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Next Generation Fiber-Reinforced Composites New Insights

Edited by Longbiao Li, António B. Pereira and Alexandre Luiz Pereira





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Contributors

Citlalli Gaona-Tiburcio, Manuel Alejandro Lira Martinez, Jesús M. Jaquez-Muñoz, Miguel Angel Baltazar-Zamora, Demetrio Nieves-Mendoza, Francisco Estupiñan-López, Facundo Almeraya-Calderón, Laura Landa-Ruiz, Marianggy Del Carmen Gomez Avila, Bahadir Birecikli, Laxmisha M. Sridhar, Timothy M. Champagne, Jawed Qureshi, Hassaan A. Butt, German V. Rogozhkin, Dmitry V. Krasnikov, Albert G. Nasibulin, Andrei Starkov, Jalal El Bahaoui, Mohamed Chairi, Francisco Mata Cabrera, Issam Hanafi, Guido Di Bella, António B. Bastos Pereira, Alexandre Luiz Pereira, Mamaru Wutabachew, Longbiao Li

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Meet the editors



Dr. Longbiao Li is a lecturer in the College of Civil Aviation, at the Nanjing University of Aeronautics and Astronautics in China. His research focuses on the vibration, fatigue, damage, fracture, reliability, safety, and durability of aircraft and aero engines. He has 200 journal publications, 10 monographs, 4 edited books, 4 textbooks, 3 book chapters, 34 Chinese patents, 2 US patents, 2 Chinese software copyrights, and more than

30 refereed conference proceedings to his credit. He has been involved in different projects related to structural damage, reliability, and airworthiness design for aircraft and aero engines, supported by the Natural Science Foundation of China, COMAC Company, and AECC Commercial Aircraft Engine Company.



António Bastos Pereira has a Ph.D. in Mechanical Engineering. Since 2000, he has been a professor and researcher at the University of Aveiro, Portugal, where he held management positions in the Department of Mechanical Engineering. He is currently a director and researcher at the Centre for Mechanical Technology and Automation, University of Aveiro. His research focuses on the study of materials, including composites and

metals. He has 80 journal articles to his credit. Dr. Pereira has collaborated on thirty scientific research projects, having been the principal investigator for eighteen.



Alexandre Luiz Pereira has a Ph.D. in Mechanical Engineering and Materials Technology. He completed a postgraduate internship at the Department of Mechanical Engineering at the University of Aveiro in Portugal. Since 2014 he has been a professor and researcher at the Federal Center of Technological Education in Rio de Janeiro (CEFET/RJ). He is currently the coordinator of the Mechanical Engineering course at the CEFET/RJ Campus

Angra dos Reis. His main research areas are materials technology (structural and hybrid composites), hyperelastic materials, and adhesive joints.

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Preface

Composite materials are composed of two or more materials with different properties by physical or chemical methods on a macroscopic scale. The comprehensive performance of composite materials is better than that of the original constituent materials, thus meeting a variety of different requirements. Compared with traditional materials, composite materials possess high specific strength and modulus, good fatigue resistance, designability, and other beneficial characteristics. This book examines the next generation of fiber-reinforced composites. It discusses their mechanical properties and experimental and numerical evaluation, the application of fiber-reinforced composites in civil engineering, and multifunction carbon nanotube-reinforced composites. Adhesive bonding is a recent technology that in many applications can replace engineering materials such as rivets and bolts and processes like welding. Adhesives have the advantage of being lightweight and cost-effective for many applications in aeronautics, renewable energy, mechanics, and more. This book provides a comprehensive overview of adhesives science and technology, recent advances, and applications that use adhesive technology.

Longbiao Li College of Civil Aviation, Nanjing University of Aeronautics and Astronautics, Nanjing, PR China

António B. Pereira

TEMA - Centre for Mechanical Technology and Automation, Department of Mechanical Engineering, University of Aveiro, Aveiro, Portugal

Alexandre Luiz Pereira Federal Center of Technological Education in Rio de Janeiro (CEFET/RJ), Rio de Janeiro, Brasil

Section 1

Next Generation Fiber-Reinforced Composites

Chapter 1

Introductory Chapter: Next-Generation Fibre-Reinforced Composites

Longbiao Li

1. Introduction

Composite materials are materials with new properties composed of two or more materials with different properties by physical or chemical methods on a macroscopic scale [1–3]. The comprehensive performance of composite materials is better than that of the original constituent materials, thus meeting a variety of different requirements. The history of the use of composite materials can be traced back to ancient times. From ancient times to the present day, the straw reinforced clay and the steel reinforced concrete are made of two kinds of materials. In the mid-1960s, the carbon fibre-reinforced composite materials appear; in the early 1970s, the composites began to be used in aircraft structures. Compared with traditional materials, composite materials possess high specific strength and modulus, good fatigue resistance, designability and other characteristics. The number of applications in aircraft structures has been rising, the amount of composite materials in the Airbus A350 aircraft has been close to 40% of the total mass of the fuselage, and the Boeing 787 wing and fuselage use more than 50% of the composite materials [4, 5].

2. Characteristics of next-generation fibre-reinforced composites

For the next generation fibre-reinforced matrix composite, the composites should possess higher mechanical properties, that are modulus, strength, high delamination resistance and lower cost, especially for the reinforcing fibres. For the composites used in the high-temperature environment, the composites should possess high mechanical properties at elevated temperatures. Ceramic-matrix composites (CMCs) have a high specific strength, high specific modulus, low thermal expansion coefficient, high resistance to ablation, fatigue, creep, etc. They are new lightweight composite materials that combine structural load bearing and resistance to harsh environments. It has great potential for application in high-temperature structural components, such as thermal protection systems (TPS), for aerospace vehicles (ASV), aero engines, rocket engines and advanced nuclear energy [6]. To ensure the operation reliability and safety of composite structures, it is necessary to perform experimental and theoretical investigation on the design and mechanical properties evaluation of the composites.

3. Summary and conclusions

For the next generation of fibre-reinforced composites, the composites should possess higher mechanical properties that are modulus, strength, fatigue or creep lifetime and lower manufacturing cost. For the composites applied for the hightemperature condition, the composites should possess higher mechanical properties at elevated temperature and improve the fatigue or creep lifetime at elevated temperature.

Author details

Longbiao Li College of Civil Aviation, Nanjing University of Aeronautics and Astronautics, Nanjing, PR China

*Address all correspondence to: llb451@163.com

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Chapter 2

Composite Materials: A Review of Polymer and Metal Matrix Composites, Their Mechanical Characterization, and Mechanical Properties

Mohamed Chairi, Jalal El Bahaoui, Issam Hanafi, Francisco Mata Cabrera and Guido Di Bella

Abstract

Unlike conventional materials, composites have become an optimal option for a range of modern, industrial, clinical, and sports applications. This is combined with their noteworthy physical, thermal, electrical, and mechanical properties, as well as low weight and cost investment funds in certain cases. This review article attempts to give an overall outline of composite materials, regularly polymer-matrix composites (PMCs) and metal-matrix composites (MMCs). Polypropylene (PP) polymer and aluminum alloy were selected as matrices for this concentrate in light of their appealing properties and their use in different applications. Various studies address the different build-up materials, material handling, and the various properties. Mechanical characterization is an important cycle process for the development and design of composite materials and their components. It includes the determination of mechanical properties, for example, stiffness and strength according to standard test techniques (i.e., tensile, compression, and shear test strategies) distributed by the ASTM and EN ISO associations. Comparable to the determination of fatigue strength and fatigue life for composite materials. With respect to mechanical properties of composite materials, this paper reports several variables and limitations that affect mechanical property estimates, including material constituents, manufacturing process, test parameters, and environmental conditions.

Keywords: composite material, reinforcement, matrix, polypropylene, aluminum alloy, mechanical characterization, standard test methods, static, fatigue, mechanical properties

1. Introduction

All materials can be divided into four classifications based on their tendency. The last classification consists of at least two separate materials from the other three material classes combined into a single design unit [1]. Composite materials have been utilized as long as mankind has been gathering experience. Basically, every material in this world is a composite material, that is, a single piece of metal is actually a polycrystal (composite) of many individual crystals. Nevertheless, there are plenty of composite models in nature where the idea of composite material comes into play, for example, wood is really a sinewy material. Bone is another example of normal composite material. It is made of short and delicate collagen strands inserted in a mineralmatrix called apatite [2]. Animal bones were used by humans in the good old days as raw, regular composite material for fishing, hunting, and farming [3]. With the growth and development of societies in the last few years of the following 100 years, the world has had difficulty promoting advanced design materials to meet their needs. The 1930s can be considered as the start of the historical development of today's composites. In the 1960s, the concept of polymer-matrix composites (PMCs) advanced the economy in many areas, especially in the aerospace industry, to develop materials that are better protected against corrosion and fatigue damage. The energy emergency during the 1970s and the significant expense of fuel had expanded the interest for superior execution composites. Extraordinary efforts were made in the improvement of metal-matrix composites (MMCs) materials in the late 1970s. Toward the end of the twentieth 100 years, research focused on the use of low-cost carriers [3, 4]. Composite materials have supplanted steel parts, bringing about a weight saving of 60–80%. This makes them design materials and is utilized for assortment applications [5].

In the broadest sense, a composite is a material that consolidated at least two constituent materials or stages that have a distinctive point of connection between them [6, 7]. By choosing a suitable blend of composite and build-up material, it is possible to deliver a composite that precisely meets the needs of a specific application [8]. The matrix phase is soft and ductile, and serves to hold the reinforcing phase that is strong and stiffer and enhances the mechanical properties of the composite. The interface or the connection point between the two material phases transfers the loads applied by the external environment to the single reinforced material [9, 10]. In light of the matrix, composites are referred to as polymer matrix composites (PMCs), metal matrix composites (MMCs), and ceramic matrix composites (CMCs). Polymer composites consist of a thermoplastic or thermosetting polymer as a matrix, while they give a wide assortment of properties [4]. PP [11, 12], polyamides (PA) [13, 14], polyetherimide (PEI) [14], polystyrene (PS) [12], polyether ether ketone (PEEK) [15], and poly(phenylene sulfide) (PPS) [14] are some examples of common thermoplastic resins. While polyesters (PE) [16], epoxides [16, 17], and vinyl esters [17, 18] are the best-known thermosetting materials. Thermoset composites are considered strong with high stiffness and strength and good fatigue and creep resistance, but have low ductility and poor impact resistance, also they cannot be recycled [19, 20]. While thermoplastic composites are harder, less brittle, and exceptionally impact resistance, but they exhibit unfortunate creep resistance compared to thermoset composites. The main benefit of thermoplastic is that they can be easily reused [20]. The ceramic matrix is usually supported by other reinforcing ceramic types. They offer exceptional corrosion resistance, excellent compressive properties, and strength at high temperatures with elevated melting points, but the main disadvantages of CMCs are the disappointing deformation. On the opposite side, metal matrix composites are considered advanced materials. The common metallic matrices used for composite applications are aluminum, copper, iron, magnesium, nickel, and titanium alloys. Metal composites offer better mechanical and thermal properties than conventional materials, particularly wear resistance and thermal conductivity [4].

The classification of composites can also be based on the reinforcements and mainly consists of three main groups: particle reinforced composites, fiber reinforced composites, and structural composites. The particles can be of different sizes. To effectively support the composite, the particles must be small, have a certain size in all directions, and be uniformly introduced into the matrix. In fiber-reinforced composites, the reinforcements are in the form of fibers or strands. These can be continuousor discontinuous-fibers depending on the orientation, three extremes are possible for discontinuous fibers: a parallel orientation and a completely or partially random orientation. The fibers, in particular, can be whiskers or wires, depending on their diameter and character. The first form has an extremely large length-to-diameter ratio and the wires have a relatively large diameter [21]. Discontinuous filaments are often not as effective as continuous fibers in enhancing some mechanical properties, such as strength, stiffness, and creep resistance, while they are particularly productive in advancing other properties, such as fracture toughness, wear resistance, and thermal and electrical conductivity [22]. Structural composites consist of a system of layers bonded together to form laminate or sandwich structures. Laminate composites typically consist of made of multiple layers (plies) of arranged or random fibers bonded together by the matrix, the lay-up of each layer depending on the planned loading of the structure in which the laminate is to be used [23, 24]. Consequently, sandwich structure results from the joining of two face sheets or skins on a lightweight foal core, which is utilized to isolate the skins [25].

The most commonly manufactured filaments in composites are carbon, glass, and aramid. Carbon fibers are classified by their modulus (UHM, HM, IM, and LM), strength (HT and SHT), precursor materials, and heat treatment temperature. Glass fiber can be adjusted for different applications, for example, E-glass, which is excellent as an electrical resistant, C-glass, which has better chemical resistance to acids, and type T-glass for thermal protection. S-glass is of high strength and is broadly utilized in composite materials [26, 27]. Aramid filaments are synthetic organic fibers formed by highly crystalline aromatic polyamides [9]. Nomex® and Kevlar® are two known aramid fibers, as claimed by Du Pont. They have high mechanical and thermal properties (tenacity, strength, strength, and melting point) although they display high resistance to most chemicals [27]. Ceramic fibers are made up of different metal oxides, nitrides, and carbides and combinations of them. They are accessible in various fiber lengths and fabrics. Ceramic reinforcements used to strengthen composites can be used in applications with temperature up to 1000°C due to the high melting point of about 2000°C for ceramic fibers [9]. Natural fibers are divided into three basic types, depending on their origins. Plant filaments so-called cellulosic or lignocellulosic fibers, such as bast strands, leaf, and wood strands. Animal fibers usually consist of proteins like collagen, keratin, and fibroin, such as wool and animal hair, and mineral fibers include the asbestos bunch, fibrous brucite, and wollastonite [28]. Natural filaments are characterized by their low density, high toughness, and acceptable strength properties, and also offer significant advantages, such as biodegradability, biodegradability, renewability, and environmental friendliness [9]. Both synthetic and natural fibers have advantages and limitations for such applications. Different classification of composites including other characterizations: material source, biodegradability.

Injection, extrusion, and compression molding are the predominant manufacturing processes for PMCs. Injection molding is the most recognized and cost-effective process for producing composites on a large scale. Both thermoplastics and thermosets are exposed to injection forming. Extrusion is probably one of the most common processing methods for producing short FRPCs. Compression molding, on the other hand, is particularly suitable for the mass production of composite parts. Therefore, the sheet molding technique is another primary approach for composite manufacturing [9, 29]. Open molding, also called contact molding, including hand lay-up and spray-up, are additionally utilized to produce thermoset composites products [30]. The resin transfer molding (RTM) procedure is generally suitable for the mediumvolume production of large parts [29]. It has a place in a category that is sometimes called liquid composite molding [31]. Thermoplastic sheet forming, thermoplastic pultrusion, and tape laying are used to produce thermoplastic advanced materials, such as continuous long fiber reinforced composites. Therefore, composite liquid forming, thermoset pultrusion, fiber winding, and autoclave are the best manufacturing processes for thermoset composites [32].

The processing strategies used for the production of MMCs are mainly divided into two classes. In the liquid-state process, the pressure causes the reinforcement to infiltrate through the liquid metal. Powder metallurgy is the most popular method in solid-state processes. This technique provides the most remarkable mechanical properties of metal composites reinforced with particles [8]. The friction stir process is also a strong solid-state process, which is characterized by the fact that it can improve the properties compared to the conventional processing technique [33]. Finally, the method of high-energy ball milling was usually used for the preparation of nanocomposites [34].

The reason for this review is to provide a compilation of information from the literature on polymer and metal matrix composites. PP and aluminum alloy composite were chosen as composites for this paper. Most of the research reviewed deals with these two materials, with a few exceptions where further results are needed.

2. Polymer and metal matrix composites: case of PP and aluminum alloys-based composites

2.1 PP matrix composites

PP is a thermoplastic polymer that comes in three explicit compositions: isotactic PP (iPP), syndiotactic polypropylene (sPP), and atactic polypropylene (aPP). There are also different types of PP: homo-polymer PP (HPP) in which the PP is composed entirely of propylene monomer in semi-crystalline solid form. Random copolymer (RCP) and impact copolymer, whose PP chains contain ethylene as a co-monomer in amounts (1–8% for RCP and 45–65% for ICP) [11].

Many engineering plastics have been replaced by PP due to this they have good mechanical performance and are easy to process and recyclable, lightweight, and economically viable. For example, PP accounts for more than 40% of the total plastics in automobiles and is strengthened with various types of fibers [35]. For this reason, since its discovery, PP has been attractive to numerous researchers and modern companies to improve and enhance its properties and make it useful for additional engineering applications. K. Okuno and R. T. Woodhams [36] investigated the viscosity of the mica flake-filled PP melts and their mechanical properties. As a result, the properties of the molded composite exhibit modulus values that are higher than unfilled PP. George C. Richardson and Jhon A. Sauer [37] exanimated the possible reinforcing effects of six different types of filler particles on composites made of thermoplastic isotactic PP. They used as reinforcements the chopped strand glass fibers,

ceramic whiskers (alumina Al2O3 and silicon carbide SiC), two different glass spheres (type E with a small diameter of 10 to 40 microns and types P with a larger diameter of 75 to 110 microns), and finally carbon spheres. Their results showed that adding a higher modulus filler to a lower modulus PP matrix increases the stiffness of the composite, as well as when the fillers are glass spheres. The addition of carbon spheres to the PP matrix leads to a reduction in tensile strength and ductility. D.M. BIGG [38] investigated the electrical, thermal, and mechanical properties of PP matrix composites loaded with randomly scattered short aluminum fibers (three types with three different aspect ratios). The results showed an increase in the electrical conductivity of the composites and in the thermal conductivity, without sacrificing the advantage of low density. Shigeo Miyata et al. [39] estimated some properties of PP matrix composites enhanced by four types of $Mg(OH)_2$ magnesium hydroxide particles. By incorporating more than 57% by weight of $Mg(OH)_2$ particles, the composite becomes nonflammable, and their stiffness also improves when the crystallite size of $Mg(OH)_2$ becomes larger in the range of up to about 2 µm. Conversely, the strengths have considerably reduced.

Since glass and carbon fibers are the most widely used reinforcing materials, they possess all the advantageous properties that made the rapid development of PP matrix composites and their use in many applications possible in recent decades. J. Karger-Krocis et al. [40] studied the fatigue crack propagation (FCP) of short- (SGF) and long-glass (LGF) fibers reinforced molded PP composites on notched compact tensile specimens (CT). The FCP response of long-glass fiber reinforced PP exhibited less sensitivity to notching and fiber arrangement than that of the short fiber reinforced versions. For further, J.A.M. Ferreira et al. [41] focused on the static and fatigue behavior of PP/glass-fiber thermoplastic composites produced from a bidirectional woven cloth mixture of E-glass fibers and PP fibers, this composite was made with a fiber volume fraction of 0.338. S.-Y. Fu et al. [42] investigated the tensile properties of short-glass (SGF) and short-carbon (SCF) fibers reinforced injection molded PP composites. The addition of both glass and carbon fibers significantly enhanced the modulus and strength of the composites, with SCF/PP composites exhibiting the most remarkable strength, the modulus values of SGF/PP were the highest, and that attributed to the high strength of carbon fibers and the high stiffness of the glass fibers on the opposite side. K. Senthil Kumar et al. [43] described the development of long-glass fiber reinforced polypropylene (LFRP) composites. LFRP pallets of different sizes were produced by extrusion with a specially developed impregnation die and then injection molded into LFRP composites. Optimum content of 5 wt.% of MA-g-PP compatibilizer was added to PP-matrix in order to get better adhesion between fiber and resin. F. Rezaei et al. [44] characterized a short-carbon fiber reinforced PP (10% SCF/PP) composite as a substitute of a steel bonnet. The effects of the length and composition of fibers on mechanical properties of SCF/PP composites fabricated by compression molding technique were additionally explored. The experimental results revealed that the carbon-fiber-reinforced PP composite is a good option for replacing steel in car bonnet, with much lower weight and higher mechanical properties. P. Russo et al. [45] observed that the use of PP in combination with maleic anhydride (PP-g-MA) as a compatibilizer improved the mechanical performance of a woven glass fabric reinforced PP composites laminates. Therefore, the addition of the compatibilizer to the PP matrix improved the adhesion between glass fibers and PP matrix, which increased the ability to transfer the load from the polymer matrix to the reinforcement phase glass and carbon fibers are used as reinforcement for PP homopolymer used in structural applications. The two types of

fibers showed advantages and drawbacks in terms of their stiffness, adhesion with the matrix, and compositions. Carbon fibers are more costly and their composites are stiff but have only moderate strengths due to poor interfacial adhesion [46].

Aramid fibers are other synthetic fibers that are generally used to reinforce PP polymer. They provide very good properties for a polymeric material. They are particularly popular because of their increasing use in industrial applications and advanced technologies [47]. N.K. Cuong & Z. Maekawa [48] investigated the effects of manufacturing conditions on the mechanical properties of thermoplastic PP composites with aramid fibers. The composite laminates were prepared by varying the impregnation time and compression pressures. Tensile properties were found to enhance with increasing the impregnation time, or the compression molding pressure led to lower void content in the composites. In any case, SEM micrographs surfaces indicated weak adhesion of the aramid fibers and the PP matrix. J. Maity et al. [49] studied the incorporation of chopped Twaron filaments into PP polymer. Also, the surface modification of Twaron fibers by direct fluorination technique, and its influence on the mechanical and thermal properties of PP/Twaron fiber composites. The surface modification leads to better adhesion with the PP matrix and, thus to an enhancement in the properties of PP/Twaron fiber composites. Xiaousi Chen et al. [50] reported in detail the mechanical and thermal properties of chopped aramid fibers (Kevlar 1414) reinforced PP polymer composites. Crystallization and flammability behavior were also investigated. Tensile strength of the composites improved with the addition of aramid fibers, with maximum strength achieved at 20 wt.% aramid fibers, which present 11.8% higher than unreinforced PP. Impact strength also improved, with PP/ AF composites able to withstand 40.1 KJ/m² at 40 wt.% AF loading, 3.7 times that of PP matrix. Moreover, the PP/AF composites had superior thermal stability and char residue relative to pure PP. In addition, the AF had an effective impact on promoting the flame retardancy of the PP/AF composites. Rajat Kapoor et al. [51] investigated the dynamic compressive behavior of PP fabric composites reinforced with Kevlar fibers under compressive loading with a high strain rate. The PP/K composites laminates were manufactured by using vacuum assisted compression molding technique, and maleic anhydride grafted-PP (Mag-PP) was added to PP for enhancing the interfacial property between Kevlar fiber and PP matrix. The influence of increasing strain rate from 1370 s⁻¹ and 4264 s⁻¹ on the dynamic compressive response of PP/Kevlar laminates was evaluated in terms of peak stress, strain at peak stress, toughness, and compressive stiffness. All of these properties had increased with increasing strain rate. As result, the peak stress multiplied threefold, toughness improved tenfold, and elongation at peak stress nearly doubled. Calvin Ralph et al. [52] investigated the potential of 10, 20, and 30 wt.% short-basalt fibers (SBF) as reinforcement for the PP matrix. Mechanical testing of short-basalt-PP composites indicated that the addition of basalt fiber significantly improved the tensile and flexural properties, but in the opposite case, the impact properties decreased. In addition, these mechanical properties were strongly influenced by fiber sizing, as adhesion increased greatly, which provided the greatest improvement in tensile and flexural strength, but led to lower impact strength.

PP can be modified with mineral fillers, in order to produce a composite material with a reduced cost and enhanced thermomechanical properties study by Trotignon et al. [53] focused on the effect of the low concentration of mineral fillers on the mechanical properties of PP homopolymer. Findings showed that all fillers, regardless of their concentration, lead to an increase in the maximum stress at around 50%. elastic and yield properties of all specimens are partially not affected by the stresses.

In this work [54], nano-sized SiO₂ and ZnO nanoparticles were obtained by the solgel process, they have distinguished-mechanical properties. UV and heat ray shielding effect, also EFKA commercial dispersant and silane coupling agent were used to improve particle dispersion and particle-polymer adhesion, respectively. Haydar U. Zaman et al. [55] studied the effects of nano- and micro-ZnO concentration on the morphology, mechanical, and crystallization behavior of iPP polymer. Three compositions of iPP/mZnO and iPP/nZnO composites were made with ZnO content ranging from 2, 5, and 8 wt.%. The nanoparticles (5 wt.% nZnO) displayed good dispersion in the matrix, whereas the microparticles (5 wt.% mZnO) showed a broad size distribution, which significantly impact the mechanical properties of the composites. Similarly, the DSC measurement showed that the crystallization temperature of iPP/ nZnO was much higher than that of iPP/mZnO. F. Mirjalili et al. [56] prepared a nanocomposite containing PP and alumina nanoparticles (α -Al₂O₃) by using a Haake internal mixer and adding a dispersant agent (titanium dioxide powder TiO_2) to the PP polymer. The alumina nanoparticles with the contribution of dispersant agent enhanced the tensile and the flexural properties of the composites with the amount of 4 wt.% and 5 wt.%, respectively.

Polymer nanocomposites reinforced with carbon fillers, for example, graphene, graphite, and carbon nanotubes (CNTs), have been investigated for use as strengthening materials for various applications due to their attractive properties, including low mass density, outstanding mechanical properties, and superior electrical and thermal conductivity [57–59]. In these studies, Kyriaki Kalaitzidou et al. [60, 61] examined the possibility of using exfoliated graphite nanoplatelets xGnP, as reinforcement in PP and fabricated by melt mixing followed by injection molding. The nanocomposites xGnP-PP exhibited outstanding mechanical and thermal properties compared to the polymer matrix, with the highest value of thermal conductivity observed for 25 vol% xGnP/PP being six times that of PP. In addition, the xGnP-PP composites exhibited better performance compared to commercial carbon fiber-reinforced PP composites due to the morphological differences between exfoliated graphite nanoplatelets and carbon fibers, as xGnP reduces the CTE in two dimensions, while carbon fibers only in the direction parallel to its axis [61]. Sok Won Kim [62] proved that the addition of low concentrations of multi-walled carbon nanotubes to the PP matrix improved the thermal conductivity and the thermal diffusivity of the polymer. The addition of 2 wt.% MWCNTs resulted in a doubling of the thermal conductivity. In addition, the thermal conductivity can be raised threefold by surface treatment of the nanotubes. Rumiana Kotsilkova et al. [63] have prepared by an extrusion process nanocomposites containing reinforcement of some weight fractions of 0.1 to 3 wt.% MWCNTs and isotactic PP (iPP) matrix. Thermogravimetric analyses (TGA) performed on the nanocomposites showed better thermal stability compared to the matrix polymer. The contribution of MWCNTs to the overall modules of the nanocomposites was highlighted. Dynamic mechanical tests demonstrated the improvement of the storage modulus with increasing MWCNTs content was demonstrated, as well as during tensile tests, the young modulus showed a maximum improvement of about 23% compared to the iPP-matrix. Young Soo Yun et al. [64] found that the morphological differences between the alkylated graphene oxides (AGOs) and the one-dimension alkylated carbon nanotubes (ACNTs) produced a different reinforcement effect in the composites. AGO fillers have been conducted to better thermal conductivity, and high performance in thermal degradation temperatures compared to ACNT fillers. Furthermore, AGO was beneficial for Young's modulus, only 0.1 wt.% AGO was required to increase it by more than 70%. However, the tensile strength and

elongation were reduced relative to the PP polymer. On the other hand, 0.5 wt.% of ACNTs was sufficient to increase the elastic modulus and tensile strength by 37% and 20%, respectively. The interfacial interaction between the fillers and the matrix also contributed to these different reinforcing effects, with AGO and PP having a greater interaction than ACNT and the PP matrix. Chien-Lin Huang et al. [65] also noted a decrease in tensile strength and maximum elongation with an improved elastic modulus of the carbon filler reinforced conductive composite. In terms of mechanical properties of the PP matrix, 20 wt.% graphene nanosheets (GNs) and carbon fibers (CFs) reinforced conductive composites showed a decrease in tensile strength by 34% and 8%, respectively, while they increased tensile strength by 150% and 158%, respectively. Moreover, GNs proved a significant influence on the crystallinity temperatures and electrical conductivity of the PP matrix as CF. Moustafa Mahmoud Yousry Zaghloul et al. [66] found a significant increase in the flexural strength and modulus of PP matrix reinforced with two different carbon fillers, namely multiwalled carbon nanotubes (PP-MWCNT) and synthetic graphite (PP-G), with more significant improvement was noticed for CNTs. In particular, the flexural modulus was 49% and 74% better at 2 wt.% and 10 wt.% loading than that of PP-G composites, respectively. A similar trend was observed in the fracture toughness tests, the inclusion of carbon fillers resulted in an impressive enhancement in fracture toughness, with the maximum load supported by MWCNT-reinforced composites being 38% higher than that of synthetic graphite-reinforced composites. Bartolomeo Coppola et al. [67] Illustrated the potential use of carbon nanotube reinforced PP nanocomposites (PP/ CNTs) as strain-gauge sensors for structural monitoring. PP/CNTs nanocomposites with 5 and 7 wt.% of CNTs have shown attractive sensing properties in terms of gauge factor (GF). Moreover, their excellent mechanical and electrical properties are made from the appropriate substitutes for sensing materials. Since their ease of preparation and flexibility, they can be easily used in several application areas.

