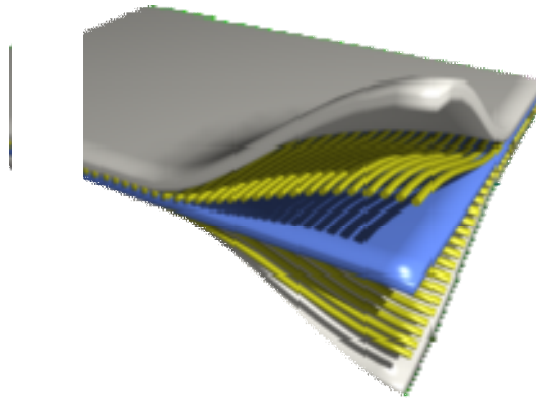
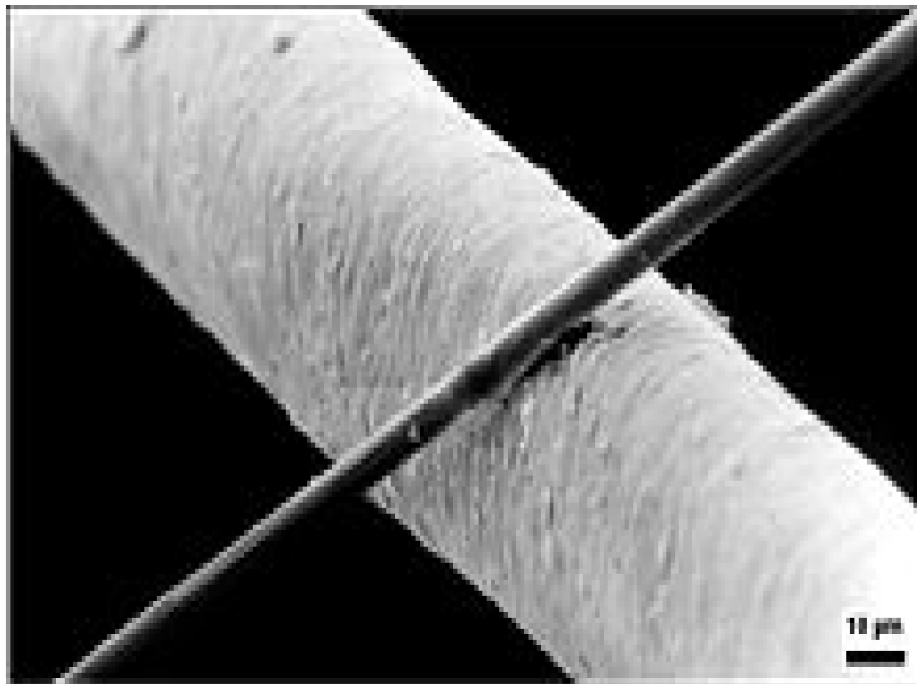


Programme-	M. Tech
Course -	COMPOSITE MATERIALS
Course Code-	MTMA-211(2)
Sem-	3rd
Year-	2020-21
Unit-	1
Topic-	Introduction to Composite Materials
Sub-Topic-	Introduction to Composite Materials: Definition, classification and characteristics of composite Materials – fibrous composites, laminated composites, particulate composites. Applications: Automobile, Aircrafts. Missiles. Space hardware, Electrical and electronics, Marine, recreational and sports equipment, future potential of composites..
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Introduction to Composite Materials -A composite material (also called a composition material or shortened to composite, which is the common name) is a material made from two or more constituent materials with significantly different physical or chemical properties that, when combined, produce a material with characteristics different from the individual components. The individual components remain separate and distinct within the finished structure, differentiating composites from mixtures and solid solutions.



Composites are formed by combining materials together to form an overall structure with properties that differ from that of the individual components.



A black carbon fibre (used as a reinforcement component) compared to a human hair.

The new material may be preferred for many reasons. Common examples include materials which are stronger, lighter, or less expensive when compared to traditional materials.

More recently, researchers have also begun to actively include sensing, actuation, computation and communication into composites,^[3] which are known as Robotic Materials.

Typical engineered composite materials include:

- Reinforced concrete and masonry

- Composite wood such as plywood
- Reinforced plastics, such as fibre-reinforced polymer or fiberglass
- Ceramic matrix composites (composite ceramic and metal matrices)
- Metal matrix composites
- and other advanced composite materials

Composite materials are generally used for buildings, bridges, and structures such as boat hulls, swimming pool panels, racing car bodies, shower stalls, bathtubs, storage tanks, imitation granite and cultured marble sinks and countertops.

The most advanced examples perform routinely on spacecraft and aircraft in demanding environments

History- The earliest synthetic composite materials were made from straw and mud combined to form bricks for building construction. Ancient brick-making was documented by Egyptian tomb paintings.

Wattle and daub is one of the oldest synthetic composite materials, at over 6000 years old. Concrete is also a composite material, and is used more than any other synthetic material in the world. As of 2006, about 7.5 billion cubic metres of concrete are made each year—more than one cubic metre for every person on Earth.

- Woody plants, both true wood from trees and such plants as palms and bamboo, yield natural composites that were used prehistorically by mankind and are still used widely in construction and scaffolding.
- Plywood 3400 BC[8] by the Ancient Mesopotamians; gluing wood at different angles gives better properties than natural wood.
- Cartonnage layers of linen or papyrus soaked in plaster dates to the First Intermediate Period of Egypt c. 2181–2055 BC and was used for death masks.
- Cob mud bricks, or mud walls, (using mud (clay) with straw or gravel as a binder) have been used for thousands of years.
- Concrete was described by Vitruvius, writing around 25 BC in his Ten Books on Architecture, distinguished types of aggregate appropriate for the preparation of lime mortars. For structural mortars, he recommended pozzolana, which were volcanic sands from the sand like beds of Pozzuoli brownish-yellow-gray in colour near Naples and reddish-brown at Rome. Vitruvius specifies a ratio of 1 part lime to 3 parts pozzolana for cements used in buildings and a 1:2 ratio of lime to pulvis Puteolanus for underwater work, essentially the same ratio mixed today for concrete used at sea. Natural cement-stones, after burning, produced cements used in concretes from post-Roman times into the 20th century, with some properties superior to manufactured Portland cement.
- Papier-mâché, a composite of paper and glue, has been used for hundreds of years.
- The first artificial fibre reinforced plastic was a combination of fiber glass and bakelite, performed in 1935 by Al Simison and Arthur D Little in Owens Corning Company.

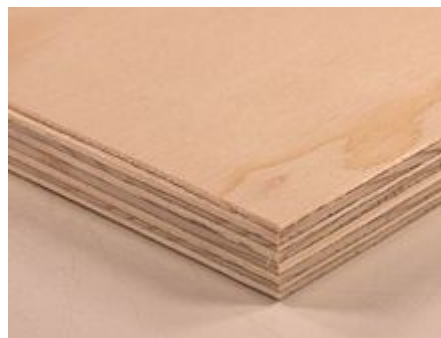
- One of the most common and familiar composite is fibreglass, in which small glass fibre are embedded within a polymeric material (normally an epoxy or polyester). The glass fibre is relatively strong and stiff (but also brittle), whereas the polymer is ductile (but also weak and flexible). Thus the resulting fibreglass is relatively stiff, strong, flexible, and ductile.

Examples- Concrete is the most common artificial composite material of all and typically consists of loose stones (aggregate) held with a matrix of cement. Concrete is an inexpensive material, and will not compress or shatter even under quite a large compressive force. However, concrete cannot survive tensile loading (i.e., if stretched it will quickly break apart). Therefore, to give concrete the ability to resist being stretched, steel bars, which can resist high stretching forces, are often added to concrete to form reinforced concrete.



Concrete is a mixture of cement and aggregate, giving a robust, strong material that is very widely used.

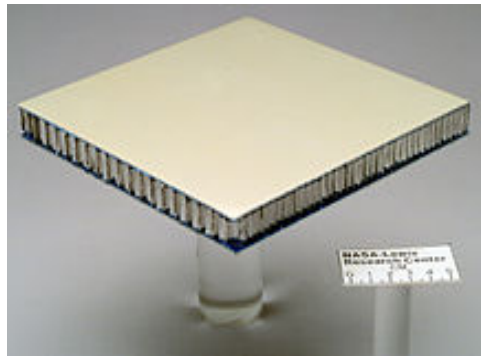
Fibre-reinforced polymers include carbon fiber reinforced polymer and glass-reinforced plastic. If classified by matrix then there are thermoplastic composites, short fibre thermoplastics, long fibre thermoplastics or long fibre-reinforced thermoplastics. There are numerous thermoset composites, including paper composite panels. Many advanced thermoset polymer matrix systems usually incorporate aramid fibre and carbon fibre in an epoxy resin matrix.



Plywood is used widely in construction

Shape memory polymer composites are high-performance composites, formulated using fibre or fabric reinforcement and shape memory polymer resin as the matrix. Since a shape memory polymer resin is used as the matrix, these composites have the ability to be easily manipulated into various configurations when they are heated above their activation temperatures and will exhibit

high strength and stiffness at lower temperatures. They can also be reheated and reshaped repeatedly without losing their material properties. These composites are ideal for applications such as lightweight, rigid, deployable structures; rapid manufacturing; and dynamic reinforcement.



Composite sandwich structure panel used for testing at NASA

High strain composites are another type of high-performance composites that are designed to perform in a high deformation setting and are often used in deployable systems where structural flexing is advantageous. Although high strain composites exhibit many similarities to shape memory polymers, their performance is generally dependent on the fibre layout as opposed to the resin content of the matrix.

Composites can also use metal fibres reinforcing other metals, as in metal matrix composites (MMC) or ceramic matrix composites (CMC), which includes bone (hydroxyapatite reinforced with collagen fibres), cermet (ceramic and metal) and concrete. Ceramic matrix composites are built primarily for fracture toughness, not for strength. Another class of composite materials involve woven fabric composite consisting of longitudinal and transverse laced yarns. Woven fabric composites are flexible as they are in form of fabric.

Organic matrix/ceramic aggregate composites include asphalt concrete, polymer concrete, mastic asphalt, mastic roller hybrid, dental composite, syntactic foam and mother of pearl. Chobham armour is a special type of composite armour used in military applications.

Additionally, thermoplastic composite materials can be formulated with specific metal powders resulting in materials with a density range from 2 g/cm³ to 11 g/cm³ (same density as lead). The most common name for this type of material is "high gravity compound" (HGC), although "lead replacement" is also used. These materials can be used in place of traditional materials such as aluminium, stainless steel, brass, bronze, copper, lead, and even tungsten in weighting, balancing (for example, modifying the centre of gravity of a tennis racquet), vibration damping, and radiation shielding applications. High density composites are an economically viable option when certain materials are deemed hazardous and are banned (such as lead) or when secondary operations costs (such as machining, finishing, or coating) are a factor.

A sandwich-structured composite is a special class of composite material that is fabricated by attaching two thin but stiff skins to a lightweight but thick core. The core material is normally low strength material, but its higher thickness provides the sandwich composite with high bending stiffness with overall low density.

Wood is a naturally occurring composite comprising cellulose fibres in a lignin and hemicellulose matrix. Engineered wood includes a wide variety of different products such as wood fibre board, plywood, oriented strand board, wood plastic composite (recycled wood fibre in polyethylene matrix), Pykrete (sawdust in ice matrix), Plastic-impregnated or laminated paper or textiles,

Arborite, Formica (plastic) and Micarta. Other engineered laminate composites, such as Mallite, use a central core of end grain balsa wood, bonded to surface skins of light alloy or GRP. These generate low-weight, high rigidity materials.

Particulate composites have particle as filler material dispersed in matrix, which may be nonmetal, such as glass, epoxy. Automobile tire is an example of particulate composite.

Advanced diamond-like carbon (DLC) coated polymer composites have been reported where the coating increases the surface hydrophobicity, hardness and wear resistance.

Products- Fibre-reinforced composite materials have gained popularity (despite their generally high cost) in high-performance products that need to be lightweight, yet strong enough to take harsh loading conditions such as aerospace components (tails, wings, fuselages, propellers), boat and scull hulls, bicycle frames and racing car bodies. Other uses include fishing rods, storage tanks, swimming pool panels, and baseball bats. The Boeing 787 and Airbus A350 structures including the wings and fuselage are composed largely of composites. Composite materials are also becoming more common in the realm of orthopedic surgery, and it is the most common hockey stick material.

Carbon composite is a key material in today's launch vehicles and heat shields for the re-entry phase of spacecraft. It is widely used in solar panel substrates, antenna reflectors and yokes of spacecraft. It is also used in payload adapters, inter-stage structures and heat shields of launch vehicles. Furthermore, disk brake systems of airplanes and racing cars are using carbon/carbon material, and the composite material with carbon fibres and silicon carbide matrix has been introduced in luxury vehicles and sports cars.

In 2006, a fibre-reinforced composite pool panel was introduced for in-ground swimming pools, residential as well as commercial, as a non-corrosive alternative to galvanized steel.

In 2007, an all-composite military Humvee was introduced by TPI Composites Inc and Armor Holdings Inc, the first all-composite military vehicle. By using composites the vehicle is lighter, allowing higher payloads. In 2008, carbon fibre and DuPont Kevlar (five times stronger than steel) were combined with enhanced thermoset resins to make military transit cases by ECS Composites creating 30-percent lighter cases with high strength.

Pipes and fittings for various purpose like transportation of potable water, fire-fighting, irrigation, seawater, desalinated water, chemical and industrial waste, and sewage are now manufactured in glass reinforced plastics.

Composite materials used in tensile structures for facade application provides the advantage of being translucent. The woven base cloth combined with the appropriate coating allows better light transmission. This provides a very comfortable level of illumination compared to the full brightness of outside.

The wings of wind turbines, in growing sizes in the order of 50 m length are fabricated in composites since several years.

Two-lower-leg-amputees run on carbon-composite spring-like artificial feet as quick as healthy sportsmen.

High pressure gas cylinders typically about 7–9 litre volume x 300 bar pressure for firemen are nowadays constructed from carbon composite. Type-4-cylinders include metal only as boss that carries the thread to screw in the valve.

Overview- Composites are made up of individual materials referred to as constituent materials. There are two main categories of constituent materials: **matrix** (binder) and **reinforcement**. At least one portion of each type is required. The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcements impart their special mechanical and physical properties to enhance the matrix properties. A synergism produces material properties unavailable from the individual constituent materials, while the wide variety of matrix and strengthening materials allows the designer of the product or structure to choose an optimum combination.

Engineered composite materials must be formed to shape. The matrix material can be introduced to the reinforcement before or after the reinforcement material is placed into the mould cavity or onto the mould surface. The matrix material experiences a melding event, after which the part shape is essentially set. Depending upon the nature of the matrix material, this melding event can occur in various ways such as chemical polymerization for a thermoset polymer matrix, or solidification from the melted state for a thermoplastic polymer matrix composite.



Carbon fibre composite part.

A variety of moulding methods can be used according to the end-item design requirements. The principal factors impacting the methodology are the natures of the chosen matrix and reinforcement materials. Another important factor is the gross quantity of material to be produced. Large quantities can be used to justify high capital expenditures for rapid and automated manufacturing technology. Small production quantities are accommodated with lower capital expenditures but higher labour and tooling costs at a correspondingly slower rate.

Many commercially produced composites use a polymer matrix material often called a resin solution. There are many different polymers available depending upon the starting raw ingredients. There are several broad categories, each with numerous variations. The most common are known as polyester, vinyl ester, epoxy, phenolic, polyimide, polyamide, polypropylene, PEEK, and others. The reinforcement materials are often fibres but also commonly ground minerals. The various methods described below have been developed to reduce the resin content of the final product, or the fibre content is increased. As a rule of thumb, lay up results in a product containing 60% resin and 40% fibre, whereas vacuum infusion gives a final product with 40% resin and 60% fibre content. The strength of the product is greatly dependent on this ratio.

Constituents

Matrices

(i) Organic- Polymers are common matrices (especially used for fibre reinforced plastics). Road surfaces are often made from asphalt concrete which uses bitumen as a matrix. Mud (wattle and daub) has seen extensive use. Typically, most common polymer-based composite materials,

including fibreglass, carbon fibre, and Kevlar, include at least two parts, the substrate and the resin.

Polyester resin tends to have yellowish tint, and is suitable for most backyard projects. Its weaknesses are that it is UV sensitive and can tend to degrade over time, and thus generally is also coated to help preserve it. It is often used in the making of surfboards and for marine applications. Its hardener is a peroxide, often MEKP (methyl ethyl ketone peroxide). When the peroxide is mixed with the resin, it decomposes to generate free radicals, which initiate the curing reaction. Hardeners in these systems are commonly called catalysts, but since they do not re-appear unchanged at the end of the reaction, they do not fit the strictest chemical definition of a catalyst.

Vinyl ester resin tends to have a purplish to bluish to greenish tint. This resin has lower viscosity than polyester resin and is more transparent. This resin is often billed as being fuel resistant, but will melt in contact with gasoline. It tends to be more resistant over time to degradation than polyester resin and is more flexible. It uses the same hardeners as polyester resin (at a similar mix ratio) and the cost is approximately the same.

Epoxy resin is almost transparent when cured. In the aerospace industry, epoxy is used as a structural matrix material or as a structural glue.

