

Inspection and Quality Control in Manufacturing
Prof. Kaushik Pal
Department of Mechanical and Industrial Engineering
Indian institute of Technology Roorkee

Lecture – 05
Testing of Composite Materials

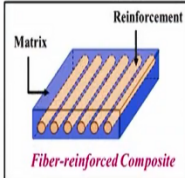
Hello my friends. So now we are going to start a new chapter on testing of composite materials. So before going to start this particular chapter as just let us know that what are composite materials? So basically, as the lay man I can say you that it's a nothing but the mixing of different materials and just to make a composite.

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What are Composite Materials ?

- Composite materials are multiphase materials (with measurable fraction of every phase), obtained by artificial combination of different materials, so as to attain properties that the individual components by themselves cannot attain.
- Composite materials are not the by-product of any chemical reaction between two or more of its constituents.

- There are two major components in a composite:
 - I. Reinforcement (Discontinuous/dispersed phase):
 - Material that provide strength to the matrix.
 - II. Matrix (Continuous phase):
 - Material that holds the reinforcement in place.



Matrix

+

Reinforcement

=

Composites

So generally, the composite materials are the multiphase materials with measurable fraction of every phase obtained by artificial combination of different materials as per our requirement generally we are wearing different __ (1.00) percent over there. So as to attain properties that the individual components by themselves cannot attain. Say suppose if I take a aluminum composites so simple aluminum or may be the virgin aluminum cannot gives that kind of properties but when I am adding maybe the silicon carbide or may be the alumina so it can give you a better properties.

Generally, the aluminum composites we are using for the aerospace applications. Composite materials are not by the by-product of any chemical reaction between two or more of its

constituents. So, there are two major components in a composite first one is called the reinforcement either it may be discontinuous or may be the dispersed phase. Materials that provide strength to the matrix and Matrix another one is called as a continuous phase. Materials that holds the reinforcement in place.


So generally, the matrix plus reinforcement is known as the composite. Now what are the role of matrix and reinforcing phases. Matrix in a composite material, matrix materials are the following functions.

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Role of Matrix and Reinforcing Phases:

Matrix: In a composite material, the matrix material serves the following functions:

- Holds the fibres together.
- Protects the fibres from environment.
- Distributes the loads evenly between fibres so that all fibres are subjected to the same amount of strain.
- Helps to avoid propagation of crack growth through the fibres by providing alternate failure path along the interface between the fibres and the matrix.
- Enhances transverse properties of a laminate.
- **Classification of Matrix Material:** Metal, Polymer, Ceramic.



Reinforcement:

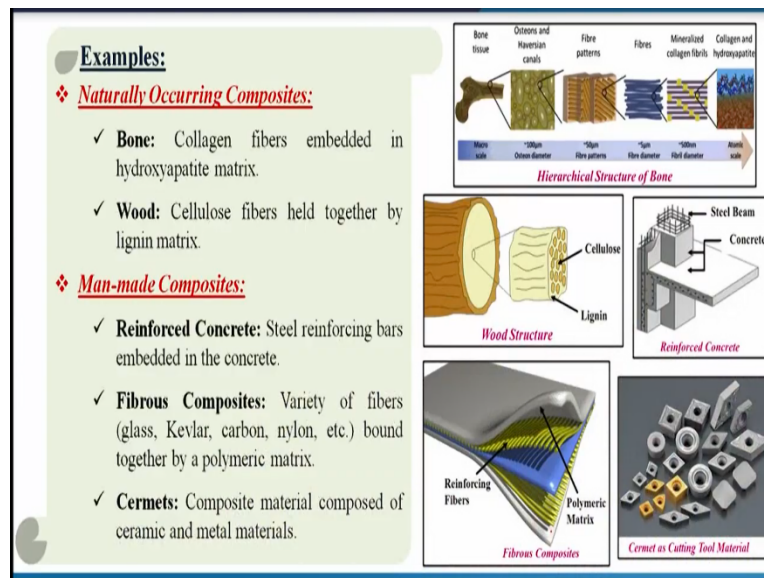
- The role of the reinforcement is to strengthen and stiffen the composite through prevention of matrix deformation by mechanical restraint.
- **Classification of Reinforcement:** Particles, Fibers (including whiskers).

First is called holds the fibers together not only the fibers if any kind of nano materials also nowadays we are using. So, it is holding any kind of additives are may be the fillers generally you can all it. Protects the fiber from the environments. Distributes the load evenly between the fibers so that all fibers are subjected to the same amount of strain. Helps to avoid propagation of crack growth through the fibers by providing alternate failure path along the interface between the fibers and the matrix.

Enhances transverse property of a laminate. Generally, the classification of matrix materials like metal, polymer, ceramic. And what are the reinforcement? the role of the reinforcement is to strengthen and stiffen the composite through prevention of matrix deformation by mechanical

restraint. Classification of reinforcement like as I told already particles, fibers including whiskers or may be other shapes.

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Examples generally for naturally occurring composites like Bone, collagen fibers embedded in hydroxyapatite matrix. Wood, Cellulose fibers held together by lignin matrix. So, from these particular image you can understand Man-made composites one is called the reinforced concrete; steel reinforcing bars embedded in the concrete. Generally, we are using it for the structural applications for making any kind of buildings structures, pillars.

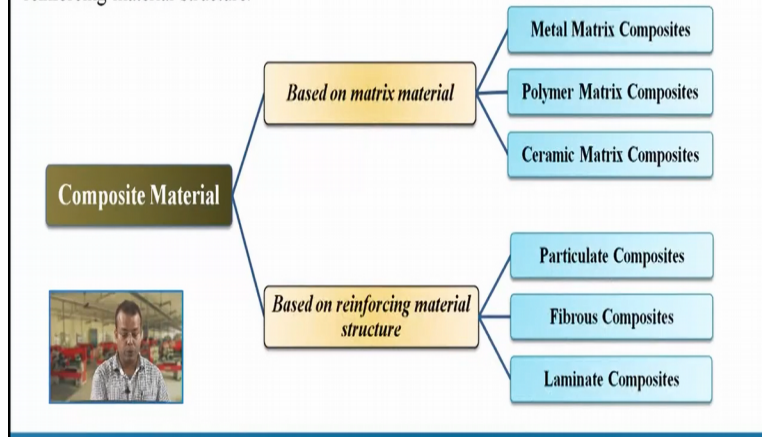
Fibers composites, variety if fibers glass, Kevlar, carbon, nylon etc. Nowadays we are using the Kevlar fiber for aero spare industries, we are making the rim liner or maybe some Boeing bound together by a polymeric matrix. And last one is the Cermets, it is nothing but the comb combinations of ceramics as well as the metals. Composite materials composed of ceramic and metal materials. So, it is known as th4e Cermets.

So, here we have given all the examples of the wood structure, then reinforce concrete, then fibrous composites it is some kind of sandwich like structure then cermet as cutting tool materials we are using it as an insert. Classification of composite materials.

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Classification of Composite Materials:

The composite materials are commonly classified based on the type of matrix material or reinforcing material structure.

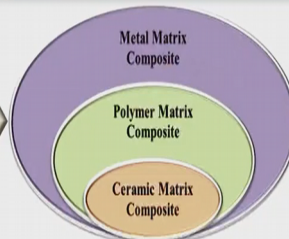


The composite materials are commonly classified based on the type of matrix material or reinforcing material structure. So Composite material based on matrix material, Metal Matrix Composite, Polymer Matrix Composite and Ceramic Matrix Composite. In short, we are calling it as MMCs, PMCs and the CMCs. Based on reinforcing material structure, it is Particulate Composite, Fibrous Composite and the Laminate Composite.

