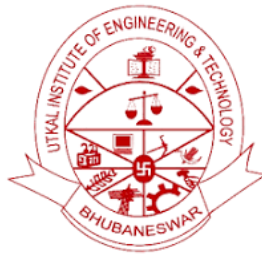


LECTURE NOTES  
ON  
COMPOSITE MATERIAL  
DIPLOMA 6<sup>TH</sup> SEMESTER

COMPILED BY  
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# Composite Materials

**CATEGORY : PE 2**

**CREDIT : 3**

## **MODULE I**

**(8 HOURS)**

Introduction: definitions and classifications; natural composites; role of matrix and reinforcement; factors which determine properties; the benefits of composites.

Reinforcements and the reinforcement matrix interface: natural fibers; synthetic organic fibers – aramid, polyethylene; and synthetic inorganic fibers – glass, alumina, boron, carbon, silicon based fibers; particulate and whisker reinforcements, reinforcement-matrix interface – wettability, interfacial bonding, methods for measuring bond strength.

## **LECTURE CHART**

<b>LECTURE NO.</b>	<b>TOPIC COVERED</b>
01	Introduction: Definition
02	Classification, natural composites; role of matrix and reinforcement; factors which determine properties
03	The benefits of composites. Reinforcements and the reinforcement matrix interface
04	Natural fibers; synthetic organic fibers – aramid, polyethylene
05	Synthetic inorganic fibers – glass, alumina, boron, carbon, silicon based fibers; particulate and whisker reinforcements
06	Reinforcement-matrix interface – wettability, interfacial bonding, methods for measuring bond strength.

# COMPOSITE MATERIALS

## Introduction : Definition

Composite materials create a new era in today's world. This is not a new term but its properties and applications make it a topic of discussion. Composites are useful in many industries such as aerospace, automobile, chemical as well as in structural and domestic purposes, electrical, production of amusing instruments.

History:

The spur to the rapid expansion over the last few decades was the development in the UK of carbon fibres and in the USA of boron fibres in the early 1960s. These new fibres, which have high elastic constants, gave a significant increase in the stiffness of composites over the well established glass fibre containing materials, and hence made possible a wide range of applications for composites.

One of the key factors was the very high strength-to-weight ratios possessed by new composites. Early employment in aviation has led, in more recent times, to wide use in other areas such as leisure and sports industry. Similarly the development of high strength SiC and Al<sub>2</sub>O<sub>3</sub> fibres which maintain their properties at elevated temperature initiated much of the current interest in composites based on metal and ceramic.

Composite materials can be studied from a no. of different viewpoints each of which requires a different kind of expertise. Thus, the development of a composite material to resist a corrosive environment is primarily within the field of materials science and chemistry. Study of composites is truly interdisciplinary.

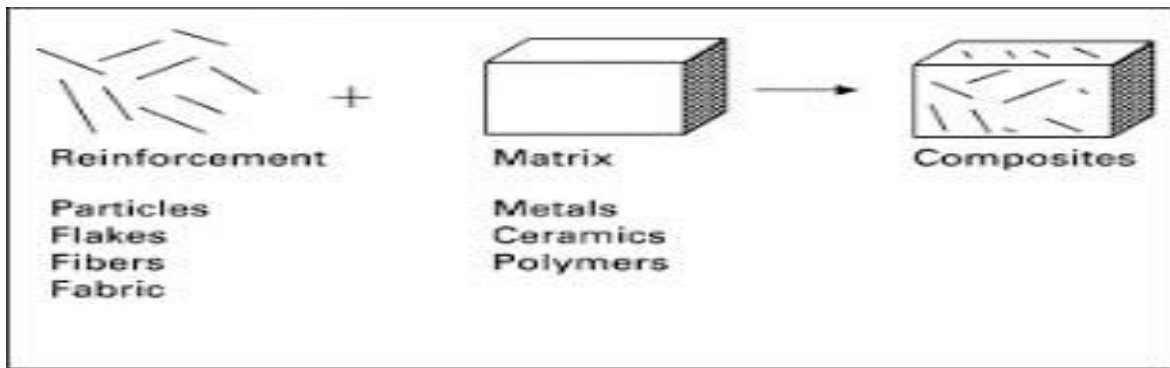
## Definition:

Composite can be defined as the mixture of two or more different constituents or phases when combined macroscopically to yield a beneficial product. But this definition is not sufficient and three other criteria have to be satisfied before a material can be said to be a composite.

1. Both constituents have to be present in a reasonable proportions say greater than 5%.
2. It is only when the constituent phases have different properties and hence the composite properties are noticeably different from properties of constituents.
3. A manmade composite is usually produced by intimately mixing and combining constituents by various means.

Thus an **alloy** which has a two phase microstructure which is produced during solidification from a homogeneous melt, or by a subsequent heat treatment whilst a solid, is **not** normally classified as a **composite**.

Composites have two (or more) chemically distinct phases on a microscopic scale, separated by distinct interface, and it is important to be able to specify these constituents.



( Fig. 01)

Difference between Matrix and reinforcement:

MATRIX	REINFORCEMENT
<ul style="list-style-type: none"> <li>This constituent is continuous and in greater quantity.</li> <li>Based up on matrix composite is of 3 types. PMC,CMC,MMC.</li> <li>Matrix transfers the load.</li> <li>It protects individual fibre from surface damage due to abrasion and oxidation.</li> <li>Example: alumina, aluminium, epoxy, polyester etc.</li> </ul>	<ul style="list-style-type: none"> <li>This may be continuous or discontinuous.</li> <li>Based up on reinforcement type it can be fibre reinforced or particulate reinforced.</li> <li>Reinforcement bears the load.</li> <li>The reinforcing phase provides strength, stiffness. In most cases these are stronger and harder than matrix.</li> <li>Example: carbon, aramid, nylon,jute etc.</li> </ul>

## CLASSIFICATION:

Classification of composite on the basis of reinforcements used.

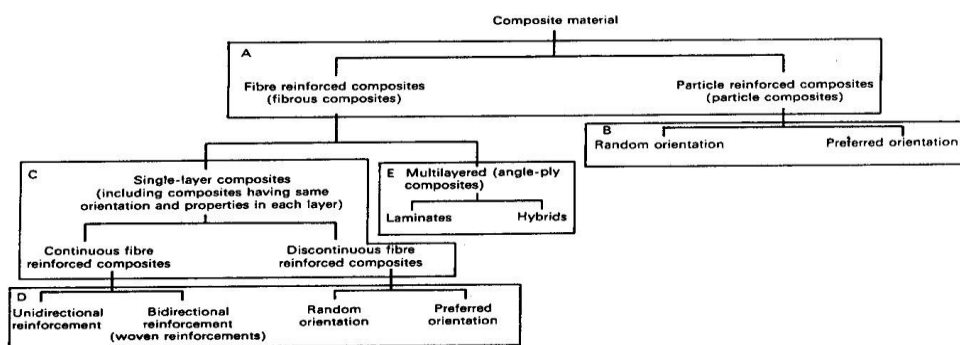


Figure 1.3 Classification of composite materials.

(Fig. 02)

The second constituent is referred to as the *reinforcing phase*, or *reinforcement*, as it enhances or reinforces the mechanical properties of the matrix. In most cases the reinforcement is harder, stronger and stiffer than the matrix, although there are some exceptions; for example, ductile metal reinforcement in a ceramic matrix and rubberlike reinforcement in a brittle polymer matrix. At least one of the dimensions of the reinforcement is small, say less than 500  $\mu\text{m}$  and sometimes only of the order of a micron. The geometry of the reinforcing phase is one of the major parameters in determining the effectiveness of the reinforcement; in other words, the mechanical properties of composites are a function of the shape and dimensions of the reinforcement. We usually describe the reinforcement as being either *fibrous* or *particulate*. Figure 1.3 represents a commonly employed classification scheme for composite materials which utilizes this designation for the reinforcement (Figure 1.3, block A).

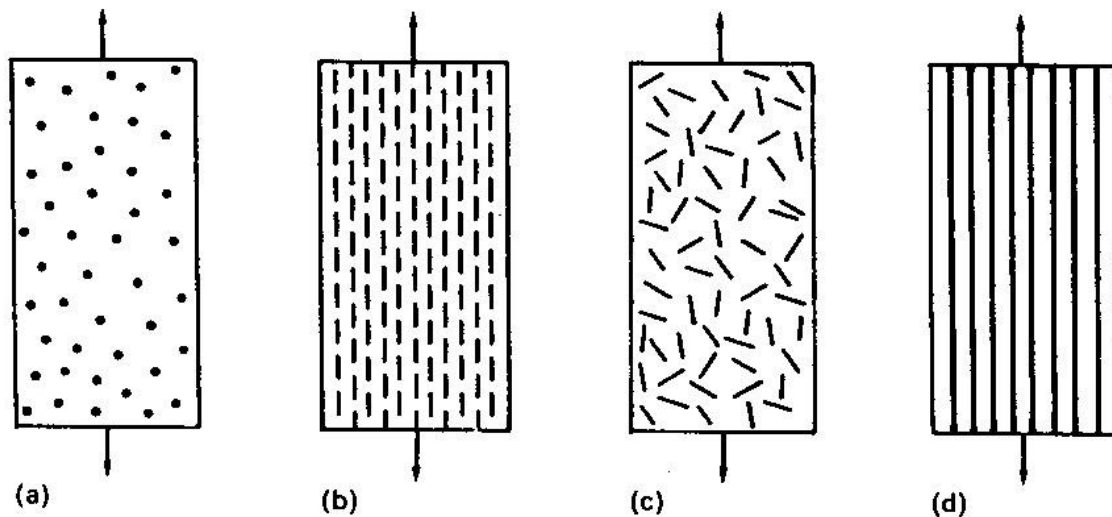
Particulate reinforcements have dimensions that are approximately equal in all directions. The shape of the reinforcing particles may be spherical, cubic, platelet or any regular or irregular geometry. The composite illustrated in the micrograph of Figure 1.2(b) has angular particles of about 10  $\mu\text{m}$  as the reinforcement. The arrangement of the particulate reinforcement may be *random* or with a *preferred orientation*, and this characteristic is also used as a part of the classification scheme (block B). In the majority of particulate reinforced composites the orientation of the particles is considered, for practical purposes, to be random (Figure 1.4(a)).