Shape memory polymer (SMP) resins have varying visual characteristics depending on their formulation. These resins may be epoxy-based, which can be used for auto body and outdoor equipment repairs; cyanate-ester-based, which are used in space applications; and acrylate-based, which can be used in very cold temperature applications, such as for sensors that indicate whether perishable goods have warmed above a certain maximum temperature. These resins are unique in that their shape can be repeatedly changed by heating above their glass transition temperature (T_g). When heated, they become flexible and elastic, allowing for easy configuration. Once they are cooled, they will maintain their new shape. The resins will return to their original shapes when they are reheated above their T_g . The advantage of shape memory polymer resins is that they can be shaped and reshaped repeatedly without losing their material properties. These resins can be used in fabricating shape memory composites.

Traditional materials such as glues, muds have traditionally been used as matrices for papier-mâché and adobe.

(ii) **Inorganic**-Cement (concrete), metals, ceramics, and sometimes glasses are employed.

Unusual matrices such as ice are sometime proposed as in pykecrete.

Reinforcements

Fiber- Reinforcement usually adds rigidity and greatly impedes crack propagation. Thin fibers can have very high strength, and provided they are mechanically well attached to the matrix they can greatly improve the composite's overall properties.



Differences in the way the fibres are laid out give different strengths and ease of manufacture

Fibre-reinforced composite materials can be divided into two main categories normally referred to as short fibre-reinforced materials and continuous fiber-reinforced materials. Continuous reinforced materials will often constitute a layered or laminated structure. The woven and continuous fiber styles are typically available in a variety of forms, being pre-impregnated with the given matrix (resin), dry, uni-directional tapes of various widths, plain weave, harness satins, braided, and stitched.

The short and long fibres are typically employed in compression moulding and sheet moulding operations. These come in the form of flakes, chips, and random mat (which can also be made from a continuous fibre laid in random fashion until the desired thickness of the ply / laminate is achieved).

Common fibres used for reinforcement include glass fibres, carbon fibres, cellulose (wood/paper fibre and straw) and high strength polymers for example aramid. Silicon carbide fibers are used for some high temperature applications.

Particle

Particle reinforcement adds a similar effect to precipitation hardening in metals and ceramics. Large particles impede dislocation movement and crack propagation as well as contribute to the composite's Young's Modulus. In general, particle reinforcement effect on Young's Modulus lies between values predicted by

$$E_c = \frac{E_m E_f}{(V_m E_f + V_f E_m)}$$

as a lower bound and

$$E_c = (V_m E_m + V_f E_f)$$

as an upper bound.

Therefore, it can be expressed as a linear combination of contribution from the matrix and some weighted contribution from the particles.

$$E_c = V_m E_m + K_c V_p E_p$$

Where K_c is an experimentally derived constant between 0 and 1. This range of values for K_c reflects that particle reinforced composites are not characterized by the isostrain condition.

Similarly, the tensile strength can be modeled in an equation of similar construction where K_s is a similarly bounded constant not necessarily of the same value of K_c .

$$(T_{ult})_c = K_s V_m S_{u,m} + K_s V_p S_{u,p}$$

The true value of K_c and K_s vary based on factors including particle shape, particle distribution, and particle/matrix interface. Knowing these parameters, the mechanical properties can be modeled based on effects from grain boundary strengthening, dislocation strengthening, and Orowan strengthening.

The most common particle reinforced composite is concrete, which is a mixture of gravel and sand usually strengthened by addition of small rocks or sand. Metals are often reinforced with ceramics to increase strength at the cost of ductility. Finally polymers and rubber are often reinforced with carbon black, commonly used in auto tires.

Cores

Many composite layup designs also include a co-curing or post-curing of the prepreg with various other media, such as honeycomb or foam. This is commonly called a sandwich structure. This is a more common layup for the manufacture of radomes, doors, cowlings, or non-structural parts.

Open- and closed-cell-structured foams like polyvinylchloride, polyurethane, polyethylene or polystyrene foams, balsa wood, syntactic foams, and honeycombs are commonly used core materials. Open- and closed-cell metal foam can also be used as core materials. Recently, 3D graphene structures (also called graphene foam) have also been employed as core structures. A recent review by Khurram and Xu et al., have provided the summary of the state-of-the-art techniques for fabrication of the 3D structure of graphene, and the examples of the use of these foam like structures as a core for their respective polymer composites.

Semi-Crystalline Polymers

Although the two phases are chemically equivalent, semi-crystalline polymers can be described both quantitatively and qualitatively as composite materials. The crystalline portion has a higher elastic modulus and provides reinforcement for the less stiff, amorphous phase. Polymeric materials can range from 0% to 100% crystallinity aka volume fraction depending on molecular structure and thermal history. Different processing techniques can be employed to vary the percent crystallinity in these materials and thus the mechanical properties of these materials as described in the physical properties section. This effect is seen in a variety of places from industrial plastics like polyethylene shopping bags to spiders which can produce silks with different mechanical properties. In many cases these materials act like particle composites with randomly dispersed crystals known as spherulites. However they can also be engineered to be anisotropic and act more like fiber reinforced composites.^[23] In the case of spider silk, the properties of the material can even be dependent on the size of the crystals, independent of the volume fraction. Ironically, single component polymeric materials are some of the most easily tunable composite materials known.

Fabrication methods

Fabrication of composite materials is accomplished by a wide variety of techniques, including:

- Advanced fibre placement (Automated fibre placement)
- Tailored fibre placement
- fibreglass spray lay-up process
- Filament winding
- Lanxide process
- Tufting
- Z-pinning

Composite fabrication usually involves wetting, mixing or saturating the reinforcement with the matrix, and then causing the matrix to bind together (with heat or a chemical reaction) into a rigid structure. The operation is usually done in an open or closed forming mould, but the order and ways of introducing the ingredients varies considerably.

References:

1. Fazeli, Mahyar; Florez, Jennifer Paola; Simão, Renata Antoun (April 2019). "Improvement in adhesion of cellulose fibers to the thermoplastic starch matrix by plasma treatment modification". *Composites Part B: Engineering*. **163**: 207–216. doi:10.1016/j.compositesb.2018.11.048.
2. Elhajjar, Rani; La Saponara, Valeria; Muliana, Anastasia, eds. (2017). *Smart Composites: Mechanics and Design (Composite Materials)*. CRC Press. ISBN 978-1-138-07551-1.
3. "Autonomous Materials Will Let Future Robots Change Color And Shift Shape". *popsci.com*. Archived from the original on 27 September 2017. Retrieved 3 May 2018.

Fibrous composites- Polymers have a low stiffness, and (in the right range of temperature) are ductile. Ceramics and glasses are stiff and strong, but are catastrophically brittle. In fibrous composites we exploit the great strength of the ceramic while avoiding the catastrophe: the brittle failure of fibres leads to a progressive, not a sudden, failure. If the fibres of a composite are aligned along the loading direction, then the stiffness and the strength are, roughly speaking, an average of those of the matrix and fibres, weighted by their volume fractions. But not all composite properties are just a linear combination of those of the components. Their great attraction lies in the fact that, frequently, something extra is gained. The toughness is an example. If a crack simply ran through a GFRP composite, one might (at first sight) expect the toughness to be a simple weighted average of that of glass and epoxy; and both are low. But that is not what happens. The strong fibres pullout of the epoxy. In pulling out, work is done and this work contributes to the toughness of the composite. The toughness is greater – often much greater – than the linear combination. Polymer-matrix composites for aerospace and transport are made by laying up glass, carbon or Kevlar fibres in an uncured mixture of resin and hardener. The resin cures, taking up the shape of the mould and bonding to the fibres. Many composites are based on epoxies, though there is now a trend to using the cheaper polyesters. Laying-up is a slow, labour-intensive job. It can be by-passed by using thermo plastics containing chopped fibres which can be injection moulded. The random chopped fibres are not quite as effective as laid-up continuous

fibres, which can be oriented to maximise their contribution to the strength. But the flow pattern in injection moulding helps to line the fibres up, so that clever mould design can give a stiff, strong product. The technique is used increasingly for sports goods (tennis racquets, for instance) and light-weight hiking gear (like back-pack frames). Making good fibre-composites is not easy; large companies have been bankrupted by their failure to do so. The technology is better understood than it used to be; the tricks can be found in the books listed under further reading. But suppose you can make them, you still have to know how to use them. That needs understanding of their properties, which we examine next. The important properties of three common composites, where they are compared with high-strength steel and high-strength aluminium alloy of the sort used for aircraft structures.

References:

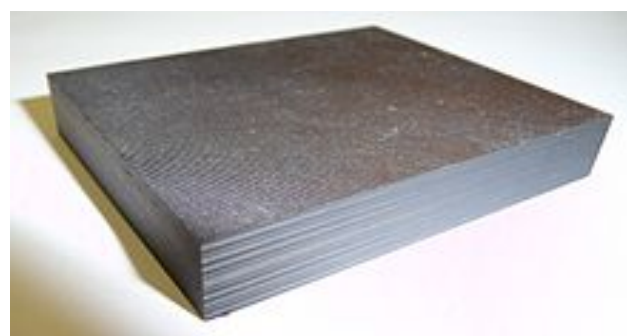
1. M. R. Piggott, Load Bearing Fibre Composites, Pergamon Press, 1980.
2. A. F. Johnson, Engineering Design Properties of GFRP, British Plastics Federation, 1974.
3. M. Grayson (editor), Encyclopedia of Composite Materials and Components, Wiley, 1983

Laminated Composites:

In materials science, a **composite laminate** is an assembly of layers of fibrous composite materials which can be joined to provide required engineering properties, including in-plane stiffness, bending stiffness, strength, and coefficient of thermal expansion.

The individual layers consist of high-modulus, high-strength fibers in a polymeric, metallic, or ceramic matrix material. Typical fibers used include cellulose, graphite, glass, boron, and silicon carbide, and some matrix materials are epoxies, polyimides, aluminium, titanium, and alumina.

Layers of different materials may be used, resulting in a hybrid laminate. The individual layers generally are orthotropic (that is, with principal properties in orthogonal directions) or transversely isotropic (with isotropic properties in the transverse plane) with the laminate then exhibiting anisotropic (with variable direction of principal properties), orthotropic, or quasi-isotropic properties. Quasi-isotropic laminates exhibit isotropic (that is, independent of direction) in plane response but are not restricted to isotropic out-of-plane (bending) response. Depending upon the stacking sequence of the individual layers, the laminate may exhibit coupling between in plane and out-of-plane response. An example of bending-stretching coupling is the presence of curvature developing as a result of in-plane loading.



References:

1. Gürdal et al. (1999), Design and optimisation of laminated composite materials, Wiley, ISBN 978-0471252764.

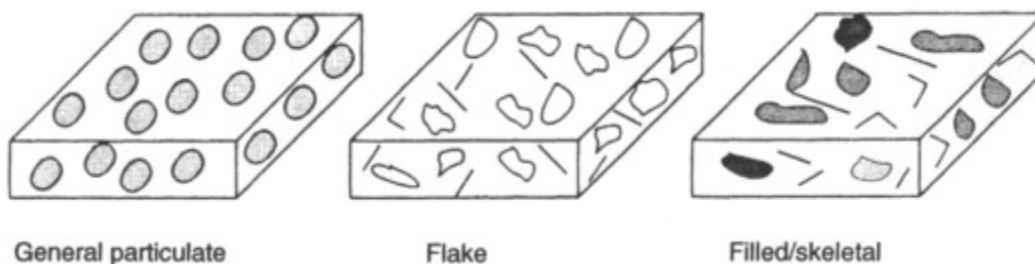
Particulate composites:

A particulate composite is characterized as being composed of particles suspended in a matrix. Particles can have virtually any shape, size or configuration. Examples of well-known particulate composites are concrete and particle board. There are two subclasses of particulates: flake and filled/skeletal

Flake. A flake composite is generally composed of flakes with large ratios of platform area to thickness, suspended in a matrix material (particle board, for example).

Filled /Skeletal. A filled/skeletal composite is composed of a continuous skeletal matrix filled by a second material: for example, a honeycomb core filled with an insulating material.

The response of a particulate composite can be either anisotropic or orthotropic. Such composites are used for many applications in which strength is not a significant component of the design. A schematic of several types of particulate composites is shown in



Particulate composites are attractive economically and a number of systems have received attention. One such system is SiC/TiO₂. Edwards (1994) has examined the residual stresses in 10%(10 μ m) SiC/(1 μ m)TiO₂ after pressing and sintering at 1100 °C, with and without a 1 minute dwell period. Using a method similar to that used by Majumdar *et al.* above, he found that the stress-free temperature dropped from 950 to 650 °C upon employing the dwell period. As a consequence, the room temperature stresses in the SiC phase fell from -700 to -500 MPa.

Zirconias containing particulate composites are of great interest, because of the potential for increasing the fracture toughness through exploitation of the structural phase transformation from the tetragonal to monoclinic form. Wang *et al.* (1984) have looked at the thermal residual strains

in $\text{Al}_2\text{O}_3/\text{ZrO}_2$ composites as a function of the ZrO_2 volume fraction (see Figure 9). That the stress given by $(1-f) \langle \sigma \rangle_{\text{Al}_2\text{O}_3} + f \langle \sigma \rangle_{\text{ZrO}_2}$ is very nearly zero in agreement with eqn [23] shows that the stress field is in self-equilibrium. Monoclinic zirconia reinforced cubic zirconia matrix composites have also been the focus of study (Majumdar *et al.*, 1991). In this case large residual stresses were expected, both because of the transformation stresses caused by the tetragonal to monoclinic transition occurring at 1000°C , as well as through differential thermal contraction upon cooling. While the residual stresses were smaller than expected, they were still significant (compressive particle stresses of around 1650 MPa). The micro mechanisms of micro cracking and creep have been proposed as causing the relaxation of stress.

Reference:

1. <https://www.sciencedirect.com/topics/materials-science/particulate-composite>

Application of Composite Materials:

1. In Aerospace-Approximately 50% component of the airspace is made from composites. The primary benefits that composite components are reduced weight and assembly simplification. The large scale use of composites in current program of development of helicopters, military fighter aircraft, small and big civil transport aircraft, satellites, launch vehicles and missiles. Various components of aircraft are fabricated by composites, e.g. rudder, spoilers, airbrakes, elevators, LG doors, engine cowlings, keel beam, rear bulkhead, wing ribs, main wings, turbine engine fan blades, propellers, Interior components etc.
2. In Automotive -Composites are being considered to make low weight, safer and more fuel-efficient vehicles. A composite is composed of a high strength fiber (carbon or glass) in a matrix material (epoxy polymer) that when combined provides magnify properties compared with the individual materials by themselves. Many components like steering wheel, dashboard, seat, roof, hatch, mats, energy absorber, instrument cluster, interior and exterior panel, leaf spring, wheels, engine cover etc. fabricated by composite materials.
3. In Medical-A composite is a nonviable material used in a medical device and intended to interact with biological system. Over the centuries, advancement in synthetic materials, surgical technique and sterilization methods have permitted the use of composite material in many ways. Medical practice today utilizes a large number of devices and implants. Composites in the form of sutures, bone and joint replacements, vascular grafts, heart valves, intraocular lenses, dental implants, pacemakers, biosensors, artificial hearts etc. widely used to replace and/or restore the function of disturbed or degenerated tissues or organs, to improve function, to assist in healing, to correct abnormalities and thus improve the quality of life of the patients.
4. In Electrical field-Composite materials have strength, high modulus; electronic composites emphasize high thermal conductivity, low thermal expansion, low dielectric constant and high/low electrical conductivity depending on the particular electronic applications. Electronics composites can use expensive fillers, such as silver particles, which serve to provide high electrical conductivity. The application of composites in electronics include interconnections, printed circuit boards, interlayer dielectrics, die attach, lids, thermal interface materials, electrical contacts, connectors, heat sinks, housings etc.
5. In Sports-Composite materials are used in sports equipment because they offer ease of transport, resistance, low weight, low maintenance and durability. Initially, natural materials, like wood, were used due to its good shock absorption, but these materials had some drawbacks. The

anisotropic nature resulted in low resistance and the variation in properties and high moisture absorption allocate various deformations. The composite material has characteristics of fatigue resistance break resistance, superior thermo stability, friction resistance, abrasion resistance and vibration attenuation, and it has light weight, high strength and high design freedom, and can be processed and shaped easily, so it is widely used in sports equipment. There are various goods made of composite materials, including the planning boats, sailing boats, sailboards tennis rackets, badminton rackets, softball bats, ice hockey sticks, bows and arrows etc .

6. In Chemical Industry-Advantages of composites of fire resistance properties, lightweight, mold ability, and resistance to chemicals has made the material used in the chemical industry. Composites are extensively used in industrial gratings, scrubbers, ducting, piping, exhaust stacks, pumps & blowers, structural supports, storage tanks, columns, reactors etc. for alkaline & acidic environments. Some applications are drive shaft, fan blades, ducts, stacks, underground storage tanks, casings, composite vessels etc. Internationally, composites applications in chemical industry are a relatively small segment in relation to the total usage of composites.

7. Other-Composites have long been used in the construction for industrial supports, buildings, long span roof structures, tanks, bridge components and complete bridge systems. With composites exhibiting excellent resistance to the marine environment. With the help of composite we make light weight doors, window, furniture, building, bridge etc. for domestic and construction purpose.