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Classification based on Matrix Material:

Based on the type of matrix material used to fabricate the composites, they can be divided into three categories:



1) Metal Matrix Composites:

- They consist of fibers or particles surrounded by a metallic matrix.
- Most metals and alloys can be used as matrices and they require reinforcement materials to be stable over a range of temperature and should be non-reactive too.
- Processing of a metal-matrix composite tends to be much more expensive than that of a polymer-matrix composite due to the high processing temperature required.

- Most common constituent materials used in this category are:

✓ Matrix Material: Al, Be, Mg, Ti, Fe, Ni, Co, and Ag

✓ Reinforcement Material: SiC, Al₂O₃, B₄C, TiC, TiB₂, W, steel fibers, graphite, and a number of other ceramics.



Now, Classification based on Matrix Material. Based on the type of matrix material used to fabricate the composites, they can be divided into three categories. As I told already Metal Matrix Composite generally we are calling it as MMCs, Polymer Matrix Composite we are calling it as a PMCs, and Ceramic Matrix Composite generally we are calling it as a CMCs.

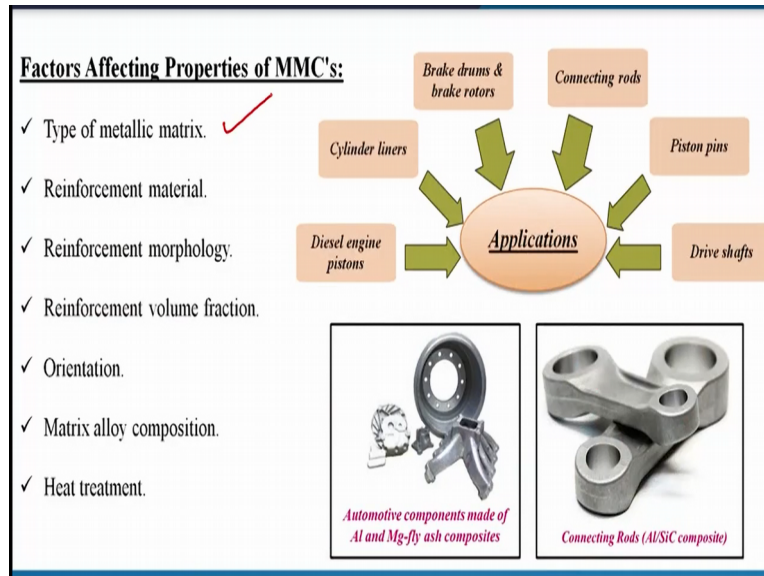
First, what is Metal Matrix Composites? They consist of fibers or particles surrounded by a metallic matrix.

Most metals and alloys can be used as matrices and they require reinforcement materials to be stable over a range of temperature and should be non-reactive too. Processing of a metal-matrix composite tends to be much more expensive than that of a polymer matrix composite due to the high processing temperature required. So, I can give you one best example in a we are using this composite from our ancient age is called the generally we are using it in village side for making the house and all these things.

We are taking the clay and then we are adding the straw over there. So now clay is the matrix and whatever the straw I am adding that is known the reinforcement. And we are using it for making the house or floats or this kind of things. So, it is coming from very old age. Most commons constituent materials used in this category are Matrix Material like Aluminum, Beryllium, Magnesium, Titanium, Iron, Nickle, Cobalt and Silver.

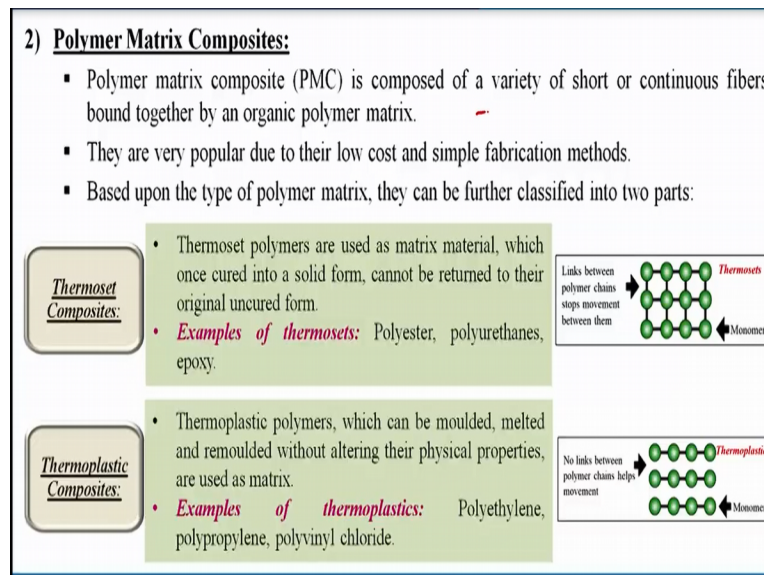
Reinforcement Material like Silicon Carbide, Al_2O_3 , B_4C , TiC, TiB₂, tungsten, steel fibers, graphite, and a number of other ceramics. That actually depends upon which type of property we need. If we need the hardest materials so we are going to add some tungsten over there or may be B_4C or maybe some other material like Silicon Carbides, if you need some materials which can hold the temperature, the high temperature if we need some material if we need the high electrical conductivities so as per our requirement, we are adding different types of fillers.

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Factors affecting properties of MMC, Types of metallic matrix, reinforcement material, reinforcement morphology, reinforcement volume fraction, orientation, matrix alloy composition and the heat treatment. So generally, what are the applications? We can use it for the diesel engine pistons, cylinder liners, brake drums and brake rotors, connecting rods, piston pins and the drive shafts. But here we have given a very limited number of examples but we are using it this metal matrix compositions for n number of applications.

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If you talk about the polymer matrix composites, generally, it is composed of a variety of short or continuous fibers bound together by an organic polymer matrix. They are very popular due to their low cost and simple fabrication methods. Based upon the type of polymer matrix, they can

be further classified into two parts. One is called the Thermoset composites and another one is called the Thermoplastic composites.

Thermoset polymers are used as matrix material, which once cured into a solid form, cannot be returned to their original uncured form. The best example is the natural rubber or maybe any kind of rubbery materials. So, if we give a particular shape to the particular rubber then after that we cannot return back to its original positions. Generally, polyester, polyurethanes, epoxy are the examples.

But when you are talking about the Thermoplastic composites, generally, different types of polymers. So, we are giving a particular shapes, then after maybe melting it or maybe some kind of load or pressure again we can give it another shapes also. So thermoplastic polymers which can be moulded, melted and remoulded without altering their physical properties, are used as matrix. What are the examples, polyethylene, polypropylene, polyvinyl chloride etc. What are the applications?

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<p><u>Applications:</u></p> <ul style="list-style-type: none">• Aerospace and military aircraft.• Automotive industry.• Construction.• Medical devices.	 <p><i>Airless Tyre</i> (glass fiber/PET composite)</p>	 <p><i>Inlet manifold</i> (polyamide composite reinforced by Al-Cu-Fe-B quastercrystals)</p>
<p><u>Drawbacks:</u></p> <ul style="list-style-type: none">• Environmental degradation.• Moisture absorption from environment causes swelling in the polymer as well as a decrease of T_g.• The moisture absorption increases at moderately high temperatures. These hydrothermal effects can lead to internal stresses in the presence of fibres in polymer composites.• A thermal mismatch between polymer and fibre may cause cracking or debonding at the interface.		

