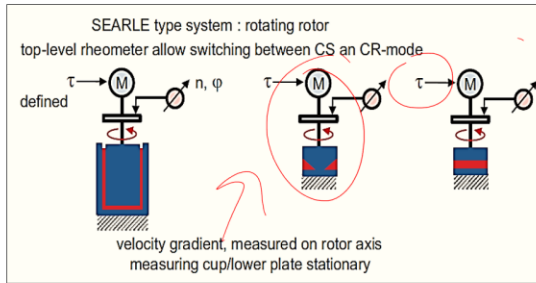
Figure; Couette type rotational rheometer



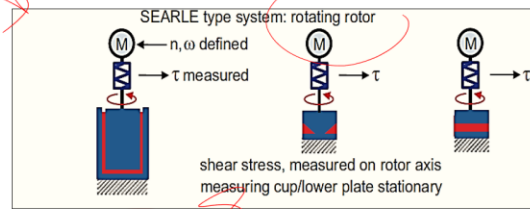
this is the Couette type of rotational rheometer. Let us talk about the cell type of measuring system the external cylinder or a lower plate is stationary and the torque value for a regulated electric motor M that drives the inner cylinder rotating body and a rotor they are stated. Any electric energy that enters the system is immediately converted into the torque values. the rotating body can only rotate at a given number of revolution or a certain velocity gradient which corresponds to the fluids viscosity because of the resistance the fluid creates to the torque or shear stress. The resulting number of revolution “n” is measured by an optical sensor whereby even a small angle of turn “φ” can be detected.

Cont...

(CS) rheometer; controlled torque defined shear stress, measure velocity gradient/deformation



(CR) rheometer/viscometer; controlled shear stress defined velocity gradient, measure shear stress



Figure; Searle type rotational rheometer

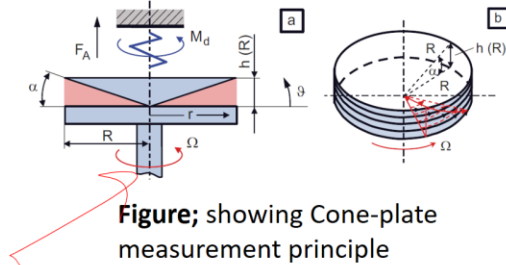
this is the rheometer the CR type of a rheometer viscometer controlled shear stress defined velocity gradient measures the shear stress. This is the cell type of a system that is the rotating rotor and here this is the CS rheometer control torque defined shear stress and measure velocity gradient and deformation. Here you see the torque motors and all these things. So velocity gradient measured on the rotor axis measuring the cup lower plate is stationary and this here the shear stress measured on rotor axis measuring the cup or lower plate is stationary. different type or different geometrical versions of a rotational rheometer they are the cone plate rheometer the plate rheometer and coaxial cylinder rheometer.

Cont...

- ✓ For small apertures α , and since $\tan \alpha \approx \alpha$, it is the case that:

$$\gamma = \frac{dW_u(r)}{dh(r)} = \frac{W_u(r)}{h(r)} = \frac{r\omega}{r \tan \alpha} = \frac{\omega}{\alpha}$$

$$\tau = \frac{3}{2\pi R^3} M_d$$



Figure; showing Cone-plate measurement principle

the cone plate rheometer a flat plate and a blunt cone they are arranged coaxially to one another in cone plate rheometer at the gap geometry between a flat plate and the cone is determined by the radius R and aperture " α " and the shear rate is only dependent on an angular speed " ω " and aperture because of aperture " α " is extremely tiny and the peripheral speed " W_s " is proportional to the radius and because of this unique design the cone plate rheometer is distinguished by a uniform shear rate distribution across its whole shear gap.

