Review

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Review

Surface Modification of Carbon Fibers

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Abstract: The purpose of the review is to provide new insight into the potential of surface modification of carbon fibers for enhancing the application of the carbon fibers many a fold. To this end a total of 429 papers on the subject of surface modification of carbon fibers by a variety of chemical and electrochemical methods published during the period from 2010-2022 have been reviewed. Astounding results of surface functionalization of carbon fibers by a variety of state of the art methods resulting in the unconventional applications of the resulting modified carbon fibers are summarized in a nut-shell in schemes from 1-6 towards the end of the review. Surface modification induces functionality to carbon fibers (CFs). The vitality of CF surface modification reactions could only be compared to the life process of respiration that sustains the multi functionality of living cells. Applicability of CFs can be drastically enhanced in incomprehensible ways by surface modification. Upon surface modification, inert and non-reactive CF surface becomes chemically active and functional with utility in diverse fields, namely, health, energy, environment, defense, catalysis, smart materials and many others. Surface modification methods can be broadly classified into chemical, electrochemical and physical methods. By these surface modification methods, the inert FC surface becomes polar. Surface properties like roughness, wettability and energy are enhanced. Modification processes like sizing, oxidation, amination, silanization, polymerization, nanoarchitecture induces multifunctionality on CF surface. Modified CFs when used as reinforcing material in carbon fiber reinforced plastics (CFRPs), improved bonding at the interface with resin matrices is observed with enhanced and outstanding mechanical properties (flexural strength, flexural modulus, IFSS, ILSS, hardness, elastic modulus, bending strength and compression strength).

Keywords: carbon fibers; surface modification; chemical; sizing; nanomaterials; oxidation; electrochemical; grafting; physical; modification; functionalization; multifunctionality; CFRPs; interface;, mechanical property

1. Introduction

Carbon fiber (CF) reinforced epoxy composites are widely used in aerospace engineering structures like, aerospace vehicles, rockets and artificial satellites owing to their excellent specific mechanical properties [1]. Carbon fibers are appropriate reinforcing material for the polymer matrix. Carbon fibers have high specific tensile strength, high modulus, and outstanding wear resistance, and are widely used for the reinforcement of advanced composite materials. CF reinforced thermoplastic composites are of interest owing to their easy processability and recycling [2]. However, the problem is that the carbon fiber surface is chemically non-polar and inert. As a result, as such carbon fibers cannot adhere strongly to the polymer matrix. It is the region between the carbon fiber (reinforcing material) and the filler matrix (polymer resin), termed as interface, which is crucial, vital and key for the ultimate performance of the CFRPs and their advanced applictions. As a result of poor adhesion between CF and polymer matrix, the strength of interfacial bonds is week and hinders the achievement of ideal mechanical properties of the composites. The structure and properties of the interface play a key role in the high temperature resistance and mechanical behavior of the integrated structure of composites. Better interface between the CF and the matrix effectively ensures the transfer of the stress load from the matrix to the CF increasing the application of CFRP's many a fold [1]. Carbon fiber reinforced polymers (CFRP's) are emerging as metal substitutes with

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applications in space exploration, military aircraft components, ship and submarine manufacturing, renewable energy industry (as blades of wind turbine), and automobile industry. In principle, CFRP's can be used in all the applications where light weight and high strength of the materials is required.

The problem of adhesion between the carbon fiber and polymer matrix can be solved by inducing polarity on to carbon fiber surface. There is a saying in Chemistry that "Like dissolves like". Since the polymer matrix is polar, by modifying the surface of CFs to be polar, makes them adhere strongly with the polymer matrix via strong covalent bonding interactions. Surface modification of CF is a well-known method to generate surface functional groups and thereby increase the interfacial adhesion between CF and the surrounding polymer matrix. The process of sizing the carbon fibers is one of the fundamental and key processes in the manufacturing of carbon fibers to induce surface polarity [3]. Oxygen rich surface functional groups can be generated on CF surface via different methods, namely, (a) oxidative treatment of CF's with a mixture of conc. H_2SO_4/conc. HNO_3, oxidation with conc. HNO_3, treatment with aq. $(NH_4)_2CO_3$, oxyfluorination, anodic oxidation and ozone treatment [2] and by a variety of physicochemical activation methods [4]. With the increase in polar functional groups and the wettability of CF, their surface roughness is increased; mechanical properties like the interfacial shear strength (IFSS), inter-laminar shear strength (ILSS) and impact toughness are enhanced. IFSS is a critical measure indicative of the interfacial adhesion between CF and polymer matrix [5]. Once the surface of the CFs is sufficiently charged, the CFs can reinforce strongly with the polymer matrix with well-defined composition, structure and properties at the interface. The bonding at the interface determines the ultimate performance of composites. Sizing is a commercial process for improving the compatibility of CF with polymer matrix with strong interfacial bonding. Owing to the significance as well as challenge of producing CFRPs with strong interface, novel and innovate methods, like modification of carbon fibers with sizing, the subject of the present review, need to be developed for improving the adhesion of CF's with the polymer matrix.

2. Role and Characterization of Sizing Agent Used for the Modification of Carbon Fibers:

2.1. Role of Sizing Agent in Enhancing the Interfacial Strength of CFRP's:

Sizing process is defined as a potential chemical method of modifying the surface of the carbon fibers. As a result of sizing the surface of carbon fibers becomes polar enriched with heteroatom (oxygen/nitrogen/sulphur) containing function groups. As a result of sizing process, CF becomes chemically more reactive with the resin matrix facilitating stronger interface formation in the CFRPs. Sizing process has commercial significance and so, much of the knowledge of sizing process, sizing agents used, sizing composition are often a subject of intellectual property [6–8] and only limited details are available in open literature [9–12]. Among several types of sizing agents, namely, epoxy resin, unsaturated resin and thermoplastic resin, the epoxy resins (for example, E44 , E51) are most widely used. Chemical structures of epoxy sizing agents E51, dimer of DGEBA (diglycedyl ether of bisphenol A), and E44, hydroxy propyl derivative of DGEBA, are shown in Figure 1 [3,13]. The carbon and proton nuclei in different chemical environments are numbered and alphabeted respectively. The choice and selection of the sizing agent for modifying the CF surface is largely based on the application dictated by the polymer resin matrix used in the preparation of carbon fiber reinforced plastics (CFRPs) [3].

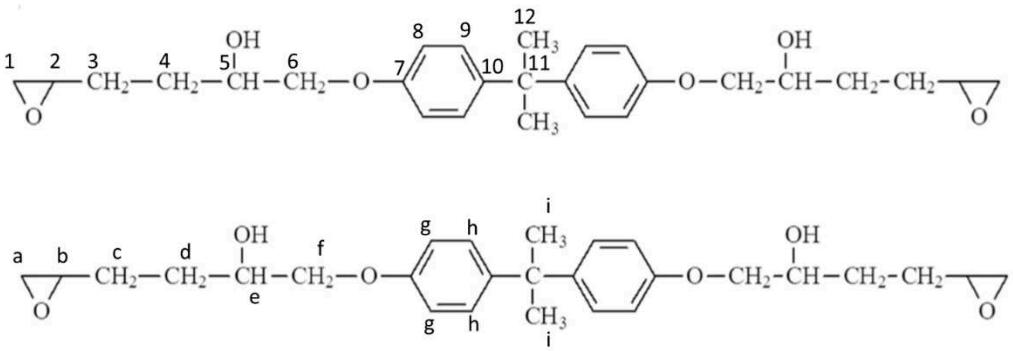

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Figure 1. Chemical Structure of epoxy resin based sizing agent, E44, hydroxyl propyl derivative of **diglycedyl ether of bisphenol A (**DGEBA) [3]. Adapted from reference 3 with permission from Elsevier.

The structure of strongly polar unsaturated resin, N-(4′4-diaminodiphenyl methane)-2 hydroxypropyl methacrylate (DMHM), that was used as sizing agent for CF (polyacryl nitrile based) is shown in Figure 2. A two fold enhancement in IFSS is observed upon using DMHM as sizing agent instead of the epoxy type sizing agent, E44 [3].

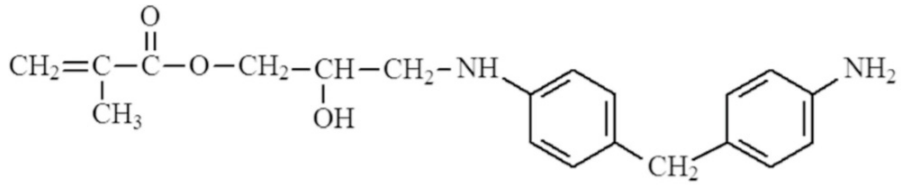

Figure 2. Unsaturated resin, N-(4′4-diaminodiphenyl methane)-2 hydroxypropyl methacrylate (DMHM), with strong polarity. Adapted from reference 3 with permission from Elsevier.

Thermoplastic resins are another class of sizing agents. Thermoplastics are different from thermosetting plastics. Typical examples of thermoplastics include, polyethylene (PE), polypropylene (PP), acrylonitrile butadiene styrene (ABS), polyamide (PA), polycarbonate (PC), polyether ether ketone (PEEK), polyetherimide (PEI), and polyether sulfone (PES). Typical advantages of thermoplastics being a choice of sizing compound/resin matrix is that they do not require curing stage, less hazardous chemical composition, improved recycling convenience and large scale production capability [2,14].

CF play crucial role as load bearing component in CFRPs. Owing to the merits of CF, CFRPs are used as a replacement to metal components in high tech fields like aerospace, automotive, wind energy, marine turbine blades. A comparison of the thermo-physical properties of CF with graphite fiber and Al metal shows the superiority of CFs with lower density value (ϱ, g/cm^3), modest heat capacity (J/g/K) and thermal conductivity (W/mK) values (where g, cm, J, m and K refers to gram, centimeter, Joule, meter and Kelvi) as shown in Table 1 [15].

Table 1. Thermo-physical properties of graphite fiber, carbon fiber and Al [15].

Material	ϱ (g/cm^3)	C_P (J/g/K)	T_c (W/mK)
Carbon fiber, CF	1.6	0.676	900
Graphite fiber, GF	2.2	0.71	100[a] (38[b])
Aluminum	2.7	0.895	237

Note: a - parallel to graphite layers; b - perpendicular to graphite layers;

Use of CFRPs is particularly advantageous over metals in space vehicles (like rocket motors, and satellites) as the amount of fuel needed to put a small weight into space is reduced considerably. Other metal replacements include helicopter blades, air craft structural components, air craft containers and chemical and ship building industries. In fact, the first ever carbon fibers were made by a group at Royal air craft establishment (RAE) with good tensile properties for high performance reinforcement applications [16]. As early as 1971, Jeffries has correctly assessed the potential of CFs and their unique properties making them ideal candidates for diverse application. Any engineering device involving rotating component will obviously be improved in its performance if manufactured from CFRP. This is because the centrifugal forces in the rotating part are dependent on the weight of the part. The structure of carbon fibers is similar to graphite. The carbon atoms are in layers or planes which are arranged almost parallel to each other. In true graphite, the atoms in each plane are arranged in a regular manner with respect to those in the adjacent planes. On the contrary, in CF, there is no such regular arrangement and the structure is described as turbostratic [16].

