COMPOSITE MATERIALS

Definition
A composite material may be defined as an artificially prepared or natural multiphase material that exhibits a significant properties of the both the constituent material such as high strength, stiffness and high coefficient of thermal expansion in which the chemically dissimilar phases are separated by distinct interface.

Applications of composites
➢ automobiles industries: Automobile parts like components of engine, spray nozzle, mud guards, tires etc
➢ Aeronautical applications: structural components like wings, body & stabilizer and fuel of aircraft, rocket army missiles in military etc
➢ Marine applications: shaft, hulls, spars and other part of ships
➢ Safety equipment like helmets
➢ Sport equipment like tennis rockets, golf sticks, other safety equipment
➢ Communication Industry like preparation of antennae and electronic circuit boards

Constituents of Composites
Two essential constituents of composites are

1. Matrix phase : It is the continuous body constituent (Dispersion phase) which encloses the composite and gives its bulk form. It may be polymer, metal or ceramic material.

2. Dispersed phase : It is the Structural constituent (Dispersed phase) which determines internal structure of the composite and gives its bulk form. It may be Fiber, Particulate, Flakes or Whiskers

Types of Composites
Based on the dispersed phase in the given matrix of composite they are classified as

A. Fiber reinforced Composite B. Particulate Composite C. Structural Composite

A. Fiber reinforced Composite
• It is Consist of dispersed phase fiber and a continuous or dispersion phase polymer or metal or metal alloy with a bonding agent. The fiber can be employed in the form of continuous length, staples or whiskers.
• Such composites possess high specific strength, specific modulus, stiffness, corrosion resistance and lowers density
• The mechanical characteristics of FRC depend on the following
  1. Properties of fiber
2. Interfacial bond between fiber and matrix
3. Fiber length like longer gives continuous, shorter length gives discontinuous or random.
   Reinforcement efficiency of continuous is higher than short fibers.
4. Fiber orientation and concentration ie if it is orderly orientation and continuous it is highly anisotropic or discontinuous or random orientation

Some important types of Fiber reinforced composites are

1. **Glass fiber reinforced polymer composite**
   Fiber glass reinforced composites can be produced by properly incorporating the continuous or discontinuous glass fibers with in a plastic matrix. Polyesters are most commonly used matrix material. most recently nylons are used.
   It is the most popular fiber reinforcement material due to Easily available, easily fabricated, highly economical and which provides stiffness, strength, impact resistance and resistance to corrosion and chemicals. 
   limitations: they can fuse or melt at high temperatures
   Applications: Automobile parts, storage tanks, floorings and plastic pipes etc

2. **Carbon fiber reinforced polymer composites**
   Carbon fibers like (graphite, Graphenes or carbon nano tubes) dispersed in the polymer matrix.
   They provide excellent resistance to corrosion, lighter density, retention of desired properties even at elevated temperatures.
   limitations: High cost
   Applications: Structural components of air craft like wings and bodies, sport equipment, fishing rods etc.

3. **Alumina oxide/ carbon fiber reinforced metal composites:**
   Fibers of alumina or carbon dispersed in metal or metal alloy matrix which possess improved specific strength, stiffness, wear resistance, creep resistance and resistance to thermal distortion etc
   Ex:1. Fiber Al2O3 / carbon in a matrix metal alloy find applications in the preparation of components of automobile engines.
   2 . Fiber Al2O3 /W2O3 in a matrix of Ni or Co based alloy find applications in the preparation of components of turbine engines.

B. **Particulate Composite**

The solid particulates of metal oxides or carbides of varying size and form dispersed in metal, metal alloy, ceramic or polymer liquid matrix.
Particle reinforced composites are further classified into the following two types

1) Large -particulate composites  2) Dispersion strengthened composites

1) Large -particulate composites

Large particle composite used with all the three major types of materials, namely metals, polymer and ceramics.

Example : 1. concrete which is composed of cement matrix and particulates of sand and gravel.

2. Automobile tire in which Carbon black particles dispersed in rubber matrix

3. Ceramic - metal composites which are known as cermets. The most commonly used cermets are

Ex: 1. Al2O3 dispersed in Cr matrix possess good strength and good thermal shock resistance.

