Chapter 15
Nonconventional Composites

We devote this chapter to some nonconventional composites. Nonconventional composites include nanocomposites (polymer, metal, and ceramic matrix), self-healing composites, self-reinforced composites, biocomposites, and laminates made of bidimensional layers. These topics are in the early stages of their development but they are potentially quite important. We provide below a brief description of these topics.

15.1 Nanocomposites

Nanocomposites are composites that have one component that has at least one dimension in the nm range; generally it will be the reinforcement phase which may be nanotubes, nanofibers, or nanoparticles. Although, they can have a polymeric, metallic, or ceramic matrix, a polymer matrix is more common (Barrera 2000; Barrera et al. 2005; Shofner et al. 2003, 2006). There is a strong potential for producing strong and wear-resistant metal matrix nanocomposites by liquid processing and powder processing routes (see, for example, Fukuda et al. 2011). Before that happens, however, researchers will need to devise processing techniques to make these materials in bulk, in a cost-effective manner, without voids or other defects.

15.1.1 Polymer Clay Nanocomposites

A special type of nanocomposite, called nanoclay composites, has made good progress into commercial production. Polymer matrix reinforced with nanoclay particles has become commercially viable (Ajayan et al. 2003; Koo 2006; Lee et al. 2005; Okada and Usuki 2006; Paul and Robeson 2008). Most of the clay additives are in the form of platelets that have been organically treated. They are commonly called nanoclays because their thickness is in the nanometer range.

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Chemically, these clays are layered magnesium aluminum silicates. Their dimensions are as follows: thickness of about 1 nm and length or width in the range of 70–150 nm, which would give an aspect ratio of over 100. Surface modification of the clays makes it possible to disperse them in thermoplastic materials. These nanoclay particles can enhance the modulus (function of amount of particles added) while lowering the coefficient of thermal expansion.

The work in this area was pioneered by Toyota researchers in 1990 when they first used clay/nylon 6 composites for timing belt covers. Some of the earliest commercial successes can be credited to Toyota, GM, and Ford. Major automakers have started making use of nanocomposites in their autos. General Motors Corp., in particular, in 2001, used a thermoplastic olefin (TPO) step-assist for the '02 GMC Astro/Safari van that was enhanced with a clay nanomaterial. GM also used clay nanocomposites in the Chevrolet Impala. These products are made of thermoplastic polyolefin–clay nanocomposite. The nanocomposites are lighter, stiffer, and cost-effective compared to the conventional polymeric materials. The cost-effectiveness part is important because the automobile industry is loath to use anything that is not cost-effective, exception being sports cars. Also, the crash requirement in autos is very important. This characteristic has to do with the energy absorbed in the impact, which in the case of autos depends more on strain than on stress.

In regard to nanoclay composites, mention should be made of Southern Clay Products Co. Cloisite® and Nanofil® are commercially available clay additives from this company that are used in automotive clay nanocomposites. These are organically modified, nanometer scale, layered magnesium aluminum silicate platelets. The key issue in nanoclay/polymer composites is that of polymer/clay compatibility at the interface. This requires that, in most cases, one must impart an organophilic character to the clay. This is commonly accomplished by adding amino acids, organic ammonium salts, or tetra organic phosphonium solution. The surface modification of platelets with an organic graft allows dispersion and miscibility with the thermoplastic matrix materials. The additives serve to reinforce thermoplastics resulting in somewhat higher stiffness and lower coefficient of thermal expansion. The additives also improve gas barrier properties of thermoplastics. The surface char formation and flame retardance of thermoplastic systems have also been improved by incorporating the nanoclay particles into the structure. In the case of hydrophilic polymers and silicate layers pretreatment is not necessary. Most polymers are hydrophobic and are not compatible with hydrophilic clays. In such cases, a treatment that results in exfoliation of the clays is needed. By exfoliation we mean peeling of clay flakes or platelets from one another, and dispersion into the polymer matrix. There is some confusion about such terminology in the literature. Let us define these terms. By intercalation we mean that some of the organic polymer is inserted between the flakes of the clay, which would increase the interlayer spacing but individual flakes stay together. In an exfoliated structure, the layers of the clay are well separated and the individual layers are distributed throughout the organic matrix. A third alternative is dispersion of complete clays within the polymer matrix; this is akin to use of the clay as conventional filler. Figure 15.1 shows schematically three different morphologies:

