Polymer Composites Carbonaceous Materials Coated

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Carbon fibre reinforced polymer composites have high mechanical properties that make them exemplary engineered materials to carry loads and stresses. Coupling fibre and matrix together require good understanding of not only fibre morphology but also matrix rheology. One way of having a strongly coupled fibre and matrix interface is to size the reinforcing fibres by means of micro- or nanocarbon materials coating on the fibre surface. Common coating materials used are carbon nanotubes and nanofibres and graphene, and more recently carbon black (colloidal particles of virtually pure elemental carbon) and graphite. There are several chemical, thermal, and electrochemical processes that are used for coating the carbonous materials onto a carbon fibre surface. Sizing of fibres provides higher interfacial adhesion between fibre and matrix and allows better fibre wetting by the surrounded matrix material.

polymer composites carbon fibre

carbon nanotube coating

graphene coating

1. Introduction

Carbon fibre (CF) reinforced polymer composites have been consistent in gaining high importance in the area of composite science and technology ^{[1][2][3]}, particularly in aerospace ^[4], defence ^[5], sports goods ^[6], and automotive industries ^[2]. The combination of polymer matrix and reinforcing fibres results in high-performance materials that offer weight reduction of more than 50% compared to those of aluminium and steel, respectively ³. CF reinforcement commands the strength and modulus properties of CF reinforced polymer composites and is the primary load element. The composite material performance usually depends on the interfacial characteristics of the reinforcement and matrix material ^[8]. It is imperative to have a high-volume fraction of fibres (around 55–60%) to obtain high mechanical performance ^[9]. Nevertheless, the low interfacial strength and the shortage of interfacial covalent bonds are caused by the even and unreactive characteristics of the CF surface, thereby affecting the mechanical properties of composites [10][11]. It is a long, prevailing, and highly important issue needed to address for the continuous progress of CF reinforced polymer composites in prospective advanced applications. Several research works have been focusing on examining and comprehending the physicochemical interactivity at the fibre/matrix interface [12][13][14][15][16]. The sufficient amount of hydrogen bond and Van der Waals forces between the CF and matrix are essential throughout composite processing for strong interfacial adhesion. The fibre/matrix interfacial adhesion energy should be higher than the cohesive energy of the matrix [17][18][19][20]. The highperformance polymeric composites' mechanical properties can be improved by modifying CF surface and structure ^{[21][22]}. Still, the importance of controlling the fibre/matrix interfacial characteristics is a key task. CF has a non-polar surface with the structure of crystallized graphitic basal planes. The chemical inertness of the CF is caused by the high-temperature carbonization or graphitization during manufacturing ^[23]. In addition, inadequate bonding with the matrix materials during the manufacturing is triggered by the surface lipophobicity, extreme smoothness and low adsorption characteristics of CF ^[24]. Thus, strong fibre/matrix interfacial adhesion for successful transfer of stress at the interface is executed by the modifications at the fibre surface to resolve the inertness of CF hierarchically reinforced composite structures ^{[25][26][27][28][29][30]}. Significant advancements have been attained in CF reinforced composites for the fibre/matrix interfacial strength and matrix governed by the thickness performance, for example, fracture toughness, fatigue life, impact strength, and interfacial shear strength on the basis of their distinctive structures and modulus and outstanding strength ^{[31][32][33]}.

Sizing technology has been recently appraised for the modification of CF surface through micro- or nanocarbon materials coating and improvement of fibre/matrix interfacial adhesion ^[34]. In regard to sizing, in the presence of fibres, retardation of the main crack and an increase in fracture work because of the debonding of fibre and matrix pull out can be possible due to the dissipation mechanism of main energy under mechanical load ^[35]. It is important to ensure good adhesion of coating onto fibre (in this limiting case; chemical bonding) for resisting the fibre oxidation, otherwise the chance of fibre getting deboned and pull out from the matrix will be higher ^{[15][36][37][38][39]} ^[40].

