Chapter 3
Matrices for Carbon Fiber Composites

Soo-Jin Park and Fan-Long Jin

Abstract Carbon fibers have become an important reinforcing material in advanced composites because of its extremely high strength, stiffness, heat resistance, and light weight. Fiber-reinforced polymer composites have gained substantial interest owing to their very high strength-to-weight and stiffness-to-weight ratios and are widely used in aerospace, engineering, marine, and automobile industries. In this chapter, we will present the various matrices and their characterization for carbon fiber composites. It is described as classification into two types of thermosetting resins and thermoplastic resins.

3.1 Thermosetting Resins

3.1.1 Introduction

Thermosetting resins are polymer materials, which cure irreversibly, generally found in the liquid form. The cure may be effected by heat [e.g., cyanate ester (CE) resins], chemical reaction (e.g., epoxy resins), and irradiation (e.g., vinyl ester resins) such as an infrared, ultraviolet light, or electron beam. Once cured, the material cannot be reheated and melted back to its original liquid form.

Thermosetting resins are easy to process and laminate, do not necessarily need either pressure or heat to form, generally inexpensive, stronger than thermoplastics, and better suited for higher temperatures compared to thermoplastics. However, they are relatively more brittle.

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Carbon fiber (CF) has become an important reinforcing material in advanced composites because of its extremely high strength, stiffness, heat resistance, and light weight. Fiber-reinforced polymer composites have gained substantial interest owing to their very high strength-to-weight and stiffness-to-weight ratios and are widely used in aerospace, engineering, marine, and automobile industries.

Among the thermosetting resins, CE, epoxy, phenolic, polyester, polyimide, and vinyl ester resins can be used as polymer matrices for CF-reinforced polymer composites [1–12].

3.1.2 Cyanate Ester Resins

CE resins belong to a class of high-performance thermosetting resins and contain at least two cyanate functional groups [13, 14]. Most commercial monomers can be represented by the structural model illustrated in Fig. 3.1.

CE resins can be cured by heating alone, either at elevated or lower temperatures in the presence of a suitable catalyst. The most common catalysts are transition metal complexes such as cobalt, copper, manganese, and zinc complexes. The chemistry of the cure reaction for CE resins involves the trimerization of three CN groups to form a triazine ring. The CE resins have two cyanate groups, which resulted in a three-dimensional polymer network structure, as shown in Fig. 3.2 [15].

CE resins will homopolymerize into a thermosetting material suitable for use in high-performance composites such as printed circuit boards, structural composites, and radomes. These resins have good processability, shelf-life, and compatibility with a variety of reinforcements.

CEs are a family of high-temperature thermosetting resins, which bridge the gap in the thermal performance between the engineering epoxy resins and high-temperature polyimides. In addition to their outstanding thermal performance, CE resins have several desirable characteristics, which justify their higher costs in many applications. CEs possess a unique balance of properties and are particularly notable for their low dielectric constant and dielectric loss, low moisture absorption, low shrinkage, and low outgassing characteristics. They can be used in high-performance adhesives, syntactic foams, honeycombs, and fiber-reinforced composites. They are often found in blends with other thermosetting resins such as epoxy, bismaleimide, and engineering thermoplastics [16, 17].

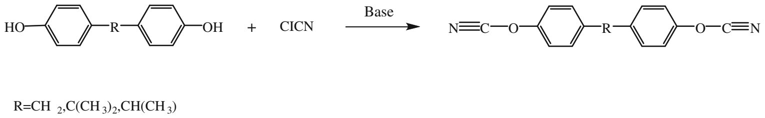


Fig. 3.1 Schematic of synthesis of CE monomers

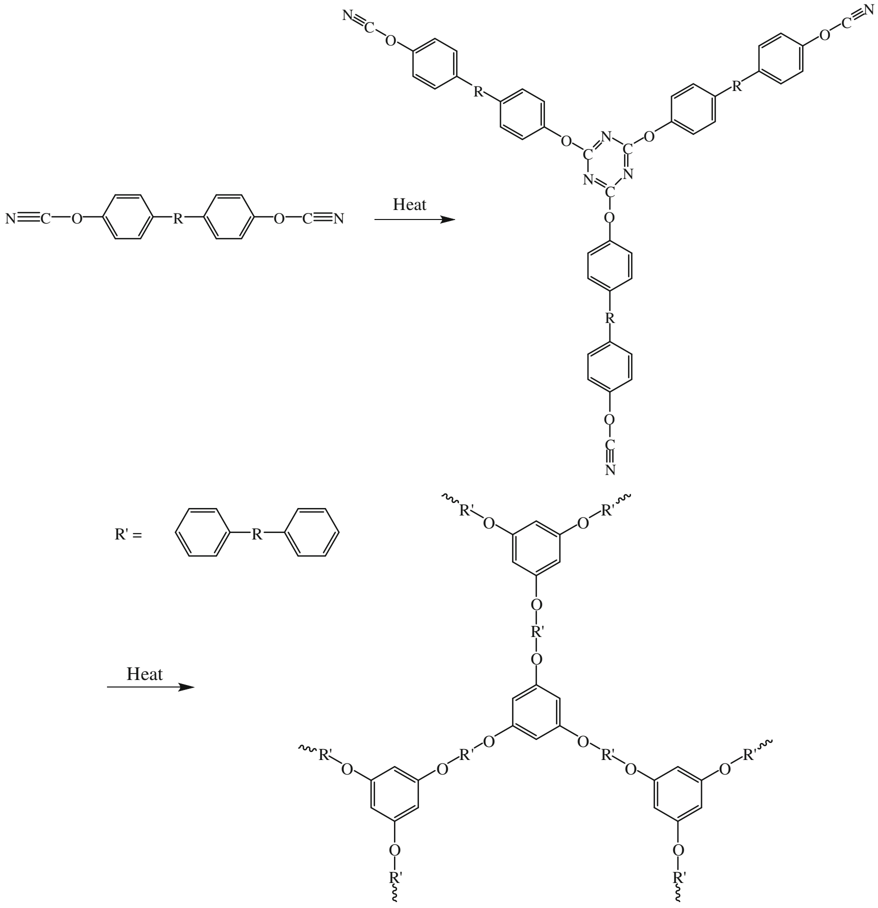
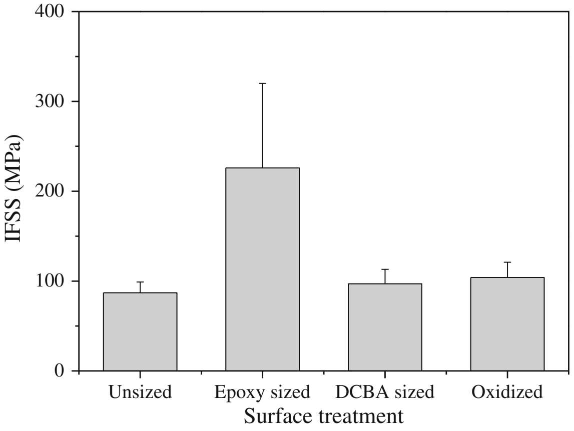


Fig. 3.2 Curing of CE resins via cyclotrimerization

Fully cured bisphenol-E CE has glass transition temperature (*T*g) and decomposition temperature (*T*d), at 5 wt% loss, of 274 and 438 °C, respectively [18, 19].

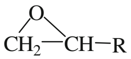
Marieta et al. [14] studied the effect of surface treatment on the interfacial shear strength (IFSS) of bisphenol-A dicyanate (DCBA)/PAN-based CF composites. Figure 3.3 shows the apparent IFSS values of the composites with different treatments. The results indicated that the commercial sizing of epoxy was found to be effective in promoting adhesion because of the chemical reactions between epoxy sizing and DCBA during the curing process.

Fig. 3.3 Apparent IFSS data of DCBA/CF composites subjected to different treatments using pull-out measurements [14]



3.1.3 Epoxy Resins

Epoxy resins are molecules containing more than one epoxide group, as shown below:



Epoxy resins are thermosetting polymers, which can be cured with a wide variety of curing agents via curing reactions. Epoxy resins have a wide range of applications, including fiber-reinforced materials, general-purpose adhesives, high-performance coatings, and encapsulating materials [20–26].

3.1.3.1 Bisphenol-A/F Epoxy Resins

Diglycidyl ether of bisphenol-A (DGEBA) is produced by reacting epichlorohydrin with bisphenol-A in the presence of a basic catalyst. Figure 3.4 depicts the chemical structure of DGEBA.

The properties of the DGEBA resins depend on the value of *n*, which is the number of repeating units commonly known as degree of polymerization. Low-molecular-weight molecules tend to be liquids, while higher molecular weight molecules tend to be either viscous liquids or solids. DGEBA has been used commercially as a raw material and is the primary chemical building block for a broad spectrum of materials [27, 28].

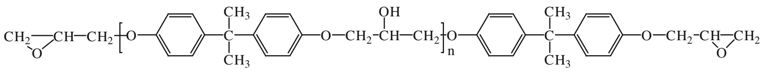


Fig. 3.4 Chemical structure of DGEBA

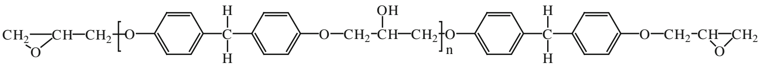


Fig. 3.5 Chemical structure of BFDGE

Diglycidyl ether of bisphenol-F (DGEBF) is synthesized by reacting epichlorohydrin with bisphenol-F in the presence of a basic catalyst. Figure 3.5 shows the chemical structure of DGEBF.