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Typical demerits of CFs namely, the smooth, non-polar, chemically inert surface with poor wettability and adhesion to resin (polymer) matrix, can be surmounted by the process of sizing [17]. Sizing process reduces the surface defects caused during fiber production process and thereby leads to improved adhesion with the resin matrix and results in enhanced mechanical properties [18]. Sizing agent performs multiple roles, namely, protecting the CF during processing, providing active functional groups on the surface of CF and improving the wettability of CF surface and the compatibility between CF and resin matrix. Solution and emulsion are the two types of sizing agents usually employed. Solution type sizing agent requires large amount of organic solvent while emulsion type comprise of organic resin dispersed in water with the help of an emulsifier. Emulsion type sizing agents are environmentally friendly and are safe to handle and so predominantly used compared to the solution type sizing agents [12]. The pH value of the sizing agent affects the IFSS of the CFRP as shown in Figure 3. Emulsion type sizing agent with dispersed reduced graphene oxide (rGO) was used to size the CF's with a concentration of 2 %. The sizing agent with acidic or neutral pH value has a minor influence on the improvement of IFSS of CF-epoxy composite. Optimum value of IFSS (92.3 MPa) was observed when the pH value of the sizing agent is 10.5. Beyond a pH value of 10.5, the IFSS value varied only slightly. When the pH value of the sizing agent increased to 12, the sizing agent became unstable due to deemulsification. ~ 20.3 % improvement in IFSS of the CF-epoxy (E51) composite is achieved when the pH of the sizing agent is increased from 4.2 to 10.5 [12]. Increasing the mechanical properties of CFRPs is a challenge and a requirement from the high technology sector like missile applications [19].

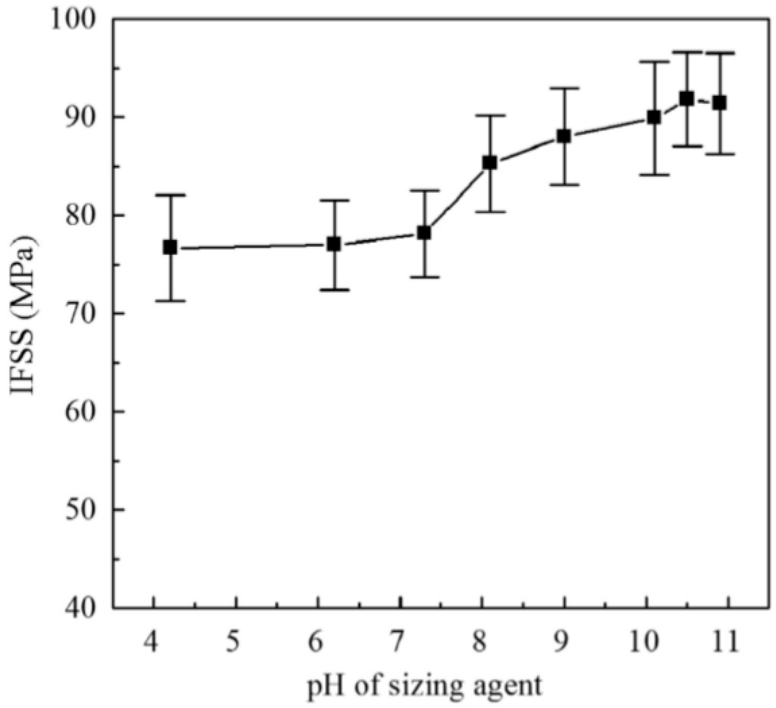

Figure 3. Effect of pH of sizing agent on the interfacial shear strength (IFSS) of CFRP. Adapted from reference 12 with permission from John Wiley and Sons.

Typical example of the measurement of the vital property of the CFRP, namely, IFSS is depicted in Figure 4. The IFSS between the single carbon fiber (SYT49) (sized with rGO modified epoxy based emulsion type sizing agent) and the resin microdroplets (E51) is determined by micro de-bonding test carried out on a interfacial evaluation equipment. The carbon fiber single filament is taped on the iron frame work. Resin microdroplets (E51) are wetted on the surface of the fiber. The resin microdroplets formed on the single fiber taped to the sample holder were cured by placing the sample in air oven at the curing conditions of 80 °C for 3 h, 140 °C for 1 h and 150 °C for 1 h. The temperature programming of the curing process is dependent on the resin matrix used in the CF-epoxy composite preparation. Two metal blades were clamped near a particular resin droplet and the sample holder is moved (in the direction of V) at a speed of 5 mm/min during the IFSS measurement. The metal

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blades were fixed during the test. The embedded length of the measured microdroplet of the resin (l_m; µm) is approximately 40-60 µm. The parameter measured during this test is the maximum force (F_{max}) required to de-bond a particular resin microdroplet from the single carbon fiber. The IFSS value of the composite is deduced by substituting the value of F_{max} in the following equation (1) [12,20]:

$$\boldsymbol{IFSS\ (MPa) = \frac{F_{max}}{\pi d_f l_m}} \quad (1)$$

where F_{max} is the maximum load force (µN)
d_f is the fiber diameter (µm)
l_m is the embedded length of the microdroplet (µm)

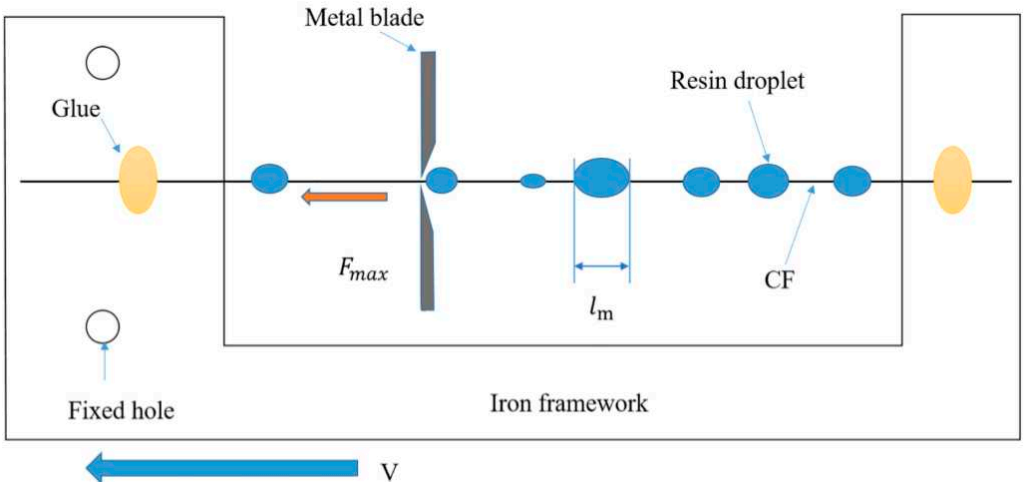

Figure 4. Schematic representation of the single carbon fiber resin microdroplet debonding test. V indicates the direction of the movement of the iron framework (sample holder). Adapted from reference 12 with permission from John Wiley and Sons.

Analogous to IFSS, ILSS is another vital mechanical property of the composites influenced by the sizing agent of the CFs. ILSS of the composites is deduced from the following equation (2) [21]:

$$\boldsymbol{ILSS\ (MPa) = \frac{3P_b}{4dh}} \quad (2)$$

where P_b - breaking load at fracture (N)
h - width of the specimen (mm)
d - thickness of the specimen (mm)

Recent advances in the design of sizing agents for the surface modification of CFs and the corresponding enhancement in the mechanical properties of the CFRPs are summarized in Table 2.

Table 2. Sizing compounds used for the modification of Carbon fibers.

Sizing compound with structure	Carbon fiber	Performance enhancement of CFRP	Reference
Maleic anhydride grafted poly (vinylidene fluoride), MPVDF, aqueous sizing agent	T300, 3K	42 % enhancement in ILSS	[5]

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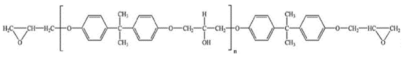
Fe_3O_4/rGO-modified polyimide aqueous sizing agent coated on the surface of CF's under an applied magnetic field	BHM3, 3K	159 % enhancement in ILSS. The enhancement was attributed to reduced agglomeration of graphene nano sheets by the orientation and uniform dispersion of Fe_3O_4 on graphene surface under applied magnetic field.	[9]
COOH-CNT modified sizing agent	JH-T800	10, 27 and 59 % enhancement in IFSS, ILSS and flexural strength compared to commercial CF-reinforced epoxy composites.	[18]
Emulsion type thermotolerant sizing agent (a resin synthesized by dodecyl amine and tetraglycidyl diaminodiphenyl-methane)	T700SC, 12K	29.16 % enhancement in ILSS	[22]
Emulsion type sizing agent prepared with modified poly (acrylonitrile-butadiene-styrene)	T700SC, 12K	26.6 % enhancement in ILSS	[23]
Modified polyacrylate emulsion	PAN based carbon fibers	14.2 % enhancement in ILSS	[24]
Nano-SiO_2 modified epoxy emulsion (AG-80) sizing agent 	PAN based carbon fibers	14 % enhancement in ILSS	[25]
Polyimide sizing performed by coating CFs with polyamic acid followed by thermal imidization;	Carbon fibers	Carbon atoms bonded to nitrogen and oxygen increased by 9.72 %; ILSS, axial compression strength and axial compression modulus of composite (with polyphenylene sulfide matrix) increased by 26.39, 26.02 and 19.64 % respectively.	[26]

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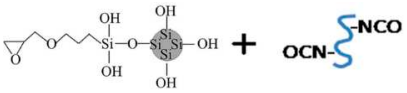
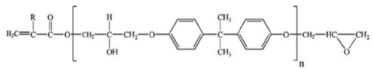
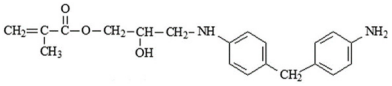
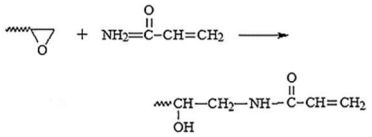
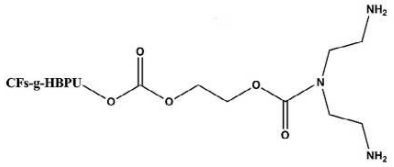
Sizing composition with MWCNT dispersion (0.5 %)	Carbon fibers	Tensile strength increased by 70.8 %; Flexural strength and ILSS, increased by 42.8 and 72.9 %	[27]
Polyurethane and nano SiO_2 hybrid sizing; Nano SiO_2 grafted with 3-glycidoxy propyl trimethoxy silane (GPTMS) coupling agent followed by dispersion in PU solution 	PAN based CFs; SYT49S (12 K) from Zhong Fushen Ying Carbon fiber limited Liability company (China)	40 % increase in the transverse fibers bundle strength	[28]
Amine functionalized CNT modified vinyl resin (M720); main polymer content in the sizing agent is 1 % 	Unsized CF (12k, 7 µm diameter) Guangwei co., China	Polar functional groups, wettability, and surface roughness enhanced; ILSS and impact toughness of modified CF/unsaturated polyester (UP) composite enhanced by 32.3 and 55.2 %	[29]
N-(4′4-diamino diphenyl methane)-2-hydroxy propyl methacrylate (DMHM) 	PAN based CFs; T700 SC CF (12k), provided by Toray company, Japan	IFSS and ILSS of sized CF vinyl ester resin (SCF/VE) enhanced by 96.56 and 66.07 %	[30]
Acrylamide functionalized epoxy (E44) sizing agent 	PAN based CFs; T700 SC CF (12k), provided by Toray company, Japan	IFSS and ILSS of the modified CFs vinyl ester resin enhanced by 86.96 and 55.61 %	[31]
Amine functionalized hyper branched polyurethane 	PAN based 6000 filaments per tow (6k) provided by Toray company (T-300 textile industry, Japan); tensile strength, 3194 MPa;	ILSS enhancement in the epoxy composite was attributed to the improvement of fiber-matrix interface through chemical interaction and mechanical locking	[32]