In recent years, with the demand for the utilization of renewable sources and sustainable materials, natural reinforcements have gained the attention of many researchers and scientists thanks to their biodegradability characteristics. P. Bataille et al. [68] studied the effects of surface pretreatment of alpha-cellulosic hardwood pulp fibers with a weighted-average fiber length of about 240 micrometers, as well as processing time and temperature, on the mechanical properties of the cellulosecontaining PP. In order to modify the interfacial interaction between the PP matrix and cellulose fiber the silane-coupling agents and maleic anhydride modified PP were used. X. L. Xie et al. [69] investigated the structural properties, as well as the mechanical behavior of injection molded composites of isotactic polypropylene (iPP) and 0, 10, 20, and 30 wt.% of chopped sisal fiber (SF) with an average length of 10 mm. The thermal stability of PP/SF composites has shown that they could be processed by injection molded in the same way as for PP homopolymers. Moreover, the stiffness of the PP/SF composites was improved by the fibers, but their tensile strength decreased due to the poor interfacial bonding between PP and sisal fibers. Byoung-Ho Lee et al. [70] fabricated bio-composites from long and discontinuous of two kinds of natural fibers (kenaf and jute fibers) and PP matrix. It was found that an optimum nominal fiber fraction of 30% by weight of kenaf fiber reinforced composites, gives the highest tensile and flexural modulus, while for the jute fiber reinforced PP composites, fiber content of 40% by weight seemed to be the optimum value. This limitation of fiber content was attributed to the void content in the bio-composites, which may be caused by nonuniform packing or lack of PP fibers-matrix. The shortness of both natural and PP fibers may help the incorporation of more natural fibers

into the biocomposites. N.A.M. Aridi et al. [71] proved that the addition of rice husk fillers to a PP polymer improved the mechanical strength and stiffness of rice husk/ PP composites. In addition, a struktol coupling agent was added to the composites to facilitate the PP fiber interaction. The 50 wt.% filler-loaded composites exhibited the maximum tensile strength, flexural strength, and flexural modulus, on the other hand, 35 wt.% of filler loading was found to be the highest elastic modulus, flexural strength, flexural modulus, and impact strength. Ngo Dinh Vu et al. [72] investigated the thermal, mechanical, and biodegradability properties of cellulose fiber (CF) reinforced PP green composites. Cellulose fibers were extracted from rice straw and prepared by the melting method. The poor thermal properties of cellulose fibers result in reduced thermal stability of PP/CFs composites with different loading levels of CFs. The strength of PP/CFs composites was also decreased, whereas their stiffness modulus was increased with the increasing CFs content. The maximum strain of pure PP showed 57.5%, but it significantly decreased with CFs loading, probably related to the weak interactions between PP-matrix and CFs, which generates stress concentration points and agglomerations. Nitish Kumar et al. [73] used animal fiber (hair) as reinforcing material and PP as the resin. The percentage of horsehair (HH) varied from 0, 10%, 20%, and 30%, where the fiber length was fixed, and the composite specimens were prepared using the film stacking method. The specimens of 20% horsehair and 80% PP (by weight) showed the best composition in mechanical characterization. Numerous other natural fiber-reinforced PP composites have been investigated, notably: Abaca fiber [74, 75], flax fiber [76], hemp fiber [77], oil palm fiber [78], banana fiber [79], kenaf fiber [80], ramie fiber [81], sisal fiber [82], cotton stalk fibers [83], coconut coir fiber [84], kapok husk [85], wood fiber [86], jute fiber [87], rice husk [71], wheat straw fiber [88], bagasse fiber [89], and pineapple leaf fiber [90].

Hybridization is an important direction for future research, which is obtained by combining two or more different types of fibers in a single matrix, hybrid composites have a balance between inherent advantages and drawbacks. Several studies have already been carried out in this regard, Himani Joshi and J. Purnima [91] developed chopped E-glass fibers and wollastonite mineral fiber reinforced PP hybrid composites (PP/GF/W), prepared by extrusion compounding and injection molding techniques. A positive hybrid effect was found for ultimate strength, while a negative hybrid effect was noticed for tensile modulus. Meanwhile, with an increase in the volume fraction of wollastonite fibers, the failure strain of the hybrid composites increased slightly. Hybridization with small amounts of mineral fibers allows these glass fiber composites to be employed in more engineering applications. R.V. Sheril et al. [92] improved the properties of homopolymer PP, by reinforcing it with two different combinations of mineral fillers, namely talc with silica (T/SI), and talc with $CaCO_3$ (T/CC). As a result, the hybrid composite CC/T showed a superior Young's modulus, whereas tensile strength was nearly the same compared to SI/T. In addition, the thermal stability of SI/T was better compared to CC/T. In terms of flammability rate, the SI/T composite showed a lower burning rate than CC/T, which indicates that the SI/T composite was less flammable. N. Gamze Karsli et al. [93] investigated the hybrid reinforcement effects of surface treated or untreated carbon nanotubes (CNTs) (0, 1, and 4 wt.%)/glass fiber (GF) (0, 30 wt.%) on the morphology, mechanical and electrical properties of PP-matrix composites prepared by means of extrusion and injection molding techniques. The hybrid-reinforced composites exhibited higher stiffness and strength values when compared with only CNTs or GF reinforced composites. In addition, the electrical resistivity measurements of the

hybrid composites decreased as the amount of nanotubes varied from 1 wt.% to 4 wt.%. The combination of glass fibers and carbon nanotubes enhances the reinforcing ability of nanotubes in polymer composites, because of the improved polarity of the system. Alok Agrawal and Alok Satapathy [94] developed hybrid composites of homopolymer PP as a matrix reinforced with aluminum nitride (AlN) microfillers and solid glass microspheres (SGMs) with an average size of $90-100 \mu m$, for microelectronics applications. The thermal conductivity of the combination PP/20 wt.% AlN/10 wt.% SGM showed the highest values and maximum value of the dielectric constant. These results highlight the potential application of this new class of polymer composites in future electronic packaging materials. Maliha Rahman et al. [95] evaluated the change in mechanical properties of PP by reinforcing it with two natural fibers, such as chopped pineapple leaf and banana fiber. The hybrid composites reinforced with discontinuous and random fibers were prepared using the compression molding technique, and the ratios of pineapple leaf and banana fiber varied of 3:1, 1:1, and 1:3. In addition, a chemical treatment with 5% sodium hydroxide was performed for both pineapple leaf and banana fiber. The combination of 5 wt.% hybrid fibers with a banana ratio of 3:1 gave the best mechanical properties of the PP-based composites. Rupam Gogoi et al. [96] developed high specific strength PP composites using short-carbon fibers (SCF) and hollow glass microspheres (HGM). The hybrid composite with 10 wt.% HGM and 8 wt.% SCF showed an improvement in mechanical strength (up by~110 and~112% over pure PP). However, the addition of just 5 wt.% of SCF and HGM reduced the impact strength by b~78%–80% of the samples compared to unfilled PP, which could be described as the poor fiber-matrix and filler-matrix interfacial adhesion that may have caused an ineffective load transfer. Mariana Desiree Reale Batista et al. [97] developed hybrid composites combining two types of cellulose fibers, long- and short-glass fibers (LGD and SGF) or talc in a homopolymeric PP matrix. All the composites were prepared by injection molding under the same processing conditions, with the total fiber mass content fixed at 30 wt.%, with the cellulose concentration varying gradually from 0 wt.% up to 30 wt.%. Significant improvements in the mechanical, thermal, and morphological properties of the hybrid composites were observed, which the LGF/cellulose A composites exhibited the best mechanical properties, while (SCF/Mica)/cellulose B, talc/cellulose B exhibited superior thermal properties. At the same time, these properties decreased with increasing cellulose content. However, an optimal amount of cellulose fiber has sufficient properties that could reduce or replace some of the inorganic reinforcements in various applications, such as automotive applications, contributing to weight and cost savings, and contributing to the sustainability of the composites. Various hybrid reinforcements have been investigated and studied, such as glass and carbon [98], boron nitride – nano-hydroxypatite [99], MgO - lignin fillers [100], kevlar basalt [101, 102], clay - graphite [103], zironica - MWCNTs [104], kenaf - carbon nanotubes [105], banana - coir [106], sisal - carbon/glass [107], bagasse - CaCO₃ [108], glass - bamboo [109], coir - betel nut fiber [110], basalt - wood [111], glass stainless steel [112], graphene - graphite [113].

2.2 Aluminum alloy matrix composites

Aluminum alloys have been the most popular and attractive choice for the automotive and aerospace industries because they possess excellent characteristics, such as lightweight, high-strength, flame retardant properties, good corrosion resistance, easy mass production, and low cost. However, they have poor high-temperature

performance and wear resistance. Al alloys of 2000 series, 5000 series, 6000 series, and 7000 series are heavily utilizable [114]. For this purpose, aluminum metal matrix composites (AMMCs) are usually developed by adding a ceramic reinforcing phase to the alloy matrix. The required properties are achieved by the high-strength ceramic particles, whiskers, or fibers uniformly distributed in the aluminum alloy-matrix. Aluminum alloys have become the most employed matrix by companies for the fabrication of metal-matrix composites [115]. The use of ceramic whiskers enables us to take advantage of the mechanical performance of ceramics, as they provided the largest increases in strength and stiffness available, as well as creep resistance. However, the difficulty in obtaining a uniform dispersion in the matrix, processing costs, and high whisker prices, make them less recommended at the current time [116]. Aluminum matrix composites (AMCs) reinforced with ceramic particles have quickly emerged as a promising material, in particular, due to their superior tribological properties and corrosion resistance behavior. Moreover, the low price and ease of manufacture of particulate reinforcement composites should be noted, while they can manufacture by the traditional methods of mechanical mixing of the particles with the matrix material [117, 118]. Carbides in the form of whiskers and particulates, such as silicon carbide (SiC) and boron carbide (B_4C), have been widely used to strengthen aluminum metal matrix, which are important structural ceramics due to their high hardness and thermal stability [119]. T. G. Nieh et al. [120] investigated the mechanical behavior of discontinuous, whiskers, and particulate SiC reinforced aluminum composites were manufactured by powder metallurgy techniques. At elevated temperature, all composites showed creep behavior that was significantly different from that of the neat matrix. They exhibited more creep resistance, with some improvement at 20 vol.% SiCw-6061Al, due to the higher creep resistance property of the whiskers. D. P. H. Hasselman et al. [121] pointed out that the thermal conductivity of a 40 vol.% SiC particulate reinforced aluminum matrix is highly dependent on the mean size of the SiC particulates ranging from 0.7 to 28 μm. They have found a decrease in thermal conductivity with decreasing SiC particle size since as the particle size decreases and the total interfacial area increases, the relative contribution of the SiC to the total conductivity of the composite decreases. N. Chawla et al. [122] characterized the mechanical behavior and microstructure of 20 vol.% SiC particulate reinforced 2080 aluminum matrix composites processed by a novel, low-cost sinter-forging technique. The microstructure of the sinter-forged composites showed relatively uniform dispersion of the SiC particles. Moreover, the sintered composites showed higher elastic modulus and tensile strength of 103 GPa and 434 MPa, respectively. The fatigue behavior of sinter-forged composites was also determined. A.A. El-Daly et al. [34] studied the mechanical properties and hardness of SiC nanoparticle reinforced aluminum matrix composites by employing the pulse-echo overlap (PEO) method as a nondestructive technique. 99.7% pure Al powder was reinforced with 2.5, 7.5, and 12.5 % by volume of nano-SiC particulates with an average size of 70 nm and fabricated by employing high-energy ball milling with cold-pressing and sintering technology at 550°C for 1 hour. As a result, a great improvement was shown with 12.5 vol.% nano-SiC particulates, elastic modulus increased by about 38%, and shear modulus by about 25% compared to the matrix material. Also, the hardness improved from 3.8 to 7.1 GPa, while Poisson's ratio was retained from 0.296 to 0.237. These improvements were essentially due to the stronger interfacial bonding. Qiyao Hu et al. [123] fabricated A356-SiC and 6061-SiC composites using the vacuum-assisted high pressure die casting (HPDC) process. A356 and 6061 aluminum alloys were reinforced with 10 vol.% SiC particles with angular

morphology. A uniform distribution of SiC particles was achieved in the composites with decreases in porosities to very low values of 1.4% for A356-SiC and 1.8% for 6061-SiC composites and good interfacial bonding. As a result, the HDPC composites exhibited significant mechanical properties, such as hardness, tensile strength, and elongation. Yuming Xie et al. [124] found that the aluminum matrix composites containing 5 wt.% of silicon carbides (SiC) and fabricated by deformation-driven metallurgy (DDM) were not only cost-effective but also process high mechanical performance, with the elastic modulus and hardness of the composites improved by 202% and 251% of the performance of pure aluminum, respectively. Jinkwan Jung and Shinhoo Kang [125] prepared a boron carbide-aluminum (B_4C -Al) composites was indicated after the addition of titanium metal and titanium-based compounds to B_4C to enhance the wettability of the molten-matrix on the boron carbide skeletons. Heat treatment of the boron carbide skeleton prior to infiltration in the temperature range of 1000–1400°C was also considered.

R.M. Mohanty et al. [126] indicated that increasing the percentage until 25 wt.% of B₄C particulate addition to the Al 1100 metal matrix resulted in a decrease in interfacial strength due to the weakening of the grain boundary of the composites when sintered at 837 K below the melting point of Al in a vacuum. In turn, the modulus and hardness improved with the increase of reinforcement from 0 wt.% to 25 wt.%, from 22 GPa to 183 GPa, and from 50 to 550 Hv5, respectively, indicating the high dependence of the modulus on the weight percentage of boron carbide particulates. E. Mohammad Sharifi et al. [127] enhanced the hardness, compressive, and wear properties of high purity aluminum by adding different amounts (5, 10, and 15 wt.%) of B_4C nanoparticles with an average size between 10 and 60 nm. With increasing content of B₄C nanoparticulates, the sample containing 15 wt.% showed the highest compressive strength of 485 MPa compared to pure Al (130 MPa), the same improvement in hardness, which increased from 33 HV for pure Al to 164 HV for the composite. The wear resistance is well known for a significant increase. K. Shirvanimoghaddam et al. [128] investigated the manufacturing of aluminum-boron carbide composites using the stir casting method. They found that changing the processing parameters (e.g., temperature, surface treatment of particles, stirring speed, and stirring time) and the volume fraction of reinforcements affected the microstructure and mechanical properties of the composites. Increasing the volume fraction of B₄C from 0 to 20 vol.%, increasing processing temperature from 800°C to 1000°C, and increasing stirring times to 12 min enhanced the mechanical properties and wettability potential of aluminum and B₄C particulates.

Titanium carbide (TiC) reinforced aluminum alloy composites are furthermore being more researched, [129, 130].

3. Conclusion

Composite materials are advanced designing materials. Thanks to the proper selection of matrix reinforcement, easy access to these constituents, their lightweight, and their exceptional physical, thermal, electrical, and mechanical properties, composite materials are becoming an alternative to conventional materials and are used in various applications. This review article provided a general overview of composite materials, explicitly PP polymer matrix composites and aluminum alloy metal matrix composites. A number of studies have been discussed in order to illustrate

the different reinforcing materials, material processing, and the different properties, such as physical, mechanical, electrical, thermal, and tribological. Various types of reinforcements were discussed in detail, along with their reinforcing effect on PP and aluminum alloy matrices. Carbon, glass, and aramid were the most commonly used synthetic reinforcing materials as they impart excellent modulus, strength, and fatigue resistance to the PP polymer. Mineral fillers and ceramic whiskers, such as mica and carbides, have been shown to improve the thermomechanical properties of the resulting polymer composites, with cost saving. Oxide fillers, including alumina, zirconium, and magnesium oxide, were investigated for their improved tribological properties and impact resistance. Graphite, graphene, and carbon nanotubes were reported to provide exceptional mechanical properties and higher electrical and thermal conductivity for the conductive composites. Natural reinforcing materials and industrial waste particles have in turn been studied by numerous researchers to benefit from their biodegradability and environmental friendliness. Some hybrid composite materials are a reviewer for both PP and aluminum alloys, weighing the benefits and drawbacks of the various hybrid reinforcement. These standards provide researchers and users of such materials with a structural guide for proper material preparation and testing to ensure their quality and accuracy. These mechanical properties are highly dependent on a variety of factors and parameters, including material constituents, manufacturing processes, testing parameters, and environmental conditions. As follows:

- In most studies, increasing the reinforcement content was found to improve the mechanical properties, as well as the fatigue strength of both PP- and aluminum alloy-based composites. However, mechanical properties tend to decrease above a certain particle content, due to the agglomeration of these particles.
- A study has found that long-fiber reinforced PP composites have better mechanical properties compared to short-fiber reinforced PP composites. On the other hand, nanoparticles were found to strengthen aluminum alloys more compared to microparticles, resulting in more cycles to failure.
- Since most composites are anisotropic, they exhibit differences in their mechanical properties depending on their orientation with respect to the direction of loading. The mechanical properties of fiber-reinforced composites are highly influenced by the number of fibers aligned with the loading direction.
- Many reinforcing materials are not compatible with the matrix, resulting in poor interfacial adhesion. Modifications, such as treating the fibers or adding a coupling agent, to the polymer can help improve adhesion between the reinforcement and matrix phases of a composite material. In the case of an alloy matrix reinforced composite, preheating or coating of ceramic particles can help.
- Moisture and temperature are the two major environmental factors affecting the fatigue behavior of composites. High moisture or high environmental conditions have been indicated to reduce fatigue life.

Author details

Mohamed Chairi¹, Jalal El Bahaoui^{1*}, Issam Hanafi², Francisco Mata Cabrera³ and Guido Di Bella⁴

1 Faculty of Science Tetouan, Department of Physics, Abdelmalek Essaâdi University, Tetouan, Morocco

2 Ecole National des Sciences Appliquées d'Al Hoceima (ENSAH), Abdelmalek Essaâdi University, Ajdir, Al Hoceima, Morocco

3 Escuela de Ingeniería Minera e Industrial de Almadén, Universidad de Castilla-La Mancha, Almadén, España

4 Department of Engineering, University of Messina, Messina, Italy

*Address all correspondence to: jelbahaoui@uae.ac.ma

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Chapter 3

Delamination and Tensile Effect of Fine z-Binder Reinforced on Fiberglass/Polyester Composite for Aerospace Applications

Citlalli Gaona-Tiburcio, Manuel Alejandro Lira Martinez, Marianggy Gomez-Avila, Jesús M. Jaquez-Muñoz, Miguel Angel Baltazar-Zamora, Laura Landa-Ruiz, Demetrio Nieves-Mendoza, Francisco Estupiñan-López and Facundo Almeraya-Calderón

Abstract

Delamination propagation in laminated composite materials is a common issue that always concerns us when we consider composites for structural purpose. Many possible solutions have been studied; the most famous is the three-dimensional (3D) woven composites materials, which have promising interlaminar fracture resistance but at the cost of increasing density, which for aerospace industry is very important. In this chapter, mode 1 double cantilever beam (DCB) interlaminar fracture toughness tests according to the American Society for Testing and Materials (ASTM) D5528 standard were performed on composite specimens made of E-Glass Saertex 830 g/ m^2 Biaxial (+/-45°) with Sypol 8086 CCP polyester resin with orthogonal z-axis oriented yarn woven of 0.22 mm diameter nylon monofilament. Four specimens were made with a longitudinal distance between the warp binders of 0.5, 1, 1.5, and 2 cm, respectively. A tensile test according to the ASTM D3039 standard was performed to study how z-binder may affect tensile resistance. The results show a considerable increase in interlaminar fracture toughness, several stress concentrators have been created because of the new yarn and premature failure in the matrix.

Keywords: composite, delamination, laminated, tensile, interlaminar

1. Introduction

A composite material, from now on just "composites," consists of 3 components known as reinforcement, matrix, and the interface between whose properties and performance are designed so that together they generate material with superior properties to its parts acting independently. In general, the "Reinforcement" is a very rigid and strong material with a distributed phase, whose function is to support the external forces in its longitudinal direction.

At the same time, the "matrix" is a material with a continuous phase, weaker, and less rigid, but more tenacious and often more chemically inert, which is responsible for keeping the reinforcement attached to the final shape of the mold in which it is located, protecting the reinforcement against external agents, supporting indirect stresses along the length of the reinforcement and helping to distribute external mechanical energy to the reinforcement, so that it be distributed and supported by the latter. The "interface" is created as the chemical interaction and bonding between reinforcement and matrix. From here, the composites are classified due to (a) the shape of the reinforcement, such as continuous or discontinuous fibers, particles, etc., and (b) the nature of the matrix, which can be a metallic, ceramic, or polymeric matrix. When composites are manufactured as a set of layers or laminates, it is simply known as laminate composites [1].

The use of composites in the aerospace industry has grown rapidly from the mid-1990s to the present, so now, it is one of the most used materials in commercial aircraft. This interest and investment in composites are because, in many cases, their properties are generally better than those of metals, especially aluminum alloys, such as lower density, better mechanical properties, they do not corrode, and with a proper design, manufacturing costs can be reduced by reducing solid joint elements such as rivets [2].

Unfortunately, laminate composites have several disadvantages over metals. One of the most important issues is delamination, a critical failure mechanism caused by high interlaminar stresses coupled with typically very low through-thickness strength. Delamination arises because fibers lying in the plane of a laminate do not provide through-thickness reinforcement, so the composite transfers most of the external load into the matrix, which usually is quite brittle and very susceptible to shear stress, so that the laminate composite starts to separate between laminates, as illustrated in **Figure 1** [3].

Specifically speaking of fiberglass/polyester composites, there are some works that report studies on their manufacture, composition, and properties, but also another more specific that analyzes thermal properties, crack growth behavior, fracture energy, the effect of mode of loading, and delamination resistance.



Figure 1. *Composite delamination.*

Bagherpour [4] published a book in which he analyzes the mechanical relationships in fiber-reinforced polyester composites. He explains its tensile, bending, and toughness properties.

Gupta [5] investigated the water absorption and thermal and mechanical properties of glass fiber reinforced polymeric composites, finding that all of them increase with the increasing number of glass fiber layers. Ganjiani [6] in 2021 considered the effect of mode II delamination and the corresponding interlaminar crack propagation during the drilling process of multilayer glass/polyester composites, using numerical and experimental approaches; and found that more than 95% of the crack propagation could be attributed to mode II under the conditions studied.

Triki [7] in his research on the influence of the fabric structure on the crack growth behavior of glass/polyester composite laminates, using fracture toughness tests, was able to determine that the balanced interface is more resistant to delamination than the balanced interface. Unbalanced interface.

Sham [8] published a review in which she highlights aspects of interlaminar and intralaminar fracture toughness studies of polymeric matrix composites and lists the different ways to assess fracture energy.

Khoramshahi [9] investigated both experimentally and numerically, the effect of mixed-mode loading on the fracture parameters of glass-reinforced polyester composite specimens, and according to the measured fracture toughness, they found the energy release rates of critical interlaminar deformation in opening and shear mode. Furthermore, her results indicated that the interlaminar cracked sample is stronger under shear loading conditions and weaker under tensile loading conditions.

More recently, Suriani [10] published a review in which she compiles other works on delamination and some common types of manufacturing defects, thereby illustrating the impact on mechanical properties and proposing alternative solutions.

Various investigations have been done on delamination due to through-thickness stresses [11–21]. However, it has been widely neglected that delamination also has a crucial role in determining in-plane strength, often leading to premature failure initiation. In-plane failure of composites is driven by the energy released when the fibers are discharged. This can occur in two ways: by fracture of the fibers or by delamination and cracks in the matrix that come together to produce a fracture in the surface without breaking the fibers. Delamination also plays a critical role in the behavior of composites under the impact, affecting both the damage caused under impact load and the subsequent response in compression after the impact [3].

Currently, four types of delamination are defined as fracture mechanisms classified by modes, and mode 1 is the one studied in this chapter, in which the normal stresses are produced perpendicular to the interlaminar crack, and the surfaces of the separate crack one from the other without there being any relative slippage of them; it is shown in **Figure 1** [22]. There are some interesting investigations about the mode-I delamination process, the effect of the reinforcement, and the Z-fiber or Z-pinned laminates, mainly using carbon fiber or epoxy [23, 24].

Many applications in aircraft parts are exposed to out-of-plane loading conditions that make it impossible to turn laminated composites into a proper material. Wind turbine blades, aircraft spars, stiffeners, and pressure vessels are examples of applications where out-of-plane loading conditions are imposed on the structure. Therefore, a delamination solution has arisen as a composite with improved full-thickness "out-of-plane" properties, known as a 3D woven composite [25].

Conventionally the reinforcement used for laminated composites is a 2-dimensional (2D) fabric; therefore, it is impossible to protect the matrix from trough thickness stresses. The three-dimensional (3D) reinforcement for composites contains a fabric that covers the 3 dimensions (length, width, and thickness) and is a technology that is having great success in the aerospace industry, such as the very know composite blades from LEAP turbofan, which is a generation of the CFM56 Aerojet produced by CFM (Snecma and GE) and SAFRAN [26].