We are using it for the aerospace and military aircraft, automotive industry, construction, medical devices, and there are also so many. What are the drawbacks? Environmental degra.. degradation yes of course now a days you we are seeing that using of plastic carry bags is a really threat for us because it is not biodegradable it is making some kind of toxic gases or it is a one kind of

hazardous material, so recycling is also very difficult so that why it is not good for the environmental point of view.

Next absorption from environment causes swelling in the polymer as well as decrease of glass transition temperature generally T_g is known as the glass transition temperature. The moisture absorption increases at moderately high temperatures. These hydrothermal effects can lead to internal stresses in the presence of fibers in polymer composites. So, if you see we are using some kind of polymering materials, after certain time it can change its shape and size. A thermal mismatch between polymer and fiber may cause cracking or debonding at the interface itself.

So, if you see some old tires you can see after certain time the rubber is coming out from the steel bead or may be the nylon cord that is nothing but the debonding in between the matrix as rubber with the filler like the cords or may be the steel cords or may be the nylon fiber.


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3) Ceramic Matrix Composites:


- These materials are those in which one or more distinct ceramic phases are intentionally added to another, in order to enhance some property that is not possessed by the monolithic ceramic materials.
- Basic reinforcements which are included in the ceramic matrices are: *carbon, glasses, glass-ceramics, oxides and non-oxides*.
- Ceramic matrix composites overcome the major *demerits (such as brittle failure, low fracture toughness and limited thermal shock resistance)* of monolithic ceramics.
- Processing temperature for CMCs is extremely high compared to polymer or metal matrix composites which leads to a very difficult and expensive processing.

Applications:

- ✓ Heat shield systems for space vehicles.
- ✓ Components for high temperature gas turbines.
- ✓ Brake disks and brake system components.
- ✓ Slide bearings components.



Turbine rotor with blades made from CMCs



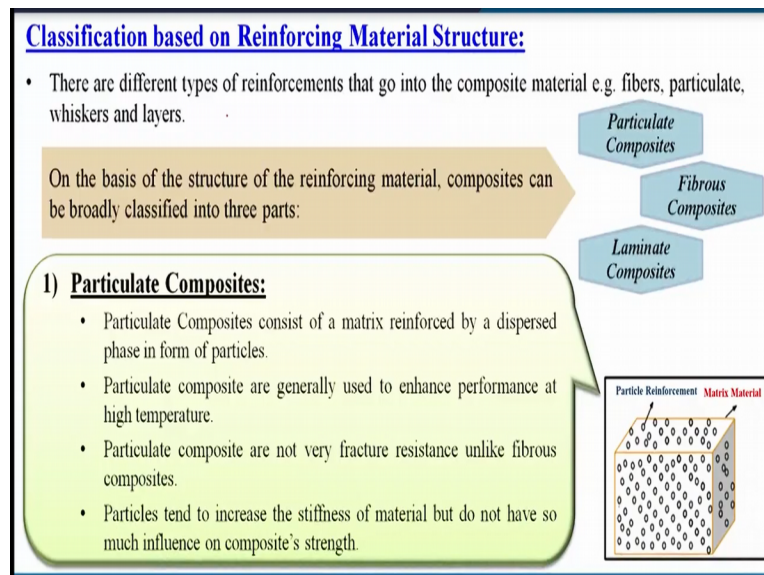
Engine nozzle made from CMCs

Next come to the Ceramic Matrix Composites. These materials are those in which one or more distinct ceramic phases are intentionally added to another, in order to enhance some property that is not possessed by the monolithic ceramic materials. So, a single ceramic or maybe a virgin ceramic cannot provide all the good properties so just to enhance the other properties we are adding the different type of reinforcement over there.

Basic reinforcements which are included in the ceramic matrices are carbon, glasses, glass ceramics, oxides and the non-oxides. Ceramic matrix composites overcome the major demerits such as brittle failure, low fracture toughness and limited thermal shock resistance of monolithic ceramics.

Processing temperature for CMCs is extremely high compared to polymer or metal matrix composites which leads to a very difficult and expensive processing. What are the applications? Heat shield systems for space vehicle, components for high temperature gas turbines, brake disks and the break system components and the slide bearing components that means where the operating temperature is more we can use this kind of ceramic matrix composite.

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Next classification based on reinforcement structure. There are different types of reinforcement that go into the composite material like fibers, particulate, whiskers and layers. So, on the basis of the structure of the reinforcing material, composites can be broadly classified into three parts. One is called the Particulate composites, fibrous composites and the laminate composites. What is Particulate composites? Particulate composite consists of a matrix reinforced by a dispersed phase in form of particles.

So small, small particles if it is in Nano meter range or maybe the size, we are calling it as a Nano particle or maybe the Nano composites. Sometimes it is better to tell that it is Nano

particle reinforced composites. Particulate composite are generally used to enhance performance at high temperature. Particulate composite are not very fracture resistance unlike fibrous composites.


Particles tend to increase the stiffness of material but do not have so much influence on composite's strength. So in this case matrix you can see it is having a particular shape and in that the particle reinforcement is there and that is homogeneously dispersed along the matrix.

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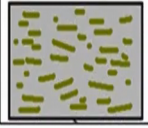
❖ Particulate composites can be further classified in to two parts on the basis of orientation of particles.

- a) Composites with random orientation of particles:
 - ✓ Orientation of particle is randomly distributed in all directions (Example: concrete).
- b) Composites with preferred orientation of particles:
 - ✓ Particle orientation is aligned to specific directions (Example: extruded plastics with reinforcement particles).

Random Orientation

A square box containing numerous small, yellow, rod-like particles scattered in various random orientations.

Preferred Orientation

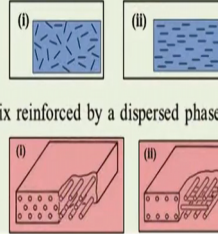
A square box containing numerous small, yellow, rod-like particles, all of which are aligned horizontally, demonstrating a preferred orientation.

Particulate composites can be further classified in to two parts on the basis of orientation of particles. One is called the Composites with random orientation of particles. Orientation of particle is randomly distributed in this particular case you can see in all directions. Example is the concrete. And composites with preferred orientation or particles. Particle orientation is aligned to specific directions. You can see all are in a particular direction. Example extruded plastics with reinforcement particles. Next called the fibrous composites.

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2) Fibrous Composites:

- They have fibres of reinforcing materials suspended in binding matrix.
- Unlike particles, a fibre has high length to diameter ratio and further its diameter may be close to its crystal size.
- These reinforcing phases may have random orientation where they are randomly distributed in all direction or may have preferred orientation where these are aligned to a specific direction.
- On the basis of fiber length, they can be further classified as:
 - a) **Short-fiber Reinforced Composites:** They consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers (length $< 100 \times$ diameter).
 - i. *Composites with Random Orientation of Fibers.*
 - ii. *Composites with Preferred Orientation of Fibers.*
 - b) **Long-fiber Reinforced Composites:** They consist of a matrix reinforced by a dispersed phase in form of continuous fibers.
 - i. *Unidirectional Orientation of Fibers.*
 - ii. *Bidirectional Orientation of Fibers.*



They have fibers of reinforcement materials suspended in binding matrix. Unlike particles, a fiber has high length to diameter ratio and further its diameter may be closer to its crystal size. These reinforcing phases may have random orientation where they are randomly distributed in all direction or may have preferred orientation where these are aligned to a specific direction. On the basis of fiber length, they can be further classified as short fiber reinforced composites.