DGEBF epoxy resins have low viscosity and are widely used in solvent-free coatings, concrete reinforcements, adhesives, electrical insulation, and filament winding. The cured DGEBF epoxy resins provide improved resistance performance against solvents compared to that of DGEBA epoxy resins [29].

Diglycidyl ether of tetrabromobisphenol-A is prepared by reacting epichlorohydrin with tetrabromobisphenol-A in the presence of a catalyst. Figure 3.6 shows the chemical structure of the diglycidyl ether of tetrabromobisphenol-A.

Diglycidyl ether of tetrabromobisphenol-A with high *T*g, heat resistance, good operation performance, fire resistance, and low moisture absorbance can be used in glass fiber laminates [30].

Park et al. [31] demonstrated the effect of fiber–polymer interactions on the fracture toughness behavior of CF-reinforced DGEBA epoxy matrix composites. Figure 3.7 shows the evolution of critical stress intensity factor (KIC) of DGEBA/CF composites as a function of the electric current density. The KIC of the composites continually increases with increasing current densities of the treatments up to 0.4 A/m^2, owing to the increased interfacial adhesion between the fibers and matrix.

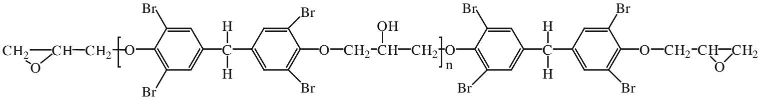


Fig. 3.6 Chemical structure of diglycidyl ether of tetrabromobisphenol-A

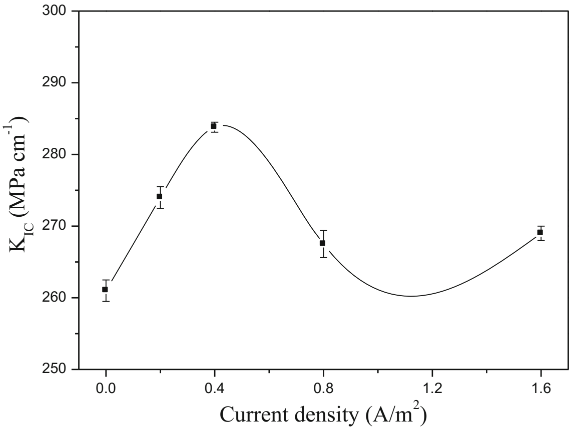


Fig. 3.7 Effect of current density on KIC of DGEBA/CF composites [31]

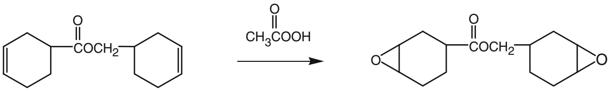


Fig. 3.8 Schematic of synthesis of 3′,4′-epoxycyclohexylmethyl-3,4-epoxycyclohexanecarboxylate

3.1.3.2 Cycloaliphatic Epoxy Resins

A cycloaliphatic epoxy resin, 3′,4′-epoxycyclohexylmethyl-3,4-epoxycyclohexanecarboxylate, is synthesized by the reaction of 3′-cyclohexenylmethyl-3-cyclohexenecarboxylate with peracetic acid, as shown in Fig. 3.8.

The cycloaliphatic epoxy resin has an aliphatic backbone and fully saturated molecular structure, which shows excellent UV stability, good weatherability, and excellent electrical properties. The epoxy resin containing high epoxide content exhibits high cross-link density, *T*g, and heat distortion temperature (HDT), which is used to fabricate structural components subjected to high temperatures. The epoxy resin containing short chains possesses low viscosity, which allows for the rapid fiber wet-out in commonly used processes, including filament winding, pultrusion, and resin transfer molding [32].

3.1.3.3 Trifunctional Epoxy Resins

A trifunctional epoxy resin, trimethylol propane-*N*-triglycidyl ether, is prepared by the reaction of trimethylol propane with epichlorohydrin, as shown in Fig. 3.9.

The epoxy resin has low viscosity, low temperature curing, noncrystallinity, and plasticity [33, 34].

3.1.3.4 Tetrafunctional Epoxy Resins

Tetrafunctional epoxy resins are synthesized through the reaction of 1,3-diaminobenzene or 4,4′-aminodiphenyl methane with epichlorohydrin as shown in Fig. 3.10.

Tetrafunctional epoxy resins with a large number of epoxy functionalities have high cross-linking densities, which are used in applications involving high temperature resistance. The cured epoxy resins show excellent chemical resistance, high modulus, UV-blocking effect, and thermal stability [35, 36].

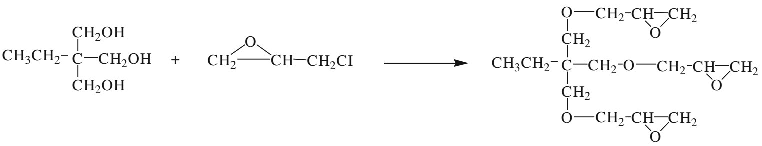


Fig. 3.9 Schematic of synthesis of trimethylol propane-*N*-triglycidyl ether

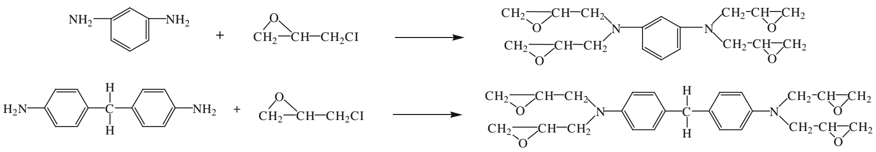


Fig. 3.10 Schematic of synthesis of tetrafunctional epoxy resins

Table 3.1 Tensile properties of resin casts [37]

Resin	Hardener	Tensile strength (MPa)	Elongation (%)	Tensile modulus (GPa)
DGEBA	MeTHPA	60 ± 5	2.1 ± 0.2	2.2 ± 0.2
	DDM	70 ± 5	2.3 ± 0.1	2.2 ± 0.1
	DDM/DETDA	72 ± 6	2.5 ± 0.2	2.0 ± 0.2
TGDDM	MeTHPA	65 ± 6	3.0 ± 0.3	2.4 ± 0.2
	DDM	71 ± 4	2.9 ± 0.3	2.7 ± 0.5
	DDM/DETDA	75 ± 6	3.1 ± 0.4	2.8 ± 0.4

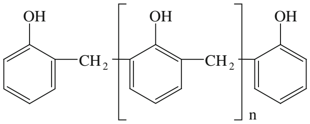
Chen et al. [18] studied the DGEBA/CF and 4,4′-tetradiglycidy diaminodiphenol methane (TGDDM)/CF filament wound composites. Table 3.1 lists the mechanical properties of the composites. It was observed that the two types of resins (DGEBA and TGDDM) containing either 4,4′-diaminodiphenyl methane (DDM) or DDM/diethyltoluene diamine (DETDA) as the hardener showed stronger tensile properties than those containing methyltetrahydrophthalic anhydride (MeTHPA) as the hardener. The TGDDM resin casts showed tensile strengths close to those of the DGEBA resin casts but with higher elongations.

3.1.3.5 Novolac Resins

Novolac resins are synthesized by reaction of a molar excess of phenol with formaldehyde in the presence of an acidic catalyst such as oxalic acid, hydrochloric acid, or sulfonate acid. Figure 3.11 shows the chemical structure of novolac resins.

Novolac resins are solids at room temperature, and soften and flow between 65 and 105 °C. The number average molecular weight (Mn) of a standard phenol novolac resin is between 250 and 900. Novolac resins are soluble in many polar organic solvents such as alcohols and acetone, but not in water [38].

Fig. 3.11 Chemical structure of novolac resins



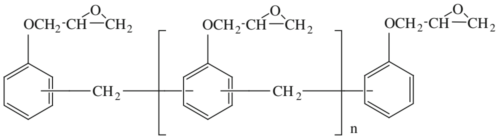


Fig. 3.12 Chemical structure of novolac epoxy resins

3.1.3.6 Novolac Epoxy Resins

Novolac epoxy resins are glycidyl ethers of phenolic novolac resins, which are synthesized by reacting phenolic novolac resin with epichlorohydrin. Figure 3.12 shows the chemical structure of novolac epoxy resins. Because an additional curing agent is required to complete the resin's cure, the industry commonly refers to novolac resins as "two-step" products.

The multiple epoxide groups in the novolac epoxy resins allow them to achieve high cross-linking density, thereby resulting in excellent resistance against temperature, chemicals, and solvents. Novolac epoxy resins are widely used to formulate the molding compounds for microelectronics packaging because of their superior performance at elevated temperatures, excellent moldability and mechanical properties, superior electrical properties, and heat and humidity resistance [39, 40].

3.1.3.7 Epoxy Diluents

The addition of epoxy diluents can reduce the viscosity of epoxy resins. The addition of reactive diluents might change the surface tension, reactivity, solvent resistance, physical strength, linear coefficient of expansion, flexibility/impact resistance, and abrasion resistance.

