CHAPTER 1

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1.1 OVERVIEW

In recent years, composite materials are finding ever-increasing use in the aerospace, marine systems, automotive, electronic, defence and other industries because of their inherently superior specific strength and specific modulus. Composite material consists of a combination of two or more materials to give unique combination of properties. This definition is more general and can include metal alloys, plastics, minerals and wood [1].

Composite materials have been utilized to solve technological problems since long time but only in the 1960s these materials started capturing the attention of industries with the introduction of polymeric-based composites [2]. Since then, composite materials have become common engineering materials and are designed and manufactured for various applications including automotive components, sporting goods, aerospace parts, consumer goods, and in the marine and oil industries [3]. The growth in composite usage also flourished because of increased awareness regarding product performance and increased competition in the global market for lightweight components. Among all materials composite materials have the potential to replace widely used steel and aluminum with much better performances, replacing steel components with composite components can save up to 60 to 80 % in component weight, and 20 to 50 % weight by replacing pure metal parts [4].

Nano-composites are a class of composite materials where one of the constituents has dimension in the range of nanometers. In the last decade, there has been a strong emphasis on the development of polymeric nano-composites. The increasing interest in the development of polymeric nano-composites is possibly not only because of their high specific strength but also for the possibility of making products with unique mechanical, electrical and thermal properties. However these properties depend on several factors such as type of nano-filler, type of polymer
At present, there are many types of nano-fillers available to produce polymer nano-composites. Some of the most commonly used nano-fillers are carbon nano-fibers (CNFs), carbon nano-tubes (CNTs) and nano clay. Both CNFs and CNTs have similar rope-like structures, but CNTs exhibits much smaller diameter and better mechanical, thermal and electrical properties compared to CNFs. However, addition of CNTs to polymeric resin significantly increases the viscosity of polymers. In addition, it is difficult to uniformly disperse CNTs into polymer due to strong van der waals forces between them. Contrary to CNTs, CNFs are drawing significant attention to form nano-composite because of its relatively better dispersion and low cost [6].

Lamination is the technique of manufacturing a material with different layers. Laminar composite is a type of composite material, which consists of two or more layers of similar or dissimilar materials that are bonded together.

**Figure 1.1 Schematic diagram of laminar nano-composite**

In case of adhesive laminated nano-composite, the layers consist of nano-adhesive materials. The synthesis of nano-adhesive materials involves incorporation of nano-fillers into
the matrix adhesive. These nano-fillers have very high modulus and high strength properties. Typical nano-filler used includes graphite, glass, boron etc. Matrix materials are adhesive based on epoxy, polyurethane, polyimide etc. [7].

Adhesive contributes highly to structural integrity, ease of manufacturing, enhanced performance, improved safety, cost and time saving [8]. With wide range of possible fillers and resins, the laminar composite materials have opened the way to an enormous range of materials with diverse chemical, thermal, physical as well as mechanical properties. These materials have an impressive and diverse range of applications in automotive, aviation, spacecraft, civil infrastructure and sports industry. For instance, laminar composite materials with high specific stiffness and strength, excellent fatigue resistance and low coefficient of thermal expansion, as applied to the space structure and being the optimal conditions that are to be met by the structural design requirements of spacecraft, space shuttle and launch vehicles [9].

In recent years, a remarkable growth in the use of polymers has been observed for technological application. Major industrial sectors like automotive industries, aircraft industries, chemical industries, railways as well as many civil construction works are increasingly using polymers adhesively bonded in secondary structures replacing traditional engineering materials such as steel and aluminum [10]. Unique, physical and chemical characteristics of polymers such as high strength to weight ratio, resistance to corrosion and relatively low cost make them attractive [11]. Amongst the polymers used in engineering applications, polyether imide (PEI) and polyether ether ketone (PEEK) are popular due to their superior thermal and mechanical properties [12]. In addition these polymers have lower rigidity leading to flexibility in design for space aviation applications [13]. Further, the lower rigidity of these polymers to have led flexibility in design for space and aviation application. Unfortunately, these polymers are
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Chemically inert and have lower surface energy. Hence, surface modification of polymers is often carried out to enhance their wetting characteristics for improved adhesion, for greater strength in metal-polymer laminar composite [14].