Where generally, the matrix reinforced by a dispersed phase in form of discontinuous fibers. Generally, Length less than 100 into its diameter. Composites with random orientation fibers and composites with preferred orientation of fibers. And long fiber reinforced composites. They consist of a matrix reinforced by a dispersed phase in form of continuous fibers. Either it maybe the Unidirectional orientation of fibers or maybe the bidirectional orientation fibers. So, its like a ply. Next is called the Laminar Composites.

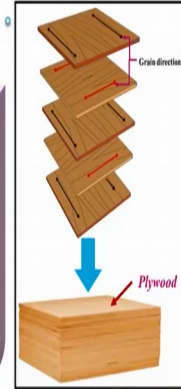
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3) Laminar Composites:

- A laminar composite is composed of several two dimensional sheets/layers with different fiber orientations cemented together.

Example: Adjacent wood sheets in plywood are aligned with the grain direction at right angles to each other.

- A laminar composite has relatively high strength in a number of directions in the two-dimensional plane.
- However, the strength in any given direction is, of course, lower than it would be if all the fibers were oriented in that direction.
- A laminar composite often uses different materials in its layers to gain the advantage of combining the particular properties of each.



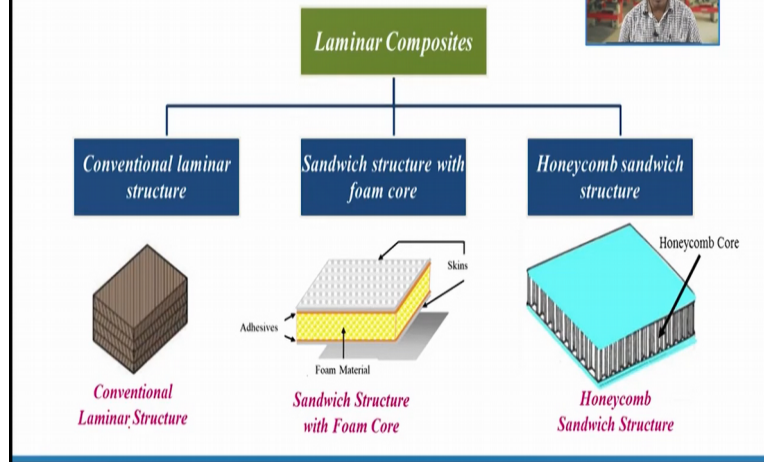
A laminar composite is composed of several two-dimensional sheets or layers with different fiber orientations cemented together. Say for example adjacent wood sheets in plywood are aligned with the grain direction at right angles to each other. You can see that all are present in the right angle. A laminar composite has relatively high strength in a number of directions in the two-dimensional plane.

However, the strength in any given direction is, of course, lower than it would be if all the fibers were oriented in that particular direction. A laminar composite often uses different materials in its layers to gain the advantage of combining the particular properties of each. So generally, we are calling it as stacking. Types of Laminar Composites.

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Types of Laminar Composites:

- There are three types of laminar composites generally found:



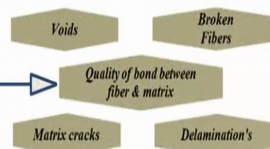
There are three types of laminar composites generally found. One is called the Conventional laminar structure so it looks like this, sandwich structure with foam core so we are having foam materials in between and so that's why it is called the sandwich structure and last one is called honeycomb sandwich structure so you can see that how reinforcement has been placed inside it.

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Testing of Composite Materials:

- Testing of material has got very much importance as it gives the required physical, mechanical and other properties for the use of that material in given application.
- The quality assessment of the composites used in the specimens to be tested must be done prior to the testing whenever possible.
- If the composite used in the specimens is of low quality with defects then the property measured are spurious.
- It can mislead the design and analysis procedure and result in a premature and catastrophic failure of the structure.
- Hence, the quality assessment of composites before it is used in specimen or actual structure fabrication is essential.

The following quality assessment in composites is essential:



Now come to the testing of this kind of Composite materials. So, testing of materials has got very much importance as it gives the required physical, mechanical and other properties for the use of that material in given application. The quality assessment of the composites used in the specimens to be tested must be done prior to the testing whenever possible. If the composite used in the specimens is of low quality with defects then the property measured are spurious.

It can mislead the design and analysis procedure and result in a premature and catastrophic failure of the structure. Hence, the quality assessment of composites before it is used in specimen or actual structure fabrication is very much essential. The following quality assessment in composition is essential like, voids, broken fibers, delamination's, matrix cracks and also the Quality of bond between the fiber and the matrix.

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Physical Characterization of Composite Materials:

- Physical characterization is determination of all the physical properties of a given material such as density, fiber volume fraction, void content, coefficient of thermal expansion, heat conduction coefficients, moisture content, etc.
- Physical properties of the composite play an important role in the measured mechanical properties.
- There is a direct dependence of mechanical properties on the physical properties.
 - For example, the mechanical properties are directly dependent on fibre volume fractions.

Here, we will consider the following physical property measurements:

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graph TD; A[Here, we will consider the following physical property measurements:] --> B((Density)); A --> C((Fiber volume fraction)); A --> D((Void content)); A --> E((Moisture content));
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Now, first come to physical characterization of composite materials. Physical characterization is determined of all the physical properties of a given material such as density, fiber volume fraction, void content, coefficient of thermal expansion, heat conducting coefficients, moisture content etc. Physical properties of the composite play an important role in the measured mechanical properties.

There is a direct dependence of mechanical properties on the physical properties. For example, the mechanical properties are directly dependent on fiber volume fractions. Here, we will consider the following physical property measurements like density, fiber volume fraction, void content and the last one is the moisture content.

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❖ Density:

- A material's density is defined as mass of the material per unit of its volume.
- It is basically a measurement of the degree of compactness of a substance.
- One of the most common use of density is in how different materials interact when mixed together. For example:
 - a) Wood floats in water because it has a lower density.
 - b) Anchor sinks in water because the metal has higher density.
 - c) Helium balloons float in air because the density of helium is lower than the air.
- The procedure for measuring the density of a composite material is same as that used for any other solid and is based on ASTM specification D792-86.

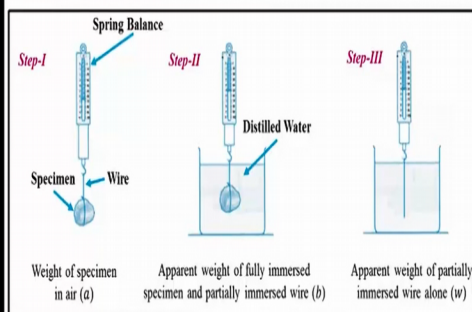
What is density? A material's density is defined as mass of the material per unit of its volume that logically we know. It is basically a measurement of the degree of compactness of a substance. One of the most common use of density is in how different materials interact when mixed together. For example, a wood floats in water because it has a lower density. Anchor sinks in water because the metal has higher density. And Helium balloons float in air because the density of helium is lower than the air.

The procedure for measuring the density of a composite material is same as that used for any other solid and is based on ASTM specification D792-86.

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Test Procedure:

- The procedure consists of the following steps:
 - i. Weigh specimen in air to the nearest 0.1 mg.
 - ii. Attach specimen to analytical balance with a thin wire and weigh while the specimen and portion of the wire are immersed in distilled water.
 - iii. Weigh wire alone, partially immersed up to the same point as in the previous step.