So this is the cone plate measuring principle and for small apertures " α " and since

$$\tan \alpha \approx \alpha$$

and for this is the case

$$\gamma = \frac{dW_u(r)}{dh(r)} = \frac{W_u(r)}{h(r)} = \frac{r\omega}{r \tan \alpha} = \frac{\omega}{\alpha}$$

it is decided what torque applies to the flat plate cone and a fluid in order to calculate the shear stress operating in the shear gap then

$$\tau = \frac{3}{2\pi R^3} M_d$$

for cone plate rheometer this can be used to determine the normal stress in addition to the shear stress rate dependency this from the measurement of the axial flow here you can see. the viscoelastic fluids elasticity creates overpressure in the shear gap which has the tendency to push the cone and plate apart.

Cont...

$f_y(r)$ shear gap surface

$$F_y(\gamma) = \int_0^R p_y(r) 2\pi r dr$$
$$= \frac{\pi R^2}{2} N_1(\gamma)$$

$$N_1(\gamma) = \frac{2F_y(\gamma)}{\pi R^2}$$

$N_1 \rightarrow$ Normal stress difference

$F_y \rightarrow$ Axial force



if we integrate the $p_y(r)$ over shear gap surface, the normal stress difference (N_1) can be determined from axial force (f_y). So

$$f_y(r) = \int_0^R p_y(r) 2\pi r dr = \frac{\pi R^2}{2} N_1(\gamma)$$

$$N_1(\gamma) = \frac{2F_y(r)}{\pi R^2}$$

Let us talk about the plate-plate rheometer. the two parallel plane plates with the radius of R and spacing H define the plate-plate rheometers. The radius of the revolving upper plate and the height of the gap both affects the velocity gradient in this particular type of measuring setup.

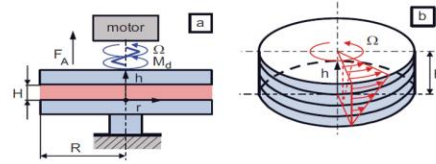
Cont...

The shear rate is the result of differentiating peripheral speed W_u according to height h :

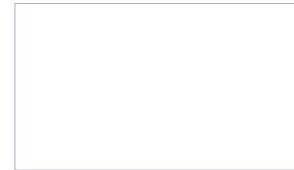
$$\dot{\gamma} = \frac{dW_u(r,h)}{dh} = \frac{d(r\omega(h))}{dh} = r \frac{\omega}{H}$$

At External radius R , shear rate is maximum

$$\dot{\gamma}_R = \frac{R\omega}{H}$$



Figure; Showing plate-plate measuring principle

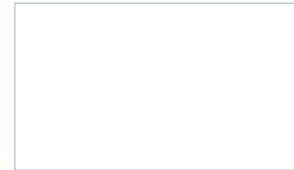


Cont...

$$\dot{\gamma} = \frac{dW_u(r,h)}{dh} = \frac{d(r\omega(h))}{dh} = r \frac{\omega}{H}$$

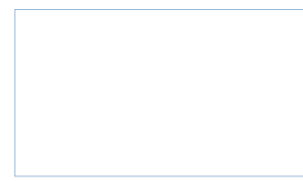
External radius R
Shear stress τ torque M_d

$$M_d = \int_A r \tau(r) dA = 2\pi \int_0^R r^2 \tau(r) dr = \frac{2\pi R^3}{3} \int_0^1 \gamma^2 \tau(\gamma) d\gamma$$



Cont...

for Newtonian fluids
 $\tau(r) = \eta_N \gamma(r) = \eta_N r \frac{\omega}{H}$
 τ_T η_N for viscosity at the plate edge
 $\tau_T = \frac{2M_d}{\pi R^3}$ and $\eta_N = \frac{2M_d H}{\pi R^3 \omega}$
 Exponential law of Ostwald and de Waele
 Non Newtonian fluid
 $\eta(\dot{\gamma}) = \frac{2M_d H (3+n)}{\pi R^3 \omega^4}$
 $= \eta_s(\dot{\gamma}) \frac{(3+n)}{4}$
 $\eta_s(\dot{\gamma})$ is the apparent viscosity