Epoxy diluents can be divided into nonreactive diluents and reactive diluents. Some examples of nonreactive diluents include benzylalcohol, dibutylphthalate, hydrocarbon resins, and pine oil. Some examples of reactive diluents include *o*-cresyl glycidyl ether (EEW: 180 g/eq, viscosity: 7 mPa s at 25 °C), hexanediol diglycidyl ether (EEW: 142 g/eq, viscosity: 14 mPa s at 25 °C), and C12–C14 alkyl glycidyl ether (EEW: 280 g/eq, viscosity: 7 mPa s at 25 °C) [41, 42]. Figure 3.13 shows the chemical structures of *o*-cresyl glycidyl ether, hexanediol diglycidyl ether, and C12–C14 alkyl glycidyl ether.

3.1.3.8 Epoxy Curing Agents

For the curing of epoxy resins, a wide variety of curing agents such as amines, polyamides, phenolic resins, anhydrides, isocyanates, polymercaptans, and catalytic curing agents have been used. The cure kinetics and *T*g of cured epoxy resins are dependent on the molecular structures of the curing agents. The stoichiometry of the

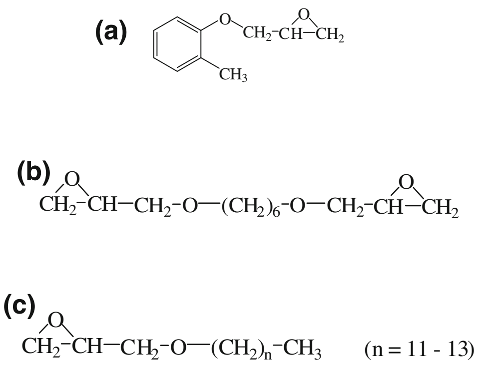


Fig. 3.13 Chemical structures of reactive epoxy diluents. **a** *o*-Cresyl glycidyl ether. **b** Hexanediol diglycidyl ether. **c** C12–C14 alkyl glycidyl ether

epoxy/curing agent system also affects the properties of the cured epoxy resins. Employing different types and amounts of the curing agent determines the final network structure.

An agent which does not participate in the reaction is known as a catalytic curing agent. A reactive curing agent is generally used in much greater amounts compared to a catalytic curing agent. Also, it actually participates in the reaction. The curing agents are reacted with molecules and coupled directly into the cured system as a structural member of the polymer.

Epoxy curing agents can be divided into amine-type curing agents (triethylenetetramine and DDM), alkali curing agents (imidazole and tertiary amine), anhydrides [phthalic anhydride (PA) and nadic methyl anhydride], and catalytic curing agents [N-benzylpyrazinium hexafluoroantimonate (BPH) and N-benzylquinoxalinium hexafluoroantimonate (BQH)] [43–46]. The chemical structures of DDM, PA, BPH, and BQH are shown in Fig. 3.14.

3.1.3.9 Curing Process

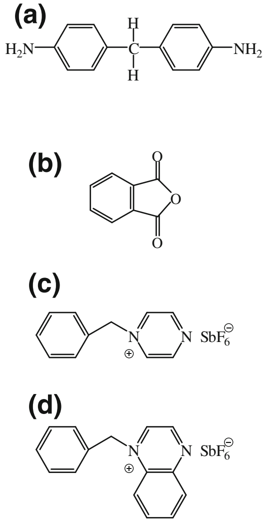
Epoxy curing process is a chemical reaction in which the epoxide groups in the epoxy resins react with a curing agent to form a highly cross-linked, three-dimensional network [47, 48].

1. Room temperature cure

   Epoxy resin cures with room temperature curing agent (such as aliphatic polyamine) at room temperature. A room temperature cure will provide lower *T*g, higher flexibility, impact resistance, and electrical and thermal shock resistance.
2. Heat cure

   Generally, epoxy resins are cured with the curing agent at elevated temperatures, and the resulting cured epoxy resins yield higher glass transition temperature, tensile strength, heat resistance, and chemical resistance.

Fig. 3.14 Chemical structures of **a** DDM, **b** PA, **c** BPH, and **d** BQH



3. Photocure

   Epoxy resins are cured using photoinitiators via an addition cure reaction in the presence of an infrared, ultraviolet light, or electron beam.

3.1.3.10 Curing Mechanism

1. Amine-based curing agents

   Primary and secondary amines react through nucleophilic addition to the epoxide ring, as shown in Fig. 3.15. The reaction is exothermic. Proton donors such as tertiary amines are generally used as catalysts to accelerate the curing process with amines. The use of an excessive amount of catalyst achieves faster curing, but usually at the expense of the active lifespan and thermal stability [49].
2. Anhydride-based curing agents

   Figure 3.16 shows the cure reaction mechanism of the epoxide with anhydrides. First, the anhydride reacts with the pendant hydroxyl groups in epoxy resins to produce ester acids, and then, the carboxyl groups react with the epoxide groups

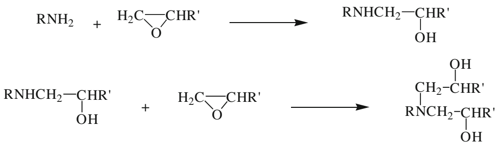


Fig. 3.15 Cure reaction mechanism of epoxide with amines

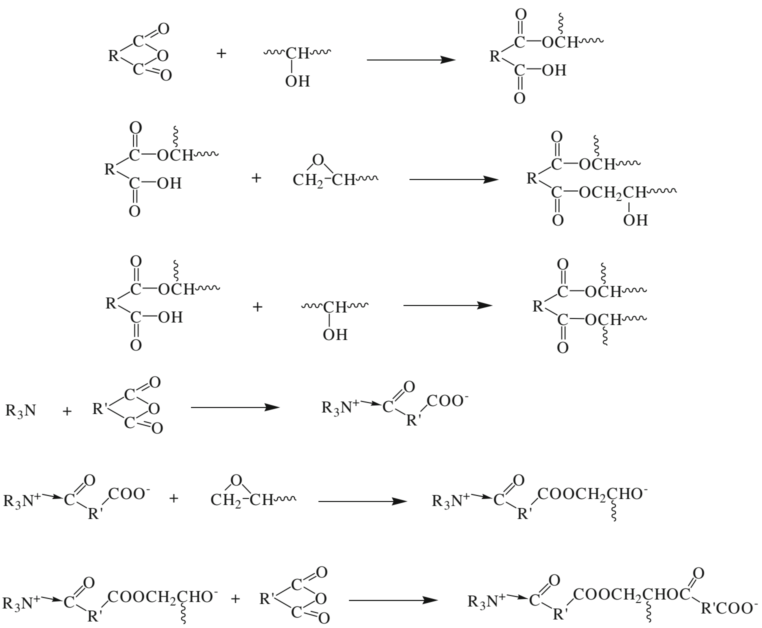


Fig. 3.16 Cure reaction mechanism of epoxide with anhydrides

to yield ester groups. The reaction with the anhydrides is usually catalyzed by tertiary amines and initiated by the activation of the anhydride with amine. Thus, the cure reaction is initiated at a relatively low temperature in the presence of tertiary amines [44, 50].

3. Catalytic curing agents

Figure 3.17 shows the suggested mechanism of the polymerization of BPH-initiated epoxides. The BPH thermally decomposed to generate a benzyl cationic initiating species. In the initial propagation step (Fig. 3.17a), the epoxide group attacked a propagating cationic species. The reaction of the OH group with BPH generated a powerful protic acid (H^+SbF6^-) (Fig. 3.17b). The propagation then continued through a subsequent attack on the epoxide groups, an alcohol attack, and proton transfer reactions (Fig. 3.17c). Also, the protic acid subsequently can attack the epoxide groups in the propagation step. Once the monomer concentration decreased, a pyrazine released from the salts can possibly attack, perhaps predominantly, the propagation species, thereby terminating the polymerization, as shown in Fig. 3.17d [51–54].