- The density of material at 23°C can be calculated as follows:

$$\rho = \frac{a}{a + w - b} \times (0.9975)$$

ρ = density, (in g/cm³)

a = weight of specimen in air,

b = apparent weight of fully immersed specimen and partially immersed wire,

w = apparent weight of partially immersed wire alone,

Density of distilled water at 23 °C (in g/cm³) =

0.9975 g/cm³

Test procedure. The test procedure consists of the following steps. Weigh specimen in air to the nearest 0.1 milligram. Attach specimen to analytical balance with a thin wire and while the specimen and portion of the wire are immersed in distilled water. So, step 1, then step 2 and then come to the step three. Weigh wire alone, partially immersed up to the same point as in the previous step. So first in this particular case, weight of specimen in air that is a, apparent weight of fully immersed specimen and partially immersed wire is b, apparent weight of partially immersed wire alone that is w.

So, the density of material at 23 degree centigrade can be calculates as follows. Rho is equal to a by a plus w minus b into 0.9975. where rho is the density generally in gram per centimeter cube; a is weight of specimen in air; b apparent weight of fully immersed specimen and partially immersed wire; w apparent weight of partially immersed wire alone. Density of distilled water at 23 degree centigrade in gram per centimeter cube is equal to 0.9975 gram per centimeter cube.


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❖ **Fiber Volume Fraction:**

- It is the percentage of fiber volume in the entire volume of a fiber reinforced composite material.
- A variety of methods exists for determination of fiber volume fraction, an important property of a composite.
- Generally, fiber volume fraction ranges from 30% to 65%. The lower end depend upon the significance of property contribution of the fibers whereas the upper depends upon the effective, defect-free packing.

Methods to Determine Fiber Volume Fractions:

- There are four main methods to determine fiber volume fraction present in a composite:
 1. Acid Digestion Method.
 2. Optical Microscopy method.
 3. Resin Burning-off Method.
 4. Gravimetric Relation Method.



Next fiber volume fraction. It is the percentage of fiber volume in the entire volume of a fiber reinforced composite material. A variety of material exists for determination of fiber volume fraction, an important property of a composite. Generally, fiber volume fraction ranges from 30% to 65%. The lower end depend upon the significance of property contribution of the fibers whereas the upper depends upon the effective, defect free packing.

Methods of determine fiber volume fractions. There are four main methods to determine fiber volume fraction present in a composite. Number one is Acid digestion method, number two is optical microscopy method, number three is the resin burning-off method and the last one is called the gravimetric relation method.

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1. Acid Digestion Method:

- This method involves the digestion of matrix material using an acid which does not attack the fibers.

Procedure:

- a. The matrix material is digested or dissolved by putting a measured volume of composite in an acid bath.
 - b. Following digestion, the remaining fibers are washed, dried, and weighed.
 - c. Knowing the initial weight of the composite specimen as well as the density of the composite, volume of the entire specimen can be calculated.
 - d. Thus, knowing the density and weight (step-2) of the fibers, the volume of fibers and the fiber volume fraction can be determined.
- One should be careful to choose the liquid for digestion such that the fibers are not digested.
 - Generally, hot nitric acid is used for carbon/epoxy composite.
 - The ASTM standard used for digestion method are D-3171-76 (1990) for polymeric composites and D3553-76 (1989) for metal matrix composites.

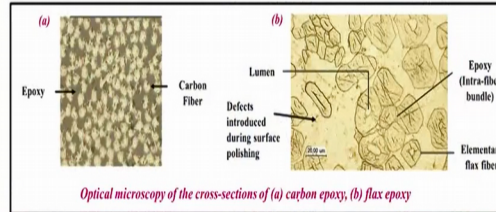
First acid digestion method. This method involves the digestion of matrix material using an acid which does not attack the fibers. Procedure. the matrix material is digese.... digested or dissolved by putting a measured volume of composite in an acid bath. Following digestion, the remaining fibers are washed, dried, and weighed. Knowing the initial weight of the composite specimen as well as the density of the composite, volume of the entire specimen can be calculated.

Thus, knowing the density and weight step 2 of the fibers, the volume of fibers and the fiber volume fraction can be determined. One should be careful to choose the liquid for digestion such that the fibers are not digested. Generally, hot nitric acid is used for carbon epoxy composite. The ASTM standard used for digestion method are D-1371-76 it was started in the year of 1990 for polymeric composites and D3553-76 it has been invented in the year of 1989 for metal matrix composites.

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2. Optical Microscopy based Techniques:

- It involves potting sectioned samples of the laminate, polished using standard metallographic techniques, and obtaining digital cross-sectional photomicrographs using an optical microscope and magnifications between 100 and 2500.
- Digital images may be recorded at a number of locations along the length and through-the-thickness of the laminate.
- Computer programs aid in the analysis of fiber ratio in the photomicrograph of the polished composite specimen.
- This method is preferred as a non-destructive approach to determining fiber volume fraction.



Next come to optical microscopy-based techniques. It involves potting sectioned samples of the laminate, polished using standard metallographic techniques, and obtaining digital cross-sectional photomicrographs using an optical microscope and magnifications between 100 and 2500. Digital images may be recorded at a number of locations along the length and through the thickness of the laminate.

Computer programs aid in the analysis of fiber ratio in the photomicrograph of the polished composite specimen. This method is preferred as a non-destructive approach to determining fiber volume fraction. So, you can see optical microscope of the cross sections of the carbon epoxy and the flax epoxy. So here we can count the number of fibers present in that particular area.

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3. Resin Burning-off Method:

- This method applies to composites with a reinforcement such as glass or ceramic that is not affected by high-temperature environments or a reinforcement such as carbon when the temperature can be adequately controlled to minimize degradation of the fiber during burn off.

Test Procedure:

- ✓ Heat up the composite to a temperature at which resin will melt and fibers remain stable.
- ✓ After burning off resin, weigh the fibers.
- ✓ Now, fiber volume fraction can be calculated from the initial weight of composite and fiber's weight.

4. Gravimetric Relation Method:

- This method determines the density of composites and then calculates the fiber volume fraction knowing the density of the fiber and the matrix.
- When it can be confirmed that the composite material has zero or negligible (less than 1%) porosity, the fiber volume fraction can be obtained from the densities of the composite and the constituents by the following gravimetric relation:

$$V_f = \frac{\rho_c - \rho_m}{\rho_f - \rho_m}$$

Where,
 V_f = Fiber volume fraction,
 ρ_f = Density of fiber,
 ρ_c = Density of composite,
 ρ_m = Density of matrix material

Then third one Resin burning-off method. This method applies to composites with a reinforcement such as glass or ceramic that is not affected by high-temperature environments or a reinforcement such as carbon when the temperature can be adequately controlled to minimize degradation of the fiber during burn off. Test procedure. Heat up the composite to a temperature at which resin will melt and fiber remain stable.

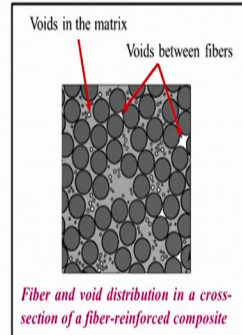
After burning off resin, weigh the fibers. Now, the fiber volume fraction can be calculated from the initial weight of composite and fiber's weight. So simple you have to separate the fibers from the composites. Next, gravimetric relation method. This method determines the density of composites and then calculates the fiber volume fraction knowing the density of the fiber and the matrix.

When it can be confirmed that the composite material has zero or negligible less than 1 percentage porosity, the fiber volume fraction can be obtained from the densities of the composition and the constituents by the following gravimetric relation. Capital V f equal to rho c minus rho m by rho f minus rho m. Where V f fiber volume fractions; rho f is the density of fiber; rho c is the density of composite and rho m is the density of matrix material.

