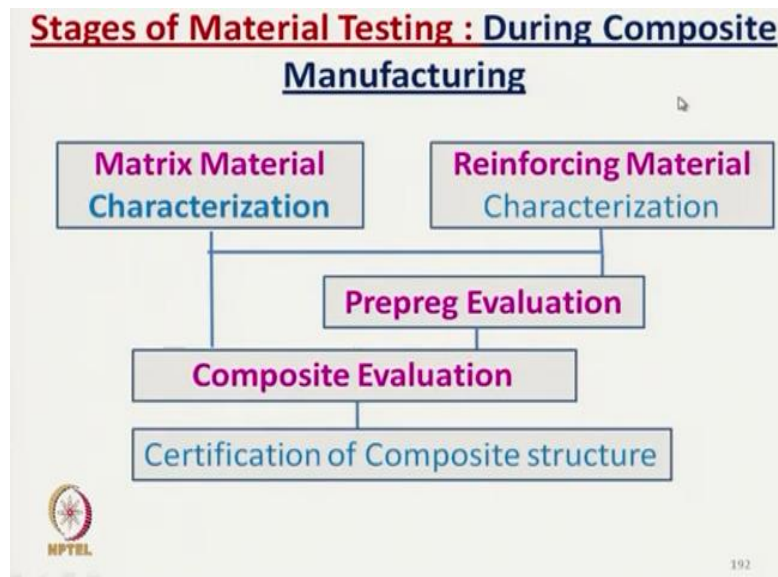


Technical Textiles
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Lecture - 10
Textile Reinforced Composites (Contd.,)

Hello everyone. Now, our next topic is that characterization of fibre reinforced composite materials. It is very important to understand the characteristics of the materials used for composite making that is matrix component and reinforcing fibre or yarn or fabric component and also the characteristics of final composite. So, in at present, we will discuss here the characterization of matrix, characterization of fibre, yarn, fabric and also characterization of final composite. We will start with the matrix material characterization.

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So, the stages are for manufacturer of composites, their raw materials are matrix materials and reinforcing materials. So, first they have to test matrix whether these matrix are as per the specification or as per the requirement. Then, we have to understand the characteristics of reinforcing material. So, we need to test the reinforcing material before we go for the composite manufacturing.

So, in reinforcing material, we can test individual form like fibre, yarn or fabric form or else we can test in addition to that, we have to test in the prepreg after that we manufacture composite and composite evaluation is extremely important for the manufacturer as well as the user of the composite. After evaluation then we can certify the composite structure,

whether it is suitable for a particular application or not. First we will start with the matrix characterization, matrix is nothing but normal polymer. And here we are not going to discuss the polymer characterization; we go a little bit quickly in the characterization process.

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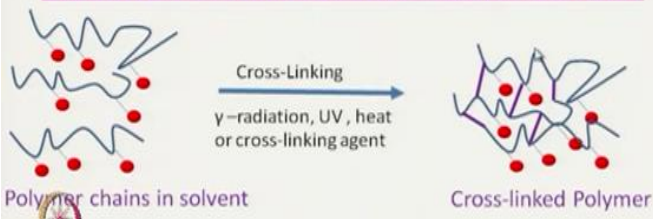
Matrix Material Characterization

Type of Matrix:

A) Thermoset B) Thermoplastic

(A) Thermoset Matrix:

- Becomes irreversibly hardened upon being cured
- Curing is the action of heat or radiation which results in extensive cross-linking between polymer chains



Example of Thermoset Polymer: **Epoxy, Phenolic resin, Unsaturated polyester etc.**

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So, you know there are 2 types of matrix materials, one is thermoset, and next is thermoplastic. Most of the characterizations of thermoset and thermoplastics are similar apart from a few additional tests for specifically for either thermoset or thermoplastic, as we know thermoplastic, thermoset matrix, they are irreversible hardened upon being cured during curing action, extensive cross linking takes place.

These are the cross link and due to this cross linking, they are not actually reversible and this cross linking is done by heating or by different radiation or using some cross linking agent. So, the examples are epoxy, phenolic resins, unsaturated polyester. So, the test methods of these thermosets are a little bit different than thermoplastic matrix to some extent for specific characteristics.