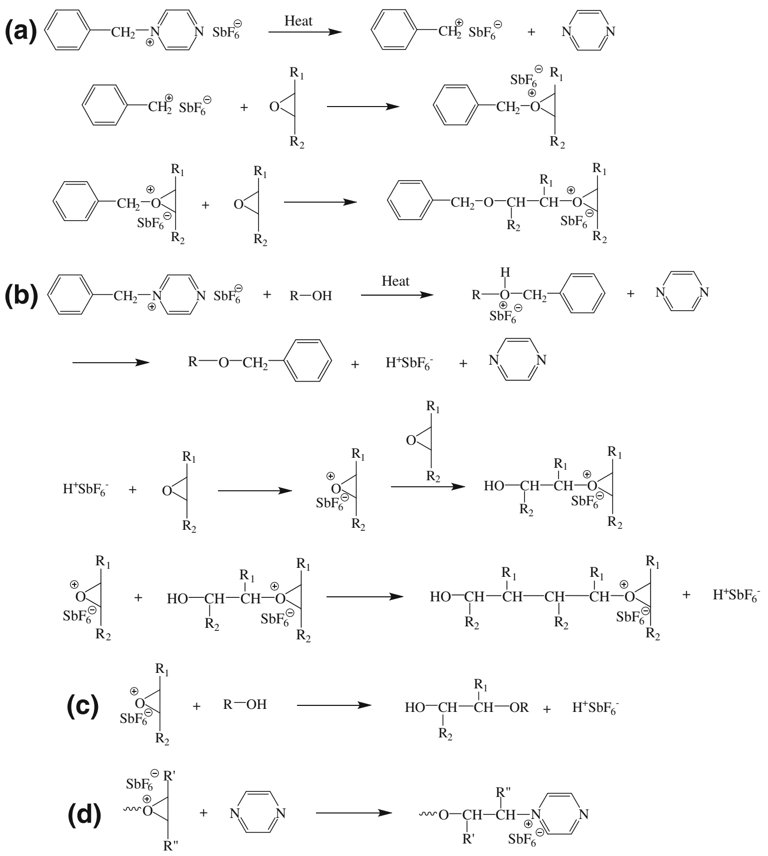


Fig. 3.17 Polymerization mechanism of BPH-initiated epoxides

3.1.4 Phenolic Resins

Phenolic resins are normally prepared from phenol and formaldehyde. On the basis of the preparation methods, phenolic resins can be divided into resol and novolac resins [55–60].

3.1.4.1 Resol Resins

Resol resins are synthesized by the reaction of a molar excess of formaldehyde with phenol in the presence of a basic catalyst. Figure 3.18 shows the chemical structure of resol resins.

When an excess of formaldehyde is used in the synthesis of resol resins, a sufficient number of methylol and dibenzyl ether groups remain reactive to

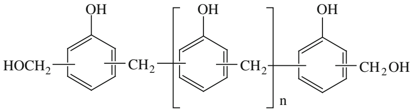


Fig. 3.18 Chemical structure of resol resins

complete the polymerization and cure the resin without the incorporation of a cure agent such as hexamethylenetetramine. For this reason, the industry commonly refers to the resol resins as "single-stage"-type products. The typical Mn of a straight phenol resol resin is between 200 and 450.

3.1.4.2 Novolac Resins

Novolac resins are synthesized by the reaction of a molar excess of phenol with formaldehyde in the presence of an acidic catalyst. Figure 3.19 shows the chemical structure of novolac resins.

The reaction creates a methylene bridge at either the ortho or para position of the phenolic aromatic rings. The final phenolic resins are unable to react further without the addition of a curing agent. Because an additional agent is required to complete the curing of the resin, the industry commonly refers to novolac resins as "two-stage" products. The most common curing agent for novolac resins is hexamethylenetetramine.

Park et al. [56] demonstrated the interfacial characteristics and fracture toughness of the electrolytically Ni-plated CF-reinforced phenolic resin composites. Figure 3.20 shows the results of the KIC tests of the composites as a function of the current density. The KIC increased with the increasing current density up to 10 A/m^2, owing to the presence of nickelized functional groups and increased number of oxide functional groups on the CF.

He et al. [61] studied the effects of novolac resin modification on the mechanical properties of the epoxy/CF composites. Figure 3.21 shows the Charpy impact strength of the unmodified and modified composites on varying the content of the novolac resin. It was observed that on increasing the content of the novolac resin from 0 to 18 wt%, the impact strength first gradually increased and then decreased. The impact strength reached the maximum at 13 wt% of the novolac resin. The increased impact properties in epoxy/novolac resin/CF composites might be owing to the novolac resin transition layer, which was bonded to the CF surface and matrix.

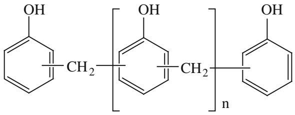


Fig. 3.19 Chemical structure of novolac resins

Fig. 3.20 Evolution of KIC of phenolic resin/CF composites with current density [56]

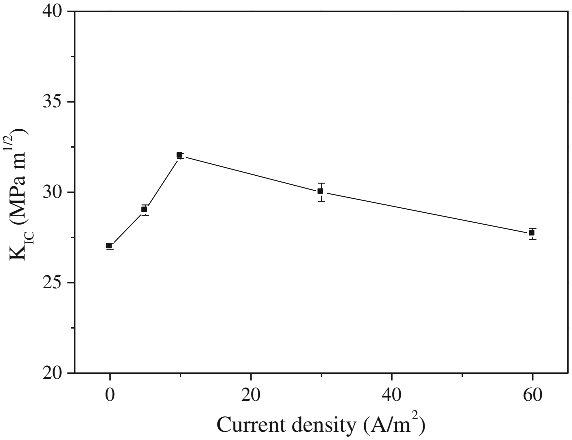
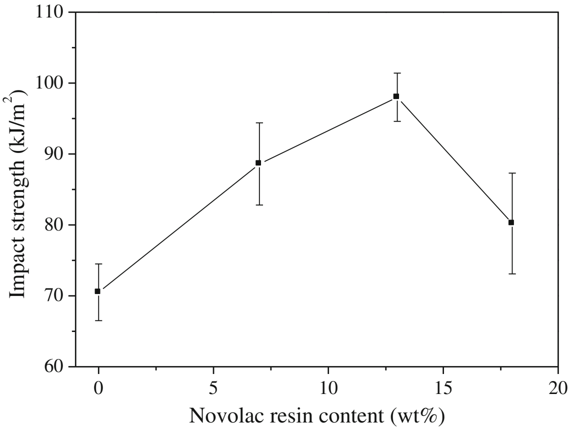


Fig. 3.21 Charpy impact strength of unmodified and modified composites with varying content of novolac resin [61]

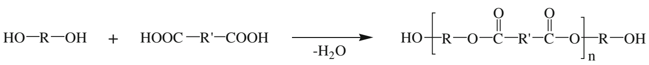


3.1.5 Polyester Resins

Polyester resins are unsaturated resins formed by the reaction of dibasic organic acids and polyhydric alcohols, as shown in Fig. 3.22.

Polyester resins have relatively good ultraviolet resistance, are long lasting, and show strong resistance to water. These resins are the most commonly used matrix in the marine and composite industries. The resins are used in sheet molding and bulk molding compounds and toners of laser printers. They are commonly used for auto repairs, casting, adhesives, and wood filling. These resins can be used with any type of fiberglass: CF or kevlar [61–65].

Polyester resins are classified as thermosetting resins and will cure exothermically to a solid when the curing agent is added. Generally, organic peroxides such as benzoyl peroxide and methyl ethyl ketone peroxide are used as the curing agents.



$$\mathrm{HO{-}R{-}OH} + \mathrm{HOOC{-}R'{-}COOH} \xrightarrow[-\mathrm{H_2O}]{} \mathrm{HO}\left[\mathrm{R{-}O{-}}\overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}}\mathrm{{-}R'{-}}\overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}}\mathrm{{-}O}\right]_n\mathrm{R{-}OH}$$

Fig. 3.22 Schematic of synthesis of polyester resins

Polyester resins are the most widely used resins in the composites industry. The resins are less expensive and more forgiving than the epoxy resins and offer corrosion resistance. The majority of all fiberglass parts are constructed using polyester resins because they are easy to use, fast curing, and resistance of the extremes of temperature and catalysts.

Some examples of commercially important linear polyesters are polyglycolic acid (PGA), polylactic acid (PLA), polycaprolactone (PCL), polyethylene terephthalate (PET), and polybutylene terephthalate (PBT).

PGA is prepared through the ring-opening polymerization of glycolide, as shown in Fig. 3.23.

PLA is synthesized through the ring-opening polymerization of lactide, as shown in Fig. 3.24.

PCL is prepared through the ring-opening polymerization of ε-caprolactone using stannous octoate as a catalyst, as shown in Fig. 3.25.

PET is produced through the polycondensation of ethylene glycol and either dimethyl terephthalate or terephthalic acid. An excess ethylene glycol is reacted with the former in the melt in the presence of a basic catalyst, as shown in Fig. 3.26.

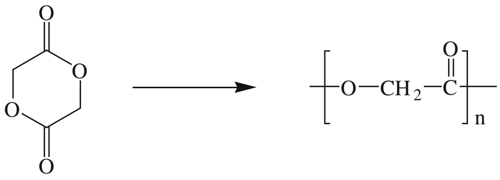


Fig. 3.23 Schematic of synthesis of PGA

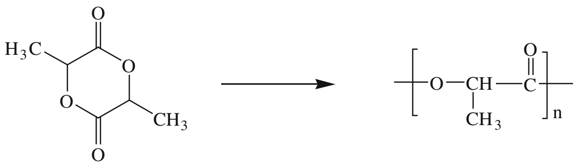


Fig. 3.24 Schematic of synthesis of PLA

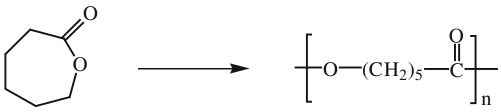


Fig. 3.25 Schematic of synthesis of PCL

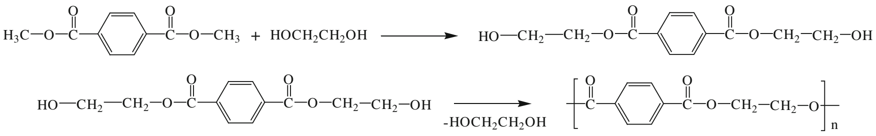


Fig. 3.26 Schematic of synthesis of PET by dimethyl terephthalate process

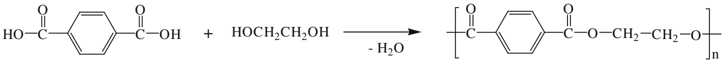
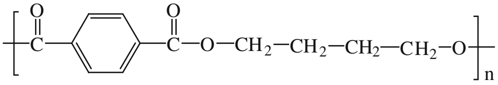


Fig. 3.27 Schematic of synthesis of PET using terephthalic acid

Fig. 3.28 Chemical structure of PBT



In the process involving terephthalic acid, the esterification of ethylene glycol and terephthalic acid is directly conducted at moderate pressure and high temperature, as shown in Fig. 3.27.

