

ME 644: COMPOSITE MATERIALS AND PROCESSING



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Syllabus

UNIT-I

- Introduction: General introduction to composites; historical background; concept of matrix and reinforcement and particulates. Matrix and reinforcement: Types of matrix and reinforcement, volume fraction and weight fraction Fiber architecture fiber packing arrangements, whiskers. (7 Hrs)

Unit-II

- Fabrication methods of polymer composites: Liquid resin impregnated routes, pressurized consolidation of resin pre-pegs, consolidation of resin molding compounds, injection molding of thermoplastics, hot press molding of thermoplastics. (7 Hrs)

Unit-III

- Fabrication of ceramic composites: Powder based routes, reactive processing, layered ceramic composites, carbon/carbon composites. (7 Hrs)

Syllabus

Unit-IV

- Fabrication routes of metal matrix composites: Squeeze infiltration, stir casting, spray deposition, powder blending and consolidation, diffusion bonding of foils. (7 Hrs)

Unit-V

- Testing and characterization: Different tests like internal stress measurement by diffraction, metallographic preparation etc with special emphasis to metal matrix, composites. (7 Hrs)

Unit-VI

- Secondary processing and application of composites: Secondary processing like machining, joining, extrusion of composites; Application and case studies (7 Hrs)

Text/Reference Book

- Introduction to Metal Matrix Composite, T. W. Clyne & P. J. Withers Cambridge press
- W.D. Callister, Jr, - Material Science & Engineering Addition-Wiley Publishing Co.
- Van Vlash - Elements of Material Science & Engineering John Wiley & Sons.
- Composite materials , S.C. Sharma Narosa Publishers.
- Metal matrix composite R. K. Everret & R.J. Arsenault, Academic press

Additional Books

- Fiber Reinforced Composites, P.K.Mallick, CRC Press
- Composite Manufacturing, S. K. Mazumdar, CRC Press
- Composites Materials, Matthews and Rawlings, CRC Press

Introduction

- Composite means “consisting of two or more distinct parts”.
- Composites materials consists of two or more chemically distinct constituents/phases with significantly different physical properties whose combination produces better properties than its constituents.
- Examples are Wood: Cellulose Fibers in Lignin Matrix; Bones: Collagen Fibers in Mineral Matrix; Concrete: Stones in Cement Matrix; Cemented carbides: WC with Co binder
- It is important to note that
 - In composites, constituents remain separate and distinct within the finished structure.
 - Composites have significantly different properties from constituents properties.

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5

Introduction

Composite materials are not multiphase materials as different phases were not formed naturally by reactions, phase transformations, or other phenomena.

In composites, generally

- One of the phase is in platelet or fibrous form.
- Volume fraction is greater than 10%.
- Property of one constituent is much greater (≥ 5 times) than other.

In metals

- Constituent phases often have nearly identical properties (e.g. modulus of elasticity).
- Phases are not generally fibrous in character.
- One of the phases is usually present in small volume fractions.

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6

Introduction

- Certain material can be classified as a composite if a combination of constituents leads to significant property changes.
- Plastics which contains small quantities of additives are not composites as their additions did not affect plastic properties significantly.
- Composite consists of a continuous (matrix) and one or more discontinuous phase (reinforcement).
- Size, distribution, volume fraction and type of reinforcement, all determines the properties of composites.

Function of Fibers

- Function of fibers are:
 - To carry the load
 - To provide strength, stiffness, thermal stability and structural properties
 - To provide electrical conductivity/insulation
- Fibrous composite are capable of achieving high strengths.
- Strength of fiber or filaments is much higher than bulk material as the large flaws present in the bulk material are minimized owing to small cross-section dimension of fibers.

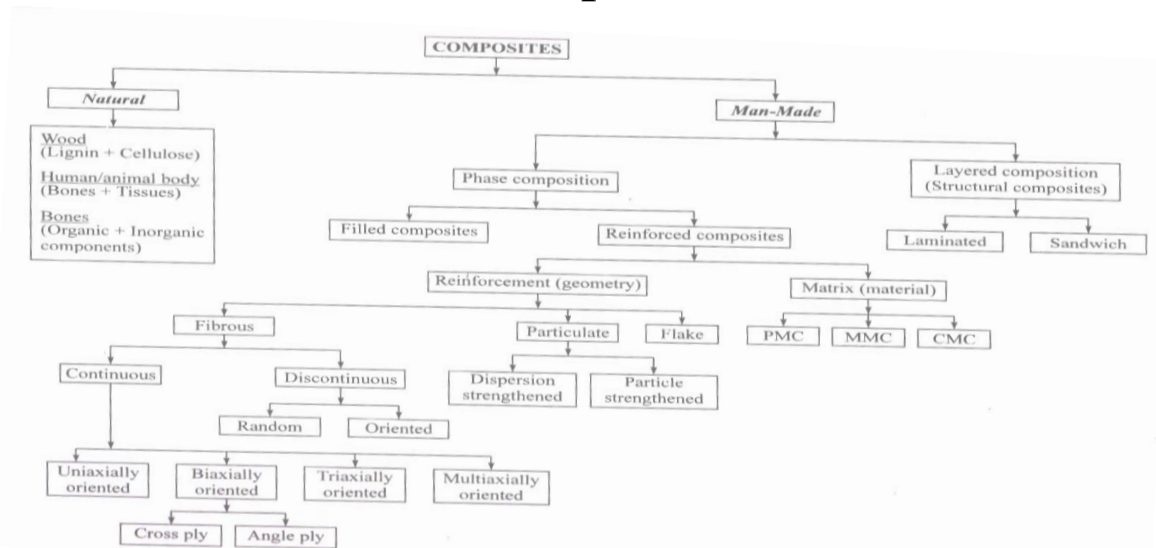
Functions of Matrix

- The matrix material binds the fibers together and transfers the load to the fibers.
- The matrix provides a good surface finish quality.
- The matrix isolates the fibers and thus stops or slows the propagation of a crack.
- The matrix protects the reinforcing fibers from chemical attack and mechanical wear and tear.
- The failure mode is strongly affected by the type of matrix material and its compatibility with the fiber.
- Matrix material influences the performance characteristics such as ductility, impact strength, etc. A ductile matrix is generally used to increase the toughness of the structure.

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9

Classification of Composites

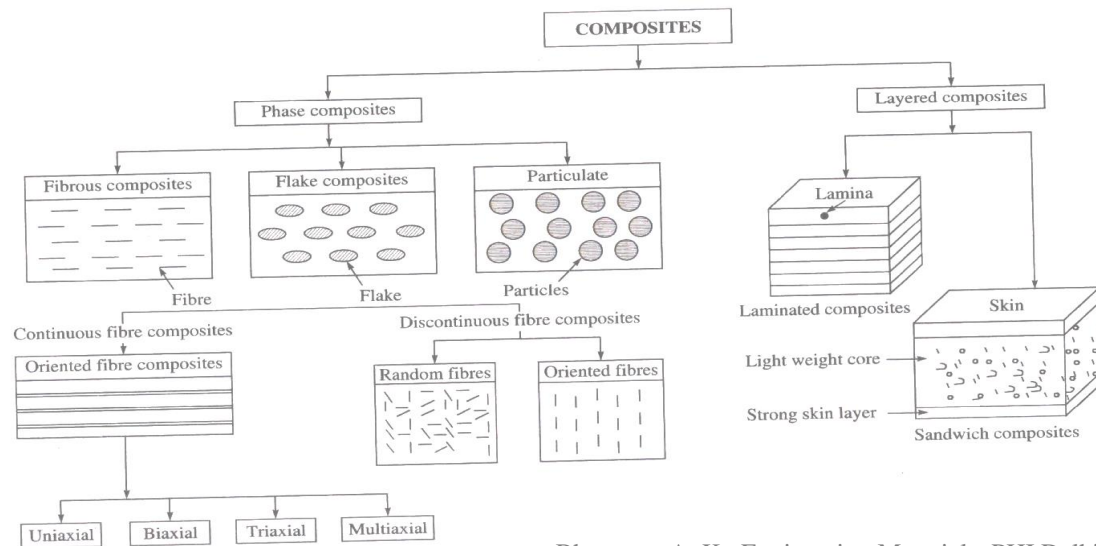


Bhargava, A. K., Engineering Materials, PHI Delhi, 2013

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10

Classification of Man-Made Composites



Bhargava, A. K., Engineering Materials, PHI Delhi, 2013

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11

Properties of Composites

- Properties of many important composites are anisotropic; this may be an advantage or a disadvantage.
- Primary advantage of composites is the ability to control anisotropy by design and fabrication.
- Orientation of reinforcements affects the isotropy of the system.
- Random oriented reinforcement in form of unequal dimension particles (short fibers) – may behave as isotropic material.
- Reinforcements in form of equiaxed particles – may behave as isotropic material.
- Particles are effective in enhancing the stiffness of the matrix but do not provide much strengthening.
- Particles are mixed in matrix to reduce friction, increase surface hardness and wear resistance, improved elevated temperature performance, reduce shrinkage and elevated temperature performance.