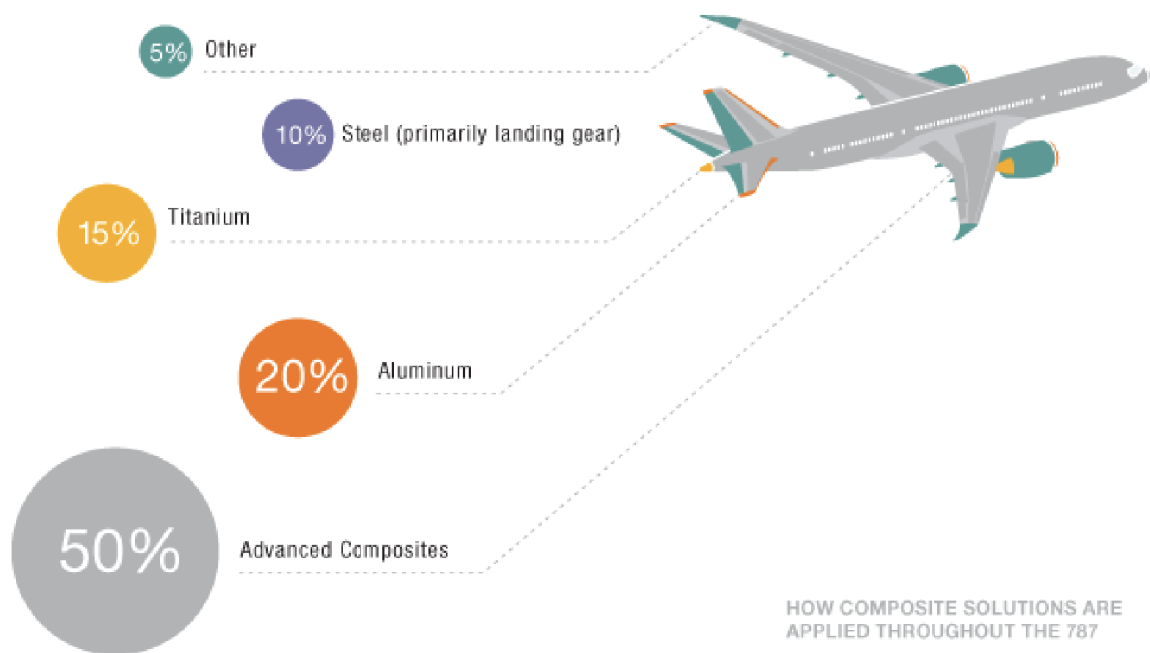
References:

1. Satyanarayana, K.G., Sukumaran, K., Mukherjee, P.S., Pavithran, C. and Pillai, S.G.K., "International conference on low cost housing for developing countries", pp. 171-181, (Rookree, 1984).
2. Satyanarayana, K.G., Pai, B.C., Sukumaran, K, and Pillai, S.G.K., "Lingocellulosic fiber reinforced polymer composites", (New York, Marcel Decker, Vol.1, pp.339.
3. Hinrichsen, J, "The Material Down-selection Process for A3XX, CAES", 19th European Conference on Materials for Aerospace Applications,Munich, pp.6-8, 2000.
4. Blythe, A.R., "Electrical properties of polymer", Cambridge University Press Cambridge, London, (1979).

Future Potential of Composites: The composites world, to me, seems like an untouched domain with an extreme abundance of applications and possibilities just waiting to be explored. Market forecasting data has showcased a few industries that will have sizeable increases in the use of composite materials. These industries will be transitioning from traditional materials like steel or iron in their products to more advanced composite materials for several reasons that I will discuss later. Composite materials are not new to the market segments I will be examining, but they will see large increases in the amount of composite materials used as well as new applications creating possibilities that were once impossible.

As a whole, the advanced composites market is expected to increase at a healthy rate. The value of the advanced composite market was at \$16.3 billion in 2014 with projected growth at a CAGR of 7.2% leading up to 2020, in which North America held the largest share by volume of the market in 2014. A growth in the North America region will be largely attributed to the growing list of applications in the defence sector as well as the Aerospace industry, which will be my focus throughout this post.

Over the next decade, the aerospace market for composite materials is anticipated to grow at elevated rates in comparison to years past. Next generation aircraft, like the Boeing 787, are exhibiting these new applications that were once untouched by the composites industry. The Boeing 787 shaves off nearly 20 percent of the aircraft's frame weight in comparison to the typical aluminium frame by using these advanced materials. With the weight savings, further travel is possible due to the fuel consumption reductions that this new composite driven airplane uses. The usage of composite materials not only reduces fuel consumption and resists corrosion, but lightning protection is now a possibility due to a composite coating that a group of researchers is working on.



The right material for the right application. Without preconceived ideas, Boeing engineers were able to specify the optimum material for specific applications throughout the airframe.

The defence sector will also see this increase in uses and applications of advanced composite materials. Though many of these will come in the form of aircraft and aircraft components for light weighting and strength as explained above, there are other new applications to assist the defence sector. From new, advanced personal protective vests that stop bullets to a new line of armor for vehicles for both personal security and military applications. For example, Morgan Advanced Materials (MAM) uses these composites to make lightweight armor products including helmets, ballistic protection jackets, and vests. According to MAM, their armor is 50 percent lighter than the comparable steel armor, while allowing for included threat protection like mine blasts, burning fluids, and fragmentation.

Mar-Bal, Inc. (MBI) is part of several different marketplaces that are growing and utilizing the beneficial advantages that these cutting-edge materials possess. One of the markets focused by MBI currently is the household appliances market. The Global Household Cooking & Appliance Manufacturing market is expected to see growth of 3.8% annually to \$230.3 billion in the years leading to 2020. The solidification of the US economy and the rise in flexible spending has created more demand for the household appliances with new finishes and sleek detailing. Composites have been used in the marketplace for the past several years, but have seen an increase due to the materials corrosion resistant and have heat-dissipating properties while keeping high aesthetics in mind. MBI's popular product line, THERMITAL™, provides a physical vapor deposition (PVD) finish, which allows for a metal-like surface appearance and feel. "Our solution won out because inside refrigerators there's condensation, and with condensation comes the potential for corrosion and rust," says Ron Poff, Director of Global marketing and Brands for Mar-Bal. "Thermoset composites gave the customer the look and feel of die cast, but with the added benefit of corrosion resistance."

The future of the advanced composite materials is extremely bright. Aerospace and defence are just two of the many market segments that will see a large increase in the use of these materials. As time progresses, these lighter weights, incredibly strong materials will dominate the materials

used in almost any given industry. With increased demand for these specialty materials, prices will be forced down, and the technology to make these advanced materials will become more readily available.

Reference:

1. <https://www.mar-bal.com/future-advanced-composite-materials>.

Unit-

II

Topic-

Metal Matrix Composites:

Sub-Topic-

Reinforcement materials, types, characteristics and selection of base metals. Need for production MMC's and its application. Fabrication Process for MMC's: Powder metallurgy technique, liquid metallurgy technique and secondary processing, special fabrication techniques.

Metal Matrix Composites : A metal matrix composite (MMC) is composite material with at least two constituent parts, one being a metal necessarily, the other material may be a different metal or another material, such as a ceramic or organic compound. When at least three materials are present, it is called a hybrid composite. An MMC is complementary to a cermet.

Composition: MMCs are made by dispersing a reinforcing material into a metal matrix. The reinforcement surface can be coated to prevent a chemical reaction with the matrix. For example, carbon fibers are commonly used in aluminium matrix to synthesize composites showing low density and high strength. However, carbon reacts with aluminium to generate a brittle and water-soluble compound Al_4C_3 on the surface of the fiber. To prevent this reaction, the carbon fibers are coated with nickel or titanium boride.

Matrix: The matrix is the monolithic material into which the reinforcement is embedded, and is completely continuous. This means that there is a path through the matrix to any point in the material, unlike two materials sandwiched together. In structural applications, the matrix is usually a lighter metal such as aluminium, magnesium, or titanium, and provides a compliant support for the reinforcement. In high-temperature applications, cobalt and cobalt–nickel alloy matrices are common.

Reinforcement: The reinforcement material is embedded into a matrix. The reinforcement does not always serve a purely structural task (reinforcing the compound), but is also used to change physical properties such as wear resistance, friction coefficient, or thermal conductivity. The reinforcement can be either continuous or discontinuous. Discontinuous MMCs can be isotropic and can be worked with standard metalworking techniques, such as extrusion, forging, or rolling.

In addition, they may be machined using conventional techniques, but commonly would need the use of polycrystalline diamond tooling (PCD).

Continuous reinforcement uses monofilament wires or fibers such as carbon fiber or silicon carbide. Because the fibers are embedded into the matrix in a certain direction, the result is an anisotropic structure in which the alignment of the material affects its strength. One of the first MMCs used boron filament as reinforcement. Discontinuous reinforcement uses "whiskers", short fibers, or particles. The most common reinforcing materials in this category are alumina and silicon carbide.

Manufacturing and forming methods: MMC manufacturing can be broken into three types—solid, liquid, and vapour.

Solid state methods

- Powder blending and consolidation (powder metallurgy): Powdered metal and discontinuous reinforcement are mixed and then bonded through a process of compaction, degassing, and thermo-mechanical treatment (possibly via hot isostatic pressing (HIP) or extrusion)
- Foil diffusion bonding: Layers of metal foil are sandwiched with long fibers, and then pressed through to form a matrix

Liquid state methods

- Electroplating and electroforming: A solution containing metal ions loaded with reinforcing particles is co-deposited forming a composite material
- Stir casting: Discontinuous reinforcement is stirred into molten metal, which is allowed to solidify
- Pressure infiltration: Molten metal is infiltrated into the reinforcement through use a kind of pressure such as gas pressure
- Squeeze casting: Molten metal is injected into a form with fibers pre-placed inside it
- Spray deposition: Molten metal is sprayed onto a continuous fiber substrate
- Reactive processing: A chemical reaction occurs, with one of the reactants forming the matrix and the other the reinforcement

Semi-solid state methods

- Semi-solid powder processing: Powder mixture is heated up to semi-solid state and pressure is applied to form the composites.

Vapor deposition

- Physical vapor deposition: The fiber is passed through a thick cloud of vaporized metal, coating it.

In-situ fabrication technique

Controlled unidirectional solidification of a eutectic alloy can result in a two-phase microstructure with one of the phases, present in lamellar or fiber form, distributed in the matrix.

Residual stress: MMCs are fabricated at elevated temperatures, which is an essential condition for diffusion bonding of the fiber/matrix interface. Later on, when they are cooled down to the ambient temperature, residual stresses (RS) are generated in the composite due to the mismatch between the coefficients of the metal matrix and fiber. The manufacturing RS significantly influence the mechanical behaviour of the MMCs in all loading conditions. In some cases, thermal RS are high enough to initiate plastic deformation within the matrix during the manufacturing process.

Applications:

- High performance tungsten carbide cutting tools are made from a tough cobalt matrix cementing the hard tungsten carbide particles; lower performance tools can use other metals such as bronze as the matrix.
- Some tank armors may be made from metal matrix composites, probably steel reinforced with boron nitride, which is a good reinforcement for steel because it is very stiff and it does not dissolve in molten steel.
- Some automotive disc brakes use MMCs. Early Lotus Elise models used aluminium MMC rotors, but they have less than optimal heat properties, and Lotus has since switched back to cast iron. Modern high-performance sport cars, such as those built by Porsche, use rotors made of carbon fiber within a silicon carbide matrix because of its high specific heat and thermal conductivity. 3M developed a preformed aluminium matrix insert for strengthening cast aluminium disc brake callipers, reducing weight by half compared to cast iron while retaining similar stiffness. 3M has also used alumina preforms for AMC pushrods.
- Ford offers a Metal Matrix Composite (MMC) driveshaft upgrade. The MMC driveshaft is made of an aluminium matrix reinforced with boron carbide, allowing the critical speed of the driveshaft to be raised by reducing inertia. The MMC driveshaft has become a common modification for racers, allowing the top speed to be increased far beyond the safe operating speeds of a standard aluminium driveshaft.
- Honda has used aluminium metal matrix composite cylinder liners in some of their engines, including the B21A1, H22A and H23A, F20C and F22C, and the C32B used in the NSX.

- Toyota has since used metal matrix composites in the Yamaha-designed 2ZZ-GE engine which is used in the later Lotus Elise S2 versions as well as Toyota car models, including the eponymous Toyota Matrix. Porsche also uses MMCs to reinforce the engine's cylinder sleeves in the Boxster and 911.
- The F-16 Fighting Falcon uses monofilament silicon carbide fibers in a titanium matrix for a structural component of the jet's landing gear.
- Specialized Bicycles has used aluminium MMC compounds for its top of the range bicycle frames for several years. Griffen Bicycles also made boron carbide-aluminium MMC bike frames, and Univega briefly did so as well.
- Some equipment in particle accelerators such as Radio Frequency Quadrupoles (RFQs) or electron targets use copper MMC compounds such as Glidcop to retain the material properties of copper at high temperatures and radiation levels.^{[9][10]}
- Copper-silver alloy matrix containing 55% by volume diamond particles, known as Dymalloy, is used as a substrate for high-power, high-density multi-chip modules in electronics for its very high thermal conductivity. AlSiC is an aluminium-silicon carbide composite for similar applications.
- Aluminium-Graphite composites are used in power electronic modules because of their high thermal conductivity, the adjustable coefficient of thermal expansion and the low density.

MMCs are nearly always more expensive than the more conventional materials they are replacing. As a result, they are found where improved properties and performance can justify the added cost. Today these applications are found most often in aircraft components, space systems and high-end or "boutique" sports equipment. The scope of applications will certainly increase as manufacturing costs are reduced.

In comparison with conventional polymer matrix composites, MMCs are resistant to fire, can operate in wider range of temperatures, do not absorb moisture, have better electrical and thermal conductivity, are resistant to radiation damage, and do not display out gassing. On the other hand, MMCs tend to be more expensive, the fiber-reinforced materials may be difficult to fabricate, and the available experience in use is limited.

References:

1. Materials science and Engineering, an introduction. William D. Callister Jr, 7th Ed, Wiley and sons publishing.

2. Wu, Yufeng; Gap; Kim, Yong (2011). "Carbon nano tube reinforced aluminium composite fabricated by semi-solid powder processing". Journal of Materials Processing Technology. 211 (8): 1341–1347. doi:10.1016/j.jmatprotec.2011.03.007.
3. University of Virginia's Directed Vapor Deposition (DVD) technology.
4. Aluminium matrix composite (AMC) inserts for reinforced brake calipers (Archived)

Unit-

III

Topic-

Polymer matrix composites (PMC)

Sub-Topic-

Reinforcement materials, types, characteristics and selection base matrix, process design of PMC's and applications. Processing of Thermoplastic composites: Types of processing methods, solution, film, lamination, sandwich etc., Advantages and limitations of each method.

Polymer matrix composites (PMC): A polymer matrix composite (PMC) is a composite material composed of a variety of short or continuous fibers bound together by an organic polymer matrix. PMCs are designed to transfer loads between fibers of a matrix. Some of the advantages with PMCs include their lightweight, high stiffness and their high strength along the direction of their reinforcements. Other advantages are good abrasion resistance and good corrosion resistance.

Matrix Materials: The function of the matrix in PMCs is to bond the fibers together and transfer loads between them. PMCs matrices are typically either thermosets or thermoplastics. Thermoset are by far the predominant type in use today. Thermoset are subdivided into several resin systems including epoxies, phenolics, polyurethanes, and polyamides. Of these, epoxy systems currently dominate the advanced composite industry.

Thermosets: Thermoset resins require addition of a curing agent or hardener and impregnation onto a reinforcing material, followed by a curing step to produce a cured or finished part. Once cured, the part cannot be changed or reformed, except for finishing. Some of the more common thermosets include epoxy, polyurethanes, phenolic and amino resins, bismaleimides (BMI, polyimides), polyamides.

Of these, epoxies are the most commonly used in the industry. Epoxy resins have been in use in U.S. industry for over 40 years. Epoxy compounds are also referred to as glycidyl compounds. The epoxy molecule can also be expanded or cross-linked with other molecules to form a wide variety of resin products, each with distinct performance characteristics. These resins range from low-viscosity liquids to high-molecular weight solids. Typically they are high-viscosity liquids.

The second of the essential ingredients of an advanced composite system is the curing agent or hardener. These compounds are very important because they control the reaction rate and determine the performance characteristics of the finished part. Since these compounds act as catalysts for the reaction, they must contain active sites on their molecules. Some of the most commonly used curing agents in the advanced composite industry are the aromatic amines. Two of the most common are methylene-dianiline (MDA) and sulfonyldianiline (DDS). SiC–SiC matrix composites are a high-temperature ceramic matrix processed from preceramic polymers (polymeric SiC precursors) to infiltrate a fibrous preform to create a SiC matrix.