The technologies of fibre coating were established a long time ago for numerous purposes. Carbon nanomaterials such as carbon nanotubes (CNTs), carbon nanofibres (CNFs), graphene, carbon black, and graphite have been popularly employed [41][42][43][44][45][46][47][48][49]. Of them, CNT and graphene have increased noteworthy developments in the electrical characteristics, tensile strength, and barrier performance of fibre [50][51]. CNTs are the precursor in this regard, due to their extraordinary inherent properties; for example, mechanical, physical, electrical, thermal, opto-electronical, and field emission [52][53][54][55][56]. The complications related to the matrix predominating properties can be minimized by the advanced fibre reinforced polymer composites materials through this coating technique. The utilization of CNT in the large volume of polymer composites is an established technique and is utilized well by researchers in-depth [57][58][59][60][61][62][63][64]. Depending on the types of precursor coating materials, several coating techniques have been proposed, including chemical vapour deposition (CVD) [65] $\frac{[66][67][68][69][70][71]}{[66][69][70][71]}$, dip coating $\frac{[70][72]}{[72]}$, electrophoretic deposition $\frac{[73][74][75]}{[73][74][75]}$, and spray coating $\frac{[76]}{[76]}$. The highly crystallized graphite basal plane sites can be functionalized by these methods and raises their surface energy. Due to the addition of reactive functional groups or pitting, the fibre surface becomes roughened. As a result, mechanical interlocking between fibre and polymer, which imparts interfacial strength to the composite materials, is improved [77][78][79]. The largest batch of nanocarbon coated fibre reinforced composites are developed by using traditional hot-walled chemical vapor deposition, which coats CNFs over woven CF materials, with composite lengths restricted to the magnitude of the stable hot zone ^[68]. The volume of the material to be coated can be maximized using rolled or scrolled woven fabric upon employing tubular furnaces [80]. Regarding the polymer composite matrix, they can be divided into two overarching classes: thermosetting and thermoplastics polymers ^[81]. Curing involves the development of a cross-linked molecular, three–dimensional structure in both cases. A few obligations are required to be performed as a matrix. It includes the transfer of loads between fibres, protecting the notch sensitive fibres from abrasion and framing a protective boundary between the filaments and environment [82].

Preventing assaults from moisture, chemicals and oxidation is also an essential requirement for matrix as a protective barrier ^[83]. The composite is needed to be provided with shear, transverse tensile, and compressive properties by the matrix. Their performance governs the behaviour of the composite under the influences of temperature ^{[84][85]}.

There are a significant number of published review articles on carbon-based polymer composites that demonstrate either the use of carbon fibre, or nanocarbon particles and nanofibres as the reinforcing fillers. However, there was no emphasize on reviewing the technology that shows how the nanocarbon are coated onto CF to reinforce polymer matrices. With innovative interface-engineered hierarchical structures, this method allows CF reinforced composites to have better strength and durability. In particular, the use of micro- or nanocarbon coating on CF show the potential of using these hybrid fibres in fabricating high-performance composites for automotive ^{[86][87]} and aerospace components ^{[88][89]}, and several other structural applications including construction ^[32] and defence ^{[73][90]}. The potentiality of such hybrid fibres for reinforcing polymer matrices are well-demonstrated by many researchers in the form of research articles, books, or patents.