This chapter is the characterization of stress fracture and Mode 1 delamination in laminated composites reinforced in cross section with a 3D fabric whose objective is the application in aeronautical structures. The transverse reinforcement was made using an industrial sewing machine as part of an external company project, and a 0.5 mm diameter thread was used. The study variable in the reinforcement was the space between reinforcement and longitudinal reinforcement to the composite, having reinforcements with a distance of 2, 1.5, 1, and 0.5 cm, respectively. The reinforced composite was manufactured, and a tensile test was carried out according to the ASTMD3039 standard [27]. Mode 1 of delamination analysis was carried out according to the ASTM D5528 standard to characterize tensile strength and its resistance to delamination [28]. Results were compared with z-axis binder and without binder, and the failures were characterized by fractographic analysis using a stereoscope. The purpose of the separation between the reinforcement is to find a possible variation in the resistance to delamination of the material since the more intertwined between the warp and weft, the greater the probability of generating stress concentrators and, therefore, reducing its interlaminar resistance. This new material is meant to be used in aircraft's control surfaces, several sources [29–32] report various issues regarding delamination on such surfaces, which is one of the most predominant uses of this kind of material.

2. Experimental procedure

The specimens were performed according to ASTM D5528 standard by resin transfer molding (RTM) process with E-Glass Saertex 830 g/m² Biaxial (+/-45°) throughthe-thickness (TTT) binder by an orthogonal weave pattern of 0.3 mm diameter Barkley FBA BGQS15–15 nylon monofilament and polyester resin Composite Envisions 1179. Four types of samples were performed with the longitudinal distance between binders of 0.5, 1, 1.5, and 2 cm, respectively, as shown in **Figure 2** and, from now on. These samples will be defined as:



Figure 2. Different samples with the longitudinal distance between binders.

- A: Binder distance of 0.5 cm.
- B: Binder distance of 1 cm.
- C: Binder distance of 1.5 cm.
- D: Binder distance of 2 cm.
- ST: Sample with no binder.

10 specimens from each sample were made and were identified by consecutive numbers, for instance, specimen A1, A2, ... A10 for sample A; B1, B2, ... B10 for samples B, but different issues were presented during tests and in the results section only is shown the results of the most relevan specimens.

The DCB tests per the ASTM D5528 standard were performed on A Sintech 20 D tensile testing machine with a constant crosshead speed equal to 5 mm/min equipped with 50 kN load cell. All test load-displacement data were recorded by Sintech software. To measure all propagation of delamination values, all tests were filmed with a Samsung S20 Ultra camera with a resolution of 4 K/120 fps and processed in Tracker software.

3. Results and discussion

3.1 Delamination test

Figure 3 shows the sequence of a bending mechanism pattern that all specimens with binder had because of the elasticity of the z-axis yarn, improving the toughness of the composite and preventing the delamination from propagating. A "lever" was generated between the force applied and the z-axis binder until the binder fractured.



Figure 3. Sequence of DCB test.

it repeats the same lever mechanism from the next closest binder in a staggered manner until (1) the piano hinge came off, (2) the tensile machine stopped, or (3) the specimen fractured causing this bending a relatively high displacement between layers δ and consequently a destructive tear for the composite.

Figure 4 shows the load-displacement curve resulting from the delamination test, and **Figure 5** shows the R-curve of most representative specimens. Specimen ST6 shows a conventional pattern for delamination composites, with pronounced noise on the Y-axis because the values were too small for the testing machine. The linear pattern indicates detachment of the fibers within the composite due to a premature



Figure 4. *Load-displacement curve from DCB tests.*



Figure 5. *R curves from DCB tests.*



Figure 6. *Pull-out z-binder and fiber detachment.*

failure in the matrix. The visible delamination (VIS) starts very early with a very minimal delamination length of "a." In the curve, 2 peaks stand out that visibly coincide with the delamination from the insert and its propagation.

Specimens A, B, C, and D had a behavior very different from conventional delamination. The beginning of delamination from the "VIS" point of specimens A6, A7, B6, B7, and D6 is highlighted by a very pronounced first peak caused by the rupture of the closest z- binder from the insert. The load decrease while increasing displacement as delamination propagates until it reaches the next z-binder. It will increase the load required to keep delamination crack growing and so on. The nonlinear zones in the curves lie in the plasticity of the z-binder trying to maintain the layers together, and the peaks above represent pull-out fracture, that is, the z-binders fractures plastically and they are pulled out from the matrix. Both fracture mechanisms are illustrated in Figure 6. The C6 curve, which also had a premature detachment of one of the piano hinges, shows the same pattern as the previous specimens with a z-binder. The pronounced peaks belong to the ductile fracture of the z-binder and, therefore, pullout. Figure 5 shows an intermediate GI value between specimens A and B. Specimen D6 had a similar performance to specimens A6 and A7, with the exception that D6 has fewer peaks due to the distance between the z-binder of 0.5 vs. 2 cm. Both maximum peaks of specimens A and D are close to 20 MPa and due to the greater number of peaks, specimen D has a smaller area under the curve and, therefore, a lower toughness modulus.

Specimens A6 and A7 were the ones that exhibited the highest toughness and maximum stresses in the DCB test specimens B6 and B7 had a premature failure in the joint of the piano hinges. However, **Figure 5** illustrated how the fiver detachment mechanism started with a linear pattern in the curve. The stresses supported were also higher than ST6. **Figures 5** B6 and B7 show that the specimens with z-binder, they had the lowest values of GI toughness. However, higher than that of the specimen without a z-binder.

Figure 7 shows all stress-displacement curves in a single plot, where the difference between them can be seen: The most predominant specimens were A7 and D7 because no issues were found in the tests. Of these 2 specimens, both had maximum stress of 18.74 MPa, but specimen A7 had a higher modulus of toughness than specimen D7, 1.035 J/mm3 vs. 0.885 J/mm3, due to a greater number of peaks that are generated



Figure 7. Load-displacement for all specimens from DCB test.

by the z-binder since it has a smaller distance between them. However, specimen D7 showed a greater displacement than specimen A7 due to the fact the z-binders blocked the latter. Despite all the drawbacks, all the samples with z-binders presented better results than those without them, which is barely visible in yellow in **Figure 7** due to their low interlaminar resistance. The instability presented in all the specimens with z-binders falls on the binder itself, where the most witnessed peaks are due to the resistance generated by them, and the pronounced valleys followed by these peaks are the rupture of the z-binders due to ductile fracture.

Table 1 shows the results from load-displacement curves from DCB tests. The specimens that registered a higher resistance against delamination were A7 and D4, with a value of 18.74 MPa ST6 was the one that registered the lowest maximum stress with 0.7 MPa. B and C show intermediate resistance values. Specimen D4 had the largest shell displacement, which for this type of test is a very large value compared to the displacement of 2.42 mm/mm that specimen ST6 had, which is a

Specimens	σ Max	δ@σMax	δ Max	Т	Failure note
_	(Mpa)	(mm)	(mm)	J/ mm ³	
ST6	0.7	1.51	2.42	0.00093	Complete delamination
A6	17.11	166.18	168.68	0.651	Specimen fractures
A7	18.74	158.76	213	1.035	Specimen fractures
B6	2.44	59.1	123.92	0.193	Piano hinge failure
B7	8.15	172.44	172.44	0.308	Tensile machine stop
C6	9.77	182.9	213.66	0.839	Piano hinge failure
D6	18.74	298.52	302.28	0.885	Specimen fractures

Table 1.Load-displacement values from DCB test.



Figure 8. *Curve R for all specimens from the DCB test.*

normal value [33–35]. A7 had the highest tenacity modulus, followed by specimens D6 and C6, with values of 1.035, 0.885, and 0.839 J/mm³, respectively.

Figure 8 shows all R-curves from the DCB test, where A6, A7, and D6 stand out. ST6 (at the bottom of the plot) shows the most delamination width because it was the only specimen to delaminate completely. Despite this, the illustrated toughness is very low compared to the other specimens.

In **Table 2**, the critical values of GI toughness are shown. The high values for A6 and D6 are due to the initial "peak" that was generated to break the first z-binder, and the very low value of ST6 was because delamination was progressive. There was no reinforcement on the interlaminar plane.

3.2 Tensile test

Figure 9 shows the stress-displacement curves of the tensile tests. The linear and semi-linear zones shown in the curves are repeated in these tests, and the mechanism

Specimens	Р	δ	a	GIMax
	(N)	(mm)	(mm)	(J/m ²)
A6	180	152.08	0.68	2367.01
A7	300	137.58	3.2139	758.4
B6	60	44.04	0.88	175.94
B7	60	44.24	0.88	176.49
C6	40	3.5	1.1	7.69
D6	420	17.11	1.93	2733.83
ST6	3.1179	0.0021	0.72	0.00004



Figure 9. Stress-displacement curves from tensile tests.

Specimens	UTS	Ε	σ _y (2%)	ε@σ _y mm/	U _R MJ/	т ј/	ϵ Max mm/	SE
	Мра	GPa	MPa	mm	m³	mm'	mm	%
А	215.79	0.96	198.95	0.25	22.46	0.061	0.48	1.92
В	192.6	0.82	180.77	0.29	22.05	0.05	0.48	3.12
С	189.37	0.72	165.48	0.27	20.13	0.045	0.44	1.34
D	178.58	0.61	157.85	0.3	21.84	0.049	0.49	4.96
ST	209.63	0.85	208.89	0.26	25.97	0.035	0.37	0.8

Table 3.

Values obtain from tensile test.

was the same as in the delamination tests. Linear zones are because of matrix cracking within the composite, and nonlinear zones because of the fibers' pull-out mechanism and the elasticity of z-binders within the composite. In **Table 3** the mechanical properties of the tensile tests are listed. Sample A has the maximum value of the UTS close to ST. Then the UTS value decreases because z-binders tend to restrain the fibers making the composite weaker, the distance between z-binders is even stronger at some point (sample A), the lesser the distance. The modulus of toughness increases on the samples with z-binder, mainly because the z-binder makes the composite absorb more energy than sample ST.

3.3 Fracture analysis

Due to the complexity of the different failure mechanisms, the specimens presented in the delamination test, the analysis was divided into 3 zones, as shown in **Figure 10**. Zone A is located in the interlaminar fracture zone where delamination occurs, Zone B is the flexural zone of the composite previously explained, and Zone C is the zone of collateral damage, where residual stresses are generated due to Zone B.



Figure 10. Scheme of failure mechanism zones in DCB test.

3.3.1 Zone A

Interlaminar fracture failure in this zone results from the composite insert according to ASTM D5528 and shows similar fracture patterns in all tests with z-binder. The ST specimens, shown in **Figure 11**, were completely delaminated. It shows an interlaminar brittle fracture, where islands of exposed fiber were found without matrix, since in some areas the matrix fractured, leaving exposed fibers. No fiber fracture was found. In



Figure 11. Fracture mechanism in ST specimens.

Figure 11c some "whitish" areas were generated because of fibers' detachment within the composite, due to internal matrix fracture, which are presented in all 3 zones.

In the case of specimens A, B, C, and D, there were various additional mechanisms to those that occurred in the ST specimen regarding delamination, as shown in **Figure 12**. Due to its nature, The z-binder shows a ductile pull-out fracture, and the filament is made of nylon. However, close to z-binders, no damage to the fibers was found. There are also fibers exposed because of brittle matrix failure. Something very relevant is shown in **Figure 12c**, where the z-binder shows a cavity in the form of residual stress concentrators that will affect the material resistance in a direction perpendicular to the fibers. A "fiber bridging" effect from z-binders was responsible for the improvement of interlaminar fracture toughness for Zone B.

3.3.2 Zone B

This area was the most affected due to the z-binder. **Figure 13** shows as the z-binder prevent the propagation of the interlaminar fracture due to its elasticity. The composite reaction in the test was to bend the upper layer, conglomerate the fibers and the z-binders in the flexing zone, and generate a hardening in the specimens as z-binders fracture progressively across the composite generating a greater flexing and at the same time a lock in the plane. These bending fractures the matrix, detaching



Figure 12. Zone A, fracture mechanism in z-binder specimens.



Figure 13. *Zone B fracture mechanism in z-binder specimens.*

the fiber as Zone A. The fracture mechanism in the detached fibers is generated since z-binders generate "knots" within the fiber, limiting the pull-out effect, hardening and embrittling the material to a point where the fibers begin to fracture due to the flexural stress generated. Additionally, delamination was found as a collateral effect due to the bending in the upper layer of the composite (where the stress is applied).

3.3.3 Zone C

As shown in **Figure 14**, this zone is the result of the propagation of the stresses generated in Zone B, distributing energy in the fibers and z-binders, generating stress concentrators. Here prevail the "whitish" zones previously explained, where a superficial fracture of the matrix was found in some zones. This zone is generated as collateral damage from Zone B since the energy absorbed is too much for the matrix. Z-binders knots were also creating the whitish zones. **Figure 14c** and **d** show that there is interlaminar damage.



Figure 14. *Zone C fracture mechanism in z-binder specimens.*

3.3.4 Tensile test fracture

Figure 15 illustrates the tensile test fracture of one sample specimen, which was angled fracture (Angle- Gauge- Middle, AGM, according to ASTM D3039 standard) with "pull-out" failure. **Figure 16** shows that all specimens had delamination. The fracture mechanisms on all z-binder specimens are shown in **Figures 17** and **18**, the layers were displaced by the tensile force and "pull-out" failure, damaging the z-binder, which were tied to the layers themselves, generating stress concentrators, fractures in the matrix and delamination. In samples "A" and "B" multiple fractures on the fibers were found because z-binders prevent their displacement and, therefore, fracture. In samples "C" and "D", the composite damage due to the z-binder is less, reducing the fracture in the fibers and the matrix in the region close to the failure.

Z-binder on 3D composites may generate collateral damage reducing mechanical properties such as compression [36–43] and/or flexural resistance [44–51]. This is mainly because such composites' manufacturing may generate resin-rich zones that can fracture prematurely and start different fracture mechanisms that depend on z-binder configuration and nature. Even though, due to their promising properties, 3D composites are of great interest for replacing metals applications, with different advantages such as corrosion resistance [52–56]. This motivates the generation of novel manufacturing processes such as the one presented in this study, a zigzag-oriented z-binder [57], or even a 2.5D pattern composite [58].



Figure 15.

Fractures of the samples tested by tension: a) ST, b) A, c), B, d) C, e) D.



Figure 16. Fractures in cross section of the samples tested by tension: a) ST, b) A, c), B, d) C, e) D.



Figure 17.

Fracture mechanisms in tension test.



Figure 18. Tensile fracture morphology.

4. Conclusions

Four fiberglass/polyester composite samples with different thin nylon z- binder reinforce lengths were tested. All samples showed an increase in mode 1 interlaminar fracture toughness over the same composite without a z-binder, but a new fracture mechanism also appears because of the elasticity of the z-binder and brittle matrix. Tensile tests were also made, and results show that tensile resistance in samples B, C, and D been compromised. Sample A had a small increase in tensile resistance, showing that the lesser the length distance between z-binder, the better tensile resistance.

All these results showed promising values. More studies need to be made on z-binder length distance smaller than 0.5 mm since Sample A had the best properties overall. Also, a matrix with superior mechanical properties than the z-binder reinforcement is recommended, since the new fracture mechanism was created because the matrix was weaker than the z-binder. With this, better values shall result.

Nomenclature

Applied load (N)
Opening Mode I interlaminar fracture toughness (J/m ²)
Interlaminar delamination length (mm)
Point at which delamination is observed visually on specimen edge
Load point deflection, mm
width of DCB specimen, mm (25.4 mm)
Delamination length, mm
Ultimate tensile strength (MPa)
Modulus of elasticity (GPa)
Yield strength at 2% (MPa)
Displacement at yield strength (mm/mm)
Modulus of resilience (MJ/m ³)
Modulus of toughness (J/mm ³)
Standard error of the mean (%)

Author details

Citlalli Gaona-Tiburcio^{1*}, Alejandro Lira-Martínez², Marianggy Gomez-Avila², Jesús M. Jaquez-Muñoz¹, Miguel Angel Baltazar-Zamora³, Laura Landa-Ruiz³, Demetrio Nieves-Mendoza³, Francisco Estupiñan-López¹ and Facundo Almeraya-Calderón¹

1 Universidad Autónoma de Nuevo León, UANL, Facultad de Ingeniería Mecánica y Eléctrica, FIME, Centro de Investigación e Innovación en Ingeniería Aeronáutica, CIIIA, Aeropuerto Internacional del Norte, Nuevo León, México

2 Universidad Autónoma de Ciudad Juárez. Instituto de Ingeniería y Tecnología, Chihuahua. México

3 Facultad de Ingeniería Civil-Xalapa, Universidad Veracruzana, Veracruz, Mexico

*Address all correspondence to: citlalli.gaona@gmail.com

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Chapter 4

Experimental and Numerical Evaluation of Tensile Strength of Horse Hair-Glass Fiber/Epoxy Hybrid Composites

Mamaru Wutabachew

Abstract

Natural fibers are environmentally friendly with less weight and energy conservation than the synthetic fibers. The present study assessed on mechanical properties of the hybrid composite which is composed of horsehair and glass fiber materials using experimental methods and Digimat simulation. The impact of weight percentage and orientation of fiber on mechanical properties such as tensile strength is investigated. The total weight of the fiber was 70% and the matrix was 30%. The specimen was prepared using the method of hand layup process. The weight percentage increment was 20% and the orientation of the fiber was $0-90^{\circ}$ and $0-90^{\circ}$. By doing Digimat simulation and testing the composite; it was rich that there is a basic effect of weight percentage and orientation in mechanical property of composite. With a different weight percentage of fiber, the maximum tensile strength was 282.97Mpa in the experiment result and 290 Mpa Digimat simulation result which is 56% of glass fiber plus 14% of horse hair fiber plus 30% of epoxy resin. Whereas the orientation of fiber 0⁰ has the maximum tensile strength which is 127.2 Mpa in the experiment result and 150.12Mpa in the Digimat simulation result. The test outcomes are drawn and the conclusions were made to compare the result of horsehair/epoxy, glass fiber/epoxy and hybrid composite with software simulation. Further investigation is recommended to test with other animal hairs.

Keywords: tensile strength, horse hair-glass fiber composites, epoxy composites

1. Introduction

The availability and distribution of horses and its hair in Ethiopia within the region have been written clearly. Ethiopia is endowed with abundant agricultural resources. The statistics show there are more than 2 million horse's lives and it is about 33.5% of the African and 3.45% of the world horse population. The population of the horse in Ethiopia ranked the first in Africa and 8th in the world. According to the livestock census of Ethiopia, the distribution of horses in the Oromia region ranked the first and highest population of the horse is found. Around 1,176,301 horses which

is 58% of the country has to live in this regime. The second-ranked region in horse population is the south nation and nationality which is 451,799 horses around 22.27% of the population of the country horse. The third one is the Amhara region, which is 396,231 around 19% of the horse population of the country. The other regions and the Tigray region contain 0.73% [1]. The horsehair is not sold in some regions, but in the Amhara region, the horsehair is sold. Commonly, the horse hair is bought from the market from the Amhara region particularly Engibara town in Awi zone.

Due to increasing the need for composite material, the development of natural fiber composite is a new topic in recent research and technology. For increasing environmental awareness, many researchers shift their research in natural fiber composite material. Natural fibers are a capability that replaces synthetic materials and their related products. Natural fibers are less weight and energy conservation applications compared with synthetic fibers. This is mainly due to their advantages compared to synthetic fibers, because of Low cost, low weight, abundant and renewable resources [2–4]. Due to this reason, this paper came up with a natural resource that is available in our environment.

As the horse hair is naturally gifted fiber and there is no extraction process, the present study focused on horsehair and glass fiber with epoxy resin hybrid composite materials mechanical properties to replace conventional material. Moreover, in this study, the mechanical properties such as the tensile strength of glass fiber and horsehair reinforced polymer hybrid composite material with different orientation and weight percentage of fiber was assessed.

2. Materials and methods

Materials used in this study include commercially available epoxy resin used as a matrix and the reinforcement materials used are commercially available horsehair/glass fiber Raw material suppliers from the market.

2.1 Description of the materials

2.1.1 Horsehair

The availability of horse hair in Ethiopia is written in the introduction part but the horse hair is bought from the market from the Amhara region Engibara town in the Awl zone. The horsehair fiber found in the animal horse. The specific strength and stiffness of horsehair have compared to those of copper wire with the same diameter [5].

2.1.2 E: Glass fiber

E–Glass fiber is one of the most important artificial classes of reinforcement material specially used in polymer composites. Glass fiber has a low thermal coefficient, low dielectric coefficient, and high electrical resistance. This property depends on additives and curing agents [6]. It is obtained from Dejen Aviation Industry which is located at the city Bishfotu in Oromia region.

2.1.3 Hardener

The hardener is used as a binder during the production. Araldite HY951 hardener is used in this study. It has low viscosity, cure at room temperature, good mechanical

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strength, and Good resistance to atmospheric and chemical degradation. The epoxy resin is obtained from Kadisco Paint Factory.

2.1.4 Remover

Wax is used to safely remove the prepared spacemen from the mold. The remover is available in the market. It is bought from the market.

2.1.5 Epoxy resin

One of the properties of epoxy resin has good additive properties. The additive property is along with its high mechanical strength, low shrinkage, chemically resistant high diffusion density, low viscous and better electric insulation capacity. It is easily reinforced with natural (horsehair) and E glass fibers. LY-501 type of epoxy is used in this study [7]. The epoxy resin is obtained from Kadisco Paint Factory which is found in Addis Ababa lafto sub city around wuha limit.

3. Procedures for preparation of laminate experimental spacemen

Each composite laminate was prepared from the mixture of Epoxy, horse hair fiber, and E-glass fibers. The horsehair used in this procedure is untreated and free from chemicals. Then an open mold of aluminum plate with a dimension of 300 × 300 mm was prepared and the prepared composite was cut for each test. Using the rule of mixtures, the various fiber weight proportions are calculated to achieve laminates with 0:100, 100:0, 80:20, 20:80, 60:40 and 40:60 ratios with former being the ratio of Fiber and the latter is horse hair. The horse hair and E- Glass fibers with the total weight of the composite fiber contained 70%. The composite material had six layers and the orientation of fiber was [0], [90], [0/90]. Laminates were composed of plates of different layer materials or layers of fiber-reinforced lamina prepared with the same matrix material.

After this the above process, horse hair and glass fiber were cut based on the length of the mold. Based on the calculation of weight proportion appropriate amount of horse hair fibers, glass fiber and epoxy resin are considered. Then the first layer was placed in the mold based on the calculated amount of fiber. Next epoxy resin LY501 and the hardener HY-951 were mixed with a ratio of 2:1 before preparing the first layered fibers and apply a mixture of epoxy resin and hardener on the first layer fiber. Unidirectional E-glass fiber had prepared into required mold size and positioned over other fibers. Again, the calculated amount of epoxy resin and hardener mixture was applied over E-glass fiber. The second layer is prepared with horse hair fibers placed over a prepared E glass fiber and again a mixture of epoxy resin and hardener was applied. The resin mixture was spread by hand layup method uniformly around the corners. After spreading resin mixture Deadweight was applied over the open mold to remove air. Then after some time, the laminate was removed from the open mold and put in suitable temperature for curing. Finally, the composite was dried for seven days and the specimen was prepared by cutting the plat with the required dimension of each test with a grinder [8].

3.1 Tensile test

According to American society of teeth manufacturing (ASTM–D 3039) standard tensile test on composite specimens where the young modulus of elasticity of glass fiber and horsehair hybrid reinforced polymer was done to observe the behavior of

hybrid material under load. The test specimen in tensile testing or tension testing was a fundamental material science test in which a test specimen is subjected to uniaxial tension until specimen failure. The results of the test were commonly used to select a material for quality control and application.

They also used to predict how a material will react under other types of forces. The material properties such as ultimate tensile strength, maximum elongation and reduction in the area are measured in a tensile test. The properties like Young's modulus and yield strength are also determined from these measurements. The aim of this photo is to show what type of testing machine i used (**Figure 1**).

3.2 Specimen size

The most commonly used specimens for American society of teeth manufacturing (ASTM 3039) were constant rectangular cross-Section 25 mm (1 in) wide, 250 mm (10 in) long and 4 mm thickness [9]. Optionally tabs were used to bonded the ends of the specimen to prevent gripping damage. For each test, a composite of three specimens were tested and the average value of each test was taken for analysis (**Figure 2**).





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Figure 2.

Hybrid spacemen sample for tensile test (photo taken by Mamaru Wutabachew, 2019).

4. Result and dissection

4.1 Validation of software and experimental result

The **Figures 3** and **4** shows the stress–strain diagram of hybrid composite with different weight percentage and hybrid composite with different orientation of fiber respectively. Both experimental and software results of the composite materials were plotted. As shown in each figure there was a deviation of result in experiment and software, but the variation of strain is nearly the same. In the graph of the software, the tensile strength is linearly increased throughout the strain. This is because the material property fill-in the software is a liner, elastic and isotropic property.







different orintation composite experiintal and software compartion

Figure 4.

Experimental and software result of composite with orientation.

But in the experiment, the graph was not linear, because the material property was a lot of losses like specimen preparation accuracy, temperature variation, testing machine adjustment; due to this reason the graph was sometimes liner other time, non-liner. The tensile strength of experimental and software results approaches each other when the load approaches maximum. Therefore, the maximum average tensile strength was taken for each composite material and the deviation of experimental and a software result was calculated. The result of each deviation (error) indicated in Table 1. The table is also showing the experimental and software result of the maximum tensile strength of 7 different composite materials.

4.2 Hybrid composite with a different weight percentage

No.	Tensile strength (Mpa)	Experimental	Software	Error
1	0-degree hybrid	127.2	150.12	0.1527
2	90-degree hybrid	4.833	9.565	0.2020
3	0–90-degree hybrid	89.5	111.33	0.1960
4	14% glass hybrids	48.87	49.758	0.0178
5	28% glass hybrids	93.7	102.95	0.0898
6	42% glass hybrids	151.733	181.41	0.1636
7	56% glass hybrids	282.97	290	0.0173

Figure 3 shows the stress-strain diagram of glass fiber and horsehair fiber with epoxy resin hybrid composite. The result shows four different weight percentages

The table shows that the deviation or error of the software and experimental result is calculated by the software result minus experimental result divided by software result.

Table 1.

Seven different composite tensile strengths with experimental and software results.

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of each fiber but it has the same orientation of fiber. Starting from the initial point to some amount of strain the stress–strain graph is far apart but, the load reaches to maximum the maximum tensile stress of the materials both experimental and software results approach each other. The graph shows that experimental and software deviation of the above **Table 1**.

4.3 Hybrid composite with orientation

The **Figure 4** shows the stress–strain diagram of glass fiber and horsehair with epoxy resin hybrid composite with different orientation of fiber. The weight percentage of the fibers was equal. The result also shows the maximum tensile strength of experimental result approaches to the software result when the increase.

5. Conclusion

The investigation of glass fiber and horsehair fiber hybrid composite leads to the following conclusions. For tensile test; 70% glass fiber/epoxy composite has the average tensile strength is 220.8Mpa but, the hybridization of 56% glass fiber and 14%horse hair the tensile strength is 282.97Mpa which is enhancing the tensile strength approximately 21.97%. In the direction of fiber orientation 0⁰ has maximum tensile strength but for other direction the tensile strength is reduced. In hybridization of horse hair and glass fiber, incasing weight proportion of glass fiber and decreasing weight proportion of horse hair fiber enhancing the tensile strength of the composite.