A fibrous reinforcement is characterized by its length being much greater than its cross-sectional dimension. However, the ratio of length to the cross-sectional dimension, known as the aspect ratio, can vary considerably. In single-layer composites long fibres with high aspect ratios give what are called *continuous* fibre reinforced composites, whereas *discontinuous* fibre composites are fabricated using short fibres of low aspect ratio (block C). The orientation of the discontinuous fibres may be random or preferred (Figures 1.4 (b) and (c)). The frequently encountered preferred orientation in the case of a continuous fibre composite (Figure 1.4(d)) is termed *unidirectional* and the corresponding random situation can be approximated to by *bidirectional* woven reinforcement (Figure 1.3, block D).

*Multilayered* composites are another category of fibre reinforced composites. These are classified as either laminates or hybrids (block E). *Laminates* are sheet constructions which are made by stacking layers (also called plies or laminae and usually unidirectional) in a specified sequence. A typical laminate may have between 4 to 40 layers and the fibre orientation changes

from layer to layer in a regular manner through the thickness of the laminate, e.g., a  $0/90^\circ$  stacking sequence results in a *cross ply* composite.

*Hybrids* are usually multilayered composites with mixed fibres and are becoming commonplace. The fibres may be mixed in a ply or layer by layer and these composites are designed to benefit from the different properties of the fibres employed. For example, a mixture of glass and carbon fibres incorporated into a polymer matrix gives a relatively inexpensive composite, owing to the low cost of glass fibres, but with mechanical properties enhanced by the excellent stiffness of carbon. Some hybrids have a mixture of fibrous and particulate reinforcement.



**Figure 1.4** Examples of composites: (a) particulate, random; (b) discontinuous fibres, unidirectional; (c) discontinuous fibres, random; (d) continuous fibres, unidirectional.

Based on matrix used composite can be classified as three types.

1. Metal matrix composite (MMC) : when matrix is a metal.
2. Ceramic matrix composite (CMC) : when matrix is a ceramic.
3. Polymer matrix composite (PMC) : when matrix is a polymer.

## NATURAL COMPOSITES

Wood is an interesting example of a natural fibre composite; the longitudinal hollow cells of wood are made up of layers of spirally wound *cellulose fibres* with varying spiral angle, bonded together with lignin during the growth of the tree. A model of good design with a composite material is the medieval longbow. In the medieval yew longbow the tensile surface was made by the bowyer from the resilient white sapwood with the bulk of the cross-section from the dark heartwood which is strong in compression. The result was a one-piece self-composite product at two levels, of remarkable strength, flexibility and stiffness.

Bone is a composite material composed primarily of organic fibres, small inorganic crystals, water and fats. The proportions of these components will vary with the type of bone, animal species and age but typically about 35% of the dry, fat-free weight of bone is the organic fibre, *collagen*.

Collagen is a fibrous protein and located around the outside of the collagen fibres are small rod-like crystals of *hydroxyapatite* with dimensions of the order of  $5 \times 5 \times 50$  nm. Therefore at this microscopic level we have a hydroxyapatite-reinforced collagen composite and this may be considered to be the basic 'building block' for bone. The different types of bone are simply composites on a macroscopic scale resulting from different arrangements of the collagen fibres; in woven bone the fibres are arranged more or less randomly whereas in lamellar bone the fibres are orientated locally in layers to form lamellae.

A long bone, such as the femur, has an outer shell of high density, low fat content bone which is termed *compact* or *cortical* bone. Cortical bone has good mechanical properties with flexural strength in the range 46–156 MPa

## ROLE OF MATRIX AND REINFORCEMENT:

Although there are many examples of composites employed mainly for their physical properties, such as the superconducting composites consisting of continuous  $\text{Nb}_3\text{Sn}$  fibres in a bronze matrix, most composites are designed to exploit an improvement in mechanical properties. Even for composites produced essentially for their physical properties, the mechanical properties can play an important role during component manufacture and service. For these reasons, in this section on the matrix and reinforcement, we will concentrate on mechanical behaviour. The properties of some fibres and their monolithic counterparts are compared in Table 1.2.

**Table 1.2** Comparison of the mechanical properties of fibres and their monolithic counterparts

	<i>Young's modulus (GPa)</i>	<i>Strength<sup>a</sup> (MPa)</i>
Alumina: fibre (Saffil RF)	300	2000
monolithic	382	332
Carbon: fibre (IM)	290	3100
monolithic	10	20
Glass: fibre (E)	76	1700
monolithic	76	100
Polyethylene: fibre (S 1000)	172	2964
monolithic (HD)	0.4	26
Silicon carbide: fibre (MF)	406	3920
monolithic	410	500

<sup>a</sup>tensile and flexural strengths for fibre and monolithic respectively.

It has already been mentioned that the reinforcement is often a strong, stiff material and this is borne out by the data presented in Table 1.2. The reader may well ask why one should not make use of the high strength and high elastic constants of these reinforcements by producing monolithic components solely from the reinforcement. In fact this is done and, for example, components manufactured from the ceramics silicon carbide

and alumina are readily available. The problem with these materials is their brittleness; although able to sustain a high stress, when they fail they do so in a catastrophic manner without warning. Moreover the stress at which nominally identical specimens, or components, fail can vary markedly. The brittleness and the variation in strength is due to failure being initiated at flaws which normally occur at the surface. There is always a distribution of flaw sizes and, in an homogeneously stressed material, failure commences at the largest flaw. The larger the failure-initiating flaw the lower the failure stress (Figure 1.6). The smaller the volume of material, the smaller on average the size of the maximum flaw and the greater the strength. Therefore the fracture stress of a small ceramic particle or fibre is greater than that of a large volume of the same material.

There are of course many properties other than strength that we have to take into account when selecting a reinforcement. In the case of fibres the flexibility is important as it determines whether the fibres may be easily woven or not and influences the choice of method for composite manufacture. The flexibility of a fibre depends mainly on the Young's modulus,  $E_t$ , and diameter,  $D$ , of the fibre:

$$\text{flexibility} \propto \frac{1}{E_t D^4} \quad (1.1)$$



Thus large diameter fibres with a high Young's modulus are not flexible.

Clearly, single fibres, because of their small cross-sectional dimensions, are not directly usable in structural applications. On the other hand the possibility exists of trying to exploit the high strength of single fibres by physically entwining the fibres in a similar manner to the manufacture of rope from hemp fibres. Unfortunately such a fabrication method would greatly restrict the shape and dimensions of components and furthermore would introduce a considerable amount of surface damage leading to a degradation in strength. These problems may be overcome by embedding the fibres in a material to hold the fibres apart, to protect the surface of the fibres and to facilitate the production of components. The embedding material is, of course, the matrix. The amount of reinforcement that can be incorporated in a given matrix is limited by a number of factors. For example with particulate reinforced metals the reinforcement content is usually kept to less than 40 vol. % (0.4 volume fraction) because of processing difficulties and increasing brittleness at higher contents. On the other hand, the processing methods for fibre reinforced polymers are capable of producing composites with a high proportion of fibres, and the upper limit of about 70 vol. % (0.7 volume fraction) is set by the need to avoid fibre-fibre contact which results in fibre damage.

Finally, the fact that the reinforcement is bonded to the matrix means that any loads applied to a composite are carried by both constituents. As in most cases the reinforcement is the stiffer and stronger constituent; it is the principal load bearer. The matrix is said to have transferred the load to the reinforcement.

#### FACTORS WHICH DETERMINE PROPERTIES:

The fabrication and properties of composites are strongly influenced by the proportions and properties of the matrix and the reinforcement. The proportions can be expressed either via the weight fraction ( $w$ ), which is

relevant to fabrication, or via the volume fraction ( $v$ ), which is commonly used in property calculations (for example, Chapter 8).

The definitions of  $w$  and  $v$  are related simply to the ratios of weight ( $W$ ) or volume ( $V$ ) as shown below.

Volume fractions:  $v_f = V_f/V_c$  and  $v_m = V_m/V_c$ . (1.2a)

Weight fractions:  $w_f = W_f/W_c$  and  $w_m = W_m/W_c$ . (1.2b)

where the subscripts m, f and c refer to the matrix, fibre (or in the more general case, reinforcement) and composite respectively.