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12

Advantages of Composites

- Lightweight
- High specific stiffness and High specific strength
- Tailored properties (anisotropic)
- Easily moldable to complex (net) shapes
- Part consolidation leading to lower overall system cost
- Easily bondable
- Good fatigue resistance
- Good damping
- Crash worthiness
- Low thermal expansion and electrical conductivity
- Stealth (low radar visibility)

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13

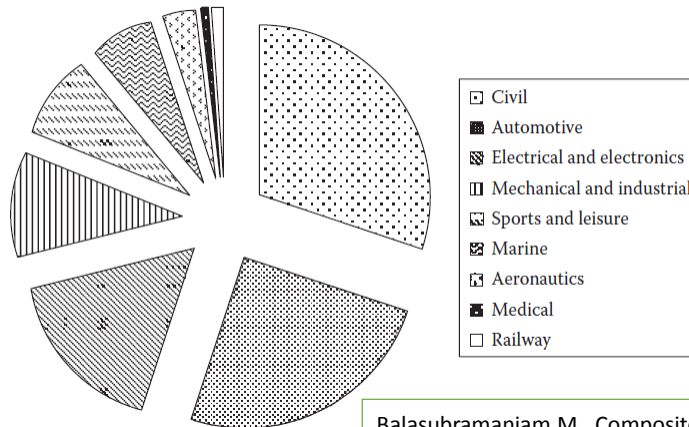
Disadvantages of Composites

- Cost of materials
- Lack of well-proven design rules
- Composite designs are seldom directly interchangeable
- Long development time
- Manufacturing difficulties (manual, slow, environmentally problems, poor reliability)
- Fasteners
- Low ductility (joints inefficient, stress risers more critical than in metals)
- Solvent/moisture attack
- Temperature limits
- Damage susceptibility
- Hidden damage

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14

Applications of Composites



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15

Applications of Composites

- High specific stiffness and specific strength are the important properties that have led to the wide usage of composites in aerospace sector.
- Polymer composites are more suitable for marine vessels and offshore structures because of their better weather resistance. Moreover, it is possible to make marine vessels with fewer joints using composites.
- Doors, door frames, partition walls, etc. are being made with polymer composites. Polymer composites also find applications as aesthetic in many buildings.
- Polymer composites are good electrical insulators. Many types of electrical insulators and the substrates of printed circuit boards are being made with glass fiber-reinforced epoxy composites.
- Many mechanical and automobile components are made with polymer composites e.g. leaf spring, automobile bodies, containers, etc.

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16

Applications of Composites

- MMCs are mainly used in aerospace and automobile sectors. Aerospace structures, turbine engine blades, landing gears, cutting tools, driving shaft, and piston.
- The processing of CMCs is even more complicated because of the requirement of very high processing temperatures.
- It is very difficult to produce good-quality composites because of adverse chemical reaction between the fibers and matrix at these high temperatures.
- At present, these materials are very expensive to make, hence their use is limited to applications that can utilize their special characteristics, such as high temperature resistance and high wear resistance.

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17

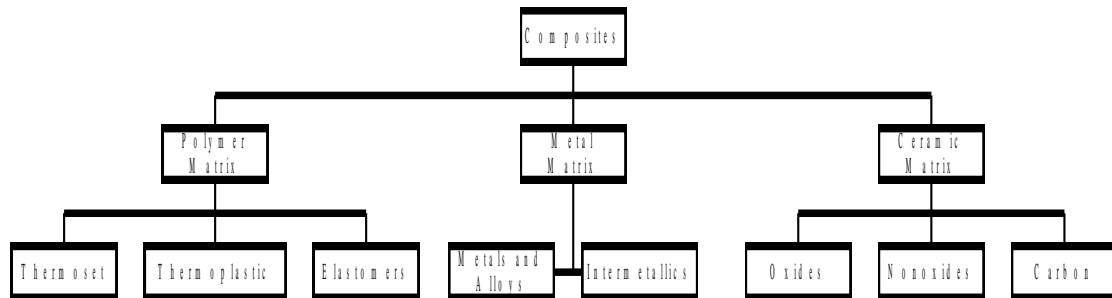
Matrix

- Matrix is the continuous phase in a composite and it binds the reinforcement together to form different shapes.
- The matrix have a little effect in the load-bearing capacity of the composite, since it transfers the load to the reinforcements but, the matrix has a significant influence on the interlaminar and in-plane shear properties.
- The interlaminar shear strength is important for the composite structures under bending loads, whereas the in-plane shear strength is important under torsional loads.
- Poor interlaminar shear strength leads to the separation of layers whereas poor in-plane shear strength leads to the debonding of fibers.
- The matrix provides lateral support against fiber buckling under compressive loading and thus influences the compressive properties of the composites also.

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18

Matrix Materials - Classification

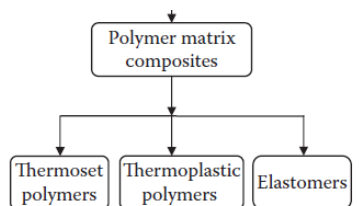


Balasubramaniam M., Composite Materials and Processing, CRC Press, 2013

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19

Polymer Matrix Composites



- PMCs are suitable for making products, which are used at ambient temperature.
- Some special polymers can also be used up to 250°C.
- In any case, PMCs are not suitable for applications where the service temperature is in excess of 350°C.

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20

Polymer Matrix Composites

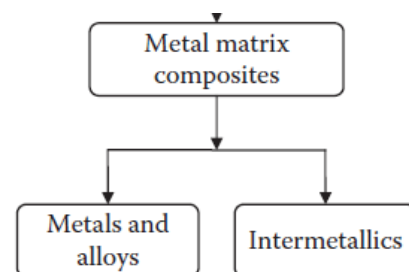
- The matrix can be a thermoset polymer, a thermoplastic polymer, or an elastomer in the PMCs.
- Thermoset polymers are widely used owing to their processing advantage.
- Thermoplastic polymers are also gaining importance now because of their relatively high toughness and most importantly, the possibility of their post-processing.
- To meet the specific property requirements, a wide variety of thermoplastic polymers are available.
- Elastomers are special polymers that are very elastic in nature. The intermolecular forces between the polymer chains are comparatively weak.
- They are lightly cross-linked and are amorphous with a glass transition temperature well below the room temperature.

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21

Metal Matrix Composites

- MMCs are suitable for applications where the service temperature is up to 1200°C.
- Generally lightweight metals such as, aluminum, titanium, and their alloys are used.
- In some special applications, heavy metals such as copper and cobalt are also used.
- Short fibers or particulates are mainly used as the dispersed phase in metal matrix.
- Metals and alloys are also reinforced with continuous fibers.
- Metals and alloys inherently have good ductility and toughness. Inclusion of reinforcements improves the Young's modulus but at the expense of ductility.



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22

Intermetallic compounds

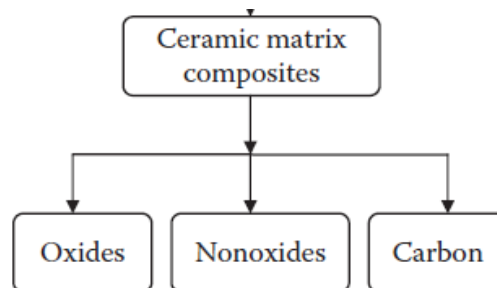
- Intermetallic compounds are generally formed between chemically dissimilar metals.
- They are combined by following the rules of chemical valence. Their properties are essentially non metallic.
- They generally have strong bonding (ionic or covalent). Examples are Mg_2Sn , Cu_2Se , $CaSe$
- Interstitial compounds : Formed between Transition metals such as Scandium, titanium, tantalum, tungsten and iron with hydrogen, oxygen, carbon, boron and nitrogen. E.g. TiC , CrN , W_2C , TiH .

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23

Ceramic Matrix Composites

- CMCs are useful for high- service temperature applications, above $1200^{\circ}C$.
- Ceramics are basically the crystalline compounds of metals (such as Al, Si, Mg, Ti, Ca, etc.) and non-metals (such as O, N, B, C) bonded by primary ionic and/or covalent bonds.
- CMCs are very expensive as they are processed at high temperature.
- In some cases, high pressure is also applied at high temperature to get a quality product.

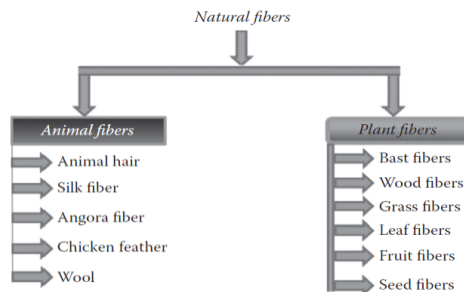


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Reinforcement

- Reinforcement in a composite may be fibrous or particulate.
- Both form are widely used but major development is in the area of fibres. Fibres can be either natural or synthetic.
- Natural fibers were obtained from plant kingdom (cotton jute, hemp, sisal) as well as animal kingdom (hair, wool and silk).