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Author details

Mamaru Wutabachew Department of Mechanical and Industrial Engineering, Debre Markos Institute of Technology, Debre Markos, Ethiopia

Address all correspondence to: mamaru.wutabachew@dmu.edu.et

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Chapter 5

Fibre-Reinforced Polymer (FRP) in Civil Engineering

Jawed Qureshi

Abstract

Construction produces a third of global carbon emissions. These emissions cause global warming and contribute to climate emergency. There is a need to encourage use of sustainable and eco-friendly materials to effectively deal with climate emergency. Fibre-reinforced polymer (FRP) is an eco-friendly material with low-carbon footprint. FRP composites in civil engineering are mainly used in three applications: (1) FRP profiles in new-build; (2) FRP-reinforcing bar in concrete members and (3) FRP in repair and rehabilitation of existing structures. This chapter presents basic properties of constituent materials (fibres and polymer resins), mechanical properties of FRP bars, strengthening systems and profiles, manufacturing processes and civil engineering applications of FRP composites. Durability, sustainability and recycling of FRP composites are also discussed.

Keywords: FRP structures, FRP in buildings and bridges, FRP in structural engineering, resins and fibres, sustainability of FRP, durability of FRP, recycling of FRP

1. Introduction

Buildings and construction sector produces 39% of global carbon emissions [1–5]. Construction uses a wide variety of materials, ranging from cement to clay, wood to steel and aluminium to glass. Traditional construction materials, such as reinforced concrete, steel, masonry and timber, have a long track record of proven strength and reliability. The construction guidelines and design standards are also well established for these materials. However, these conventional materials have limitations as well. Steel can corrode; concrete and masonry are weak in tension; and timber can shrink and rot. The conventional materials are usually energy-intensive to produce. To reduce carbon emissions and protect and restore the natural environment, there is need to develop and invest in new sustainable construction technologies and materials. Fibre-reinforced polymer (FRP) composite is such an eco-friendly material with lower ecological impact than the usual construction materials [6–9]. Use of FRPs in new-build and repair of existing structures has been increasing over past few decades [10]. There are three main FRP shapes in civil engineering: (1) all-FRP profiles for new-build; (2) FRP-reinforcing bars in concrete members; and (3) FRP sheets for repair of existing structures.

Fibre-reinforced polymer (FRP) composites have been used in various civil engineering applications, buildings and bridges included, for over five decades. Their use in aerospace, marine and automotive industries even goes back to 1930s. FRPs also have their applications in sports and rail sector and wind turbines [7, 8]. For structural use, FRP composites are usually made by embedding fibres in a polymer matrix. The matrix consists of polyester, vinylester, or epoxy resins and fibres include glass, carbon, or aramid fibres. The resin binds the fibres together, while fibres provide strength and stiffness to the finished FRP product. The main aim is to produce a lightweight strong and stiff component [11].

FRP composites have desirable properties for use in structural engineering. Lightweight, chemical and corrosion resistance, low ecological footprint, fast deployment, electromagnetic transparency and thermal insulation of glass FRPs, and high strengthto-weight ratio, offsite fabrication and modular construction, superior durability and mouldability are some of the main benefits of FRP for structural use [12]. FRP composites are versatile and customisable. The ability to mould into complex shapes creates new aesthetic possibilities and provides geometrically efficient design solutions [12, 13]. Some FRPs using aramid have high impact resistance and are often used in bulletproof vests, helmets, and automotive crash attenuators [7, 8, 14]. But structural use of FRP with aramid fibres is limited. FRP composite material is not an ideal material though. Like classical structural materials, FRPs have shortcomings too. The notable weakness is the brittle nature of the FRP material. It is linear elastic up to failure. FRPs fail in a sudden brittle manner without giving warning. However, in a real world, FRP components are never loaded to failure. They are normally loaded up to a third of their failure load. Anisotropy and low transverse properties of FRPs are few other drawbacks. Lack of ductility and limited knowledge about fire and durability performances and no agreed design codes for FRP structures are some of the main setbacks hindering wider acceptance of this material.

FRP composites are suitable in structural applications where challenging environmental conditions exist and fast installation is needed. Due to their chemical, corrosion and environmental resistances, FRPs perform better in harsh environments compared with the traditional materials. Besides use in repair market, and as rebars in concrete members, full FRP profiles are used in chemical and food processing plants, wastewater treatment plants, cooling towers, foot and road bridges, bridges decks and edge elements, and railway platforms as primary structural elements. FRP elements are also used in secondary structures, such as insulated ladders, floor gratings, stairways with handrails, working platforms and walkways, and building façade panels [1, 7].

This chapter is organised into six sections. First section gives the context and background to use of FRP material in civil engineering applications. Constituent materials and manufacturing processes of FRP products are presented in Section 2. Input materials, such as fibres and polymer resins, are discussed in the section. FRP manufacturing methods including automatic and manual processes are also explained in Section 2. Section 3 is focused on applications of FRP material in civil engineering. Three main applications include FRP profiles, rebars and strengthening systems. Section 4 relates to durability aspects of FRP composites. Various environmental factors, structural health monitoring and field evaluation of FRP materials and structures are described in the section. Section 5 is about sustainability of FRP composites. Lifespan of FRP composites, including extraction and production of FRP material, manufacturing, use and end-of-life disposal are discussed in this section. Section 5 also expands on recycling methods of FRP, such as incineration, thermal, chemical and

mechanical recycling. Finally, Section 6 highlights the key conclusions of the work presented in the chapter.

2. Constituent materials and manufacturing processes

Composite materials are formed by combining two or more materials to represent the best properties of the constituent materials. The resulting composite material accounts for weaknesses of the individual materials and leads to strong and stiff structural components. Constituent materials and manufacturing processes of FRPreinforcing bars, structural profiles and strengthening sheets are described in this section.

2.1 Materials

FRP composites consist of the fibres embedded in a polymer matrix. Fibres provide strength and stiffness. The matrix serves as a glue that ensures transfer of forces among the fibres, the applied loads and the composite component [7].

2.1.1 Fibres

Typical fibres used in strengthening and new-build applications are glass, carbon and aramid. These are man-made synthetic fibres [1]. More recently, the research focus has moved to sustainable composites with natural fibres, such as basalt fibres [15]. Typical mechanical properties of various fibres are listed in **Table 1**. The strength and modulus in this table are for plain fibres; the values for manufactured FRP composites, such as pultruded profiles, bars and sheets, will be considerably lower than the plain fibres. All fibres have linear elastic stress–strain response with no yielding [16].

Glass fibres are the most commonly used fibres in structural composites. They are used in structural profiles, reinforcing bars and strengthening applications. Glass fibres are available in four different grades: E-glass (electrical glass), A-glass (window glass), C-glass (corrosion resistant, also known as AR-glass or alkali-resistant glass) and S-glass (structural or high-strength glass). E-glass is the most popular one due to its relatively low cost and electrical insulation properties. S-glass has higher tensile strength and modulus than E-glass. S-glass is normally used in aerospace industry due to its high strength [1, 7, 8, 14, 18]. S-glass is almost four times more expensive than Eglass [1]. Except AR-glass, all other glass types are prone to alkaline attack. Glass fibres are non-conductive to electricity and can be easily used near electrified railway lines, communication facilities and power lines [19]. Glass fibres are commercially available as unidirectional rovings, as shown in **Figure 1(a)**.

Carbon fibres are the strongest of all fibres. They are used for strengthening applications, such as CFRP strips, sheets, rebars and prestressing tendons. Carbon fibres possess high tensile strength and modulus, high fatigue and creep resistances, and superior chemical resistance [7]. Due to these properties, carbon fibres are highly resistant to aggressive environments. The key disadvantages of carbon fibres are their high cost, thermal conductivity and anisotropy. Carbon fibres are 10–30 times more expensive than E-glass fibres [1, 16, 19]. As carbon fibres are conductive to electricity, they should be electrically isolated from any steel parts. Usually, the resin provides the electrical insulation, but glass fibres should be used instead in conductive

Material		Grade	Density (g/cm ³)	Tensile modulus (GPa)	Tensile strength (MPa)	Max elongation (%)	Fibre architecture	Glass transition temperature (°C)
Fibre	Glass	Е	2.57	72.5	3400	2.5	Isotropic	
		А	2.46	73.0	2760	2.5		
		C	2.46	74.0	2350	2.5		
		S	2.47	88.0	4600	3.0		
	Carbon	Standard	1.70	250.0	3700	1.2	Anisotropic	
		High strength	1.80	250.0	4800	1.4		
		High modulus	1.90	500.0	3000	0.5		
		Ultrahigh modulus	2.10	800.0	2400	0.2		
	Aramid	I	1.40	70.0–190.0	2800–4100	2.0–2.4	Anisotropic	1
	Basalt	I	2.6–2.8	90–110	4100–4800	3.2	Anisotropic	I
Polymer	Polyester		1.20	4.0	65	2.5	Ι	70–120
resin	Epoxy	I	1.20	3.0	06	8	Ι	100–270
	Vinylester		1.12	3.5	82	9		102–150
	Phenolic		1.24	2.5	40	1.8	Ι	260
	Polyurethane		varies	2.9	71	5.9		135–140 [17]

 Table 1.

 Properties of plain fibres and thermosetting polymer resins [1, 7, 8, 16].

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Figure 1.

Different fibre system for pultrusion (adapted from Bank [8]): (a) glass roving on a spool; (b) E-glass continuous filament mat (CFM) or continuous strand mat (CSM); (c) woven glass fabric; (d) stitched glass fabric; (e) carbon fibre tows; (f) polyester veil.

environments [16]. Carbon fibres come in long and continuous tows, containing bundles of 1000 to 16,000 parallel filaments [19], as shown in **Figure 1(e)**. Carbon fibres have four different strength grades: standard modulus (SM), intermediate modulus (IM), high strength (HS) and ultrahigh modulus (UHM). Glass and carbon fibres are not sensitive to the ultraviolet (UV) light [16].

Though not very common in structural engineering applications, aramid or Kevlar fibres are still used in FRP rebars and prestressing tendons. Aramid fibres have their compressive strength 20% less than the tensile strength. Their behaviour is linear elastic and brittle under tension, and non-linear and ductile under compression. They exhibit large plasticity in compression when subjected to bending. This behaviour increases the impact resistance of aramid fibres [19, 20]. Due to high energy absorption and toughness resistance, aramid fibres are used in bullet-proof vests and helmets [14]. Aramid fibres are affected by UV light; they change colour under UV and the strength is reduced. Aramid fibres are resistant to most chemical attacks, except few acids and alkalis. They can crack at high moisture content [16, 19]. Relatively low compressive strength (500–1000 MPa), sensitivity to UV light and tendency to stress rupture make aramid fibres less suitable for structural applications [7]. However, AFRP bars are sometime preferred over CFRP-reinforcing bars in high alkaline environments due to their relatively lower cost [21].

Basalt fibres are single-component materials produced by melting crushed volcanic lava deposits. Basalt is a natural material found in these volcanic rocks. Basalt rock is abundant; about 33% of Earth's crust is basalt. The manufacturing process of basalt fibres is similar to glass fibres, but with no additives. This makes basalt fibres less expensive than glass or carbon fibres. Basalt fibres have similar mechanical properties as glass fibres. The benefits of basalt fibres include heat and fire resistance, excellent thermal and acoustic insulation, cheaper cost than carbon and glass fibres, resistance to UV, chemicals and moisture, excellent dielectric insulation and excellent temperature resistance from -260°C to 700°C. Research in structural use of basalt fibres is still at very early stages. Experimental studies are available on other natural fibres, such as hemp, sisal, flax and bamboo fibres. But commercial FRP products using these fibres are not available yet [7, 15, 19, 21, 22]. With more focus on climate emergency and global warming, the key drivers for future FRP composites will be sustainability, recycling and reuse, and eco-friendliness of materials. Possible replacement of synthetic fibres with natural fibres is reviewed in a recent paper [23]. Fibres are used in various forms [14]:

- Rovings—parallel bundles of continuous untwisted filaments (Figure 1(a))
- Yarn—bundles of twisted filaments
- Fibre mats with chopped or continuous fibres (Figure 1(b))
- Woven and non-woven fabrics (Figure 1(c))
- Stitched fabrics, grid, mesh and fleece (Figure 1(d))
- Carbon fibre tows (Figure 1(e))

2.1.2 Resins

Matrix, or simply polymer or resin are different names for polymer resins. Resins bind the fibres together. It is a non-fibrous part of the FRP composite [8]. The resin serves many functions: it protects fibres from environmental degradation (moisture) and mechanical abrasion, keeps the fibres in position within the composite component, transfers load between fibres and prevents fibres from buckling in compression. The matrix constitutes 30–60% by volume of a FRP composite system [7, 19]. Resins are of two types—thermosetting and thermoplastic resins. These resins are different based on how the polymer chains are connected. Material properties of thermosetting resins are given in **Table 1**. The glass transition temperature (T_{g} , °C) of a polymer resin is the temperature at which an amorphous polymer moves from a hard or glassy state to a softer, often rubbery, or viscous or sticky state. The glass transition temperature of the unidirectional FRP composite component is usually taken equal to the glass transition temperature of the resin matrix [8].

In thermosetting resins or polymers, molecular chains are cross-linked and have strong bonds. This means once the thermosetting polymer is set after curing, it cannot be remoulded to a different shape. Excellent binding properties and low viscosity (flowy nature) of thermoset resins allow easy placement of fibres within the FRP composite system. Thermoset resins include polyester, epoxy, vinylester, phenolic and polyurethane. Conversely, thermoplastic resins are mouldable due to weak molecular bonds. Their molecular chains are not cross-linked too. They can be reshaped, repeatedly softened and hardened by temperature cycles above their forming temperature. They remain plastic and do not set. They can also be recycled and reprocessed. Due to high viscosity (gluey nature) and poor adhesion properties, it is hard to impregnate fibres in thermoplastic resins. This increases the manufacturing cost of FRP composites. There are four types of thermoplastic matrices: polypropylene, polyamide, polyethylene and polybutylene. Their strength and stiffness are lower than the thermosetting resins. Thermoplastic resins are used in aerospace engineering. Their use in structural engineering applications is rare. Most FRP products in civil engineering applications use thermoelastic resins [1, 7, 8, 19]. Most resins are

susceptible to UV light. Special additives and surface fleece/veil are needed for their protection. Resins are isotropic non-linear viscoelastic materials [7, 14].

FRP profiles and bars mainly use polyester and vinylester resins. Almost 75% of FRP products use polyester resins [7]. Polyester resin is less expensive compared to vinylester. Identical FRP structural profiles using both polyester and vinylester resins are produced [8] by many manufacturers [24–26]. FRP reinforcement bars utilise vinylester resin due to its corrosion resistance and durability performance. Phenolic resins have excellent fire resistance and are the oldest resins. They cost the same as polyester resins. However, their use in structural FRP products is scarce due to difficulty in reinforcing and curing them. They are only used in walkway gratings and strengthening strips for timber structural components [8]. Polyurethane resin matrix characterises high toughness. When used with glass fibres, it can produce high tensile and impact resistant FRP part. The cost of polyurethane is similar to the vinylester resin [1].

2.1.3 Additives and fillers

FRP structural products contain more ingredients than simply fibres and resins. Fillers are added to the polymer resin to reduce the cost of FRP products and improve some properties. Filler content varies from 10% to 30% of the resin weight. Fillers increase the hardness, creep, fatigue and chemical resistances of FRP composites. They also reduce the shrinkage cracks and improve the fire behaviour of FRP parts. Additives are also added to the resin system to improve certain properties. Additive content is usually less than 1% of the resin weight. Resins contain various additives, such as catalysts, accelerators, hardeners, curing agents, pigments, ultraviolet stabilisers, fire retardants and mould release agents. Additives and fillers alter the physical and mechanical properties of FRP components [7, 8].

2.2 Manufacturing processes

FRP products, such as rebars, strips and profiles, are produced using two methods: automatic process (pultrusion) and manual process (hand or wet layup). FRP rebars, strips and profiles use pultrusion. While, hand layup is used for FRP sheets for onsite strengthening of existing structures [7, 8]. There are other specialised methods, such as filament winding, centrifugation, resin transfer moulding (RTM), resin infusion moulding (RIM) and vacuum-assisted resin transfer moulding (VARTM). FRP tubular sections and piles are made through filament winding method. FRP decks and components are produced by RTM, RIM and VARTM methods. More recently, 3D-printed continuous FRP composites have also been produced; further details can be found in [27, 28].

2.2.1 Pultrusion

Pultrusion is an automatic process of producing constant cross-sectional FRP profiles, rebars and strips. Open sections, like wide-flanged sections, closed tubular sections and multicellular profiles can be produced using pultrusion. The part has to be straight; curved section cannot be pultruded [8, 29, 30]. Schematic diagram of pultrusion process including different stages of pultrusion is shown in **Figure 2**. Pultrusion machines have fibre and matrix units. The fibre unit contains fibre bundles, mats and surfaces veils. Roving is the term used for glass fibre bundles and tows



Figure 2.

Schematic diagram of pultrusion process (courtesy of Strongwell [24]).

for carbon fibre bundles. The unidirectional rovings or tows provide strength and stiffness in the longitudinal direction. While the continuous filament or strand mat (CFM or CSM) and stitched or woven fabric provide strength in the transverse direction. Surface veils are also used for UV and corrosion protection. Pultruded parts are produced by impregnating dry fibres with resin and guiding them through a heated die (mould) and allowing them to cure. The cured material is then pulled through the die to give it the desired tensile strength. The part is cut at the end of the die to the required length [7, 8, 31, 32]. The pultruded products including FRP profiles, rebars, plates and strips are shown in **Figure 3**.

Pultruded FRP parts mainly use glass and carbon fibres in structural engineering applications. Glass fibres are more common due to their low cost. Use of aramid fibres is limited in pultrusion. Carbon fibres are used in FRP strengthening strips because of their high modulus. A pultruded FRP profile has a middle layer with unidirectional rovings and two outer layers with continuous filament mat (CFM)/chopped strand mat (CSM) or woven fabrics. Polyester surface veils are also added to outer layers for UV and corrosion protection. FRP profiles have 35–50% fibre volume, while FRP bars and strips have 50–60% fibre volume of the total volume [1, 8, 31, 32, 34]. The mechanical properties of typical FRP profiles are shown in **Table 2**. Comparison of steel and FRP bars in terms of tensile properties are given in **Table 3**. The properties of



Figure 3.

Pultruded FRP shapes, rebars and strips: (a) FRP structural profiles or shapes [24]; (b) FRP-reinforcing bars [33]; (c) FRP plates and strips [19].

Estimated fibre volume		25–40%	
Fibre architecture	Roving and mat		
Strength (MPa)	Tensile	Longitudinal	207–317
		Transverse	48-83
	Compressive	Longitudinal	207–359
		Transverse	110–138
	Shear	In-plane	31–48
		Out-of-plane	27–31
	Flexural	Longitudinal	207–338
		Transverse	69–131
	Bearing	Longitudinal	207–269
		Transverse	179–234
Modulus (GPa)	Tensile	Longitudinal	18–28
		Transverse	6–10
	Compressive	Longitudinal	18–26
		Transverse	7–13
	Shear	In-plane	3.0-3.4
	Flexural	Longitudinal	11–14
		Transverse	6–12
Poisson's ratio	Longitudinal		0.33-0.25

Table 2.

Mechanical properties of pultruded FRP wide-flanged profiles (glass-reinforced vinylester shapes 6–13 mm thick) [2, 6, 7].

Property	Material			
	Steel	GFRP	CFRP	AFRP
Density (kg/m ³)	7850	1750–2180	1430–1670	1300–1450
Longitudinal modulus (GPa)	200	35–60	100–580	40 -125
Longitudinal tensile strength (MPa)	450–700	450–1600	600–3500	1000–2500
Ultimate tensile strain (%)	5–20	1.2–3.7	0.5–1.7	1.9–4.4

Table 3.

Comparison of tensile properties of steel and FRP rebars (with volume fraction of fibres ranging from 50 to 75%) [19].

commercially produced FRP strengthening strips using glass and carbon fibres are shown **Table 4**.

2.2.2 Wet or hand layup

Wet or hand layup is a manual method for producing FRP strengthening sheets and fabrics. Typical properties of commercially produced FRP sheets and fabric are

	Standard-modulus carbon-reinf. Epoxy strip	High-modulus carbon-reinf. Epoxy strip	Glass-reinf. Epoxy strip	Carbon-reinf. Vinylester strip
Fibre volume (%)	65–70	65–70	65–70	60
Fibre architecture	Unidirectional	Unidirectional	Unidirectional	Unidirectional
Thickness (mm)	1.2–1.9	1.2	1.4–1.9	2.0
Width (mm)	50–100	50–100	50–100	16
Longitudinal modulus (GPa)	155–165	300	41	131
Longitudinal tensile strength (MPa)	2690–2800	1290	900	2070
Ultimate tensile strain (%)	1.8	Not reported	2.2	1.7

Table 4.

Typical properties of FRP strengthening strips [8].

	Standard modulus carbon fibre tow sheet	High-modulus carbon fibre tow sheet	Glass fibre unidirectional fabric	Carbon fibre multiaxial fabric
Fibre architecture	Unidirectional	Unidirectional	Unidirectional	Various
Thickness (mm)	0.165–0.330	0.165	0.356	Not reported
Width (mm)	600	600	1200	Not reported
Longitudinal modulus (GPa)	230	370	72	230
Longitudinal tensile strength (MPa)	550	510	220-470	508
Ultimate tensile strain (%)	1.67–1.7	0.94	2.1–4.5	1.7

Table 5.

Typical properties of FRP sheets and fabric strengthening materials [8].

given in **Table 5**. This method can be used in situ or offsite. Various fibres are stacked in the resin matrix and allowed to cure in the mould. The cured FRP part takes the shape of the mould. Due to high adhesive properties, epoxy resin is commonly used for strengthening applications with carbon or glass fibres. Some applications that employ hand layup, such as, FRP sandwich panels in bridges, require offsite fabrication. A method using prepregs is also a type of hand layup method. In this method, the resin is pre-impregnated onto the unidirectional fibres and partially cured in sheetlike products termed as prepregs. The prepregs are cut and placed in different orientation in a mould and allowed to cure in an autoclave. Many manufacturers produce prepregs for automotive and aerospace industries [8, 16].

3. FRP applications in civil engineering

FRP composites are used in various primary and secondary structural applications in buildings and bridges. All-FRP profiles and structures, FRP rebars and prestressing

tendons, and FRP strengthening systems for existing structures are few of the most popular applications of FRP composites in civil engineering.

3.1 All-FRP structures using FRP profiles

Pultruded FRP profiles have been used in various all-FRP new structures and bridges. Where large single-component elements for bridges are required, resin transfer moulding (RTM), resin infusion moulding (RIM) and vacuum-assisted resin transfer moulding (VARTM) manufacturing processes are used. Here, the focus will mainly be on pultruded FRP shapes. Pultruded FRP profiles resemble structural steel sections, but their behaviour is more or less like timber structures [35]. Pultruded FRP profiles are produced as close or open sections. Some common sections include wideflanged sections, I-sections, parallel flange channels, rectangular hollow sections, and square hollow sections. FRP shapes have been used in chemical and food processing plants, cooling towers, lightweight foot and road bridges, building systems, railway platforms, marine structures and structures where electromagnetic transparency is needed [36-42]. Chemical inertness, corrosion resistance, lightweight and low maintenance are the key drivers behind the use of FRP structural components. Electrical and magnetic non-conductivity of glass FRPs make them suitable for use in telecommunication and other electronic industry. Cooling tower industry has seen a major development in the use of FRP profiles. Several manufacturers [24-26] produce bespoke cooling tower FRP elements as well. University of Arizona Cooling Tower using custom-made glass FRPs and standard pultruded FRPs by Creative Composites [25] is shown in **Figure 4(a)**.

Multistorey frame commercial or residential buildings have not seen much growth in the use of standard pultruded FRP profiles. This is mainly due to difficulty in finding an efficient and economical way of joining FRP components in buildings. The current design practice uses steel-like joint detailing, which is not optimised for FRP frame joints. The first demountable and mobile prototype FRP multistorey office building named the Eyecatcher was constructed by Fiberline composites in 1999 (**Figure 4(b**)). This 15-m-tall five-storey building was showcased at the Swiss Building Fair in Basel, Switzerland. After the exhibition, the Eyecatcher building was relocated to another place in Basel, where it is still being used as an office building. The building had three adhesively bonded parallel trapezoidal FRP frames connected by wooden floor decks. Bolting was only employed where needed for dismantling the structure [8, 35, 44].



Figure 4.

All-FRP structures: (a) University of Arizona Cooling Tower using pultruded glass FRP [43]; (b) the eyecatcher five-storey FRP framed building Basel Switzerland [44]; (c) Aberfeldy Scotland footbridge [45].

FRPs have been used in bridges since 1970s. Fast deployability, corrosion and fatigue resistance, high strength-to-weight ratio and mouldability are some of main properties of FRPs making them suitable for bridges. FRP components, standard or custom-made, are used in bridge decks and superstructure. Aberfeldy cable-stayed bridge in Scotland is the world's first major FRP footbridge, completed in 1992 (Figure 4(c)). It carried pedestrians and golf buggies. Except concrete foundations, the entire superstructure was made up of FRP composites. The bridge was 113 m long having glass FRP decks supported by aramid FRP composite cables [8, 46–48]. This was followed by construction of the Bonds Mill Lift bridge near Gloucester, UK, in 1994, which was the world's first FRP composite road bridge. This bridge is constructed over a canal with a mechanical lifting mechanism for navigation purposes. It included a multicellular FRP box girder filled with structural foam. The aim of this filled girder was to resist the local bending from wheel loads [1, 46–49]. The other notable FRP composite bridges include no-name Creek bridge, Kensas, USA, in 1996 [49], Kolding Denmark FRP pedestrian bridge, 1997 [26, 50], Pontresina bridge Switzerland in 1997 [35], Dawlish rail footbridge Exeter UK in 2011 [51], and the Pont y Ddraig or the Dragons bridge at Rhyl Harbour, North Wales, built in 2013 [49, 52]. More details about FRP composite bridges can be found elsewhere [3].

3.2 FRP reinforcement for concrete members

FRP-reinforcing bars, grids and prestressing tendons are used in concrete members to reinforce or prestress concrete members. FRP-reinforcing bars have been used in construction since 1970s but gained popularity by late 1980s [8, 19]. There are many reasons for using FRP reinforcements in concrete. The main reason is superior durability of FRP rebars; and the other reasons include high strength, lightweight and electromagnetic neutrality of glass FRPs. Due to noncorrosiveness of FRP, it is likely to find structural applications in or near marine environments, and in chemical and industrial plants. Glass, carbon and aramid FRP rebars are commercially available. The mechanical properties of GFRP, CFRP and AFRP rebars are given in Table 6. FRP reinforcements perform well in internal and external aggressive environmental conditions that can affect durability of reinforced concrete members. These aggressive environmental factors include the influence of moisture, temperature, sustained stress, chlorides, alkali, UV actions, carbonation and acid. Detailed discussion on effects of these durability parameters on concrete members using various FRP rebars can be found in fib 40 [19].

Research in fibre-reinforced polymer rebars is very well developed. This is reflected in various specific design guides available for design of concrete structures using FRP bars in Europe and USA. The Task Group 5.1, formerly known as Task Group 9.3, is responsible for producing design guides for FRP reinforcements in concrete in the CEB-FIP Model Code design format. In 2007, the group produced the technical report *'fib* 40 – FRP reinforcement in RC structures' [19]. There are number of design guides produced in the USA as well, including 'ACI 440.1R-15: Guide for the Design and Construction of Structural Concrete Reinforced with Fiber-Reinforced Polymer (FRP) Bars' [33] and 'NCHRP research report 907: Design of Concrete Bridge Beams Prestressed with CFRP Systems' [53]. Recent research on FRP rebars, rods and cables is reported in the papers [54–56].