2. Tungsten carbide(WC) dispersed in Co matrix finds application in preparation of Valves, Spray nozzles and machine parts which require high surface hardness.

2) Dispersion strengthened composites

Very small particles of the range 10-100nm size are used in this which improve strength and hardness.

Metals and Metal alloys may be hardened and strengthened by the uniform dispersion of high volume percent of very hard and inert materials, the strength is achieved due to interactions between particle and dislocations within the matrix. example Thoria-dispersed Nickel

Precipitation hardening: The strength and hardness of some metal alloys may be improved by the formation of extremely small uniformly dispersed particles of a second phase within the original phase matrix with the help of appropriate heat treatment. This process called Precipitate hardening or Age hardening

C. Structural composites

Structural composites are prepared by Compressing the stacking of layers of fiber reinforce composites

These are of two types 1. Laminated composites 2. Sandwich composites.

1. Laminated Composites.

A Laminar composite consists of two-dimensional sheets or panels that have preferred high-strength direction, successive oriented fiber reinforced layers of these are stacked and then
cemented together in such a way that the orientation of the high strength varies with each successive layer
Example: Plywood, Copper bottom steel articles
2. Sandwich panels

These usually consist of two strong outer sheets called faces, separated by a layer of less dense material called core which is of lower strength and lower stiffness.
face materials: plywood, titanium, steel, and aluminum alloy
Core materials: Synthetic rubber, Foamed polymer

SMART MATERIALS

Smart polymers are materials that respond to small external stimuli like salt, temperature, UV radiations, pH, magnetic or electric field, or ionic factors etc. and shows promising applications in various fields like drug delivery, textile engineering, tissue engineering etc.

Smart polymers are Stimuli respond materials or Intelligent materials.

That characteristic features of these materials is their ability to respond to very slight changes in the surrounding environment which bring structural changes in their structure but also these transitions are reversible, i.e., these systems are able to recover their initial state when the stimuli ends.

These materials are biocompatible, strong, resilient, flexible, easy to sharpen and colour.

The responses of the materials with the above external stimuli are manifested as changes in one or more of the following -
Shape, surface characteristic, solubility, formation of an intricate molecular assembly, a sol gel transition and others.

Classification of smart polymers

Classification based on Physical change
1. Linear smart polymers, 2. Cross linked smart polymers, 3. Branched or Chain grafted smart polymers

Classification based on Stimuli
1. Physical stimuli (Temperature, light, ultra sound, mechanical, electrical or magnetic forces)
2. Chemical Stimuli (pH, and ionic strength)
3. Biological stimuli (enzymes and bimolecular compounds)

Applications of smart polymers
1. In drug delivery system
2. As reversible bio catalyst
3. In textile engineering
4. As Glucose sensor
5. In oil recovery
6. In bio - separation
7. In protein folding and in purification
8. Gene therapy
9. In tissue engineering
10. As molecular gates and Switches
Nanotechnology Definition
Nanotechnology is manipulation of matter in atomic, molecular or supramolecular scale in the size range of 1nm - 100nm at least one dimension in its shape.
Nanochemistry is the study of atoms or molecular interactions of the materials in the size range of 1nm-100nm.

Sol Gel Process
Sol Gel process is a chemical solution deposition technique may be described as Formation of oxide network through hydrolysis and polycondensation reactions of a molecular precursor in a liquid.
In this process dissolve the compound in a liquid in order to bring it back as a solid in a controlled manner.
Sol is stable dispersion of colloidal particles or polymers in a solvent
A Gel consists of a three dimensional continuous network, which encloses a liquid phase. In a colloidal gel the network built from agglomeration of colloidal particles.
Sol gel chemistry based on the hydrolysis and condensation of alkyl metal oxide M(OR)z such as Si(OEt)4 can be described as follows
\[
\text{MOR} + \text{H}_2\text{O} \rightarrow \text{MOH} + \text{ROH} \quad \text{(Hydrolysis)}
\]
\[
\text{MOH} + \text{ROM} \rightarrow \text{MOM} + \text{ROH} \quad \text{(Condensation)}
\]
Sol gel process can be characterized by series of distinct steps
step 1: Formation of different stable solutions of the alkoxide metal precursor (sol)
Step 2: Gelation resulting from the formation of an metal oxide or metal hydroxide bridged network by polycondensation which increase in viscosity of the solution
Step 3: Ageing of the gel, during which the polycondensation reaction continue untill the gel transformation into solid mass.
Step 4: Drying of the gel, when water and other volatile liquids are removed from the gel network (xerogel)
Step 5: Dehydration which is achieved by calcining the monolith at temp up to 800°C. (Aerogel)
Step 6: Densiification and decomposition of the gel at high temp,ie >800°C.(gel film)

Advantages
Low temperature, cheap technique. Avoids co precipitation & mixture of precursors can be taken and grown

Limitations
Controlling the growth of the particle, production rate is very slow.