Several other types of curing agents are also used in the advanced composite industry. These include aliphatic and cycloaliphatic amines, polyaminoamides, amides, and anhydrides. Again, the choice of curing agent depends on the cure and performance characteristics desired for the finished part. Polyurethanes are another group of resins used in advanced composite processes. These compounds are formed by reacting the polyol component with an isocyanate compound, typically toluene diisocyanate (TDI); methylene diisocyanate (MDI) and hexamethylene diisocyanate (HDI) are also widely used. Phenolic and amino resins are another group of PMC resins. The bismaleimides and polyamides are relative newcomers to the advanced composite industry and have not been studied to the extent of the other resins.

Thermoplastics: Thermoplastics currently represent a relatively small part of the PMC industry. They are typically supplied as nonreactive solids (no chemical reaction occurs during processing) and require only heat and pressure to form the finished part. Unlike the thermosets, the thermoplastics can usually be reheated and reformed into another shape, if desired

Dispersed Materials

Fibers: Fiber-reinforced PMCs contain about 60 percent reinforcing fiber by volume. The fibers that are commonly found and used within PMCs include fiberglass, graphite and aramid. Fiberglass has a relatively low stiffness at the same time exhibits a competitive tensile strength compared to other fibers. The cost of fiberglass is also dramatically lower than the other fibers which are why fiberglass is one of the most widely used fiber. The reinforcing fibers have their highest mechanical properties along their lengths rather than their widths. Thus, the reinforcing fibers may be arranged and oriented in different forms and directions to provide different physical properties and advantages based on the application

Drawbacks of Polymer matrix :

1. Environmental degradation
2. Moisture absorption from environment causes swelling in the polymer as well as a decrease of Tg.
3. The moisture absorption increases at moderately high temperatures. These hydrothermal effects can lead to internal stresses in the presence of fibres in polymer composites.
4. A thermal mismatch between polymer and fibre may cause cracking or debonding at the interface.

Reference:

1. https://en.wikipedia.org/wiki/Polymer_matrix_composite.

Production of polymer matrix composites: Polymers are reinforced with fibres which are 8 to 12 μm in diameter either as continuous single or chopped multi-filaments that are woven into cloth and other types of preformed textiles. These fibres are then impregnated into the matrix polymer in liquid form by injection, extrusion, pressing or stamping and then cured to produce the final composite.

During the fabrication and shaping of polymer matrix composites into finished products, often the formation of the material itself is incorporated in the fabrication process. These processes include

- Hand lay-up
- Vacuum moulding
- Spray lay-up
- Pultrusion
- Resin transfer moulding (RTM)
- Filament winding

PMC used for: PMCs are regarded due to their low cost and straightforward fabrication methods. Applications for PMCs include.

Automotive industry - Body panels, leaf springs, driveshaft, bumpers, doors, racing car bodies, and so on.

Aircraft and aerospace industry - Used in the construction of structural parts for military aircraft, space shuttles, and satellite systems. The main purposes of using PMCs are to reduce aircraft weight, which can improve its performance, and to reduce its costs.

Marine - Fibreglass boat bodies, as well as canoes and kayaks.

Sports goods - Used in performance footwear, sports equipment and other sporting goods because of their lightweight and high-strength properties.

Biomedical applications - Medical implants, orthopaedic devices, MRI scanners, X-ray tables, and prosthetics.

Electrical - Panels, housing, switchgear, insulators, and connectors. It also covers electronic devices like capacitors, Li-ion and flexible batteries and covers for digital portable equipment like headphones, etc.

Protective equipment - Since polymer matrix composites can withstand extreme hot or cold and other hazardous conditions, they are often made as raw materials for bulletproof vests and other armour.

Industrial - Chemical storage tanks, pressure vessels, pump housing, and valves. PMCs are also used in impellers, blades, blower and pump housings, and motor covers.

Structural - Polymer matrix composites are used to repair bridges and other construction materials and equipment like booms and cranes.

References:

1. M. Akay, 2015, An introduction to polymer matrix composites,” from: https://www.academia.edu/37778336/An_introduction_to_polymer_matrix_composites
2. S.G. Advani, K.T. Hsiao, 2012, Manufacturing Techniques for Polymer Matrix Composites (PMCs), UK: Woodhead Publishing ltd.
3. Chapter 3 Polymer Matrix Composites,” n.d., from: <https://www.princeton.edu/~ota/disk2/1988/8801/880106.PDF>.
4. Processing of polymer composites,” 2012, from: <http://www-materials.eng.cam.ac.uk/3C1archive/handout6.pdf>.

Unit-

IV

Topic-

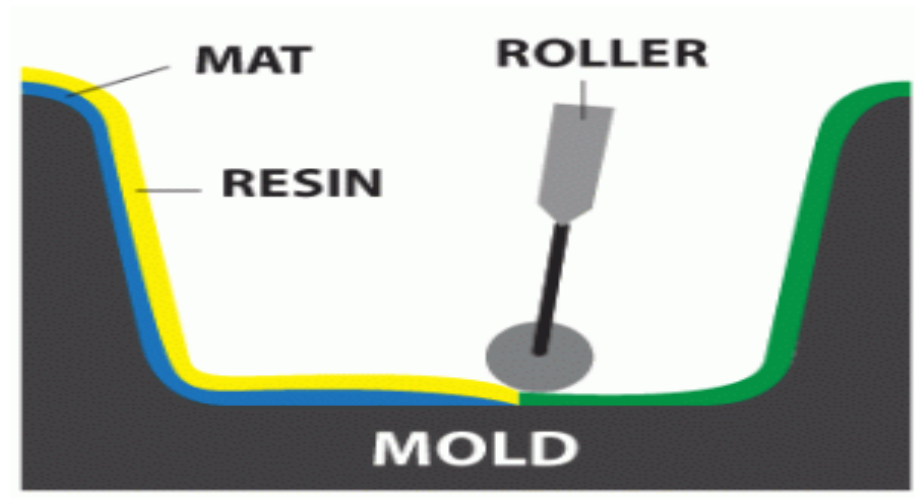
Processing of Thermoses composites

Sub-Topic-

Hand layup method, compression and transfer moulding, pressure and vacuum bag process, filament winding, protrusion,

reinforced RIM, RRIM, Injection moulding of thermoses, SMC and DMC, Advantages and limitations of each method. Mechanical testing of composites: Tensile testing, Compressive testing, impact, bending strength. Basic concepts of fracture mechanism, Inter laminar shear testing, Fracture testing.

Hand layup method:



Overview

Hand lay-up is a molding process where fiber reinforcements are placed by hand then wet with resin.

Raw Material

The manual nature of this process allows for almost any reinforcing material to be considered, chopped strand or mat. Similarly, the resin and catalyst blend can be manipulated to allow for ideal processing conditions.

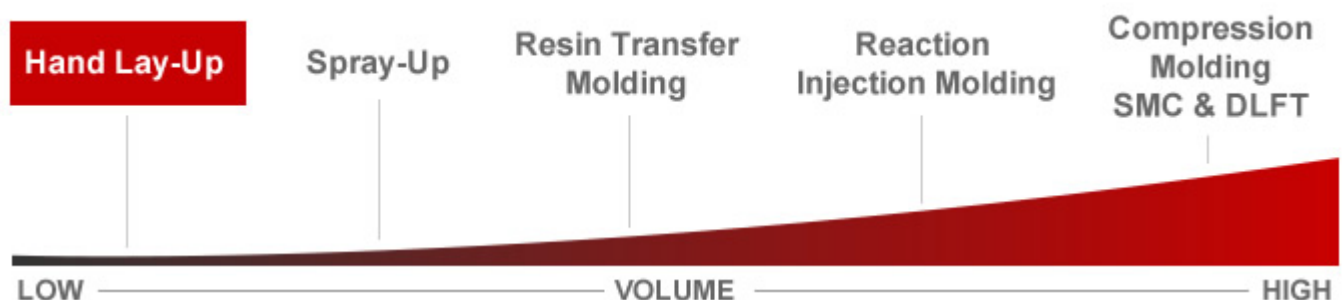
Tooling

Single sided; typically composite tools are used for this process

Finishing

Hand layup parts normally have one visible, smooth side. A gel coat can be added during molding to produce cosmetic finished surfaces.

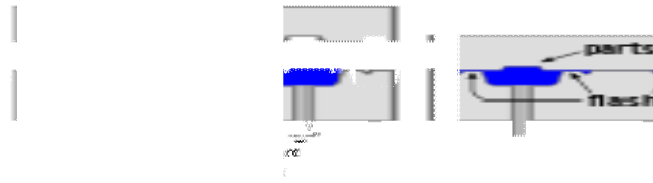
Benefits	Limitations
<ul style="list-style-type: none"> Flexibility in material design Lower cost to tool Ideal for lower volumes 	<ul style="list-style-type: none"> Higher cycle times Produces only one smooth surface Somewhat inconsistent in part thickness



Reference:

1. <http://www.coremt.com/processes/hand-lay-up>.

Compression Moulding: Compression Moulding is a method of moulding in which the moulding material, generally preheated, is first placed in an open, heated mould cavity. The mould is closed with a top force or plug member, pressure is applied to force the material into contact with all mould areas, while heat and pressure are maintained until the moulding material has cured. The process employs thermosetting resins in a partially cured stage, either in the form of granules, putty-like masses, or preforms.



Compression molding - simplified diagram of the process

Compression molding is a high-volume, high-pressure method suitable for molding complex, high-strength fiberglass reinforcements. Advanced composite thermoplastics can also be compression molded with unidirectional tapes, woven fabrics, randomly oriented fiber mat or chopped strand. The advantage of compression molding is its ability to mold large, fairly intricate parts. Also, it is one of the lowest cost molding methods compared with other methods such as transfer molding and injection molding; moreover it wastes relatively little material, giving it an advantage when working with expensive compounds.

However, compression molding often provides poor product consistency and difficulty in controlling flashing, and it is not suitable for some types of parts. Fewer knit lines are produced and a smaller amount of fiber-length degradation is noticeable when compared to injection molding. Compression-molding is also suitable for ultra-large basic shape production in sizes beyond the capacity of extrusion techniques. Materials that are typically manufactured through compression molding include: Polyester fiberglass resin systems (SMC/BMC), Torlon, Vespel, Poly(p-phenylene sulfide) (PPS), and many grades of PEEK.

Thermoplastic matrices are commonplace in mass production industries. One significant example are automotive applications where the leading technologies are long fibre reinforced thermoplastics (LFT) and glass fiber mat reinforced thermoplastics (GMT).

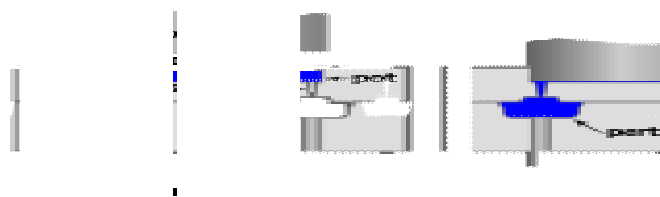
In compression molding there are six important considerations that an engineer should bear in mind

- Determining the proper amount of material.
- Determining the minimum amount of energy required to heat the material.
- Determining the minimum time required to heat the material.
- Determining the appropriate heating technique.
- Predicting the required force, to ensure that shot attains the proper shape.
- Designing the mold for rapid cooling after the material has been compressed into the mold.

References:

1. Introduction to Compression Molding". eFunda. Retrieved 19 March 2013.
2. What is Compression Moulding?". *Coventive Composites*. Retrieved 2018-10-01.

Transfer molding: Transfer molding (BrE moulding) is a manufacturing process in which casting material is forced into a mold. Transfer molding is different from compression molding in that the mold is enclosed [Hayward] rather than open to the fill plunger resulting in higher dimensional tolerances and less environmental impact. Compared to injection molding, transfer molding uses higher pressures to uniformly fill the mold cavity. This allows thicker reinforcing fiber matrices to be more completely saturated by resin. Furthermore, unlike injection molding the transfer mold casting material may start the process as a solid. This can reduce equipment costs and time dependency. The transfer process may have a slower fill rate than an equivalent injection molding processes.

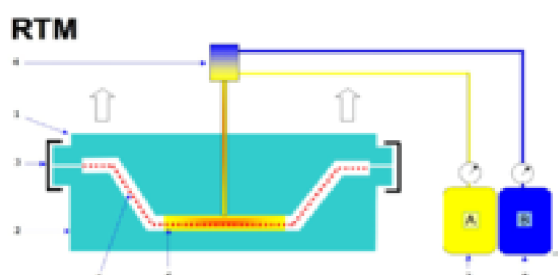


Transfer Molding basic process

Process: The mold interior surfaces may be gel-coated. If desired, the mold is first pre-loaded with a reinforcing fiber matrix or preform. Fiber content of a transfer molded composite can be as high as 60% by volume. The fill material may be a preheated solid or a liquid. It is loaded into a chamber known as the pot. A ram or plunger forces material from the pot into the heated mold cavity. If feed-stock is initially solid, the forcing pressure and mold temperature melt it. Standard mold features such as sprue channels, a flow gate and ejector pins may be used. The heated mold ensures that the flow remains liquid for complete filling. Once filled the mold can be cooled at a controlled rate for optimal thermoset curing

Variations: The industry identifies a variety of processes within the transfer molding category. There are areas of overlap and the distinctions between each method may not be clearly defined.

Resin transfer molding: Resin transfer molding (RTM) uses a liquid thermoset resin to saturate a fiber preform placed in a closed mold. The process is versatile and can fabricate products with embedded objects such as foam cores or other components in addition to the fiber preform.



Resin Transfer Molding

Vacuum assisted resin transfer molding: Vacuum assisted transfer molding (VARTM) uses a partial vacuum on one side of a fiber mat to pull the resin in for complete saturation. VARTM uses lower plunger forces which allows molding to be carried out with cheaper equipment. The use of a vacuum may allow the resin to adequately flow and or cure without heating. This temperature independence allows thicker fiber preforms and larger product geometries to be economical. VARTM can produce parts with less porosity than regular transfer molding with a proportional increase in casting strength

Micro transfer molding: Also called transfer micro molding, micro transfer molding is a process that uses a mold to form then transfer structures as small as 30 nm onto thin films and micro circuitry. Unlike normal scale transfer molding, the micro form can and is used with metals as well as non metals

References:

1. Kendall, K. N.; Rudd, C. D.; Owen, M. J.; Middleton, V. (1992-01-01). "Characterization of the resin transfer moulding process". *Composites Manufacturing*. 3 (4): 235–249. doi:10.1016/0956-7143(92)90111-7.
2. Heider, Dirk; Graf, A.; Fink, Bruce K.; Gillespie, Jr., John W. (1999-01-01). "Feedback control of the vacuum-assisted resin transfer molding (VARTM) process". *Process Control and Sensors for Manufacturing II*. 3589: 133–141. doi:10.1117/12.339956.

Vacuum Bag Process: By reducing the pressure inside the vacuum bag, external atmospheric pressure exerts force on the bag. The pressure on the laminate removes entrapped air, excess resin, and compacts the laminate, resulting in a higher percentage of fiber reinforcement.



Vacuum bagging can be used with wet-lay laminates and prepreg advanced composites. In wet lay up bagging the reinforcement is saturated using hand layup, then the vacuum bag is mounted on the mold and used to compact the laminate and remove air voids. In the case of pre-impregnated advanced composites molding, the prepreg material is laid up on the mold, the vacuum bag is mounted and the mold is heated or the mold is placed in an autoclave that applies both heat and external pressure, adding to the force of atmospheric pressure. The prepreg-vacuum bag-autoclave method is most often used to create advanced composite aircraft and military products.

Structures fabricated with traditional hand layup techniques can become resin rich and vacuum bagging can eliminate the problem. Additionally, complete fiber wet-out can be accomplished if the process is done correctly. Improved core bonding is also possible with vacuum bag processing.

Process: In the simplest form of vacuum bagging, a flexible film (PVA, nylon, mylar, or polyethylene) is placed over the wet **layup**, the edges are sealed, and a vacuum is drawn. A more advanced form of vacuum bagging places a release film over the laminate, followed by a bleeder ply of fiberglass cloth, non-woven nylon, polyester cloth, or other material that absorbs excess resin from the laminate. A breather ply of a non woven fabric is placed over the bleeder ply, and the vacuum bag is mounted over the entire assembly. Pulling a vacuum from within the bag uses atmospheric pressure to eliminate voids and force excess resin from the laminate. The addition of pressure further results in high fiber concentration and provides better adhesion between layers of sandwich construction. When laying non-contoured sheets of PVC foam or balsa into a female mold, vacuum bagging is the technique of choice to ensure proper secondary bonding of the core to the outer laminate.

Molds:

Molds are similar to those used for conventional open mold processes.

Reference:

1. <http://compositeslab.com/composites-manufacturing-processes/closed-molding/vacuum-bag-molding/>

Filament winding: Filament winding is a fabrication technique mainly used for manufacturing open (cylinders) or closed end structures (pressure vessels or tanks). This process involves winding filaments under tension over a rotating mandrel. The mandrel rotates around the spindle (Axis 1 or X: Spindle) while a delivery eye on a carriage (Axis 2 or Y: Horizontal) traverses horizontally in line with the axis of the rotating mandrel, laying down fibers in the desired pattern or angle. The most common filaments are glass or carbon and are impregnated in a bath with resin as they are wound onto the mandrel. Once the mandrel is completely covered to the desired thickness, the resin is cured. Depending on the resin system and its cure characteristics, often the rotating mandrel is placed in an oven or placed under radiant heaters until the part is cured. Once the resin has cured, the mandrel is removed or extracted, leaving the hollow final product. For some products such as gas bottles, the 'mandrel' is a permanent part of the finished product forming a liner to prevent gas leakage or as a barrier to protect the composite from the fluid to be stored.