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(A) Thermoset Polymer Characterization

Tests for Neat Resin :

- i) Infrared Spectroscopy (IR-Spectroscopy)
- ii) High Performance Liquid Chromatography (HPLC)
- iii) Viscosity
- iv) Gel Time
- v) Moisture Content
- vi) Mechanical properties
- vii) Density



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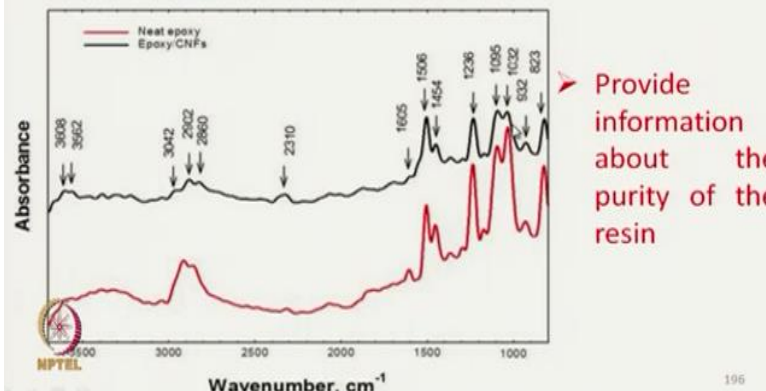
So, this thermoset matrix to evaluate the characteristics we may use IR spectroscopy, HPLC high performance liquid chromatography, viscosity which is extremely important for composite manufacturing because the melt flow or flow length here it is not melt flow here of flowing of resin into the structure that is between the reinforcing material is important, gelation time, gel time is the characteristics which is specifically for thermoset polymers.

It is not for thermoplastic polymer, gel time means with the normal temperature, room temperature or with a certain other temperature or during cross linking the solidification from the liquid state or lower viscosity state to higher viscosity state that timing is very important for composite making, we must know the gel time, moisture content is extremely important, because moisture present in the polymer will actually deteriorate the characteristics of composite, mechanical properties are important, density of polymer we must know.

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(i) Infrared Spectroscopy (IR-Spectroscopy)

- Gives an idea about the different functional groups present by showing peaks in the spectra at different wave-length

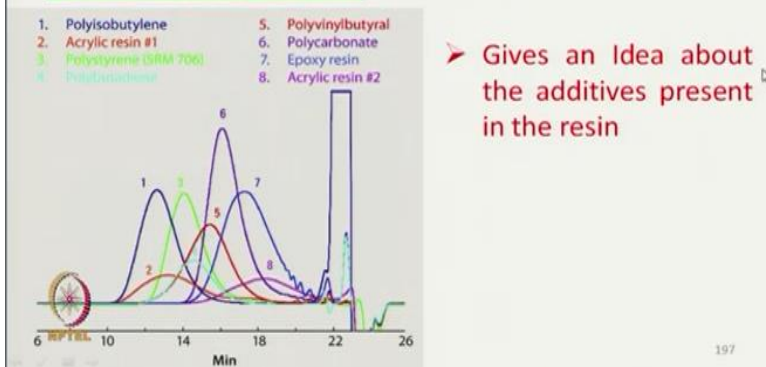


Now, IR spectroscopy here it gives an idea about different functional group present. We must know, what are the different functional group present, just to know the purity of the polymer, if the polymer is not pure as we know that it will affect the characteristics of final composite. So, IR spectroscopy is required. So, different functional groups will show different peaks in the spectra at different wavelength. So, from this peaks we will come to know the presence of different functional groups.

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(ii) High Performance Liquid Chromatography (HPLC)

- HPLC is an analytical technique which is used to identify and quantify each component present in the mixture
- During HPLC a resin sample is injected in a Chromatographic column



HPLC, so by high performance liquid chromatography, we just we can identify and quantify each component present in the mixture, if we take the mixture or if we take any polymer, so, where whether there are any mixture or not that we can identify using the HPLC. ,t gives an idea about the additives present in the resins. So, what are the different additives present that we can identify.