As a result the distance between the plates or the angular viscosity velocity over a sizable area these can be changed to alter the shear rate in a plate-plate system. In plate-plate rheometer the shear rate varies with the different radii and the torsion on the cylindrical bar is the equivalent to the shear. The shear rate is the result of differentiating the peripheral speed (W_u) according to the height (h). So,

$$\gamma = \frac{dW_u(r, h)}{dh} = \frac{d(r\omega(h))}{dh} = \frac{r\omega}{H}$$

At external radius R , the shear rate has its maximum. So,

$$\gamma = \frac{R\omega}{H}$$

The shear stress (τ) is calculated from torque M_d and torque can be expressed by integrating the shear stress over shear surface. So,

$$M_d = \int_0^R r\tau(r)dA = 2\pi \int_0^R r^2\tau(r)dr = \frac{2\pi R^3}{\gamma_R^3} \int_0^{\gamma_R} r^2 t(r) d\gamma$$

For Newtonian fluids,

$$\tau(r) = \eta_N \gamma(r) = \frac{\eta_N r \omega}{H}$$

From this the $\tau(r)$ result for shear stress and η_N for viscosity at the plate edge.

This can be represented as

$$\tau_R = \frac{2M_d}{\pi R^3} \text{ and } \eta_N = \frac{2M_d H}{\pi R^4 \omega}$$

Assuming the exponential law of Ostwald-de waele the viscosity resulting at the plate H of non-Newtonian fluid can be calculated as,

$$\eta \gamma_R = \frac{2M_d H (3 + h)}{\pi R^4 \omega^4} = \frac{\eta_S(\gamma_R)(3 + h)}{4}$$

Here, $\eta_S(\gamma_R)$ is the apparent viscosity.

The coaxial cylinder rheometer a cylinder cylindrical vessel with the radius (R_a) and an interior cylinder with a radius (R_i) are arranged coaxially to form the coaxial cylinder rheometer. Shear load can be produced by rotating either the internal or external like or type of cylinder while keeping the other cylinder fixed and there are numerous coaxial measurement devices used and each having a unique working principle and set of geometrical specification. So there are another standard that is DIN53018 this measurement tool and practice they are standardized.

Considering the laminar stationary flow in the measuring gap and the fluid adhesion to the wall (i.e., angular velocity of internal cylinder $\omega_i = 0$), the shear stress usually is caused by

$$\tau = f(r) = \frac{M_d}{2\pi r^2 h}$$

For Newtonian fluid the resulting torque is

$$M_d = \frac{4\pi R_i^2 R_a^2 h}{R_a^2 - R_i^2} \eta \omega = C_n \Omega_a$$

Cont...

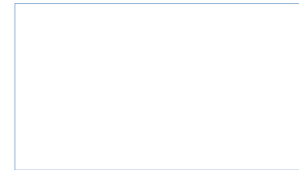
Considering laminar, stationary flow in the measuring gap and fluid adhesion to the wall (angular velocity of the internal cylinder $\omega_i = 0$), shear stress is caused by:

Newtonian fluid the resulting torque is

$$\tau = f(r) = \frac{M_d}{2\pi r^3 h}$$
$$M_d = \frac{4\pi h R_a^2 R_i^2}{R_a^2 - R_i^2} \eta \omega = C \eta \omega$$

C → device constant

$$\tau = \frac{M_d}{2\pi R_i R_a h}$$



Where, C is the device constant this with

$$\tau = \frac{M_d}{2\pi R_i R_a h}$$

$$\gamma = \frac{2R_i R_a}{R_a^2 - R_i^2} \omega$$

So the apparent viscosity η_s can be calculated as;

$$\eta_s = \frac{R_a^2 - R_i^2}{4\pi R_i^2 R_a^2 h} (M_d / \Omega_a)$$

So apparent viscosity for the Scale type with $\omega_a = 0$.