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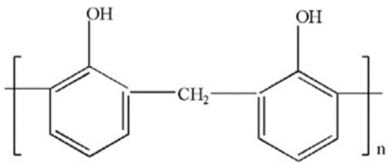
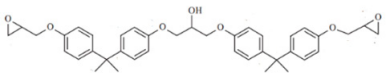
	density, 1.77 g/cc; diameter, 7.8 μm; CF surface was electrooxidised with conc. HNO_3 to generate COOH functionality to facilitate grafting with the sizing agent		
Novolac resin as sizing compound for the surface modification of CF; sizing was done in conventional process by immersing fiber tows in a novolac resin solution 	PAN based CFs (3 k) provided by continuous polyacrylonitrile based CFs; diameter, 7.1 μm Tensile modulus, 211 GPa; tensile strength,, 4.05 GPa	ILSS and impact strength enhanced by 22.5 and 56.8 %	[33]
CNT modified epoxy sizing agent; sizing composition comprise of CNTs (0.75 wt.%), epoxy resin (E51), tweet-80, and span-60; stability of sizing emulsion enhanced by the presence of CNT in the sizing composition sedimentation is prevented by the addition of CNT to sizing composition; 	PAN based carbon fibers, MT300, 3 K	Surface oxygen functional groups enhanced by 45.96 %; surface roughness increased by 73.1 %; contact angle reduced by 11.9 %; IFSS of the composite (bisphenol A epoxy resin matrix) enhanced by 14.7 %	[34]

Sizing plays an important role in improving the interfacial properties of CFRPs. The sizing layer on the CF act as additional compatibilizing phase where in some physical properties (for instance, stiffness modulus) of the composite have gradients, facilitating stress transfer from the polymer matrix to the CFs. In addition, sizing leads to formation of active functional groups on the CF surface enhancing the compatibility of CFs with polymer matrix. Particle size of the sizing agent and its distribution (PSD) is a crucial parameter determining the adhesion, stability, maximum solid content, drying time, rheological and optical properties of the sizing composition. The work of Yuan et al., on the surface modification of CFs with sizing agent (polyacrylate) and engineering the interface of sized CFs and polymer resin matrix (E44, DGEBA) has significantly improved the understanding of the region of interface that is critical for mechanical properties of the CFRPs with applicability in

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high technology sectors [24,35]. Sizing emulsions (water borne polyacrylate sizing agent) with five different particle size distributions, PSD, (P1, P2, P3, P4 and P5) with a respective mean particle diameters of 12.32 nm, 95.35 nm, 110.35 nm, 594.28 nm and 1.67 μm were prepared by controlling the PSD by physical filtration through differently sized filter screens (Figure 5). Irrespective of the PSD, all the sizing compositions have a zeta potential (ζ) value of ~ 40 mV with a low value of polydispersity coefficient (0.198-0.217). Usually, ζ value of more than 30 mV imply stability of the emulsion particles due to high electrostatic repulsion. Thus all the sizing emulsions (P1-P5, water borne polyacrylate sizing agents with varying particle size distributions) are thermostable. The aim of the study was to know the effect of PSD of the sizing composition on the adhesion strength of the polymer (epoxy resin) with the CF, and to find the optimal value of the PSD that can fill up the surface defects and interstitial sites on the CF surface and there by enhance the mechanical locking between fiber and the matrix. The authors have judiciously employed various physico-chemical interface characterization techniques like AFM and XPS to study the interface formed by the sizing compound at the interface of the CF and epoxy resin matrix.

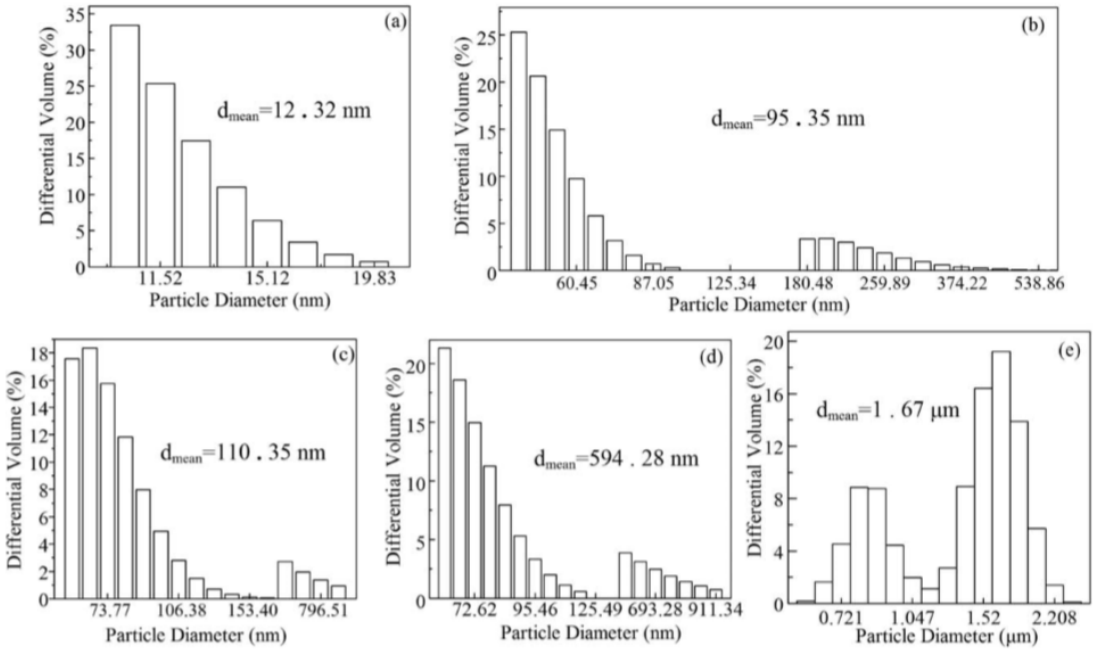

Figure 5. Images of the PSD of sizing agents: (a) P-1, (b) P-2, (c) P-3, (d) P-4, (e) P-5; d_{mean} represents the statistical mean diameter of emulsion droplets in different sizing emulsions (water borne polyacrylate sizing agent). Adapted from reference 35 with permission from John Wiley & Sons.

Load and stress transition role of interfacial phase (formed by sizing layer) due to the polarity matching between polymer matrix and sized CF was analyzed by force modulation mode of AFM [35–37]. The stiffness of various phases, namely, the CF reinforcement, the sizing layer (poly acrylate, P5, P3, and P1) on the CF surface and the polymer resin matrix (epoxy E44) as well as the thickness of the interface (formed by the sizing layer that bridges the CFs and the resin matrix) is examined by imaging the phase of the sizing layer at the interface using force modulation microscopy. The change in the stiffness modulus at the interface is a qualitative measure to know the crosslinking density between the fiber and the matrix as well as to understand the transition area between the fiber and the matrix formed by the sizing layer. Using the oscillating cantilever tip that probes the CFRP surface, the stiffness of various phases in the unidirectional CF-epoxy composite is examined. The force modulus images obtained from the cross-section of the composites reinforced by differently sized carbon fibers, namely, with sizing agents P5, P3 and P1 are shown in Figure 6 a-c respectively. It should be noted that the relative stiffness image of CF's is brighter than the surrounding epoxy matrix (E44). The corresponding relative stiffness modulus value variation [the gradation of the voltage generated from the cantilever deflection of the AFM as the tip moves from

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right (CF phase) to left (epoxy matrix) along the white line (through the interfacial phase of the sizing layer) in the images Figure 6 a-c] at the interface of the sizing layers formed by P5, P3 and P1 (along the white line in the images 6 a-c respectively) is shown in Figure 6. d-f, respectively.

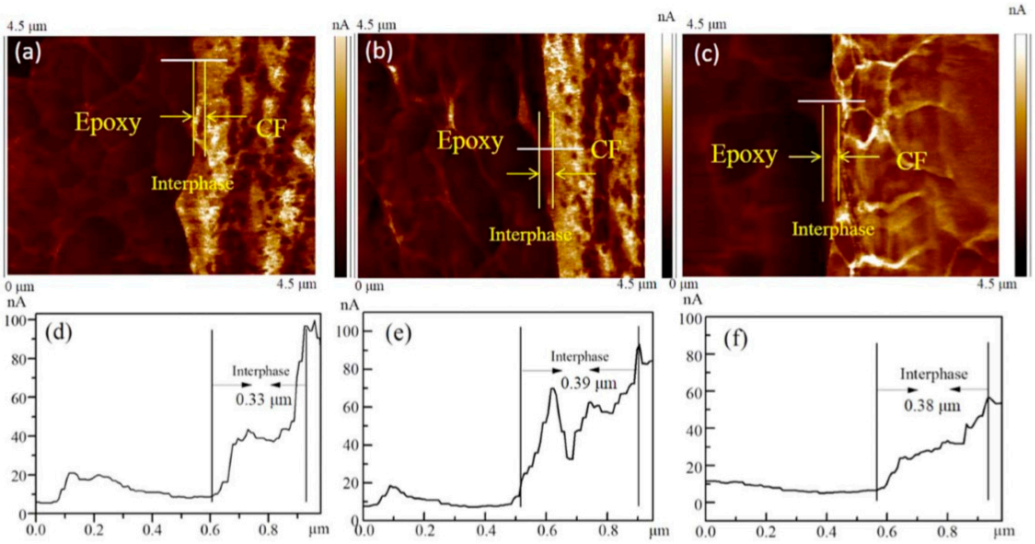

Figure 6. (a, b and c) Relative stiffness image of cross-section areas of P-5, P3 and P-1 sized composites respectively; (d, e and f) stiffness distribution of cross-section in the composites corresponding to (a, b and c respectively; along the white line). Adapted from reference 35 with permission from John Wiley & Sons.

The stiffness image, wherein P5 sizing is employed for the CFs in the CFRP, a clear distinction between the CFs and the surrounding epoxy matrix is observed (Figure 6 a). On the contrary, the boundary between the CF and the epoxy matrix becomes blurrier and a light brown interfacial phase is observed in P3 and P1 sized composites (Figure 6 b and c respectively), signifying the role of optimal particle size distribution (P3 with mean particle diameter of ~ 110 nm) in forming an effective interface between CFs and epoxy matrix. In P5 sized CFRPs (Figure 6d), the magnitude of stiffness modulus drops suddenly (as reflected in the magnitude of current measured in nA and plotted on the Y-axis of Figure 6 d-f), along the white line from right to left. The thickness of the sizing phase at the interface is 0.33 µm which is lower than the thickness of interface formed with sizing agents P3 (0.39 µm) and P5 (0.38 µm). These parameters illustrate that large particle size (P5 - 1.67 µm) of the sizing emulsion plays a negative role in the surface modification (wettability and roughness) and adhesion ability of CF surface with the epoxy resin matrix. In contrast, the stiffness modulus at the interface of CFRPs sized with P3 and P1 sizing agents decrease gradually (from right to left, from CF surface to the matrix along the white line in the images Figure 6 e-f respectively) indicating the gradient sizing distribution in the interface layer forming thicker interface (~ 0.39 µm). Thus relatively small size (~ 110 nm, P3) sizing droplets particles of the polyacrylate spread out on the CF surface uniformly healing the penetrating into the surface defects and interstitial sites facilitating the stress transfer efficiency. These observations were further confirmed by the evaluation of IFSS values of CFRPs without sizing agent (81.2 MPa) and CFRPs sized with P1-P5 sizing compounds (88.3-92.5 MPa). The CFRPs sized with P3 showed a 13.9 % enhancement compared to unsized CFRPs (with no sizing agent) and 4.7 % enhancement compared to P5 sized CFRP's [10].