Vijay K. Thakur, Green Composites, CRC Press, 2014

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25

Mechanical Properties of Natural Fibers

Fiber	Density (g/m ³)	Length (mm)	Diameter (μm)	Elongation at break (%)	Tensile strength* (MPa)
Cotton	1.21	15–56	12–35	2–10	287–597
Coir		0.3–3.0	7–30	15–25	
Flax	1.38	10–65	5–38	1.2–3	343–1035
Jute	1.23	0.8–6	5–25	1.5–3.1	187–773
Sisal	1.20	0.8–8	7–47	1.9–3	507–855
Hemp	1.35	5–55	10–51	1.6–4.5	580–1110
Henequen	1.4		8–33	3–4.7	430–580
Ramie	1.44	40–250	18–80	2–4	400–938
Kenaf (bast)	1.2	1.4–11	12–36	2.7–6.9	295–930
Kenaf (core)	0.31	0.4–1.1	18–37		
Pineapple	1.5	3–8	8–41	1–3	170–1627
Bagasse	1.2	0.8–2.8	10–34	0.9	20–290
Southern yellow pine	0.51	2.7–4.6	32–43		
Douglas fir	0.48	2.7–4.6	32–43		
Aspen	0.39	0.7–1.6	20–30		

Kim L. Pickering, Properties and performance of natural-fibre composites, Woodhead Publishing Ltd, 2008.

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26

Natural Fibers - Advantages

- Natural fibers are cheap (approximately 30% lower than glass fibers) and produced from renewable sources, but their mechanical properties are poor compared to synthetic fibers.
- The density values of natural fibers are roughly one-half of the density of glass fiber. Hence, the specific strength and modulus values are comparable to glass fibers.
- Suitable for applications where the load-bearing capacity is not very critical.
- A major advantage of natural fibers is their biodegradability. The polymer composites made using natural fibers can be thermally decomposed easily, whereas glass fiber causes problem in combustion furnaces.

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27

Natural Fibers - Disadvantages

- There is a wide variation in dimensions and properties as fiber quality depends on the place of cultivation and weather conditions.
- The price of the fiber depends on the harvest results and agricultural policies.
- The strength and stiffness of these fibers are generally lower than synthetic composites.
- Even though the specific strength and stiffness of natural fibers are comparable to glass fibers, the actual mechanical properties are lower.
- Moisture absorption is a major problem of the natural fibers as they swell on moisture absorption. Hence, they are only suitable for indoor applications.
- The maximum processing temperature of composites is limited due to the degradation of natural fibers.

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28

Synthetic Fibers

The production, structure and properties of synthetic fibers all affects the composite properties.

Material	Diameter (μm)	Density (ρ) (g/cm ³)	Tensile Modulus (E) (GPa)	Tensile Strength (σ) (GPa)	Specific Modulus (E/ρ)	Specific Strength	Melting Point (°C)	% Elongation at Break	Relative Cost
Fibers									
E-glass	7	2.54	70	3.45	27	1.35	1540+	4.8	Low
S-glass	15	2.50	86	4.50	34.5	1.8	1540+	5.7	Moderate
Graphite, high modulus	7.5	1.9	400	1.8	200	0.9	>3500	1.5	High
Graphite, high strength	7.5	1.7	240	2.6	140	1.5	>3500	0.8	High
Boron	130	2.6	400	3.5	155	1.3	2300	—	High
Kevlar 29	12	1.45	80	2.8	55.5	1.9	500(D)	3.5	Moderate
Kevlar 49	12	1.45	130	2.8	89.5	1.9	500(D)	2.5	Moderate
Bulk materials									
Steel		7.8	208	0.34–2.1	27	0.04–0.27	1480	5–25	<Low
Aluminum alloys		2.7	69	0.14–0.62	26	0.05–0.23	600	8–16	Low

S. K. Mazumdar, Composites Manufacturing, CRC Press, Indian Edition 2010

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29

Tensile strength of fibers

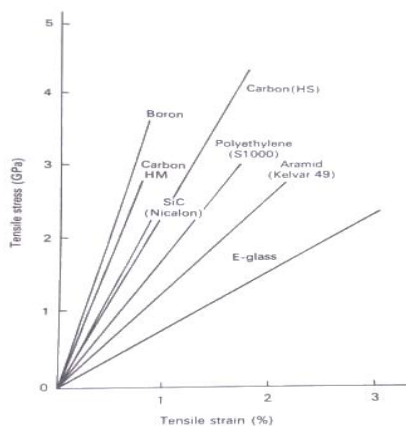


Fig. Stress-Strain curve of various fibers

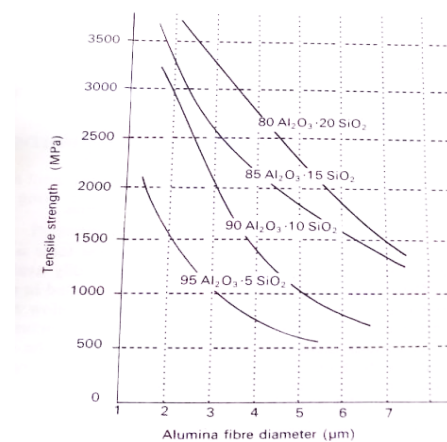


Fig. Tensile strength as a function of fiber diameter

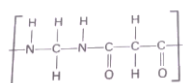
Matthews F. L. and Rawlings R. D., Composite Materials, CRC Press, 1999

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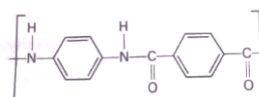
30

Synthetic organic fibers - Aramid

- Aramid is the short form of aromatic polyamide. Aromatic polyamides contain rigid rodlike polymer molecules.
- Kevlar, Twarlon and Technora are some examples of aramids.
- Aramid fibers provide the highest tensile strength-to-weight ratio among reinforcing fibers.
- Aramids can be considered as nylon with extra benzene rings in polymer chain.



a) Nylon



b) Aramid

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31

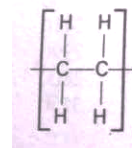
Synthetic organic fibers - Aramid

- Aramid fibers are produced by extruding an acidic solution (a proprietary polycondensation product of terephthaloyl chloride and *p*-phenylenediamine) through a spinneret.
- The filaments are drawn through several orifices. During the drawing operation, aramid molecules become highly oriented in the longitudinal direction.
- They provide good impact strength. Like carbon fibers, they provide a negative coefficient of thermal expansion.
- The disadvantage of aramid fibers is that they are difficult to cut and machine.

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32

Synthetic organic fibers -Polyethylene



- Polyethylene is a simple polymer with $\text{--CH}_2\text{--CH}_2\text{--}$ repeating units.
- Generally the molecules in bulk polyethylene material assume the random coil configuration resulting in poor mechanical properties.
- Polymer chains need to be aligned with full extension to get better properties of this polymer.
- Theoretical elastic modulus of covalent C-C bond in fully extended form is 220 GPa along the direction of chain.
- Although this is not fully achieved by oriented structure of fibre, value of 172GPa is impressive.
- Polyethylene has lowest density of readily available fibers but has a lowest melting point of 135 °C so use is restricted to temperature below 100 °C.
- These fibers can be treated with gas plasma for increased strength and stiffness but reduced toughness.

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33

Synthetic inorganic fibers - Glass

- Glass is a noncrystalline material with a short range network structure.
- It has no distinctive microstructure and the mechanical properties are isotropic.
- Silica, oxynitride, phosphate and halide glasses are group of glasses but from the point of composite, only silica glasses are important.
- There are four main types of *glass* used in fiberglass
 - A-glass (Alkali it contains)
 - C-glass (Chemical attack resistant)
 - E-glass (Electrical applications –alkali free)
 - S-glass (Stiff – High Strength)
- Most widely used fiber e.g. piping, tanks, boats, sporting goods
- Advantages are low cost and Corrosion resistance
- Disadvantages are relatively low strength, high elongation, moderate strength and weight.

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34

Synthetic inorganic fibers - Alumina

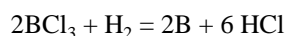
- Alumina can adopt several crystal structures, some of which are metastable.
- Different alumina phases are alpha-(α), delta-(δ), gamma (γ), and eta (η) alumina.
- Alumina has a high melting point in excess of 2000°C and a relatively low viscosity in molten stage.
- Alumina fibers are polycrystalline and are not 100% Al_2O_3 but contains other oxides often in appreciable quantities.
- The most common oxide addition is SiO_2 and fibers upto 20% SiO_2 are manufactured.

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35

Synthetic inorganic fibers - Boron

- Boron fiber is produced by chemical vapor deposition from boron trichloride on a heated substrate.
- High melting point substrate is required due to high temperature involved.
- Heated tungsten wire of 10 μm diameter is most commonly used substrate.
- Below reaction give an approximately 50 μm thick coating of boron with grain size of only 2-3 μm .