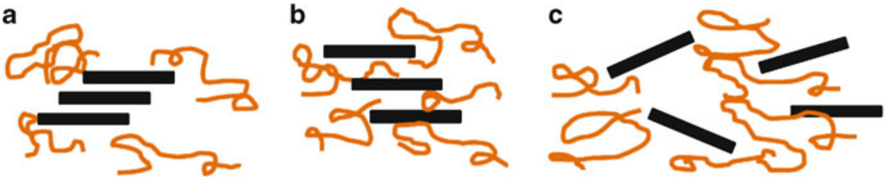

Fig. 15.1 Three different morphologies of platelets (*black*) dispersed in a polymeric matrix: (**a**) immiscible, (**b**) intercalated, and (**c**) miscible. Note the increased spacing between sheets in (**b**)

immiscible, intercalated, and miscible platelets. The treatment or process of exfoliation can result in nanoclay sheets with aspect ratios between 100 and 1,000.

It should be pointed out that, in general, the nanoclays improve the strength and stability of the resin, i.e., they are more functional than conventional "fillers" such as talc or mica used in sheet molding processes. Also, the use of nanoclays results in enhanced aesthetics such as surface quality, color appearance, and enhanced scratch resistance. However, generally the amount of nanoparticles used is very small, 2–3 % by weight.

As mentioned above, the important issue is polymer/clay compatibility at the interface. This requires that, in most cases, one must impart an organophilic character to the clay. This is commonly done by adding amino acids, organic ammonium salts, or tetra organic phosphonium solution. In the case of hydrophilic polymers and silicate layers pretreatment is not necessary. Most polymers, however, are hydrophobic and are not compatible with hydrophilic clays. Following methods can be used to make the clays compatible with polymers.

Solution induced intercalation. In this method, one solubilizes polymer in an organic solvent, and the clay gets dispersed in the solution, following which either the solvent is evaporated or the polymer precipitated. The clay dispersion is not very good, and the process is expensive because of the high cost of the solvents required, and health and safety issues.

In situ polymerization. In this method the clay layers are dispersed in the matrix by polymerization. The silicate layers are mixed with the monomer together with the polymerization initiator and/or the catalyst.

Melt processing. In this case the silicate layers are dispersed into the polymer melt. The silicates need to be surface treated by organo-modification prior to introducing in the polymer melt.

15.2 Self-Healing Composites

Self-healing or self-repairing of materials is a very interesting concept. The concept is quite general inasmuch as it is applicable to monolithic materials as well, not just composites. For an overview of this field, the reader is referred to van der Zwaag (2007). The concept of self-healing is taken from the field of medicine. The basic premise is that, in a self-healing system, when damage occurs, the system realizes that something is wrong. Then, without human intervention, the self-healing system makes the necessary adjustments and returns to normal operation. The process has been called an autonomic healing process (White et al. 2001). The term autonomic, again borrowed from medicine, refers to automatic functioning of a system. The self-healing process mimics the spontaneous healing processes observed in living systems when they are damaged. In a living system, damage (for example formation of a bruise) triggers an autonomic healing process, i.e., when an organism is damaged, there is a natural healing response. Researchers have applied this concept to synthetic material design and have developed self-healing materials. The basic idea is to incorporate a healing agent in the material of interest which will come into action when damage in the form of a crack appears. It is easy to appreciate the appeal of this concept in composites by recalling that increasing amounts of PMCs are being used in civilian infrastructure (bridges, decks, columns, etc.).