The geometry and location of sizing agent/compound and the mechanism of interlocking of CF and resin matrix through sizing agent (poly acrylate) at the interface with optimal PSD was demonstrated by Yuan et al., in the typical example of CF-epoxy (E44) composite [35].

Sizing agent with optimal multimodal PSD fit into the defects and interstices on CF surface and increase the packing efficiency and uniformity of surface distribution. The surface groves and defects of CFs are reduced and rectified. Relatively small particle size of sizing agent (~110 nm as in the above example) results in the formation of interface of moderate thickness and even distribution of sizing compound permeating through the bundles of CFs. As a result, surface defects are reduced, chemically active surface is formed leading to strong interfacial adhesion and effective stress

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transfer from polymer matrix to CF and thereby the load bearing ability with high tensile strength of CFs is utilized in various applications as metal replacement [35]. The chemical interlocking (by covalent and hydrogen bonding interactions) of the CF surface and epoxy matrix is facilitated by the increase in oxygen functional groups at the interface due to sizing by polyacrylate emulsion particles of optimum PSD (~ 110 nm) as deduced from XPS analysis [35].

2.2. Physical and Chemical Methods of Surface Modification of Carbon Fibers:

Apart from sizing, a variety of chemical and electrochemical methods were recently developed for the surface modification of carbon fibers. Vedrtnam and Sharma classified surface modification methods into five broad classes, namely, wet methods (sizing, electrochemical, acid treatment), dry methods (plasma, thermal, high energy irradiation), using nanomaterials (CNT's , ZnO, TiO_2, SiO_2, SiC, graphene oxide), oxidative methods (oxidation with acids, gases, plasma) and nonoxidative methods (pyrolytic carbon deposition, plasma polymerization, whiskerization) [38]. Zang et al., reviewed the progress in carbon fiber surface modification methods, namely, grafting, coating, oxidation, and plasma treatment and proposed that chemical grafting method standout owing to its advantages (precision of grafting polymeric species on to CF surface) resulting in improvement in surface roughness, and mechanical properties (interfacial adhesion) in composites [39]. Zang et al., classified surface modification methods into three groups, namely, wet modification methods (liquid phase oxidation, anodic electrolytic oxidation and sizing treatment), dry modification methods (gas phase oxidation, plasma oxidation and irradiation treatment) and nanoparticle modification (functionalization with graphene, CNT and metal oxide NPs). Merits and demerits of various modification methods for application of modified CF's for carbon fiber reinforced polyamide (CFRPA) were discussed [40]. Raphael et al., pointed out that grafting is an effective and efficient alternative to oxidation functionalization for the surface modification of CF [41]. Raphael et al., reviewed various methods of modification of CF and the resulting property enhancement for the reduction of interfacial tension so as to utilize the potential of the reinforcing matrix to the fullest. The authors have classified surface modification methods into five groups, namely, electrochemical oxidation, chemical oxidation, plasma treatment, gamma ray or laser irradiation and treatment with rate earth elements [41]. Hung et al., reviewed method of surface modification of CF's with graphene-related materials (GRM) derived from graphite. GRM are effective in improving the properties of the CF's in stress transfer from matrix to the CF through the interface comprising of GRM. The authors pointed out that the property enhancement of the composite materials under cryogenic environment are crucial for new applications involving aircraft and space engineering development [42]. Corujerira-Gallo reviewed the changes in the surface chemistry and surface microstructure of CFs as a result of plasma treatment. Surface contaminants or weekly bonded graphitic layers are removed by the process of etching, surface roughness increased, the surface chemistry and the polarity are altered [43]. Naito reviewed the effect of surface modification of CFs, by grafting with CNTs, coating with polymeric materials and other hybrid methods, on the resulting tensile properties of both pitch and PAN based CF's. A linear relationship between Weibull modulus and average tensile strength on a log-log scale was observed for all surface modified CFs. While all modifications resulted in improvement of tensile strength and Weibull moduli, hybrid modifications (grafting followed by vapour deposition, grafting-dipping hybridization) resulted in the highest values [44]. An exemplary account of the chemical and electrochemical methods invented for the surface modification of carbon fibers enhencing their applicability is provided in Table 3 and Table 4 respectively.

Table 3. Chemical methods for the surface modification of carbon fibers.

Carbon fibers	Chemical method	Functionality improvement	Reference
CFs were obtained from Toray industries (diameter, 7 μm; tensile strength, 3500 MPa)	Salicylaldehyde (SLDH) was grafted chemically through 3-aminopropyltriethoxyilane (3-APS) bridges onto the surface of CFs	Contact angle with water decreased from 78.5 to 52.1° and the surface energy increased from 35.9 to 55.0 mNm^{-1}; IFSS of the composite (CF-g-SLDH and methyl phenyl silicone resin, MPSR) was enhanced	[45]

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		by 48.66 %; Hydrothermal aging resistance was enhanced by 18.5 %;	
Carbon fiber (no further details available)	Deposition of hydrophobic (n-hexyl amine) layer on CF surface followed by a coating of outer catalyst layer (isolated single atomic sites of cobalt/hallow N-doped carbon spheres)	Modified CF used in the fabrication of microelectrode for sensing oxygen content in brain; four electron reduction of oxygen is achieved; H_2O_2 formation pathway suppressed preventing cell toxicity	[46]
Carbon fiber (PAN, asphalt, rayon, graphite)	Polymerization modification	Wettability of CFs with resin improved; physical and chemical interactions between CFs and resins improved	[47]
T700 12 K; unidirectional; areal density, 200 gm^{-2}; produced by Toray industrial Co. Ltd., Japan	Aqueous solution coating of CFs; polyelectrolyte complexes (PEC) were formed by the reaction between oppositely charged PEI (polyethylene imine) and APP (ammonium polyphosphate) through an ion exchange reaction by soaking the CFs in the PEC at room temperature;	Phosphorous containing PEC coating on CFs (PEC@CF) imparted fire retardancy property to the carbon fiber reinforced epoxy composite; properties of CFRP enhanced as follows: Limiting oxygen index enhanced by 43 %; reduction in the peak heat release rate, 47 %; mechanical properties and glass transition temperature were enhanced; UL-94 V-0 rating achieved for the composite;	[48]
Carbon fibers (further details are not accessible)	Amine functionalized CFs were further grafted with polyhedral oligomeric silsesquioxane (POSS) species on the surface of CFs; uniform continuous layer of siloxane oligomers formed on CF surface;	ILSS and oxygen erosion resistance of interface of CF-epoxy resin composite improved; material suitable for environment in low-earth orbit for space exploration vehicles;	[49]
Long PAN based CFs (3×10^3 single filaments per tow); diameter, 6-7 µm; density, 1.76 gcm^3; tensile strength, 3500 MPa; obtained from Toray Industries, Inc Tokyo, Japan	Cardanol molecules were granted onto CF surface by in situ polymerization; Initially, hydroxyl groups were formed on CF surface by treating CFs (suspended on a glass frame) with isopentyl nitrite and 4-amino phenol; polar hydroxyl groups served as grafting sites to adhere Cardanol by in situ polymerization of hexachlorocyclotriphosphazene (HCCP) and cardanol;	Wettability and surface energy enhanced by cardanol functionalization; Water contact angle decreased from 78.5 to 66.82 °C; surface energy increased from 35.86 to 47.53 $mN.m^{-1}$; ILSS of CF-cardanol-UPR (unsaturated polyester resin) composite increased by 41.35 %;	[50]
Carbon fibers (PAN based; petroleum pitch based and coal pitch based)	Coating organic layer followed by oxidation;soaking CFs in organic solution (phenolic resin slurry or aliphatic hydrocarbon) followed by drying and low temperature heat treatment (180-220 °C); further, the organic layer was oxidized to form surface oxygen functionality in the presence of oxidizing gas (chlorine gas or SO_2) carried by inert carrier gas (He or Ar)	Though not disclosed organic layer coating followed by oxidative treatment might have resulted in the formation of oxygen rich surface functional groups; high bonding strength achieved on CF-polyamide 66 thermoplastic composite.	[51]
Carbon nanofibers prepared from polypropylene (PP) by melt force spinning technology	CN-groups are formed on CF surface by grafting acrylonitrile and methacrylic acid monomers; cyano groups were converted to amidoxime groups by treatment with hydroxyl amine solution followed by treatment with alkali	Amidoxime functionalized CFs used as adsorbent for U (VI) (83.24 mg/g in 60 min at pH = 4) from simulated sea water.	[52]

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	for enhancing the sorption capacity of amidoxime group to form complex with metal ions.		
PAN fibers, provided by Jilin chemical industrial company, Ltd., China	Chemical treatment with hydroxylamine hydrochloride and mono ethanolamine followed by thermal oxidation in air; amidoxime groups formed on CF surface as a result of chemical treatment followed by oxidation.	Thermal stability, flame retardant and mechanical properties were enhanced;	[53]
Carbon fibers (CFs), supplied by Sigma Aldrich; diameters, 100 μm Length, 20-200 μm	CFs were dispersed by ultrasonic treatment in the aqueous solution of Triton X-100; the well dispersed CFs were surface functionalized (O, N, S) by oxidative treatment with H_2SO_4 and HNO_3 (3:1) at 80 °C for 90 min;	Amount of oxygen functional groups on CNF surface increased by Triton X-100 mediated acid oxidation	[54]
PAN based carbon fibers; Diameter, 7 μm; Fibers surface treated with epoxy resin based sizing agent (1.3 %); Supplied from Toho Tenax Europe GmbH, Japan, in the form of carbon fiber tape;	Surface oxidation by plasma treatment; prior to Ar plasma treatment the sizing was removed by treatment with acetone at 50 °C for 18 h; Ar plasma treatment was done by cold radio frequency (RT) plasma multi-jet at atmospheric pressure;	Wettability measured by glycerol contact angle decreased from 90-35°; oxygen functionalities (alcohol, ether, carboxyl, ester groups) increased; ILSS increased from 20.8 to 31 MPa	[55]
Carbon fibers based on 3K-1200-200 twill weave fabrics;	Thermal oxidation (300-500 °C for 30 min in air atmosphere)	Oxygen functionalities (hydroxyl, ethereal, carbonyl, carboxyl and epoxy) were formed on the CF surface; ILSS of CF-Poly sulfone composites increased from 42.8 to 62.9 MPa; formation of chemical bonds between CF and polysulfone matrix lead to enhancement in ILSS;	[56]
T300 PAN based carbon fiber plain cloth from Toray, Japan	CF-epoxy composite was treated with conc. H_2SO_4 (97 wt.%) followed by $CaCl_2$ (1 M). The composite was surface functionalized with SO_3H and Ca^{2+} species;	Biocompatibility of CF-epoxy composite enhanced by the presence of $–SO_3H$ and Ca^{2+} species on the surface ; uniform dense coating of apatite was formed on the composite when soaked in simulated body fluid (SBF) indicating the application in orthopedic and dental implants	[57]
Carbon fibers, PAN based	Oximation (treatment with Na_2CO_3 and hydroxyl amine hydrochloride lead to formation of amidoxime groups) followed by alkaline hydrolysis (lead to partial hydrolysis of amidoxime groups to carboxylate groups); oxygen content increased from 2.71 to 35.92 % upon modification;	Radioactive waste mitigation; Adsorption of U (VI) from radioactive waste, 163 mg/g; efficient and economic removal of U (VI) from radioactive waste; U (VI) coordinates with amidoxime (=N-OH/=N-O-), $-NH_2$/-NH-, and carboxyl groups through a penta-coordination complexation;	[58]
Carbon fibers, T700SC, 12k	Desized CFs were pulled through PEI (polyetherimide) sizing agent solution in N-methyl-pyrrolidone (NMP) followed by in situ functionalization with zeolitic	40.5 % enhancement in IFSS of modified CF/PEEK	[59]