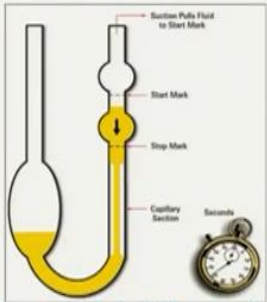
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(iii) Viscosity

- It is a measure of resistance to flow
- Unit of viscosity is Poise which is equivalent to Pascal second (Pa·s), or (N·s)/m²

Types of viscometer

- U-tube viscometers
- Falling ball viscometer
- Vibrational viscometers
- Rotational viscometers

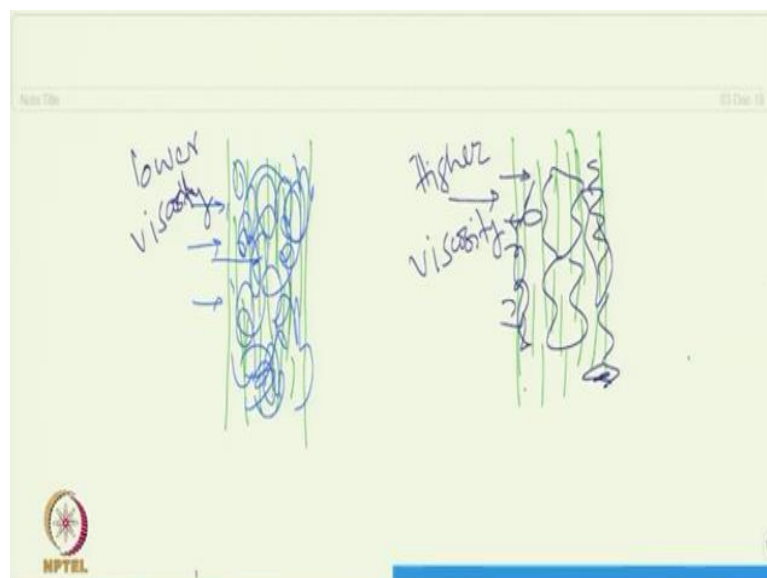


- It gives an idea about the molecular weight of the resin
- Low resin viscosity improves the resin distribution in the composite structure and reduces the void content the composites

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Viscosity is has already been mentioned, it is a very important characteristics here. So, it is a measure of resistance to flow. It is a unit is Poise which is Pascal second. The resistance to flow is extremely important for composite manufacturing. If the matrix material does not flow smoothly or easily within the reinforcing material structure, the quality of composite which we get will be inferior in quality, like this if viscosity is high and due to that the matrix material if it cannot penetrate within the structure, so, that will create void.

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So, let us see, suppose this is the reinforcing structure, this is another reinforcing structure. Now, this is a polymer with lower viscosity, they will penetrate easily inside the structure and with the higher viscosity if we take, so, this is higher viscosity they will not be able to

penetrate clearly, so there will be void created within the structure they are not able to penetrate properly.

So, that will actually deteriorate the composite characteristics. So, this is measured using the U-tube viscometer, falling ball viscometer, vibrational viscometer, rotational viscometer and here the technique is that, so viscosity with a lower than polymer with the lower viscosity will flow at higher rate so that the flowing time will give us idea about the viscosity.

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(iv) Gel Time

- Also Known as gelation time
- Gelation is a phenomenon describing the transition of a material from a viscous liquid to an elastic solid during curing
- This transition does not occur instantaneously, the viscosity of the system increases gradually, which makes the precise gel time determination very difficult
- Gel time is a useful parameter for quality control of both resins and preregs

Methods for Determination of the Gel Time

- Manual Method
- Automatic measurements using gel-timer

Manual method is used in the industry since it does not require any expensive equipment

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Now gel time is actually it is known as a gelation time. Gelation is a phenomenon describing the transition of material from viscous liquid to an elastic solid during curing. So, the curing may be done using heating or maybe some radiation and this transition does not occur instantaneously, it takes time. It is gradual. So that is why it makes it difficult to get precise gelation time, but at least we will get some idea.

So it is a useful parameter for quality control of both resins and preregs. So you can use manual technique or automatic measurement technique. So, manual method is basically used in industry. Since it does not require any expensive equipment, at least we get some idea about the gelation time that helps in planning the curing time.