2. Types of Coating

2.1. Carbon Nanotubes and Nanofibres

Carbon nanotube (CNT) and carbon nanofibre (CNF) coating on CF reinforced polymer composites exhibit excellent properties that are essential in many high-performance applications including automotive [86][87] and aerospace [88][89]. High strength (>150 GPa), modulus (~1 TPa), thermal conductivity, electrical capacity and thermal stability are the common characteristics of this coating ^[91]. Several research works have been done based on CNT coated CF reinforced polymer composites [65][67][89]. Importantly, an optimal loading for CNT coating in the polymer composites is a key criterion to employ its outstanding mechanical characteristics in the composites. It has been studied that increasing CNT loading can primarily lead to an increase in tensile strength and tensile modulus, however, increasing CNT loading has an adverse effect on the tensile modulus and strength of the composite beyond a critical weight fraction ^[92]. For example, the deterioration of tensile strength, failure strain and elastic modulus of polystyrene composites has been found beyond a critical CNT mass fraction [93]. Nanotubes can cause agglomeration that decreases the interphase region and produces stress concentration sites of the composites, thereby leading to failure [94]. This can cause a reduction in the load carrying capacity of the composite as well. Therefore, it is necessary to grow CNT directly on the CFs surface. The processing parameters such as the growth time, catalyst, and growth temperature play an important role in the extent of CNT coating and the fibre surface coverage. Agnihotri et al. [95] have studied the effects of CNT coating on the CNT-coated CF/polyester composites' properties. The researchers have found that the optimization of the multiscale composite properties can be achievable by changing the reactor duration for chemical vapor deposition (CVD). Dispersion, degradation of the CNTs and matrix viscosity are some challenging issues for CNT coating ^[96]. In some cases, the dispersion techniques of CNTs coating in the polymer composites resulted in low CNT graphitization, poor nanotube alignment, CNT agglomeration, inadequacy to small weight percentage (wt%) accumulations, deficiency of morphology control, and reduced matrix infusion capability while using infusion techniques, such as resin transfer

molding (RTM) and vacuum assisted resin transfer molding (VARTM). The role of CF as a substrate for CNT coating permits well dispersed and high-density CNTs for the incorporation in the composite. The link among the adjoining CFs, generating electrical and thermal percolation routes throughout the composite and improving the mechanical characteristics of CF-based composites can be possible by CNTs. The interfacial adhesion between the resin-dominated (i.e., volume or weight fraction of resin is much higher than the filler) processes and the CFs can also be improved by the CNTs. The effect of sonication time on the mechanical properties of CNTs reinforced multiscale composites. Compared with neat epoxy resin, the residual heat, rate of reaction and heat flow and conversion at the beginning stage of the cure course has been enhanced by involving non-woven CNFs in the composites. Sharma and Lakkad ^[91] investigated the CF before and after the growth of CNTs/CNFs. According to the TEM images (Figure 1A), the centre of the CNT has a hollow core, which is very narrow. It is clear that the CNT covered the fibre surface uniformly and grew long enough with various diameter and length. As shown schematically in (Figure 1B), the long CNTs may orient themselves with the direction of drawing of the fibre when it is being pulled out through the very small orifice of the wire drawing die. The CNTs/ CNFs grown on CF is shown in Figure 1B(I) whereas the schematic diagram of polymer matrix dipped fibre and its pulling through the wire drawing die is portrayed in Figure 1B(II). The CNTs/carbon nanofibre coated CF after pulling out through the die is shown in **Figure 1**B(III), which displays an expected partial alignment of long CNTs/CNFs along the fibre axis, whereas the short CNTs/CNFs are still expected to remain unchanged. The cause of the alignment is due to the viscous force of the polymer matrix and also the frictional force due to the compactness of the opening of the die. Throughout the fibre pulling across the die, the exertion of the forces took place. The increase in the composites' tensile strength is due to this partial alignment.



Figure 1. (**A**) TEM images of the carbon nanotubes: I. aligned bundles of hollow-core MWCNTs; II. isolated MWCNTs. (**B**) Schematic diagram illustrating: I. carbon fibre after the growth of CNTs/CNFs; II. fibre dipped in polymer matrix and drawn through the die to fabricate a single fibre tow composite specimen; III. carbon fibre after

drawing through the die showing partial alignment of CNTs/CNFs along the fibre axis due to the viscous force of the matrix and the force due to the smallness of the orifice. Reprinted with permission from ref. ^[91], Copyright 2011 Elsevier Ltd.

It has been reported that the tensile strength increased by 69% for the multiscale composites compared to the reference composites made of CF while undergoing similar heat treatment processes as the CNTs coated CF. The authors have attributed that the presence of CNT on the CF surface improved the tensile properties of the composites. It is without a doubt that the addition of CNTs influences the properties of the composites. The most simplistic route is to add the CNTs to the polymeric matrix directly before fabricating the fibre-based polymer composite. Boroujeni et al. ^[97] have shown noteworthy enhancements for vibration attenuation (25.8%), impact energy absorption (21.3%) and axial strain to failure (12%) by adding multi-walled CNTs of 2.0 wt% to an epoxy matrix of a fibre reinforced polymer.