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Property	GFRP	CFRP	AFRP
	E-glass/epoxy	Carbon/epoxy	Kevlar 49/epoxy
Fibre volume fraction	0.55	0.65	0.60
Density (kg/m ³)	2100	1600	1380
Longitudinal modulus (GPa)	39	177	87
Transverse modulus (GPa)	8.6	10.8	5.5
In-plane shear modulus (GPa)	3.8	7.6	2.2
Major Poisson's ratio	0.28	0.27	0.34
Minor Poisson's ratio	0.06	0.02	0.02
Longitudinal tensile strength (MPa)	1080	2860	1280
Transverse tensile strength (MPa)	39	49	30
In-plane shear strength (MPa)	89	83	49
Ultimate longitudinal tensile strain (%)	2.8	1.6	1.5
Ultimate transverse tensile strain (%)	0.5	0.5	0.5
Longitudinal compressive strength (MPa)	620	1875	335
Transverse compressive strength (MPa)	128	246	158

Table 6.

Mechanical properties of GFRP-, CFRP- and AFRP-reinforcing bars [19].

3.3 FRP strengthening systems

Externally bonded FRP systems have been used since the mid-1980s to strengthen and repair/retrofit exiting structures [57]. FRP sheets, plates, strips and fabrics can be used to strengthen or repair concrete, timber, steel and masonry structures [21]. Strengthening enhances the load-carrying capacity or ductility of the structures. While, retrofitting or repair restores the original strength or ductility of the deteriorated structure. However, the terms strengthening, repair and retrofitting have been used interchangeably in the past. The deterioration happens due to environmental factors, design errors, accidental events or lack of maintenance [8, 20].

The emphasis here is on FRP strengthening for concrete structures. The initial research on FRP strengthening for concrete structures happened in Europe and Japan in the 1980s. Externally bonded FRP systems were developed as an alternative approach to column jacketing and steel plate bonding to concrete members. Bonding steel plates to tensile zone of concrete beams increases its flexural strength. Steel plates can corrode resulting in a weaker bond between the plate and concrete member. Steel plates are heavy too and require heavy equipment for handling. On the other hand, FRP strengthening systems are lightweight and non-corrosive.

Researchers in Switzerland and Japan started developing FRP systems for flexural strengthening of bridges and concrete confinement of columns by around 1980s. Since then, significant research on FRP strengthening systems has been undertaken, especially in the last two decades. The development of design codes in Europe, Japan, Canada and the USA is ongoing [57]. Several design guides have been produced as a result. In the UK, The Concrete Society produced the 'Technical Report No. 55 on Design guidance for strengthening concrete structures using fibre composites' in 2012 [16]. In Europe 'CEB-FIP fib bulletin 14' [20] and in America 'ACI 440.2R-17' [57] are the design guides

for externally bonded FRP strengthening systems. Other design guides for FRP strengthening systems in the USA, Japan and Italy are included elsewhere [58–66]. A comparison of various design guides for FRP strengthening is presented in [67].

FRP strengthening can be applied to concrete members using wet layup, prepreg, precured and near-surface mounted (NSM) systems. The wet layup system consists of dry unidirectional or multidirectional fibre sheets or fabrics impregnated with saturated resin. Both the resin infusion and curing take place onsite. The prepreg systems have partially cured fibre sheets or fabrics, pre-impregnated offsite. The prepreg system is fully cured onsite. The precured FRP systems, as the name suggests, are manufactured and cured offsite. An adhesive is used to bond these FRP systems to concrete members. The NSM FRP system consists of circular or rectangular bars or plates bonded into grooves made on the surface of concrete members. The mechanical properties of all FRP strengthening systems are affected by environmental factors, such as high temperature, chemical exposure and humidity. A reduction factor is usually applied to the mechanical properties of the FRP strengthening system to account for the environmental factors. Details of these reduction factors can be found in 'ACI 440.2R-17' guide for FRP strengthening system [57]. Figure 5 shows various FRP strengthening systems in real structures. Qualitative comparison of FRP strengthening sheets using carbon, aramid and E-glass fibres is presented in Table 7. The criterion in this table is only applicable to strengthening applications of FRP.



Figure 5.

FRP strengthening systems [68]: (a) column strengthened with woven unidirectional carbon fibre fabric; (b) CFRP plates and strips bonded to a bridge concrete beam; (c) externally bonded CFRP plates for flexural strengthening; (d) near-surface mounted (NSM) flexural strengthening of bridge decks with CFRP rods near negative moment regions.

Criterion	Fibre composite sheet made up of:		
	Carbon fibres	Aramid fibres	E-glass fibres
Tensile strength	Very good	Very good	Very good
Compressive strength	Very good	Inadequate	Good
Young's modulus	Very good	Good	Adequate
Long-term behaviour	Very good	Good	Adequate
Fatigue behaviour	Excellent	Good	Adequate
Bulk density	Good	Excellent	Adequate
Alkaline resistance	Very good	Good	Inadequate
Price	Adequate	Adequate	Very good

Table 7.

Qualitative comparison among high tensile—carbon, aramid and E-glass fibres in FRP strengthening application [69].

4. Durability aspects of FRP composites

Environmental factors affect durability performance of FRPs in terms of reduction in strength and stiffness. These environmental factors include moisture, ultraviolet exposure, elevated temperature, alkaline or acidic and saline solutions, freezing–thawing cycle and high humidity. Various testing and mitigation measures are available in the design guides [16, 19, 20, 33, 57] for FRP rebars and strengthening systems. The available testing for durability includes hot-wet cycling, alkaline immersion, freeze–thaw cycling, ultraviolet exposure, salt water and dry heat. Protective coatings can be applied to FRP composite part to account for various environmental factors. Carbon fibre is resistant to alkaline/acidic environment, whereas plain glass fibre degrades in these environments. In high alkaline and high moisture environments, carbon FRPs should be used in place of glass FRPs.

GFRP and AFRP are non-conductive (electrical insulators), while carbon FRP is conductive. Carbon-based FRP should not be used in direct contact with steel elements to avoid galvanic corrosion. CFRP composites are also resistant to creep rupture under sustained loading and fatigue failure under cyclic loading [33, 57]. Durability of externally bonded FRP composites in concrete structures is reviewed in a recent paper [70]. There is a lack of long-term durability and performance data for FRP profiles and their joints [71]. Durability of FRP composites exposed to elevated temperature and fire, ultraviolet radiation, creep and fatigue loads, freeze–thaw conditions and moist environment is discussed in a comprehensive book by Karbhari [72]. Structural health monitoring and field evaluation of FRP composites' durability are also explained in the book [72] and the recent review paper [73]. Ageing effects on mechanical properties of FRP are discussed in [74, 75].

5. Sustainability of FRP

Sustainability is about meeting the needs of the present without compromising on the needs of future generations, as per Brundtland report [76]. In the past, good structural design used materials and resources efficiently with focus on performance and economy. The sustainable design approach is based on material that considers environmental, economic and social factors and energy and resource consumption in addition to performance criteria. The evaluation of sustainability of materials involves life cycle assessment from cradle to grave including raw material procurement, fabrication and processing, construction, maintenance, recycling and disposal. An ideal sustainable material would have a closed life cycle that utilises renewable resources, energy and zero waste with low impact on environment, people and society [10].

Due to lightweight and ease in transportation, FRP composites are generally less energy-intensive to produce. FRPs also have minimal ecological and carbon footprint compared to the traditional materials. FRPs have no corrosion with superior performance in chemically aggressive environments. FRP composites resist creep and fatigue loads better than other materials. This leads to low maintenance for structures that use FRP materials. Resultantly, the expected durability of structures is enhanced by using FRPs. Sustainability of FRPs can be better understood by evaluating various stages of their lifespan and their impact on environment. In this way, better insights can be gained into the life cycle assessment of FRPs.

5.1 Lifespan of FRP composites

FRP composites are considered to have long lifespans. There are four different stages of FRP's lifespan: (1) extraction and production of FRP material; (2) manufacturing; (3) use and (4) end-of-life disposal.

5.1.1 Material extraction and production

The first stage involves sourcing raw materials for producing input materials for FRP composites. After extraction, the materials are processed and refined to become input materials for FRP manufacturing. The extraction and production of FRP input materials requires energy [6]. **Table 8** shows the energy intensity required for extraction and production of different materials. Thermosetting polymer resins are created through energy-intensive chemical process. Carbon fibres have relatively very high energy consumption compared with other synthetic fibres. Due to high energy demand, most pultruded FRP profiles use glass fibres rather than carbon fibres. Carbon fibres have far better strength though compared to conventional materials. The lightweight of FRPs reduces energy required for transportation at a later stage in their life cycle. The carbon emission due to transportation of traditional material is significantly higher than FRP composites. Using FRP composite as an alternative to steel reduces the weight of the structural component to about 60–80% [6, 77].

5.1.2 Manufacturing

While FRP composites have promising properties for structural engineering, their production on mass scale is yet to be realised. The main barriers to their widespread use include low production, lack of automation and high cost. The energy consumption of various manufacturing processes of FRP composites are shown in **Table 9**. The consumption is just for the manufacturing process, not for constituent materials. Manual methods, such as hand layup and prepreg, require significant energy compared to automatic processes, like pultrusion and filament winding. As FRP composite materials require more than two materials, additional energy may be needed for fibre impregnation, surface preparation, additives, fabrics and solvents. Pultrusion and filament winding have very low energy inputs of 3.1 and 2.7 MJ/kg [77], respectively.

Low energy intensity input materials and manufacturing combined with automation can reduce the cost of FRP composites significantly. Pultrusion process benefits

Material		Energy input (MJ/kg)
Polymers	Polyester resin	63–78
	Epoxy resin	76–80
Fibres	Glass fibre	13–32
	Carbon fibre	183–286
Metals	Steel	30–60
	Stainless steel	110–210
	Aluminium	196–257

Table 8.

Energy content for extraction and production of input materials [77].

Manufacturing process	Energy consumption (MJ/kg)
Pultrusion	3.1
Resin transfer moulding (RTM)	12.8
Vacuum-assisted resin infusion (VARI)	10.2
Filament winding	2.7
Hand/wet/spray layup	14.9
Injection moulding	19.0
Autoclave moulding	21.9
Prepreg	40

Table 9.

Energy consumption for manufacturing of FRP composites [77].

from low energy input, high production rates and automation. Even though pultrusion requires the least energy to produce FRP parts, it cannot produce very complicated shapes. Pultrusion is only limited to making very simple section profiles, such as tubes, wide-flanged sections, parallel flange channels, railings, poles and ladders [77]. Other energy-intensive processes like RTM, infusion moulding and prepreg are used to produce complex shapes. Single-component FRP bridge elements are usually manufactured by resin-infused/prepreg methods [48].

5.1.3 Use

FRP composites have been used in various industries, such as aerospace, automobile, construction, marine, consumer products and appliances. Especially, advanced FRP composites have been adopted well in aerospace industry. The Boeing 787 Dreamliner contains 80% of FRP composite materials by volume. The Airbus A380 is the first aircraft to have CFRP composite wing box. The boats have also been constructed from FRP composites. Today, 90% of the hulls of modern boats consist of FRP composites. In automotive industry, 90% of truck bodies are made of FRP composites. In these industries, the lightweight of FRP composites reduces fuel consumption and carbon emissions. In military applications, aramid fibre composites are used in bulletproof jackets and other impact resistant body outfits. Construction industry uses about a quarter of globally produced FRP [6, 77].

5.1.4 End-of-life disposal

Reuse of FRP composites for another application is a sustainable way to dispose of FRP waste. Ideally, FRP waste should be reused as FRP part in another application. However, due to special production and application of FRPs, reuse potential for FRP parts is very limited as compared to the traditional materials, such as steel and timber. FRP composites can be disposed of in three ways: dumping, incineration and reuse/ recycling. *Dumping in landfill* is the cheapest way to dispose of FRPs. The scrap FRP composites must be sorted and separated. However, separating high value fibres from cured resins is difficult in FRP products, as steel and other parts might be attached to the FRP products [78].

5.2 Recycling of FRP composites

Thermoplastic-based FRP materials are easier to recycle by remelting and remoulding due to weaker molecular bonds in thermoplastic resin matrix. Thermosetbased FRP composites are difficult to recycle due to cross-linked nature of thermoset resins [79]. There are three main processes for recycling thermoset resin-based FRP waste materials: (1) incineration-with partial energy recovery from heating of the organic part and co-incineration-with both raw material and energy recovery; (2) thermal and chemical recycling-with decomposition processes to partially recover fibres and energy; (3) mechanical recycling-with breakdown of FRP composites by shredding, grinding and milling resulting in smaller fibrous or powdered products.

5.2.1 Incineration and co-incineration

Incineration and *co-incineration* are the methods of energy and/or material recovery for thermoset-based FRP materials. *Incineration* results in 'partial energy recovery from heat generated during combustion of the organic part'. While, *co-incineration* leads to recovery of both energy and raw material [79]. In incineration process, 50% of the waste remains as ash, which still needs to be landfilled. Air pollution resulting from incineration is one of the setbacks of incineration. Co-incineration has been tried in Germany to convert FRP waste into energy and clinker (the raw material) for cement manufacturing [80]. No ash is produced in this method, two-thirds of composite waste is converted into clinker and one-third is recovered as fuel for kiln. One drawback of co-incineration is that the composite waste material needs to be reduced to small particles suitable for cement kiln. Incineration and co-incineration can be classified as reuse methods.

5.2.2 Thermal recycling

Thermal recycling decomposes the FRP waste material into raw recovered fibre and results in partial energy recovery. It is only applied to CFRP composite waste, where the value of recovered fibre and energy is more than the cost of thermal recycling. It requires large amount of FRP material waste to justify the cost of recycling. The most common thermal recycling method is *pyrolysis*. It involves heating the FRP waste in an inert atmosphere to recover the polymer as oil. Another thermal recycling process is *oxidation in fluidised beds*. The resin matrix is combusted in a hot and oxygen-rich flow in this method resulting in fibre recovery. Strength and shape degradation may happen to the recovered fibres in thermal recycling. Thermal recycling is still far away from becoming commercially viable recycling process [79].

5.2.3 Chemical recycling

Chemical recycling consists of dissolving the resin using chemicals at low temperatures. This is a gentle thermal stress-free method. The recovered fibres retain their original strength in this process. Some limitations of this method include use of hazardous solvents, reduction in length of recovered fibres and lack of adhesion capacity of the recovered fibres. Like thermal recycling, chemical recycling is yet to become an economically viable solution. It is just limited to low-volume CFRP recycling [79].

5.2.4 Mechanical recycling

Among all methods, *mechanical recycling* is the most developed and viable process to recover reusable fibres [79]. This consists of shredding, grinding and milling the waste FRP material into smaller-sized filler material. The extracted materiel can either be used in new FRP products, which are based on bulk or sheet moulding processes or in concrete with cementitious, asphaltic or polymer binders [78]. The mechanical recycling has several economic and environmental benefits. These include no air pollution, no complicated and expensive equipment, and recycling ability on a larger scale. Some drawbacks consist of risk of ignition during shredding and low value recovered fibres. Carbon fibres can retain their strength though, while glass fibres lose their strength after thermal treatments [78, 79]. Use of glass and carbon FRP waste material in concrete and its life cycle assessment are presented in a recent paper by Singh et al. [74].

6. Conclusions

Research in use of FRP as rebars and strengthening systems is well developed with several design guides produced worldwide. There is a lack of legal design codes and awareness among structural engineers for FRP structural profiles and their joints. Some evolving design guides for pultruded FRP shapes have been produced in last two decades, such as Eurocomp design code [81], ASCE Pre-Standard for pultruded FRP [82], CIRIA C779 FRP bridges—guidance for designers [49] and Italian guide for pultruded FRP elements [83]. These guides are not legally binding for all-FRP structural design. Work is in progress in Europe and America for developing agreed design codes for all-FRP structures. Several automatic and manual manufacturing processes of FRPs were discussed. Pultrusion is the most cost-effective and eco-friendly process for producing constant cross-sectional FRP profiles at high production rates by means of automation.

The long-term durability and performance data for FRP profiles and their joints is limited. Some data is available, though, for FRP strengthening systems and rebars. Glass FRPs are by far the most common FRP composites with a market share of 90% of the FRP produced worldwide. Due to low viscosity, thermosetting resins (polyes-ter, vinylester and epoxy) are the most popular polymer matrices for producing FRP structural products. The recycling methods for FRP including incineration, chemical, thermal and mechanical processes were discussed. Recycling of FRPs seems to be the most challenging aspect of sustainable design. Only a limited percentage of FRPs can be recycled, which may not be economically feasible keeping in mind the cost of recycling process.

Author details

Jawed Qureshi Senior Lecturer in Structural Engineering and Design, University of East London, London, UK

*Address all correspondence to: j.qureshi@uel.ac.uk

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Chapter 6

Multifunctional Carbon Nanotube Reinforced Polymer/Fiber Composites: Fiber-Based Integration and Properties

Hassaan A. Butt, German V. Rogozhkin, Andrei Starkov, Dmitry V. Krasnikov and Albert G. Nasibulin

Abstract

Carbon nanotubes are one of the most versatile nanomaterials currently used to modify the properties of both thermoplastic and thermoset-based composites, both with and without the use of a fibrous reinforcement phase. Electrically and thermally conductive by nature, their addition to traditional fiber-reinforced polymer composites has not only heralded increased mechanical properties in terms of flexural, tensile, impact, and interlaminar properties, but also allowed imparting inherent conductivity to the final composites, allowing the creation of specialized, isotropic, anisotropic, and hierarchically graded composites with applications ranging from self-diagnostic damage detection, de-icing to energy storage and conversion. The purpose of this book chapter is to focus on the methods used to integrate carbon nanotubes, both anistropically and anisotropically via techniques that focus solely on the fibrous reinforcement phase and not the matrix, into fiber-reinforced polymer composite materials. The chapter aims to review the properties that may result from such integration of the various techniques, provide a current state of the art of the multifunctional properties, which have been achieved thus far, and outline possible future dimensions of investigation and application.

Keywords: carbon nanotube, nanocomposite, fiber reinforcement, fiber-reinforced polymer, glass fiber, carbon fiber, functional composite

1. Introduction

Carbon nanotubes (CNTs) are one of the few materials that have found a plethora of applications in almost all fields, stemming from their exceptional inherent properties. Being inherently electrically and thermally conductive [1–3], showing relatively high stability in elevated temperatures [3, 4], and having the ability to be integrated with a wide variety of materials [5] while showing a level of chemical inertness [6], CNTs are probably one of the most widely investigated nanomaterials for applications in their pure form, in macrostructures, or when combined to form nanocomposites.

The term CNT is a generalization, which encompasses the three most widespread forms of this material, categorized by the number of walls shown by the tubes, as shown in **Figure 1**. CNTs may display single (SWCNT), double (DWCNT), or multiple walls (MWCNT, more than 2), which in effect causes changes to their properties, hence the further categorization [7]. CNTs may also be classified according their ends being opened or closed [8], by their electronic structure and properties [9], or by additional features added to their structure during synthesis [10]. However, one trend remains: reducing the number of walls displayed by the CNTs with the length remaining constant, or by keeping the diameter constant and increasing the length, increases the aspect ratio of the CNTs [11]. This is inherently important for CNT/polymer nanocomposites since percolation theory and percolative behavior are entirely dependent on this ratio [12]. Almost all bulk functional properties are dependent upon dispersion, the percolation network, and its formation and manipulation [12–14].

All polymer composite materials consist of two distinct phases: the matrix material, generally comprising the polymer itself, and the reinforcement material, which may be particulate or fibrous in nature. Since this book chapter deals with fiber-reinforced polymer composites (FRPCs), this chapter shall focus on elaborating on the fibrous reinforcement phase. As stated, FRPCs are made up of two distinct phases, and, thus, two distinct approaches to including CNTs into FRPCs exist: the first being through modifying the matrix material and the second integrating CNTs on the fibrous reinforcement phase. Although this chapter is dedicated to the latter approach, it is necessary to lightly touch upon the former to draw comparisons and be able to compare the advantages and disadvantages of the two.

Integrating CNTs into matrix materials is the more popular approach when literature for the topic is reviewed, both for thermoset [15–18] and thermoplastic polymer nanocomposites [19–23]. In this method, CNTs are dispersed within the polymer matrix to obtain either homogeneous or heterogeneous dispersion of structures [24]. Following this modification of the polymer phase of the composite, this dispersion is then used and transferred to the fibrous reinforcement phase, by method such as vacuum infusion [25], resin transfer molding [26], injection molding [27], compression molding [19], extrusion [28], and pultrusion [29], depending upon the type of polymer and fibers used. Integrating CNTs through the matrix is extremely popular since it relies on cheaper processing equipment, is facile and robust, can be combined with other nanomaterials to form complex composites without major changes to processing regimes; moreover, it can be used with predispersed masterbatches, and the quality of the percolation network is dependent



Figure 1.

The structures of CNTs, most commonly used for classification; (a) SWCNT, (b) DWCNT, and (c) MWCNT.

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upon processing parameters, which can easily be optimized. This method has been shown to successfully be used for improving mechanical properties [30, 31], while promoting multifunctionality such as flame retardancy [32], electrical conductivity [15, 19], thermal conductivity [16, 33], piezoresistive response [15, 34], selfdetection of damage [35], increased wear resistance [36], and improved fracture and failure resistance [37, 38].

However, the technique has some drawbacks. Due to the fact that high-performance composites usually use continuous fibers as the reinforcement material, it is common for nanoparticles such as CNTs to be "filtered" by the fibers, causing uneven distribution of the nano-reinforcements, in turn causing regions of inhomogeneity [39, 40]. Also, since matrix integration usually employs ultrasonication in one form or another, the CNTs become susceptible to damage and loss in properties [41], and the same is seen for some forms of mechanical dispersion [42, 43]. The addition of CNTs to polymer matrices greatly increases the viscosity of the matrix while processing [44], leading to the need for more expensive and high-tech machinery to obtain optimum dispersion and higher weight percentages [45, 46], not to mention a decreased amount of fiber wetting. The addition of CNTs to the polymer matrix may also negatively influence the polymerization degree, causing a loss in mechanical properties [47, 48].

Considering these drawbacks, fiber-based techniques, although more intensive than matrix-based in terms of capital and technological investment, may have certain advantages for large-scale production. The following sections elaborate on the techniques included in fiber-based integration, their advantages and drawbacks, current status of selected results, and possible future implications and directions.

2. Fiber-based CNT integration

2.1 Integration techniques

As stated in the introduction, the integration of CNTs into composites to form nanocomposites by deposition on fibers holds several advantages over the alternative technique. Through this approach, CNTs are not subjected to the filtration effect associated with matrix-based composite manufacturing techniques. Secondly, the CNTs, which are already deposited onto the fibers, allow for a strong interfacial connection between the matrix and the fibers [49]. In addition, although the curing of the matrix may be affected by the CNTs themselves in the local vicinity, this effect is theoretically lower than for homogeneously dispersed CNT/polymer nanocomposites, where the entire volume of the polymer may be affected. By using the fibers as the integrating material, specialized hierarchical composites where directional alignment of CNTs is maintained allow for anisotropic properties to be obtained [50]. Fiber-based integration techniques may roughly be broken down into the following categories: (1) direct deposition of CNTs on the fibers from the CNT source or by direct growth on the fibers, (2) deposition of CNTs via the spray technique, (3) dip coating, and (4) dry transfer of CNTs to the fibers. Additional techniques, which have been shown to be successful in transferring CNTs on the other types of materials, include using a doctor blade for direction alignment and thin film creation [51], the Meyer rod technique for creating thin films [52], slot casting for creating continuous layers with a matrix [53], and inkjet printing [54]. Since these latter mentioned techniques either have not found widespread usage in large-scale fiber-reinforced composite

manufacturing or application, or rely on a matrix to be present and retained, they have not been discussed in great detail in the further sections.

2.2 Direct deposition and growth

In this method of CNT deposition, CNTs are deposited directly on the fibers of FRCPs from the CNT reactor, as shown in **Figure 2**. In essence, instead of CNTs being collected on a filter substrate as is normally done, the fibers act as the substrate and collect the CNTs directly onto their surface [55]. This technique is, however, the most complicated and least scalable of the techniques associated with fiber-based CNT integration. Among a few of the problems associated with this technique are enlarging the collection area in the reactor to be able to place the fibers, the fact that control of the flow rate for CNT production to the reactor needs understanding and mastering of the complex ensemble flow of the precursors, catalyst and reactor design, as well as flow tuning for CNT production to the reactor (without additional reactor modifications) [56–60]. This makes controlling the area of deposition troublesome and requiring detailed knowledge of CNT synthesis parameters.

A more intriguing method, whereby a catalyst is deposited on fibers, which are then subjected to reactor conditions to grow CNTs directly on the fibers, was deemed to be more suitable as it allowed passing the fibers through a portion of the reactor, allowing increased controllability of deposition without the need for upscaling the reactor itself [61]. The technique has been investigated by a number of authors all showing the feasibility of growing CNTs directly on the fibers through catalyst deposition, CVD variable adjustment, or a combination of both [62, 63].

Studies utilizing this technique have shown that growth on fibrous substrates can successfully be performed while providing a number of multifunctional properties [64, 65]. He *et al.* showed that the technique, when applied in various configurations, could lead to directionally anisotropic conductivity, with up to eight times in difference according to directions [55]. Rahmanian *et al.* successfully grew vertically aligned CNTs [62], which have been shown to provide increased interlaminar adhesion and strengthening in other publications [66, 67], as well as the ability to detect



Figure 2. Direct deposition of CNTs onto fibers with a CNT reactor.

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polymerization degree based on piezoresistive response [68]. Zhao et al. used a novel flame synthesis technique to grow CNTs directly on glass fibers, resulting in an electrical conductivity increase of more than 10 magnitudes with a simultaneous reported maximum thermal conductivity of ~0.5 W/mK [69]. Pozegic et al. showed that by growing CNTs directly on fibers, they were able to enhance the electrical conductivity of the final composite made via vacuum infusion, leading to electrical conductivity enhancement of up to 450%, with potential applications in lightning strike damage mitigation and de-icing in the aerospace industry [70]. The same authors showed that the technique results in no major loss in infusion ability, ~140% increase in Young's modulus, 20% increase in ultimate shear stress, and 83% increase in the initial fracture toughness of the final composites [71]. Veedu et al. reported that this method of CNT introduction into FRCPs resulted in lower flexural deflection, higher flexural modulus, strength and toughness (105, 240, and 524% increase), higher interlaminar fracture toughness (~350% improvement), a shear sliding fracture toughness of 54%, all the while showing a thermal and electrical conductivity increase of 151% and roughly 5 magnitudes, respectively [72]. Further studies in the field have shown that the growth on fibers allows not only mechanical property enhancement when used for FRCPs but also possible applications in electrochemical detection [73], structural batteries and energy storage [74], low power resistive heaters for advanced composite structures [75], and electromagnetic shield [76].