Filament winding is well suited to automation, and there are many applications, such as pipe and small pressure vessel that are wound and cured without any human intervention. The controlled variables for winding are fibre type, resin content, wind angle, tow or bandwidth and thickness of the fiber bundle. The angle at which the fibre is wound has an effect on the properties of the final product. A high angle "hoop" will provide circumferential strength, while lower angle patterns (either polar or helical) will provide greater longitudinal / axial tensile strength.

Products currently being produced using this technique range from pipes, golf clubs, Reverse Osmosis Membrane Housings, oars, bicycle forks, bicycle rims, power and transmission poles, pressure vessels to missile casings, aircraft fuselages and lamp posts and yacht masts.

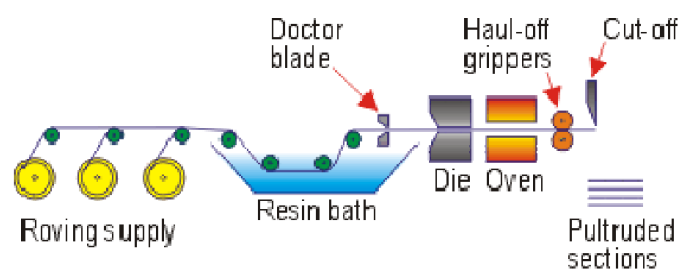
Filament winding machines: The simplest winding machines have two axes of motion, the mandrel rotation and the carriage travel (usually horizontal). Two axes machines are best suited to the manufacture of pipes only. For pressure vessels such as LPG or CNG containers (for example) it is normal to have a four axis winding machine. A four axes machine additionally has a radial (cross-feed) axis perpendicular to carriage travel and a rotating fibre payout head mounted to the cross-feed axis. The payout head rotation can be used to stop the fibre band twisting and thus varying in width during winding.

Machines with more than four axes can be used for advanced applications, six-axis winding machines usually have 3 linear and 3 rotation axes. Machines with more than 2 axes of motion have computer/CNC control, however these days new 2-axis machines mostly have numeric control. Computer controlled filament winding machines require the use of software to generate the winding patterns and machine paths, such software can normally be provided by filament winding machine manufacturers or by using independent products such as Cadfil or Cadwind, a review of programming techniques for CNC machines can be found in.^[3] An example of such a winding process can be found all throughout the web.

References:

1. Advanced Filament winding software.
2. Cadwind filament winding software.
3. Stan Peters, "Composite Filament Winding", 2011, ch 4, ISBN 1615037225.

Pultrusion: In the pultrusion process dry reinforcements are impregnated with a specially prepared low viscosity liquid resin system and drawn through a die heated to about 120-150°C where curing occurs. The solid laminate, which has assumed the shape of the die, is withdrawn by a series of haul off grippers and cut to length or coiled. Pultrusion is unique among the processes under consideration in that it is capable of producing complex components on a continuous basis. The process can basically produce any shape that can be extruded. It is also not allied to any one industry and applications range from civil engineering to electrical. These factors combine to give pultrusion one of the highest predicted growth rates of all composite process.



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Schematic of the pultrusion process.

Currently, the requirements of pultruders and their suppliers are stronger market development and higher productivity to capitalise on these projections. Faster production rates are one way of achieving this, but the industry itself has recognised the need for greater consistency of raw materials and process operation, coupled with the ability to monitor the product quality more closely.

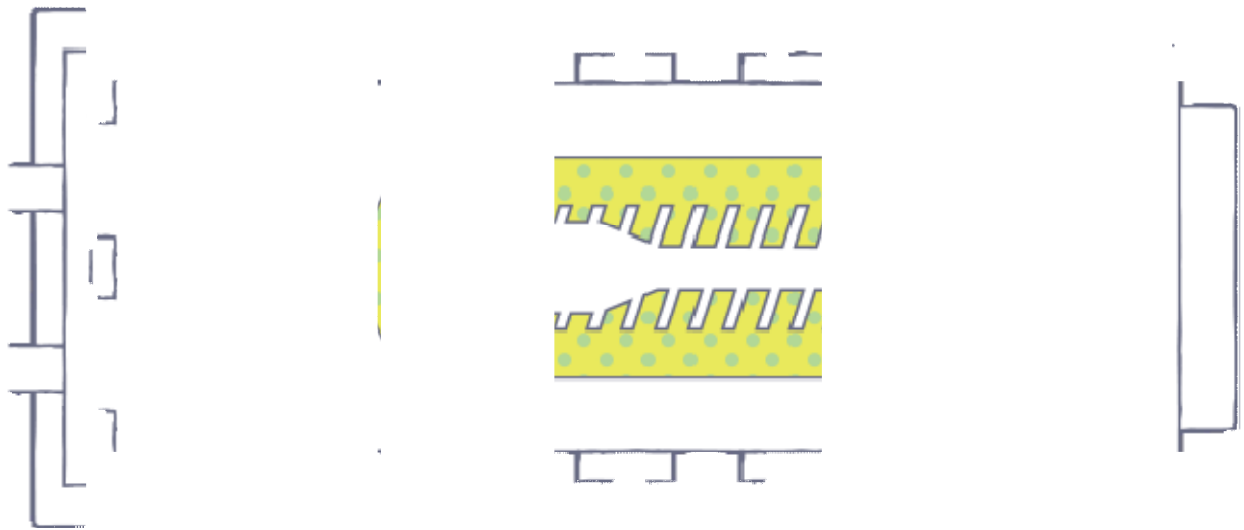
Reference:

1. <https://www.azom.com/article.aspx?ArticleID=352>.

Reinforced reaction injection molding (RRIM): is an alternative to the standard RIM process used to create lightweight, durable parts in larger sizes than ever before. It has made great strides within the automotive and transportation industries, allowing polyurethane injection molding to be used to make body panels, fascia, bumpers, spoilers, floor panels and more.

RRIM differentiates itself from standard reaction injection molding in that fibers, usually glass or carbon, are added to the resin during the molding process. The fibers reinforce the thermoset polymer and create a stronger, more impact resistant overall product.

Recently, long fiber injection molding (LFI) has emerged as a competitor to RRIM in terms of creating strong, lightweight, durable materials. However, RRIM is still a cost-efficient process which is extremely useful in the production of large parts for automobiles and heavy machinery.



The Reinforced Reaction Injection Molding Process: Reinforced reaction injection molding is a two-step process similar to its non-reinforced variation. It utilizes a closed mold, often created from a lightweight, low-cost material such as aluminum. Low pressure can be used, but high temperatures (often between 300 and 350 degrees Fahrenheit / 149 and 177 degrees Celsius) are required due to the presence of the reinforcing fibers.

As with standard RIM, the RRIM process begins with two components, polyol and isocyanate, stored in separate containers in liquid form. Both are injected into a mold, where they are mixed using a combination of low pressure and high velocity and then allowed to cure and form a solid thermoset polymer.

However, where RRIM differs from RIM is in the addition of reinforcing fibers to the polyol before its injection into the mold. Traditionally, glass fibers are used, however, recently, some RRIM processes have begun to use carbon fiber instead. Carbon fiber is both stronger and more lightweight than glass, and many believe that it may represent RRIM. However, glass is still a cost-effective option that is extremely viable especially in larger production runs.

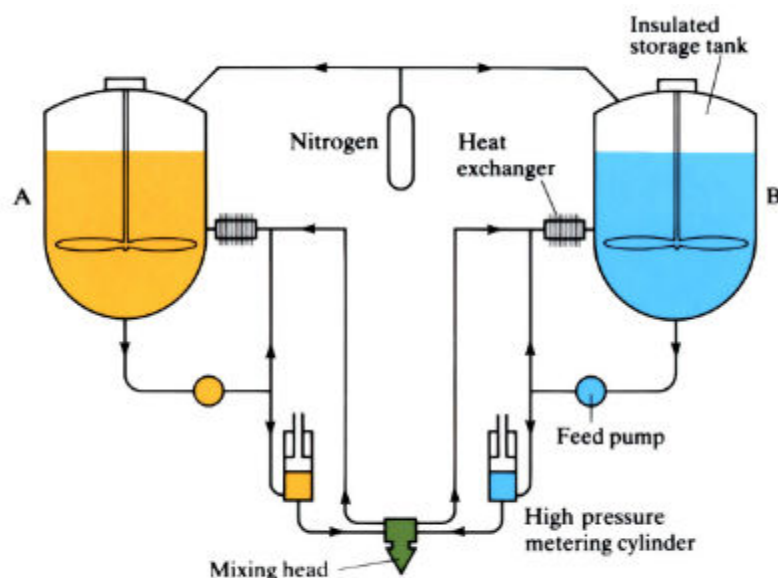
The fibers which are used during the RRIM process are either milled or chopped using a specialized machine before they are added to the polyol resin. The standard length of fibers utilized in RRIM-produced parts is extremely short, between 0.009 in and 0.02 in (0.2 and 0.5mm). In addition, some RRIM manufacturers choose to use glass flake rather than the standard chopped or milled fibers.

Only certain resins are compatible with the RRIM process. The most popular are epoxy, polyester, nylon, and polyurethane, with polyurethane emerging as the most widely used RRIM resin in recent years. Typically, thermoset polyurethane in an elastomeric variety is used rather than thermoplastic due to its shorter cure time and minimal need for post-mold processing.

Reference:

1. <https://romeorim.com/rrim>.

Reaction injection moulding (RIM): In reaction injection moulding, two types of monomers are mixed together before being injected into a mould, where they polymerise to form the plastic component. In reinforced reaction injection molding, reinforcing agents are added to the monomer mixture.

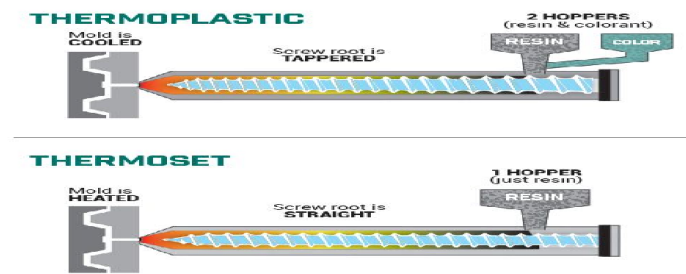


Advantages and disadvantages: Reaction injection molding can produce strong, flexible, lightweight parts which can easily be painted. It also has the advantage of quick cycle times compared to typical vacuum cast materials. The bi-component mixture injected into the mold has a much lower viscosity than molten thermoplastic polymers, therefore large, light-weight, and thin-walled items can be successfully RIM processed. This thinner mixture also requires less clamping forces, which leads to smaller equipment and ultimately lower capital expenditures. Another advantage of RIM processed foam is that a high-density skin is formed with a low-density core.

The disadvantages are slow cycle times, compared to injection molding, and expensive raw materials.

1. Ashida, Kaneyoshi (2006). Polyurethane and related foams: chemistry and technology. CRC Press. pp. 79–81. ISBN 978-1-58716-159-9.
2. Hutchinson, D.J. (Apr 1987). "Wash Studies of IMR Containing PU-RIM". Journal of Elastomers and Plastics. 19 (2): 109–119. doi:10.1177/009524438701900204. Retrieved 2010-09-15.

Injection Molding Process: In *thermoset* injection molding, cold material is injected into an extremely hot mold to create a part. This process cures the part so it can never be melted again.



In thermoplastic injection molding, plastic material is melted and injected into a mold to create a part. Once this part cools, the mold opens and the part drops out.

Availability

The very first plastic materials created were thermosets, but today, thermoset injection molding is not as common *except* with electrical contact applications and liquid silicone. Thus, thermoset injection molders are typically more difficult to find than thermoplastic injection molders.

Uses

Thermosets are most frequently used in situations where the part must be able to withstand high temperatures. Medical parts are a good example; dental tools with silicone handles and metal parts must be able to withstand the autoclaving process in order to be sanitized and reused over and over again, so the silicone must be thermoset. Higher voltage electrical applications also use thermosets.

Thermoplastic parts are more likely to be used for consumer plastics that either won't come in contact with high temperatures, like the plastic used in milk jugs, or will need to withstand moderately high temperatures. Certain polycarbonates, for example, are resistant to hot liquids and often used to make plastic to-go mugs—but they can still deform or melt at high-enough temperatures.

When considering thermoset vs. thermoplastic parts, you'll also want to consider whether the part will regularly be contact with certain chemistries. **If you're** manufacturing plastic casing for an EKG, for example, the material used will need to be able to withstand any hospital-grade chemical disinfectants.

Cost

The cost difference between thermoset and thermoplastic injection molding is not black and white. While the thermoset process is typically slower—which equates to a higher cost—the material difference in thermoplastic could range from 90 cents to \$10 per pound, depending on the properties. That said, cost considerations should not be enough to sway an engineer toward one type or another—a decision should be based far more on material properties and the functionality of the part needed.

Recyclability

Thermoplastics are created by melting pellets and then cooling them, which ensures the finished part can also be remelted. This allows thermoplastics to be recycled. Of course, certain properties

are no longer the same once you've melted a part down, so recycling a thermoplastic doesn't ensure you'll get the same quality plastic part—but it is worth noting.

Thermosets, on the other hand, cannot be remelted—so while thermoplastic parts could, hypothetically, be ground up and used in a sandbag, their recyclability factor is limited.

Reference:

1. <https://www.micronproducts.com/blog/thermoset-vs-thermoplastic>.

Sheet Moulding Compounds: Until the mid 1980s the moulding of Sheet Moulding Compounds (SMCs) was considered a slow process, but nevertheless capable of delivering both small and large non-structural components with a surface finish superior to all other composite processing methods. SMC can refer to both the material and the process. Complex shapes and details that

may not be possible with sheet metal can often be achieved relatively easily with SMC. The material usually comprises a filled polyester resin and glass fibre, which is either chopped, continuous, or a mix of both. When ready to mould, it normally has a consistency similar to that of thick putty, as it will be partially cured (B staged). As the name suggests, SMC is supplied in sheet form and in many respects can be considered a low performance prepreg. The sheets are essentially compression moulded to final shape.

Cycle Times

Commonly achieved cycle times were of the order of four minutes, resulting in processors operating multiple dies and ancillary equipment to keep up with production demands from their main customer, the automotive industry. Whilst this increases cost, it also enhances further the risk of process or part variations. A major deficiency of SMC components has been their structural inconsistency caused by uneven distribution of reinforcement during flow of the charge material. Much effort has been focused on the SMC moulding process in recent years, which has led to the 1 part per minute barrier being challenged. A key development enabling this improvement has been vacuum assisted moulding.

Vacuum Assisted Moulding

In conventional SMC processes, charge loading and press closure speeds are selected with the primary aim of forcing out entrapped air. Typically, 'stacks' of charge are placed in the mould, covering only 40% of the mould surface, and mould closure rates are in the order of 0.1 m.min⁻¹. With vacuum assistance, a one atmosphere vacuum is applied prior to closing the mould. This enables the charge to be spread to cover up to 90% of the mould surface and closure speeds at 0.9 m.min⁻¹ to be achieved without entrapping air.

The increase in mould surface coverage by the charge has several additional benefits associated with the reduced materials flow. Wave patterns and flow lines are eliminated and localised strength of components is enhanced due to better retention of fibre orientation. Specially introduced orientation is now feasible and a high strength SMC process is available where unidirectional fibres are arranged at specific locations. Raw material suppliers are developing faster reacting resin systems designed to take advantage of the lower flow, higher speed requirements. Equipment manufacturers are investing in automation and control of the process. These continuing activities are positioning SMC for a resurgence of interest in high volume semi structural applications.

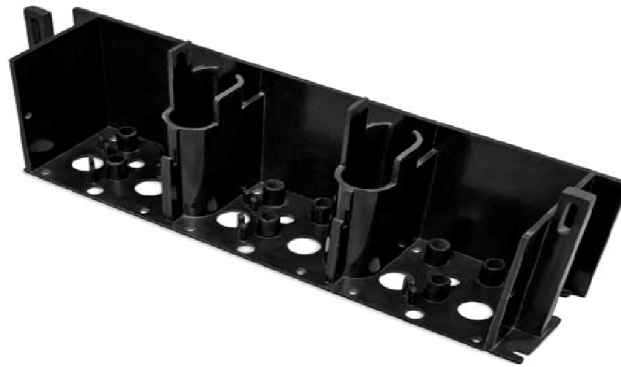
Reference:

1. <https://www.azom.com/article.aspx?ArticleID=352>.

Dough Molding Compound (DMC): Dough Molding Compound (DMC) is offered as a dough or extrudate for small but highly intricate moldings. Meant for electrical switchgear and other non-structural applications. Indigenously manufactured machinery combined with knowledge from Menzolit enable the production of high quality DMC. These molding compounds are reactive molding materials made primarily of Unsaturated Polyester Resin, Glass Fiber and Fillers. They form into solid components of desired sizes, shapes and properties on application of

heat and pressure in a matched die mold. Different techniques include compression and injection molding.

The demand for these products is expected to increase significantly as they are replacing conventional materials. Mahindra Composites is well equipped to cater to the Indian and global markets.