2.2. Graphene

Graphene coated CF reinforced polymer composites are used due to graphene's exceptional electrical, thermal, and mechanical properties [98][99][100]. Numerous studies have been done by incorporating graphene or graphene oxide (GO) sheets onto a wide range of polymer matrices [98][101][102][103]. He et al. [104] have introduced the graphene/CF hybrid as a multi-functional interfacial nano-reinforcement of CF reinforced polytetrafluroethylene composites for the enhancement of the mechanical and electrical properties. A slight increase in the tensile strength was also observed. Accordingly, an effective method is necessary for depositing graphene oxide onto the surface of CFs. For optimum improvement in mechanical properties of polymer matrix composites, the major key issues for favorable interaction are the alignment and dispersion of graphene on the polymer and the surface modification. Agglomeration of the graphene sheet is often caused by the entangled structure produced during the synthesis of graphene and interlaminar Van der Waal forces. While mixing with polymer, major complications arise due to the dispersion and peeling of graphene in media. Poor agglomeration and dispersion of graphene sheets can trigger nano defects in laminated composites, which can lead to poor enhancement of the mechanical properties. Multifunctional sizing agents such as graphene oxide can be consistently dispersed and robustly adsorbed on the CF surface to form an additional hierarchical reinforcement. It has the capability to generate in enormous numbers at a relatively low cost and holds outstanding mechanical properties. For the time being, it covers numerous oxygen functional groups, for example; epoxide, hydroxyl, and carbonyl groups. It has been studied that graphene oxide treatment on the surfaces of short CF can significantly enhance the adhesion at the interface of CF/polyethersulfone composites owing to the hydrophilic oxygen-functional groups in the basal planes of graphene oxide. Li et al. [105] has examined graphene coated short CF reinforced polyethersulfone composites to be used in cryogenic engineering field. The cryogenic mechanical performance was examined in the cryogenic tensile and bending test systems (Figure 2A) as demonstrated by Okayasu et al. [106]. In the work conducted by Li et al. ^[105], the sizing was conducted in a method similar to the Hummers method through acid oxidation of graphite powders. The preparation of pure polyethersulfone was conducted using the injection molding technique, which helps to observe the effect on the mechanical properties of the polyethersulfone matrix when an extrusion compounding process is used. Dried graphene oxide-coated short CFs were re-dispersed in deionized water and intensively stirred. The coating of short CFs was conducted in order to assess the coating efficiency of graphene oxide on short CFs in the physical adsorption process. According to Figure 2B, the graphene oxide was still robustly adsorbed on the short CFs surfaces, as shown in the SEM image of the collected short CFs. The researchers have reported an increase in tensile and flexural moduli when graphene oxide coating is used due to their outstanding elastic modulus. The optimal graphene oxide content of 0.5 wt% can efficiently improve the overall mechanical performance of the composite. Moreover, the usage of reduced graphene oxide can improve the interfacial property and mechanical properties of CF. The reduced graphene oxide coated CF has been found to be more effective than CF to enhance the unsaturated polyester-based composites in terms of electromagnetic interference shielding property. Chen et al. [98] have obtained that the shielding effectiveness of the composite at the frequency range 8.2–12.4 GHz (x-band) of reduced graphene oxide-based CF having 0.75% mass fraction can reach 37.8 dB, which is a 16.3% increase compared to that of CF unsaturated polyester based composite (32.5 dB) for the same mass fraction. Directly grafting graphene on CF can contribute to enhancing the mechanical properties of polymer composites. Figure 2C shows the SEM image of grafted GO on CF by ester linkage in a low temperature (>100 °C) solvothermal process [107]. Zhang et al. [108] have studied the novel hierarchical reinforcement of CF on which graphene oxide is directly grafted. The interphase strength between the CF and resin matrix was boosted due to the grafting of graphene oxide onto CF. Surface modification can effectively increase the polarity and wettability of the surface of CF without compromising the tensile strength [1]. The efficient graphene oxide reinforcement between CF and matrix resin results in these improvements. The ample potential in highperformance CF polymer composites is demonstrated due to the enhanced mechanical performance of this kind of hierarchical reinforcement.