The idea of autonomic healing in a composite works is shown schematically in Fig. 15.2. The healing or repair agent (a kind of cross-linking polymer) encapsulated in a hollow microsphere (or a hollow fiber) can be embedded in the matrix. The polymer matrix has a catalyst distributed in it that serves to polymerize the healing agent. Damage in the form of a crack is shown in Fig. 15.2a. When the crack propagates and breaks the microsphere, the healing agent is released through capillary action. Polymerization occurs when the healing agent encounters the catalyst and the crack faces are closed by bonding as shown in Fig. 15.2c. A self-healing PMC can be made by using a microencapsulated healing agent and a solid chemical catalyst, both dispersed within the polymer matrix phase. Healing or closure of a crack will occur when the crack propagates through the microcapsules releasing the healing agent onto the crack plane. The healing agent, under the action of chemical catalyst, initiates polymerization and bonding of the crack faces. Kessler et al. (2003) used this approach in a carbon fiber/epoxy composite. The self-healing composite system consisted of a healing agent, microcapsule shell, chemical catalyst, epoxy matrix, and carbon fiber. They used the monomer, dicyclopentadiene (DCPD) as the healing agent, which possesses low viscosity and excellent shelf life when stabilized with 100–200 ppm *p*-*tert*-butylcatechol. The healing agent was encapsulated in poly-ureaformaldehyde by in situ polymerization (White et al. 2001) to yield the self-healing PMC.

Self-healing was demonstrated on width-tapered double cantilever beam fracture specimens in which a mid-plane delamination was introduced, which was allowed to heal. Specifically, healing at room temperature yielded as much as 45 % recovery of undamaged value of interlaminar fracture toughness, while healing at 80 °C increased the recovery to over 80 % (Kessler et al. 2003). An extension of this concept was reported by Kirkby et al. (2007). They embedded shape memory alloy (SMA) wires in the composite and observed improvements in performance, which were attributed to three effects: (1) crack closure, which reduces the crack volume and the amount of healing agent required; (2) heating of the healing agent during polymerization, which improved the cross-linking; and (3) mechanical registration of the two crack faces, which resulted in a reduced crack volume on closure.

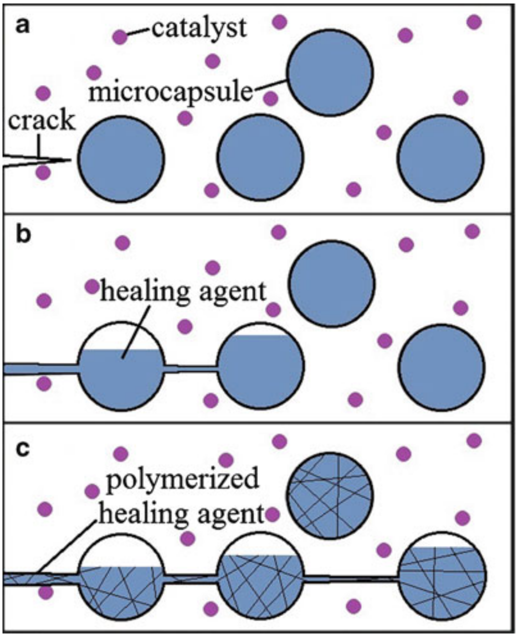

Fig. 15.2 The concept of autonomic healing in a composite. The healing agent is encapsulated in hollow microspheres. (**a**) Damage in the form of a crack. The matrix has catalyst in it and microcapsules containing the healing agent. (**b**) When the crack breaks the microsphere, the healing agent is released. (**c**) Polymerization occurs and the crack faces are closed

15.3 Self-Reinforced Composites

A new class of polymer matrix composites, called self-reinforced or single polymer composites (SPC), has emerged. A common example is polypropylene reinforced with glass fibers to increase strength and stiffness. This, however, reduces the recyclability of polypropylene, one of the hallmarks of thermoplastics such as polypropylene. It also causes problems in achieving an acceptable surface finish. Enter the concept of self-reinforcement, i.e., use aligned polypropylene to reinforce polypropylene; such a material will have an intermediate stiffness, good impact performance, a “class A” surface finish, and it is fully recyclable (Capiati and Porter 1975; Matabola et al. 2009). By aligned polypropylene one means polypropylene fiber wherein the molecular orientation of chains has been introduced, e.g., by hot drawing.