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Gel Time Evaluation: Manual Method

- Based on “by eye” evaluation of the rheological behavior of the resin by operator
 - The gel times obtained by such methods depend very heavily on the experience of the operator
 - Resin samples with cross-linkers were kept in a test-tube and maintain a constant temperature in the oil bath with string
 - A glass-rod is used as a probe to determine resin viscosity
- The time when the solidified resin string-line is broken is termed as Gel time



So, based on by eye evolution technique, of the rheological behavior of resin by operator. The gel time obtained by such method depends very heavily on the experience of the operator because otherwise it will add to error. So resin samples with crosslinker were kept in a test tube, this is test tube and maintained at a constant temperature in oil bath and we start studying a glass rod is used as a probe to determine the resin viscosity the time when it starts solidifying.

The resin start solidifying the string line is broken, so that the string line is not continuous. And that means it is gel time has reached it is gelation has reached, so that time is recorded and we get gelation time. It gives an idea about the solidification time of the composite, it should not be too high or also it should not be too low. If it is too high, that means the actual process time is very high, it is longer that will make the manufacturing method, it is not viable. On the other end, if gelation time is very short, even if it is solidifies before proper penetration of matrix within the structure that will also create problem.

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(v) Moisture Content

$$\text{Moisture Content} = \frac{W-D}{W} \times 100$$

Where D = Dry weight of the sample

W = Wet weight of the sample

In general the matrix properties deteriorate with increasing Moisture Content



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Next important characteristics is moisture content. It is nothing but percent moisture present on the material; so, we can use the drying technique, oven drying technique. So, dry weight of the sample we can take and this is the weight of sample with water. So, this is moisture content. So, in general the matrix properties deteriorate with the increase in moisture content. So, moisture content of say polymer if it is high then, matrix characteristics will deteriorate. So, we must select the matrix material with lower moisture content.

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(B) Thermoplastic Polymer Characterization

Thermoplastic Matrix: Becomes pliable or moldable above a specific temperature and solidifies upon cooling

These polymers are recyclable

Example: Polypropylene, Polyethylene, Nylon, PLA etc.

Tests for Neat Resin :

i) Infrared Spectroscopy (IR-Spectroscopy)

ii) Moisture Content iii) Melting Temperature (DSC)

iv) Melt Flow Index v) Mechanical properties



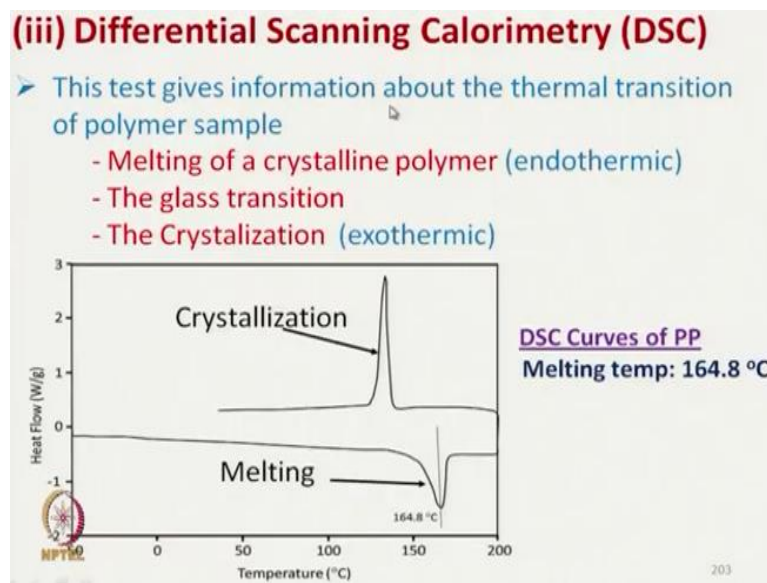
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Now, specifically for thermoplastic polymer characterization, we have a few extra tests we have to do which is not required for thermoset matrix because thermoplastic matrix it becomes pliable or moldable, above specific temperature and solidifies upon cooling. So, that means we must know the softening temperature, T_g or maybe that is a melting temperature we must know.