2.3 Spray deposition

A seemingly simple, yet technologically intensive technique used to transfer CNTs onto the fiber is the method of spray deposition. In this technique, dispersions of CNTs made with solvents and dispersants are sprayed directly onto fibers using a spraying device, as depicted in **Figure 3**. The technique allows CNT powders to be used, which is the most popular commercial form available. Although relatively simple in procedure, this technique does have its drawbacks. First, a homogeneous dispersion of CNTs must be prepared with time and resource-consuming techniques. The CNTs utilized must be carefully purified from the synthesis byproducts. To



Figure 3. Spray deposition of CNTs on fibers.

detangle CNT bundles usually mechanical mixers, ultrasonicators, high-speed homogenizers, or three-roll milling machines must be used. To avoid subsequent CNT agglomeration, surfactants are usually added in the dispersion. And finally, to get rid of large agglomerates, centrifugation techniques may also be necessary to apply. Then, to provide homogeneous dispersion on the surface of the fibers, the dwell time, spray rate, and coverage area all need to be monitored or planned. Further, to make sure that proper adhesion of the CNTs with the fibers takes place, the fibers themselves may need to be de-sized before the procedure. The solvents and dispersants that may be used in this technique also need to be checked for compatibility with the fibers and their sizing. Another major drawback of this technique is that the method randomly disperses CNTs on the fibers' surface, with alignment of the CNTs not being possible (without electrostatic modifications), which in turn makes the anisotropic properties of the end nanocomposite difficult to engineer.

Perry *et al.* showed that the spray deposition technique can be used to create laminar composites, which displayed Mode 1 fracture property increase (20%) with weight percentages as low as 0.057% [77]. Wang *et al.* showed that the process of deposition can be conducted using a simple commercial mechanical sprayer when coupled with a suitable dispersant [78]. The study showed that the flexural strength of natural FRPCs can be increased by up to ~38% and that the interfacial shear strength can be raised by ~25%. The study did, however, show that the deposition of the CNTs using this mechanical spray device was not as effective and uniform as other compressed gas-based spray devices. Lee *et al.* showed that by using a more efficient and standardized spray method, increases in tensile strength, modulus, and ultimate tensile strength were possible [79]. Li et al. dedicated a study to understanding the parameters that affect the final morphology and deposition degree of CNTs on fibers when the spraying technique is upgraded to electro-spraying [80]. The technique combines the traditional spraying technique with the application of voltage and electric connections, causing deposition to be aided by electrostatic forces. The study showed that with such modification to the process, the voltage is the main parameter dictating the deposition, especially when electrically conductive filaments such as carbon fiber are used. After voltage, it was found that the distance between the spray source and the deposition substrate (fibers) also influences the end morphology and deposition degree. The same electrodeposition technique was utilized by Sabri et al., where a 34% increase in fracture toughness was noted in samples with CNTs, the mechanism of toughening being attributed to fiber bridging [81]. Fogel et al. conducted a more systematic study where along with mechanical properties of the composites, the electrical properties were also determined [82]. The study showed that the electrical conductivity of the composites made from CNTs reached almost twice the value of reference samples, whereas DC spectroscopy methods showed this value to be two and a half times the value. Dynamic mechanical analysis showed no major changes in behavior for the polymer matrix, whereas slight decreases in mechanical properties such as interlaminar shear strength and fracture toughness were noted.

Cao *et al.* reported the electromagnetic shielding properties of composites produced from this method of integration [83]. The electromagnetic shielding efficiency for these composites was as high as 76% for weight percentages as low as ~0.5%. At various weight percentages studied in the article, tensile strength showed the highest increase of 4.1% at a loading of ~0.1% by weight, flexural modulus increased by 28.6% at a loading of ~0.3% by weight, elastic modulus showed an increase of 5.4% at ~0.1% weight loading, and flexural modulus showed an increase of 24.6% at ~0.3% by weight loading. Electrical conductivity was noted to increase by 68% at a Multifunctional Carbon Nanotube Reinforced Polymer/Fiber Composites: Fiber-Based... DOI: http://dx.doi.org/10.5772/intechopen.108810

loading of ~0.5%, with an SE value of 25.4 dBm/m. Holubowitch *et al.* described the electrochemical application of CNTs dispersed through the spray technique, although without any fibrous reinforcement phase [84]. The work showed that composites manufactured from this technique can be compared in performance to those that are currently used as scientific standards. Zhang *et al.* demonstrated that the application technique can be used to manufacture self-diagnostic materials capable of detecting damage through piezoresistive response even at extremely low CNT additions of ~0.01% weight [85]. Simultaneously, these composites showed a marked increase in fracture toughness and interlaminar shear strength. A similar work by Gonzales et al. showed that nanocomposites produced with this method displayed simultaneous marked increases in flexural strength, electrical conductivity, and interlaminar shear strength at weight percentages below 1.0%, with piezoresistive gauge factors for self-monitoring as high as 6.5 [86]. The materials were also noted to have good cyclic response without drift or major variation. Li et al. described hybrid nanocomposites manufactured through this method with enhanced electrical (increase by a factor of 4) and thermal conductivities (increase of seven times) [87]. Zakria et al. reported similar increases in mechanical and thermal conductivity via the same technique coupled with electrostatic assistance [88].

2.4 Dip coating

This technique for integrating CNTs in FRPCs is inherently simple yet requires a liquid medium for effective transfer, as shown in **Figure 4**. In this technique, CNTs are pre-dispersed in a liquid, which may consist of a polymer matrix, solvent, dispersant, or all of the mentioned chemicals. Once an effective dispersion is created, the fibers are soaked in the liquid dispersion, causing the CNTs in the dispersion to be transferred to the fibers. The fiber medium, after a certain soaking time, is removed from the container and dried to remove the unwanted chemical species, leaving



Figure 4. *Schematic representation of the dip coating technique.*

behind the CNTs as a coating on the fibers. This technique, like spray coating, is also often coupled with electrostatic methods to ensure that effective and even transfer of CNTs to the fibers takes place.

Although this technique seems relatively simple, it is the method of integrating CNTs onto fibers that may result in the highest amount of variance. Firstly, to attain a dispersion of CNTs within a liquid requires processing machinery such as mechanical mixers, ultrasonicators, high-speed homogenizers, or three roll mills and extruders. Secondly, this solution needs to be optimized so that the dispersion is stable and agglomeration of the CNTs is minimized. In addition to the CNTs in the dispersion, the dispersants need to be carefully chosen so as to not cause any chemical or physical damage to the fibers being used. When soaking or transfer is taking place, this method provides no control as to how much or how many CNTs may be transferred onto a certain location of the fibrous reinforcement phase. Electrostatic techniques often help in this regard, controlling the amount of CNTs deposited on the fiber surface through voltage control. Finally, when inserting or removing the fibers from the dispersion used for coating, the movement and flow of the dispersion may cause an uneven amount of deposition, making the technique cumbersome and requiring a certain level or automation or delicacy.

Awan *et al.* showed than the dip coating method resulted in a better quality of CNT grafting and deposition than the aforementioned spray technique [89]. Rong et al. in their early publication showed that the technique may result in an increase of tensile strength from ~10 to ~25% [90]. Jamnani et al. reported that the technique of dip coating coupled with dispersants and chemical treatment resulted in an ultimate tensile strength increase of 38% and an interfacial shear strength increase of 116% at the optimum weight percentage and soaking time [91]. A work by Tzounis et al. focused on understanding the effect of chemical grafting and physical adsorption of CNTs on fibrous reinforcements and their end mechanical properties in the composites [92]. The study, which used dip coating to transfer CNTs from a solution to the intended fibrous reinforcement phase, showed that the composites produced with chemically bonded CNTs had lower electrical conductivities (~2 S/cm compared to 20 S/cm) and lower interfacial adhesion strength (48% lower than physically bonded CNT composites). Dip coating supported by electrophoretic deposition was shown to be feasible by Tamrakar et al. for controlling the thickness of the deposited CNTs $(200 \text{ nm}-2 \mu\text{m} [93])$. The authors of the study showed that not only did the electrical conductivity of the composites increase, but the interfacial shear strength increase was as high as 58% compared with unmodified fibers. A similar study by Kwon et al. reported on hybrid nanofillers containing CNTs deposited on fibers using the electrophoretic deposition technique, which allowed increased interfacial adhesion of the fibers to the matrix, a ~10% increase in flexural strength, and simultaneously increased electrical conductivity of the final nanocomposite by ~1400% [94].

The multifunctional properties of FRCPs manufactured using this technique were described by Liu *et al.* [95]. The fibers produced during this study showed low electrical resistance at low weight percentages ($\sim 10^{-2}$ S/cm at 0.12–0.5% weight) while simultaneously showing major increases in tensile strength, Young's modulus, reduced Poisson ratio, and increased interfacial shear strength. Natural fiber-reinforced composites were manufactured and investigated using this manufacturing technique by Zhuang Liu *et al.* [96]. The study showed that even with natural fibers, electrical resistivity values may drop up to five magnitudes at low weight percentage addition of CNTs. The study showed that directional conductivity values were different along the path of the fibers and through the thickness of the composites.

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The composites also showed sensitivity to thermal changes as thermistors, showing sensitivity to relative humidity as well as piezoresistive response with gauge factors of up to 12. One work by Liu *et al.* showed that by using this technique, simple cotton fabric can be functionalized and thus holds promise for applications in fiberreinforced applications [97]. The work showed that simple cotton fabric can be made more mechanically stable, flame retardant, can be used as a UV blocking material not materials and be made superhydrophobic simultaneously, all with CNT loading percentages of less than 6%. A work by Lima et al., in which a multifunctional cottondoped CNT fiber was developed, showed that this technique was used to modify a fibrous structure and endow it with antibacterial and electrochemical performance, while being able to self-heat under the application of electricity [98]. The study showed potential applications of CNT/fiber composites for personal smart devices. Tzounis et al. showed similar multifunctional behavior of composites manufactured through the same method, where the final composites were electrically conductive (~10 S/cm), could be used for self-determining the degree of curing of the composite via piezoresistive response, were sensitive to UV radiation while at the same time could be used as a thermoelectric energy harvester due to the semiconductive nature of the CNTs used to produce the nanocomposite [99].

In addition, this versatile technique has been shown to be feasible for the production of transparent and flexible fiber-based electroluminescent devices [100], wearable electronics [101], and special fiber-based electrodes for human neuromodulators [102].

2.5 Dry transfer of CNTs

The last method described in this chapter is that of dry transfer. Probably the simplest method of integrating CNTs into fibrous reinforcement phases conceptually, dry transfer involves taking CNTs that have been collected on a substrate, bringing the CNT layer into contact with the intended fiber layer, applying a small amount of force to transfer the CNTs onto the fibers, and then removing the original collection substrate. This technique can be done both manually and with some degree of automation to make sure that the transfer is done efficiently. Since CNTs may be collected on a substrate randomly or in an aligned way during their synthesis, both types of CNT films have been investigated in literature for their use in manufacturing multifunctional nanocomposites. The basic dry transfer process has been schematically shown in **Figure 5** for randomly oriented films and **Figure 6** for aligned films.

Although seemingly simple, the technique does have its drawbacks. The application of force does not guarantee homogeneous removal of the CNTs from the collection substrate, often causing wrinkles and defects to appear in large-scale transfers. Furthermore, even when the application of force is uniform, since the receiving fibers are often woven, their surface is uneven and may cause defects in the film. CNT thin films themselves are delicate and easily break, which make the process extremely susceptible to damaging the films in one way or another. Another factor adding to the difficulty of this integration technique is that thin films often negatively impact the infusion capacity of the composites they are integrated in, especially when thick films are applied. Even though such drawbacks exist, many publications have shown that with proper process streamlining, the technique can produce large-scale multifunctional nanocomposites, as is seen in the following paragraphs of this section.

Randomly oriented CNT films have been shown to be feasible for integration into large-scale FRPCs while endowing them with multifunctional properties. Early



Figure 5.

Schematic showing the dry transfer technique for randomly oriented films.



Figure 6. Schematic showing the dry transfer technique for aligned films.

publications on the topic such as by Wang *et al.* [103], which used randomly oriented thin films as interplies for FRPCs, showed that their inclusion could cause an increase in tensile strength, tensile modulus and provide significant directional electrical conductivity. However, the work did highlight that the inclusion of such thin films may cause voids and defects, owing to the fact that resin infiltration in the large-scale composites may be negatively impacted.

Zhang *et al.* [104] reported on multifunctional composites made by the integration of CNT thin films, where the end composite could be used for the self-sensing of strain and damage, showing gauge factors between ~1.5 and 6 and that such material systems had a future in structural health monitoring. A similar work by Pan *et al.* successfully created ultrathin and flexible carbon fiber composites with CNT thin film interlayers [105]. The composites showed a good load transfer between the layers, reduced delamination, and improved interfacial bonding along with increased damping capability. A comparable tensile strength and tensile modulus to that of the unmodified composite were noted, with the electrical conductivity of the nanocomposite being 2 magnitudes higher. Li *et al.* studied in detail the effects of thickness of
the films used for reinforcing FRPCs as well as the testing conditions used for interlaminar fracture toughness, with the results indicating that for quasistatic loading, two films applied together provided the best results whereas for Mode 1 fracture, the greater the number of films, the tougher the composite [106]. However, the work did show that increasing the number of layers did reduce the impregnation of the films by the matrix, leading to crack origination and propagation at the CNT interlayers.

While such interlayers have been investigated for their influence on mechanical properties, authors have also focused on their specific multifunctional property enhancement abilities. Ribeiro *et al.* showed that the incorporation of randomly oriented CNT thin films provided no major benefits to the composite in terms of interlaminar shear and compressive shear strength, yet managed to improve the thermal stability and glass transition temperature. The study also showed that such composites could provide electromagnetic shielding in the X-band region, with a \sim 99% attenuation rate [107]. The same author showed in a later publication that the electromagnetic shielding of such composites is frequency-dependent and can thus be tailored if needed [108]. Xu et al. showed that such thin films incorporated into composites can be used for resistive heating and curing of the composites themselves during manufacturing, with no major differences noted in the glass transition, curing degree, or tensile properties when compared with traditional oven curing [109]. The study also showed that such films can be incorporated into composites for the practical application of de-icing during the service conditions of typical composites. Lu et al. reported on the usage of CNT thin films for the manufacturing monitoring of FRPCs [110]. The authors described the different stages of polymer curing during the manufacturing process through piezoresistive changes, while the interlayer was also able to show sensitivity to the amount of solvents typically used during composite manufacturing. In addition, the work showed that the CNT thin film interlayer was also sensitive to temperature changes and showed a range of responses dependent upon the temperature range. In their work, Slobodian et al. presented results confirming the structural health monitoring ability of such composites to deformation, with gauge factors for pristine films being ~5, with an increase to ~500 when additional doping is applied [111]. Han et al. utilized composites incorporating CNT thin films for lightning strike protection [112]. The work, which combined electrical and nondestructive testing techniques, proved that for both visible and underlying damage of the composites, incorporation of an electrically conductive CNT film was essential to providing superior protection and post-strike mechanical properties. Li et al. reported on the flame-retardant properties of CNT thin films compounded with polyethersulfone, showing that the films help reduce the amount of heat released during combustion and help form a char, which helps to extinguish flames [113]. Hao et al. showed in their work that CNT interlayers can be combined with electrical impedance tomography to identify subsurface structural damage in composite materials [114].

Aligned CNT films are usually used as interlayers to maintain the alignmentdependent properties of the CNTs and provide direction or anisotropic properties to the composites they are integrated in. Garcia *et al.* utilized aligned CNT interlayers as joining surfaces between the layers of a FRPC [115]. The study showed that due to the interlayer integration, a 1.5–2.5 times increase in Mode 1 and three times increase in Mode 2 fracture toughness were seen. The aligned CNTs acted as reinforcing agents, which provided a crack bridging effect. The same author showed in another work that the interlaminar fracture toughness increased by 155% [116]. Ni *et al.* showed similar results in a recent work [117], where a so-called nanostitch layer of aligned CNTs was integrated in FRPCs without a change in final thickness. The work showed that the toughness of the CNT layer caused intralaminar fracture to take place as opposed to interlaminar. Villoria *et al.* from the same research group showed that the same interlayers may cause a 30% increase in tension-bearing critical strength, 14% increase in open-hole compression ultimate strength, and an increase in L-section bending energy and deflection by more than 25% [118]. Bhanushali *et al.* reported that by including aligned CNT films into composites as interlayers, the fracture toughness of the composites may increase by ~20-47% but only when interlayer thickness is low [119]. No changes in tensile behavior were reported in the study, but an increase in electrical conductivity was noted. Aly *et al.* showed in their work that the addition of aligned CNT films can increase the compressive strength of FRPCs [120]. In combination with this increase in compressive strength, structural health monitoring for damage could be conducted via the piezoresistive response and gauge factors of between 25 and 45 were reported. The publication showed that a smaller number of films, where the resistance is presumably higher, showed a greater overall piezoresistive response as compared with a large number of films. The response of the films was also asymmetric, with tensile loading leading to an increase in resistance while compression led to a decrease. This was confirmed in cyclic testing, where the cyclic piezoresistive response of the composites was also confirmed. Hallander et al. examined the mechanics of deformation of aligned CNT films in FRPCs and found that there was an increase in both intraply shear stiffness and interply friction when the film is used for composite manufacturing [121]. It was also found that the aligned film was more prone to shear than buckling when subjected to testing.

A number of researchers have identified fields and applications where the multifunctional properties of aligned CNT films may be exploited. Lee et al. proved that the electrical conductivity of such layers can be used for the heating and curing of composites without the need for an external autoclave or oven [122]. The work showed that no significant difference existed for composites made from internal heating of the CNT film embedded within the composite as compared with traditional autoclave or oven heating in terms of degree of cure, dynamic mechanical analysis, shear beam test, and double notch-based tensile testing. The technique, however, did show itself to be more energy-efficient [123]. The same author showed that such aligned CNT films can be used for the *in-situ* cure monitoring of thermoset matrices, often used to manufacture high-performance composites [68]. Work by Tarfaoui et al. exploited the self-heating characteristics of such films for the purpose of de-icing composite structures, showing low heating times with films as low as 60 µm in thickness [124]. Meng et al. demonstrated the combination of aligned CNT films along with glass fiber and Kevlar to produce structural composite batteries, which showed an energy density of \sim 1.4 Wh/kg, an elastic modulus of 7 GPa, and tensile strength exceeding 0.27 GPa [125]. Tensile testing showed stability of operation of the material during uniaxial loading and proved the feasibility of potential application. Aly et al. elaborated on the strain sensing ability of aligned CNT film integrated composites for the purpose of structural health monitoring in the interlaminar regions of FRPCs [126]. The study showed that no major difference in mechanical properties occurred with the inclusion of the aligned CNT films, gauge factors of up to 20 were seen during tensile testing, and that pre-straining the films caused a consecutive increase in sensitivity as cyclic testing progressed. The same author studied the structural health monitoring ability of FRPCs with aligned CNT films in three locations; in the middle of the composite and at the top and bottom most plies [127]. This study showed results pertaining to both monotonic and dynamic flexural loading, with gauge factors of ~6 being recorded. It was also shown that the film on the tension side of the

samples showed a greater sensitivity to loading than the one on the compression side, while the layer in the middle showed greater sensitivity as the load increased. Conway *et al.* reported that the use of aligned CNT films can increase the impact resistance of carbon fiber-reinforced polymers, raising the residual strength by up to 16% [128]. A more exotic application was shown by Li [129], where in combination with carbon fiber sheets, aligned CNT films were shown to be remarkable electrode materials for potential applications in supercapacitors. Besides the low electrical resistance of the material, the electrodes showed good chemical and cyclic stability and strong resistance to water impact owing to the mechanical properties of the structure. In addition to the studies listed, aligned CNT films have been shown to have potential in applications such as the creation of CNT based macrostructures [130], dry electrochemical electrodes [131], composite pressure sensors [132], gas sensors [133], 3-D printing materials [134], thermal management materials [135], and a host of biology-related applications [136–140].

3. Conclusions

This book chapter aims to provide an overview for the techniques used to integrate CNTs into FRPC materials. The chapter overviews notable work regarding the main techniques and focuses on the published results by authors who have built both the foundations of the field and are currently working on the cutting edge of fiber-based integration techniques. The chapter touches upon the trends seen in mechanical properties attained by the various techniques discussed while combining the discussion with multifunctional properties such as electrical conductivity, electromagnetic shielding, flame retardancy, thermal conductivity and management, and sensors based on these materials.

Although a large amount of research work has been conducted on CNTs and their integration in FRPCs, the modern works clearly show that with new characterization techniques and application fields, an increasing number of doors for the practical application of such composites are opening, especially since the field of composites in general is shifting toward the large-scale integration of nanomaterials for smarter, stronger composite systems. Thus, CNTs and their application techniques and final properties of the end nanocomposites are a field that continues to grow and is expected to keep pace of growth for the forthcoming future.

In the near future, the authors see that CNT incorporating nanocomposites, which are hierarchical in nature, utilizing a combination of nanoparticles and multilevel reinforcements are going to become popular and the center of research attention. Combining CNTs with specialized high-performance fibers, shape memory matrices, self-healing nanoparticles, adaptive materials and making them from precursors and matrices which are biodegradable are avenues which the authors expect to be explored in the coming years. Such advanced materials will not only revolutionize the industry, but how we deal with composite lifecycle management and long-term usage.

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Author details

Hassaan A. Butt^{1*}, German V. Rogozhkin¹, Andrei Starkov², Dmitry V. Krasnikov¹ and Albert G. Nasibulin^{1,3}

1 Skolkovo Institute of Science and Technology, Moscow, Russia

2 National University of Singapore, Singapore

3 Aalto University School of Chemical Engineering, Espoo, Finland

*Address all correspondence to: hassaan.butt@skoltech.ru

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Section 2

Adhesives: Science, Technology, and Applications

Chapter 7

Introductory Chapter: The Importance of Adhesives in the World

António B. Pereira and Alexandre Luiz Pereira

1. Introduction

An adhesive is a natural or synthetic product that can hold pieces together, usually by surface connection. These materials can be metals, composites, ceramics, etc., and combinations between ones. Its use has been going on for many years, but from the year 40, the technology of adhesives evolved considerably [1]. It is due to the use of synthetic polymers in the adhesives.

These polymers easily adhere to most materials and can transmit stress considerably. There are several types of adhesives, each of which is naturally more suitable for each application.

The range is extensive, from epoxy, and polyurethane, to polyimide, with one or more components. They can be applied, e.g., in the form of a paste, liquid, film, or pellets. There are hot melt adhesives, reactive hot melt, thermosetting, thermoplastic, pressure sensitive, and contact. The applications can be of the structural kind, in cases where high mechanical strength is usually required, but there are also applications for other purposes, such as silicone sealing. The adhesives may also contain additives in your composition, such as metal nanoparticles (e.g., copper, nickel, or silver), water, oil, etc., to improve their properties and increase their durability.

2. Applications of the adhesives

Aeronautical applications have been one of the main motivations for the development of adhesive technology with the use of adhesively bonded joints. However, today, other areas are also gaining from adhesive bonded technology. They are the automotive, naval, sports industries, and so on. Reducing the weight of an automotive vehicle generates fuel savings, an increase in speed, and a decrease in pollutant levels, for this, lighter materials such as aluminum, composites, and plastics have been used in their projects. The binding of these materials by traditional methods (bolts, rivets, welds, brazing, and other interference connections) is difficult to make, hence the preference for adhesive bonded [2]. Composites and adhesives develop together because bonding with adhesive is better in this case. It is possible to find in the literature several works made of composites reinforced with vegetable fibers and their adhesively bonded [3]. Another example in the naval area is the use of composites or metal-composites glued to repair pipelines, it is due to corrosion resistance and low weight, also the bonding in the composite with adhesive is better [4]. In the industry in general, it has become a common practice to repair pipelines using adhesive joints [5]. In the sports area, equipment such as bicycles, helmets, rackets, etc., that use lighter materials such as plastics and composites in their projects, also use this bonding technology through adhesively bonded joints [1]. Adhesives are used in almost all consumer products. You can bond almost anything, from rock (civil construction, decorative items), and metals to plastics, including natural materials such as sisal fibers. In a general way, the use of adhesives ranges from simple pens to much more sophisticated pieces, like some components of a spaceship.

Adhesive bonded and their projects for adhesively bonded joints are areas that need knowledge of various sciences and technologies: namely, physics, chemistry, mechanics, the study of surfaces, types of polymers for adhesives, the mechanical design of adhesive joints, as well as knowledge of economics [1]. Thus, the study of adhesives is a multidisciplinary area of great growth and technological importance today.

3. Some advantages and limitations of the adhesives

There are several advantages to using adhesively bonded joints, some are uniform stress distribution in the bonded area, vibration dampening, joining on surfaces of different materials, (e.g., vegetable-fiber-reinforced polymer matrix composites bonded with metals [6]), allows joining surfaces with irregular geometries, may be more economically viable [1].

Adhesive applications still have limitations (compared with traditional mechanical methods), therefore, the importance of the study and development of this area. In an adhesive bonded, stresses such as cleavage and peel on adhesive bonded must be avoided, shear stresses are preferable, avoid geometries that present localized stresses, a careful preparation of the surfaces to be bonded (cleaning and degreasing with solvents, abrasion, etc.) [1]. **Figure 1** shows the types of stresses that should be avoided in adhesively bonded joints.

Figure 2 shows a single-lap adhesive joint working in shear stresses. This is a better condition, as the stresses are parallel to the adhesive bonded and are also better distributed.

Mechanical strength in structural metal applications still has limitations. Really, for example, the bonding of two stainless steel pieces is very compromised being done with adhesives. Just think that steel has an ultimate strength of 600 MPa, while the adhesive resists, at most 10% of that, i.e., 60 MPa. In metal of the thickness thin, these limitations are less.



Figure 1. Stresses that should be avoided in adhesively bonded joints: (1) cleavage, and (2) peel.

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Figure 2. Single-lap adhesive joint working in shear stresses.

Author details

António B. Pereira^{1*} and Alexandre Luiz Pereira²

1 TEMA – Centre for Mechanical Technology and Automation, Department of Mechanical Engineering, University of Aveiro, Aveiro, Portugal

2 Federal Center of Technological Education in Rio de Janeiro (CEFET/RJ), Brazil

*Address all correspondence to: abastos@ua.pt

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Chapter 8

Mechanical Strength of Adhesively Bonded Metals

António B. Pereira and Alexandre Luiz Pereira

Abstract

Adhesive joints are nowadays widely used in fields ranging from packaging to aeronautics. Nevertheless, the absence of accurate failure criteria remains an important obstacle that often prevents the use of adhesive joints in structural applications. The main objective of this work is to be an introduction to the subject, and it was for this to evaluate the factors that most influence the strength of overlap adhesive joints.

Keywords: adhesive joints, overlap adhesive joints, strength of adhesive joints

1. Introduction

Glued joints currently have a wide range of applications, from the packaging industry to the demanding aeronautical industry. The characteristics of the so-called structural polymeric adhesives allow the increasing use of primary adhesive joints, that is, connections whose performance is critical for the integrity of the structure in which they are inserted. Among the main advantages of glued joints, we can mention:

- high mechanical strength if the joint is well designed
- weight and number of parts savings compared to bolted and riveted connections
- minimization of corrosion problems, especially in the connections of different materials
- sealing and thermal insulation capacity
- vibration dampening, due to the viscoelastic behavior of the glues
- good resistance to fatigue, to which the absence of holes and the respective stress concentration effect strongly contribute
- good esthetic appearance
- the fact that they are often the cheapest option.

Glued joints are particularly interesting for joining advanced high-strength materials, such as polymer matrix composites. Alternative riveted and bolted connections are much less efficient than metallic materials due to the low ductility and poor crush strength of composites.