The main driving force for this is the fact that they are easily recyclable and thus bring in economic and environmental benefits. The mechanical properties of these self-reinforced composites are slightly less than, but comparable to, the conventional fiber reinforced composites. The main difficulty in processing them is the rather small difference in the melting point of the fiber and the matrix. The processing window is generally very short, 1–2 °C; however, the sheets can be post-formed over a range of 20 °C because the remelted skins of the tapes have a lower softening temperature. As we saw in Chap. 2, drawing of thermoplastics can result in some chain alignment, which in turn can lead to an increase in modulus. Polypropylene (PP) fibers in a polypropylene matrix, called self-reinforced polypropylene, are available commercially: Curv® and Moldable Fabric Technology (MFT) are self-reinforced 100 % PP composites that contain PP fibers in a PP matrix. The process of making such self-reinforced composites is to heat and compact highly oriented, small-diameter fibers under suitable time, temperature, and pressure conditions to form a composite wherein a substantial proportion of the original molecular orientation of fibers is retained. Generally, in practice, this means that some 10–20 % of the original fiber gets melted and loses the superior mechanical characteristics but serves to produce matrix. Figure 15.3 shows the microstructure of such a composite. The material properties are enhanced through molecular orientation, resulting in *self-reinforcement*.

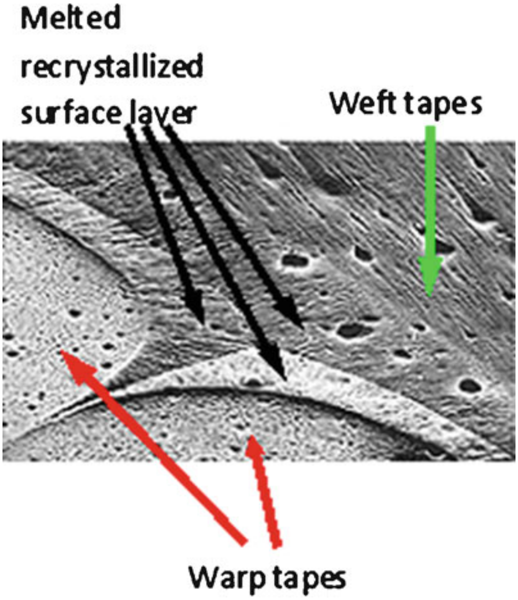

Fig. 15.3 Microstructure of a self-reinforced composite (PP fabric in a PP matrix)

The tapes are woven into fabric to provide ease of handling and balance. This material offers excellent impact resistance, lightweight, and high stiffness compared to non-drawn PP. Curv® is used in automotive exterior (bumpers, body panels, underbody shields, wheel arch liners), interior (load floors, pillar trims, door and head liners), audio products, sporting and travel goods, as well as ballistic protection.

15.4 Biocomposites

Biocomposites can be defined as composites that have at least one component that is derived from biological or natural sources. There is a growing interest in these composites (see, for example, Fuqua and Ulven 2008; Mohanty et al. 2005; Silva et al. 2008; Sui et al. 2009; Wool and Sun 2005). The main attributes of biocomposites that are driving the effort in this area are the following:

- Biodegradable
- Renewable
- Cheap
- Natural resource

Biocomposites, based on these attributes, are environmentally friendly. However, the ultimate test for their success in the commercial arena will depend on whether they meet the application requirements. Among the problem areas in their widescale acceptance are the following:

- High moisture absorption
- Poor wettability of fibers by polymeric resins leading to poor interfacial bonding
- High viscosity of bioresins making processing more difficult

There are many examples of composites found in nature. Shells (abalone and mussel) are good examples of how living organisms synthesize layered composites that show high strength and high toughness. Layers of calcium carbonate ($CaCO_3$, orthorhombic crystal structure) and an organic adhesive are stacked in a brick and mortar type structure. The mortar gluing together the bricks of calcium carbonate is a thin film of proteins and polysaccharides. This biocomposite consists of about 95 % inorganic mineral and 5 % organic polymer, with electrostatic forces acting at the interface. The organic glue is strong enough to hold layers of calcium carbonate together, but weak enough to allow the layers to slide and thus absorb the energy of an impact or to deflect an oncoming crack instead of letting it propagate in a catastrophic manner through the brittle calcium carbonate. Such ingenius materials science and engineering work done by nature is a source of work in the field of biomimetics. Some work related to synthetic laminates is described in the next section.