We note that

$$v_f + v_m = 1, \text{ and}$$

$$w_f + w_m = 1.$$

We can relate weight to volume fractions by introducing the density,  $\rho$ , of the composite and its constituents. Now

$$W_c = W_f + W_m$$

which, as  $W = \rho V$ , becomes

$$\rho_c V_c = \rho_f V_f + \rho_m V_m,$$

or

$$\begin{aligned} \rho_c &= \rho_f (V_f/V_c) + \rho_m (V_m/V_c) \\ &= \rho_f v_f + \rho_m v_m. \end{aligned} \quad (1.3)$$

It may also be shown that

$$\frac{1}{\rho_c} = \frac{w_f}{\rho_f} + \frac{w_m}{\rho_m} \quad (1.4)$$

Also we have

$$w_f = \frac{W_f}{W_c} = \frac{(\rho_f V_f)}{(\rho_c V_c)} = \frac{\rho_f}{\rho_c} v_f,$$

and similarly

$$w_m = \frac{W_m}{W_c} = \frac{(\rho_m V_m)}{(\rho_c V_c)} = \frac{\rho_m}{\rho_c} v_m. \quad (1.5)$$

We can see that we can convert from weight fraction to volume fraction, and vice versa, provided the densities of the reinforcement ( $\rho_f$ ) and the matrix ( $\rho_m$ ) are known.

Equation 1.3 shows that the density of the composite is given by the volume fraction adjusted sum of the densities of the constituents. This

equation is not only applicable to density but, in certain circumstances, may apply to other properties of composites. A generalized form of the equation is

$$X_c = X_m v_m + X_f v_f, \quad (1.2)$$

where  $X_c$  represents an appropriate property of the composite, and, as before,  $v$  is the volume fraction and the subscripts m and f refer to the matrix and reinforcement respectively. This equation is known as the *Law of Mixtures*.

Most properties of a composite are a complex function of a number of parameters as the constituents usually interact in a synergistic way so as to provide properties in the composite that are not fully accounted for by the law of mixtures. The chemical and strength characteristics of the *interface* between the fibres and the matrix is particularly important in determining the properties of the composite. The interfacial bond strength has to be sufficient for load to be transferred from the matrix to the fibres if the composite is to be stronger than the unreinforced matrix. On the other hand, as the reader will learn in Chapter 11, if we are also concerned with the toughness of the composite, the interface must not be so strong that it does not fail and allow toughening mechanisms such as debonding and fibre pull-out to take place.

Other parameters which may significantly affect the properties of a composite are the shape, size, orientation and distribution of the reinforcement and various features of the matrix such as the grain size for polycrystalline matrices. These, together with volume fraction, constitute what is called the *microstructure* of the composite.

However it should be noted that even for properties which are microstructure dependent, and which do not obey the law of mixtures, the volume fraction still plays a major role in determining properties. The volume fraction is generally regarded as the single most important parameter influencing the composite's properties. Also, it is an easily controllable manufacturing variable by which the properties of a composite may be altered to suit the application.

A problem encountered during manufacture is maintaining a uniform distribution of the reinforcement. Ideally, a composite should be homogeneous, or uniform, but this is difficult to achieve. *Homogeneity* is an important characteristic that determines the extent to which a representative volume of the material may differ in physical and mechanical properties from the average properties of the material. Non-uniformity of the system should be avoided as much as possible because it reduces those properties that are governed by the weakest part of the composite. For example, failure in a non-uniform material will initiate in an area of lowest strength, thus adversely affecting the overall strength of a component manufactured from that material.

The orientation of the reinforcement within the matrix affects the *isotropy* of the system. When the reinforcement is in the form of equiaxed particles, the composite behaves essentially as an isotropic material whose elastic properties are independent of direction. When the dimensions of the reinforcement are unequal, the composite can behave as if isotropic, provided the reinforcement is randomly oriented, as in a randomly oriented, short fibre-reinforced composite (Figure 1.4(c)). In other cases the manufacturing process may induce orientation of the reinforcement and hence loss of isotropy; the composite is then said to be *anisotropic* or to exhibit *anisotropy* (Figure 1.7).

In components manufactured from continuous fibre reinforced composites, such as unidirectional or cross-ply laminates, anisotropy may be desirable as it can be arranged for the maximum service stress to be in the direction that has the highest strength. Indeed, a primary advantage of these composites is the ability to control the anisotropy of a component by design and fabrication.

It is clear from the preceding discussion that many factors may be important in determining the properties of composites; this should be borne in mind when studying theoretical models as all the factors are seldom accounted for in the development of the models.

Numerical :

Question: A continuous and aligned glass FRC consist of 40% volume of glass fiber having modulus of elasticity 69GPa and polymer resin is having modulus of elasticity 3.4GPa. Compute modulus of elasticity of composite.

Ans: ( $E_c = E_m \cdot v_m + E_f \cdot v_f$ ) [Law of mixture]

$$(E_c = E_m \cdot v_m + E_f \cdot v_f)$$

$$=(3.4 \cdot 0.6) + (69 \cdot 0.4)$$

$$=29.94 \text{ GPa}$$

$$=30 \text{ GPa} \quad (\text{Ans})$$

#### **BENEFITS OF COMPOSITES :**

Composite materials have fully established themselves as workable engineering materials and are now relatively commonplace around the world, particularly for structural purposes. Early military applications of polymer matrix composites during World War II led to large-scale commercial exploitation, especially in the marine industry, during the late 1940s and early 1950s. Today, the aircraft, automobile, leisure, electronic and medical industries are quite dependent on fibre-reinforced plastics, and these composites are routinely designed, manufactured and used. Less exotic composites, namely particulate or mineral filled plastics, are also widely used in industry because of the associated cost reduction. Some typical applications of polymer matrix composites are listed in Table 1.3.

<i>Industrial sector</i>	<i>Examples</i>
Aerospace	wings, fuselage, radomes, antennae, tail-planes, helicopter blades, landing gears, seats, floors, interior panels, fuel tanks, rocket motor cases, nose cones, launch tubes
Automobile	body panels, cabs, spoilers, consoles, instrument panels, lamp-housings, bumpers, leaf springs, drive shafts, gears, bearings
Boats	hulls, decks, masts, engine shrouds, interior panels
Chemical	pipes, tanks, pressure vessels, hoppers, valves, pumps, impellers
Domestic	interior and exterior panels, chairs, tables, baths, shower units, ladders
Electrical	panels, housings, switchgear, insulators, connectors
Leisure	motor homes, caravans, trailers, golf clubs, racquets, protective helmets, skis, archery bows, surfboards, fishing rods, canoes, pools, diving boards, playground equipment

( PMC APPLICATIONS)

The success of fibre composites with thermosetting (epoxies and polyesters) or thermoplastic matrices, largely as replacements for metals, results from the much improved mechanical properties of the composites compared with the matrix materials. The good mechanical properties of the composites are a consequence of utilizing the special properties of glass, carbon and aramid fibres (Table 1.2).

It can be seen from a comparison of Tables 1.1 and 1.4 that on the basis of strength and stiffness alone, fibre-reinforced polymer composites do not have a clear advantage over conventional materials particularly when it is noted that their elongation to fracture is much lower than that for metallic alloys of comparable strength. The advantage that polymer matrix composites have over metals is their low density,  $\rho$ . The benefit of a low density becomes apparent when the Young's modulus per unit mass,  $E/\rho$  (*specific modulus*), and tensile strength per unit mass,  $\sigma_T/\rho$  (*specific strength*), are considered. The higher specific modulus and specific strength of these composites means that the weight of certain components can be reduced. This is a factor of great importance in moving components, especially in all forms of transport where reduction in weight results in greater efficiency leading to energy and hence cost savings.



However the specific modulus and specific strength alone are only capable of specifying the performance under certain service conditions. Let us illustrate this point by first considering specific modulus. The specific modulus is an acceptable parameter for comparing materials for rod-like components under tensile loading as the lightest rod that will carry a given tensile force without exceeding a given deflection is that made from material with the greatest value of  $E/\rho$ . On the other hand under compression loading of a rod, elastic buckling may be the limiting factor and the lightest rod that will support a compressive force without buckling is constructed from material with the greatest value of  $E^{1/2}/\rho$ . Finally if we consider a panel loaded in bending, we find that the lightest panel which will support a given load with minimum deflection should be manufactured from material with the greatest value of  $E^{1/3}/\rho$ .

It is concluded therefore that a single *performance indicator*, such as specific modulus, is insufficient for assessment of materials. It is preferable to compare materials, and classes of materials, by means of *materials property charts* (Figure 1.8) where one property (Young's modulus in this case) is plotted against another (the density) on logarithmic axes, as these can incorporate a number of performance indicators. On the chart of Figure 1.8 are plotted guide lines for material selection for the three service situations, and hence performance indicators, previously described. Consider the guide line drawn for  $E^{1/3}/\rho$ ; this corresponds to a specific set of service conditions for a panel supported at both ends and subject to a central force. Any

Furthermore it should be noted that a ceramic matrix composite may also have improved strength and stiffness. An example of an application of a ceramic matrix composite which arises from the enhanced toughness and strength, together with the intrinsic hardness associated with the matrix, is industrial cutting tools; tools manufactured from alumina reinforced with SiC whiskers are said to improve the rate of cutting greatly and to have a longer life compared with conventional tool components.