Ω_a is the angular velocity of the external cylinder.

$$\eta_s = \frac{R_a^2 - R_i^2}{4\pi R_a^2 R_i^2 h} \left(\frac{M_d}{\Omega_i} \right)$$

Cont...

$$\gamma = \frac{2R_i R_o \omega}{R_o^2 - R_i^2}$$

The apparent viscosity η_s can be calculated

$$\eta_s = \frac{(R_o^2 - R_i^2)}{4\pi R_i^2 R_o^2 h} \frac{M\dot{\gamma}}{\dot{\gamma}}$$

Apparent viscosity for the ~~for~~ ~~scale~~ ~~type~~ with $\omega_a = 0$
(ω_a angular velocity of external cylinder)

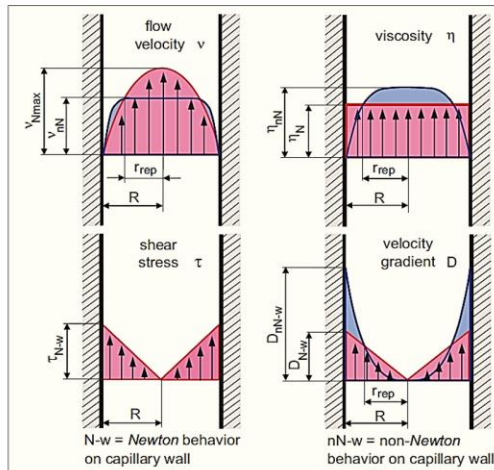
$$\eta_s = \frac{(R_o^2 - R_i^2)}{4\pi R_i^2 R_o^2 h} \frac{M\dot{\gamma}}{\dot{\gamma}}$$



Let us talk about the capillary rheometers. The fluid being measured must pass through a capillary with a circular or circular ring or square cross section slit in order for a capillary rheometer to function. Both low viscosity fluid and the fluid with the high viscosity can be measured using the capillary rheometer and they operate on the basis of gravity for low viscosity fluid but require the application of the proper flow process pressure for extremely viscous fluid. there are 2 type of a capillary rheometer the low pressure capillary rheometer and high pressure capillary rheometer.

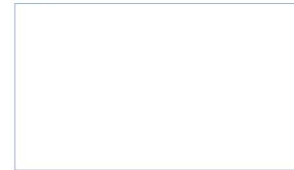
The in principle after adjusting the temperature to the proper level the fluid to be characterized is fed through a capillary either by gravity or under pressure from a reservoir. When the capillary reaches its determinants the pressure is equal to the atmospheric pressure and the volume going through the capillary per unit of time and the pressure gradient are measured and the resulting rheological parameters are determined. So when using the capillary viscometer for measuring the pressure rho can pressure difference rho can be defined and the resulting volume flow Q or pressure difference can be specified and the volume flow Q can be measured. here this particular figure showing the principle curve of the flow rate shear rate stress and viscosity of Newtonian or non-Newtonian fluids. So based on force and mass balance a shear stress method and the marginal circumstances flow processes in the capillaries are described.

Cont...



Figure; Showing the principle curve of flow rate, shear rate, shear stress and viscosity of Newtonian or non-Newtonian fluids.

Based on force and mass balances, a shear stress method, and marginal circumstances, flow processes in capillaries are described.



So, the shear stress dependent on the radius r is calculated by

$$\tau_r = \frac{r}{2\Delta l} \Delta P$$

Where, Δl is the capillary length between pressure measuring points and ΔP is the pressure drop over capillary segment Δl . So the volume throughput may be used to calculate the velocity gradient and that is the capillary radius dependent. So,

$$\gamma_r = \frac{4}{\pi r^3} Q$$

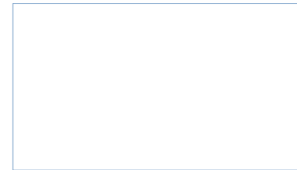
Cont...

The shear stress dependent on radius r is calculated by

$$\tau_r = \frac{r}{2\Delta l} \Delta P \rightarrow \text{Capillary length between pressure measuring ports}$$

$\Delta P \rightarrow$ pressure drop over capillary segment Δl

$$\tau_r = \frac{4}{\pi r^3} Q$$



The Hagen Poiseuille relation this can be used to compute the time-dependent volume flow $Q = \frac{v}{t}$ through a tube of length Δl at a pressure drop of ΔP which determines the viscosity. So,

$$\frac{dv}{dt} = \frac{\pi r^4 \Delta P}{8\eta \Delta l}$$
$$\eta = \frac{\pi r^4 \Delta P}{8v \Delta l} t = \frac{\pi r^4 \Delta P}{8\Delta l Q}$$

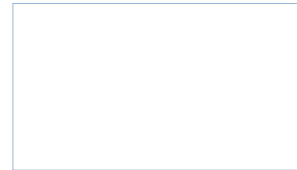
Cont...