Figure 2. (**A**) Schematic diagrams of cryogenic testing system for three-point bending and tensile tests. Reprinted from ref. ^[106], Copyright 1969 Elsevier Ltd. (**B**) SEM image of GO coated SCFs. Reprinted with permission from ref. ^[105], Copyright 2016 Elsevier Ltd. (**C**) SEM images of graphene oxide grafted on CF. Reprinted with permission from ref. ^[107], Copyright 2020 Elsevier Ltd.

2.3. Carbon Black

The usage of carbon black coated CF reinforced polymer composites has been considered in some researches due to their microcrystalline structure and different functional groups available on the carbon black surface ^[22]. Comprising virtually pure elemental carbon, carbon black is found in the form of colloidal particles (pellet or powder) synthesized by incomplete combustion or thermal decomposition of gaseous or liquid hydrocarbons ^[76]. The carbon atoms have hexagonal planes, resembling that of graphite, and the arrangement of these atoms of carbon black crystallite is ordered in graphite layers individually ^[109]. However, quasi-graphite crystals have a disordered array of carbon atoms in the adjoining layers. A high temperature is required for the manufacturing process of carbon black ^{[110][111]}. The primary structure is formed by the generation of adjoining particles simultaneously, melted into a chain, and occupying three degrees of space. This primary structure forms loose secondary structure steadily because of the Van der Waals force or physical adsorption. These secondary

structures can also be called temporary structures due to their susceptibility to mechanical damage during processing. Dong et al. [22] applied a carbon black coating on the surface of CF to enhance the mechanical properties of CF/epoxy composites. The interfacial strength between resin matrix and CFs was calculated using interfacial shear strength (IFSS) tests in this study. As shown in Figure 3A, IFSS of 5 min modified CF (CF-5) increased by 44.4% (49.45 MPa to 71.91 MPa) compared to untreated CF (CF). The surface energy was enhanced by the homogeneous distribution of carbon black, leading to enhanced wettability between CF and epoxy resin. The development in the surface roughness leads to an increase in bonding between the fibres and resin. Interface shear strength of 10 min modified CF (CF-10) significantly rose to 54.05 MPa at increasing carbon black growth time. The variability of the secondary structures of carbon black created caused the microdroplet composites to slide with relative ease from the fibre surface, even though the roughness and wettability of the fibres were improved. The properties of polymeric matrix and CFs as well as the efficacy of the interfacial adhesion between the matrix and the CFs are completely responsible for the composites' outstanding mechanical properties. Figure **3B** illustrates the correlation between the different growth time of the carbon black and its effect on the CF/epoxy composites' mechanical properties. The interlaminar shear strength (ILSS) of untreated CF composites was observed to be 47.6 MPa, however, the shear strength value of CF-5 min was 22.0% and CF-10 min was 13.55% higher compared to that of untreated CF composites due to the weak bonding between fibre and matrix. The structural integrity of composites was improved by the carbon black deposited on the CF surface that shifts the load successfully from the matrix resin to the fibre. The defects on the fibre surface coverage and stress concentration reduction can be possible by the carbon black coating, thereby increasing composites' mechanical properties (Figure 3C). The carbon black's secondary structure deteriorated the interfacial adhesive force between the resin and the fibres with time. The deposition of carbon black on the untreated-CF, CF-5, and CF-10 can be seen in Figure 3D-F. There was an enhancement in the surface energy of CF and in the wettability between the carbon black and matrix. As no holes were reported in the composite and epoxy matrix, we can say that the mechanical properties and interfacial adhesion of CF reinforced matrix were considerably enhanced. Several gaps between the fibre and the resin were detected while decreasing the number of voids and drawn fibres. Collectively, carbon black can increase the surface energy, the wettability, the CF surface roughness and cover the defects of CE surface ^[22].