- Fibers are subjected to annealing and chemical treatment to reduce residual stresses and strength respectively.

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36

Synthetic inorganic fibers - Carbon

- Carbon has two well known crystalline forms (diamond and graphite) but it also exists in quasi-crystalline and glassy states.
- One of the important advantages of using carbon fibers in composites is superior fatigue resistance.
- Unlike glass or aramid fibers, carbon fibers do not undergo stress rupture but undergo complete elastic recovery upon unloading.
- Graphite is most widely used form of carbon in fiber technology.
- Another important characteristic of carbon fiber is its negative coefficient of thermal expansion (CTE) in the axial direction at room temperature.
- CTE is slightly negative for low-modulus carbon fibers and more negative for high-modulus carbon fibers. However it becomes positive above 700°C.

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37

Synthetic inorganic fibers - Silicon

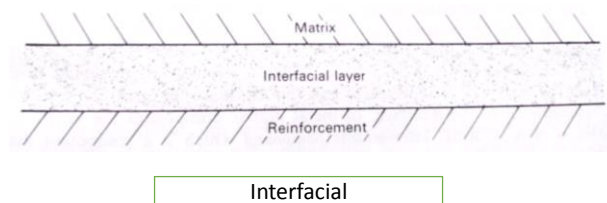
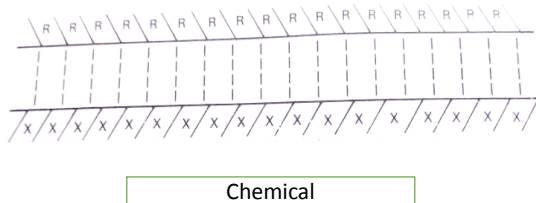
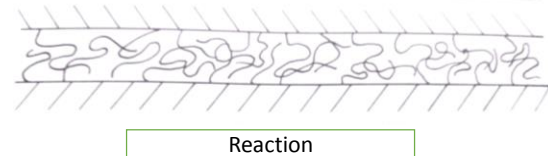
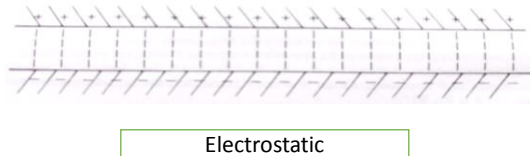
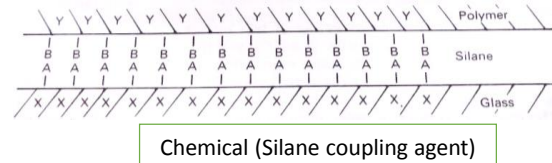
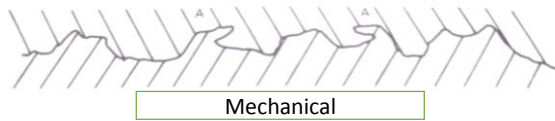
- SiC is widely used as reinforcement for ceramics.
- SiC bonding is covalent so the fibers are brittle but have a high modulus.
- Density of SiC fibres is high so specific properties are inferior to carbon and polyethylene.
- SiC is produced from carbon containing silane

$$\text{CH}_3\text{SiCl}_3 \longrightarrow \text{SiC} + 3\text{HCl}$$
- Substrate core is usually tungsten although carbon is also used

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38

Interfacial Bonding



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Matthews F. L. and Rawlings R. D., Composite Materials, CRC Press, 1999

39

Mechanical Bonding

- Rougher the surface \Rightarrow greater interlocking \Rightarrow more effective bonding.
- Shear strength is considerable.
- Tensile strength is low unless there is a high density of re-entrant angles.

Electrostatic Bonding

- Bonding occurs when one surface is +ively charged and other -ively leading to electrostatic attraction.
- Electrostatic interactions are short range and are only effective over atomic distances.
- Matrix and reinforcement should be in intimate contact.

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40

Chemical Bonding

- Bond formed between chemical groups of reinforcement and compatible groups in matrix.
- Strength depends on no. of bonds per unit area.
- Coupling agents were used for better chemical bonding.

Reaction Bonding

- Forms due to intertwining of molecules.
- Strength depends on extent of entwined distance and entanglement of molecules, number of molecules per unit area

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41

Interdiffusion Bonding

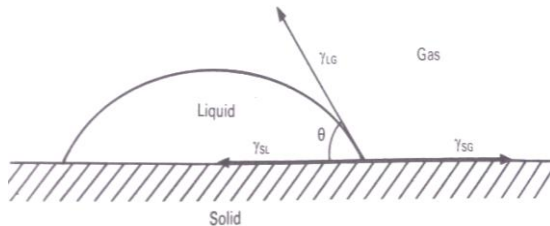
- Interdiffusion of two components produces interfacial layer of different composition and structure from either of two components.
- Different mechanical properties from either of two components.
- Interfacial layer is often a brittle intermetallic compound.

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42

Wettability

Wettability defines the extent of spread of a liquid over a solid surface.



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

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

$$\cos \theta = (\gamma_{SG} - \gamma_{SL}) / \gamma_{LG}$$

If $\theta = 0$, perfect wetting

$0^\circ < \theta < 180^\circ$, wetting will increase as θ decreases

Liquid does not wet the surface if

$$\theta > 90^\circ$$

$$\gamma_{SL} dA + \gamma_{LG} dA \leq \gamma_{SG} dA$$

$$\gamma_{SL} + \gamma_{LG} \leq \gamma_{SG}$$

Matthews F. L. and Rawlings R. D., Composite Materials, CRC Press, 1999

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43

Volume and weight fractions

$$v_c = v_f + v_m$$

$$V_f = \frac{v_f}{v_c} \quad V_m = \frac{v_m}{v_c}$$

$$w_c = w_f + w_m$$

$$W_f = \frac{w_f}{w_c} \quad W_m = \frac{w_m}{w_c}$$

$$\rho_c v_c = \rho_f v_f + \rho_m v_m$$

$$\rho_c = \rho_f \frac{v_f}{v_c} + \rho_m \frac{v_m}{v_c}$$

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

$$\rho_c = \frac{1}{(W_f/\rho_f) + (W_m/\rho_m)}$$

$$W_f = \frac{w_f}{w_c} = \frac{\rho_f v_f}{\rho_c v_c} = \frac{\rho_f}{\rho_c} V_f$$

$$W_f = \frac{\rho_f}{\rho_c} V_f$$

$$W_m = \frac{\rho_m}{\rho_c} V_m$$

$$V_f = \frac{\rho_c}{\rho_f} W_f$$

$$V_m = \frac{\rho_c}{\rho_m} W_m$$

$$\rho_c = \sum_{i=1}^n \rho_i V_i$$

$$\rho_c = \frac{1}{\sum_{i=1}^n (W_i/\rho_i)}$$

$$W_i = \frac{\rho_i}{\rho_c} V_i$$

$$V_i = \frac{\rho_c}{\rho_i} W_i$$

44

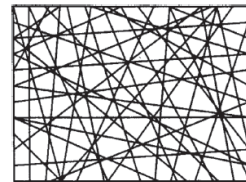
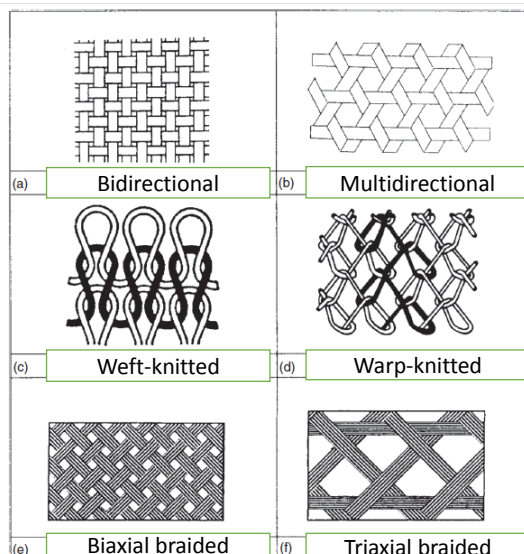
Fiber architecture

- Arrangement of fibers in a composite.
- Mechanical properties are affected by fiber continuity, orientation, crimping and interlocking.
- During processing fiber architecture determines
 - Void content
 - Fiber wetting
 - Fiber distribution
 - Dry area
- Fiber architecture can be 1D, 2D or 3D.