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	imidazolate frame work-67 (ZIF-67) by immersing PEI sized CF tows in 2 – methylimidazole followed by reaction with $Co(NO_3)_2$ 6 H_2O (24 h, RT); PEI sizing layer controls the relative amount of ZIF-67 crystals adhered to the CF surface;		
Carbon fibers, 3 x 10^3 single filaments per tow; diameter, 7 μm; density 1.76 g/cm^3; tensile strength, 3500 MPa; tensile modulus, 230 GPa; provided by Toray industries, Inc.,	Hyper branched polysiloxane grafting by sol-gel polymerization;	Water contact angle decreased from 78.5 to 52.1°; surface energy increased from 35.9 to 55.5 mNm^{-1}; ILSS and impact strength of CF-MPSR composite increased from 56.02 and 33.78 % respectively;	[60]
Carbon fibers (no further information accessible)	Polyurethane coated CFs used as reinforcing material for polyamide46/polyphenylene oxide (PA46/PPO)	Mechanical, tribological and heat resistance properties enhanced; volumetric wear resistance decreased by 95 %; tensile strength increased from 82 to 282 MPa.	[61]
Carbon fibers (T800SC), PAN based; used in the form of carbon fabric provided by Nanjing Tianniao corporation, China	Carbon coated CFs by hydrothermal process (180 °C) using glucose as carbon precursor;	BET specific surface area increased from 6 to 44 m^2/g; specific capacitance enhanced from 15 to 106 F/g; IFSS increased from 39.8 to 51.3 MPa; applicable for wearable (flexible textile) electronic devices	[62]
Carbon fibers in the form of carbon fabric (density, 1.55 g/cc), provided by Torayca, Japan	Air plasma (6 kW processing power) oxidation and etching using atmospheric pressure plasma jet (APPJ) system; polar functional groups formed on the surface; active species (OH, N_2, O) are generated during plasma irradiation;	Water contact angle decreased by 49.7 %; surface free energy increased by 41.3 %; Sheer strength of modified CF-reinforced plywood increased by 50 %;	[63]
Carbon fibers, T700 based non-crimp fabric (NCF UD, 400 gsm)	Hydrophobic (C8, unsaturated fluorinated monomer) coating on CF surface by inert gas plasma treatment;	71 % growth of loss factor; CFRP (epoxy matrix, biresin CR80) material with reduced sound emission due to reduction of the fiber-matrix bonding via hydrophobic and non-polar treatment;	[64]
Ultrahigh molecular weight polyethylene (UHMWPE) fibers; diameter, 40 μm; provided by Dacheng Advanced Material Co., Ltd., China	Chromic acid pretreatment; bamboo like polyethylene crystals were formed on the surface of pretreated UHMWPE using solution crystallization; pretreated UHMWPE were added to the p-xylene solution of polyethylene (110 °C for 10 min); the mixture cooled to 96 °C and allowed to crystallize for 3 h	Strength and toughness of modified UHMWPE epoxy composites improved simultaneously; tensile modulus increased from 57.8 to 73.5 GPa; bending modulus increased from 45.4 %; ILSS enhanced by 71.9 % ;	[65]
Carbon fibers prepared from oxidized coal, polyacrylonitrile and pitch	Depositing pitch on the modified carbon surface and subsequent carbonization;	Super hydrophobic CFs with a water contact angle of 159.5°; used for gravity driven oil/water separation ; 98 % separation efficiency; flux, 3600 L/m^2 h;	[66]
Polyimide (PI) organic carbon fibers	Surface modification with silane coupling agent (KH-550) is better than SiO_2 sol-gel or ozone treatment; formation of Si-O-Si groups at the interface of PI/epoxy composite;	Polar surface functionality (Si-O-Si) increased; surface roughness increased by an order of magnitude; water contact angle decreased from 111.1 to 82.3°; IFSS and ILSS of PI/Epoxy composites enhanced by 15.6 and 18.2 % respectively.	[67]

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Rayon carbon fibers	Oxidation and grafting modification to generate enriched oxygen and nitrogen groups;	Improved electric double layer performance; lower capacitative impedance; high sensitivity marine electric field sensor;	[68]
Carbon fibers	Inductively coupled RF plasma (ICP) treatment for surface oxidation; carboxyl and hydroxyl groups are formed;	Interfacial property of composites with bismaleimide (BMI) resin enhanced; surface roughness, surface free energy, and wettability improved	[69]
Carbon fibers (length, 2 mm) provided by Nanjing WeiDa composite Material Co Ltd., Nanjing, China	Conc. HNO_3 treatment (60 °C, 3 h)	Flexural strength and modulus of CF reinforced ultrahigh molecular weight polyethylene (UHMPE) increased to as high as 157 Ma and 9.82 GPa respectively;	[70]
Carbon fibers from Good fellow, Huntingdon, England	Polymer (PEI) surface coating;	Limit of detection of 3-methoxy tyramine, 3 MT; 3, 4-dihydroxyphenyl acetic acid, DOPAC, 58.2 nM (enhanced by 80 %)	[71]
Carbon fibers (T700-12K-50C, average diameter, 7 μm) Toray Industries Inc.,	Copolymerization of dopamine and poly (amidoamine);	IFSS, ILSS and impact strength increased by 78.57, 62.39 and 75.12 % respectively	[72]
Carbon fibers (PAN based, T 300, 3K, diameter 7 μm; density, 1.76 gcm^{-3}, tensile strength, 3.53 GPa)	Layer by layer (LbL) selfassembly to deposit graphene oxide/silica (GO/SiO_2) multilayers on CF surface based on electrostatic interactions;	IFSS, ILSS and flexural strength increased by 86.1, 89.3 and 30.4 %	[73]
Carbon fibers supplied by Jilin carbon co. Ltd., ; coated with epoxy resin; density, 1.76 gcm-3; diameter, 6.44 μm;	Desizing, oxidation with $AgNO_3$ and $K_2S_2O_8$ (to form CF-COOH); treatment with $LiAlH_4$ (to produce CF-OH); grafting with γ-amino propyl triethoxy silane (γ- APS) as coupling agent	IFSS, flexural strength, and flexural modulus enhanced by 52.9, 29.1 and 42.6 %	[74]
Carbon fiber tow (3K, T300, Toray co, Ltd., Japan)	Uniform TiC/Ti_2AlC coating by in situ reaction with molten salts; subsequent pyrolytic carbon layer deposition through pyrolysis of phenolic resin; desizing followed by coating with TiC/Ti_2AlC with Ti and Al powders as respective precursors;	High resistance to temperature, oxidation and corrosion; application in high performance CF/SiC composites for aerospace, and nuclear industries;	[75]
CF fabric (3K-T300-plain) supplied by Toray industries, Inc., Tokyo, Japan	Aminated polyether ether ketone (PEEK-NH_2) grafted to CF surface;	ILSS improved by 33.4 %; Modified CF is compatible with PEEK matrix for CFRP formation	[76]
Carbon fiber, PAN based with epoxy/polyurethane sizing (H TS 40 FB, Teijin Carbon Europe GmbH,, Wuppertal, Germany)	Oxyfluorination (F_2/N_2 mixture with 10:90 vol %) at room temperature for 180 S	Desized; surface bound fluorine content and oxygen functionality enhanced; strength of fibers increased by 10 %	[77]

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Carbon fiber T700SC-12000-50C; diameter, 7 μm; tensile strength, 4.9 GPa; with 1 % sizing on the surface	Amine functionalization of CF surface using 4-(aminomethyl benzene derivative; grafting tosylated lignin derivative to arylamine functionalized CF	IFSS increased by 27 and 65 % with epoxy and cellulose propionate matrices	[78]
Carbon fibers	Grafting of aryl sulfate with aryl amine pendant groups; Exposure of fibers to an atmosphere of sulfuryl fluoride (SO_2F_2) gas lead to fluoro sulfate functionality ; exchange with aryl silyl ether to form pendant aryl amino group	IFSS improved by 130 % in the epoxy resin composite	[79]
Carbon fibers, PAN based; diameter, 7 μm; density, 1.76 gcm^{-3} ; provided by Sino steel Jilin carbon co, China	Desizing; oxidation with $AgNO_3$ and $K_2S_2O_8$; CF-COOH is grafted with polyethylene imine in supercritical methanol	Polarity, wetness, roughness of modified CFs enhanced; Water contact angle decreased from 85.68 to 36.54°; surface energy increased from 31.3 to 66.62 mNm^{-1} IFSS of the modified CF epoxy composite increased by 40.60 %	[80]
Carbon fibers, PAN based; based; diameter, 7 μm; density, 1.76 gcm^{-3} ; provided by Sino steel Jilin carbon co, China	Polyether amines; covalently grafted and coated onto oxidized CF surface (CF-COOH) to form CF-g-PEA	Polarity, wettability, roughness, and IFSS increased	[81]
Carbon fibers	In situ formation and deposition of graphitic carbon nitride (g-C_3N_4) on CF surface	Polar functional groups, roughness, wettability enhanced; water contact angle decreased from 75.37 to 46.37°; surface energy increased by 65.6 %; ILSS, IFSS, tensile strength, absorbed energy of impact enhanced by 16.4, 43.2, 16.9 and 30.3 %	[82]
Carbon fibers, T300; diameter 7 μm provided by Toray, Japan	Evaporation induced surface modification; deposition of polyether imide (PEI) nanoparticles on the CF surface; PEI, optimal conc. 0.2 %;	IFSS enhanced by 44.02 %	[83]
SIGRA FIL® C30 T050 supplied by SGL carbon GmbH	Low pressure ammonia plasma treatment	Nitrogen content of the fibers enhanced; wettability and surface energy are enhanced	[84]
H TS 340, provided by Toho Lamac Co., Ltd., Tokyo, Japan	Grafting of maleic anhydride followed by deposition of intumescent flame retardant (IFR)	Synergistic activity of maleic anhydride grafting and intumescent flame retardant (P, N and O) on the enhancement of flame retardency and mechanical property	[85]