Recent interest has concentrated on transport applications and consequently the light metals, particularly aluminium and its alloys, have received the most attention. The Young's modulus of aluminium and its alloys is relatively low for metals and hence there is considerable potential for improvement by reinforcement. However the reader will recall that the forte of metals is their good ductility and toughness and obviously it is desirable that these properties are not degraded in metal matrix composites.

Industrial sector	Application	
	Ceramic matrix	Metal matrix
Aerospace	afterburners, brakes, heat shields, rocket nozzles	struts, antennae
Automobile	brakes	piston crowns
Manufacturing	thermal insulation, cutting tools, wire drawing dies	
Electrical		superconductors, contacts, filaments, electrodes
Medical	prostheses, fixation plates	

( CMC AND MMC APPLICATIONS)

## REINFORCEMENT AND REINFORCEMENT-MATRIX INTERFACE:

### NATURAL FIBRES :

Examples of natural fibres are silk, wool, jute, cotton, hemp, sisal etc. Mostly these are animal and plant based, used in textile, rope, twine production. It has cellulose fibre in an amorphous matrix of lignin and hemicelluloses. It has a high aspect ratio of greater than 1000. **Aspect ratio** can be defined as the ratio of length to diameter of fibre.

Although the plant-based organic fibres are occasionally used in synthetic composites, the natural fibre which has been exploited most widely is *asbestos*. Asbestos is the name given to a group of minerals which exist in fibrous form and are often found separated from the surrounding rock. The best source of asbestos is one of the serpentine minerals known as *chrysotile*, which is a hydrated magnesium silicate,  $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$ . This occurs as silky fibres of up to several centimetres in length, which have good flexibility, stiffness and strength (Table 2.3).

The other forms of asbestos are not so commercially important. Of these *crocidolite*, or blue asbestos, which belongs to the amphibole minerals, is the best known. Its fibres are usually short and although they have good stiffness and tensile strength, as can be seen from the data in Table 2.3, their flexibility is poor.

Asbestos is used to reinforce some synthetic resins but mainly cement and plasters such as gypsum. In this context it is worth mentioning that asbestos is resistant to the very corrosive cement matrix environment during both setting and service. Asbestos has excellent thermal resistance and retains its properties to intermediate temperatures of about 600 °C and 400 °C for *chrysotile* and *crocidolite* respectively. However asbestos does have a major disadvantage of being a hazard to health if ingested. The concentration in air is limited by health standards in many countries; in the UK the limit is

2 fibres per millimetre except for *crocidolite* where the limit is even more restrictive name 0.2 fibres per millimetre.

## SYNTHETIC FIBRE:

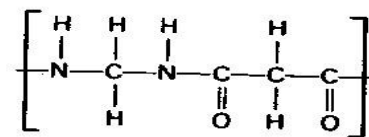
Different fibres are manufactured for the reinforcement in composites and some typical properties. The manufacture of fibres involves a no. of processing steps and variability of properties from one fibre to another is large even when made by the same process. Therefore resulting microstructure and properties are different.

## SYNTHETIC ORGANIC FIBRES:

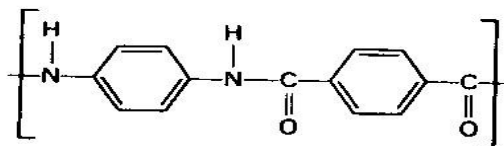
### ARAMID :

There are a number of commercially available *aramid* fibres, e.g., Kevlar (Du Pont), Twarlon (Akzo) and Technora (Teijin); of these *Kevlar* is the most well known. The aramids can be viewed as nylon with extra benzene rings in the polymer chain to increase stiffness (Figures 2.4(a) and 2.4(b)); alternative nomenclature that the reader might encounter is aromatic polyamide or poly(phenylene terephthalamide).

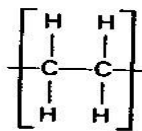
The stiff aramid molecule is aligned to a certain extent even in solution or in the melt. The extended chains are arranged approximately parallel to



(a)



(b)

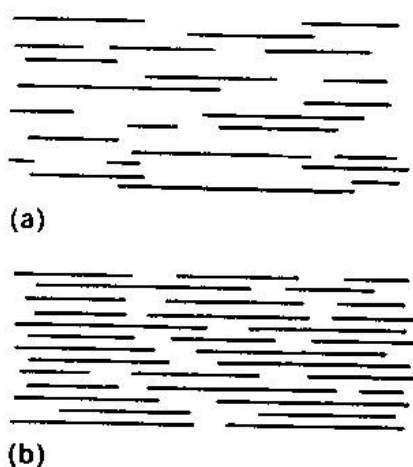


(c)

**Figure 2.4** The molecular structure of: (a) nylon; (b) aramid; (c) polyethylene.



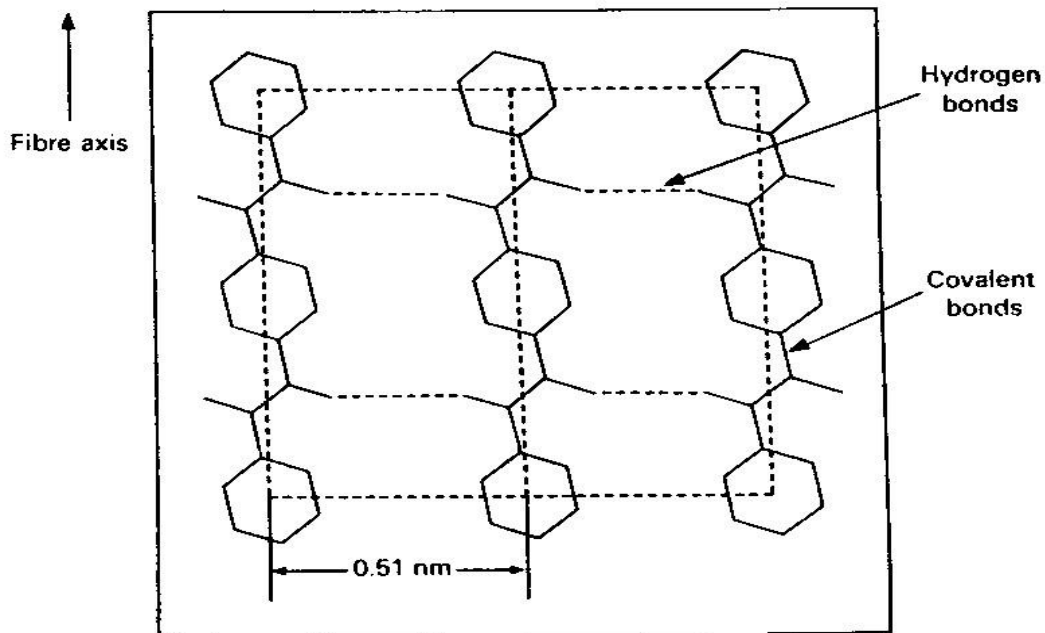
their long axis although the chain centres are randomly distributed; this structure is known as a *nematic liquid crystal* and is schematically illustrated in Figure 2.5(a). The alignment of the molecules is enhanced during fibre production by spinning and extrusion, and by any subsequent mechanical treatment (Figure 2.5(b)). The production route is as follows. A solution of the aramid in a suitable solvent such as sulphuric acid is held at a low temperature and then extruded at an elevated temperature into a coagulation bath to remove the residual solvent. This results in fibres of relatively low strength and Young's modulus of about 850 MPa and 5 GPa respectively. Finally the fibre is stretched and cold drawn to align the structure further and to increase the mechanical properties to those quoted in Table 2.4.



**Figure 2.5** Schematic illustration of aramid: (a) in solution: nematic liquid crystal; (b) after alignment.

The molecules are arranged in planar sheets (Figure 2.6) with strong covalent bonds in the molecular chains parallel to the fibre axis but weak hydrogen interchain bonding in the transverse direction. The planar sheets are in turn pleated and stacked to give the three-dimensional orientated structure with radial symmetry of Kevlar 49 shown schematically in Figure 2.7. The low modulus Kevlar (Kevlar 29) is simply a structural variation; the structure of Kevlar 29 is less perfect and less orientated but can be converted to the Kevlar 49 structure by a suitable heat treatment.

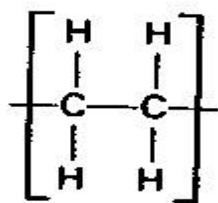
Aramid fibres have good high temperature properties for a polymeric material. They have a glass transition temperature of about 360°C, burn with difficulty and do not melt like nylon. A loss in performance due to carbonization occurs around 425°C but they can be used at elevated temperatures for sustained periods and even at 300°C for a limited time. Aramid fibres do have a tendency to degrade in sunlight. Their dimensional stability is good as the coefficient of thermal expansion is low (approximately  $-4 \times 10^{-6} \text{ K}^{-1}$ ). Other properties which are important in certain applications are low electrical and thermal conductivity and high thermal capacity.



**Figure 2.6** The planar arrangement of the aramid molecules showing the weak hydrogen and strong covalent bonds. (Source: Dobb *et al.*, 1980.)