Figure 3. Mechanical properties of carbon black coated CFs/epoxy composites: (**A**) IFSS tests results of the composites. (**B**) ILSS of the composites. (**C**) Impact test results of the composites and single fibre tensile strength. SEM morphologies of the fracture surface of composites (**D**) the untreated-CF, (**E**) CF-5 min, (**F**) CF-10 min. Reprinted with permission from ref. ^[22], Copyright 2017 Elsevier Ltd.

2.4. Graphite

Graphite coated CF reinforced polymer composites graphite are gaining interest because graphite is considered in nature as the stiffest material and has outstanding electrical and thermal conductivity owing to its layered structure. Exfoliated graphite nanoplatelets (xGnP) can be formed from graphite flakes interpolated with extremely concentrated acids which, at high temperatures, can be increased over a hundred times over their initial volume. The particles can be made of singular or a few layers of graphene sheets with considerable exfoliation. Park et al. [112] have fabricated xGnP/Cu coated CFs epoxy composites. Excellent electrical and thermal conductivity and good mechanical properties of xGnP polymer nanocomposites were found at low xGnP content of even less than 2 vol%. Due to the high surface area and aspect ratio of xGnPs, a percolated conducting network within the polymer is generated with a concentration of less than 2 vol%, thereby resulting in outstanding properties of these polymer

nanocomposites. In Figure 4A, the SEM images of the coated CFs with varying applied voltages using the copper plates as anode are shown. As shown in Figure 4A(II), except at the applied voltage of 10 V for 5 min, the surface of CFs treated with different process conditions was seen to have a relatively good deposition of xGnP with a size of 1 µm on the CFs in the ESEM images. As shown in Figure 4A(V,VI), there is redundant deposition above 40 V for 5 min. It is provident that the quantity of xGnPs on CFs steadily rises with an increase of the voltage applied at constant deposition time. Further investigation of the morphology of the coated fibres specified that there were additional constituents such as granular and co-deposited particles, as signified in Figure 4A(III,IV), other than xGnP. It has also been found that the xGnP/Cu coated CF reinforced epoxy composites' flexural strength and modulus are greater because of the coating of xGnP/Cu on the CFs, which has a reinforcing effect. In one study, xGnP coated CFs by using a solution of xGnP was produced, and a 19% increase in interlaminar shear strength was obtained with 3 wt% xGnPs [113]. In another research, the thermal conductivity was 13% higher for xGnP coated fibre with the addition of 1 wt% of xGnP $\frac{114}{1}$. Kostagiannakopoulou et al. $\frac{115}{1}$ showed that there is a 176% increase in thermal conductivity in nanomodified polymers and a 48% increase in thermal conductivity for CF reinforced polymer composite with the addition of 15 wt% GnP into the epoxy matrix. Due to its conformability, graphite has a very low contact resistance, which is why, for composite bipolar plates, methods of graphite coating were developed. Yu et al. [116] have conducted a study for polymer electrolyte membrane fuel cell based on graphite-coated CF epoxy composite bipolar plate. In this research, the cross-sections of the composite plate surface were shown (Figure 4B) when the thicknesses of the graphite layer are 2 µm and 50 µm (Figure 4B). It was found that the graphite layers with a thickness of 2 µm and 50 µm coating on the carbon/epoxy composite bipolar plates had 14% and 10%, respectively, of the total electrical resistance compared to that of the composite bipolar plate without surface treatment under 1 MPa of compaction pressure. This proves that the composite bipolar plate coated with graphite would be an appropriate substitute to metal bipolar plates.



Resin

Figure 4. (**A**) SEM images for the surface of CFs at different applied voltages for 5 min. Reprinted with permission from ref. ^[112], Copyright 2008 Elsevier Ltd. (**B**) SEM images: I. composite plate coated with a 2-µm-thick graphite layer; II. composite plate coated with a 50-µm-thick graphite layer. Reprinted with permission from ref. ^[116], Copyright 2011 Elsevier B.V.

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