PBT is prepared through the polycondensation of terephthalic acid with 1,4-butanediol. Figure 3.28 shows the chemical structure of PBT.

Vilčáková et al. [66] demonstrated the electrical conductivities of polyester resin/CF composites in the percolation threshold region. Table 3.2 shows the average values of conductivity of the composites at laboratory temperature. A steep conductivity increase at a relatively low CF content (0.7 vol.%) appeared, which was due to a special mechanism involved in the formation of the intrinsic fibrous structure of the composites.

3.1.6 Polyimide Resins

Polyimide (PI) is a polymer of imide monomers. Figure 3.29 shows the structure of PI [67–69].

On the basis of the composition of the main chain, the PIs can be divided into aliphatic PI, wherein the atoms of the imide group are part of a linear chain, and aromatic PI, wherein the imide group is part of a cyclic unit in the polymer chain, as shown in Fig. 3.30.

PI possesses a greater resistance to heat compared to any other unfilled organic material. Unlike most plastics, the PIs are available in laminates, molded parts, and stock shapes. PI parts are fabricated using techniques ranging from powder metallurgy to conventional injection, transfer, and compression molding, and extrusion.

Table 3.2 Conductivity of polyester/CF composites [66]

CF content (vol.%)	Conductivity (S/cm)
0	$(4.09 \pm 0.00) \times 10^{-12}$
0.70	$(1.38 \pm 0.34) \times 10^{-11}$
0.77	$(1.41 \pm 1.51) \times 10^{-7}$
0.84	$(1.26 \pm 1.43) \times 10^{-6}$
0.91	$(2.38 \pm 1.15) \times 10^{-6}$
0.98	$(4.43 \pm 1.67) \times 10^{-6}$
1.05	$(3.70 \pm 1.03) \times 10^{-6}$
1.75	$(3.73 \pm 0.88) \times 10^{-6}$
2.10	$(1.11 \pm 0.24) \times 10^{-5}$
2.80	$(4.19 \pm 0.47) \times 10^{-5}$
3.85	$(2.42 \pm 0.05) \times 10^{-4}$
4.20	$(1.24 \pm 0.03) \times 10^{-4}$
4.55	$(1.71 \pm 0.02) \times 10^{-3}$
4.90	$(8.75 \pm 0.02) \times 10^{-3}$
5.25	$(2.64 \pm 0.03) \times 10^{-2}$
5.60	$(8.58 \pm 0.02) \times 10^{-2}$
6.00	$(1.32 \pm 0.00) \times 10^{-1}$
6.30	$(2.13 \pm 0.00) \times 10^{-1}$

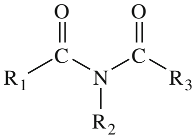


Fig. 3.29 Chemical structure of PI

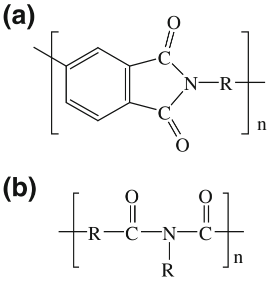


Fig. 3.30 Chemical structures of aromatic and linear PIs. **a** Aromatic PIs. **b** Linear PIs

PIs possess low dielectric constant, flexibility, polishability to angstrom-level surface finishes, excellent dimensional stability, low water absorption, high temperature stability, excellent machinability, low outgassing and noncontaminating quality, exceptional mechanical strength, and low coefficient of thermal expansion. Consequently, PIs are used in place of metals and glass in many high-performance applications in the electronics, automotive, and even the aerospace industries.

PIs are solid, heat-resistant, incombustible substances having predominantly amorphous structures, with Mn of 50,000–150,000 and density of 1.35–1.48 g/cm^3 at 20 °C. Most PIs do not dissolve in organic solvents, are inert to the action of oils, and remain virtually unchanged under the action of dilute acids. However, they are hydrolyzed by alkalis and superheated steam. PIs are resistant to ozone, γ-rays, and fast electrons and neutrons, and resistant to heat.

Aromatic PIs with imide rings in the main chain have acquired practical significance because of their valuable physicochemical properties, which remain unchanged for long periods over a wide range of temperature (ranging from −270 to 300 °C). The most industrially valuable PI is polypyromellitimide, prepared through the –diamine route, as shown in Fig. 3.31. The PI melts above 600 °C and exhibits thermal stability up to 500 °C in an inert atmosphere [70–73].

Li et al. [74] studied the friction and wear properties of surface-treated CF-reinforced PI composites under oil-lubricated conditions. As shown in Fig. 3.32, the friction coefficients of air-oxidized and rare-earth-solution-treated CF-reinforced PI composites are lower compared to those of untreated CF-reinforced PI composites, thereby indicating that the surface modification can improve the interfacial adhesion and compatibility between the CF and PI matrix.

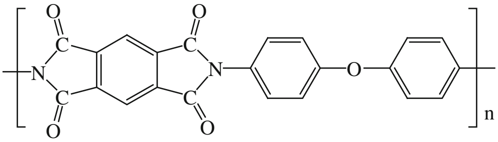


Fig. 3.31 Chemical structure of polypyromellitimide

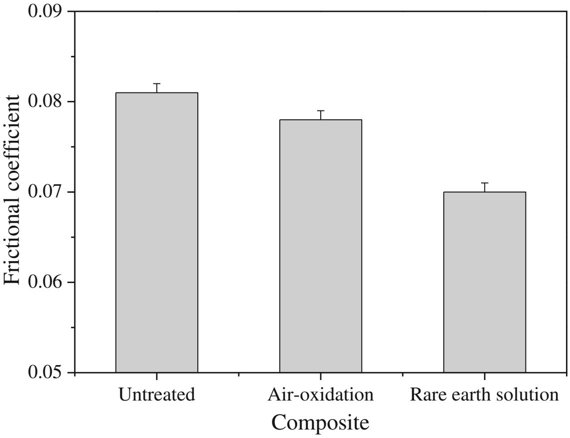


Fig. 3.32 Effect of surface treatment on friction of PI/CF composites [74]

3.1.7 Vinyl Ester Resins

Vinyl ester (VE) is a resin produced by the esterification of an epoxy resin with an unsaturated monocarboxylic acid, thereby combining the advantages of epoxy and polyester resins. The resins possess higher flexibility, analogous to the epoxy resins, and ease of processing, analogous to the polyesters [75].

VE resins are stronger than the polyester resins and cheaper than the epoxy resins. Vinyl ester has lower viscosity compared to the polyester and epoxy resins. VE resins offer better resistance to moisture absorption compared to the polyester resins. It is also known that vinyl ester resins bond well with fiberglass, but weakly so with kevlar and CF owing to the nature of their exotic fibers [76].

Bisphenol-A epoxy-VE resin, as shown in Fig. 3.33, is based on bisphenol-A epoxy resin and provides resistance to a wide range of acids, alkalis, bleaches, and organic compounds for use in several chemical processing industry applications.

VE resins are exceptionally reactive and cure rapidly. Peroxides such as methyl ethyl ketone peroxide (MEKP), dicumyl peroxide, di(t-butylperoxy) cyclohexane, and t-butyl peroxybenzoate have been used as initiators for curing VE systems [77].

Cured VE is more flexible and possesses greater toughness compared to the polyester. VE can also withstand temperatures of up to approximately 200 °C without distortion.

Yamada et al. [78] demonstrated the plasma-graft polymerization of a monomer with double bonds onto the surface of CF and its adhesion to a VE resin. Figure 3.34 shows the pull-out force of yarns, which were grafted using poly(adipic acid divinyl ester) (AADE) chains and later embedded in the cured VE resin/

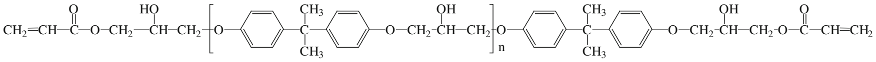
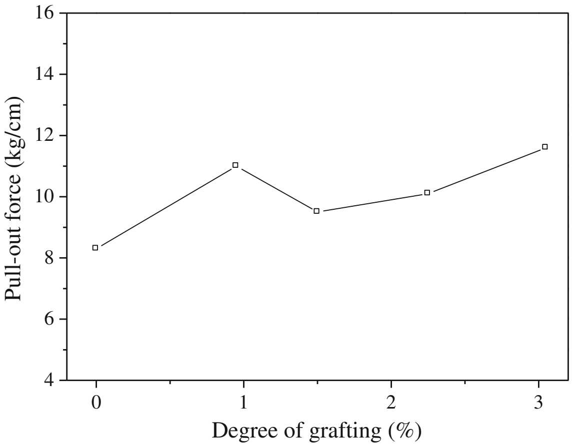


Fig. 3.33 Chemical structure of bisphenol-A epoxy vinyl ester

Fig. 3.34 Pull-out force of grafted carbon yarn as a function of degree of grafting in AADE [78]



benzoyl peroxide/N,N-dimethylaniline mixture, as a function of the degree of grafting. The pull-out force increased with the increasing degree of grafting. The degree of grafting was proportional to the number of graft chains formed on the surface of the yarn because the average molecular weight of the graft chains did not depend on the degree of grafting.