#### POLYETHYLENE:

In the late 1980s a number of *polyethylene* fibres became commercially available including Spectra (Allied Signal) and Dyneema (Dutch State Mines, DSM). As can be seen from Figure 2.4(c), the polyethylene molecule with its carbon-carbon (C-C) backbone is simpler than that of aramid. The theoretical elastic modulus of the covalent C-C bond in the fully extended polyethylene molecule is 220 GPa along the direction of the chain. Although this is not fully achieved by the orientated structure of the fibres, the value of 172 GPa is impressive. Polyethylene has the lowest density of any readily available fibre and hence its specific properties are good and superior to those of Kevlar. However polyethylene has a low melting point of 135°C and readily creeps at elevated temperature, so use is restricted to temperatures below 100°C. The fibres can be surface treated with gas-plasma to replace H atoms with polar groups which gives improved bonding in composites and hence increased strength and stiffness but reduced toughness.



[ POLYETHYLENE ]

#### SYNTHETIC INORGANIC FIBRES:

## 1. GLASS FIBRE

Glass have the composition of  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{ZrO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$ . As per it's composition and properties these are used in different purposes. The E in E-glass is an abbreviation for electrical. This glass is based on the eutectic in the ternary system  $\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  with some  $\text{B}_2\text{O}_3$  substituting for  $\text{SiO}_2$  and some  $\text{MgO}$  for  $\text{CaO}$ . S –glass known as R-glass in Europe is based on  $\text{SiO}_2 - \text{Al}_2\text{O}_3 - \text{MgO}$  system. This glass fibre has higher stiffness and strength than E-glass. C –glass is known as chemical glass and E-CR glass is electrical- corrosion resistant glass, AR is alkali resistant glass.

The required oxides melted and molten glass is exuded under gravity from a melting tank through an orifice and rapidly pulled to draw it down to a  $10\mu\text{m}$  diameter fibre. Normally 204 orifices are used on the same melting tank giving fibres which are gathered into a strand, sized with starch-oil emulsion to minimize surface damage and wound on to a drum at speeds up to  $50\text{m/s}$ . These strands can be chopped into  $2.5\text{-}5\text{cm}$  length, wound parallel rovings and ribbon. Freshly drawn and carefully handled fibres have tensile strengths of approximately  $E_f/20$  but a typical value may be near to  $E_f/50$ , where  $E_f$  is Young's modulus.

## 2. ALUMINA FIBRE

Alumina has a high melting point  $2000^\circ\text{C}$  and a relatively low viscosity when molten. Hence it is not amenable for fibre production in melt spinning techniques. So it can be produced in two ways. i) Slurry forming + firing method, ii) Sol-gel process + heat treatment

### 1. SLURRY FORMING+FIRING METHOD

Slurry formation(aqueous suspension of fine alumina particles + polymer/ deflocculents)



Slurry extruded to green fibres and dried



Fired to remove additives and sintering occurs to produce solid fibres, controlled amount of porosity and grain size

plus heat treatment. The first stage in the former process is to form a slurry which is simply an aqueous suspension of fine alumina particles, together with additions to stabilize the suspension (deflocculents) and polymers to modify the viscosity. The slurry is then extruded into 'green' fibres and dried. Finally the 'green' fibres are fired during which additives are driven off and sintering occurs to produce solid fibres with a controlled amount of porosity and grain size. The most established fibre produced by this route is  $\text{Al}_2\text{O}_3$ -FP manufactured by Du Pont. This is a 99%  $\alpha$ -alumina fibre of diameter  $10\text{--}20\mu\text{m}$  with a grain size of about  $0.5\mu\text{m}$ . The mechanical properties at room temperature are good, but the fibres are susceptible to grain growth and to creep at elevated temperatures.

### 2. SOL-GEL PROCESS + HEAT TREATMENT

High concentrated solution of aluminium compound(nano particles)

↓  
Do extrusion(100-200micro meter holes if continuous fibre required)

↓  
After spinning fibres are dried and heat treated

↓  
Volatile metals driven off

In *solution* or *sol-gel* spinning a viscous, highly concentrated solution of an aluminium compound is used for spinning. The details of the solution and the spinning technique depend on whether continuous or discontinuous fibres are to be produced, e.g., continuous fibres produced by extrusion through 100–200  $\mu\text{m}$  holes require a solution of high viscosity (10–100  $\text{Ns m}^{-2}$ ) whereas the production of discontinuous fibres by gas-attenuation spinning generally employs solutions of lower viscosities of less than about 2  $\text{Ns m}^{-2}$ .

After spinning the fibres are dried and heat treated. During heat treatment the remaining volatiles are driven off and then crystallization takes place to the various crystal forms of alumina. The final crystal and microstructure of the fibre depends on the composition (especially the silica content), the atmosphere during decomposition, and the time and temperature of heat

Effect of heat treatment on properties of different types of alumina fibres:

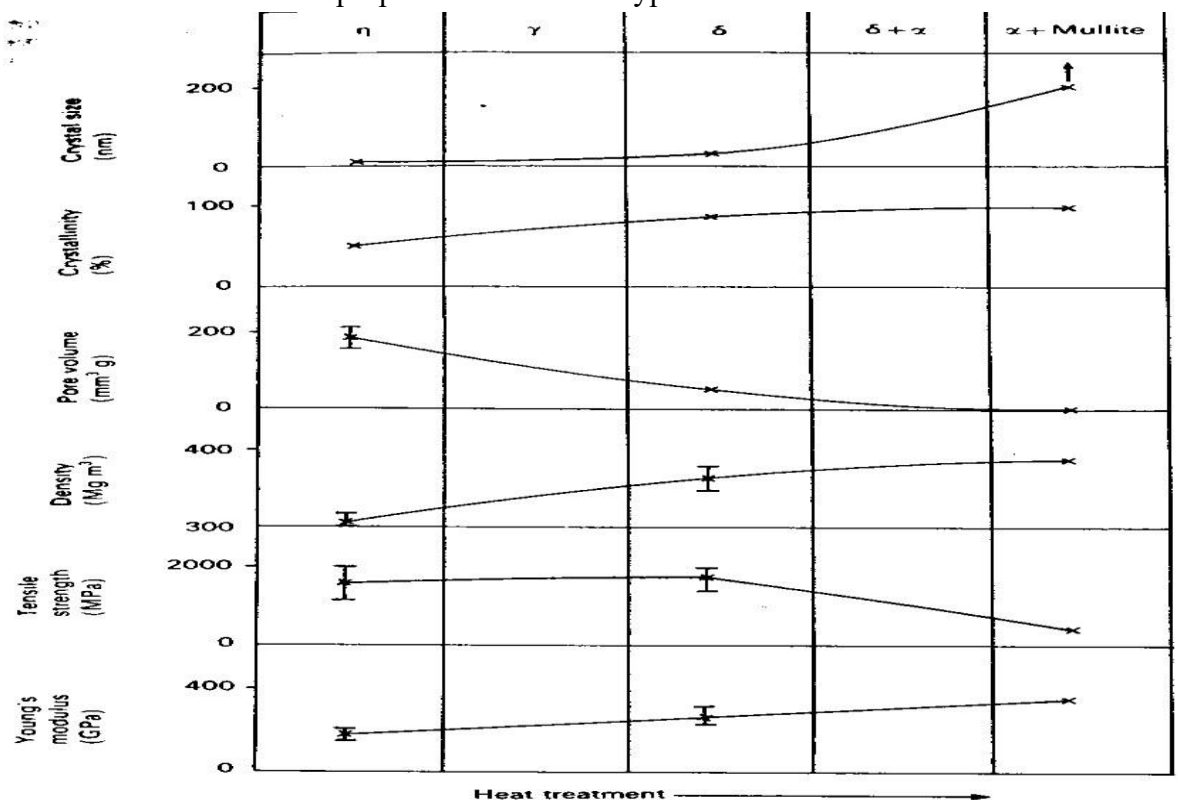
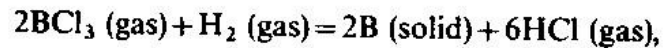


Figure 2.9 Effect of heat treatment on the structure and properties of alumina fibres (Saffil) produced by solution spinning. (Source: Dinwoodie and Horsfall, 1987.)

### 3. BORON FIBRE:

**Boron fibre** is produced by *chemical vapour deposition* from boron trichloride on to a heated substrate. The temperatures involved are high and so the substrate has to have a high melting point; the most commonly used substrate is a heated tungsten wire of about 10 µm diameter. Continuous lengths of up to 3000 m have been made. The boron trichloride is mixed with hydrogen gas and decomposes according to:



to give an approximately 50 µm thick coating of boron with an extremely small grain size of only 2–3 nm. The boron exists in two crystalline forms, namely rhombohedral and tetragonal. The former is thought to be predominant in the fibres but the details of the structure and properties depend critically on the deposition temperature and rate. Interaction of the boron with the tungsten core can occur to yield tungsten borides.

After formation, the fibre may be subjected to annealing to reduce residual stresses and to a chemical treatment to remove surface flaws and hence increase strength. Supplementary coatings may also be applied to reduce the extent of reaction with the matrix; for example, a 0.25 µm thick coating of silicon carbide is applied for use with aluminium alloy matrices. Boron fibres have a very high Young's modulus (Table 2.2 and Figure 2.2) but are not widely used because of their cost (production is about 50 000 kg/y in the USA). Substitution of the tungsten core with a less expensive and lower density material would make boron fibres more attractive. Carbon fibres have been widely investigated as a possible alternative to tungsten but have been found to have a major disadvantage. During the deposition process the boron fibre may elongate by as much as 5% which results in fracture of the carbon fibre core. This in turn leads to 'hot spots' along the fibre length and local variations in the deposition rate.