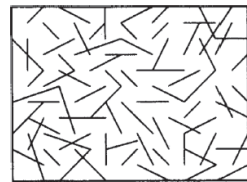
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45

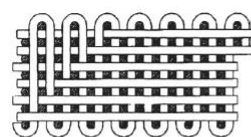
Fiber Architecture



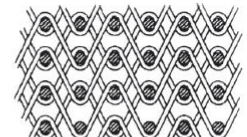
Continuous fiber mat



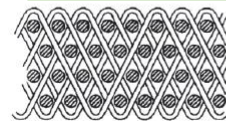
Chopped strand mat



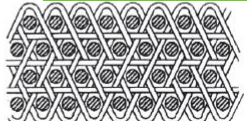
(i) 3D



(ii) 2D or angle interlock



(iii) 3X



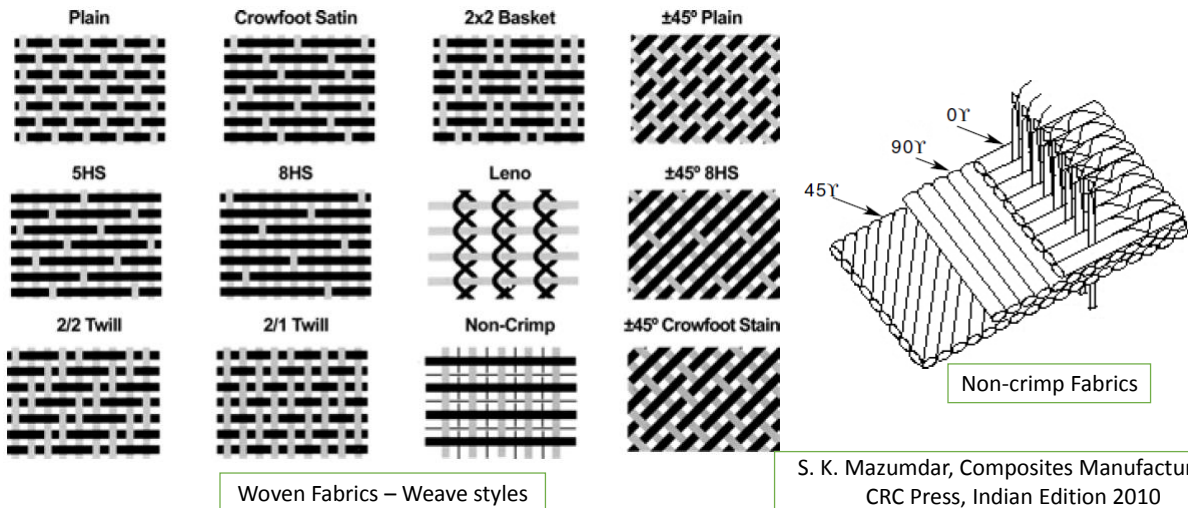
(iv) 3X with warp stiffer yarn

P. K. Mallick, Fiber Reinforced Composites, CRC Press Boca Raton, 2013

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46

Woven and Non-crimp Fabrics



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47

Whiskers

- On the basis of diameter and character fibers are of three types:
 - Whiskers, fibers and wire
- Whiskers are very thin single crystals with extremely large l/d ratio.
- They have a high degree of crystalline imperfection and are virtually flaw free.
- They are among the strongest known materials.
- Whiskers material include graphite, silicon carbide, silicon nitride and aluminum oxide.
- They are extremely expensive so not used extensively as reinforcement.

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48

Unit 2: Polymer Matrix Composites

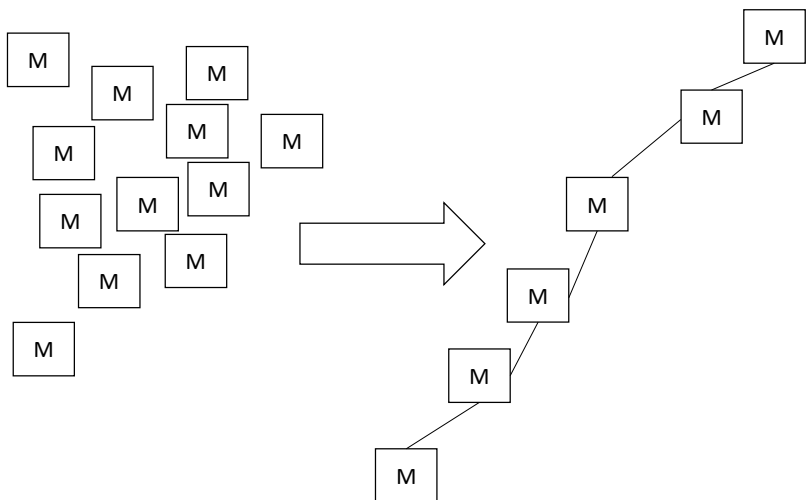


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1

Polymer: Basics

- Polymer means many units.
- A single molecular unit that made a polymer is called a monomer.
- Monomers have a specific and consistent grouping of atoms.
- Monomers are linked together into a single long chain called as polymer molecule



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2

Polymer: Basics

- Resins are polymeric materials that are not in their final form and shape. Most of the common resins are liquids although they exist also as solid materials.
- Plastics refer to the final shape of polymeric materials.
- A resin is changed into a plastic through some forming or molding process.
- “Polymer” and “resin” refer to naturally occurring and synthetic materials both, whereas plastic is used only to refer to synthetics.
- The process of linking of small units (monomers) into long chains (polymers) is called polymerization.
- The two most common mechanisms are addition polymerization and condensation polymerization.

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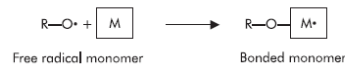
3

Addition Polymerization

Step 1: Free radical formation



Step 2: Bonding with the monomer (monomer activation)



Step 3: Chain growth



Step 4: Further chain growth showing many (n) repetitions



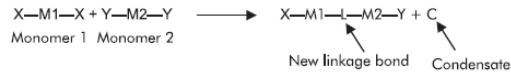
Step 5: Termination

General representation: $\left[\text{M} \right]_n$

A. Brent Strong, Fundamentals of Composites Manufacturing, Society of Manufacturing Engineers, Dearborn, Michigan 2008

Condensation Polymerization

Step 1: Monomers react to form a new molecule and a condensate



Step 2: Reaction of an end of the linked molecule with a new monomer



Step 3: Reaction with a new monomer



General representation: -(M1-L-M2)-_n

A. Brent Strong, Fundamentals of Composites Manufacturing, Society of Manufacturing Engineers, Dearborn, Michigan 2008

Thermosets

- Thermosets are resins that are usually liquids (or easily melted solids) at room temperature. They are placed into a mold and then solidified into the desired shape by a process other than freezing.
- The molded material is set in its shape by a heating process in which bonds are formed between the molecules. These bonds or crosslinks change the basic nature of the material.
- After the polymer material has been heated and the crosslink bonds formed, which is called curing, the thermoset material can no longer be melted. Hence, thermosets will not melt upon heating in contrast to thermoplastics, which are capable of being remelted.
- Most common thermosets are epoxy, polyester, vinylester, phenolics, cyanate esters, polyimides.

Thermoplastics

- Thermoplastics are resins that are solids at room temperature. They are melted or softened by heating, placed into a mold or other shaping device when in the molten stage, and then cooled to give the desired shape.
- Several molding processes, such as extrusion, injection molding, blow molding, and thermoforming, are based on this type of resin behavior.
- Some common thermoplastics are Nylon, Polyester, Polyethylene, Teflon, Polyetheretherketone (PEEK), Polypropylene (PP).

Glass transition temperature (T_g)

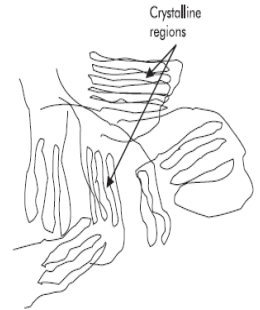
- Thermal transition that occurs in the solid phase and marks a change from a rigid solid to a more pliable state is T_g .
- Atoms motion in a rigid, solid material below T_g are generally simple vibrations in rigidly fixed positions.
- Above T_g but below the melting point, the vibrations and rotations are more expansive and the atoms may move laterally for short distances. However, they are still locked into relatively fixed positions due to continued entanglements.
- Between the T_g and the melting point, the material becomes somewhat pliable, although still a solid. The state above the glass transition is referred to in these materials as the “leathery state.”

Amorphous and Crystalline Structure

- In thermoplastics, several interactions less permanent than crosslinks occurs. Any interaction (entanglement or intertwining or secondary bonding) or association between molecules increases the melting point of the polymer.
- Some polymers pack closely together in the solid state and are therefore called crystalline regions. The crystalline regions are held together by secondary bonds, or crystalline bonds, which increase the melting point in these regions.
- Most polymers having crystalline regions are thermoplastics as the crosslinks in thermosets interferes with the packing of the polymers. Molecular motion in a crystalline region is highly restricted.
- The crystalline part of a group of molecules would not have a T_g , but other regions in the same molecular mass where no crystallinity exists could have a T_g . Such mixed polymers are called semicrystalline.



a. Amorphous (random entanglement)



b. Semi-crystalline or crystalline (regular packing)

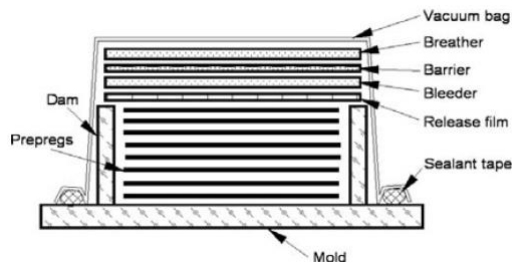
A. Brent Strong, Fundamentals of Composites Manufacturing, Society of Manufacturing Engineers, Dearborn, Michigan 2008

Basic Steps of Composite Manufacturing

- **Impregnation**
Fibers and resins are mixed together to form a lamina.
- **Lay-up**
Composite laminates are formed by placing fiber resin mixture or preregs at desired angles.
- **Consolidation**
Creates intimate contact between each layer of prepreg or lamina.
- **Solidification**
Final step is solidification or curing.