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Mesophase pitch based CFs without sizing	Microwave oxidative etching of CF surface (dispersed in water) in the presence of micro plasma ; etching of surface GO layer	Oxygen containing functionalities enhanced; nitrogen heterocyclic rings of the bulk of CFs were exposed; IFSS of modified CF with epoxy resin composite enhanced	[86]
T300 grade CFs (3K) produced by the institute of coal, chemistry, Chinese Academy of Sciences	Grafting of acrylamide on CF surface	IFSS and ILSS of polyimide composites enhanced by 86.96 and 55.61 %	[87]
CF (3K; diameter, 7 μm) provided by Toray industries	Hydroxyl groups were formed on CF surface (by aryl diazonium reaction) followed by chemical grafting with octamaleamic acid-polyhedral oligomeric silsequioxanes (OMA-POSS)	Polarity and roughness increased with grafting density; IFSS and ILSS enhanced in the silicon composites by 65.87 and 70.89 %	[88]
PAN based CFs from Jilin Jiyanhigh technology fiber, Co Ltd., Jilin, China	Methyl acrylate grafting to generate carboxyl functionality	Shear and tensile strength of modified CF's enhanced by 90.3 and 78.7 %; wear rate decreased by 52.7 % in the phenolic composite of methyl acrylate grafted CF's	[89]
Carbon fibers (length, 10 mm; diameter, 7 μm)	Desized by boiling in propan-2-ol; chemical prefunctionalization by treatment with either H_2O_2 or aqua regia followed by treatment with 1 M $CaCl_2$ at 80 °C for 24 h; deposition of calcium phosphate crystals on CF surface;	Mechanical properties (strength and work of fracture) of calcium phosphate cement (CPC) were enhanced by injectable and load bearing substitutes; strength increased by a factor of 4 and work of fracture increased by 2 orders of magnitude;	[90]
UHMWPE (ultrahigh molecular weight polyethylene) fibers (diameter, 20 μm; molecular weight, 4.5 million)	Ozone treatment (generate oxygen functionality) followed by UV irradiation polymerization grafting of glycidyl methacrylate (GMA) onto UHMWPE fiber (ultrahigh molecular weight polyethylene)	Interfacial adhesion force of UHMWPE fibers with rubber matrix enhanced by 79 %	[91]
Carbon fibers	Desizing CFs were coated with a layer of polydopamine with di-coating method;	Bending strength and modulus increased by 71.3 and 36.9 %	[92]
Carbon fibers	Hydrothermal treatment with glucose followed by carbonization to form amorphous carbon layer on CF surface	ILSS of CF/polyimide composite enhanced; IFSS of CF/polyether ether ketone is enhanced; oxygen functionality (C-O, C=O) generated	[93]
T700SC-12000-60E CFs, epoxy sized, provided by Toray carbon Europe	Grafting of hydroxyethyl methacrylate on CF surface by free radical polymerization of vinylic monomers	Tensile strength and ILSS increased by 10 and 20 %	[94]
CF, T700 SC 12K, Toray, Japan	Co deposition of polyethyleneiminie/polydopamine (PEI/PDA) on CF surface by oxidation co-polymerization	Surface energy enhanced by 70.5 %; 45.7 % increase in apparent activation energy (290 kJ/mol) which is a reflection of strong interaction between PEI/PDA layer and matrix;	[95]
Carbon fibers	Mechano chemical modification by simple rubbing	Surface modification facilitated surface oxidation; oxygen functionalities (hydroxyl, alkoxide, carbonyl and carboxyl groups were generated); modification confined to first few atomic layers	[96]

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Carbon fibers, JT-400A-3K, diameter, 6.8 μm; linear density, 0.175±6 g/m; density, 1.76 g/cm3; provided by Jilin Shen Zhou Carbon fiber Co Ltd.,	Chemical grafting of hyper branched polyglycerol (HPG) via anionic ring-opening polymerization	IFSS and ILSS the epoxy composite enhanced by 90.69 and 49.83 %	[97]
Carbon fibers (5631 12K) provided by Toho Tenax Co Ltd., Tokyo, Japan	Sol-gel coating prepared by the hydrolysis and condensation reactions between tetraethyl ortho silicate (TEOS) and one of the four organic compounds namely, (3-amino propyl) trimethoxy silane, (3-mercaptopropyl) trimethoxy silane, 2-(3, 4-epoxy cyclo hexyl) ethyl trimethyoxy silane and methyl trimethoxy silane, depending on the kind of functionality required	Si-O-Si, epoxy, SH, CH_3 functionalities were generated on CF surface; Stiffening and clumping of fibers is prevented by functionalization; oxide network structure with organic functional groups is generated; surface enhancement is maximum in coatings leading to the formation of epoxy functional groups	[98]
Carbon fibers (T700SC-12000-50C, 12k); tensile strength, 4.9 GPa; diameter, 7 μm; density, 1.8 gcm^{-3}	Surface functionalization using poly(oxopropylene) diamines (D-400) as coupling and curing agents	Polarity, wettability and surface roughness enhanced; tensile strength increased by 8.2 %; IFSS of modified CF/epoxy composite enhanced by 79.1 %	[99]
PAN based CFs (CH-CF 1k), provided by Shanghai Chem Fiber Technology Co, Ltd, China Single filaments per two, 12000; tensile strength, 4.9 GPa; tensile modulus, 230 GPa; diameter, 7 μm; elongation, 2.1 %	Supersonic atmospheric plasma spraying; the power composition used for spraying is 30-50 wt.% Si, 30-40 % $KMnO_4$, 10-20 wt. % C and 4-8 wt.% TiO_2; slurry of the spray powder prepared in distilled water with polymeric binder	ILSS of the polypropylene/polystyrene (blends) composites with modified CFs enhanced; water contact angle of the composite enhanced by 50 %; O/C ratio enhanced by 67.9 %	[100]
Carbon fibers produced by Toho Tenax Co, Ltd., USA	Hydrogen plasma treatment, via plasma enriched chemical vapour deposition (working temperature, 500 °C; pressure 360 mTorr);	Density of functional groups increased; tensile properties enhanced both at room temperature and high temperature (150 °C);	[102]
Carbon fiber rovers (bundles)	Coating thin layers (2-30 nm) of polymers, namely, poly(acrylic acid) and poly(hydroxyl ethyl methacrylate) using electron spray-ionization (ESI);	Adhesion between CF and polymer matrix (epoxy resin) enhanced; IFSS enhanced by 170-285 %	[103]
PAN based chopped carbon fibers provided by Hangzhou Gaoke composite Co Ltd.,	Oxidation with conc. HNO_3 followed by thermal treatment of 400 °C for 2 h;	Oxygen functionality generated (C-O; O-C=O); tensile modulus, flexural strength, flexural modulus of composites (nylon 12, PA12, matrix) enhanced by 11, 11, and 5 %	[104]
Carbon fibers (T700-12000-50C) from Toray industries	Oxidation of CFs by treatment with HNO_3 at 80 °C for 4 h followed by depositing nanoporous Zr based metal organic frame works (UiO-66-NH_2) on CF surface;	Surface energy and tensile strength enhanced by 102 and 11.6 % respectively; ILSS enhanced by 50.2 %	[105]
PAN based CFs provided by Sino steel Jilin carbon Co, China	Step wise growth of melamine-based dendrimers	Surface roughness, and wettability enhanced; IFSS and impact strength of the epoxy based composite enhanced by 61.8 and 39.9 %	[106]

Micrometer sized magnetic carbon fiber (MSMCF)	Coating polydopamine/polyethyleneimine complex on micrometer sized magnetic carbon fiber (MSMCF)	Sorbent for extracorporeal blood-cleansing in hemoperfusion	[107]
Carbon fibers	Grafting poly (glycidyl methacrylate) chains onto CF surface by RAFT polymerization	Compression strength, tensile strength, flexural strength and ILSS of epoxy composite enhanced by 63.1m 37.9, 55.6 and 122.5 % respectively	[108]
Tenax® HTS 45 12K 800 tex CFs provided by Toho Tenax	Oxidation with conc HNO_3 (70 °C, 90 min); co oxide and Fe_2CoO_4 were deposited on CF;	Flexural modulus and flexural strength enhanced by 16 and 62 % respectively	[109]
JT-400A-3K, diameter, 6.8 μm; linear density, 0.175±6 g/m; density, 1.76 g.cm3; provided by Jilin Shen Zhou carbon fiber co.,	Chemical grafting of linear amine terminated poly (amido amine), PAMAM, dendrimers	ILSS enhanced by 53.13 % in the modified CF epoxy composite	[110]
JT-400A-3K, provided by Jilin Shen Zhou carbon fiber co.,	Grafting of polyhedral oligomeric silsesquioxane (44) through poly (amido amine) PAMAM, coupling agent to generate CF-PAMAM-POSS	Surface energy and wettability enhanced; ILSS and IFSS of composites (epoxy matrix) increased by 48 and 89 %	[111]
PAN based T800 unsized CF's	Doping with para-amino benzoic acid (PABA)	cytotoxicity minimized; compressive strength enhanced by 27.4 %	[112]
Pitch based carbon fibers (XN-50®, Nippon graphite fiber Co.,); strength, 3.9 GPa; modulus, 520 GPa	Coating of ester functionalized phenoxy resin based material on CF surface;	ILSS enhanced by 20 % in the composites with polyamide 6 (PA 6) matrix	[113]
PAN based CFs provided by Toray, Japan	Treatment with silicone peroxide [tris(tert-butyldioxy)-vinyl-silane (VTPS)] Si-CH=CH_2 groups generated that acted as covalent bonding bridges between CF and resin matrix (polydimethyl siloxanes, PDMS)	Tensile strength and tear strength enhanced by 52.3 and 340 %	[114]
Carbon fibers	Oxidative treatment with conc. H_2SO_4 and HNO_3 in the presence of $KMnO_4$ for 16 h; modified CF was woven into a fabric and cut into a certain width of base band for application as ribbon for printer (printeronix p7010 and p7003H)	Improved life time of the printer ribbon (50 million/number of characters); CF based materials fulfil the nylon 66 ultra-high ribbon requirements	[115]
High strength PAN based CF (T1000 GB; Toray industries Inc.) High-modulus pitch based carbon fibers (K 13 D; Mitsubishi plastics Inc.,)	Amine functionalized CFs obtained by treatment with different agents, namely, ethylene diamine; 4, 4 diaminodiphenyl sulfone, and p-amino benzoic acid (PAB);	N/C ratio increased ; IFSS of the composite (epoxy matrix) enhanced	[116]
T700S, average diameter, 7 μm; provided by Toray industries, Inc., Japan	Coating of amine capped poly(cyclotriphosphazene-co-4, 4'-oxydianiline) through in-situ polymerization under mild reaction conditions	Tensile strength increased by 10 % by the healing of surface defects by the formation of poly phosphazene sheath; IFSS increased by 70.5 %	[117]