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❖ Void Content:

- A void is a pore that remains unoccupied in a composite material.
- A void is typically the result of an imperfection from the processing of the material and is generally deemed undesirable.
- Because a void is a non-uniformity in a composite material, it can affect the mechanical properties and lifespan of the composite.
- Voids can act as a crack nucleation site as well as allow moisture to penetrate the composite and contribute to anisotropy of the composite.
- For aerospace applications, a void content of approximately 1% is appropriate for performance while other grades of composites can have between 3%-5% void content.



Next come to a void content. A void is a pore that remains unoccupied in a composite material. So that means it create some kind of hollow space inside the composites. A void is typically the result of an imperfection from the processing of the material and is generally deemed undesirable. Because a void is a non-uniformity in a composite material, it can affect the mechanical properties and lifespan of the composite itself.

Voids can act as a crack nucleation site as well as allow moisture to penetrate the composite and contribute to anisotropy of the composite. So from the void itself inside the matrix it can generates a crack or maybe the moisture can come over there and it can deposit at that particular point. For aerospace applications, a void content of approximately 1 percent is appropriate for performance while other grades of composites can have between 3 to 5 percent void content. So you can see these are all known as the void between fibers and these all are the voids in the matrix.

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Void Content Calculation:

- Consider a composite consisting of fiber and matrix. Take the following symbol notations:

$v_{c,f,m}$ = volume of composite, fiber, and matrix, respectively.

$\rho_{c,f,m}$ = density of composite, fiber, and matrix, respectively.

- For composites with a certain volume of voids v_v the volume fraction of the voids V_v is defined as:

$$V_v = \frac{v_v}{v_c}$$

- The total volume of composite (v_c) with voids is given by:

$$v_c = v_f + v_m + v_v \quad \dots \text{Eq. (1)}$$

- By definition of the experimental density ρ_{ce} of a composite, the actual volume of the composite is:

$$v_c = \frac{w_c}{\rho_{ce}} \quad \dots \text{Eq. (2)}$$

- By the definition of the theoretical density ρ_{ct} of the composite, the theoretical volume of the composite is

$$v_f + v_m = \frac{w_c}{\rho_{ct}} \quad \dots \text{Eq. (3)}$$

Void content calculations. Consider a composite consisting of fiber and matrix. Take the following symbol notations. Like v_c v_f v_m volume of composites v_c , v_f fibers, and v_m is for matrix. ρ_c ρ_f ρ_m is for density of composite, ρ_f for fiber, and ρ_m for the matrix. For composites with a certain volume of voids v_v the volume fraction of the voids V_v is defined as capital V_v equal to small v_v by small v_c . The total volume of composite small v_c with voids is given by small v_c equal to small v_f plus small v_m plus small v_v .

By definition of the experimental density ρ_{ce} of a composite, the actual volume of the composite is small v_c equal to small w_c by ρ_{ce} . By the definition of the theoretical density ρ_{ct} of the composite, the theoretical volume of the composite is small v_f plus small v_m equal to w_c by ρ_{ct} .

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- Substituting eq. 2 and 3 in 1, we have

$$\frac{w_c}{\rho_{ce}} = \frac{w_c}{\rho_{ct}} + v_v$$

- The volume of void is given by:

$$v_v = \frac{w_c}{\rho_{ce}} \left(\frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \right) = v_c \left(\frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \right)$$

- Now volume fraction of the voids is

$$V_v = \frac{v_v}{v_c} = \left(\frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \right)$$



In general, if void content < 1% => good composite
if void content > 5% => poor composite

Now substituting equations 2 and 3 in 1, we have small w_c by ρ_{ce} is equal to small w_c by ρ_{ct} plus small v_v . The volume of void is given by, small v_v is equal to small w_c by ρ_{ce} into ρ_{ct} minus ρ_{ce} by ρ_{ct} so we can get this one. Now volume fraction of the void is this one that is capital V_v is equal to small v_v by small v_c which is nothing but the ρ_{ct} minus ρ_{ce} by ρ_{ct} .

In general, if void content is less than 1 percent it is known as a very good composite if it is more than 5 percent then it is known as the bad or maybe the poor composite.

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❖ Moisture Content:

- Moisture content or water content is the quantity of water contained in a material.
- Composite materials when exposed to the environment or water absorb moisture.
- Absorption of the moisture results in the expansion and affects the degradation of the mechanical as well as thermal properties.
- Moisture content in a composite is given in terms of moisture by weight.

Procedure: To measure the moisture content, there are following steps as:

- A sample is weighed at the ambient conditions.
- Then, sample is dried and weighed again.
- The average amount of absorbed moisture in material can be calculated, as follows:

$$M, \% = \frac{W_i - W_o}{W_o} \times 100$$

$M, \% = \text{Percentage moisture content,}$
 $W_i = \text{Initial specimen mass,}$
 $W_o = \text{Oven-dry specimen mass.}$

- The difference in these two weights per unit weight of the dry sample gives the weight change due to moisture content.

Precautions:

- ❖ While drying, care should be taken to avoid excessively high drying temperatures and high thermal excursions that may induce thermal cracking, oxidation, or mass loss, or combinations of the three in the material.
- ❖ Wear clean, non-linting gloves when handling specimens.

Now, come to the moisture content. Moisture content or water content is quantity of water contained in a particular material. Composite materials when exposed to the environment or water absorb the moisture. Absorption of the moisture results in the expansion and affects the degradation of the mechanical as well as thermal properties. Moisture content in a composite is given in terms of moisture by weight. Procedure. To measure the moisture content, there are following steps as.

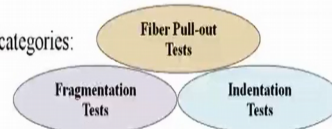
That number 1, a sample is weighed at the ambient conditions. Then, the sample is dried and weighed again. The average amount of absorbed moisture in material can be calculated, as follows. $M, \text{ percentage} = \frac{W_i - W_0}{W_0} \times 100$. So $M, \text{ percent, percentage of the moisture content}$; W_i initial specimen mass; W_0 oven-dry specimen mass. The difference in these two weighs per unit weight of the dry sample gives the weight change due to moisture content.

Now what are the precautions we have to take? While drying, care should be taken to avoid excessively high drying temperatures and high thermal excursions that may induce thermal cracking, oxidation, or mass loss, or combinations of the three in the material. So, we should not raise the temperature to high. Wear clean, non-linting gloves when handling specimens.

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Mechanical Behaviour Testing of Composite Materials:

- In an effort to understand the mechanical behaviour of composites at the microscopic level, a number of test techniques have been and continue to be developed.
- The principal aim of these tests is to evaluate the shear strength of fiber resin interface and any frictional effects that may be present.
- These micro-mechanical test methods fall into three categories:



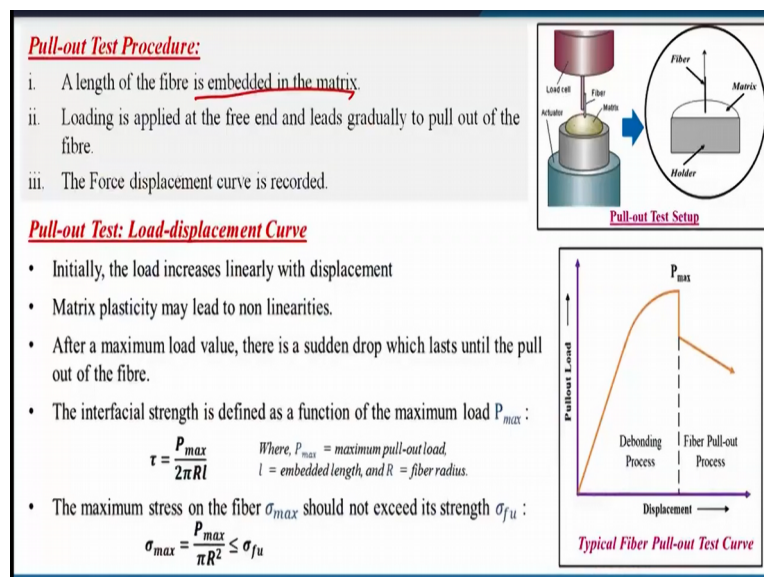
a) Fiber Pull-out Tests:

- The fiber/matrix interface plays an important role in controlling the macroscopic mechanical properties of fiber composites and it is often characterized by Fiber Pull-out Test.
- In this test, a single fiber is embedded in a thin sheet or film of matrix material and the force required to pull the fiber out of the film is determined.
- Interfacial shear strengths are calculated using pull-out forces for fibers with a range of embedded lengths.