Reference:

1. <https://www.mahindrachie.com/what-we-do/technology/composites/products/compounds/dough-molding.html>

Mechanical testing of composites: The objective of the mechanical testing of composite is the determination of mechanical parameters such as strength and stiffness that will be later used on the design of a composite structure. Referring to the classical laminate theory, 5 elastic properties (E_1 , E_2 , ν_{12} , ν_{23} and G_{12}) are necessary to calculate the elastic behaviour of a long fibre reinforced composite. This can be reduced to 4 parameters (E_1 , E_2 , ν_{12} and G_{12}) in case of thin laminate composite in plane stress condition. These parameters are usually determined by testing flat coupons in tensile and shear modes.

For the ultimate strength and failure prediction of a composite, further tests on compression, shear and interlaminar strength are required for determining the proper parameters of the failure criterion model.

The test methods outlined in this section merely represent a small selection available to our customers. However, taking into account the terms and conditions of the corresponding standard (DIN, ASTM, EN, ISO,...), we perform measurements using only calibrated test equipment



Tensile testing

For tensile tests the fiber-reinforced plastic samples are prepared with tabs to prevent a break in the restraint. For the tensile measurement either a video extensometer or a plug-on extensometer available in various sizes is used. For the different power ranges we use force sensors. Mechanical parameters are determined and the thermal behavior is investigated in the temperature range between -269°C and 600°C.

Determination of interlaminar energy release rate

Here, the interlaminar energy release rate G_{IC} is determined, which describes the required total energy for the production of a defined crack growth under normal stress. The tensile force required for crack propagation is recorded on the traverse and a force-displacement diagram is created. The tests are carried out between -196°C and 600 °C taking into account DIN 65563.

Compression tests: modified Celanese

For our compression tests we employ the modified Celanese fixture which is most widely used. The according ASTM standard, which was also the first one specifically written for the compression testing of composite materials, is ASTM D 34101. The so-called Celanese compression test method was developed by the Celanese Corp in the early 1970s and was improved by the so called modified Celanese. This method required the use of a tabbed specimen of precise thickness, gripped between split cones. The specimen has to be prepared accurately and the test has to be performed very carefully, for otherwise the results are likely to be erratic. The tests are carried out between -269°C and 600°C .

Bending tests

For measuring the deflection and bending strength of fibre reinforced plastics we offer 3-point as well as 4-point bending tests. The mechanical parameters are determined between -269°C and 600°C by use of a moving coil extensometer or cross head movement of the machine.

Inter-laminar shear strength testing (ILSS)

The inter-laminar shear strength of carbon fibre reinforced plastics and unidirectional laminates are determined in a 3-point bending test. The resistance to interlaminar shear stress parallel to the layers of the laminate is measured in accordance with DIN EN 2563. This test provides information about the quality of the resin-fiber bond.

Basic concepts of fracture mechanism: tensile test results apply to material that does not contain cracks or stress concentrators, such as brittle inclusions. When crack like defects are present either as surface cracks or internal ones, failure may begin at much lower applied stresses. The applied stress is greatly magnified at the crack tip due to zero area (theoretically). For a ductile material, it can deform locally when the stress is high, blunting the crack tip reducing the intensity of stress. For brittle material, the crack will propagate through the stressed region with little deformation. The small scale plastic region around the crack will continue to propagate across the specimen. Fracture may be defined as the mechanical separation of a solid owing to the application of stress. Fractures of engineering material are categorized as ductile or brittle fractures. Ductile fractures absorb more energy, while brittle fractures absorb little energy, and are generally characterized by fracture with flat surfaces. Fracture toughness is related to the amount of energy required to create fracture surfaces. In brittle materials such as glass the energy required for fracture is simply the intrinsic surface energy of the material, as demonstrated by Griffith. For structural alloys at room temperature considerably energy is required for fracture because plastic deformation accompanies the fracture process. The application of fracture mechanics concepts has identified and quantified

the primary parameters that affect structural integrity. These parameters include the magnitude and range of the applied stresses, the size, shape, orientation of cracks / crack like defects, rate of propagation of the existing cracks and the fracture toughness of the material. Two categories of fracture mechanics are Linear Elastic Fracture Mechanics (LEFM) and Elastic-Plastic Fracture Mechanics (EPFM). The Linear Elastic Fracture Mechanics (LEFM) approach to fracture analysis assumes that the material behaves elastically at regions away from the crack, except for a small region of inelastic deformation at the crack tip. The fracture resistance is determined in terms of the stress- intensification factor, K and strain energy release rate G . The energy released during rapid crack propagation is a basic material property and is not influenced by part size. According to ASTM the stress intensity factor K can be written as $(gfaKI\pi\sigma = (1))$ Where 'a' is the initial crack length, 'f (g)' is the dimensionless factor for the specimen geometry and loading condition and the K_I , the Mode I critical stress intensity factor. The specimen size must be chosen such that there is small scale plasticity around the crack tip. If a large plastic zone develops ahead of the crack tip then the condition of "small scale yielding" for LEFM applicability are not met . One of the underlying principles of fracture mechanics is that the unstable fracture occurs when the stress intensity factor at the crack tip reaches a critical value, K_{IC} . The greater the value of fracture toughness, the higher the intensity of stress required to produce crack propagation and the greater the resistance of the material to brittle fracture. The critical stress intensity factor is determined using relatively simple laboratory specimen, the limiting value being $K_{IC} / K_{IIC} / K_{IIIC}$. The Elastic-Plastic fracture mechanics is used when there is large scale crack tip plasticity (blunting).

References:

1. John M Barsom, Stanley, T Rolfe. (1987). Fracture and Fatigue control in structures: Application of Fracture Mechanics. Second Edition. Prentice Hall. Inc. USA.
2. Todd M Mower, Victor C Li. (1987). Fracture characterization of random short fiber reinforced thermoset resin composites. Engineering fracture Mechanics. 26(4): 593-603.

Unit-	V
Topic-	Thermal Methods
Sub-Topic-	Introduction, principle, theory, applications, advantages and limitations of Differential scanning calorimeter (DSC), thermo gravimetric analysis (TGA), Dynamic mechanical analysis (DMA), and thermo mechanical analyzer (TMA). Morphological studies: Introduction, principle, theory, applications, advantages and limitations of- Optical microscopy, Scanning Electron Microscopy (SEM), TEM and AFM. Introduction: Hybrid polymer composite, Green composites and

Nano composites -fabrication, Characterization and applications.

Differential scanning calorimeter (DSC): Differential scanning calorimeter (DSC) is a thermo analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned.

The technique was developed by E. S. Watson and M. J. O'Neill in 1962, and introduced commercially at the 1963 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The first adiabatic differential scanning calorimeter that could be used in

biochemistry was developed by P. L. Privalov and D. R. Monaselidze in 1964 at Institute of Physics in Tbilisi, Georgia. The term DSC was coined to describe this instrument, which measures energy directly and allows precise measurements of heat capacity.



Differential scanning calorimeter

Types of DSC: here are two different types of DSC: Heat-flux DSC in which heat flux remains constant and Power differential DSC in which power supply remains constant.

Heat-flux DSC: With Heat-flux DSC, the changes in heat flow are calculated by integrating the ΔT_{ref} curve. For this kind of experiment, a sample and a reference crucible are placed on a sample holder with integrated temperature sensors for temperature measurement of the crucibles. This arrangement is located in a temperature-controlled oven. Contrary to this classic design, the distinctive attribute of MC-DSC is the vertical configuration of planar temperature sensors surrounding a planar heater. This arrangement allows a very compact, lightweight and low heat capacitance structure with the full functionality of a DSC oven

Power differential DSC: For this kind of setup, also known as *Power compensating DSC*, the sample and reference crucible are placed in thermally insulated furnaces and not next to each other in the same furnace like in Heat-flux-DSC experiments. Then the temperature of both chambers is controlled so that the same temperature is always present on both sides. The electrical power that is required to obtain and remain this state is then recorded instead of the temperature difference of the two crucibles.

Detection of phase transitions: The basic principle underlying this technique is that when the sample undergoes a physical transformation such as phase transitions, more or less heat will need to flow to it than the reference to maintain both at the same temperature. Whether less or more heat must flow to the sample depends on whether the process is exothermic or endothermic. For example, as a solid sample melts to a liquid, it will require more heat flowing to the sample to

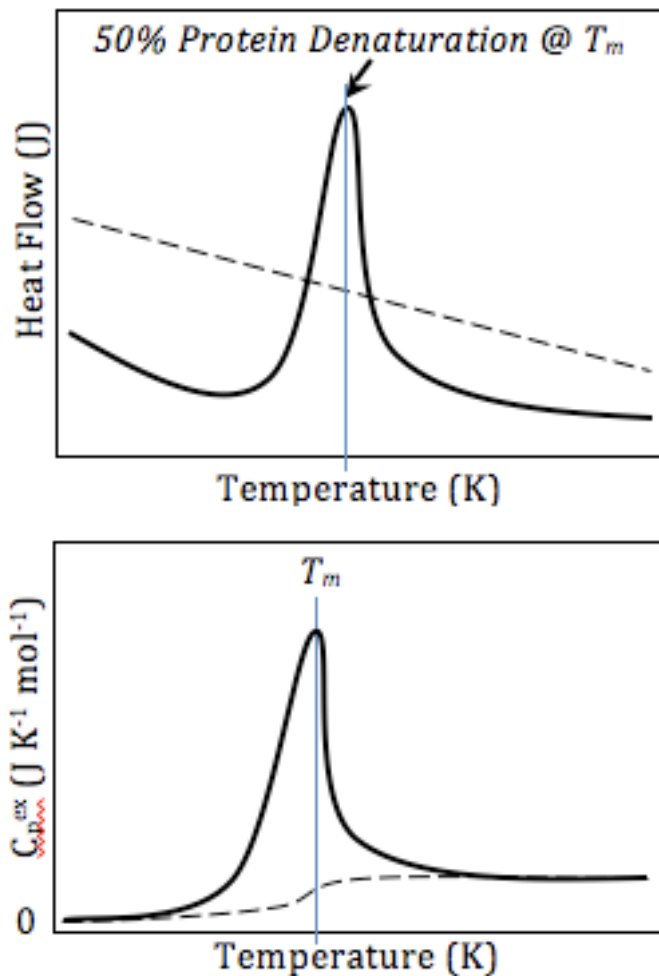
increase its temperature at the same rate as the reference. This is due to the absorption of heat by the sample as it undergoes the endothermic phase transition from solid to liquid. Likewise, as the sample undergoes exothermic processes (such as crystallization) less heat is required to raise the sample temperature. By observing the difference in heat flow between the sample and reference, differential scanning calorimeters are able to measure the amount of heat absorbed or released during such transitions. DSC may also be used to observe more subtle physical changes, such as glass transitions. It is widely used in industrial settings as a quality control instrument due to its applicability in evaluating sample purity and for studying polymer curing

DTA: An alternative technique, which shares much in common with DSC, is differential thermal analysis (DTA). In this technique it is the heat flow to the sample and reference that remains the same rather than the temperature. When the sample and reference are heated identically, phase changes and other thermal processes cause a difference in temperature between the sample and reference. Both DSC and DTA provide similar information. DSC measures the energy required to keep both the reference and the sample at the same temperature whereas DTA measures the difference in temperature between the sample and the reference when the same amount of energy has been introduced into both.

DSC curves: The result of a DSC experiment is a curve of heat flux versus temperature or versus time. There are two different conventions: exothermic reactions in the sample shown with a positive or negative peak, depending on the kind of technology used in the experiment. This curve can be used to calculate enthalpies of transitions. This is done by integrating the peak corresponding to a given transition. It can be shown that the enthalpy of transition can be expressed using the following equation:

$$\Delta H = K A$$

Where ΔH is the enthalpy of transition, K is the calorimetric constant, and A is the area under the curve. The calorimetric constant will vary from instrument to instrument, and can be determined by analyzing a well-characterized sample with known enthalpies of transition



Top: A schematic DSC curve of amount of energy input (y) required to maintain each temperature (x), scanned across a range of temperatures. Bottom: Normalized curves setting the initial heat capacity as the reference. Buffer-buffer baseline (dashed) and protein-buffer variance (solid).

Applications: Differential scanning calorimeter can be used to measure a number of characteristic properties of a sample. Using this technique it is possible to observe fusion and crystallization events as well as glass transition temperatures T_g . DSC can also be used to study oxidation, as well as other chemical reactions.

Glass transitions may occur as the temperature of an amorphous solid is increased. These transitions appear as a step in the baseline of the recorded DSC signal. This is due to the sample undergoing a change in heat capacity; no formal phase change occurs.

As the temperature increases, an amorphous solid will become less viscous. At some point the molecules may obtain enough freedom of motion to spontaneously arrange themselves into a crystalline form. This is known as the crystallization temperature (T_c). This transition from amorphous solid to crystalline solid is an exothermic process, and results in a peak in the DSC signal. As the temperature increases the sample eventually reaches its melting temperature (T_m).

The melting process results in an endothermic peak in the DSC curve. The ability to determine transition temperatures and enthalpies makes DSC a valuable tool in producing phase diagrams for various chemical systems.

Differential scanning calorimeter can also be used to obtain valuable thermodynamics information about proteins. The thermodynamics analysis of proteins can reveal important information about the global structure of proteins, and protein/ligand interaction. For example, many mutations lower the stability of proteins, while ligand binding usually increases protein stability. Using DSC, this stability can be measured by obtaining Gibbs free energy values at any given temperature. This allows researchers to compare the free energy of unfolding between ligand-free protein and protein-ligand complex, or wild type and mutant proteins. DSC can also be used in studying protein/lipid interactions, nucleotides, drug-lipid interactions. In studying protein denaturation using DSC, the thermal melt should be at least to some degree reversible, as the thermodynamics calculations rely on chemical equilibrium.

Examples: The technique is widely used across a range of applications, both as a routine quality test and as a research tool. The equipment is easy to calibrate, using low melting indium at 156.5985 °C for example, and is a rapid and reliable method of thermal analysis.

Polymers: DSC is used widely for examining polymeric materials to determine their thermal transitions. Important thermal transitions include the glass transition temperature (T_g), crystallization temperature (T_c), and melting temperature (T_m). The observed thermal transitions can be utilized to compare materials, although the transitions alone do not uniquely identify composition. The composition of unknown materials may be completed using complementary techniques such as IR spectroscopy. Melting points and glass transition temperatures for most polymers are available from standard compilations, and the method can show polymer degradation by the lowering of the expected melting temperature. T_m depends on the molecular weight of the polymer and thermal history.

Liquid crystals: DSC is used in the study of liquid crystals. As some forms of matter go from solid to liquid they go through a third state, which displays properties of both phases. This anisotropic liquid is known as a liquid crystalline or mesomorphous state. Using DSC, it is possible to observe the small energy changes that occur as matter transitions from a solid to a liquid crystal and from a liquid crystal to an isotropic liquid.

Oxidative stability: Using differential scanning calorimetry to study the stability to oxidation of samples generally requires an airtight sample chamber. Usually, such tests are done isothermally (at constant temperature) by changing the atmosphere of the sample. First, the sample is brought to the desired test temperature under an inert atmosphere, usually nitrogen. Then, oxygen is added to the system. Any oxidation that occurs is observed as a deviation in the baseline. Such analysis can be used to determine the stability and optimum storage conditions for a material or compound.

Safety screening: DSC makes a reasonable initial safety screening tool. In this mode the sample will be housed in a non-reactive crucible (often gold or gold-plated steel), and which will be able to withstand pressure (typically up to 100 bar). The presence of an exothermic event can then be used to assess the stability of a substance to heat. However, due to a combination of relatively poor sensitivity, slower than normal scan rates (typically 2–3 °C/min, due to much heavier crucible) and unknown activation energy, it is necessary to deduct about 75–100 °C from the initial start of the observed exotherm to suggest a maximal temperature for the material. A much more accurate data set can be obtained from an adiabatic calorimeter, but such a test may take 2–3 days from ambient at a rate of a 3 °C increment per half-hour.

Drug analysis: DSC is widely used in the pharmaceutical and polymer industries. For the polymer chemist, DSC is a handy tool for studying curing processes, which allows the fine tuning of polymer properties. The cross-linking of polymer molecules that occurs in the curing process is exothermic, resulting in a negative peak in the DSC curve that usually appears soon after the glass transition.

In the pharmaceutical industry it is necessary to have well-characterized drug compounds in order to define processing parameters. For instance, if it is necessary to deliver a drug in the amorphous form, it is desirable to process the drug at temperatures below those at which crystallization can occur.

General chemical analysis: Freezing-point depression can be used as a purity analysis tool when analysed by differential scanning calorimetry. This is possible because the temperature range over which a mixture of compounds melts is dependent on their relative amounts. Consequently, less pure compounds will exhibit a broadened melting peak that begins at lower temperature than a pure compound.