3.2 Thermoplastic Resins

3.2.1 Introduction

Thermoplastic resins consist of long polymer molecules, which are generally not cross-linked. The resin can be repeatedly melted and reused. Usually, no chemical change occurs when thermoplastic is cured.

Thermoplastic resins are often supplied as granules and heated to permit fabrication using conventional molding methods such as injection molding, rotational molding, extrusion, vacuum forming, and compression molding.

Thermoplastic resins have high impact strength, recyclability, and zero emissions. They can bond to other thermoplastics and be molded or shaped with reheat.

Among the thermoplastic resins, acrylonitrile butadiene styrene (ABS), polyamide, polycarbonate (PC), polyetheretherketone (PEEK), polyetherimide (PEI), polyethersulfone, polyethylene (PE), polyphenylene sulfide, and polypropylene (PP) resins can be used as polymer matrices for CF-reinforced polymer composites [79–81].

3.2.2 Acrylonitrile Butadiene Styrene Resins

ABS is a terpolymer of acrylonitrile, butadiene, and styrene. Figure 3.35 shows the chemical structure of ABS. Many industrial methods such as bulk, bulk-suspension, emulsion, and emulsion graft-doped legitimate are used to prepare the ABS resins.

The typical compositions contain 50 % styrene and the remaining 50 % consisting of butadiene and acrylonitrile. The higher the content of acrylonitrile, the better the heat resistance, rigidity and solvent resistance; however, the worse the flow character. Its heat resistance and solvent resistance are better compared to those of the high-impact polystyrene (HIPS). The impact strength, tensile strength, and surface hardness of ABS are better compared to those HIPS. ABS resin is

Fig. 3.35 Chemical structure of ABS

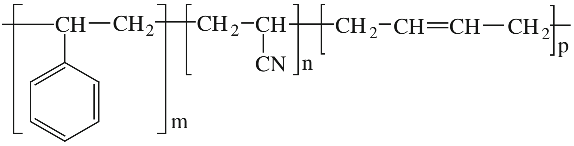
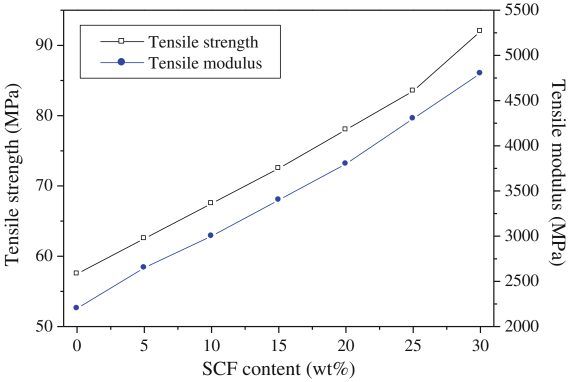


Fig. 3.36 Tensile properties of ABS/SCF composites [83]



tough, light, economic, heat- and stain-resistant, and an excellent engineering plastic [82, 83].

ABS materials can be processed using any of the standard thermoplastic processing methods. ABS resins have a relative density of 1.03–1.07, Izod impact strength of 186.2–215.8 J/m, tensile strength of 34.3–49 MPa, and Rockwell hardness of 62–118 [84].

ABS resins are widely used in fields such as automotive and electrical/electronic applications, housing, business equipment, and computer components.

Li et al. [83] studied the CF surface treatment and addition of PA6 on the tensile properties of ABS composites. Figure 3.36 shows the effects of the shot carbon fiber (SCF) content on the tensile properties of ABS/SCF composites. Increasing the SCF content increased the tensile strength and modulus of the composites, in agreement with the well-known equation of Kelly and Tyson for mixing short fiber-reinforced composite materials.

3.2.3 Polyamide Resins

Polyamide (PA) is a polymer containing amide monomers joined by peptide bonds. PAs possess the CONH functional groups in the polymer backbone. They can be produced either by the interaction between an amine (NH_2) group and a carboxyl (COOH) group, or the polymerization of amino acids or amino-acid derivatives. PAs can occur both naturally and artificially. They are commonly used in textiles, automotives, carpets, and sportswear owing to their extreme durability and strength [85–87].

On the basis of the composition of the main chain, PAs are classified as aliphatic PAs, polyphthalamides, and aromatic PAs. On the basis of the number and types of repeating units, polyamides can be divided into homopolymers (PA 66, made from hexamethylenediamine and adipic acid) and copolymers (PA 6/66, made from caprolactam, hexamethylenediamine, and adipic acid). On the basis of their crystallinity, polyamides can be divided into semicrystalline and amorphous.

Poly(hexamethylenediamine adipamide) (Nylon 66, PA66) is one of the most widely used semicrystalline engineering thermoplastic resins. Nylon 66 is synthesized by reacting adipic acid with hexamethylene diamine, as shown in Fig. 3.37 [88, 89].

Nylon 66 is a semicrystalline polyamide, commonly used in fiber applications such as carpeting, clothing, and tire cords. It is also used as an engineering material in bearings and gears owing to its good abrasion resistance and self-lubricating properties [90]. Nylon 66 has good sliding properties, abrasion resistance, high tensile strength and melting point, and electrical insulation. It is widely used as gear wheels, friction strips, piston guides, impact plates, cam disks, etc. [91].

Nylon 66 has a melting point of 265 °C, which is high for a synthetic fiber, though not comparable to either polyesters or aramids such as Kevlar. Its long molecular chain results in more sites for hydrogen bonds, thereby creating chemical "springs," and making it very resilient.

The molten Nylon 66 undergoes a spinning process, wherein Nylon 66 is extruded and sent through a spinneret, which is a small metal plate with fine holes. Nylon 66 is then air-cooled to form a wide range of fiber types.

Botelho et al. [86] studied the mechanical behavior of CF-reinforced PA composites. Table 3.3 shows the tensile and compression properties of Nylon 66/CF composites. An increase in the fiber volume fraction improved the mechanical properties of the composites.

3.2.4 Polycarbonate Resins

PC is produced by the reaction of bisphenol-A with phosgene $COCl_2$. The overall reaction is shown in Fig. 3.38.

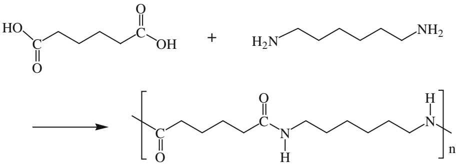


Fig. 3.37 Schematic of synthesis of Nylon 66

Table 3.3 Tensile and compression properties of Nylon 66/CF composites [86]

Properties	CF content (vol.%)		
	40	50	60
Tensile strength (MPa)	408 ± 24	443 ± 31	512 ± 44.2
Tensile modulus (GPa)	35.4 ± 2.8	39 ± 1.6	51.1 ± 2.3
Compressive strength (MPa)	290 ± 29	352 ± 46	391 ± 45
Shear compression (MPa)	22.0 ± 3.0	25.5 ± 4.9	27.8 ± 2.3
Density (g/cm^3)	1.31	1.36	1.39

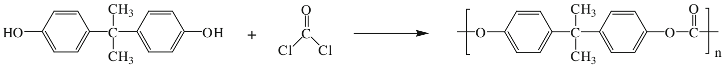


Fig. 3.38 Schematic of synthesis of PC

PC resins are a particular group of thermoplastic polymers. PC is a novel, highly flexible, durable and nonbreakable, and cost-effective means of domestic and industrial usage. PC possesses high impact strength and exceptional clarity and is widely used in applications such as bulletproof windows, break-resistant lenses, and compact discs [92, 93].

PCs are used mainly as molding compounds. The commercially important PCs use bisphenol-A and diphenyl carbonate. These polymers are clear plastics with a slight yellow coloration. They have excellent electrical properties and high impact strength.

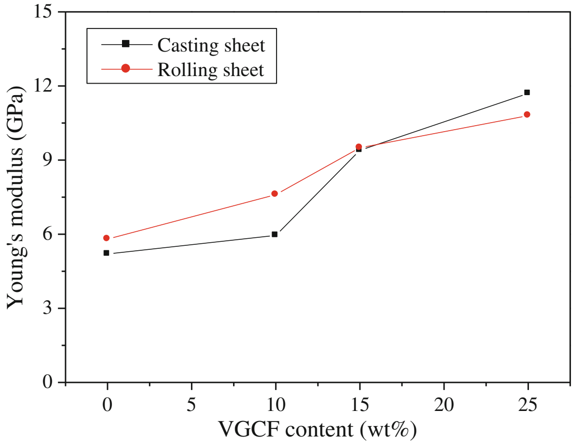
PCs are easily worked, molded, and thermoformed. The plastics are widely used in the modern chemical industry. A balance of useful features, including temperature resistance, impact resistance, and optical properties, position the PCs between commodity plastics and engineering plastics [94].