#### 4. CARBON FIBRE

Carbon fibres are produced from a variety of precursors. The mechanical properties vary greatly with types of precursors used and the processing conditions employed as these determine the perfection and alignment of crystals. There are three main precursors used. Such as PAN, cellulose or rayon, petroleum or coal- tar pitches.

**Controlled heating of cellulose or rayon fibre** converts the fibre to graphite. The decomposition is complex but essentially loss of weight and shrinkage takes place during *pyrolysis* in the temperature range 200 to 400 °C as the organic precursor decomposes to carbon. This is followed by *carbonization*, i.e., gradual ordering of the structure, and finally *graphitization* at higher temperatures. The yield is low, only 15–30%, and although the fibre is crystalline the alignment is poor. Thus at this stage the fibres have low strength and stiffness, the latter being of the order of the modulus in the *c*-direction of the graphitic structure. However hot stretching by up to 500%

50% at temperatures in the range 2700 to 3000 °C increases the modulus and strength by developing a preferred orientation in the fibres and reducing porosity (Table 2.7). The preferred orientation is such that there is a tendency for the basal planes to be aligned parallel to the axis of the fibre, although the alignment is far from perfect. This manufacturing route is declining because of the decreasing availability of the main source of suitable rayon, the low yield and the problems involved in stretching at such high temperatures.

#### PAN Process:

The first stage involves stretching and oxidation. The fibres are initially stretched 500–1300% to improve molecular alignment and then heated in air, while still under tension, to 200–280 °C. This results in intramolecular rearrangement to give a ladder-like polymer; chemical cross-links between the oriented chains in three dimensions are formed so that chains can no longer bend or distort on heating (Figure 2.13(b)). This is followed by oxidation whereby some two-thirds of the oxygen sites are filled slowly with the release of H<sub>2</sub>O (Figure 2.13(c)); this is sometimes called oxyPAN. Heating oxyPAN in nitrogen or argon at 900–1200 °C produces low modulus, high strength carbon fibres, which have fine well-oriented crystals parallel to the fibre axis. However they have a fair degree of porosity and the density is only 1.74 Mg/m<sup>3</sup>. The idealized formation of the carbon ring structure is shown in Figure 2.13(d); it can be seen that there is an operational hazard at this stage as toxic volatiles such as HCN are given off. Heating in argon up to 2800 °C causes *graphitization* and produces high modulus fibres of increased density up to 2.00 Mg/m<sup>3</sup> (the density of graphite is 2.26 Mg/m<sup>3</sup>). A three-dimensional representation of the arrangement of the basal planes in PAN-based carbon fibre is given in Figure 2.14. The value of the modulus increases with increasing temperature of graphitization as shown in Figure 2.15. In fact the properties can be varied over a wide range by suitable heat treatment conditions but it is difficult to

combine high strength with high modulus. Refinements in fibre process technology over the past twenty years have led to considerable improvements in strength and in strain to fracture for PAN-based fibres. These can now be supplied in three basic forms, high modulus (HM), intermediate modulus (IM) and high strength (HS) as shown in Table 2.7. The most recent developments of the high strength fibres have led to what are known as high strain fibres, which have strain values approaching 2% before fracture. Figure 2.16 shows the stress-strain curves for a range of carbon fibres. The strain is elastic up to failure and a lot of stored energy, given by the area under the stress-strain curve, is released when the fibres break in a brittle manner.

#### Petroleum and coal-tar pitches:

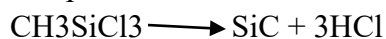


Petroleum and coal-tar pitches contain a complex mixture of high molecular weight aliphatic and aromatic hydrocarbons and they have a high carbon content, hence is suitable for precursor. Pitch heated to

**350°C to polymerize it to molecular weights of about 1000. The structure of the molecules is such that an ordered liquid-crystal or mesophase is formed. The polymer is extruded through the holes in a hot-rolled metal cylinder whilst the cylinder rotates. This process, which is known as melt-spinning, orientates the hot mesophase pitch. After oxidation to induce cross-linking, and hence prevent the fibres remelting and sticking together, the fibres are carbonized at temperatures up to 2000 °C giving low modulus carbon fibres. The carbon yield is high at about 80%. The degree of graphitization is controlled by heat treatment at temperatures up to 2900 °C. A high degree of perfection of the graphite may be obtained, together with good axial alignment of the basal planes, and not surprisingly this leads to high density, high modulus fibres (see for example P-120S in Table 2.7).**

#### 5. SILICON BASED FIBRES: CVD TECHNIQUE:

SiC produced from a carbon containing silane



- Substrate used is W. this SiC make reaction with W at SiC interface after prolonged use at temp. above 1000°C and form W<sub>2</sub>C, W<sub>5</sub>Si<sub>3</sub> up to thickness of 100-150micrometer dia.
- Due to this high thickness and high young's modulus filaments are not flexible, so it cannot be woven.
- To reduce surface damage thin coating of pyrolytic carbon is used to increase abrasion resistance.

#### DECOMPOSITION OF PRECURSOR:

- Nicalon (59%Si-31%C-10%O) is thermally decomposed at high pressure (10MPa) in autoclave by vacuum distillation to form poly dimethyl silane.
- Then it melted and oxidized at 200°C. Temp increases to 1300 °C to form SiC.
- This is not pure. Some oxygen at low temp may present and excess Si, C also there. It may form SiO<sub>2</sub>.

#### PARTICULATE AND WHISKER REINFORCEMENT

Most particulate reinforcements are produced easily by

- From bulk material grinding it to powder
- Precipitation from solution
- Sol gel processing
- Gas atomization

What is a **Whisker** ?

Whisker is a small single crystal normally less than one micron diameter. It is more pure and structurally more stable at elevated temperature.

## **PRODUCTION OF SiC WHISKER:**

PRODUCE BY PYROLYSIS OF RICE HUSK(15-20Wt% SiO<sub>2</sub> cellulose)

Heat at 700°C in an oxygen free atmosphere (Remove volatile /organic/inorganic constituents)



At higher temp 1500-1600°C heat treatment in an inert and reducing temp. ( $3C + SiO_2 = SiC + 2CO$ )



Residue heated at 800°C to remove free carbon(10% whisker SiC,Remaining are particles)

Disadvantages in production:

1. Difficult for homogeneous distribution
2. Costly
3. May be hazardous
4. Some orientation lead to anisotropic properties

## **REINFORCEMENT- MATRIX INTERFACE:**

Reinforcement-matrix is an important topic of discussion as matrix transfers the load and reinforcement bear the load. Thus reinforcement must be strongly bonded to matrix if their high strength and stiffness imparted to the composite. A weak interface results in low strength and stiffness but high resistance to fracture, whereas a strong interface results in high strength and stiffness but low resistance to fracture. Other properties of a composite such as resistance to creep, fatigue and environmental degradation also affected by characteristics of interface.

The interface is also important as the reinforcement may be continuous, discontinuous, short fibres or whisker type. Interface role also depend upon type of reinforcements used and their distribution.

### **Wettability:**

Wettability can be defined as the extent up to which a liquid will spread over a solid surface. Good wettability means that the liquid (matrix) will spread over the reinforcement covering every bump and dip of rough surface of reinforcement and displace the air.



Wetting will only occur if the viscosity of the matrix is not too high and if wetting results in a decrease in the free energy of the system. Let us consider the latter in more detail by studying a thin film of liquid (matrix) spreading over the solid (reinforcement) surface. All surfaces have an associated energy and the free energy per unit area of the solid-gas, liquid-gas and solid-liquid interfaces are  $\gamma_{SG}$ ,  $\gamma_{LG}$  and  $\gamma_{SL}$  respectively. For an increment of area  $dA$  covered by the spreading film, extra energy is required for the new areas of solid-liquid and liquid-gas interfaces. This extra energy is  $(\gamma_{SL} dA + \gamma_{LG} dA)$ , whereas  $\gamma_{SG} dA$  is recovered as the solid surface is covered. For the spreading of the liquid to be spontaneous it has to be energetically favourable and therefore we must have

$$\gamma_{SL} dA + \gamma_{LG} dA < \gamma_{SG} dA.$$

Dividing by  $dA$  gives

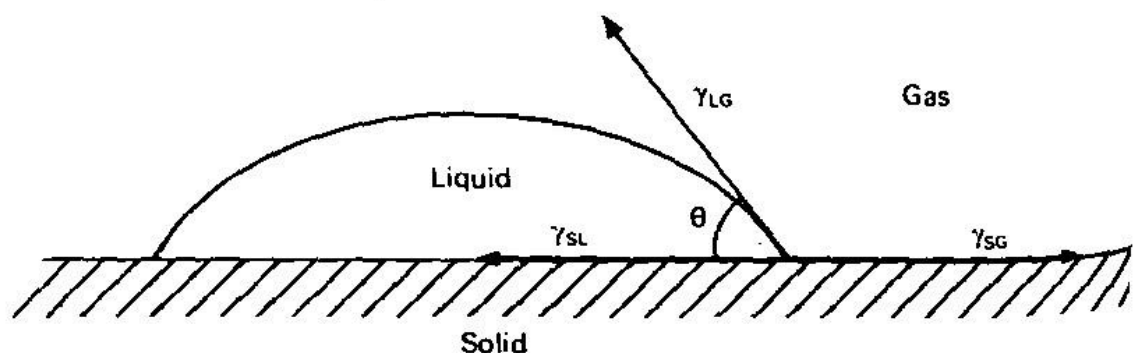
$$\gamma_{SL} + \gamma_{LG} < \gamma_{SG}.$$

From this relationship the *spreading coefficient* SC is defined as

$$SC = \gamma_{SG} - (\gamma_{SL} + \gamma_{LG}), \quad (2.1)$$

which has to be positive for wetting. It follows from equation 2.1 that if  $\gamma_{SL}$  is similar to or less than  $\gamma_{LG}$  then wetting will not occur. Thus an epoxy resin with  $\gamma_{LG}$  of about  $40 \text{ mJ/m}^2$  might be expected to readily wet aluminium ( $\gamma_{SG} \sim 1100 \text{ mJ/m}^2$ ) but not polyethylene which has a low value of  $\gamma_{SG}$  of about  $30 \text{ mJ/m}^2$ .