15.5 Laminates

Laminates are multilayered composite materials. We discussed fiber reinforced laminates made by appropriately stacking fiber reinforced plies. The laminates we are discussing in this section are in general made by putting together sheets, except for variety called fiber metal laminates (see below). Some of these laminates have been in commercial use for sometime while others are potential candidates for a variety of applications because of their superior properties: high strength (ambient and high temperature), fatigue resistance, wear resistance, and biocompatibility.

Examples of laminated structural materials can be found in nature and in historical manmade objects. The unique structure of laminates found in nature offers desired functional properties. Examples include bones, tooth, shells of mussels, etc. For example, abalone shell consists of protein layers separating the mesolayers. Among historic examples, we should mention metallic laminates made of steel that have been used to make Samurai swords. The famous iron pillar of Delhi consists of laminae of wrought iron. Other mundane examples include copper/aluminum and copper/stainless laminates for use in kitchen utensils. Researchers have explored multilayered composites consisting of metal/metal and metal/ceramic laminates. Extensive work on ultrahigh carbon steel (UHCS) laminates, which have compositions very similar to ancient Damascus or Wootz steels, showed excellent impact properties (Verhoeven et al. 1998; Lesuer et al. 1996).

A general view of the structure of laminates at different length scales is provided in Fig. 15.4 (Deng et al. 2005; Woerden et al. 2003; Gavens et al. 1999). Here we wish to highlight the microstructure of the multilayered Al/SiC composites made by DC/RF magnetron sputtering. Figure 15.5a shows the nanolaminate as observed in SEM while Fig. 15.5b shows the interface in this system as observed in a transmission electron microscopy (TEM); no reaction product is observed at the interface

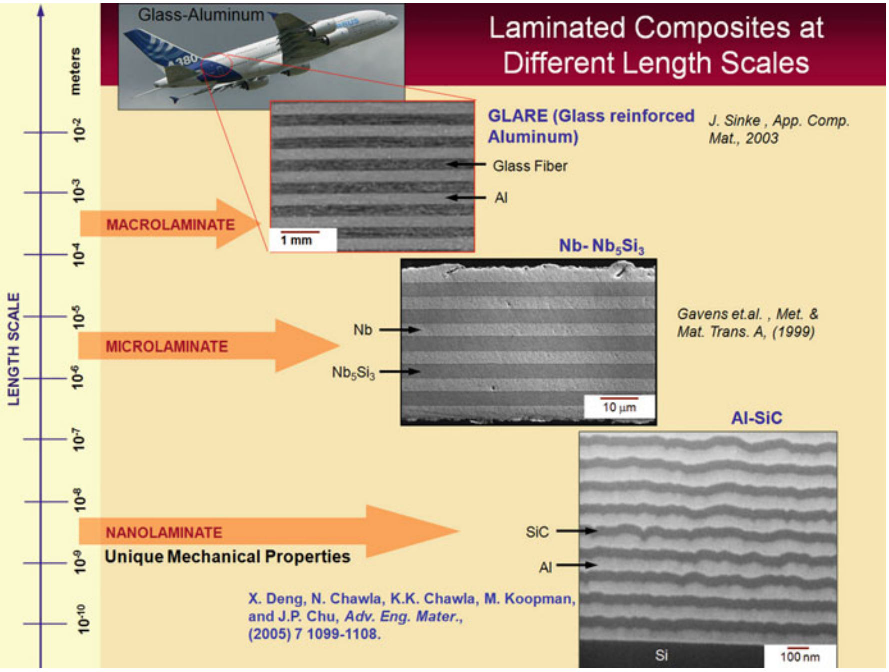

Fig. 15.4 A general view of the structure of laminates at different length scales