The Hagen-Poiseuille relation can be used to compute the time-dependent volume flow $Q = \frac{V}{t}$ through a tube of length Δl at pressure drop Δp , which determines viscosity;

$$\frac{dV}{dt} = \frac{\pi r^4 \Delta p}{8 \eta \Delta l}$$
$$\eta = \frac{\pi r^4 \Delta p}{8 V \Delta l t} = \frac{\pi r^4 \Delta p}{8 \Delta l Q}$$

height h width w $h \ll w$

$$\tau_w = \frac{h \Delta p}{2 \Delta l}$$



The shear stress at the valve of capillary with a square cross section that is a slit with the height (h) and width (w) can be calculated for $h \ll w$ and this can be calculated as;

$$\tau_w = \frac{h \Delta p}{2 \Delta l}$$

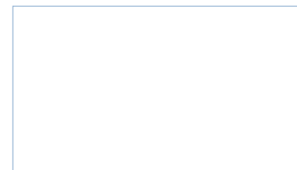
Cont...

velocity gradient it follows that

$$\tau_w = \frac{6}{w h} Q$$

for viscosity Correspondingly

$$\eta = \frac{w h^3 \Delta p}{12 \Delta l Q}$$



So for velocity gradient it follows that

$$\gamma_w = \frac{6}{wh^2} Q$$

for viscosity, correspondingly

$$\eta = \frac{wh^3 \Delta P}{12 \Delta l Q}$$

Melt flow index measurement:

The melt flow index which indicates in gram the amount of material flowing through a capillary of a specified dimension at a specific pressure and temperature during ten minutes it is known as MFR value or melt flow mass flow rate. This can be represented as

$$MFR = m \frac{600}{t}$$

where (m) is the median value of mass segment and (t) is the interval for cutting off.

In ISO 1133 the determination of the melt flow index is standardized and the melt flow index is given in unit of gram into 10 minute inverse.

➤ Melt-Flow Index Measurement

- ✓ The melt-flow index, which indicates in grams the amount of material flowing through a capillary of specific dimensions at a specific pressure and temperature during ten minutes, is known as the MFR value (melt mass-flow rate).

$$MFR = \frac{m \cdot 600}{t}$$

Where,

'm' is median value of mass segment

't' is time interval for cutting off



According to the material and a load is specified in the test standard a certain testing temperature is chosen. In addition to the melt flow index expressed in gram over 10 per 10 minutes the melt volume flow rate MVR which determines the volume per time unit

and eliminates the influence of melt density this can also be calculated. The MVR the melt volume flow rate stated in cubic centimeter per 10 minute is calculated as;

$$MVR = \frac{427l}{t}$$

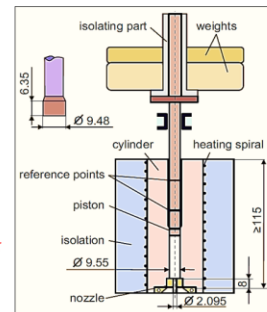
where (l) is the traverse piston path. The figure shows the melt flow indexer. The viscosity at relatively low shear rate is merely one value that the melt flow index represents and it is impossible to compare directly to viscosity reading obtained from high pressure capillary rheometer.

Cont...

- ✓ MVR (Melt volume flow rate) stated in $\text{cm}^3/10\text{min}$ is calculated as;

$$MVR = \frac{427.l}{t} \quad \text{Where } l \text{ is traversed piston path}$$

- ✓ The viscosity at relatively low shear rates is merely one value that the melt-flow index represents.
- ✓ It is impossible to compare it directly to viscosity readings obtained from high-pressure capillary rheometers.