Structure, properties and applications of fullerenes
- Fullerenes are the third allotropic form of carbon material after graphite and diamond.
- It is a spherical surface entirely built up from pentagons and hexagons.
They are called fullerenes because Fuller was renowned for his geodesic domes, those are based on hexagons and pentagons.

Fullerenes are composed entirely of carbon. They can be found in three different forms: spherical, elliptical and in the form of tubes.

Buckyball Structure

- The structure of the C\textsubscript{60} Buckyball is a combination of 12 pentagonal and 20 hexagonal rings, forming a spheroid shape with 60 vertices for 60 carbons.
- The structure of the molecule, which reveals how the pentagonal rings sit at the vertices of an icosahedron such that no two pentagonal rings are next to each other.
- In these each carbon atom is bonded to three others and is sp\textsuperscript{2} hybridized.
- The average C-C bond distance measured using nuclear magnetic resonance (NMR) is 1.44 Å. A diameter of 7.09 Å is calculated for the C\textsubscript{60} based on the fact that the C-C distance is equal to 1.40 Å for the hexagon bonds and 1.46 Å for the pentagonal bonds length.
- As hexagon bonds has shorter bond length which consist of 'double bond'
- C\textsubscript{60} is not "superaromatic" as it tends to avoid double bonds in the pentagonal rings, resulting in poor electron delocalisation.
- As a result, C\textsubscript{60} behaves like an electron deficient alkenes and reacts readily with electron rich species.
- The geodesic and electronic bonding factors in the structure account for the stability of the molecule.

Synthesis of Fullerenes

- When the graphite electrodes contact arcs passing alternating or direct current through them in an atmosphere of helium in approximately 200 torr. The evaporated graphite takes the form of soot, which is dissolved in a nonpolar solvent.
The solvent is dried away and the C60 and C70 fullerenes can be separated from the residue. Optimal current, helium pressure and flow rate leads to yields of up to 70% of C60 and 15% of C70 with this method.

Applications

Fullerenes can be used as:
- organic photovoltaics (OPV),
- powerful antioxidants, reacting readily and at a high rate with free radicals which are often the cause of cell damage or death.
- catalysts,
- in water purification and biohazard protection
- Hydrogen gas storage
- Sensors
- Preparation of composites

Carbon nanotubes
- These are cylindrical fullerenes, hollow tubes of very small dimensions.
- These are two types: 1. Single wall nano tube (SWNT) 2. Multi Wall Nano Tube (MWNT)
- SWNT consist only of a single graphene sheet with atomic layer in thickness
- MWNT is formed from 2 to several graphene sheets arranged concentrically into tube structure
- These tubes have either closed ends or open ends. They show excellent properties like high tensile strength, electrical conductivity, ductility and chemical reactivity etc.
- These show applications in electronic industry, drug delivery, composites etc.
ELECTRON MICROSCOPY:

Electron microscopes are scientific instruments that use a beam of energetic electrons to examine objects on a very fine scale. Earlier Light microscopes were used to examine the objects but these magnifies only to limited extent, i.e up to 1000 X magnification. The scientific desire to see the fine details of the interior structure of organic cells demands 10,000 X plus magnification which was not possible by current optical microscopes.

Electron Microscopy is an imaging tool with much better imaging resolution as compared to Optical Microscopy.

In electron microscopy a beam of highly accelerated electrons are focused on the surface of materials, and scan materials on a fine and very small scale, thus creating the image.

There are two main types of electron microscopes based on their interaction of beam of electrons with the sample

1. Scanning Electron Microscope (SEM) detects scattered electrons emitted from the surface.
2. Transmission Electron Microscope (TEM) detects transmitted electrons.