Adhesive joints, however, have several limitations:

- the current difficulties in the rigorous design of joints, which lead to the adoption of empirical methods or rather conservative calculation processes
- sensitivity to cleavage loads
- need for cleaning and surface preparation procedures
- the time it may take to develop strength (curing time for thermosets)
- some inspection difficulties
- · impossibility of dismantling without destroying the joint
- sensitivity to environmental exposure (temperature, humidity, UV radiation, etc.) and creep.
- Figure 1 shows the main types of adhesive joints. The most used are single-lap and double-lap joints. Stair and ramp joints have high performance and are mainly applied in aeronautics for parts made of composite materials with a relatively high thickness (above 5 mm) [1]. The manufacturing costs of these joints are much higher than the costs of overlap joints.

Adhesive joints load can be ordered in three main ways, namely (1) shear; (2) tensile; and (3) cleavage (**Figure 2**).

A fundamental principle in the design of bonded connections is that the adhesive should preferentially transmit shear forces. Cleavage loads are highly harmful. Tensile demands are also to be avoided, as unavoidable misalignments cause cleavage efforts.



Figure 1.

The most common types of glued joints: (1) single-lap; (2) double-lap; (3) stair; (4) ramp.

Mechanical Strength of Adhesively Bonded Metals DOI: http://dx.doi.org/10.5772/intechopen.108872



Figure 2. Fundamental loading ways: (1) shear; (2) tensile; (3) cleavage.

It should be noted, however, that, in the overlap joints, there are always localized cleavage stresses.

Joint failure can occur in three ways:

- adhesive breakage, that is, by detachment at one of the adherent/adhesive interfaces
- cohesive rupture of the adhesive
- fracture of one of the adherents.

One of the main causes of adhesive breakage is inadequate surface preparation [2]. The specific action of the preparation normally consists of:

- increasing the surface roughness, in order to promote mechanical contact with the adhesive
- cause chemical changes that favor electrostatic attraction at the atomic level, through van der Waals forces.

The procedures naturally depend on the materials to be connected and are often the subject of standards, which are particularly well established for several metal alloys [3, 4]. The first stage of the preparation is cleaning the surfaces, especially in terms of degreasing, using solvents, detergent solutions, trichloroethane vapor (toxic), ultrasound, etc. The surface roughness can be increased by applying fine abrasive paper or by shot blasting, after which it is necessary to remove the loose particles. In the case of metals, it is recommended to carry out a chemical attack with appropriate solutions, or even electrochemical treatments, as is the case of anodizing Al alloys. The application of primers favors the durability of the connection.

Several studies have already been presented on the effect of surface preparation on failure mode and on the strength of bonded joints [2, 5]. The conclusions, however, do not always go in the same direction, either in terms of failure modes or in terms of the classification of surface treatments. In [5] it is considered that the interfacial rupture is due to deficient bonding procedures, namely inadequate preparation or contamination of the surfaces. However, in [6], where a vast amount of experimental results of glued Al joints were reviewed, there were cases of interfacial ruptures even when sophisticated treatments were used. Interfacial ruptures even seem to be quite frequent after more or less prolonged exposure to environments of relatively high temperature and humidity. In a large-scale study carried out in Japan [7], frequent

adhesive failures were observed in joints with steel adherents. However, according to [5], there are cases of apparent interfacial rupture in which more sophisticated analysis methods allow us to verify the presence of a very thin adhesive layer on the fracture surfaces.

Another factor to be controlled is the thickness of the adhesive layer, for which there is an optimal range, generally between 0.1 and 0.3 mm [8]. The strength of the joint decreases markedly with the thickness of the adhesive layer above certain values, due to the greater probability of the existence of defects. On the other hand, thicknesses that are too thin considerably increase the risk of failure of the adhesive layer. Thickness control can be done through the clamping devices used in the gluing operation. In other cases, small glass spheres can be added to the adhesive that guarantee a certain thickness. The use of adhesives in the form of films allows better control of the thickness of the joint, although with generally higher costs.

Finally, the proper choice of adhesive is critical to joint performance. Structural adhesives are normally thermosetting polymers, as thermoplastics are more susceptible to creep and property degradation from environmental exposure. The most common types of adhesives are epoxides, polyurethanes, modified acrylics, and cyanoacrylates. Epoxy adhesives are the most used, given their good chemical resistance and good creep behavior. There is a great variety of formulations, which are relatively fragile based on, but which become very ductile with the addition of rubber or thermoplastic particles. Curing generally takes place at temperatures between 20 and 120°C, so heating means may be required. Polyurethane adhesives cure by reaction with ambient humidity, have excellent toughness and moderate cost. Resistance to environmental exposure and creep are the main limitations, which are shared by acrylic adhesives, said to be modified, as they are derived from thermoplastic formulations. These, however, have good cleavage strength, moderate cost, and are less demanding in surface preparation. Cyanoacrylates cure quickly and have good cleavage strength, but bond durability is relatively low.

2. Characterization of adhesives

The characterization of the behavior of the adhesives is somewhat delicate. In fact, the most common tests of adhesive joints do not directly provide the mechanical properties of the adhesives, having mainly a comparative or quality control value. Cleavage assays are clearly in this category. **Figure 3(1)** and **(2)** represent the two most common specimens, specified by ASTM D 1876 [9] and ASTM D 3762 [10], respectively. In the first case, the force necessary to progressively break the joint is



Figure 3. Adhesive joint cleavage tests: (1) ASTM D1876; (2) ASTM D3762.

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measured, while in the second test, the advance of the crack in the joint relative to the position of the wedge is normally measured. These tests only allow comparing adhesives and/or surface preparation techniques, as well as evaluating the effect of environmental exposure.

The shear test of simple overlap joints is also widely publicized at ASTM D 1002 [11] standard for metals (**Figure 4**).

The overlap length L is determined in such a way that there is no yielding of the adherents before the joint breaks, since it is intended to measure the ultimate stress at the average shear of the adhesive. Once again, this test has only comparative value, as it does not allow measuring the true shear strength of the adhesive. In fact, the distribution of the shear stress along L is not uniform (**Figure 5**). On the other hand, the eccentricity of the load causes bending of the adherents (**Figure 6**) and cleavage stresses at the ends of the bond.

The tests that allow obtaining the mechanical properties of the adhesives are more complex. The shear strength can be obtained from the so-called "thick tack" test (ASTM D 5656) [12]. It is again a simple overlap joint with 9.5 mm thick adherents to minimize bending deformations and cleavage stresses. The overlap length L is proportionately small (9.5 mm), so that the shear stress distribution is approximately uniform. The use of a strain gauge also makes it possible to obtain the shear modulus of the adhesive, Ga.



Figure 4. ASTM D 1002-10 test sample.



Figure 5.

Distribution of shear stresses in a lap joint.



Figure 6. Bending effect on a simple lap joint.

3. Strength of overlap adhesive joints

There are important difficulties in the design of glued joints. In stress analysis, there is a singularity in the adherent/adhesive interface that makes it difficult to use the stresses obtained with Finite Element (FE) models. Therefore, simplified analyses are normally used, which, despite the inevitable limitations, are still recommended by design codes [13]. These analyses apply mainly to joints with adherents in tensile mode. **Figure 7** shows the case for single-lap joint in shear.

The best-known analysis is that of Goland-Reissner [14], which takes into account the effect of bending in the simple lap joint, but which is clearly unrealistic in assuming linear elastic behavior for the adhesive. Instead, the Hart-Smith analysis [15] considers the plasticization of the adhesive through an elasto-perfectly plastic approach.

In either case, the fundamental dimensioning parameter is the overlap length L. This must be sufficient to prevent failure due to cleavage stresses and that the average shear stress is too high, promoting excessive creep deformations. However, beyond a certain value, there is no advantage in increasing L, as it penalizes the joint in terms of weight without any gains in joint strength. At this stage, the difficulty lies in the absence of a sufficiently stringent failure criterion. Hart-Smith [15] found that, in the short term, joints can reach breaking loads close to the smallest of the following values:

$$P_{1} = \sqrt{2\tau_{p}t_{a}} \left(\frac{\gamma_{e}}{2} + \gamma_{p}\right) E_{i}t_{i} \left(1 + \frac{E_{i}t_{i}}{E_{o}t_{o}}\right)$$
(1)

$$P_{2} = \sqrt{2\tau_{p}t_{a}\left(\frac{\gamma_{e}}{2} + \gamma_{p}\right)E_{o}t_{o}\left(1 + \frac{E_{o}t_{o}}{E_{i}t_{i}}\right)}$$
(2)

However, given the uncertainties, the design philosophy is mainly aimed at guaranteeing the joint's durability and creep resistance. Hart-Smith [15] suggests that the plastic zones at the ends of the joint be dimensioned to fully support the applied load, while the inner elastic zone is reserved to give the joint resistance to fatigue and creep.

Another type of approach to the problem of predicting the rupture of bonded joints consists of the application of fracture mechanic. The most well-known fracture tests are: the "Double Cantilever Beam" (DCB), mode I (**Figure 8**) [16], and the "End Notched Flexure" (ENF), mode II (**Figure 9**) [17].

The aforementioned tests allowed to obtain a failure criterion expressed as a function of the critical rates of energy release GIc and GIIc, as well as the percentage



Figure 7. Single-lap joint.

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Figure 8. DCB test [16].



Figure 9. ENF test [17].

of solicitation modes. This criterion was then applied to predict the failure of overlapping joints.

4. Conclusions

From the literature review carried out, it is evident that there are still many aspects to be clarified in relation to the structural performance of adhesive joints. We highlight three key issues here:

- the relevance of interfacial decohesion as a mode of rupture of adhesive joints
- the characterization of adhesives, to obtain properties that allow their selection for structural applications
- the best approach to predicting joint failure: fracture mechanics or criteria based on maximum stresses/strains.

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Conflict of interest

The authors declare no potential conflicts of interest concerning the research, authorship, and publication of this article.

Author details

António B. Pereira^{1*} and Alexandre Luiz Pereira²

1 Department of Mechanical Engineering, TEMA—Centre for Mechanical Technology and Automation, University of Aveiro, Campus de Santiago, Aveiro, Portugal

2 Federal Center of Technological Education in Rio de Janeiro (CEFET/RJ), Brazil

*Address all correspondence to: abastos@ua.pt

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Chapter 9

The Effect of Different Adhesive Types on Failure Load

Bahadir Birecikli

Abstract

In this study, bonding joint with double zigzag type geometry was used. There are four types of overlap angles in the bonding geometry: 30°, 45°, 60° and 75°, respectively. Composite materials that are made of glass fiber have been used in this adhesive bonding geometry. These materials were produced by using prepreg (pre-impregnated) technique and [0°/90°] of orientation angle. Thickness of composite material is 3 mm. Ductile and brittle-type adhesives were used for the bonding joint. DP460 adhesive type shows ductile material properties while ATLAC580 adhesive type shows brittle material properties. The effect of adhesive type on the failure load was investigated experimentally. Test results demonstrated that failure load values were higher in the ductile-type adhesive.

Keywords: adhesives, composite material, failure load, bonding geometry, tensile test

1. Introduction

Industrial adhesives are a joining method used as an alternative to mechanical joining methods such as bolts, rivets, welding, and soldering. They have found a suitable area for development, as the bonding process is carried out below the melting temperatures of the joined parts. In addition, industrial adhesives do not create stress concentrations that occur in welding, soldering, and other connection types. The use of industrial adhesives, which are used as an alternative to existing bonding methods, is rapidly increasing. There are many applications joining with adhesive, especially in the aerospace, aviation, and automotive industries.

In this study, a mechanical analysis of the bonding joint geometry was realized using different types of adhesives.

The use of adhesive, which is a more efficient joining method, has become common instead of traditional joining methods [1]. An adhesive is defined by ASTM (Standard test method for strength properties of adhesives in shear by tension loading) as "a substance that can hold materials together by surface contact" [2]. In another definition of adhesive, it is a polymeric material that can hold surfaces together and prevents separation when applied to surfaces [3]. Neto et al. [4] carried out an experimental study on the bonding joint of composite materials. They used two different adhesives, brittle and ductile type in a single lap joint with different lap lengths between 10 and 80 mm. It was seen that failure load increased with growing overlap length in the ductile-type adhesive joint. It was determined that 30 mm overlap length failure load occurred in the brittle-type bonding joint. Sawa et al. [5] analyzed the single lap joint formed by different types of adhesives subjected to tensile loads. They showed that the material thickness and the modulus of elasticity have an extremely large influence on the stress distributions in bonding area. Guess et al. [6] studied analytically and experimentally the strength of adhesively bonded single lap joints using two different adhesives. Apalak et al. [7] made analysis on corner joints. Adhesive is considered as a linear elastic material, and its effects on the bonding stresses created in corner joints were investigated.

Pinto et al. [8] experimentally investigated the mechanical behavior of single lap joint under tensile load by using two different adhesives, rigid and flexible. They stated that there was an insignificant decrease in the strength of the joint in the flexible adhesive type, but there was an increase in the bond strength when rigid adhesive was used. Ozel et al. [9] carried out an experimental study on a single lap joint under bending load using two different types of adhesives. They demonstrated that thickness of material has an extremely major effect on bonding joint performance. Wu et al. [10] applied a method they developed on single lap joints formed using different adhesives of different thicknesses. Temiz [11] stress analysis was performed using a flexible and rigid adhesive on single lap joint. It has been shown that the use of flexible adhesive reduces stress concentrations and increases the strength on the bonding joint. Kline [12] studied the effect of thickness on stress distribution in bonding layer. The alteration of stresses along the thickness is considered linear. He investigated the effect of parameters on the stress distribution in the bonding layer. Dean and Duncan [13] prepared bulk samples with thicknesses varying between 0.5 and 4.0 mm and examined whether the mechanical properties of the adhesive change with thickness. They used four different types of structural adhesives. One and two component epoxies, two-component polyurethane, and two-component acrylic adhesives were tested on samples with different thicknesses. According to the tensile test results, they determined that the material properties did not change with the sample thickness.

The goal of this study is to experimentally analyzed the effects of Vinylester Atlac 580 brittle-type adhesive produced by Huntsman Company and DP460 ductile-type adhesive produced by 3 M Company on failure load.

2. Material and method

DP460 is the center formed by epoxy and accelerator in a 2:1 volume ratio. It is used in a facility from metal, ceramics, and glass.

ATLAC 580 is a low-viscosity epoxy-based vinylester resin that is heat-resistant and flexible. It can also be used for wrapping and spraying in fabrication methods. It is resistant to acid and salt solutions. It is cured with accelerator and hardener mixture.

Curing conditions of brittle type of ATLAC580 and ductile type of DP460 adhesives used in the experiment are given in **Table 1**.

The mechanical properties and stress-strain diagram of DP 460 adhesive are taken from Akpınar's [14] doctoral thesis. Also, the mechanical properties and stress-strain diagram of ATLAC 580 adhesive are taken from Adin's [15] doctoral thesis as given in **Figures 1** and **2**.

Type of adhesive	Component	Curing conditions	State
ATLAC580	Epoxy/accelerator	120°C/60 min	Liquid
DP460	Epoxy/accelerator + hardener	100°C/180 min	Liquid

Table 1.

Curing conditions of adhesives.



Figure 1. Stress-strain diagram of DP 460 ductile adhesive

The mechanical properties of ATLAC580 and DP460 adhesives used in the experiment are given in **Table 2**.

The high strength of adhesive bonding joint depends on surface preparation methods. The samples must be cleaned from foreign materials such as oil, dirt, and dust that will prevent adhesion. The surfaces were first washed with pure water and then wiped with microfiber cloths. Then, the surfaces to be bonded with pure alcohol were washed and kept on hold until the alcohol exactly evaporated from the surfaces.

Consistency of the adhesive thickness on the surface is possible with the use of a well-designed mold. For this, the thickness of the adhesive was kept constant at 0.20 mm so that the sample length is kept constant, it was placed in a certain mold.

2.1 Experimental study

The composite plates used in the experiments were cut in CNC milling device in accordance with ASTM standards and in desired geometric dimensions. The length of each test specimen is 250 mm. About 25 mm jaw margin is left for the specimens to be fixed to the tensile test device.



Figure 2.

Stress-strain diagram of ATLAC580 brittle adhesive.

	DP 460	ATLAC 580
Modulus of elasticity; E, Mpa	2077.10	442.46
Poisson's ratio; v, (–)	0.38	0.37
Max. Tensile Stress; σ, Mpa	44.615	40.618

Table 2.

Mechanical properties of ductile and brittle adhesive.

Glass fiber composite materials were used in the experiment. Glass fiberreinforced composite materials are produced as prepreg (pre-resin-impregnated wet fiber). Composite materials were prepared for testing with a sample thickness of 3 mm and fiber orientation [0°/90°] and are given in **Figure 3**.

In the experimental study, four different types of adhesive joints with overlap angles of 30°, 45°, 60° and 75° were used as seen in the **Figure 3**. The bonding length of each angle was kept constant at 60 mm.



Figure 3. Composite samples with four different overlap angles.
The Effect of Different Adhesive Types on Failure Load DOI: http://dx.doi.org/10.5772/intechopen.107335

The Shimadzu AG-X model tensile test device was used in the experiment (**Figure 4**). The device has an integrated extensometer and with a capacity of 100 kN.

The test device was calibrated before it was started. The test device was given a preload of 0.10 Mpa. Experimental tests 1 mm/min carried out at pulling speed as seen in the **Figure 4**. The experiment was terminated after the samples were completely detached from the bonding area.

2.2 Experimental results

Tensile tests were performed at four different overlap angles. Composite materials are produced in [0/90°] fiber orientation and 3 mm sample thick. Failure load values of each sample were determined by experiment. For the precision of these values, three samples of the same bonding type were produced, and the test was repeated. Results in graphs are the average of three test specimens.

Failure load and displacement graphs for DP460 (ductile) and ATLAC580 (brittle) adhesives are shown in **Figures 5–8**.



Figure 4. Tensile test device and composite sample.



Figure 5. Failure load-displacement graph for ductile and brittle adhesive at 30° angle.



Figure 6.

Failure load-displacement graph for ductile and brittle adhesive at 45° angle.



Figure 7.

Failure load-displacement graph for ductile and brittle adhesive at 60° angle.



Figure 8.

Failure load-displacement graph for ductile and brittle adhesive at 75° angle.

Failure loads increased with increasing overlap angle in the same bonding area. Increasing the overlap angle increased the failure load by approximately 81%. The highest failure load value was observed at 75° overlap angle. Test results demonstrated that failure load values were higher in the ductile-type adhesive. The Effect of Different Adhesive Types on Failure Load DOI: http://dx.doi.org/10.5772/intechopen.107335



Figure 9.

Failure load-displacement graph for all overlap angles for ductile adhesive.



Figure 10.

Failure load-displacement graph for all overlap angles for brittle adhesive.

Failure load and displacement graph for entire overlap angles for DP460 (ductile) adhesive is shown in **Figure 9**.

Failure load and displacement graph for all overlap angles for ATLAC580 (brittle) adhesive is shown in **Figure 10**.

The failure load values of the ductile-type adhesive were bigger than the failure load values of the brittle-type adhesive. Modulus of elasticity of the ductile-type adhesive is 2077.10 MPa, while the modulus of elasticity of the brittle-type adhesive is 442.46 MPa.

As a result of the experimental study, it has emerged that the adhesive type has a significant effect on the failure load.

When the whole graphs of both adhesive types were examined, more displacement was obtained in the ductile-type adhesive, while almost half of this amount of displacement was obtained in the brittle-type adhesive.

When the bonding surfaces were examined, cohesion damage was observed in the ductile type of adhesive, and adhesion damage was observed in the brittle type of adhesive.

3. Conclusions

In this study, glass fiber composite materials were used at different overlap angles and with different adhesive types by using a single lap joint exposed to tensile loads. As a result of the experiment, it has been revealed that the failure load increases with the increase of the overlap angle value in the bonding joint geometry.

Cohesion damage was occurred in the ductile type of adhesive, and adhesion damage was occurred in the brittle type of adhesive.

In addition, it can be said that the ductile type of adhesive increases the strength of the bonding joint.

Author details

Bahadir Birecikli Vocational School of Technical Sciences, Batman University, Batman, Turkey

*Address all correspondence to: bahadirbirecikli@gmail.com

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Chapter 10

High-Performance Reworkable Underfill Adhesives Based on Dicyclopentadiene Epoxy Thermoset

Laxmisha M. Sridhar and Timothy M. Champagne

Abstract

A highly reliable and reworkable underfill adhesive based on thermoset epoxy resin possessing thermally reversible dicyclopentadiene (DCPD) moiety is described. The adhesive can be cured rapidly at moderate temperatures resulting in high Tg cured network, which gives high reliability to the bonded semiconductor components. The inherent thermal reversibility of DCPD moiety causes network breakdown at high temperatures enabling easy removal of defective semiconductor chips. A discernible trend between loading level of the thermally reversible epoxy resin and high-temperature die shear strength was observed. Using this novel adhesive system, both high reliability and reworkability can be achieved concurrently, which is normally not possible with other thermoset adhesive systems. The epoxy resin used in the study was scaled up to multi-kg quantities demonstrating industrial applicability of the approach.

Keywords: epoxy, dicyclopentadienedicarboxylic acid diglycidyl ester, thermoset, reworkable, underfill adhesive, thermally reversible

1. Introduction

The popularity of handheld display devices (HHDDs) has made their demand increase dramatically in recent years. Manufacturing throughput has consequently been challenged to meet the growing demand. One area that is particularly troublesome for manufacturers is the treatment and handling of defective semiconductor chips on a circuit board. For instance, during the manufacture of a circuit board subassembly, a multitude of semiconductor devices are electrically connected to the circuit board in chip scale packages ("CSPs"), ball grid arrays ("BGAs"), land grid arrays ("LGAs"), and the like [1]. The board may then be tested to evaluate function and sometimes the board fails. In such cases, it is desirable to identify the semiconductor device that caused the failure, remove it from the board, and reuse the board with the remaining functioning semiconductor devices. This would save cost for the manufacturer of HHDDs.



Figure 1.

A cross-sectional representation of Si die bonded to a circuit board through solder balls without an underfill adhesive.

Ordinarily, semiconductor devices (also called as chips) are connected to electrical conductors such as Cu pads on printed circuit boards (PCBs) by solder connection as shown schematically in **Figure 1A**. The coefficient of thermal expansion (CTE) of the Si die and the solder balls is significantly different. When the resulting subassembly in a HHDD is exposed to mechanical shocks such as vibration, distortion, drop (**Figure 1C**), or rapid temperature change (thermal shock, for example, when a device is left in a car during winter or summer), the reliability of the solder connection between the circuit board and the chip often becomes suspect. There are other modes of failure that can occur in a subassembly such as electrical shorting or stress cracks in solder balls (B & D, respectively, **Figure 1**).

Underfill adhesives are widely used to improve the overall thermal shock resistance, mechanical and electrical reliability of the assembly [2]. After a chip is mounted on a circuit board, the space between the chip and the PCB is filled with an underfill adhesive resin. Once the adhesive is cured, the stress is uniformly distributed throughout the bondline rather than just at the contact point between the solder ball and the chip. This enhances the overall thermal and mechanical shock properties and thus the reliability of the assembly [3, 4]. The adhesive formulation is typically a low-viscosity liquid (<1000cPs), which penetrates the gap between the chip and the PCB by capillary action when dispensed. The circuit board can also be slightly heated to about 50°C to lower the viscosity further and to accelerate the capillary fill process. In a high-throughput assembly, the dispensing and the fill process is completed in less than a minute, which requires low viscosity in the underfill resin. The adhesive resin is typically cured in the temperature range 120–130°C in less than 10 minutes. A picture of two CSPs with cured underfill adhesive is shown in **Figure 2** (adhesive is shown in black color indicated with arrows).

The underfill adhesives are typically thermosetting resin compositions that form cross-linked networks when cured. With conventional thermoset adhesives, it is difficult to remove the chip without damaging the subassembly in the event of a



Figure 2.

Two CSPs with cured underfill adhesive filling the gap between the chip and the PCB (indicated with arrows).

failure of a semiconductor chip on the circuit board. Several approaches have been published in the literature on reworkable, reversible, or degradable thermoset networks used in a number of adhesive applications [5–9]. Reworkability or removability is also a valuable attribute of adhesives used for electronic packaging applications including underfill adhesives [10]. It is highly desirable for an underfill adhesive to provide good electrical reliability, mechanical and thermal shock resistance, while allowing for the semiconductor chips to be easily separated in a defective assembly without causing damage to the circuit board. Several chemistry approaches have been explored to make the underfill adhesive reworkable at high temperature [11, 12]. For good reliability (mechanical and thermal shock resistance), high modulus and Tg are essential for underfill adhesives, which allow them to have lower CTE at the service temperature of the HHDDs or during T-cycle tests (hence reduced mismatch in CTEs between bonded substrates) since the CTE of thermosets increases rapidly above the Tg. However, with conventional thermosets, it is difficult to achieve both high reliability and reworkability without a built-in rework chemistry mechanism. The results shown below demonstrate that by using a carefully designed thermoset resin system, both high reliability and reworkability can be achieved concurrently.

2. Results and discussion

The concept of highly reliable and reworkable thermoset underfill adhesive system is shown schematically in **Figure 3**. The adhesive needs to be a highly cross-linked system (high modulus and Tg) for high reliability at the service temperature of the



Figure 3. Highly reliable and reworkable thermoset adhesive concept. HHDD or during thermal cycle (T-cycle) tests. The thermoset adhesive needs to undergo network break down at rework temperatures (typically around 220°C for this application) for easy removability of the faulty semiconductor chip. The design needs to be such that the rework temperature is sufficiently high so that there is no network breakdown occurring during adhesive curing or thermal cycling reliability tests. Typically for T-cycle tests, the bonded components are subjected to a temperature ramp from -40°C to 80°C (thermal shock) with a 15-minute hold time at each temperature. For good reliability performance, 1000 T-cycles without electrical failure are required, and hence, the need for a high-performance underfill adhesive system.

2.1 Resin system design

For the resin system design, the built-in reworkable or reversible chemistry was carefully chosen so that the cross-linked polymer network breakdown takes place at sufficiently higher temperature than that used for adhesive curing. The dicyclopentadiene backbone was chosen because of its high Tg and relatively high retro-Diels-Alder temperature. Based on previous literature report on activation energy required for retro reaction [13], we sought to incorporate electron withdrawing ester groups on dicyclopentadiene double bonds such that the network breakdown occurs at the correct temperature range for good reworkability. Thiele's acid 1 (Figure 4) was chosen as starting material for the resin design. The synthesis of this acid was first reported more than a century ago [14], and design of thermally reversible polymeric system based on alkylation of potassium salt of this acid with dihalides has been reported [15]. A cyclic diester of this diacid, also known as "mendomer," has been made and used in thermally remendable composite design [16]. The temperature used in this study for remendability was above 150°C, which is consistent with the expected retro reaction temperature of dicyclopentadiene-based systems. Prior to our discovery, the diglycidyl ester 2 and its use in reworkable underfill adhesive system have not been reported [11].