The above analysis demonstrates the significance of the relative values for the surface energies involved in the wetting process but does not yield a parameter that specifies intermediate conditions of wetting. Figure 2.2 illustrates an example of a drop of liquid which has been allowed to read



equilibrium and has partially wet the solid. The free energy of an interface is measured in  $\text{J/m}^2$  and can be shown to be equal to the surface tension, which has units of force per unit length ( $\text{N/m}$ ). Therefore, as we are at equilibrium, the forces can be resolved horizontally to give.

$$\gamma_{\text{SG}} = \gamma_{\text{SL}} + \gamma_{\text{LG}} \cos \theta$$

where  $\theta$  is called the *contact angle*. Rearranging gives

$$\cos \theta = \frac{(\gamma_{\text{SG}} - \gamma_{\text{SL}})}{\gamma_{\text{LG}}} \quad (2.2)$$

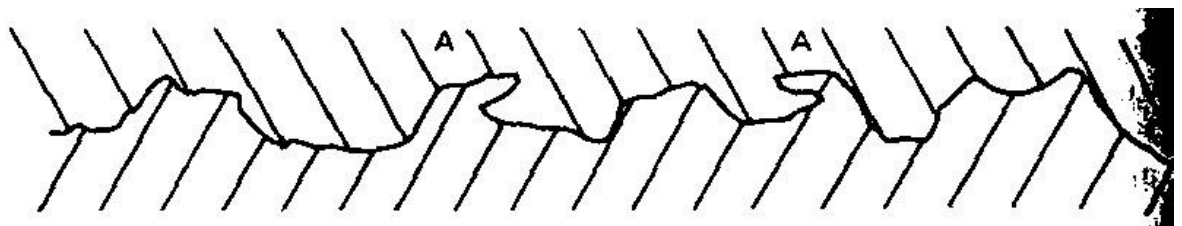
which may be used as a measure of the degree of the wettability. For a contact angle of  $180^\circ$ , the drop is spherical with only point contact with the solid and no wetting takes place. At the other extreme,  $\theta = 0$  gives perfect wetting. For intermediate values of  $\theta$ , i.e.,  $0^\circ < \theta < 180^\circ$  the degree of wetting increases as  $\theta$  decreases. Often it is considered that the liquid does not wet the solid if  $\theta > 90^\circ$ .

#### INTERFACIAL BONDING:

Once the matrix has wet the reinforcement, and is therefore in intimate contact with the reinforcement, bonding will occur. A number of different types of bond may be formed. Furthermore, for a given system more than one bonding mechanism may be operative at the same time, e.g., mechanical and electrostatic bonding, and the bonding mechanism may change during the various production stages or during service, e.g., electrostatic bonding changing to reaction bonding. The type of bonding varies from system to system and depends on fine details such as the presence of contaminants or of added surface active agents (coupling agents). We now discuss the main features of the different bonding mechanisms.

#### MECHANICAL BONDING:

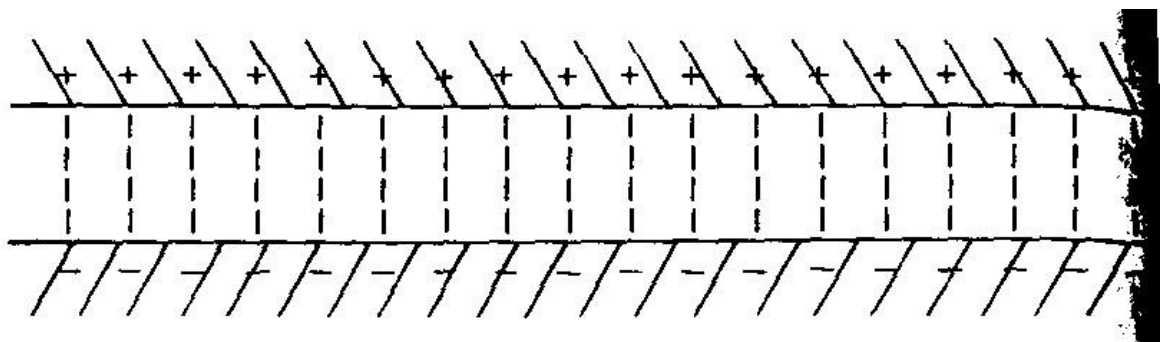
Mechanical interlocking or keying of two surfaces, as shown in Figure 2.8, can lead to a reasonable bond. Clearly the interlocking is greater, hence the mechanical bonding more effective, the rougher the interface. Also, any contraction of the matrix onto the reinforcement is favourable for bonding.



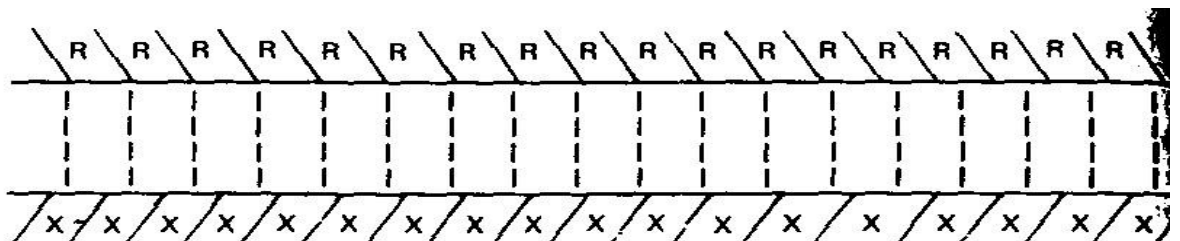
Mechanical bond is most effective when the force is applied parallel to the interface, in other words, the shear strength may be considerable. On the other hand, when the interface is being pulled apart by tensile force, the strength is likely to be low unless there is a high density of interlocking (designated A in Figure 2.22(a)) with re-entrant angles. In most cases, mechanical bond is not encountered and mechanical bonding is in conjunction with another bonding mechanism.

#### (b) Electrostatic bonding

Bonding occurs between the matrix and the reinforcement when one surface is *positively charged* and the other *negatively charged* (Figure 2.22(b)). This leads to an electrostatic attraction between the components of the composite which will depend on the difference in charge on their surfaces. Electrostatic interactions are short range and are only effective over small distances of the order of atomic dimensions: the reader will therefore appreciate that it is essential that the matrix and reinforcement are in intimate contact and that surface contamination and entrapped gases will decrease the effectiveness of this bonding mechanism.

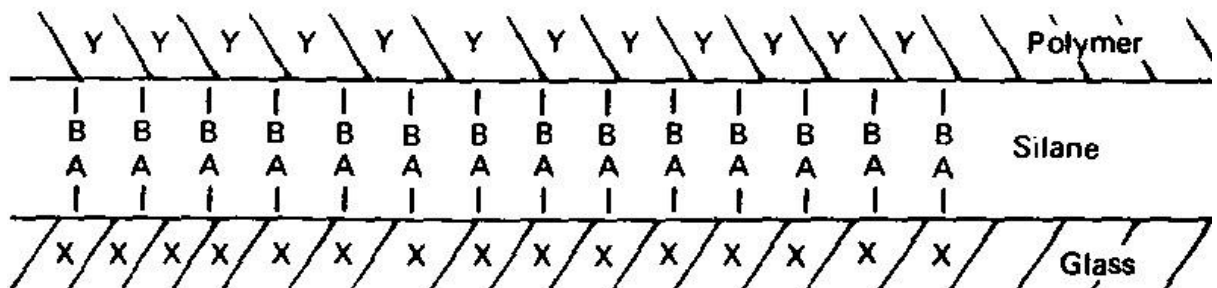


#### C. CHEMICAL BONDING



In the context of composite science, chemical bonding is the bond formed between chemical groups on the reinforcement surface (marked X in Figure 2.22(c)) and compatible groups (marked R) in the matrix. Not surprisingly the strength of the chemical bond depends on the number of bonds per unit area and the type of bond.