References:

1. U.S. Patent 3,263,484.
2. Molecular biology (in Russian). 6. Moscow. 1975. pp. 7–33.
3. Wunderlich B (1990). Thermal Analysis. New York: Academic Press. pp. 137–140. ISBN 0-12-765605-7.
4. Missal W, Kita J, Wappler E, Gora F, Kipka A, Bartnitzek T, Bechtold F, Schabbel D, Pawlowski B, Moos R (2010). "Miniaturized Ceramic Differential Scanning Calorimeter with Integrated Oven and Crucible in LTCC Technology". Procedia Engineering 5. Elsevier. pp. 940–943. ISSN 1877-7058

Thermo gravimetric analysis (TGA): Thermo gravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption, adsorption and desorption; as well as chemical phenomena including chemisorptions, thermal decomposition, and solid-gas reactions (e.g., oxidation or reduction).



A typical TGA system

Thermo gravimetric analyzer: Thermo gravimetric analysis (TGA) is conducted on an instrument referred to as a thermo gravimetric analyzer. A thermo gravimetric analyzer continuously measures mass while the temperature of a sample is changed over time. Mass, temperature, and time are considered base measurements in thermo gravimetric analysis while many additional measures may be derived from these three base measurements.

A typical thermo gravimetric analyzer consists of a precision balance with a sample pan located inside a furnace with a programmable control temperature. The temperature is generally increased at constant rate (or for some applications the temperature is controlled for a constant mass loss) to incur a thermal reaction. The thermal reaction may occur under a variety of atmospheres including: ambient air, vacuum, inert gas, oxidizing/reducing gases, corrosive gases, carburizing gases, vapors of liquids or "self-generated atmosphere"; as well as a variety of pressures including: a high vacuum, high pressure, constant pressure, or a controlled pressure.

The thermo gravimetric data collected from a thermal reaction is compiled into a plot of mass or percentage of initial mass on the y axis versus either temperature or time on the x-axis. This plot, which is often smoothed, is referred to as a TGA curve. The first derivative of the TGA curve (the DTG curve) may be plotted to determine inflection points useful for in-depth interpretations as well as differential thermal analysis.

A TGA can be used for materials characterization through analysis of characteristic decomposition patterns. It is an especially useful technique for the study of polymeric materials,

including thermoplastics, thermosets, elastomers, composites, plastic films, fibers, coatings, paints, and fuels.

Type of TGA: There are three types of thermo gravimetry.

1. **Isothermal or static thermo gravimetry-** In this technique, the sample weight is recorded as a function of time at constant temperature.
2. **Quasistatic thermo gravimetry-** In this technique, the sample temperature is raised in sequential steps separated by isothermal intervals, during which the sample mass reaches stability before the start of the next temperature ramp.
3. **Dynamic thermo gravimetry-** In this technique the sample is heated in an environment whose temperature is changed in a linear manner.

Applications:

- Thermal stability
- Oxidation and combustion
- Thermo gravimetric kinetics
- Operation in combination with instruments

References:

1. Coats, A. W.; Redfern, J. P. (1963). "Thermogravimetric Analysis: A Review". *Analyst*. 88 (1053): 906–924. Bibcode:1963Ana....88..906C. doi:10.1039/AN9638800906.
2. Liu, X.; Yu, W. (2006). "Evaluating the Thermal Stability of High Performance Fibers by TGA". *Journal of Applied Polymer Science*. 99 (3): 937–944. doi:10.1002/app.22305.
3. Marvel, C. S. (1972). "Synthesis of Thermally Stable Polymers". Ft. Belvoir: Defense Technical Information Center.

Thermo mechanical analyzer: Thermo mechanical analysis (TMA) is a technique used in thermal analysis, a branch of materials science which studies the properties of materials as they change with temperature.

Thermo mechanical analysis is a subdiscipline of the thermomechanometry (TM) technique.

Related techniques and terminology: Thermomechanometry is the measurement of a change of a dimension or a mechanical property of the sample while it is subjected to a temperature regime. An associated thermoanalytical method is thermomechanical analysis. A special related technique is thermodilatometry (TD), the measurement of a change of a dimension of the sample with a negligible force acting on the sample while it is subjected to a temperature regime. The associated thermoanalytical method is thermodilatometric analysis (TDA).

TDA is often referred to as zero force TMA. The temperature regime may be heating, cooling at a rate of temperature change that can include stepwise temperature changes, linear rate of change, temperature modulation with a set frequency and amplitude, free (uncontrolled) heating or cooling, or maintaining a constant increase in temperature. The sequence of temperatures with respect to time may be predetermined (temperature programmed) or sample controlled (controlled by a feedback signal from the sample response).

Thermomechanometry includes several variations according to the force and the way the force is applied.

Static force TM (sf-TM) is when the applied force is constant; previously called TMA with TD as the special case of zero force.

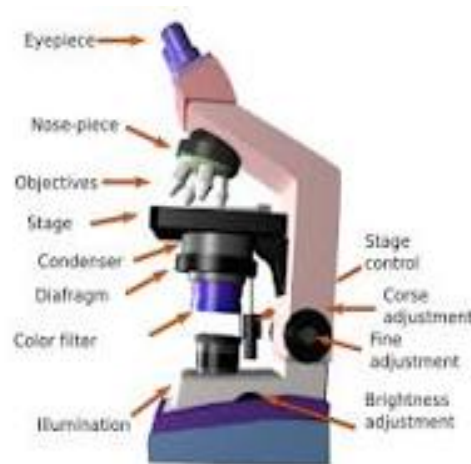
Dynamic force TM (df-TM) is when the force is changed as for the case of a typical stress–strain analysis; previously called TMA with the term dynamic meaning any alteration of the variable with time, and not to be confused with dynamic mechanical analysis (DMA).

Modulated force TM (mf-TM) is when the force is changed with a frequency and amplitude; previously called DMA. The term modulated is a special variant of dynamic, used to be consistent with modulated temperature differential scanning calorimetry (mt-DSC) and other situations when a variable is imposed in a cyclic manner.

References:

1. International Confederation of Thermal Analysis and Calorimetry (ICTAC), Nomenclature Committee, Recommendations for names and definitions in thermal analysis and calorimetry, Document IND98030.
2. Menard K. P., (1999), Dynamic Mechanical Analysis; A Practical Introduction, CRC Press, Boca Raton, Chapter 3
3. Wellisch E., Marker L., Sweeting O. J. (1961), Viscoelastic properties of regenerated cellulose, J. Appl. Polym. Sci., 5, 647-654.

Optical microscopy: optical microscopy is a technique employed to closely view a sample through the magnification of a lens with visible light. This is the traditional form of microscopy, which was first invented before the 18th century and is still in use today.



An optical microscope, also sometimes known as a light microscope, uses one or a series of lenses to magnify images of small samples with visible light. The lenses are placed between the sample and the viewer's eye to magnify the image so that it can be examined in greater detail.

Types of optical microscopes: There are many types of optical microscopes. They can vary from a very basic design to a high complexity that offers higher resolution and contrast. Some of the types of optical microscopes include the following:

- Simple microscope: a single lens to magnify the image of the sample, similar to a magnifying glass.
- Compound microscope: a series of lenses to magnify the sample image to a higher resolution, more commonly used in modern research.
- Digital microscope: may have simple or compound lenses, but uses a computer to visualize the image without the need for an eyepiece to view the sample.
- Stereo microscope: provides a stereoscopic image, which is useful for dissections.
- Comparison microscope: allows for the simultaneous view of two different samples, one in each eye.
- Inverted microscope: views the sample from underneath, which is useful to examine liquid cell cultures.

Other types of optical microscopes include petrographic, polarizing, phase contrast, epifluorescence, and confocal microscopes.

Images: An optical microscope can generate a micrograph using standard light-sensitive cameras. Photographic film was traditionally used to capture the images.

Technological developments have now enabled digital images to be taken with CMOS and charge-couple device (CCD) cameras for optical microscopes. As a result, the image can be projected onto a computer screen in real time to examine a sample with these digital microscopes. This increases the convenience of use as eyepieces are no longer needed.

The power of magnification of a compound optical microscope depends on the ocular and the objective lenses. It is equal to the product of the powers of these lenses (e.g. for a 10x ocular lens and 100x objective lens used together, the final magnification is 1000x.)

Operation, applications and limitations: In order to use an optical microscope effectively, it is important to ensure that the microscope is set up correctly.

The objective lens should be brought close to the study sample to allow the light inside the tube of the microscope. This creates an enlarged, inverted image of the sample, which can be viewed through the eyepiece of the microscope.

Optical microscopy is commonly used in many research areas including microbiology, microelectronics, nanophysics, biotechnology and pharmaceutical research. It can also be useful to view biological samples for medical diagnoses, known as histopathology.

There are some instances when optical microscopy is not well suited to the task at hand due to limitations of the technique. For example, at very high magnifications airy disks may be visible, which are fuzzy discs surrounded by diffraction rings, which appear in place of point objects.

When the limitations of optical microscopy are significant, alternative types of microscopy may be more useful.

Alternative types of microscopy: There are several other types of microscopy that may be used as alternatives to optical microscopy. These include:

- Scanning electron microscopy
- Transmission electron microscopy
- Fluorescence microscopy
- Atomic force microscopy
- Scanning ion conductance microscopy
- Scanning tunneling microscopy

- Ultraviolet microscopy
- X-ray microscopy

Unlike optical microscopy, these types of microscopy do not use visible light to view the sample

References

1. <http://web.utk.edu/~prack/MSE%20300/Lightmicroscopyhandout.pdf>.
2. <http://www.leica-microsystems.com/science-lab/the-optical-microscope-some-basics/>
3. <https://micro.magnet.fsu.edu/primer/pdfs/microscopy.pdf>.

Scanning Electron Microscopy (SEM): The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). The design and function of the SEM is very similar to the EPMA and considerable overlap in capabilities exists between the two instruments.



A typical SEM instrument, showing the electron column, sample chamber, EDS detector, electronics console, and visual display monitors

Fundamental Principles of Scanning Electron Microscopy (SEM): Accelerated electrons in an SEM carry significant amounts of kinetic energy, and this energy is dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample. These signals include secondary electrons (that produce SEM images), backscattered electrons (BSE), diffracted backscattered electrons (EBSD that are used to determine crystal structures and orientations of minerals), photons (characteristic X-rays that are used for elemental analysis and continuum X-rays), visible light (cathodoluminescence--CL), and heat. Secondary electrons and backscattered electrons are commonly used for imaging samples: secondary electrons are most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples (i.e. for rapid phase discrimination). X-ray generation is produced by inelastic collisions

of the incident electrons with electrons in discrete orbitals (shells) of atoms in the sample. As the excited electrons return to lower energy states, they yield X-rays that are of a fixed wavelength (that is related to the difference in energy levels of electrons in different shells for a given element). Thus, characteristic X-rays are produced for each element in a mineral that is "excited" by the electron beam. SEM analysis is considered to be "non-destructive"; that is, x-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyze the same materials repeatedly.

Scanning Electron Microscopy (SEM) Instrumentation - How Does It Work:

Essential components of all SEMs include the following:

- Electron Source ("Gun")
- Electron Lenses
- Sample Stage
- Detectors for all signals of interest
- Display / Data output devices
- Infrastructure Requirements:
 - Power Supply
 - Vacuum System
 - Cooling system
 - Vibration-free floor
 - Room free of ambient magnetic and electric fields

SEMs always have at least one detector (usually a secondary electron detector), and most have additional detectors. The specific capabilities of a particular instrument are critically dependent on which detectors it accommodates.

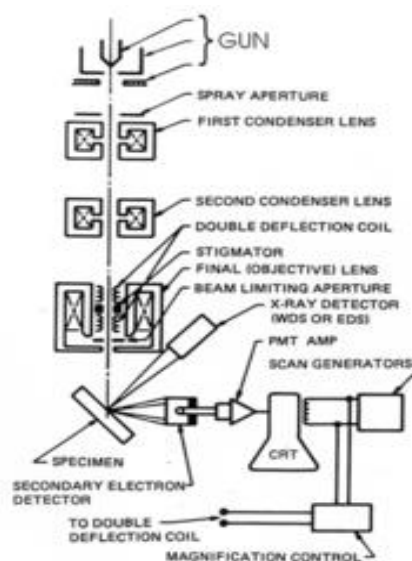


Figure 1.11. Schematic drawing of the electron and x-ray optics of a combined SEM-EPMA.

Applications: The SEM is routinely used to generate high-resolution images of shapes of objects (SEI) and to show spatial variations in chemical compositions: 1) acquiring elemental maps or spot chemical analyses using EDS, 2) discrimination of phases based on mean atomic number (commonly related to relative density) using BSE, and 3) compositional maps based on differences in trace element "activators" (typically transition metal and Rare Earth elements) using CL. The SEM is also widely used to identify phases based on qualitative chemical analysis and/or crystalline structure. Precise measurement of very small features and objects down to 50 nm in size is also accomplished using the SEM. Backscattered electron images (BSE) can be used for rapid discrimination of phases in multiphase samples. SEMs equipped with diffracted backscattered electron detectors (EBSD) can be used to examine microfabric and crystallographic orientation in many materials.

Reference:

1. https://serc.carleton.edu/research_education/geochemsheets/techniques/SEM.html.

Transmission Electron Microscopy (TEM): The transmission electron microscope is a very powerful tool for material science. A high energy beam of electrons is shone through a very thin sample, and the interactions between the electrons and the atoms can be used to observe features such as the crystal structure and features in the structure like dislocations and grain boundaries. Chemical analysis can also be performed. TEM can be used to study the growth of layers, their composition and defects in semiconductors. High resolution can be used to analyze the quality, shape, size and density of quantum wells, wires and dots.

The TEM operates on the same basic principles as the light microscope but uses electrons instead of light. Because the wavelength of electrons is much smaller than that of light, the optimal resolution attainable for TEM images is many orders of magnitude better than that from a light microscope. Thus, TEMs can reveal the finest details of internal structure - in some cases as small as individual atoms.

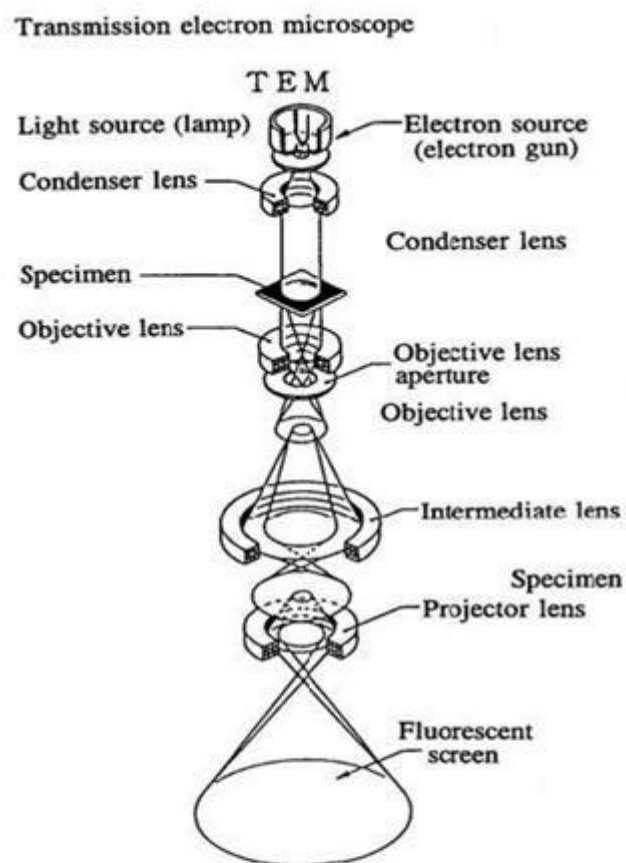


Fig 1

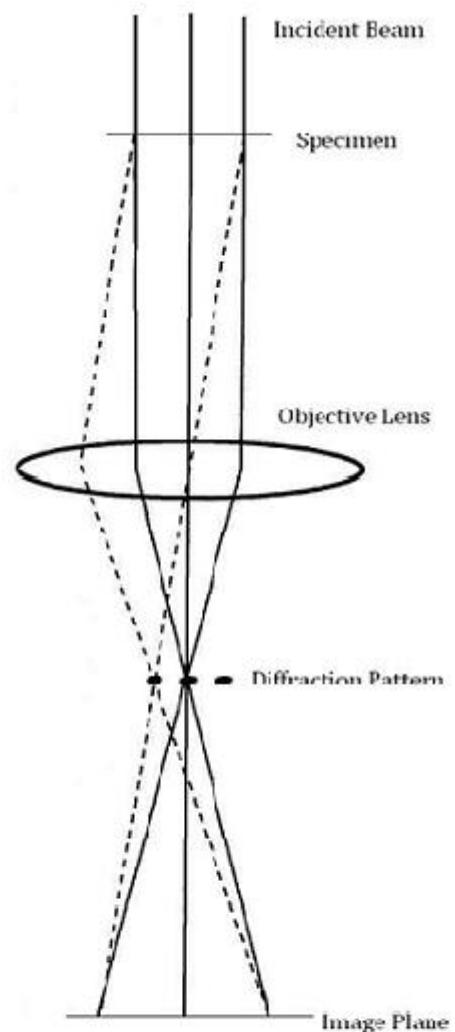


Fig 2

Imaging: The beam of electrons from the electron gun is focused into a small, thin, coherent beam by the use of the condenser lens. This beam is restricted by the condenser aperture, which excludes high angle electrons. The beam then strikes the specimen and parts of it are transmitted depending upon the thickness and electron transparency of the specimen. This transmitted portion is focused by the objective lens into an image on phosphor screen or charge coupled device (CCD) camera. Optional objective apertures can be used to enhance the contrast by blocking out high-angle diffracted electrons. The image then passed down the column through the intermediate and projector lenses, is enlarged all the way.