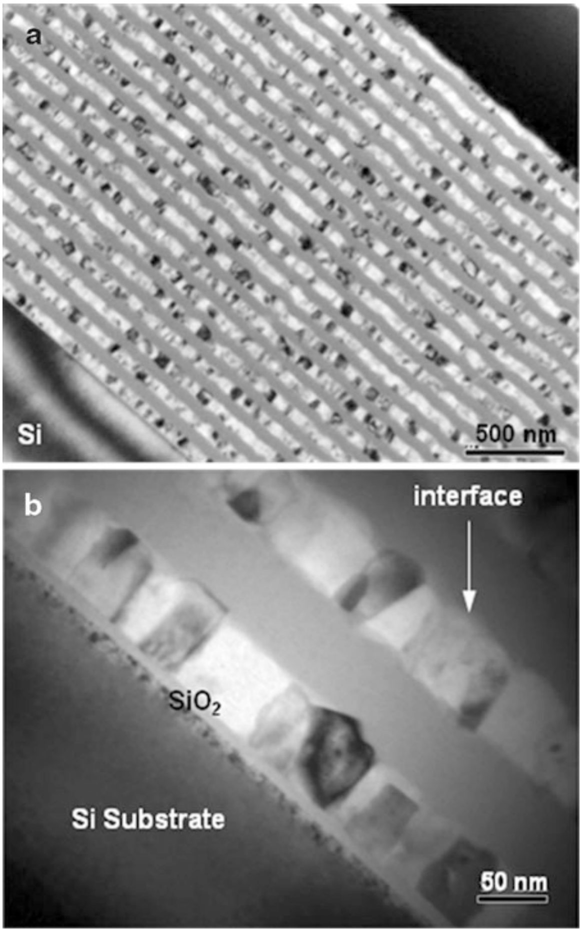

Fig. 15.5 (**a**) Microstructure of SiC/Al nanolaminate. (**b**) TEM micrography shows the well-bonded interface and absence of any reaction product at the interface. SiO$_2$ forms on the surface of SiC

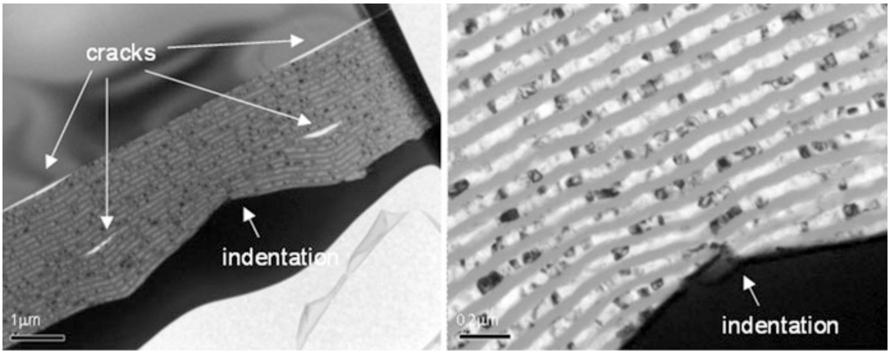

Fig. 15.6 Deformation of SiC/Al nanolaminate in a nanoindenter. Note the well-bonded interface and the extreme flexibility of SiC, an inherently brittle material

between Al and SiC. The elastic and plastic behavior of the multilayered materials, as investigated by nanoindentation, showed a high degree of flexibility of brittle SiC layers, see Fig. 15.6, before and after indentation. Cracking under indentation started at a depth of 1,000 nm in SiC layers. This occurred at sites where kinks formed in SiC which appeared to occur at sites of shear localization.

15.5.1 Ceramic Laminates

Multilayered ceramic capacitors (MLCCs) represent a commercially very important and successful ceramic/metal laminate system. Barium titanate ($BaTiO_3$) is a ferroelectric material with a high dielectric constant. MLCCs are laminates made of alternate layers of $BaTiO_3$ dielectric and inner electrodes of Pd, Pt–Ag, or Ni. The inner electrodes are exposed at the opposite sides of the laminate. MLCCs are widely used in the electronics industry. These capacitors, manufactured by alternately layering electrodes and thin dielectric, are small, efficient, reliable, and cost-effective, and used in virtually every electronic circuit.