Melt Flow indexer



Cont...

Mass of add-on weight (kg)	Piston force (N)	Piston Pressure (bar)	Apparent shear (Pa)
0.325	3.2	0.45	2.9×10^3
1.20	11.7	0.17	1.1×10^4
2.16	21.2	3.00	1.9×10^4
3.8	37.3	5.28	3.5×10^4
5.0	49	6.95	4.5×10^4
10.0	98.1	13.90	9.1×10^4
15.0	147.1	20.84	1.4×10^5
21.6	211.8	30.01	1.9×10^5



Source: Wolfgang Grellmann, Sabine Seidler, Paul I. Anderson Polymer testing, 2nd edition, Hanser Publications Cincinnati (2013), E-Book ISBN 978-1-56990-549-4.

37

In this particular table we have enlisted based on that particular equipment the mass of add on weight then the piston force whatever piston force is applied and the piston pressure and by this way how we have calculated the apparent shear. Let us talk about the extensional rheometer. The behaviour of viscoelastic flow differs from shear loading under the extensional deformation. because of this it is essential to comprehend both the behaviour under the shear loading and that under the extensional deformation which is what gave rise to the extensional rheometry. Melt flows inside mould channels, uniaxial melt spinning method or film blowing processes all exhibits the extensional melt deformation.

Elongational flow has a velocity gradient and that is parallel to the flow direction as opposed to the shear flow which has a velocity gradient and that is perpendicular to the flow.

$$\dot{\epsilon} = \frac{dv_x}{dx}$$

The calculation of the tensile stress and elongational velocity necessary to strain a solution is the foundation of their operation. The extensional rheometers which work with temporarily constant tensile stress or elongational velocity this can assure isothermal uniform elongation they are employed for highly viscous melt. The Meissner and Munstedt extensional rheometer they are the most well-k n instrument for the same.

Cont...

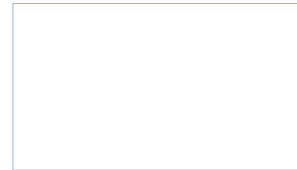
- ✓ Elongational flow has a velocity gradient that is parallel to the flow direction as opposed to shear flow, which has a velocity gradient that is perpendicular to the flow.

$$\dot{\epsilon} = \frac{dv_x}{dx}$$



Note;

- ✓ The calculation of the tensile stress and elongational velocity necessary to strain a solution is the foundation of their operation.



The figure shows the Meissner and Munstedt extensional rheometer. This device can be used to achieve the constant elongational velocity $\dot{\epsilon}$ over the constant length L_0 when the number of revolution n_1 or n_2 which they are constant. So here you see that this is the load cell you can put the specimen over here. This is the oil bath and this is surrounded by the heating bath and there are couple of steel straps the motor and a control power units and all these things are there and you can insert the heating liquid from this outer jacket. When we need to calculate the elongational velocity it can be calculated as;

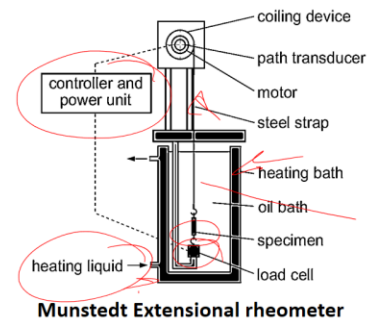
$$\dot{\epsilon} = \frac{2\pi R(n_1 + n_2)}{L_0}$$

Cont...

■ Meissner and Munstedt Extensional rheometer

A Meissner device can be used to achieve constant elongational velocity $\dot{\epsilon}$ over constant length L_0 when the number of revolutions n_1 or n_2 is constant:

$$\dot{\epsilon} = \frac{2\pi R (n_1 + n_2)}{L_0}$$



Source: Wolfgang Grellmann, Sabine Seidler, Paul I. Anderson Polymer testing, 2nd edition, Hanser Publications Cincinnati (2013), E-Book ISBN 978-1-56990-549-4.