Because this if the softening temperature is very low that means it is application will be limited. So, polypropylene, polyethylene, nylon these are the examples of thermoplastic polymer. So, IR spectroscopy is done as we have seen in case of thermoset, moisture content, mechanical properties are required, density is also required for both for thermoplastic and thermoset. Here we do not need gelation time but we need melting temperature that is melting point we have to measure using DSC.

And also we must know the melt flow index that means, at certain force, how much polymer is flowing through certain hole that we measure. This is very well flowing this is very important for composite making, because if the melt flow index is low that means, the total amount of the polymer flow per unit time is low that means, it will create problem in proper penetration of matrix within the structure.

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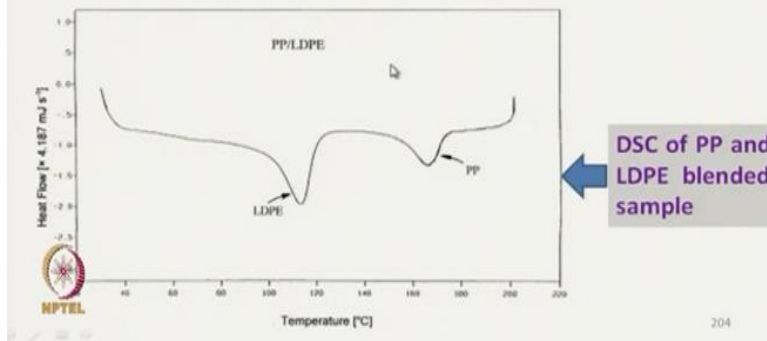


So, DSC differential scanning calorimetry is used for measuring the melting point.

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Differential Scanning Calorimetry ... cont

- A DSC curve of a polymer also provide information about the purity of the sample
- In case of impure sample, peak responsible for melting shifts its position or two separate peaks are observed



And also from DSC, we can get an idea about the purity of the material. If it is the polymer is pure, then we will get a single peak. But in case of mixed impure sample, the polymer will show the multiple peaks like here example, PP and LDPE, the mixture it gives 2 peaks.

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(iv) Melt Flow Index

- Measure the ease of flow of the Melted polymer

i) Melt-Mass Flow Rate (MFR): The mass of Material flowing through a die at a specified temperature (g/10min)

ii) Melt Volume Rate (MVR): The volume of Material flowing through a die at a specified temperature (cm³/10min)

$MVR = MFR / \text{Material density}$

Factor affecting MFI:

- Temperature accuracy
- Moisture in sample
- Method parameters (e.g. Die size)
- Material compactness

And, as I have already mentioned the melt flow index, it measures the ease of flow of the melted polymer how easily it will flow through the reinforcing material through the structure. It is actually it is measured in 2 parameters. One is melt mass flow rate, MFR. So, melt mass flow rate which is the mass of material flowing through a die of specific dimension at a specific temperature that is a gram per 10 minute.

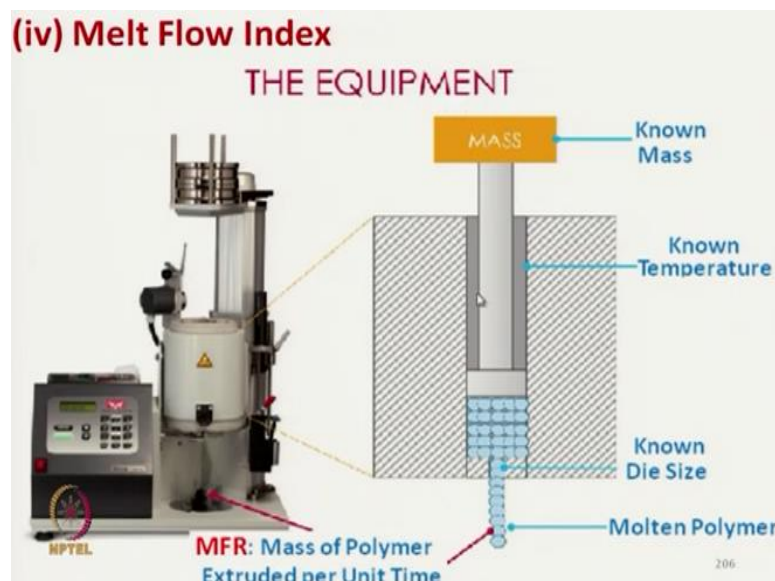
How much material is flowing through a die? And melt volume rate, this is the volume of material flowing through a die. So, at specific temperature it is important because if we

increase the temperature, the viscosity will reduce and mass flow will change. So, we normally we should use the temperature which is specified for application in composite manufacturing.