Laminated glass is an example of glass/polymer laminate that is widely used but not appreciated as a sheet laminate composite, which is what it really is. One of the flat glass products is the automobile windshield (about 25 % total flat glass production is consumed by the automobile industry in the USA) that amounts to over $500 million market. The USA and many other countries require by law that automobile windshields be made of laminated glass. The glass composition is mostly SiO_2 with some $Na_2O + CaO + K_2O + MgO + Al_2O_3$. The laminate is made of two sheets of tempered glass (each 0.75 mm thick) bonded by a layer (2.5 mm) of tough plastic (PVB—poly vinyl butaryl) in an autoclave. PVB/glass has good adhesion. PVB is transparent and can be tinted to protect against UV radiation. The finished laminated glass is virtually indistinguishable from ordinary glass, because the PVB is invisible and covered on both sides by the glass. The main advantage is that if the glass laminate gets broken, the glass shards stick to the plastic interlayer rather than cleanly breaking apart and do a lot more damage. The minor disadvantage is that the PVB laminated glass has less strength than ordinary glass of the same size and thickness.

15.5.2 Hybrid Composites

A novel composite material, erroneously named fiber metal laminate (FML), has been developed. It consists of a metal sheet (~ 0.3 mm thick) which can be aluminum, steel, titanium, magnesium, etc. and a polymer matrix composite (PMC) prepreg which consists of fiber reinforced polymer; see Fig. 15.7. The composite consists of alternate layers of PMC and metal sheet. In principle, the fiber can be continuous or not; it can be glass, aramid, or carbon and the resin

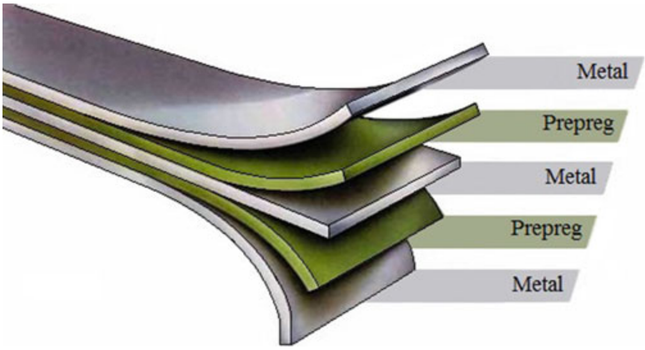

Fig. 15.7 A fiber metal laminate (FML) made of a metal sheet and a polymer matrix composite (PMC) prepreg which consists of a fiber reinforced polymer composite

matrix can be any polymer but is commonly epoxy. The nomenclature scheme goes as follows:

- GLARE: *GLA*ss fiber *RE*inforced laminates
- ARALL: *AR*amid fiber reinforced *AL*uminum *L*aminates
- CARE: *CA*rbon fiber *RE*inforced laminates
- TIGR: *TI*tanium/*GR*aphite-epoxy laminates

Common aircraft fuselage material is an aluminum alloy. In the Airbus A 380 jumbo aircraft the panels for the upper fuselage section of the aircraft are made GLARE which consists of laminates made of alternate layers of glass fiber/epoxy (a PMC) and aluminum sheet. Such hybrid composites combine advantages of metals and PMCs, to wit:

- Excellent fatigue and impact properties
- High strength and stiffness
- Flame resistance
- Easily machined or formed
- Mechanical fastening capability

There is some interesting history with the use of GLARE in aircraft. Earlier, fuselage construction with ARALL was tried. It was observed that under loading conditions encountered in an aircraft fuselage, the aramid fibers around a fatigue crack would break. Once the fiber was broken, the crack was no longer inhibited and would grow. The growing crack would lead to delamination, which placed a compressive stress on the aramid fibers, causing buckling. Low compressive stress of aramid fibers is well known (see Chap. 2; Chawla 1998). It was observed that aramid fibers would break under rather low compressive loads and it also became evident that ARALL was also sensitive to strength reductions caused by any holes that might be drilled in it. Such holes are unavoidable in a large structure such as a fuselage; they would lead to premature fatigue cracks to develop. Although use of ARALL was not suitable for fuselage construction, the weight savings by using this material were very attractive. Earlier, ARALL was used in the C-17 military transport plane. Because of heavy weight problems toward the rear of the plane, the large aluminum cargo door was replaced with ARALL.