Adhesion of solid surfaces depends on wetting [15]. Wetting is quantitatively defined by reference to a sessile liquid drop resting on a solid surface at equilibrium. For perfect wetting, contact angle, \( \theta \) should be equal to 0°. At \( \theta < 0° \) there is no equilibrium and the situation corresponds to spreading wetting [16]. It is also possible for liquid adhesive to spread and wet a solid surface even when \( \theta > 0° \) but this requires the application of force to the liquid adhesive to spread it forcibly over the solid surface [17]. A widely used method for approximating the surface energy of solid substrate in the context of wetting was developed by Zisman (1950) by introducing the concept of critical surface tension, \( \gamma_C \), the minimum surface tension of the liquid below which the liquid spreads on solid spontaneously. The critical surface tension \( \gamma_C \) has been taken as an approximate measure of the surface energy \( \gamma_{SV} \) of a solid. Polyether imide and polyether ether ketone have low energy surfaces. Thus, these materials are difficult to wet and bond as it requires adhesive of surface tension \( \gamma < \gamma_C = \gamma_{SV} \). On the other hand metals have high values of \( \gamma_{SV} \) and may be readily wetted by many organic adhesives exhibiting good bonding [18].

The surface energies of liquid adhesives are less than 100 mJ/m². Surface energies of metals are about 500 mJ/m² whereas that of polymers is less than 100 mJ/m². Therefore, the liquid adhesives spread freely on solids of high surface energy like metals, since there is a large decrease in the free energy of the system accompanying spreading and consequently, it shows high work of adhesion. But the surface energies of liquid adhesives are comparable to those of
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low energy solids like polymers; hence the system shows non-spreading and low work of adhesion [19].

Polymers exhibit chemically inactive surfaces having low surface energy, in general. In the context of adhesion, it is well known that low energy surface lead to poor adhesion. Therefore, it is necessary to modify the surfaces by introducing various polar groups so as to increase their surface energy for better adhesion. Glow discharge under low-pressure plasma is a popular technique, which results in better uniformity in surface modification of the polymers [20]. The temperature of gases in a glow discharge generally remains low and the plasma plays a predominant role in the surface modification of polymers. It is possible to strike glow discharge with either a direct current (DC) or radio frequency (RF) power source. Therefore, to improve adhesion of polymers, an effort will be made in the present work to increase surface energy of polymers by DC glow discharge [21].

Apart from the surface modification of polymer by glow discharge, adhesion may also be improved by inducing strain on a polymer surface, which provides improved joint strength. Further the metal surface may also be subjected to plasma nitriding, which also improves the strength of plastic-metal joint [22].

Many adhesives are used for joining, depending upon the mode of application, setting, chemical composition, cost and sustainability of various adherends. The broad general classification as structural and non-structural adhesives is generally made on the basis of application. The structural adhesive is normally defined as one, which is employed in joints or load carrying member associated with primary design. These types of adhesives are subjected to large stresses. Non-structural adhesives are not capable of supporting appreciable loads and are
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generally required to locate parts in an assembly. Based on these considerations the commonly used structural adhesives include polyurethane, polysulfide, polyester, acrylic and epoxies. These adhesives after mixing with hardener are employed on the surface of the substrate in liquid form having low viscosity at the time of application to the surface of the substrate. However, to develop high cohesive strength it is necessary to harden the adhesives through curing. Curing is formation of cross-links between adhesive molecules with the help of hardener and it becomes hard by the reaction polymerization [23].

Mechanical properties of a polymer-metal adhesive joint are governed by various factors, such as surface characteristics of the polymer and the metal as well as the physic-chemical properties of these materials which govern stress distribution in the joint, thereby influencing both the mode of failure and the joint strength. Apart from these properties, geometrical factors; such as adhesive layer thickness, design and dimension of the polymer and the metal, overlap length of the joint, strain rate and environmental conditions during mechanical testing play important role in deciding the mechanical properties of the joint [24].

Despite these all-positive aspects of utilizing laminar composite materials, if the structures are required to operate for an extended period of time in space, they may have difficulty in maintaining their thermal, mechanical and optical performance against the harsh space environment. These demanding conditions are limiting the use of plastic-metal joint materials for space and aviation industry [25].
1.2 OBJECTIVE OF PRESENT WORK

Based on above-mentioned considerations, attention is given to develop nanofibers reinforced laminar composites. The incorporation of nanotechnology in the field of composites has opened new horizons towards the development of advanced materials with unique functional properties. Due to high mechanical, thermal and electrical properties, nano-composites are widely used in the aerospace industry to manufacture load-bearing structures such as rudders, flaps, and undercarriage doors. Therefore, in order to meet to requirement of aerospace industry, research is continue to develop new high performance plastics (Polyether ether ketone and Polyether imide)-titanium laminar nano-composite, which can maintain the desired properties under working environmental condition for longer duration time and serve their purpose properly.
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