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Carbon fibers	Plasma treatment; environmentally safe method	IFSS of the micro composite (poly phenylene sulfide, PPS matrix) increased by 17 %	[118]
Carbon fibers (3K), provided by Toray industries, diameter 7 μm; density, 1.76 gcm^{-3}	Grafting of octaglycidyl polyhedral oligomeric silsesquioxane (gly-POSS) and tetraethylene peptamine (TEPA) were grafted to CF surface in succession	Fiber polarity, wettability and surface energy were enhanced; ILSS and impact toughness of composite (with methyl phenyl silicone, MPSR resin matrix) enhanced by 56.6 and 34.9 %	[119]
Carbon fibers in the form of reinforcement cloth bought from Shanghai Yingjia Special fiber Material Co Ltd., Shanghai, China	Oxidation with conc. HNO_3 for 3 h; *Candida tropicalis* immobilized on CF surface for Xylitol fermentation	Hydrophilicity generated; immobilization efficiency of *Candida tropicalis* enhanced (0.98 g/g modified CF); xylitol yield (70.13 %) and productivity (1.22 $gL^{-1}h^{-1}$) were enhanced	[120]
PAN derived CFs, UKN-M-12K provided by Argon Ltd, Russia; Specific weight, 1.75 g/cc; diameter , 7 μm; elastic modulus, 220 GPa; tensile strength, 3.0 GPa	Coating of Al_2O_3 (3.3 wt. %) (by soaking in $Al(OH)_3$ followed by annealing at 800 °C) as protective layer followed by grafting with CNT	IFSS of the composite (with polyurethane matrix) enhanced by 144 %; composite stiffness and thermal conductivity enhanced; flexible composites with outstanding delamination resistance	[121]
PAN based T800 CFs supplied by Petro China Jilin Petrochemical company (Jilin, China)	Controlled chemical oxidation with $H_3PO_4/H_2SO_4/HNO_3$; soaking in mixed $H_3PO_4/H_2SO_4/HNO_3$ for 1 h at 60 °C;	Anode electrode material for Li ion batteries; presence of H_3PO_4 in the electrolyte restricted the formation of surface defects during modification; tensile strength of the original CF's preserved	[122]
PAN based CFs, T300, with 3000 single filaments per tow; provided by advanced fiber research center, Iran	Oxygen plasma treatment (1 min; 125 W; oxygen flow rate, 1000 1 cm^3_{STP}/min); high power and long exposure reduced the tensile strength and ILSS of CFs	ILSS of the composite (epoxy matrix) enhanced by 28 %; reactive functional groups increased; surface roughness enhanced	[123]
Carbon fibers	Microwave irradiation in ionic liquid [1]-ethyl-3-methyl imidazolium bis (trifluoromethyl sulfonyl) imide] and organic solvent, 1, 2-dichlorobenzene	28 % enhancement in interfacial adhesion	[124]
PAN based CFs, T300 B, provided by Toray Co.,	Vinyl groups of coupling agent N-(4-amino-phenyl-2-methyl-acrylamide (APMA) were grafted CF surface, APMA-CF	IFSS and the flexural strength of composite (vinyl ester resin matrix) enhanced by 90.53 and 19.4 % respectively	[125]

Table 4. Electro-chemical methods for the surface modification of carbon fibers.

Carbon fibers	Electro-chemical method	Functionality improvement	Reference
Toray T700, 7 μM diameter (polyacrylonitrile based carbon fibers)	Electrochemical oxidation:Carried out in a two-electrode cell (PAN anode and graphite cathode) with H_3PO_4 (0.5 M) as electrolyte with a	Due to oxidation, the amount of oxygen functional groups, namely, hydroxyl, carbonyl and carboxylate increased; these groups made the carbon fibers hydrophilic;	[126]

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	current density of 5 A/g for 5 min.	contact angle decreased from 142 to 52°	
High modulus carbon fiber (HMCF, 6 K), diameter 5.2 µm; Bulk density – 1.846 g/cm^3 Linear density – 0.238 g/m Tensile strength – 4.0 GPa Tensile modulus – 410 GPa	HMCF was anodized (oxidized) by electrochemical oxidation in the electrolyte $(NH_4)_2SO_4$ followed by electrochemical grafting with diethylenetriamine (DETA) by electro chemical grafting;	As a result of the two stage electrochemical oxidation and grafting, oxygen and nitrogen containing surface functional groups were formed on the surface of the highly inert and unreactive HMCF; ILSS of functionalized HMCF/epoxy composites increased by 257 %;	[127]
Carbon fiber (no further details available)	A ultrasound (40 kHz; ultrasonic bath) assisted anodic oxidation (+2 V) in alkaline medium; sono anodization was performed by immersing electrochemical cell in a ultrasonic clean bath; kinetics of electro-oxidation increased by sonication;	C-O functionality increased at the expense of C-C and C=C; carbon edge defects increased; contact angle decreased from ~115 to ~40°; increase in wettability is attributed to formation of carboxyl and C-OH functionalities;	[128]
HT40 E13 6K 400 tex provide by TohoTenax®-E (PAN derived CF, epoxy sized diameter, 7 µm; tensile modulus, 238 GPa; tensile strength, 3.9 GPa)	Active screen plasma functionalization; radicals generated in the plasma using gas mixtures of N_2-H_2-Ar modify the CF surface; the applied voltage was in the range of 300-400 V between the active screen (cathode) and the wall of the furnace (anode);	Structural disorder of modified CFs reduced; surface crystallite size and tensile strength of the modified CFs increased; Flexural strength of the CF-epoxy composite increased by 3.8 % and ILSS increased from 43.7 to 43.9 MPa;	[129]
HF 100 PAN based carbon fiber (12 K, tensile strength ≥ 3530	Anodic oxidation (current density: 0-6 Am^{-2}) with 5 wt.% NaOH followed	IFSS of CF-epoxy composite is enhanced by 15 % when CFs are treated	[130]

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Mpa; elastic modulus, 230 GPa; density, 1.789 cm^{-3}; diameter, 7 μm); provided by Jiangsu Hengshen Incorporated Company (Jiangsu, China); obtained by carbonization without surface treatment and sizing;	(electrolyte) followed by rinsing with HCl (0.1 M)	in NaOH electrolyte followed by HCl treatment	
Carbon fibers without sizing agent; supplied by Hengshen Co Ltd., Jiangsu, China; diameter, 7 μm; density, 1.78 g/cm^3	Electrophoric deposition (EPD) of graphene oxide (particle size distribution, 0.5-10 μm) on CFs followed by electro polymerization of itaconic acid and p-aminobenzoic acid; For EPD process,	ILSS of CF-epoxy (E51) composite enhanced by 37.6 %; tensile strength of CF monofilament increased from 3.01 to 3.22 GPa; water contact angle decreased from 119.9 to 30.2°; T_g increased by 7.15 °C and storage modulus (E′ at 35 °C) increased by 73.9 %	[131]
Pure carbon fiber was provided by Suzhou Institute of Nano-Tech and Nano-Bionics (SINANO), Chinese Academy of Sciences	Ni coated on CF surface was achieved by electroplating method; $NiSO_4$ 6 H_2O was the main Ni source; CF is used as cathode placed between two nickel anode plates in the electroplating bath;	Electro thermal conversion efficiency of Ni coated carbon fiber (NCCF) bundles was 32.5 % higher than untreated fiber based composites; The composite material has E heating (de-icing) application in aerospace, wind turbine, civil infrastructure building for cold climatic regions	[132]
Pristine unsized CF, 12k tow, Carbon Nexus	Electro reductive grafting of aryl diazonium salts followed by in situ polymerization of monomers onto CF surface to impart colour to CF;	Blue CFs with application in smart composites produced; exposure to different solvents varied the colour of CFs from blue through the visible spectrum; tensile strength of treated fibers increased	[133]

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		by 12 % ; 300 % enhancement in the fiber to matrix (epoxy) adhesion observed compared to control;	
Carbon fibers, PAN based with epoxy sizing supplied by Toho Tenax (Tenax® - EHTS40 F13 12 K yarn fineness 800 tex Toho Tenax Europe GmbH Wuppertal, Germany)	Oxygen containing calcium species (calcite) was coated on CF by anode electro oxidation in cement pore solution (3 V, 15 M);	Shear bond strength of CF increased by 37.9 %; compatibility and mechanical properties of modified CF-cement based composites enhanced.	[134]
Carbon fibers, PAN based, 6000 filaments per tow; prepared in the laboratory	Two stage process of surface modification; electro oxidation (enhanced oxygen functionalities) and grafting of silane coupling agent (KH 550)	IFSS and ILSS of CF/EP composites increased by 73.1 and 61.2 % respectively.	[135]
HMCF (high modulus carbon fiber), tensile modulus, 410 GPa; tensile strength, 4.0 GPa; diameter, 52 μm, 6 K) obtained by the carbonization of PAN based CF (at 2400 °C)	Multi stage anodization (electron oxidation, with NH_4HCO_3 followed by $(NH_4)_2SO_4$;	ILSS increased by 299 % (from 23.3 to 93.1 Mpa)	[136]
Carbon fibers (unsized and unoxidised); provided by Carbon Nexus at Deakin University, Australia	Electrochemical reduction of nitro aryl diazonium salt to graft amine groups of CF surface followed by depositing perfluoroalkyl chains on CF surface	Hydrophobicity generated; fiber to matrix adhesion increased as high as 216 %; water contact angle increased from 98.4 to 135.5°	[137]
Carbon fibers (PAN based) HTA 40 E13 3K CF from Toho Tenax Europe GmbH (HTA)	Thermal desizing followed by electrochemical oxidation	Interfacial fracture toughness enhanced; surface oxidation of CF improved the siliconization	[138]

	with NH_4HCO_3 solution as electrolyte	(liquid silicon infiltration) leading to enhancement of mechanical property	
Continuous carbon fibers (T700 SC-12K), diameter, 7 μm; tensile strength, 4.51 GPa; tensile modulus, 248 GPa	Electro oxidation with 1 M HNO_3, at 150 mA, for 1 h	Reactive oxygen and nitrogen functional groups formed (C-O, C=O, C-N, C=N, O=C-O); surface roughness enhanced; interfacial adhesion between modified CFs and high density polyethylene (HDPE) enhanced	[139]
Carbon fiber fabric	Electrolytic plasma spraying for coating SiO_2/SiC on carbon fiber fabric using Na_2SiO_3. $9H_2O$ as electrolyte; spraying distance (15 mm) is a crucial parameter;	Oxidation resistance temperature increased up to 1000 °C; tensile strength enhanced by 48.77 %;	[140]
Carbon fiber (5 μm diameter)	Electro deposition of gold thin film on CF surface	Improved performance as microelectrode for the detection of catecholamine and dopamine	[141]
Carbon fibers (unsized, electrolytically oxidized) provided by carbon Nexus at Deakin University, Australia	Surface grafting through Kolbe decarboxylation reaction; electrochemical oxidative grafting of benzyl groups (-CH_2-Ar-CH_2-Ar)	IFSS increased by 112 %	[142]
Ni coated carbon fibers, 12k 50 NiCCF tow, provided by Toho-Tenax	Desizing followed by electro oxidation in NaOH (0.1 M) in the potential range of 1600-1800 mV vs RHE	Oxygen evolution reaction (in the water electrolysis for H_2 production) reaction kinetics enhanced from the reduction of charge transfer resistance and modification of Tafel polarization slopes	[143]