Scanning Electron Microscope (SEM)

A Scanning electron microscope is a type of electron microscope that images a sample by scanning it with a high energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain qualitative information about the samples surface topography, morphology, composition, orientation of grains, crystallographic information other properties of a material.

- Topography: The surface feature of an object and its texture, smoothness or roughness
- Morphology: The shape and size of the particles
- Composition: Elements and compounds that constitute the material, while
- Crystallography: The arrangement of atoms in the materials.

Principle

The SEM instrument is based on the principle that the primary electrons emitted from the electron gun are scanned across the sample surface by scanning coils in a raster pattern. Once the primary electron beam hits the sample surface, then the impinging electrons emit a variety of signals such as secondary electrons (SEs), backscattered electrons (BSEs), photons (X-rays).
• Secondary electrons indicate sample morphology and topography.
• Backscattered electrons are used for demonstrating the composition.
• X-rays provide chemical information i.e elemental analysis of the sample.

The signals are gathered by electron collectors (detectors), which are then manipulated by the computer to form the required image.

**Instrumentation**

SEM is an electronic and optical system which consists of the following components:

(i) Electron gun
(ii) Vacuum
(iii) Column: condenser lens, scanning coil, objective lens, stigmator, sample holder and detector

In principle, first the gun emits the electron beam which is held within a vacuum which follows a vertical travel path through electromagnetic fields and lenses. The electron beam is focused by objective lens on the specimen. Then the focused beam scans over a specific area of the specimen surface where the focused beam is raster across the surface of the materials with help of deflector coils, which is controlled by the scan generator. Magnification controls the size of the raster pattern. The changes in magnification change the size of the raster area on the sample. When the electron beam hits the material, this strike produces a huge number of signals, i.e. electrons and X-rays are emitted from the specimen. These signals are detected by the detector.
Advantages

**Applications of SEM**
1. To know information about topography, morphology and composition of materials
2. To examine microscopic objects like bacteria and viruses
3. To know the crystalline structure of samples
4. SEM is used in industries like microelectronics, semiconductor, food processing to examine the surfaces
5. To know the shape and size of the particles in cosmetic industry

**Transmission Electron Microscope (TEM)**

Transmission electron microscopy is a technique in which a beam of electrons is transmitted through a specimen to form an image. The specimen is most often an ultrathin section less than 100 nm thick or a suspension on a grid.

Image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen. The image is then magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic film.

TEM offers invaluable information on the inner structure of the sample, such as
- crystal structure,
- morphology and
- stress state information,

**Instrumentation:**

In a conventional transmission electron microscope, a thin specimen is irradiated with an electron beam of uniform current density. Electrons are emitted from the electron gun and illuminate the specimen through a two or three stage condenser lens system. Objective lens provides the formation of either image or diffraction pattern of the specimen. The electron intensity distribution behind the specimen is magnified with a three or four stage lens system and viewed on a fluorescent screen. The image can be recorded by direct exposure of a photographic emulsion or an image plate or digitally by a CCD camera.
Applications of TEM

1. TEM can be used to study the growth of layers, their composition and defects in semiconductors.
2. High resolution can be used to analyze the quality, shape, size and density of quantum wells, wires and dots.
3. TEM provide topographical, morphological, stress, compositional and crystalline information.
4. TEM is used in cancer research, virology, and materials science as well as pollution.

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<tr>
<th>S.N</th>
<th>SEM</th>
<th>TEM</th>
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<tr>
<td>1</td>
<td>Detects scattered electrons emitted from surface of the sample</td>
<td>Detects transmitted electrons through the sample</td>
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<td>2</td>
<td>Sem provides topography and morphology</td>
<td>Tem provides Internal composition, stresses or magnetic domains</td>
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<td>3</td>
<td>Sample preparation is easy specimen may be at any thickness</td>
<td>Sample thickness should be &lt; 100nm</td>
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<td>4</td>
<td>Accelerate voltage range from 10-40 kv</td>
<td>&gt; 100kv</td>
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<td>5</td>
<td>Low resolution</td>
<td>High resolution</td>
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Comparison of OM, TEM and SEM

Principal features of an optical microscope, a transmission electron microscope and a scanning electron microscope, drawn to emphasize the similarities of overall design.