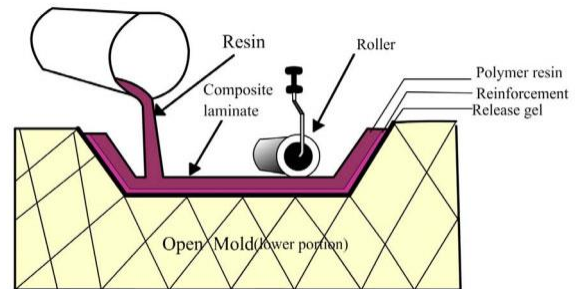
Hand Layup Process

• Prepreg Lay-up Process



S. K. Mazumdar, Composites Manufacturing, CRC Press, Indian Edition 2010

• Wet Lay-up Process



<https://www.eppcomposites.com/hand-layup-process.html>

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11

Advantages and Limitations

Pre-preg layup

Advantages

- allows production of high fiber volume fraction (more than 60%)
- Simple to complex parts can be easily manufactured.
- low tooling cost investment for the autoclave.
- Very strong and stiff parts can be fabricated using this process.

Limitations

- Labor intensive
- Not suitable for high-volume production applications.
- The parts produced are expensive

Wet lay-up

Advantages

- Very low capital investment
- very simple and versatile
- cost of making a prototype part is low

Limitations

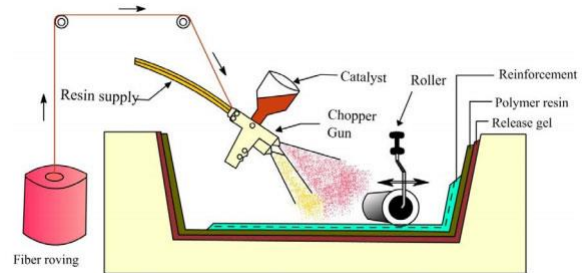
- labor intensive process
- styrene emission is a major concern
- quality is not consistent from part to part
- High fiber volume fraction parts cannot be manufactured

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12

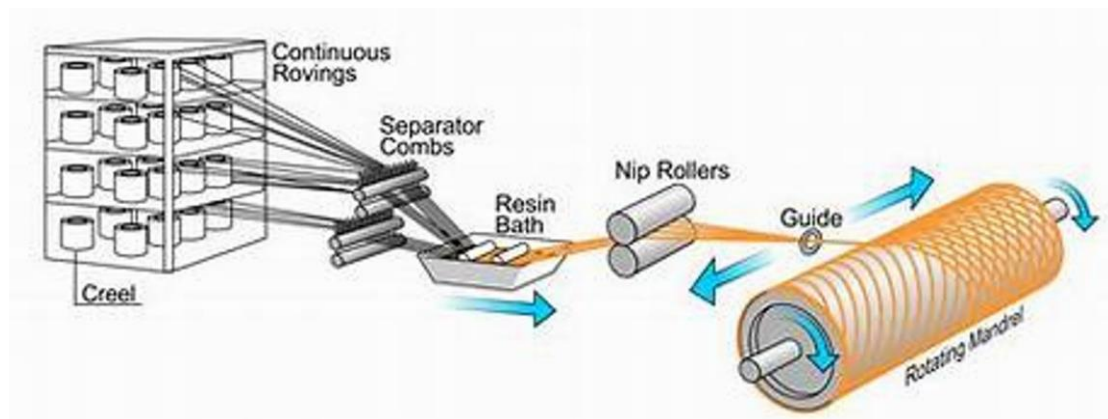
Spray-up Process

- This is partial automation of hand lay-up process.
- Two spray systems were used
 - External mixing (two nozzles)
 - Internal mixing (single nozzle)



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Filament Winding



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<https://idsiofga.com/filament-winding>

Filament Winding

Methods of Applying Heat and Pressure

- The pressure during filament winding is applied by creating fiber tension.
- In general, 1 lbf to 6 lbf fiber tension is created using some tensioning device or by passing the fibers through the carriage unit in such a way that it creates tension.
- Composites thus fabricated are cured at room temperature, or in an oven at a higher temperature.
- For large-volume production, the process of part fabrication is automated.
- In an automated line, the filament wound part with the mandrel is moved to a heated chamber using a robot.
- The part slowly moves in the heated chamber and comes out after partial or full cure of the composite part.
- The part is then sent to the mandrel extracting station where the mandrel is extracted and sent back to the filament winding machine for winding purposes.

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16

Filament winding

Advantages

- Filament winding is the only method that can be used to make cost effective and high-performance pressure vessels and fuel tanks
- Utilizes low-cost raw material systems and low cost tooling
- Filament winding can be automated for high volume production

Limitations

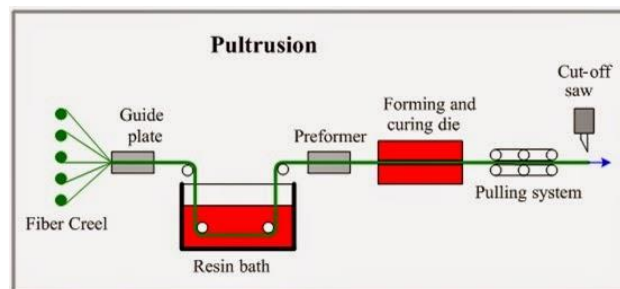
- It is limited to producing closed and convex structures.
- Not all fiber angles are easily produced during the filament winding process.
- Low fiber angles (0 to 15°) are not easily produced.
- Maximum fiber volume fraction attainable is only 60%.
- It is difficult to obtain uniform fiber distribution and resin content throughout the thickness of the laminate.

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17

Pultrusion Process

- In Pultrusion process resin-impregnated fibers are pulled through a heated die at a constant speed to make the part.
- Process is similar to the extrusion process, but instead of pushing the metal metal through the die it is pulled through die in this process.



Dr. Vikas Upadhyay, MED, NIT Patna <http://pultruder.blogspot.com/2014/05/what-is-pultrusion.html>

Pultrusion Process

- Used to fabricate a wide range of solid and hollow structures with constant cross-section.
- Typically used for making parts with unidirectional fibers. Fabrics and mats are also used for bi or multi direction properties.
- Unsaturated polyester is the most common resin material. Vinyl esters and epoxies can be used but they are difficult to process and pulling speeds are also low.
- Steel dies are used to provide desired shape to resin impregnated fibers.

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19

Pultrusion Process Contd.

Advantages

- It is a continuous process.
- Can be completely automated to get the finished part.
- Suitable for making high-volume composite parts.
- Typical production speeds are 2 to 10 ft/min.
- Low-cost fiber and resin are used.

Limitations

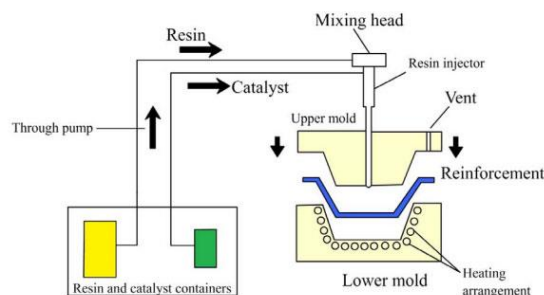
- Suitable for parts with constant cross-sections along their length.
- Tapered and complex shapes cannot be produced.
- Very high-tolerance parts can not be produced
- Thin wall parts cannot be produced.
- Fiber angles on pultruded parts are limited to 0°.

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20

Resin transfer Molding

- RTM is a liquid transfer molding process.
- A pre-catalyzed resin is supplied under pressure to a heated mold cavity that contains a porous fiber preform.
- It offers the fabrication of near net shape complex parts.
- Resin flow, curing and heat transfer in porous media are important parameters in RTM.



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<https://www.eppcomposites.com/rtm-process.html>

Basic Steps

1. A thermoset resin and catalyst are placed in tanks of the dispensing equipment.
2. A release agent is applied to the mold for easy removal of the part. Sometimes, a gel coat is applied for good surface finish.
3. The preform is placed inside the mold and the mold is clamped.
4. The mold is heated to a specified temperature.
5. Mixed resin is injected through inlet ports at selected temperature and pressure. Sometimes, a vacuum is created inside the mold to assist in resin flow as well as to remove air bubbles.
6. Resin is injected until the mold is completely filled. The vacuum is turned off and the outlet port is closed. The pressure inside the mold is increased to ensure that the remaining porosity is collapsed.
7. After curing for a certain time (6 to 20 min, depending on resin chemistry), the composite part is removed from the mold.