Next mechanical behavior testing of composite material. In an effort to understand the mechanical behavior of composites at the microscopic level, a number of test techniques have been and continue to be developed. The principal aim of these tests is to evaluate the shear strength of fiber resin interface and any frictional effects that may be present. These micro mechanical test methods fall into three categories. One is called the Fiber pull out tests, fragmentation test and the indentation test.

What is Fiber pull out tests? The fiber matrix interface plays an important role in controlling the macroscopic mechanical properties of fiber composites and it is often characterized by fiber pull out test. In this test, a single fiber is embedded in a thin sheet or film of matrix material and the force is required to pull the fiber out of the film is determined. Interfacial shear strengths are calculated using pull out forces for fibers with a range of embedded lengths.

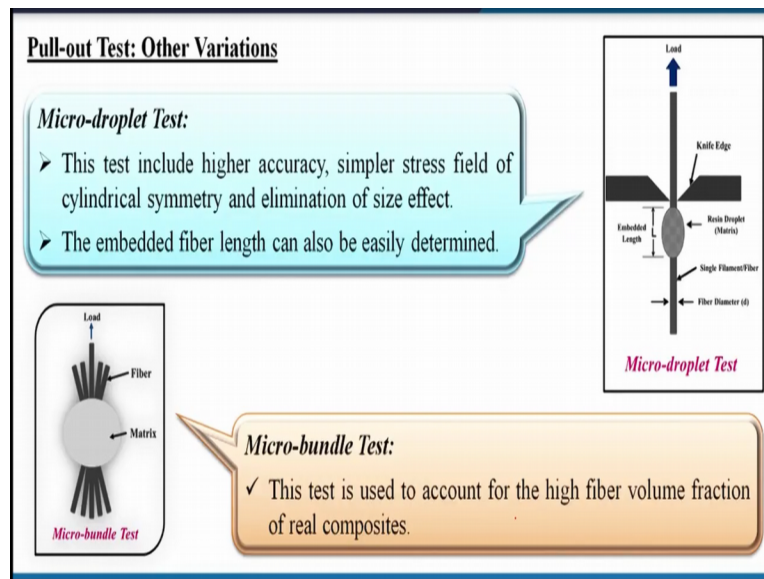
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Pull out test procedure. A length of the fiber is embedded in the matrix. So first, we are having the matrix, and then we are putting the fiber over there and just we are trying to pull out that fiber from that particular matrix. Loading is applied at the free end and leads gradually to pull out of the fiber. The force displacement curve is recorded. Pull out test; loading displacement curve. Initially, the load increases linearly with displacement. Matrix plasticity may lead to non-linearities. After a maximum load value, there is a sudden drop which lasts until the pull out of the fiber.

The interfacial strength is defined as a function of the maximum load P_{max} ; τ is equal to P_{max} by $2\pi Rl$, where P_{max} maximum pull out load; small l embedded length and capital R is the fiber radius. The maximum stress on the fiber σ_{max} should not exceed its strength σ_{fu} . So, σ_{max} is equal to P_{max} by πR^2 which is less than equal to σ_{fu} .

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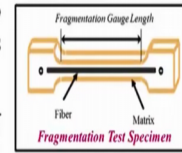


Now pull out test, other variations. One is called Micro droplet test. This test include higher accuracy, simpler stress field of cylindrical symmetry and elimination of size effect. The embedded fiber length can also be easily determined. Another on is called the Micro bundle test. So, this one. This test is used to account for the high fiber volume fraction of real composites.

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b) Fragmentation Tests:

- This test is developed from the early work of Kelly and Tyson, who investigated brittle tungsten fibers that broke into multiple segments in a copper matrix composite.
- Each test specimen for the fragmentation test consists of one fiber encapsulated in a chosen matrix and normally has a *dogbone* shape.



Fragmentation Test Procedure:

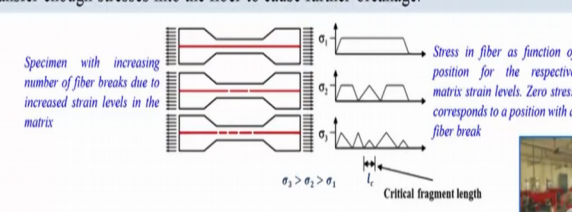
- ✓ Specimens are elongated in a tensile tester, which results in fiber breakage.
- ✓ Experiment is done under a light microscope so that fragmentation process can be observed in-situ.
- ✓ The fiber inside the resin breaks into increasingly smaller fragments at locations where the fiber's axial stress reaches its tensile strength.
- ✓ When the fiber breaks, tensile stress at the fracture location reduces to zero. Due to constant shear in the matrix, tensile stress in the fiber increases roughly linearly from its ends to a plateau in longer fragments.

Next, Fragmentation test. This test is developed from the early work of Kelly and Tyson, who investigated brittle tungsten fibers that broke into multiple segment in a copper matrix composite. Each test specimen for the fragmentation test consist of one fiber encapsulated in a chosen matrix and normally has a dog bone shape. So, you can see the shape, this shape is called the dog bone shape. Fragmentation test procedure. specimens are elongated in a tensile tester, which results in fiber breakage.

Experiment is done under a light microscope so that fragmentation process can be observed in-situ. The fiber inside the resin breaks into increasingly smaller fragments at locations where the fiber's axial stress reaches its tensile strength. When the fiber breaks, tensile stress at the fracture location reduces to zero. Due to constant shear in the matrix, tensile stress in the fiber increases roughly linearly from its ends to the plateau in longer fragments. So first, it will elongate, elongate, elongate and after certain time it will fell.

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✓ The higher the axial strain, the more fractures will be caused in the fiber, but at some level (known as saturation stage) the number of fragments will become constant as the fragment length is too short to transfer enough stresses into the fiber to cause further breakage.



Specimen with increasing number of fiber breaks due to increased strain levels in the matrix

Stress in fiber as function of position for the respective matrix strain levels. Zero stress corresponds to a position with a fiber break

$\sigma_3 > \sigma_2 > \sigma_1$

Critical fragment length l_c

Calculation of Interfacial Shear Strength:

- To derive interfacial strength, the stress field must be defined. For constant shear, the average shear strength at the interface is given by:

$$\tau = \frac{\sigma_{fu} d}{2l_c}$$

Where, σ_{fu} is the fiber strength at the critical length, d is the fiber diameter and l_c is the critical fragment length of the fiber (the average fragment length at saturation stage i.e. no more breaks occur when applying further strain to the specimen).

The higher the axial strain, the more fractures will be caused in the fiber, but at some level known as saturation stage, the number of fragments will become constant as the fragment length is too short to transfer enough stresses into the fiber to cause further breakage. So, in this particular case you can see the specimen with increasing number of fiber breaks due to increased strain levels in the matrix itself.