For the proof-of-concept study, the diacid **1** was made following a literature process by reaction of sodium cyclopentadienide (supplied as a THF solution, Boulder Scientific Company) with dry ice followed by acidification [17]. The diglycidyl diester **2** was synthesized by reacting the potassium salt of **1** with epibromohydrin in DMSO as solvent in 60–70% yield [11]. The literature process for making diacid **1** resulted in several isomers with the isomer represented by structure **1** being the major component. Since diacid **1** was a mixture of isomers, synthesis of **2** also resulted in a mixture of isomers. Both **1** and **2** were made in multi-kg scale in a production plant demonstrating industrial scalability. However, significant process change was necessary for the scale-up of **1** in the plant. The diacid **1** has been obtained as a single isomer before by a multistep process, but the synthesis used a different synthetic route [18]. During the process development for **1**, a proprietary isomerization process was developed to



Figure 4. Synthesis of diglycidyl ester **2** from diacid **1**.

convert mixture of isomers into mainly one isomer represented by the structure **1**. This allowed for better characterization of the diacid by ¹H NMR and other analytical techniques. The ¹H NMR of **1** thus obtained (**Figure 5**) matches closely with that reported in the literature [18]. For better structural confirmation, the diglycidyl ester **2** was also made starting from single isomer of **1**. While trace isomerization was observed during the synthesis, the ¹H NMR of **2** matches well with the assigned structure and shows predominantly one isomer (**Figure 5**). During formulation and testing, no significant dependence of isomer ratio in **2** on reliability or reworkability performance was seen. This is likely because the reactive epoxy functionality is remote from the adduct forming double bond carbon centers (sterics) to be consequential for polymerization reactivity. Also, all of the reported main isomers of **1** have the two carboxylic acid groups on the norbornene and cyclopentene rings of the DCPD unit, and thus, they are not expected to significantly affect the network breakdown [19].

Both **1** and **2** were analyzed for weight loss using thermogravimetric analysis (TGA). The onset of weight loss began around 160–170°C for both compounds consistent with the expected retro reaction temperature of the dicylopentadiene units. While the TGA weight loss progressed up to a temperature of about 250°C for both resins, it reached a plateau for **2** above this temperature (**Figure 6A**). It is likely that onset of homopolymerization of epoxy functionality in **2** at higher temperature stabilizes the weight loss while no such effect is possible for diacid **1**.

To investigate if facile uncatalyzed homopolymerization of epoxy functionality in **2** takes place above 200°C, a differential scanning colorimetry (DSC) thermogram



Figure 5.

¹H NMR spectra of **1** (CDCl₃ with two drops of DMSO d6) and **2** (CDCl₃). ¹H NMR spectra were run on Varian 300 MHz instrument.



Figure 6.

A) TGA thermograms for 1 & 2 (Tests performed using Discovery TGA 55 instrument at a ramp rate of 10°C per minute). B) DSC thermogram for neat resin 2 at a ramp rate of 10°C per minute (DSC was run using Q-100 DSC from TA Instruments).

was run using neat resin without any added epoxy hardeners at a scan rate of 10°C/ minute. The DSC thermogram shows an exotherm with a peak position at 272.86°C (**Figure 6B**). The magnitude of the exotherm indeed confirms facile homopolymerization of the epoxy group taking place above 200°C without the need for added hardeners. The onset of exotherm is around the same temperature as the weight loss was seen stabilizing for resin **2** in the TGA (**Figure 6A**).

2.2 Development of underfill formulations

Epoxy resin 2 was formulated in several epoxy-only and epoxy-acrylic underfill formulations (**Table 1**). Typical epoxy-only underfill formulations contain bisphenol-A epoxy resin and bisphenol-F epoxy resins, which contribute to high Tg and modulus of the cured adhesives. The use of reactive diluents such as 4-tert-butylphenyl glycidyl ether is essential to lower viscosity to below 1000cPs for better capillary flow. The epoxy formulations also contain a blend of epoxy-imidazole adduct hardeners to balance cure rate and work life. Several epoxy-only formulations were developed (F1, F2, F3, and F4) by using varying levels of the key epoxy resin 2. An epoxy-acrylic formulation F5 was also developed consisting of high Tg acrylic cross-linker such as tricyclodecane dimethanol diacrylate (saturated DCPD backbone), a high Tg acrylic diluent such as isobornyl methacrylate, and hybrid resins such as glycidyl methacrylate, which presumably links the epoxy and acrylic networks to form interpenetrating type networks (IPN). A radical initiator along with a combination of epoxy hardeners was also used for the curing of epoxy-acrylic hybrid formulation F5. Table 1 shows several epoxy-only and epoxy-acrylic hybrid formulations (F1-F5) where the amount of the key reworkable resin 2 was varied to study its impact on properties such as Tg, modulus, adhesion, and reworkability. In formula F5, 5% of 2 was used for direct comparison with the corresponding epoxy-only formula F3. Formula F5 also contained 5% of commercially available cyanate ester bisphenol E cyanate ester. Cyanate esters have been known to co-cure well with epoxy resins, and they help lower the viscosity and improve the Tg and modulus of cured networks [20]. The formulations also contain additives such as carbon black color, dispersants, inhibitors, and silane adhesion promoters.

For good mechanical and thermal shock resistance, the underfill adhesive needs to exhibit a stable storage modulus in the service temperature and T-cycle test

Materials	Formula function	Epoxy-only formulas				Epoxy-acrylic formulas	
	-	F1	F2	F3	F4	F5	
Bisphenol A epoxy	Resin	41.4	42.3	43.9	45.4		
Bisphenol F epoxy	-	10.6	11.7	13.1	14.6	10.0	
2	-	10.0	8.0	5.0	2.0	5.0	
Naphthyl 1,6-diglycidyl ether	-					20.0	
Trifunctional epoxy	-					5.0	
Tricyclodecane dimethanol diacrylate	-					9.9	
Isobornyl methacrylate	-					5.0	
Bisphenol E cyanate ester	-					5.0	
p-tert-Butylphenyl glycidyl ether	Diluent	15.3	15.3	15.3	15.3	6.0	
Epoxy-imidazole adduct	Hardener	16.7	16.7	16.7	16.7	20	
Cationic polymer	Dispersant	0.7	0.7	0.7	0.7		
Ethyl/ethyl-hexyl acrylate copolymer	_					0.4	
Epoxy silane	Adhesion Promoter	0.7	0.7	0.7	0.7		
Carbon black in epoxy	Color	0.9	0.9	0.9	0.9	5	
Dicyandiamide	Hardener	3.6	3.6	3.6	3.6		
Barbituric acid	Inhibitor	0.1	0.1	0.1	0.1		
Butylated hydroxytoluene (BHT)	-					0.1	
Glycidyl methacrylate	Crosslinker					8	
tert-Butyl peroxy-2-ethylhexanoate	Radical initiator					0.6	
Total		100	100	100	100	100	

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Table 1.

Epoxy-only and epoxy-acrylic underfill formulations containing key reworkable resin 2.

temperature range (-40 to 85°C). Since underfill adhesives distribute stress along the bondline rather than at the point of contact between the die and solder ball, the mechanical reliability improvement by their use has been well established (1, 2). Higher Tg and modulus in the adhesive further improve the mechanical reliability due to lower CTE mismatch below the Tg of the cured adhesive. At rework temperatures (typically > solder melting temperature), a low modulus adhesive would facilitate easy removal of defective chips. The storage modulus vs temperature plots for cured samples of formulations F-1 to F-4 were run using dynamic mechanical analysis (DMA) and compared with controls (**Figure 7**). During a typical application process with low volume applied, the adhesives were cured at 130°C for 10 minutes. For material property testing, the same temperature conditions were used but with longer cure times to ensure full cure. Two control formulations, Loctite Eccobond UF 3800 (epoxy-acrylate hybrid underfill) and Loctite Eccobond E-1216M (epoxy-only



Figure 7.

Storage modulus vs temperature plots for formulas F1, F2, F3, F4, and controls UF3800 and E1216M. ASTM D5023 method was used for DMA analysis.

underfill), were used for the initial screening work. UF 3800 has inferior reliability because of its lower Tg, and it shows rapid decrease in storage modulus beginning around 70°C (Figure 7), which is well within the temperature range used for T-cycle reliability tests (-40°C to 85°C). This formula however has very good reworkability as it displays low modulus at the rework temperature (220°C). In contrast, formula E1216M exhibits stable modulus in the temperature range -55 to 120°C (good reliability), but its high temperature modulus is higher than UF3800, which is consistent with its poorer reworkability. It is important to note that the good reworkability seen with UF3800 formula (discussed later in the chapter) is a manifestation of relatively low modulus at higher temperatures. This formula does not contain any resin with built-in chemistry that causes network breakdown at higher temperatures. All the new epoxy-only formulations (F1, F2, F3, and F4) show a storage modulus profile similar to the highly reliable E1216M in the temperature range –55°C to 85°C (**Figure 7**). As expected at high temperatures, these formulas show storage moduli much lower than the best reworkable control UF3800, which indicates superior reworkability. The drop in storage moduli at higher temperatures corresponds well with the relative level of epoxy resin 2 present in these formulas. F-1 with the highest level (10%) shows the lowest high temperature modulus while F-4 with lowest level (2%) shows the highest modulus. The magnitude of modulus decreases seen at higher temperature with increased loading of 2 is consistent with expected higher network breakdown caused by the retro Diels-Alder reaction.

The DMA storage modulus plot for formula F-4 with the lowest level of resin **2** (2%) was also compared with three benchmark underfill formulas, UF3810, UF3808, and UF3800. None of these benchmarks have a built-in chemistry feature, which

causes network breakdown at higher temperatures. As discussed previously, UF 3800 shows good reworkability at high temperature and has inferior reliability among the controls. In contrast, UF3808 has highest reliability and lower reworkability. UF3810 has an intermediate balance of both reliability and reworkability. The reliability and reworkability performance of these benchmark formulas have been well established by their use in commercial products. The mechanical reliability (shock, vibration, and drop resistance) performance of UF3808 and UF3810 correlates well with their higher Tg and modulus profiles shown in **Figure 8**. The T-cycle reliability performance of these three formulas also correlates well with their storage modulus profiles and is discussed separately later in this chapter. As compared with the benchmark formulas, F4 shows stable storage modulus over a wider temperature range (Figure 8B), which suggests superior mechanical and T-cycle reliability. A closer examination revealed that in the temperature range 130–180°C, F4 exhibited sharper modulus decrease than benchmarks. This phenomenon cannot be explained entirely by expected modulus drop typically seen above the Tg of the cured networks and is strongly indicative of network breakdown occurring in this temperature range. A similar modulus trend was also seen with epoxy-acrylic hybrid formula F5 (Figure 8A). It appears that the impact of resin 2 loading in F5 (5%) on storage modulus at higher temperature is lower as compared with the corresponding epoxy-only formula F3, which contains the same amount of **2** (Figure 7). The likely reason for this is discussed separately in the following section.

Table 2 shows comparison of Tg and storage modulus numbers (MPa) at different temperatures (-75°C, 25°C, 85°C, 125°C, and 220°C) for formulas F1–F5 along with those for controls. The relative level of epoxy resin 2 in formulas F1–F5 did not appear to affect the Tg of cured networks significantly as measured by DMA. Formulas F2, F3, F4, and F5 show similar Tg as the highly reliable UF3808 control. In addition, they also exhibit relatively stable modulus numbers in the temperature range -75°C to 125°C similar to or better than UF3808 indicating good reliability performance. The storage modulus numbers for F1–F5 formulas at 220°C correlate well with the relative loading of resin 2, as discussed previously. It is interesting to note that UF3800, which has proven reworkability in commercial HHDD products, has a higher modulus number at 220°C than formulas F1–F5. These results strongly correlate with superior reworkability for F1–F5 formulas arising from adhesive network breakdown caused by the retro Diels-Alder reaction. The reworkability tests performed on these formulas (discussed later) confirm this further.



Figure 8.

Comparison of storage modulus of formulas F4 and F5 with control formulas. ASTM D5023 method was used for the DMA analysis.

Formula	E'(MPa) (-75°C)	E'(MPa) (25°C)	E'(MPa) (85°C)	E'(MPa) (125°C)	E'(MPa) (220°C)	Τg (tanδ)
F1	2757	2159	1871	662	11.6	140
F2	3069	2322	2024	994	16.5	143
F3	2713	2092	1869	1188	22.4	145
F4	2627	1986	1807	1326	33.7	148
F5	3241	2507	2005	809	40.6	147
UF3800	2459	1860	1110	185	57	124
UF3808	3320	2502	2059	945	106	145
UF3810	4160	3031	1369	451	72	145
E1216M	3750	2858	2653	1452	88	144

Table 2.

Storage modulus numbers at different temperatures and Tgs for formulas F1–F5 and those for controls. ASTM D5023 method was used for the DMA analysis.

2.3 Die shear adhesion properties

An important property of underfill adhesives is good die shear adhesion at room temperature (25°C) and the peak temperature experienced during an assembly reflow process (260°C). In contrast, reworkable underfill adhesives need to exhibit high die shear adhesion at room temperature for high reliability performance while that measured at higher temperature needs to be low enough for easy removal of faulty chip at the rework temperature (220°C), yet high enough to prevent any solder extrusion during a reflow step (< 1–2 min @ 260°C). The die shear tests were performed on Dage 4000 instrument from Dage Precision Industries following MIL-STD-883 2019.9 die shear method. The 25°C die shear tests were completed using 3 mm² size SiN dies on bismaleimide-triazine (BT) substrate. Adhesion at 260°C using the same 3 mm² size dies was too low among all underfills for direct comparison. Therefore, 260°C die shear tests were performed using 7.6 mm² size dies for better response and comparison of underfill adhesion. The die shear adhesion of reworkable formulas F1–F5 was measured and compared with controls.

Figure 9 shows the comparison of die shear strengths at room temperature. Formulas F1–F5 show similar or superior die shear strength than the highly reliable underfill benchmark UF3808 while another control formula UF3800 shows the lowest strength among formulas tested. There appears to be no discernible trend between loading level of resin 2 and die shear adhesion at room temperature. However, results from the die shear tests performed at 260°C discussed next show a clear trend, which further confirms network breakdown happening at higher temperature in formulas F1–F5.

The die shear tests performed at 260°C further corroborate the storage modulus results shown in **Figures 7** and **8**. The 260°C die shear strengths for formulas F1–F5 and their comparison with benchmarks are shown in **Figure 10**. The shear strengths correlate well with the amount of reworkable resin **2** used in the experimental formulations. F1, which uses the highest (10%), shows the lowest while F4, which contains the lowest amount (2%), shows highest die shear values. These results further confirm that the network breakdown arising from the retro Diels-Alder reaction of the DCPD moiety takes place at high temperature resulting in lower bond strengths in the



Figure 9.

Die shear strength comparison of F1–F5 with benchmark formulas (UF3800, UF3808, and UF3810) at 25°C. MIL-STD-883 2019.9 die shear standard was used for the die shear tests using 3 mm² size dies on BT substrate. The tests were performed at a load of 0.5 kg and speed of 13.77 mil/sec at a height of 11 mil (settings on Dage 4000).





Figure 10.

Die shear strength comparison at 260°C for formulas F1–F5 and benchmark underfill formulas following MIL-STD-883 2019.9 die shear standard. The tests were performed using 7.6 mm² size dies on BT substrate at a load level of 0.5 kg and a head speed of 13.77 mil/s at a height of 11 mil.

adhesive. The results are also consistent with the storage modulus results discussed previously for these formulas. The only slight deviation appears to be the hybrid epoxy-acrylic formula F5, which shows higher 260°C die shear adhesion as compared



Figure 11. DSC thermogram for neat resin **2** in the presence of 10 wt% of tert-butyl peroxy-2-ethylhexanoate. DSC thermogram was run using Q-100 DSC from TA Instruments at a scan rate of 10°C/minute.

with the corresponding epoxy-only formula F3 both of which have the same amount (5%) of epoxy resin 2. While the formulation components are different, the higher 260°C die shear value seen with formula F5 is likely resulting from relatively lower network breakdown occurring at this temperature. Presumably, the norbornene double bond in 2 (more strained as compared with cyclopentene) undergoes some radical copolymerization with acrylic components in F5 effectively lowering the level of thermally reversible adducts in the cured network. To validate this, a DSC thermogram of neat resin 2 was run in the presence of 10% tert-butyl peroxy-2-ethylhexanoate radical initiator. The resulting thermogram (Figure 11) showed an exotherm with a peak at 132.96°C that can be ascribed to the radical homopolymerization of the norbornene double bond while no such peak was seen in Figure 6B discussed previously. While the magnitude of this exotherm is relatively small (116 J/g), it is likely that the norbornene double bond possessing an electron-withdrawing ester group (as present in structure 2) would exhibit significantly higher reactivity in radical copolymerization with other acrylic components. Radical copolymerization reactivity of norbornene double bond connected to electron withdrawing ester group has been reported before [21].

2.4 Reworkability study

Select experimental underfill formulas F1 (contains 10% of 2), F4 (2% of 2), and F5 (5% of 2, epoxy-acrylic) were tested for reworkability and compared with controls. An underfill board array containing 0.4 mm CSP (chip scale package or simply called chip, die or semiconductor or component) test board (ID# ACEM 94V0 1612, nomenclature 1502009) was used for the study. Sample pictures of the (**A**)

unpopulated test board, (B) populated and underfilled board, and (C) zoom-in of a single underfilled die assembly unit are shown in **Figure 12**. When a faulty chip is identified after assembly in such a board with a multitude of semiconductors, the chip needs to be removed, residues cleaned up, and the chip assembly process is repeated so that board can be reused again. Figure 13 illustrates the whole rework process. (A) A typical rework station used for this study, (B) chip removal tool with a focused heating element, (C) the scavenger nozzle for removing glue and solder residues after chip removal, (**D**) diagram of the scavenger nozzle illustrating the heating and vacuum suction to remove residues. A typical rework process consists of localized heating of the circuit board on a hot plate (heated to around 180-200°C), and the faulty die on top is heated using a hot air nozzle (typically around 220°C, picture B, Figure 13) for a few seconds. The die is then removed using suction tool optionally with additional force using a metal tool. The adhesive and solder residues are then cleaned using suction nozzle (picture C, Figure 13) to make the board reusable again. When a good reworkable adhesive is used, the whole rework process is typically completed in under 2 minutes.

Table 3 shows qualitative evaluation guideline and score card for the board rework process. The rating of the rework process considers a multitude of factors such as ease of die removal, amount of underfill left on the board after cleaning, number of pads and traces damaged on the board, total cleaning time, and solder mask damage, each with its own weightage (total adds up to 1). The ease of rework is rated on a scale of 1–10 where a rating of 1 indicates poor reworkability and 10 best reworkability.

Benchmark underfill formulas UF3800, UF3808, and E1216M were compared with F1, F2, and F5 formulas for reworkability performance. A test board similar to that shown in **Figure 12** was used to bond the chips using benchmarks and the experimental formulas. **Table 4** shows total reworkability score for these formulas involving a multitude of factors discussed before. As expected, UF 3800 showed a high rating of 8 while the other two benchmark formulas UF3808 and E1216 formulas



Figure 12.

Photographs of (**A**) unpopulated 0.4 mm BGA test board used for reworkability and reliability study; (**B**) test board array populated with 6 mm² WLCSP (wafer level chip scale package) and underfilled; (**C**) zoom-in of a single, underfilled WLCSP on test board.



Figure 13.

Photographs of (A) a typical rework station; (B) chip removal tool with focused heating element; (C) the scavenger nozzle for removing glue and residues after component removal; (D) diagram of the scavenger nozzle illustrating the heating and vacuum suction.

fared poorer for reworkability. The reworkability score for formulas F1, F4, and F5 was similar to or better than UF3800. The hybrid epoxy-acrylic formula F5 showed slightly inferior reworkability score than F1 and F2. The likely reason for relatively lower rework score for F5 as compared with F1 and F4 was discussed in the previous section and is suspected to be from the norbornene double bond copolymerization, which would result in partially (depending on extent of copolymerization) non-cleavable networks.

Figure 14 shows pictures of the test board after removal of the bonded die for the control formulas UF3800 and UF3808. The images on the left side show the substrate after removal of the die before rework and cleaning while those on the right show substrate board after cleaning. As expected, UF 3800 results in a clean board after die removal, rework, and cleaning. In contrast, UF 3808 causes damage to the board during die removal and leaves lots of adhesive residue even after cleaning. **Figure 15** shows images of the substrate board after die removal (on the left side) and after the rework process and cleaning (on the right) for formulas F1, F4, and F5. Consistent with the storage modulus profile and high-temperature die shear results discussed previously, all of these formulas enable easy rework process that is similar to or slightly better than reworkable UF3800 benchmark as evidenced by the clean substrate board obtained after the rework process. This result further demonstrates that resin **2** with built-in thermally reversible DCPD moiety enables good reworkability of underfill formulations.

Weight	Score	1	3	5	7	10
0.3	Ease of removal of die	Suction+high force, cannot be removed	Suction+high force to turn & remove	Suction+medium force to turn & remove	Suction+low force to turn & remove	Removes on suction
0.2	Amount of UF left after cleaning	>75%	50-75%	25–50%	0-25%	0%0
0.1	# of pads damaged	>9	6–9	3–6	1–2	0
0.1	# of traces damaged	>9	6–9	3–6	1–2	0
0.2	Time to clean	>150s	120–150s	90–120s	45–90s	<45s
0.1	Solder mask damage	Significant damage (>15% area)	Damage 10–15% area	Damage 5–10% area	Damage to <5% area	No damage

Table 3. Qualitative evaluation guideline and score card for board rework. Higher score indicates easier rework process.

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Formulas	UF3800	UF3808	E1216M	F1	F4	F5
Resin 2 content	0	0	0	10	2	5
Amount of UF left ¹	1.33	0.87	0.93	1.07	1.4	1.13
Ease of Removal ²	1.95	1.55	1.50	2.70	2.10	1.95
# Pads Damaged ³	0.85	1.00	0.90	0.92	1.0	1.0
# Traces Damaged ⁴	0.95	1.00	0.90	0.95	1.0	1.0
Time to clean ⁵	1.90	0.60	1.70	2.00	2.0	1.70
Solder mask damage ⁶	1.0	0.62	0.5	0.92	1.0	0.95
Final rework score	8.0	5.6	6.4	8.6	8.5	7.7

¹*Highest score is 2. Higher number indicates lower amount of underfill left after die removal.*

²Highest score is 3, higher number indicates easier removal of die.

³Highest score is 1, higher number indicates less damage to the pads on the board.

⁴Highest score is 1, higher number indicates less damage to the traces on the board.

⁵Highest score is 2, higher number indicates less time taken for cleaning.

⁶Highest score is 1, higher number indicates less solder mask damage on the board.

Table 4.

Rework test scores for control formulas and F1, F4, and F5 formulas (higher total score indicates superior reworkability).



Damage to the board

Figure 14.

Picture of substrates after removal of semiconductor before rework and cleaning for controls UF 3800 (top left) and UF3808 (bottom left). Picture of substrate board after underfill and solder residue removal for UF 3800 (top right) and UF3808 (bottom right). The marks indicated by the arrow are caused by removal of the adhesive that is not reworkable (UF3808).

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Figure 15.

Picture of substrates after removal of die before rework and cleaning for F1, F4, and F5 (on the left side). Picture of substrate board after cleaning (underfill and solder residue removal) for the same formulas (on the right side).

2.5 T-cycle reliability tests

Since the HHDDs with bonded semiconductor components can be subjected to thermal shocks as described previously, the bonded components were tested for T-cycle reliability using an underfill board array containing 15 bonded semiconductor components similar to that shown in **Figure 12**. The reliability test was performed by using a 30-minute temperature ramp from -40°C to 85°C per cycle with a 15 min hold time at both temperatures (-40°C and 85°C). Air-to-air thermal cycling where the bonded parts cycle between an oven chamber and a freezer chamber in air was used for the study. The cycle is repeated several times, and the semiconductors on the board were tested for electrical failure. For good T-cycle reliability performance of HHDDs, no chip failure up to 1000 cycles is required.

Formula	200 cycles	400 cycles	600 cycles	800 cycles	1000 cycles	1200 cycles	2000 cycles
UF3800	0/15	0/15	3/15	7/15	9/15	10/15	15/15
UF3808	0/15	0/15	0/15	0/15	0/15	0/15	0/15
E1216M	0/15	0/15	0/15	0/15	0/15	0/15	0/15
F1	0/15	0/15	0/15	0/15	0/15	0/15	0/15
F4	0/15	0/15	0/15	0/15	0/15	0/15	0/15
F5	0/15	0/15	0/15	0/15	0/15	0/15	0/15

Fifteen bonded semiconductors were tested together for each formulation and the number of failures is indicated on the left side out of the total 15 components tested.

Table 5.

Air-to-air thermal cycling $(-40^{\circ}C \leftarrow \rightarrow 85^{\circ}C, 30 \text{ min/cycle}, 15 \text{ min hold}).$

Table 5 shows comparison of reliability performance of three benchmark formulas and the experimental underfill formulas F1, F4, and F5. The highly reworkable UF3800 formula started showing chip failures after 600 T-cycles (three failed chips) and complete failure of all the bonded chips after 2000 T-cycles (Table 5). In contrast, the highly reliable UF3808 and E1216 controls showed no failures up to 2000 T-cycles. As expected, none of the experimental formulas F1, F4, and F5 showed failures up to 2000 cycles indicating that the T-cycle reliability is similar to the highly reliable UF3808 and E1216M. The loading level of thermally reversible resin 2 did not appear to impact the reliability performance (compare F1 vs F4, **Table 5**). This result is consistent with reliability model established based on the storage modulus results discussed previously (Figure 7). As noted previously, the cured underfill adhesives also improve the mechanical reliability performance (shock, vibration, and drop) by distributing stress uniformly through the bondline. Higher Tg and modulus adhesives improve the reliability further similar to the reliability model shown before for T-cycle. Thus, the results discussed in this chapter demonstrate that using resin 2 with built-in reworkability feature, both high reliability and reworkability can be achieved concurrently with underfill formulations.

3. Conclusions

Synthesis of a new diglycidyl ester epoxy resin possessing dicyclopentadiene backbone was described. The resin was formulated in underfill adhesive formulations to provide cured adhesives with high Tg and modulus. Clear dependence of loading level of thermally reversible resin 2 on high-temperature die shear strength was seen. The formulated adhesives provided high mechanical and T-cycle reliability performance to the bonded semiconductor components consistent with the reliability model established based on storage modulus results. At high temperatures, the retro Diels-Alder reaction of the DCPD unit caused network breakdown enabling easy removability/reworkability of the bonded components. The network breakdown occurred at sufficiently above the cure temperature of the adhesive to not interfere during the cure of the adhesive. Thus, using this novel adhesive system, both high reliability and reworkability can be achieved simultaneously, which is generally not possible with

other thermoset adhesive system. The key epoxy resin was scaled up to several kg's demonstrating industrial applicability of the high-performance reworkable adhesive technology.

Conflict of interest

The authors declare no conflicts of interest.

Author details

Laxmisha M. Sridhar^{1*} and Timothy M. Champagne^{2*}

1 Henkel Corporation, Rocky Hill, CT, USA

2 Henkel Corporation, Irvine, CA, USA

*Address all correspondence to: laxmishasridh@gmail.com and timothy.champagne@henkel.com

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Composite materials have become an optimal option for a range of modern, industrial, clinical, and sports applications, offering a material with the desired properties and a limitless choice of building and composite levels. They possess noteworthy physical, thermal, electrical, and mechanical properties, in addition to being lightweight and cost-effective. This book focuses on the next generation of fiber-reinforced composites and adhesives, including recent advances in their development as well as their applications in numerous fields.

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