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22

Advantages

1. Initial investment cost is low because of reduced tooling costs and operating expenses as compared to compression molding and injection molding.
2. Moldings can be manufactured close to dimensional tolerances.
3. RTM processing can make complex parts at intermediate volume rates.
4. RTM provides for the manufacture of parts that have a good surface finish on both sides.
5. RTM allows for production of structural parts with selective reinforcement and accurate fiber management.
6. Higher fiber volume fractions, up to 65%, can be achieved.
7. Inserts can be easily incorporated into moldings and thus allows good joining and assembly features.
8. A wide variety of reinforcement materials can be used.
9. RTM offers low volatile emission during processing because of the closed molding process.
10. RTM offers production of near-net-shape parts.
11. The process can be automated.

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23

Limitations

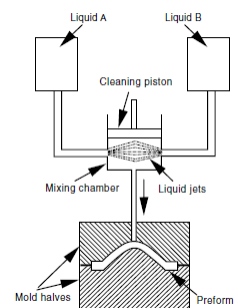
1. The manufacture of complex parts requires a good amount of trial and-error experimentation or flow simulation modeling to ensure that porosity- and dry fiber-free parts are manufactured.
2. Tooling and equipment costs for the RTM process are higher than for hand lay-up and spray-up processes.
3. The tooling design is complex.

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24

Structural Reaction Injection Molding

- In the SRIM process, two resins A and B are mixed in a mixing chamber at a very high velocity just before injecting into the mold.
- The resin flows at a speed of 100 to 200 m/s and collides in the mixing chamber.
- The pressure generated during collision is in the range of 10 to 40 MPa although the resin is injected into the mold at a pressure of less than 1 MPa.
- Low pressure is used to prevent the wash-out of fibers at the injection port.
- The resin used for the SRIM process is of very low viscosity and the most common resin is polyisocyanurate.



S. K. Mazumdar,
Composites Manufacturing,
CRC Press, Indian Edition
2010

25

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Advantages

1. It is very suitable for making high-volume structural parts at low cost, in particular for making automotive parts.
2. Small- to large-sized parts with complex configurations can be made with this technique.

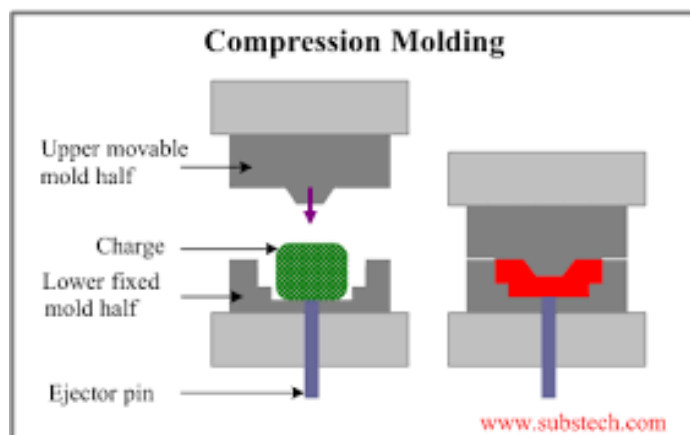
Limitations

1. It requires a large capital investment in equipment.
2. The tooling cost for the SRIM process is high.
3. A high fiber volume fraction cannot be attained by this process; the maximum fiber volume fraction achievable is about 40%.

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26

Compression Molding



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27

Compression Molding

- SMC is cut into rectangular sizes (Charge) and placed on the bottom half of the preheated mold.
- The charge usually covers 30 to 90% of the total area, and the remaining area is filled by forced flow of the charge.
- The mold is closed by bringing the upper half of the mold to a certain velocity (Typically 40 mm/s)
- Molds are usually preheated to about 140°C.
- With the movement of mold, the charge starts flowing inside the mold and fills the cavity.
- After a reasonable amount of cure under heat and pressure, the mold is opened and the part is removed from the mold.

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28

Sources of variation

1. *Press parallelism.* The thickness variation in a molded part depends on press parallelism tolerances as well as mold tolerances. Each equipment supplier has its own tolerances on parallelism. Advance-control presses provide low tolerance variation, but they are expensive.

2. *Mold tolerances.* Every mold and tool has tolerances that can affect part tolerances. The use of computer math data in cutting the part surface of the mold increases the accuracy of the mold over large surface areas.

3. *Molded datum features.* In dimensioning, geometric characteristics of a part are established from a datum. A datum feature is an actual feature of a part that is used to establish a datum. The use of molded datum features will increase the repeatability in bonded assemblies. The molded datums reduce the need to locate and maintain datums on the secondary fixtures and thus reduce additional sets of tolerances.

4. *Material shrinkage.* Shrinkage is the reduction in volume or linear dimension caused by curing of the resin as well as by thermal contraction of the material. Curing shrinkage occurs because of the rearrangement of polymer molecules into a more compact mass. The volumetric shrinkage of cast polyester and vinylester resins is in the range of 5 to 12%, whereas for epoxy, it is 1 to 5%. The addition of fibers and fillers reduces the amount of shrinkage.

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29

Guidelines for mold parts

1. *Avoid flatness.* On flat panels, reflected highlights often make the panel look distorted and therefore a contoured surface is recommended to eliminate objectionable highlights on high-gloss surfaces.
2. *Maintain uniform thickness.* While designing the panel, maintain a uniform thickness across the length for uniform flow of material as well as for uniform cooling rate. This will avoid the risk of part distortion and telegraphing of thickness changes through the surface.
3. *Inner and outer radii.* Sharp corners must be avoided during design. A minimum inside corner radius of 0.08 in. and minimum outside corner radius of 0.06 in. are recommended for better material flow along the corner as well as for ease in part removal.
4. *Create mash-offs for hole location.* A mash-off is a localized reduction in panel thickness for creating holes during secondary operation. It is a membrane-like thin area that can be easily removed. Charge can be placed directly over the mash-off area. This promotes a more homogeneous material flow through the hole location, which improves mechanical properties around the hole.

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30

Advantages

1. High-volume production and thus is suitable for automotive applications. The mold cycle time is only 1 to 4 min.
2. Low-cost components at high volume because it utilizes SMC, which is fairly inexpensive.
3. The process offers high surface quality and good styling possibilities.
4. Multiple parts can be consolidated into one single molded part.
5. Compressive molding of SMC offers shorter production runs and more rapid design-to-production schedules..

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31

Limitations

1. The initial investment for the process is high because of high equipment and mold costs. However, this initial investment is low compared to sheet metal stamping processes.
2. The process is not suitable for making a small number of parts or for prototyping applications.
3. Compression molding of SMC provides nonstructural parts; but by utilizing ribs and stiffeners, structural parts can be manufactured.

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32

Injection Molding

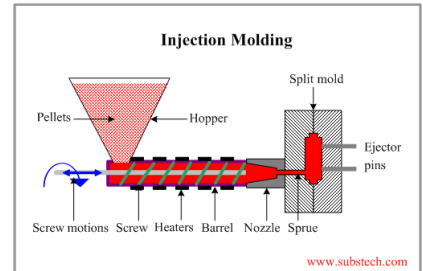
- In injection molding, a fixed amount of material is injected into the heated mold cavities.
- After the completion of cross-linking, the mold opens and the part is dropped into a receiving bin.
- Injection molding has the shortest process cycle time compared to any other molding operation and thus has the highest production rate. Typically, the complete process takes about 30 to 60 s.
- The production rate can be further increased by having a multiple-cavity mold.
- Injection molding is widely used for making small sized parts, but it can also be used to make large structures.

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33

Units of Injection Molding Machine

- **Plasticating and injection unit:** Plasticating unit melts the polymer, accumulates the melt in the screw chamber, injects the melt into the cavity and maintains the holding pressure during cooling.
- **Clamping unit:** It opens and closes the mold, and hold the mold tightly to avoid flash.
- **Mold cavity:** It distributes melted polymer in the cavities, shapes and cools the melt and ejects the finished product.

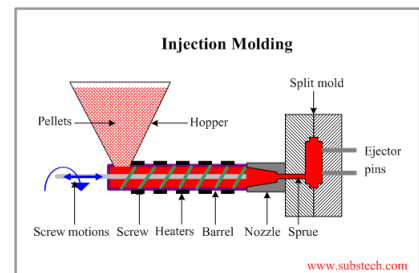


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34

Making of Part

- Polymer material in form of pellets is fed into an Injection Molding machine through a hopper. The material is then conveyed forward by a feeding screw and forced into a split mold, filling its cavity through a feeding system with sprue gate and runners.
- Injection Molding machine is similar to Extruder. The main difference between the two machines is in screw operation. In extruder screw rotates continuously providing output of continuous long product (pipe, rod, sheet).
- Screw of injection molding machine is called reciprocating screw since it not only rotates but also moves forward and backward according to the steps of the molding cycle. It acts as a ram in the filling step when the molten polymer is injected into the mold and then it retracts backward in the molding step.
- Heating elements, placed over the barrel, soften and melt the polymer. The mold is equipped with a cooling system providing controlled cooling and solidification of the material.
- The polymer is held in the mold until solidification and then the mold opens and the part is removed from the mold by ejector pins.