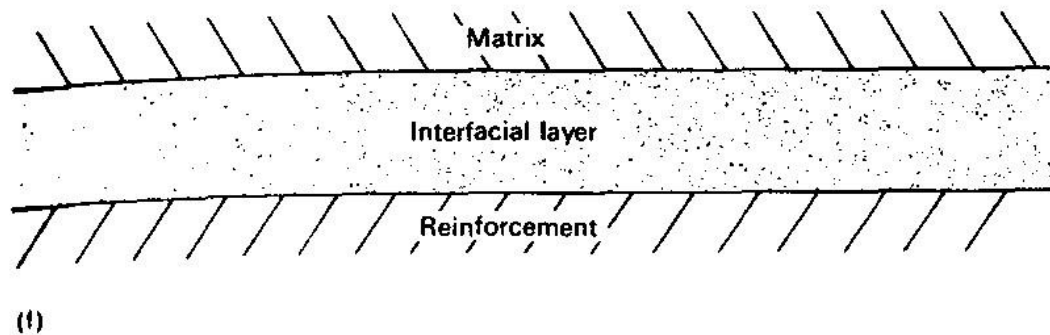
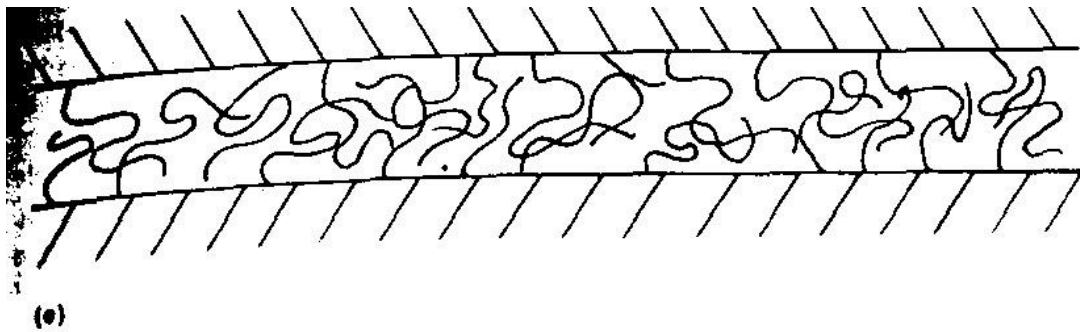
It is thought that chemical bonding may account for the success of some coupling agents. For example *silanes* are commonly employed for coupling the oxide groups on a glass surface to the molecules of a polymer matrix. At one end (A) of the silane molecule a hydrogen bond forms between the oxide (silanol) groups on the glass and the partially hydrolyzed silane, whereas at the other end (B) it reacts with a compatible group in the polymer (Figure 2.22(d)).



#### D. REACTION OR INTERDIFFUSION BONDING

The atoms or molecules of the two components of the composite may interdiffuse at the interface to give what is known as reaction or interdiffusion bonding. For interfaces involving polymers this type of bonding can, in simple terms, be considered as due to the intertwining of molecules (Figure 2.22(e)). Many factors control the strength of this interface including the distance over which the molecules have entwined, the extent of the entanglement of the molecules and the number of molecules per unit area of interface.

For systems involving metals and ceramics the interdiffusion of species from the two components can produce an interfacial layer of different composition and structure from either of the components (Figure 2.22 (f)). The interfacial layer will also have different mechanical properties from either the matrix or reinforcement and this consequently greatly affects the characteristics of the interface. In metal matrix composites the interfacial layer is often a brittle *intermetallic compound* which is a compound that exists at or around a stoichiometric composition such as  $AB$ ,  $A_2B$ ,  $A_3B$ , e.g.,  $CuAl_2$ .



One of the main reasons why interfacial layers are formed is that ceramic matrix and metal matrix composite production invariably involve high temperatures. Diffusion is rapid at high temperatures as the rate of diffusion, or the *diffusion coefficient*,  $D_d$ , increases exponentially with temperature according to the Arrhenius-type equation

$$D_d = D_0 \exp(-Q_d/RT), \quad (2.3)$$

where  $Q_d$  is the activation energy for diffusion,  $D_0$  is a constant,  $R$  is the gas constant and  $T$  is the temperature. Temperature has a marked effect on the diffusion coefficient; substituting a typical value for  $Q_d$  of 250 kJ/mol into equation 2.3 it can be shown that  $D_d$  is  $2 \times 10^{34}$  greater at 1000 °C than at room temperature! The extent of the interdiffusion  $x$ , and hence the thickness of the reaction layer, depends on time,  $t$ , as well as temperature and is given by the approximate relationship

$$x = (D_d t)^{1/2}. \quad (2.4)$$

An interface can change during service. In particular interfacial layers can form during service at elevated temperature. In addition, previously formed layers may continue to grow and complex multilayer interfaces may develop.

#### BOND STRENGTH MEASUREMENT:

### (a) Single fibre tests

The most direct test involves pulling a partially embedded single fibre out of a block of matrix material (Figure 2.23(a)). This test, although simple in principle, is difficult to carry out especially for thin brittle fibres. However if successful, from the resulting tensile stress versus strain plot the shear strength of the interface and the energies of debonding and pull-out may be obtained. These energies are discussed in more detail in Chapter 11 which considers toughening mechanisms.

The interfacial shear strength  $\tau_i$  may also be evaluated using a specimen consisting of a block of matrix material with a single, completely embedded short fibre which is accurately aligned longitudinally in the centre of the specimen (Figure 2.23(b)). On testing in compression, shear stresses are set up at the ends of the fibres as a consequence of the difference in elastic properties of the fibre and matrix (see Chapter 10). The shear stresses eventually lead to debonding at the fibre ends and  $\tau_i$  may be evaluated from the compressive stress  $\sigma_c$  at which debonding occurs as

$$\tau_i \sim 2.5\sigma_c.$$

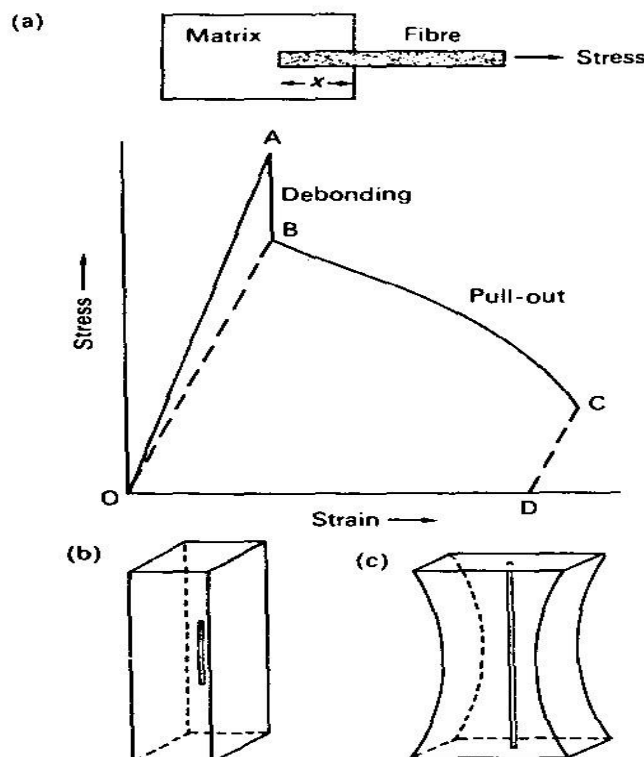


Figure 2.23 Single fibre tests: (a) fibre pull out test; (b) compression test for interfacial shear strength; (c) compression test for interfacial tensile strength.

### BULK SPECIMEN TEST:



Of these the *three-point bend* is the simplest and the most widely employed. The three-point bend geometry and the variation in the shear and tensile stresses in the loaded specimen are illustrated in Figure 2.24. At a given load  $P$ , the maximum tensile stress  $\sigma$ , which is parallel to the specimen length, lies on a line on the surface below the centre loading point (Figure 2.24(b)) and is given by

$$\sigma = \frac{3}{2} \frac{(PS)}{(BD^2)}, \quad (2.5)$$

where  $B$ ,  $D$  and  $S$  are defined in Figure 2.24(a);  $D$  and  $B$  are the height (thickness) and breadth of the specimen respectively, and  $S$  is the span between the outer loading points.

Using this testing arrangement with an aligned composite with the fibres perpendicular to the specimen length (Figure 2.24(d)) enables the tensile strength  $\sigma_i$  of the interface to be evaluated by substitution of the failure load into equation 2.5.

We have to consider the shear stresses in a specimen if we wish to determine the interfacial shear strength. The shear stress has a maximum at the midplane of the specimen (Figure 2.24(c)) of

$$\tau = \frac{3}{4} \frac{P}{BD}. \quad (2.6)$$

Combining this equation with equation 2.5, gives

$$\frac{\tau}{\sigma} = \frac{D}{2S} \quad (2.7)$$

### (c) Micro-indentation test

As the name suggests, this test employs a standard micro-indentation hardness tester. The advantages of the micro-indentation test are that the specimen is small and does not have to receive any special preparation except that one surface has to be polished to a finish suitable for microscopic examination. The indenter is loaded with a force  $P$  on to the centre of a fibre, whose axis is normal to the surface, and causes the fibre to slide along the fibre-matrix interface. The surface of the fibre is therefore depressed a distance  $u$  below the surrounding matrix surface (Figure 2.26). For a specimen of reasonable thickness the sliding distance is less than the specimen thickness and in these circumstances the interfacial shear strength is given by

$$\tau_i = P^2 / 4\pi u R^3 E_f,$$

where  $R$  and  $E_f$  are the radius and Young's modulus of the fibre,