So, melt mass melt volume rate it is expressed in cubic centimeter per 10 minute and the relationship is MVR equal to MFR divided by material density at particular temperature. So, this is the material density here. So, if we divide this by this MFR, if we divide by material density we will get MVR. So, the factor affecting the melt flow index, these are the factors temperature accuracy.

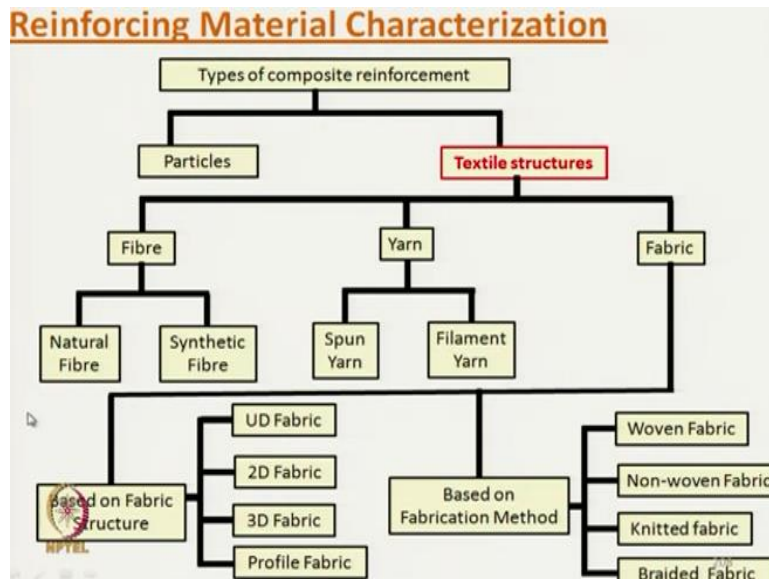
So, if we cannot maintain the temperature properly, this will affect the ultimately the melt flow index below. So, you must accurately maintain the temperature, moisture in the sample, method of pyramidal die size. So, if we have to keep the die size standard, otherwise it will change the melt flow index value and material compact because, the melt flow if the material is compact that will actually give us the lower melt flow index.

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This is the standard equipment. Known mass of material is placed here with a known temperature, known die size and molten polymer, we can measure take the volume or take the mass of the polymer extruded, we can take. Next is that reinforcing material characteristics. So, after matrix we must understand the characteristics of reinforcing material.

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So, reinforcing material at this stage we will use the textile structure we are not going to discuss the particle reinforcement. So, textile structure are it is made of fibre. So, natural fibre, synthetic fibre characterization is required yarn characterization, spun yarn, filament yarn characterizations are required and also fabric characterizations are required that different structure 2D, 3D, unidirectional. So, woven, non-woven, knitted, braided. So, all this textile structure characterizations are required.

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Reinforcing Material Characterization

Fibre Characterization

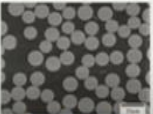
1) Fibre Identification:

The fibre (type) can be identified in the following ways


- i) Burning Test
 Example: cellulosic fibres smell like burring paper
- ii) Fibre-sectional view



Bast Fibre



Glass Fibre



Aramid Fibre
- iii) Solubility test
- iv) Element-analysis

First fibre, I will go quickly because we all this characterization we know. So, we are not going to spend much time. So, fibre identification by a burning test or by fibre solubility test. This we can identify the fibre what type of fibres are there, whether it is a natural fibre, synthetic fibre, cellulosic fibre, so that we can identify the fibre.