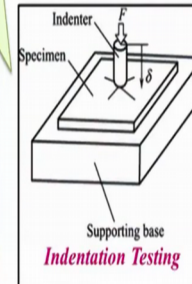
So, this is the small l_c is known as the critical fragment length. In this particular case σ_3 is more than σ_2 is more than σ_1 . So, stress in fiber as function of position for the respective matrix strain level. Zero stress corresponds to a position with a fiber break.

Calculation of interfacial shear strength. To derive interfacial strength, the stress field must be defined. For constant shear, the average shear strength at the interface is given by τ is equal to $\sigma_{fu} d$ by $2l_c$. where σ_{fu} is the fiber strength at a critical length, small d is the fiber diameter and small l_c is the critical fragment length of the fiber. The average fragment length at saturation state that is no more break occur when applying further strain to that particular specimen.

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c) Indentation Tests:

- This test gives knowledge about damage from concentrated out-of-plane forces, which is one of the major design concerns of many structures made of advanced composite laminates.
- It is performed on a grinded and polished surface.
- A flat, square composite plate is subjected to an out-of-plane, concentrated force by slowly pressing a hemispherical indenter into the surface.
- The damage resistance is quantified in terms of a critical contact force to cause a specific size and type of damage in the specimen.
- The damage response is a function of the test configuration; comparisons cannot be made between materials unless identical test configurations, test conditions, etc. are used.

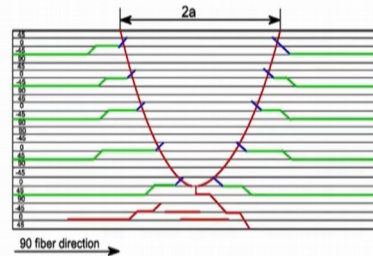


Next indentation tests. This test gives the knowledge about damage from concentrated out of place forces, which is one of the major design concerns of many structures made of advanced composite laminates. It is performed on a grinded and polished surface. A flat square composite plate is subjected to an out of plane, concentrated force by slowly pressing a hemispherical indenter into the surface.

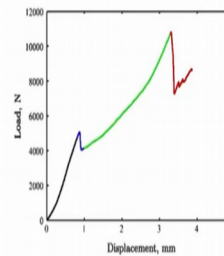
The damage resistance is quantified in terms of a critical contact force to cause a specific size and type of damage in the specimen. So simple, you have to make a specimen over there which is polished and clean and then after that through that indenter you are going to make a mark over there. The damage response is a function of test configuration; comparisons cannot be made between materials unless identical test configurations, test conditions, etc. are used.

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Indentation Damage in Composite Laminates:



Schematic representation of indentation damage



Related load-displacement curve

- These figures summarize the sequence of damage events observed in indentation tests along with its correlation with the load-displacement curve.
- The load displacement curve is plotted using four different colours, each colour corresponding to the damage mechanism represented in another figure of the same colour.

So generally, the Indentation damage in composite laminates is look like this schematic representation of indentation damage so it is for the 90 fiber directions and right-hand side related load displacement curve. These figures summarize the sequence of damage events observed in indentation test along with its correlation with the load displacement curve. The load displacement curve is plotted using four different colors, each color corresponding to the damage mechanism represented in another figure of the same color.

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Failure Modes in Micro-mechanical Test Methods:

- Specimen configuration and loading conditions determine failure modes that may be present.

Test	Specimen	Load	Failure Modes Present
Pull-out	Single fiber	Tension	Interracial failure (cohesive or adhesive), matrix yielding, fiber end surface loaded
Micro-droplet	Single fiber	Tension	Interfacial failure (cohesive or adhesive), matrix yielding, non-uniform loading
Micro-bundle	Single fiber in bundle	Tension	Interfacial failure (cohesive or adhesive), matrix failure, fiber failure
Fragmentation	Single fiber	Tension	Progressive damage accumulation by fiber failure, local matrix yielding, local matrix cracking, fiber-matrix debonding
Indentation	Single fiber in composite	Compression	Interfacial failure, fiber crushing

- Not all tests are appropriate for testing the properties of the three classes of composites and the primary limitation of using a particular test is the fabrication of the specimen.
 - ✓ **PMC**- Specimens for all the tests can be made.
 - ✓ **MMC**- These are currently restricted to fragmentation and indentation tests.
 - ✓ **CMC**- These are confined to the indentation test because of the low interfacial shear stresses in these systems.

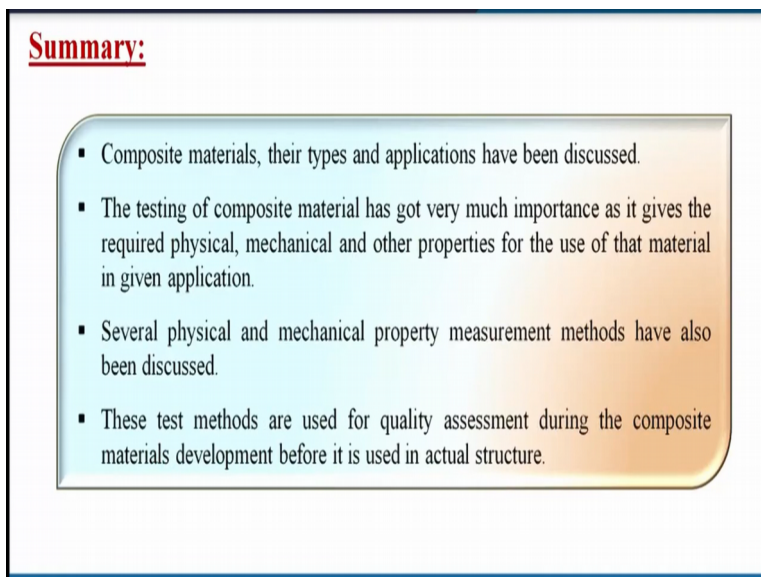
Then failure modes in micro mechanical test methods. Specimen configuration and loading conditions determine the failure modes that may be present. So generally, test is pull out specimen is the single fiber; load is the tension; inter interracial failure cohesive or adhesive

matrix yielding, fiber end surface loaded. So generally, the interfacial failure either it may be cohesive or adhesives, matrix yielding, fiber end surface loaded.

Say suppose for micro droplet it's about the single fiber tension interfacial failure, matrix yielding, non-uniform loading. Say suppose for indentations it is single fiber in composite, load is compression interfacial failure, or may be the fiber crushing. So, not all tests are appropriate for testing the properties of the three classes of composites and the preliminary limitation of using a particular test is the fabrication of the specimen. In PMC, Polymer matrix composite, the specimens for all the tests can be made.

For MMC, these are currently restricted to fragmentation and indentation tests. For Ceramic matrix composites these are confined to the indentation test because of the low interfacial shear stresses in these particular systems. Now we have come to the last slide of this particular lecture. So, in summary we can say, generally the composite materials, their types and applications we have broadly discussed in this particular lecture.

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Summary:

- Composite materials, their types and applications have been discussed.
- The testing of composite material has got very much importance as it gives the required physical, mechanical and other properties for the use of that material in given application.
- Several physical and mechanical property measurement methods have also been discussed.
- These test methods are used for quality assessment during the composite materials development before it is used in actual structure.

The testing of composite material has got very much importance as it gives the required physical, mechanical and other properties for the use of that material in given application. Several physical and mechanical property measurement methods have also been discussed in this particular

lecture. These test methods are used for quality assessment during the composite materials developed before it is actually used in applications. Thank you for your patience.