Choi et al. [95] demonstrated the production and characterization of PC composite sheets reinforced with vapor grown carbon fiber (VGCF). Figure 3.39 shows the Young's modulus of PC/VGCF cast and rolling composites reinforced with various amounts of VGCF. The Young's modulus of both the composites increased with the increasing VGCF content.

3.2.5 *Polyetheretherketone Resins*

PEEK resins are synthesized using step-growth polymerization by the dialkylation of bisphenolate salts. A typical reaction involving 4,4′-difluorobenzophenone and

Fig. 3.39 Young's modulus of PC/VGCF cast and rolling composites reinforced with various amounts of VGCF [95]



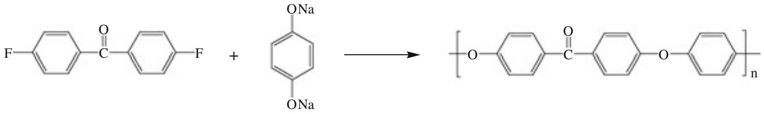


Fig. 3.40 Schematic of synthesis of PEEK

disodium salt of hydroquinone, which is generated in situ by deprotonation with sodium carbonate, is shown in Fig. 3.40 [96].

PEEK is a semicrystalline thermoplastic with excellent mechanical and chemical resistance properties, which are retained at high temperatures. The Young's modulus is 3.6 GPa, while its tensile strength is over the range 90–100 MPa. PEEK has a *T*g of approximately 143 °C and melts at approximately 343 °C. PEEK carries a V-0 flammability rating and exhibits low smoke and toxic gas emissions when exposed to flame. PEEK has excellent chemical resistance, low moisture absorption, inherently good wear, and abrasion resistance and remains unaffected by continuous exposure to either hot water or steam [97].

PEEK is generally used as an engineering material because it has high strength, low wear, and good resistance to corrosion and operates effectively in an ultrahigh vacuum environment [98, 99].

Gebhard et al. [100] studied the wear of aqueous lubricated short carbon fiber (SCF)-reinforced PEEK composites. Figure 3.41 shows the fiber corrosion depths of the composites under different experimental and environmental conditions. The fiber corrosion depth of TGC (after a sliding experiment with simultaneous galvanic corrosion) showed the highest value in this study. This means that there is not only an increase in the wear rate owing to the occurrence of fiber corrosion, but also a supplementary effect of the tribological stress-enhanced CF corrosion.

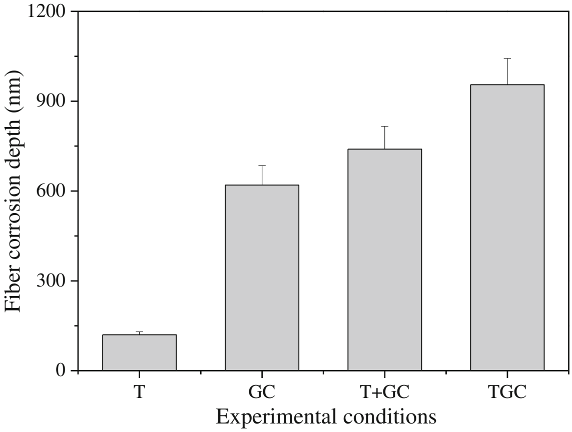


Fig. 3.41 Fiber corrosion depths of PEEK versus stainless steel pairing under different experimental and environmental conditions [100]. *T* pure sliding, *GC* galvanic corrosion, *T* + *GC* mathematical addition, *TGC* sliding experiment with simultaneous galvanic corrosion

3.2.6 Polyetherimide Resins

PEI is an amorphous engineering thermoplastic resin. Figure 3.42 shows the chemical structure of PEI. PEI has high heat resistance, excellent electrical properties, high strength and modulus, and excellent processibility. *T*g and amorphous density at 25 are 216 °C and 1.27 g/cc, respectively. Unmodified PEI resin is transparent and has inherent flame resistance and low smoke evolution [101].

PEI can be processed on conventional thermoplastic molding equipment. PEI resin is available for general-purpose injection molding, blow molding, foam molding, and extrusion. PEI is extruded to produce profiles, coated wires, sheets, and films. The glass fiber reinforcement of the PEI provides it with both greater tensile strength and rigidity while simultaneously improving the dimensional stability [102].

PEI resists a broad range of chemicals under varied conditions of stress and temperatures. PEI is compatible with aliphatic hydrocarbons and alcohols such as gasoline and gasohol, mineral-salt solutions, dilute bases, and fully halogenated hydrocarbons [103].

PEI is often used instead of polysulfone and commonly specified for aerospace, automotive, medical, and packaging applications. PEI is resistant to UV and gamma radiation. This material can be easily machined and provides cost savings over ceramics in electrical applications [104].

Xian et al. [105] demonstrated the influence of SCF reinforcement on the sliding wear of PEI composites. Figure 3.43 shows the evolution of the specific wear rates

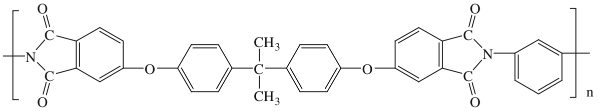
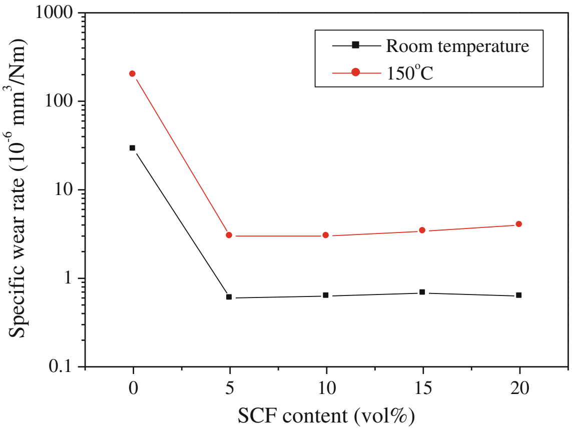


Fig. 3.42 Chemical structure of PEI

Fig. 3.43 Specific wear rate of neat PEI and its SCF composites at room temperature and 150 °C, measured with a pin-on-disk testing rig, 1 m/s, and 2 MPa [105]



with the SCF fraction. It was found that the addition of SCF reduced the specific wear rate of PEI by approximately 60 times at room temperature, and 80 times at 150 °C, respectively, owing to several functions of SCF, i.e., reinforcing, lubricating, and thermal conducting functions.

3.2.7 Polyethersulfone Resins

Polyethersulfone (PES) is an excellent heat-resistant, transparent, noncrystalline engineering plastic containing ether and sulfone groups in their backbone chains. Figure 3.44 shows the chemical structure of PES.

PES resins exhibit high-temperature performance along with a high *T*g, good dimensional stability, outstanding rigidity even at high temperatures, excellent insulation properties, biocompatibility, and inherent flame retardation [106].

PES can be molded using conventional plastics processing equipment such as injection molding, extrusion, compression molding, solution casting, and sintering. Because of its amorphous nature, PES has excellent dimensional stability and can be easily processed with highly polar solvents, which are suitable for applications requiring similar tolerances and little dimensional change over a wide temperature range. PES composites offer distinct advantages over thermosets, namely shorter processing times, better toughness, reduced storage and scrap, and reparability [107].

Wu et al. [108] studied the processing and properties of solution impregnated CF-reinforced PES composites. Table 3.4 lists the flexural test results of the composites using different molding cycles. A higher molding temperature and longer molding time increased the flexural strength from 44 to 71 MPa.

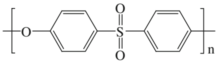


Fig. 3.44 Chemical structure of PES

Table 3.4 Effects of molding cycle on transverse flexural properties of solution-processed CF-reinforced PES composites [104]

Sample	Molding temperature (°C)	Molding time t_1 (min)	Molding time t_2 (min)	Transverse flexural strength (MPa)	Transverse flexural modulus (GPa)
1	350	15	10	44	9
2	385	50	10	71	9

3.2.8 Polyethylene Resins

PE is a thermoplastic polymer consisting of long chains of the ethylene monomer, as shown in Fig. 3.45. PE is a light, versatile synthetic resin made by the polymerization of ethylene. PE can be produced by polymerization techniques, including radical, anionic addition, cationic addition, and ion coordination. Its simple basic structure can be either linear or branched to a greater or lesser degree.

PE is a member of the important family of polyolefin resins. It is the most widely used plastic worldwide, products ranging from clear food wraps and shopping bags to detergent bottles and automobile fuel tanks. It can also be slit or spun into either synthetic fibers or modified to take on the elastic properties of rubber [108–110].

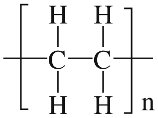
PE is available over a range of flexibilities and other properties depending on the production process. PE can be formed using a wide variety of thermoplastic processing methods and is particularly useful when moisture resistance and low costs are necessary.