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www.substech.com

35

Thermoplastic Composites

- Thermoplastic composites are widely used in aerospace and automotive industries because of their higher toughness, higher production rate, and minimal environmental concerns.
- Commercial thermoplastic manufacturing techniques are injection molding, compression molding, and, to some degree, the autoclave/prepreg lay-up process.
- Most of the manufacturing processes (filament winding and pultrusion) used for thermoset composites can be applied for the production of thermoplastic composite parts.
- In thermoplastics, processing takes place in seconds. Thermoplastics processing is entirely a physical operation because there is no chemical reaction as in thermoset composites.

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36

Autoclave Processing

- In an autoclave process, a thermoplastic composite part is made by placing the prepregs on the surface of the open mold, similar to the thermoset autoclave process.
- Here, the difference is that the prepreg layers are spot welded to avoid relative motion between plies.
- Prepregs can easily be laid on a complex shape with thermoset prepregs, whereas the laying of thermoplastic prepregs is difficult on a complex shape.
- Once the prepreg layers are laid in the desired sequence, the entire assembly is vacuum bagged similar to the thermoset autoclave process.

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37

Processing Steps

- Thermoplastic prepregs do not have good tack and drapability, significant numbers of air pockets remain at the interface.
- Once all the tapes are laid down, entire assembly is vacuum bagged for consolidation.
- After vacuum bagging, the entire assembly is placed inside the autoclave. Heat and pressure are applied similar to thermoset autoclave processing

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38

Processing steps contd.

- Pressure is applied in two ways: (1) by creating a vacuum inside the vacuum bag, or (2) external pressure by the autoclave.
- Pressure removes air from the interface and creates intimate contact. It also helps in the flow of resin material during consolidation.
- Once two adjacent layers come into intimate contact, a bonding process between the interfaces begins.
- Bonding is primarily caused by the autohesion process for two similar thermoplastic interfaces. During autohesion, segments of the chain-like molecules diffuse across the interface. The extent of the molecular diffusion and hence the bond strength increase with time.

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39

Processing steps contd.

- The amount of molecular diffusion depends on the temperature and duration, as is evident from Equations given below

$$D_{au} = \chi \cdot t_a^{1/4}$$

where t_a is the time elapsed from the start of the autohesion process and χ is a constant. The parameter χ depends on the temperature T as shown in the following Arrhenius relation.

$$\chi = \chi_0 \exp\left(-\frac{E}{RT}\right)$$

where χ_0 is a constant, E is the activation energy, and R is the universal gas constant.

- After bond formation assemble is taken out and the vacuum bag is removed

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40

Advantages

- It provides fabrication of structural composite components with a high fiber volume fraction.
- It allows production of any fiber orientation.
- It is simple and basically a replicate of autoclave processing of thermoset composites.
- The same autoclave equipment as in thermoset processing is used for thermoplastic autoclave processing.
- It is suitable for making prototype parts.
- The tool design is simple for autoclave processing.

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41

Limitations

- Due to lack of tack and drapability, prepreg lay-up during autoclave processing of thermoplastic composites is labor intensive.
- A high capital investment is required.
- Processing of thermoplastic composites is difficult as compared to thermoset composites.
- Higher temperatures and pressures are required due to the high melt temperature and higher viscosity of thermoplastics.

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42

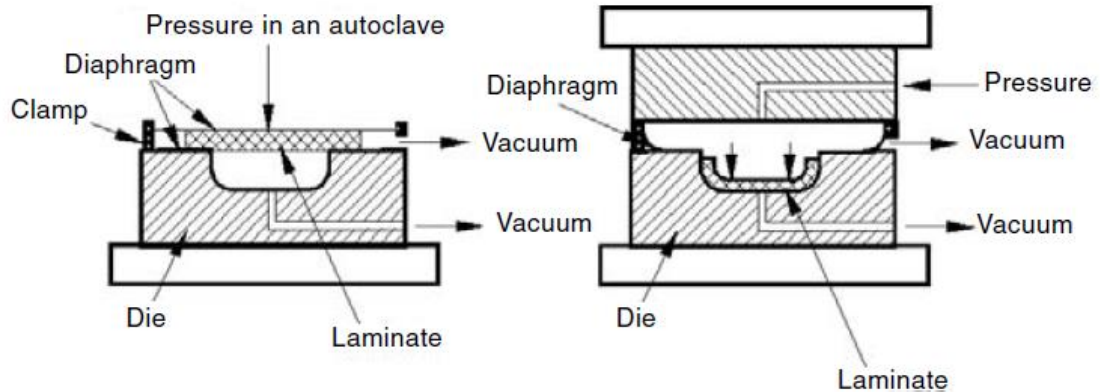
Diaphragm Forming Process

- In the diaphragm forming process, prepreg layers in the form of a composite sheet are placed between two flexible diaphragms and then formed under heat and pressure against a female mold.
- Vacuum ports are fabricated in the mold for creating vacuum between the lower diaphragm and the female mold surface.
- The diaphragm materials are usually superplastic aluminum alloys, polyimide films, Upilex, or sheet rubber.
- The diaphragm controls the forming process. The stiffness of the diaphragm is a critical factor in getting a good quality part.
- For simple shapes, compliant diaphragms are preferred; whereas for complex shapes, stiffer diaphragms are selected.

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43

Diaphragm Forming Process



S. K. Mazumdar, Composites Manufacturing, CRC Press, Indian Edition 2010

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44

Advantages

- It offers excellent structural properties because continuous fibers are used in making the part.
- Reasonably complex shapes with uniform thickness can be produced with reasonably high production efficiencies.

Limitations

- The process is limited to making parts that have constant thickness.
- Maintaining uniform fiber distribution during the manufacture of complex shapes is a challenge.
- In the diaphragm forming process, composite layers float between diaphragms and are free to have all the allowable modes of deformation resulting in significant reorientation of the reinforcing fibers.

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45

Injection Molding – Raw material

- Initial thermoplastic composite materials used in this process are in pellet or granular form.
- These pellets are formed by pultruding composite rods and then cutting them into small pieces about 10 mm in length.
- Another way to make fiber reinforced pellets is by passing a continuous strand through a coating die. Coated strands are then chopped, typically to a length of 10 mm.
- The final molded parts contain fibers that range from 0.2 to 6 mm in length. Fiber breaks when it passes through a screw barrel, nozzle, or other part of the equipment and mold.
- Primarily, glass fibers are used with various types of thermoplastics such as PP, nylon, PET, polyester, etc.

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46

Injection Molding - Process

- In injection molding, the pellets/molding compounds are first dried and then fed into the hopper.
- The feedstock moves through a heated chamber where it starts softening.
- Screw mechanism moves the feedstock close to the nozzle and completely melts the resin.
- The melted material is injected into the mold cavity at a pressure of 100 to 200 MPa.
- Once the resin fills the cavity, a holding pressure of about 50 to 100 MPa, depending on material type, is maintained to prevent the back-flow of filled resin.
- The duration to maintain this pressure depends on part size, melt temperature, and mold temperature.
- The mold is kept at 50 to 100°C so that the resin does not immediately solidify as it nears the cavity. The mold is maintained at that temperature by circulating coolant.
- With thermoplastic composites, the resin material does not have to come to room temperature for parts to be removed from the mold.

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47

Advantages

- This process allows production of complex shapes in one shot as inserts and core materials can be used in part fabrication.
- Part repeatability is much better in injection molding than in any other molding process. It offers tight dimensional control (± 0.002 in.).
- The process is a high-volume production method with a mold cycle ranging from 20 to 60 s.
- The process allows fabrication of low-cost parts because of its capacity for high-volume production rates. The process has very low labor costs.
- Small (5 g) to large (85 kg) parts can be made using this process.
- The process allows for production of net-shape or near-net-shape parts. It eliminates finishing operations such as trimming and sanding.
- The quality of the surface finish is very good.
- The process has very low scrap loss. Runners, gates, and scrap are recyclable.

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48

Limitations

1. The process requires significant capital investment. Lack of expertise in product design, manufacturing, and machine maintenance can cause high start-up and running costs.
2. The process is not suitable for the fabrication of low-volume parts because of high tooling costs.
3. The process is not suitable for making prototype parts. To get an idea of the design, rapid prototyping is preferred for visualization of the part before going for final production.
4. The process requires a longer lead time because of the time involved in mold design, mold making, computer simulation of the manufacturing process, debugging, trial and error, etc.
5. Because there are so many process variables (e.g., injection pressure, back-pressure, melt temperature, mold temperature, shot size, etc.), the quality of the part is difficult to determine immediately.

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49