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Reinforcing Material Characterization

Fibre Characterization

2) Fibre Fineness
 Generally express in terms of mass per unit length such as
Denier = mass in gram per 9000 meter length
Tex= mass in gram per 1000 meter length
 In case of synthetic fibre with uniform circular cross-section, such as glass, carbon etc., the fibre fineness is also express in terms of fibre diameter

3) Fibre Length

➤ **Reinforcing fibre length should be more than the critical fibre length**
Critical fibre length (L_c): the length above which the fibres start contributing to the composite strength

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Then fibre fineness, we can measure the diameter of fibre or linear density in terms of Denier or Tex of fibre. So, Denier means mass in gram per 9000 meter length Denier and Tex. So, these are typically used for filament also, fibre length for say staple fibre short fibre, we can use the measure the fibre length and critical length as already been discussed, it is the length above which the fibre start contributing to the composite strength, because if the fibre length is short, very short it will not contribute to that extent, it will start coming out from the composite. Fibre length distribution which is an important characteristics we must know.

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Fibre Length

Fiber length can be expressed as,

- ✓ Staple length (STPL)
- ✓ Mean length (ML)
- ✓ Upper quartile length (UQL)
- ✓ Effective length (EL)
- ✓ Modal length (MoL)
- ✓ Span length (SL)
- ✓ Upper half-mean length (UHML)

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So, fibre length this can be expressed in terms of staple length, mean length, upper quartile length, effective length, modal length, span length, upper half mean length this we are not going to discuss, these are very basic fibre characteristics.

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Fibre Length

The parameters used to estimate **fiber length variations**

- ✓ Dispersion%
- ✓ Uniformity index (UI)
- ✓ Uniformity ratio (UR)
- ✓ Short fiber content (SFC), and
- ✓ Floating fiber index (FFI)



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So, length variation if we want to know it is very important for any composite manufacturing, we must know the fibre length variations. So, variation, fibre length variation is dispersion percentage, uniformity index, uniformity ratio, short fibre content, floating fibre index.

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Mean Length

The mean length of the fibers is defined as **“the average length of all fibers in the test specimen based on weight-length data”**.

• Mean length based on weight: $ML_{(w)} = \frac{w_1 l_1 + w_2 l_2 + w_3 l_3}{w_1 + w_2 + w_3} m$

It can also be calculated by **number-length data** as an alternative.

Mean length based on number: $ML_{(n)} = \frac{l_1 + l_2 + l_3}{3} mm$



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Mean length can be measured either mass based. So, weight length data, so mean length of fibre is defined as the average length of all fibres in the test specimen based on weight length data, this is the weight length suppose l_1 length, total weight of total mass of fibre say w_1, w_2, w_3 . 3 different fibres are mixed together and with l_1, l_2, l_3 lengths where their weight components are w_1, w_2, w_3 .

So, with this formula we can measure the weight based mean length. This is important for composite manufacturing to know the weight based length, mean length, because composite,

the fibre volume fraction of fibre weight fraction is based on total mass of fibre present. It can be measured the based on the number length. So, 1 1, 1 2, 1 3 for different number of fibres we can measure there are 3 fibres of 1 1, 1 2, 1 3 based we can measure the based on the length based number.

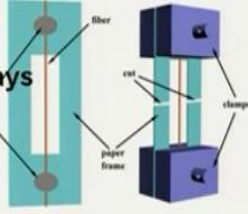
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Reinforcing Material Characterization

Fibre Characterization

5) Fibre tensile properties

- Fibre strength is measured in two ways
 - i) Bundle Strength (ASTM D1445)**
 Gauze length 3.2 mm
 bundle length: 15 mm
 bundle mass: mg/15 mm
 - ii) Single Fibre strength (ASTM D3822)**
 Gauze length: 25 mm for manmade fibres
 : 10 mm for natural fibres



Fibre	Density (g/cm ³)	Sp. Strength (MPa)	Sp. Modulus (GPa)
E-glass	2.5	800-1400	28
Flax	1.5	400-1200	19
Jute	1.48	300-800	17.5

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So, fibre tensile properties can also be measured by bundle strength in case of short fibre very short fibre. So, bundle strength can be measured by ASTM D1445 techniques, where gauze length is 3.2 millimeter, bundle length 15 millimeter, total bundle length and bundle mass per 15 millimeter is, milligram per 15 millimeter is measured. Single fibre strength can also be measured. Basically manmade fibre for filament we can measure here using 25 millimeter gauze length as far as ASTM D3822 and 10 millimeter for natural fibre.