This was followed by replacing aramid with glass fibers in the new FML called GLARE, which has superior blunt notch strength and the ability to handle greater compressive stresses than ARALL. GLARE is also more resistant to impact than ARALL and for this reason the first commercial application of GLARE was in the cargo floor of Boeing's 777 plane where the excellent impact properties of GLARE were put to use. The next stage of use of Glare was in the form of panels for the fuselage of Airbus's A380 aircraft. At first the manufactured flat panels lacked the cost-effectiveness necessary for the material to break ahead of conventional aluminum structures despite the excellent mechanical properties. The cost of manufacture of GLARE composites was roughly 7–10 times that of aluminum per kilogram and the next step was to lower the cost of manufacture (Vlot and Gunnick 2001). The dimensions of metal sheet with the required 0.3–0.4 mm thickness were limited to a width of 1.65 m. For the aluminum/S2-glass fiber in epoxy system, failure occurred primarily at the metal/adhesive interface. The thicker specimens debonded faster than thinner ones, hence the need for very thin aluminum sheet to increase the fatigue resistance of GLARE. The manufacture of flat panels of GLARE in an autoclave, although expensive, was an important step in GLARE production. The main source of fatigue resistance in Glare is the intact bridging fibers in the wake of the crack; they restrain crack opening. Figure 15.8 shows the slow fatigue crack growth characteristics of GLARE compared to monolithic aluminum alloy.

Yet another type *hybrid composite* is a composite containing more than one type of fiber. We described this in Chap. 13. Such composites, by using two or more types of fibers, extend the idea of tailor-making a composite material to meet specific property requirements. Partial replacement of expensive fibers by cheaper but adequate fiber types is another attractive feature of hybrid composites. Additionally, there is the possibility of obtaining a synergistic effect in the fatigue behavior of hybrid composites. Such synergistic results are by no means universal. For example, in the case of the flexural fatigue strength vs. the number of cycles for 100 % unidirectional carbon fiber/polyester, 100 % chopped glass fiber/polyester, and unidirectional carbon fiber faces over chopped glass core, the hybrid composite curve was intermediate between the 100 % carbon and 100 % glass curves (Riggs 1985).

Another version of hybrid laminated MMCs is a family of layered composites consisting of metallic outer skins with a viscoelastic core material (for example, polyethylene, nylon, polypropylene, paper, or cork). Such composites will be useful where sound and vibration damping are required. The viscoelastic layer provides a high loss factor, i.e., a high capacity to convert vibrational energy to heat.

Although such composites are commonly made of metal and thermoset matrix PMCs containing continuous fibers, this concept of hybrid composites has been extended to thermoplastics and discontinuous fibers (Abdullah and Cantwell 2006; Cortes and Cantwell 2006; Kulkarni et al. 2007).

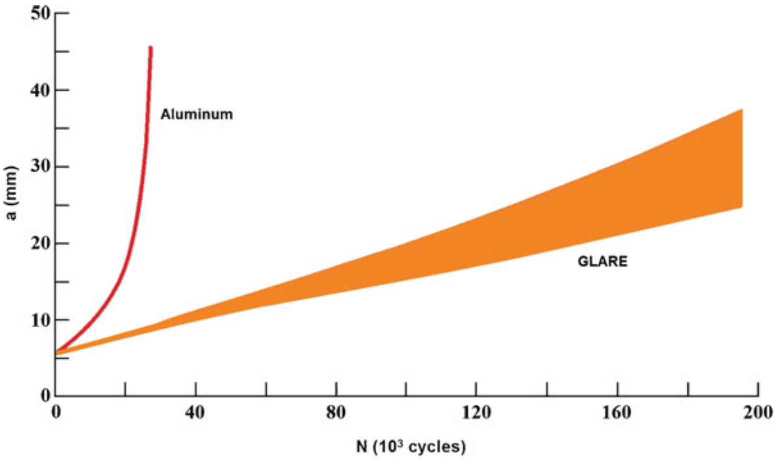

Fig. 15.8 Fatigue crack growth of GLARE compared to monolithic aluminum alloy

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