The mechanical properties of PE depend significantly on variables such as the extent and type of branching, crystal structure, and molecular weight. PE is classified into varieties such as low-density PE (LDPE), linear low-density PE (LLDPE), and high-density PE (HDPE). LDPE, LLDPE, and HDPE typically have a density value ranging from 0.91 to 0.925 g/cm^3, 0.918 to 0.94 g/cm^3, and 0.935 to 0.96 g/cm^3, respectively [111, 112].

LDPE is prepared from gaseous ethylene under high pressures and temperatures in the presence of oxide initiators. LDPE has a high degree of short and long chain branching, and weak intermolecular forces as the attraction between the instantaneous-dipole and induced-dipole is less. This results in low tensile strength and increased ductility. The high degree of branches with long chains endows the molten LDPE with unique and desirable flow properties. LDPE is a flexible material with a melting point of approximately 110 °C. LDPE is used for both rigid containers and plastic film applications [113].

LLDPE is made through the copolymerization of ethylene with 1-butene and small amounts of 1-hexene and 1-octene, using either Ziegler–Natta or metallocene catalysts. LLDPE is a substantially linear polymer, with significant numbers of short branches. LLDPE exhibits higher tensile strength, impact, and puncture resistance compared to LDPE. LLDPE is used predominantly in the packaging film owing to its toughness, flexibility, and relative transparency [114].

HDPE can be produced at low temperatures and pressures, using Ziegler–Natta and metallocene catalysts or activated chromium oxide (Phillips catalyst). HDPE has a low degree of branching and consequently, stronger intermolecular forces and



$$\left[\mathrm{CH_2-CH_2} \right]_n$$

Fig. 3.45 Chemical structure of PE

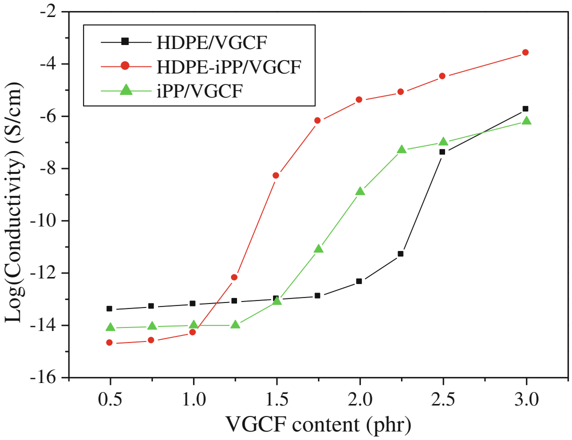


Fig. 3.46 Dependence of electrical conductivity on VGCF content for VGCF-filled HDPE, iPP, and HDPE/iPP (50/50) blends molded at 190 °C for 15 min [116]

tensile strength. HDPE is used in products and packaging such as milk jugs, detergent bottles, margarine tubs, garbage containers, and water pipes [115].

Zhang et al. [116] demonstrated the selective location and double percolation of CF-filled HDPE/isotactic polypropylene (iPP) blends. Figure 3.46 shows the dependence of the electrical conductivity on the VGCF content for HDPE, iPP, and HDPE/iPP blends. The HDPE samples exhibited a percolation threshold at 2.5 phr (parts per hundred parts resin) VGCF content, whereas for the iPP samples, the percolation threshold was 1.75 phr VGCF content. This was owing to the different filler dispersion states in the two polymers.

3.2.9 Polyphenylene Sulfide Resins

Polyphenylene sulfide (PPS) is formed by the reaction of sodium sulfide with *p*-dichlorobenzene, as shown in Fig. 3.47. PPS is an organic polymer having a symmetrical, rigid backbone chain consisting of recurring *p*-substituted benzene rings and sulfur atoms. PPS is a semicrystalline, high-performance engineering thermoplastic.

PPS exhibits outstanding chemical resistance, thermal stability, dimensional stability, and fire resistance. The extreme inertness of PPS toward organic solvents, and inorganic salts and bases makes for an outstanding performance as a corrosion-resistant coating, suitable for contact with foods [117].

PPS can be molded, extruded, or machined to high tolerances. In its pure solid form, it may be opaque white to light tan in color. PPS resins are available as

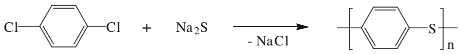


Fig. 3.47 Schematic of synthesis of PPS

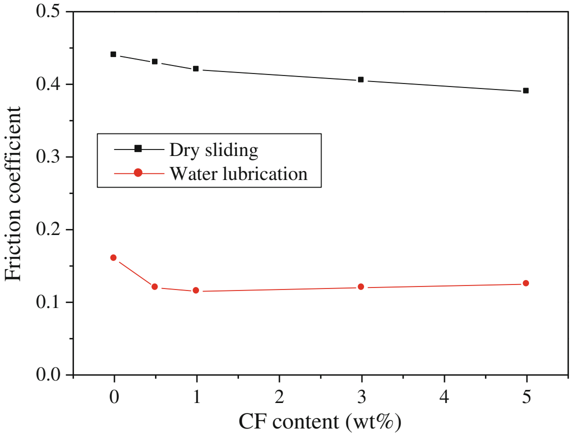


Fig. 3.48 Variations in friction coefficient of PPS/CF composite coating as a function of CF content [118]

powders for slurry coating and electrostatic spraying. Because of its low viscosity, PPS can be molded with a high loading of fillers and reinforcements, which is needed to compensate for its inherent brittleness.

Fillers and reinforcements can also increase the strength, surface properties, dimensional stability, electrical properties, and overall cost. Glass-fiber-reinforced PPS is used for the mechanical and electronic applications requiring high mechanical strength, impact resistance, and insulating characteristics [118].

Xu et al. [118] studied the tribological behavior of PPS/CF composite coating under dry sliding and water lubrication. Figure 3.48 shows the variations in the friction coefficient of the composite coating with the sliding content of CF at the rate of 0.43 m/s under 200 N. This showed that the friction coefficient of the composite coating appeared to slightly decrease with the increasing CF content, both under dry- and water-lubricated conditions. At the same time, the friction coefficient under water-lubricated conditions was much lower than that under dry sliding.

3.2.10 Polypropylene Resins

PP is a thermoplastic polymer with a linear structure made from the propylene monomer using Ziegler–Natta polymerization and metallocene catalysis polymerization, as shown in Fig. 3.49.

The short segments of PP show examples of isotactic and syndiotactic tacticity, as depicted in Fig. 3.50.

PP resins have cheap, lowest density (0.90–0.92 g/cm^3), high tensile and compressive strength, melting point of 160 °C, excellent dielectric properties, and nontoxicity and are used in a wide variety of applications, including packaging and labeling, textiles, stationery, plastic parts and reusable containers of various types, laboratory equipment, loudspeakers, automotive components, and polymer

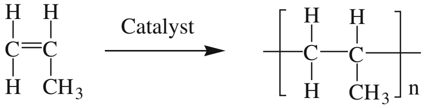


Fig. 3.49 Schematic of synthesis of PP

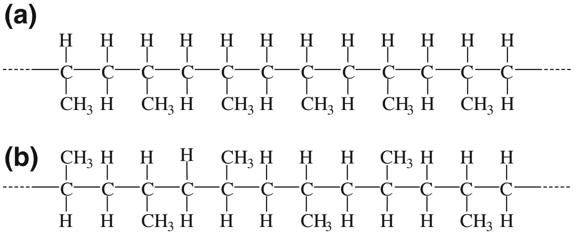


Fig. 3.50 Chemical structures of isotactic and atactic PPs. **a** Isotactic PP. **b** Atactic PP

banknotes. As an addition polymer made using the propylene monomer, it is unusually resistant to several chemical solvents, bases, and acids [119–121].

In general, homopolymers can be used for housing, housewares, packaging, cassette holders and fibers, monofilaments, and film tapes; copolymers are preferred for all applications exposed to cold environments and widely used for pipes, containers, boat hulls, seat shells, and automotive parts.

In order to improve a few properties, PP formulas may include additives such as pigments, carbon black, rubbers, antioxidants, and UV stabilizers. PP is available in the form of molding powder, extruded sheets, cast films, textile staples, and continuous filament yarns [122–124].

Li et al. [125] demonstrated the interfacial compatibility of PP composites filled with surface-treated CF. Figure 3.51 shows the three-point bending value of PP/CF composites as a function of treatment time. It was observed that the bending strength and modulus increased with the increasing treatment time, thereby indicating that the interfacial adhesion between CF and PP matrix increased.

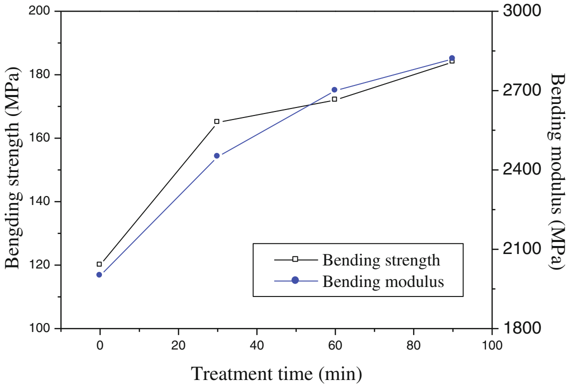


Fig. 3.51 Bending strength and modulus of PP/CF composites [125]

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