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Reinforcing Material Characterization

Fibre Characterization

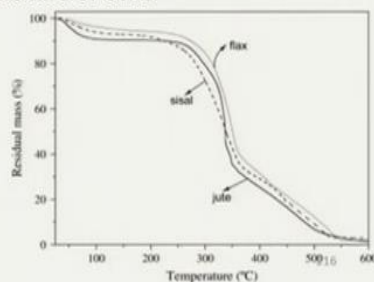
6) Moisture content

$$\text{Moisture Content} = \frac{W-D}{W} \times 100$$

Where D = Dry weight of the sample; W = Wet Weight of the sample

7) Thermo Gravimetric Analysis (TGA)

- TGA measures the amount and rate of weight change of a material with respect to temperature or time
- It gives an Idea about the thermal stability of the material
- Natural fibres like sisal, flax, jute etc. are thermally stable up to 260 °C



Moisture content can also be measured as has already been discussed. So, in case of matrix TGA thermo gravimetric analysis, it is basically amount and rate of weight change of material with respect to temperature or time. This is important because, we must know the thermal stability of fibre or textile structure. It gives an idea about the thermal stability of the material, natural fibre like jute, sisal, flax. These are the thermally stable fibres up to 260 degrees Celsius. Above that, they start deteriorating.

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Reinforcing Material Characterization

Fibre Characterization

8) Fibre surface characteristics

Surface roughness: Express in terms coefficient of friction.
Rougher the surface better will be the fibre-matrix bonding.

Surface energy:

- Contact angle - provide an indirect measurement of fiber surface free energy
- Measurement of contact angles on small diameter fibers is difficult
- A simple force balance is used to determine the contact angle by measuring the force induced by immersing the fiber into a liquid of known surface free energy
- Natural fibres (hydrophylic) are not compatible to hydrophobic matrices, hence they are subjected to some surface modification treatment

Fibre surface characteristics is important. So, surface roughness express in terms of friction. So, this fibre surface roughness is very important particularly for composite making. Because this is actually if the fibre coefficient of friction is high so, it will give better surface and better fibre matrix bonding particularly by mechanical keying, this we will discuss subsequently. Surface energy is also one of the surface characteristics.

Which is measured by contact angle, which provides an indirect measurement of fibre surface energy, contact angle is very important particularly for composite manufacturing, if the composite that reinforcing fibre has very high contact angle they do not weight quickly or easily then composite characteristics will deteriorate, natural fibre that hydrophilic fibres are not compatible to hydrophobic matrices. Hence, they are subjected to some surface modification treatment as has already been mentioned. In case of flax PP polymer, we have treated the flax fibre with MAgPP to change the surface characteristics fibre.

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Reinforcing Material Characterization

Yarn Characterization

Following parameters are characterized for reinforcing a yarn into the polymer matrix

- i) Constituent fibre parameters
- ii) Yarn type : filament or staple spun
- iii) Yarn twist
- iv) Yarn strength
- v) Size or coating material content



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Then we must know the yarn characteristics, that in the yarn what are the parameters of constituent fibre we must know before we use for composite, we must know whether the yarn is filament type or staple, accordingly we have to set our parameters yarn twist we must know yarn strength and size and coating material in the yarn. So, these are the tests required for the composite manufacturing.

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Reinforcing Material Characterization

Fabric Characterization

Following parameters are characterized for reinforcing a fabric into the polymer matrix

- i) Fabric thickness
- ii) Type of fabric: woven, nonwoven, braided etc.
- iii) Constituent fibre parameters
- iv) Constituent yarn parameters
- v) Fabric construction

Example: areal density, ends and picks per inch (for woven fabric), fabric weave pattern etc.

- vi) Fabric mechanical properties

Example: Tensile properties, bending rigidity etc.



- vii) Drape of fabric

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In fabric characteristics we must know the fabric thickness, type of fabric woven, nonwoven, braided, constituent fibre parameters, constituent yarn parameters, fabric construction, fabric mechanical properties, drape characteristics, we must know all these characteristics before we take or we select them those fabrics for composite manufacturing. Here will stop. In next segment we will discuss the composite testing. Till then thank you.