41

Cont...

- ✓ A force sensor installed on a gear set measures the elongation force F .
- ✓ Cross-head force and momentary cross-sectional surface, which decreases exponentially as elongation rises, are used to calculate tensile stress:

$$\sigma = \frac{F}{A} = \frac{F \cdot \exp(-\epsilon)}{A_0}$$

- ✓ Understanding the extension-rheological behavior of polymer melts is crucial when designing processing techniques like film blowing, blow moulding, or fiber spinning that place high uniaxial or biaxial strain on the melt.



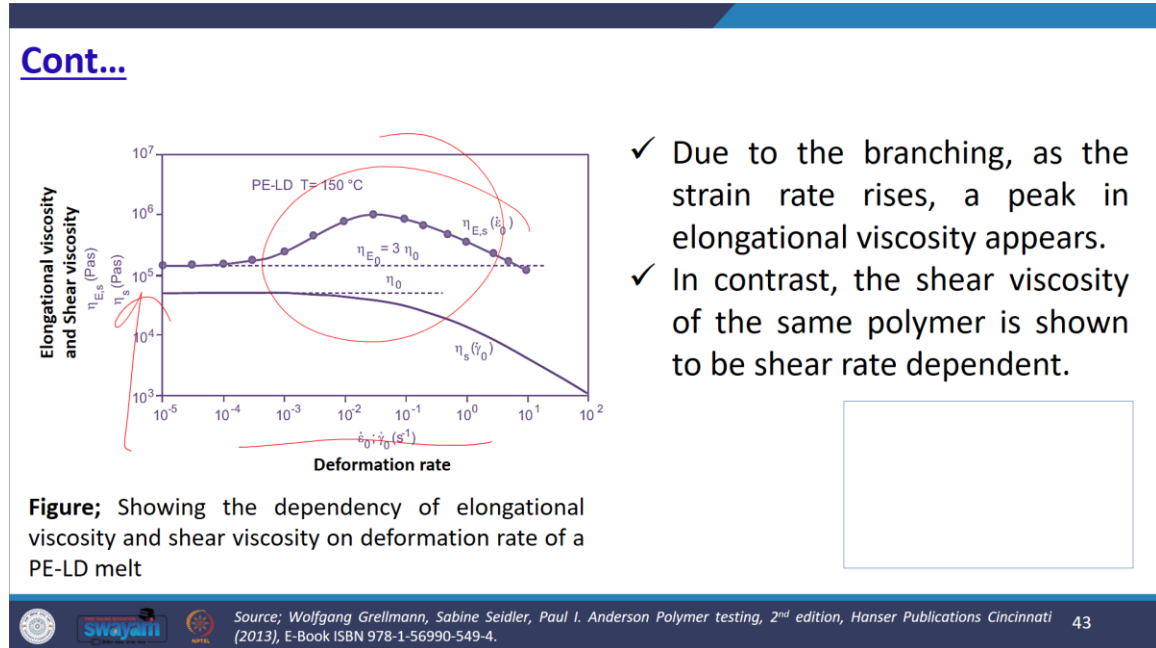
42

A force sensor installed on gear set measures the elongation force F and the cross-sectional force and momentary cross-sectional surface which decreases exponentially as elongation rate they are used to calculate the tensile stress. This can be calculated as;

$$\sigma = \frac{F}{A} = \frac{F \cdot \exp(-\epsilon)}{A_0}$$

So understanding the extensional rheological behaviour of polymer melt which is crucial when designing processing techniques like film blowing, blow molding or fibre spinning

that place highly uniaxial or biaxial strain on the melt. here you see the dependency of elongational viscosity and the shear viscosity on deformation rate of PE-LD melt. So due to the branching as the strain rate rises a peak in the elongation where velocity viscosity appears.



In contrast the shear viscosity of the same polymer is shown to be shear rate dependent. So dear friends in this particular segment we discussed the rheological behaviour of the polymeric system and for your convenience we have enlisted several references which if you require then you can use those references for your further studies. Thank you very much.