The image strikes the phosphor screen and light is generated, allowing the user to see the image. The darker areas of the image represent those areas of the sample that fewer electrons are transmitted through while the lighter areas of the image represent those areas of the sample that more electrons were transmitted through.

Diffraction: Fig2. shows a simple sketch of the path of a beam of electrons in a TEM from just above the specimen and down the column to the phosphor screen. As the electrons pass through the sample, they are scattered by the electrostatic potential set up by the constituent elements in the specimen. After passing through the specimen they pass through the electromagnetic objective lens which focuses all the electrons scattered from one point of the specimen into one point in the image plane. Also, shown in fig 2 is a dotted line where the electrons scattered in the same direction by the sample are collected into a single point. This is the back focal plane of the objective lens and is where the diffraction pattern is formed.

TEM Applications: A Transmission Electron Microscope is ideal for a number of different fields such as life sciences, nanotechnology, medical, biological and material research, forensic analysis, gemology and metallurgy as well as industry and education.

TEMs provide topographical, morphological, compositional and crystalline information.

The images allow researchers to view samples on a molecular level, making it possible to analyze structure and texture.

This information is useful in the study of crystals and metals, but also has industrial applications.

TEMs can be used in semiconductor analysis and production and the manufacturing of computer and silicon chips.

Technology companies use TEMs to identify flaws, fractures and damages to micro-sized objects; this data can help fix problems and/or help to make a more durable, efficient product.

Colleges and universities can utilize TEMs for research and studies.

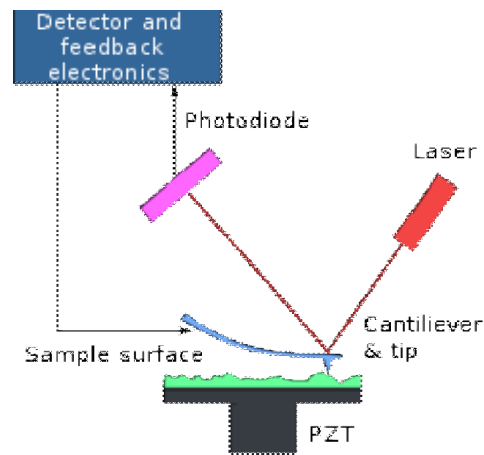
Although electron microscopes require specialized training, students can assist professors and learn TEM techniques.

Students will have the opportunity to observe a nano-sized world in incredible depth and detail.

Reference:

1. https://serc.carleton.edu/research_education/geochemsheets/techniques/SEM.html.

Atomic Force Microscope : Atomic force microscopy (AFM) or scanning force microscopy (SFM) is a very-high-resolution type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit.



An AFM generates images by scanning a small cantilever over the surface of a sample. The sharp tip on the end of the cantilever contacts the surface, bending the cantilever and changing the amount of laser light reflected into the photodiode. The height of the cantilever is then adjusted to restore the response signal resulting in the measured cantilever height tracing the surface.

Overview: Atomic force microscopy (AFM) is a type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit. The information is gathered by "feeling" or "touching" the surface with a mechanical probe. Piezoelectric elements that facilitate tiny but accurate and precise movements on (electronic) command enable precise scanning.

Principles: The AFM consists of a cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers. When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law.[9] Depending on the situation, forces that are measured in AFM include mechanical contact force, van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces (see magnetic force microscope, MFM), Casimir forces, solvation forces, etc. Along with force, additional quantities may simultaneously be measured through the use of specialized types of probes (see scanning thermal microscopy, scanning joule expansion microscopy, photothermal microspectroscopy, etc.).

The AFM can be operated in a number of modes, depending on the application. In general, possible imaging modes are divided into static (also called contact) modes and a variety of dynamic (non-contact or "tapping") modes where the cantilever is vibrated or oscillated at a given frequency

Abilities: The AFM has three major abilities: force measurement, topographic imaging, and manipulation.

In force measurement, AFMs can be used to measure the forces between the probe and the sample as a function of their mutual separation. This can be applied to perform force spectroscopy, to measure the mechanical properties of the sample, such as the sample's Young's modulus, a measure of stiffness.

For imaging, the reaction of the probe to the forces that the sample imposes on it can be used to form an image of the three-dimensional shape (topography) of a sample surface at a high resolution. This is achieved by raster scanning the position of the sample with respect to the tip and recording the height of the probe that corresponds to a constant probe-sample interaction (see section topographic imaging in AFM for more details). The surface topography is commonly displayed as a pseudocolor plot. Although the initial publication about the atomic force microscopy by Binnig, Quate and Gerber in 1986 speculated about the possibility of achieving atomic resolution, profound experimental challenges needed to be overcome before atomic resolution of defects and step edges in ambient (liquid) conditions was demonstrated in 1993 by Ohnesorge and Binnig. True atomic resolution of the silicon 7x7 surface - the atomic images of this surface obtained by STM had convinced the scientific community of the spectacular spatial resolution of scanning tunneling microscopy – had to wait a little longer before it was shown by Giessibl.

In manipulation, the forces between tip and sample can also be used to change the properties of the sample in a controlled way. Examples of this include atomic manipulation, scanning probe lithography and local stimulation of cells.

Simultaneous with the acquisition of topographical images, other properties of the sample can be measured locally and displayed as an image, often with similarly high resolution. Examples of such properties are mechanical properties like stiffness or adhesion strength and electrical

properties such as conductivity or surface potential. In fact, the majority of SPM techniques are extensions of AFM that use this modality.

Image formation: When using the AFM to image a sample, the tip is brought into contact with the sample, and the sample is raster scanned along an x-y grid (fig 4). Most commonly, an electronic feedback loop is employed to keep the probe-sample force constant during scanning. This feedback loop has the cantilever deflection as input, and its output controls the distance along the z axis between the probe support (2 in fig. 3) and the sample support (8 in fig 3). As long as the tip remains in contact with the sample, and the sample is scanned in the x-y plane, height variations in the sample will change the deflection of the cantilever. The feedback then adjusts the height of the probe support so that the deflection is restored to a user-defined value (the setpoint). A properly adjusted feedback loop adjusts the support-sample separation continuously during the scanning motion, such that the deflection remains approximately constant. In this situation, the feedback output equals the sample surface topography to within a small error.

Historically, a different operation method has been used, in which the sample-probe support distance is kept constant and not controlled by a feedback (servo mechanism). In this mode, usually referred to as 'constant height mode', the deflection of the cantilever is recorded as a function of the sample x-y position. As long as the tip is in contact with the sample, the deflection then corresponds to surface topography. The main reason this method is not very popular anymore, is that the forces between tip and sample are not controlled, which can lead to forces high enough to damage the tip or the sample. It is however common practice to record the deflection even when scanning in 'constant force mode', with feedback. This reveals the small tracking error of the feedback, and can sometimes reveal features that the feedback was not able to adjust for.

The AFM signals, such as sample height or cantilever deflection, are recorded on a computer during the x-y scan. They are plotted in a pseudocolor image, in which each pixel represents an x-y position on the sample, and the color represents the recorded signal.

Applications: The AFM has been applied to problems in a wide range of disciplines of the natural sciences, including solid-state physics, semiconductor science and technology, molecular engineering, polymer chemistry and physics, surface chemistry, molecular biology, cell biology, and medicine.

Applications in the field of solid state physics include (a) the identification of atoms at a surface, (b) the evaluation of interactions between a specific atom and its neighboring atoms, and (c) the

study of changes in physical properties arising from changes in an atomic arrangement through atomic manipulation.

In molecular biology, AFM can be used to study the structure and mechanical properties of protein complexes and assemblies. For example, AFM has been used to image microtubules and measure their stiffness.

In cellular biology, AFM can be used to attempt to distinguish cancer cells and normal cells based on a hardness of cells, and to evaluate interactions between a specific cell and its neighboring cells in a competitive culture system. AFM can also be used to indent cells, to study how they regulate the stiffness or shape of the cell membrane or wall.

In some variations, electric potentials can also be scanned using conducting cantilevers. In more advanced versions, currents can be passed through the tip to probe the electrical conductivity or transport of the underlying surface, but this is a challenging task with few research groups reporting consistent data (as of 2004)

References:

1. "Measuring and Analyzing Force-Distance Curves with Atomic Force Microscopy" (PDF). afmworkshop.com.
2. Ohnesorge, Frank (1 January 1993). "True atomic resolution by atomic force microscopy through repulsive and attractive forces". *Science*. 260 (5113): 1451–6. Bibcode:1993Sci...260.1451O. doi:10.1126/science.260.5113.1451. PMID 17739801.

Hybrid polymer composite: A combination of two or more types of fibers in a single polymeric matrix (also known as the hybrid composite) can produce greater stiffness and strength in comparison with the individual corresponding reinforced polymer composites. Different fibers have different properties and characteristics. Thus, if these different properties are known, different fibers can be employed in a way that they can superpose their properties towards reaching an optimum behavior when subjected to a certain mechanical loading as a whole. Commonly, some types of fibers in hybrid composites have low modulus and/or lower cost, such as glass and Kevlar fibers, whilst the other types have high modulus and/or high cost such as boron and carbon fibers. Low modulus and inexpensive fibers make hybrid composites more tolerant to damage and reduce the overall cost, while the more expensive fibers with high modulus improve load bearing capabilities and composite stiffness. Hence, hybridization of composites can provide high stiffness and strength, improve the impact and fatigue resistance, provide high fracture toughness and simultaneously cut the total weight and cost. Hybridization is one of the effective methods of achieving desired properties of laminated composites. Hybrid composites are produced by incorporating two or more types of fibers in a laminated composite in order to achieve enhanced properties in comparison with the corresponding single type fiber-reinforced composites. Hybridizing the composite laminates provide the opportunity of benefiting from the advantages of various fibers and suppressing their weaknesses. Hybridization has been used in order to improve the fracture toughness, fatigue resistance and reduce the total cost or weight of composite laminates.

References:

1. R. B. Heslehurst, Defects and Damage in Composite Materials and Structures (CRC Press, 2014).
2. S. C. V. Talreja, Damage and Failure of Composite Materials (Cambridge University Press, 2012).

Green composites: The first natural fibre based composites were appeared in 1908. Green composites are a specific part of bio-composites, in which bio-based polymer matrix is reinforced by natural fibers; they symbolize a developing area in polymer science. In a situation like increment in oil price, the use of green composites is helpful not only in making the environment better but also from an economical perspective. The newest development in the field of bio-composites is the substitution of oil-derived polymers with polymers from renewable resources as the matrix component. Such materials are termed as “green composites”. Green composites are less in cost and are decomposable. There is a mounting need to develop bio- based products and other advanced skills that can decrease our dependence on fossil fuel. Bio-based resources include industrial goods, wood, wood wastes and remains. Bio composites which are eco-friendly are new materials they are not just a solution to growing environmental danger but also as a solution to the ambiguity of petroleum supply. Green composites, which are a mixture of natural fibers and bio plastics, have appeared as hopeful replacements to conventional glass fiber composites because they carry a widespread range of benefits, such as biodegradability, renewable and low density. In recent years, efforts have been made to lessen the use of expensive glass, carbon fibers and also reduce the car’s weight by taking advantage of the lower density natural fibers. Natural fibres like sisal, jute, abaca and coir are been in use as reinforcement in composites. Most of the composites are built using polymers and synthetic fibers. These polymers are threat to the environment. As a result, bio based composites have drawn much attention. As compared to synthetic fibres, natural plant-based fibres have various advantages. These natural fibres are low in cost, high thermal insulation and biodegradability. At present, numbers of biodegradable and green composites have been advanced with enhanced mechanical properties using different natural fibers. The performance of green composites relies on the properties of the natural fibers used for reinforcement. Though, green composites have some drawbacks, but these natural fibers don’t have any effect on the environment hence, are useful.

Mechanical Properties: Georgios Koronis et.al (2013) have explored about the use of numerous green composites in automobile sector. Green composites made with the help of resins and plants (using plant fibres) are already been in use in automobile. Some tests were performed on the green composites to check their various performances for different application. Table 1 shows results of various tests done on several reinforced bio-resins with different kinds of natural fibres. From the data in Table 1 most of the green composites are made up of Poly Lactic Acid (PLA), Poly-L-Lactide (PLLA) and other natural fibres. Increment in Flax performance when reinforced with PLLA as compare to PP-fibreglass and highest tensile strength was shown by jute fibres as compared among other natural fibres but reinforcement of jute fibre also reported with the low mechanical properties on comparing to abaca fibres. The lower in performance in the mechanical

properties could be due to the different manufacturing method used. Several physical and mechanical properties are presented in Table 1. For the structural performance of automobile panels and the automobile design specific stiffness and strength happens to be important indicators, since these have special distinction in application. The different values of tensile strength and young's modulus have been attributed to the different harvesting seasons.

Table 1.

Mechanical properties of several green composites fibers and PP + GFR composites

S. No.		Elongation to Break (%)	Tensile Strength (MPa)	Young's Modulus (GPa)
1	Starch + 30% jute	2 ± 0.2	26.3 ± 0.55	2.5 ± 0.23
2	PLA + 30% ramie	4.8 ± 0.2	66.8 ± 1.7	n.s
3	PLA + 30% jute	1.8 ± 0	81.9 ± 2.9	9.6 ± 0.36
4	PLA + 25% hemp	n.s	62 ± 2	7.2 ± 0.3
5	PHBV + 30% jute	0.8 ± 0	35.2 ± 1.3	7 ± 0.26
6	PLLA + 30% flax	2.3 ± 0.2	98 ± 12	9.5 ± 0.5
7	PHB + 30% flax	7 ± 1.5	40 ± 2.5	4.7 ± 0.3
8	PLA + 30% flax	1 ± 0.2	53 ± 3.1	8.3 ± 0.6
9	PP + 30% flax	2.7 ± 1.5	29.1 ± 4.2	5 ± 0.4
10	PP + 30% jute	1.4 ± 0.1	47.9 ± 2.7	5.8 ± 0.47
11	PP + 30% fiberglass	3.01 ± 0.22	82.8 ± 4.0	4.62 ± 0.11

Adapted from Georgios Koronis et.al (2013)

E-glass is clearly better in terms of specific strength on the other hand is lower in term of specific stiffness comparing with kenaf, hemp and ramie. Therefore more factors should be considered to choose the ideal material.

Reference:

1. <https://www.igi-global.com/chapter/green-composites-and-their-properties/156906>.

Nanocomposites –fabrication: Nanocomposites are materials that incorporate nano sized particles into a matrix of standard material. The result of the addition of nano particles is a drastic improvement in properties that can include mechanical strength, toughness and electrical or thermal conductivity. The effectiveness of the nano particles is such that the amount of material added is normally only between 0.5 and 5% by weight.

Nano particles have an extremely high surface to volume ratio which dramatically changes their properties when compared with their bulk sized equivalents. It also changes the way in which the nano particles bond with the bulk material. The result is that the composite can be many times improved with respect to the component parts. Some nano composite materials have been shown to be 1000 times tougher than the bulk component materials

Improved Properties: Nanocomposites can dramatically improve properties like:

- Mechanical properties including strength, modulus and dimensional stability
- Electrical conductivity
- Decreased gas, water and hydrocarbon permeability
- Flame retardancy
- Thermal stability
- Chemical resistance
- Surface appearance
- Optical clarity

Applications:

Nanocomposites are currently being used in a number of fields and new applications are being continuously developed. Applications for Nanocomposites include:

- Thin-film capacitors for computer chips
- Solid polymer electrolytes for batteries.
- Automotive engine parts and fuel tanks
- Impellers and blades
- Oxygen and gas barriers
- Food packaging

Characterization of Nanocomposites Particles: Characterization refers to the study and evaluation of the material's qualitative as well as quantitative features such as its composition, microstructure and various properties like physical, chemical, electrical, mechanical, magnetic etc. Nano particle characterization provides an idea and understanding regarding synthesis parameters and application oriented properties, and carried out by using a variety of different techniques and approaches, drawn from materials science. The experimental determination of mechanical properties reported in the literature is limited only to fracture toughness and hardness through micro indentation. Most of the reports are regarding fabrication of sintered compacts of nano-and NcPs at bulk and then evaluation of mechanical properties

Nanocomposites Particles For Engineering Applications: The electronic structure of a substance is responsible for its physio-mechanical properties, and enormously increased surface area at nano size proportionately increases the sensitivity and catalytic properties. The bonding strength at the surface of a nano size particle changes hence the implications for the reactivity of the surface can be significant. The NcPs synthesized for engineering applications are prepared through chemical or mechanical processing routes and investigated mainly for the application oriented characteristics.

References:

1. Zsigmondy, R. (1909). Colloids and the Ultra Microscope. Journal of the American Chemical Society, 31(8), 951-952.
2. Goodstein, D., & Goodstein, J. (2000). Richard Feynman and the history of superconductivity. Physics in Perspective, 2(1), 30-47.
3. Peppas, N. A., Hilt, J. Z., Khademhosseini, A., & Langer, R. (2006). Hydrogels in biology and Medicine: from molecular principles to bionanotechnology. Advanced Materials, 18(11), 1345-1360.