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Carbon fibers unsized and electronically oxidized, produced by carbon Nexus at Deakin University, Australia	Reductive electrochemical deposition of diazonium salt to generate pendant amine and carboxylic groups	Composites of CF with amine pendant groups with epoxy matrix showed 172 % enhancement in IFSS	[144]
Carbon fibers	Electrochemical copolymerization of acrylamide and acrylic acid using cyclic voltammogram in H_2SO_4 electrolyte	Tensile strength of the composite (with epoxy matrix) enhanced	[145]
Toray carbon fibers (T-700-12k-60E); strength, 4.9 GPa; modulus, 230 GPa; density, 1.80 g/cm^3; diameter, 7 μm; sizing type, 60E, sizing volume, 0.3 vol %	Functionalization with active screen plasma treatment (300 V for 5 min) with a gas mixture of 25 % N_2 and 75 % H_2;	surface enriched with nitrogen functionality; amine and pyridone type nitrogen were introduced; quinone (C=O) and ester (O-C=O) type oxygen functionalities were formed; wettability increased; water contact angle decreased from 80 to 41°	[146]
PAN based carbon fibers (T700 SC, 12K tow fibers) provided by Toray industries Inc.,	Electrochemical oxidation of CF in H_2SO_4 electrolyte followed by grafting with 4, 4'-diphenyl methane diisocyante (MDI)	Tensile strength of MDI-CF composites (polyurethane matrix) increased by 99.3 % ; friction loss decreased by 49.09 %	[147]
Carbon fibers	Coating of polyacrylic acid layer of CF surface by the elctropolymerization of acrylic acid	ILSS of the composite (epoxy matrix) enhanced by 123 %	[148]
Carbon fibers HTS40, Toho Tenax	Adsorption of poly(methyl) methacrylate (PMMA)	IFSS enhanced	[149]

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	particles on the desized CF surface by electrophoresis		
PAN based CFs; 48k; A-company, Jeonju, Korea	Electrochemical oxidation; electrolyte (ABC) undisclosed	Oxygen (-OH, C=O, COOH) and amine functionalities were introduced; toughness of CFs increased; IFSS of composite (with epoxy matrix) enhanced by 144 %; Surface energy increased from 70.6 to 82.5 mN/m (16.8 %)	[150]
PAN based CFs	Electrochemical coating using electrolytes; fatty alcohol polyethylene ether ammonium phosphate (O-3PNH-4; 5 % mass fraction) showed best performance	Hydrophilic acidic functional groups formed; surface energy enhanced	[151]
T700 carbon fibers provided by Toray, Japan	nanofibers of polyaniline (PANI) were grown on modified CF surface by in situ electrochemical polymerization;	Functionalization of CF with nitrogen containing compounds enhanced the electrochemical performance of PANI composite for supercapacitor application; faster charge transfer; smaller internal resistance and improved electro chemical performance	[152]
CFs based on PAN provided by carbon nexus, Australia	Covalent surface modification; electrochemical grafting of biphenyl groups using diazo benzene precursor	Electric resistance of CFs increased by 250 %; IFSS of epoxy composites enhanced by 19 %	[153]

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Carbon fibers	Immobilization of Prussian blue crystals on CF surface by electrochemical modification	Potential adsorbent for Cs^+ over a wide range of pH values (acidic and basic)	[154]

2.3. Surface Modification of Carbon Fibers with Nanoparticles:

Kakhki reviewed the surface modification of CFs by enzymes and nanoparticles (like Pt, Rh, Au) for enhanced performance of the modified CF's as electrodes with application in analytical chemistry, electrochemistry, environmental chemistry, health science, and renewable energy (battery, supercapacitors, fuel cells and biofuel cells) [155]. Vedrtnam and Sharma reviewed the potential of nanospecies (CNTs, ZnO nanorods, SiO_2 nanotubes) in the surface modification of CFs for enhancing the performance (interfacial strength, vibration attenuation and delamination resistance) of the resulting CFRPs and proposed that the potential of CNTs is underutilized for modifying CF surface [38]. A brief account of the recent advances in the surface modification of carbon fibers with nanoparticles is provided in Table 5.

Table 5. Nanoparticles functionalized carbon fibers.

Carbon fibers	Modification method	Functionality enhancement	Reference
Carbon fibers from Donghua university (no further details available)	Hydrophilic Fe_3O_4 NPs were grafted on the surface of CFs using polyol-assisted hydrothermal method (CFs@ Fe_3O_4);	Presence of hydrophilic Fe_3O_4 NPs improved interfacial adhesion of CFs; Hydrophobic CFs were converted to hydrophilic magnetic CF, CFs@Fe_3O_4@SiO-C/Ni;	[156]
T300, diameter 7 µm, provided by Toray, Tokyo, Japan	Coating nanoparticles of polyetherimide (PEI) on carbon fibers by evaporation induced surface modification;	IFSS enhanced by 20.5, 37.7, 52.7, 49.6, 42.5 and 58.0 % as the thermoplastic resin in the composite is varied from PVC, PC, PA6, PP, PA66 to PEI respectively	[157]
Carbon fibers (no further details provided)	Desized carbon fibers added to a solution (solvent is atleast one of water, alcohol, acetone, and tetrahydrofuran) of ferrocene derivative Subsequently the ferrocene functionalized CFs were either oxidized or electrooxidized	Though not disclosed, the surface modification of CFs might have formed nanoparticles of Fe_3O_4 on CF surface making the CFs magnetic and hydrophilic leading to enhancement of their performance in composite materials and catalysis.	[158]
T300 (average filament diameter 7 µm); 3 K plain weave mat type carbon fibers; tensile strength, ~3500 MPa; supplied by Toray, Japan	Grafting graphene based nanofillers to CF surface by cathodic electrophoretic deposition (EPD)	IFSS and flexural strength of composite (CF-DGABE type epoxy) enhanced by 35 and 26.6 % respectively	[159]

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T700-12 K (bidirectional PAN derived); supplied by Toray	Functionalization with MWCNTs by dip-coating technique	Structural, electrical and thermal properties of the composite (modified CF with epoxy matrix) increased	[160]
Carbon fibers in the form of carbon cloth (1 K, T300); supplied by Toray Inc., Japan, China	Silicon carbide nanowire functionalized CFs prepared by Si coating followed by thin carbon layer coating and graphitization;	Contact angle decreased by 32°; wear resistance increased by 78 %	[161]
Carbon fibers (3K, diameter, 7 μm; Tensile modulus, 230 Gpa; tensile strength, 3500 Mpa; provided by Toray industries, Inc.,	Halloysite nanotubes (HNTs) were grafted to CF surface through diethylenetriamine-penta acetic acid bridges	Surface polarity, energy and wettability increased; 17.95 % increase in storage modulus; glass transition temperature enhanced by 17 °C in the CF-MPSR (methyl-phenyl silicone resins) composite; water contact angel decreased from 78.5 to 42.76°; surface energy increased from 35.87 to 62.71 $mN.m^{-1}$; ILSS and IFSS enhanced by 76.52 and 72.73 %	[162]
Carbon fibers (no further information accessible)	Coating oxidized graphite NPs on CF surface with ultrasonically assisted direct current electrophoretic deposition (at 50 V for 5 min)	Transverse fiber bundle test strength enhanced by 113 %; aspect ratio of CF surface enhanced upon modification with oxidized graphite NPs.	[163]
Carbon fibers, Diameter, 7 μm; Provided by Toray industries, Tokyo, Japan.	Functionalization with SiO_2 NPs through the hydrolysis of tetra ethyl ortho silicate (TEOS) onto poly dopamine coated CFs;	ILSS and IFSS of CF/PDA/SiO_2-silicone composites enhanced by 57.28 and 41.84 %	[164]
Carbon fibers (no further information accessible)	Functionalization with nanoparticles of Ti, Al by reacting CF with nano powers of Titanium, Aluminum in the presence of KCl, NaCl and phenolic resin and subjecting the treated CF to carbonization at 900 °C for 60 min followed by treatment of the carbon film coated Ti, Al functionalized CFs with LiF and HCl.	ILSS of the composite increased by 20-30 % ; thermal stability of the resulting fiber is claimed to be enhanced upto 900 °C; probably, TiC and AlC NPs might have formed on the CF surface imparting thermal stability to the material	[165]

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Carbon fibers supplied as carbon fiber cloth (CFC, PAN based) by Avantor performance materials, Poland, S. A.,	Ag NPs were deposited on TiO_2 by wet impregnation and $NaBH_4$ reduction; Ag NP coated TiO_2 was supported on carbon fiber cloth (CFC) by spraying and drying method; Ag/TiO_2-CFC	Photo catalyst for the partial oxidation of NO to N_2O; NO_2 formation hindered by Ag NPs; NO removal rate, 95 %; Possibility of N_2O reduction to N_2 by modifying Titania	[166]
Carbon fiber bundles (24 K, pristine, unsized) supplied by Carbon Nexus, Waurn Ponds, Australia	Deposition of Fe_3O_4 (magnetite NPs) using ammonium iron (II) sulphate precursor; thermal treatment in N_2 at 1000 °C	IFSS improved by 84.3 %; surface energy enhanced by 5.5 % ; ILSS enhanced by 25.9 %	[167]
3 K plain weave mat carbon fiber (T 300), filament diameter, 7 µm	Electrophoretic deposition (EPO) of graphene based nano fillers (GBN), graphene, graphene oxide, graphene hydroxyl, graphene carboxyl;	Graphene-COOH modified CF should highest enhancement in flexural strength (9.6 %) and inter laminar shear strength (22.9 %)	[168]
Carbon fiber (diameter, 7 µm, length, 0.1-1 mm) provided by Toray, Japan	Spark plasma sintering for coating SiC;	Improvement in oxidation resistance; temperature (on set) of oxidation increased by 140 °C; (usually untreated CFs gets oxidized rapidly at 500 °C)	[169]
T300-3K, 7 µM (from Toray Industries, Inc, Tokyo, Japan)	Uniform layer of SiO_2 nanoparticles was coated on the surface of CFs using a polydopamine (PDA) resulting in a nanohybrid coating (PDA/SiO_2).	Surface energy of CF was enhanced by 42.5 % due to nano hybrid layer coating. Likewise, water contact angle decreased from 78.5 to 41.89°; ILSS of the composite, CF-PDA/SiO_2-methyl phenyl silicone resin (PMSR) increased by 63.59 % by the modification of CF's with nanohybrid coating.	[170]
Carbon (polyphenylene terephthalaide, p-PPTA) fibers, Kevlar® K-29, dtex 3300; provided by Changzhou Gaoyuan group co., Ltd., Changzhou, China	Grafting polydopamine (PDA) plyethyleneimine (PEI)-graphene oxide onto CF surface;	Adhesive properties of CF to rubber matrix enhanced;	[171]

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Carbon fiber (T-650), 7 μm diameter; Cytec Engineering Materials, West Peterson, NJ	Carboxylated nanodiamond (ND) functionalized CFs	Carbon fiber microelectrodes (CFMEs) for neuro transmitter (for example dopamine) detection; sensitivity of detection increased by 2.1 times; limit of detection improved to 3 ± 1 nm	[172]
Carbon fibers (tensile strength, 3500 MPa, Toray industries, Inc.,)	Chemical grafting of silica nanoparticles (SiO_2 NPs) using the bridging toluene 2, 4-diisocyanate	Water contact angle decreased from 78.5 to 44.12°; surface energy increased from 35.87 to 50.36 mN/m; ILSS and IFSS enhanced by 50.97 and 35.92 %	[173]
Carbon fibers provided by Toho Tenax, HTS 45 E23, linear density 800 tex, Japan	CNT deposition by aerosol spray deposition	Tensile strength of the modified CF/epoxy composite enhanced	[174]
Carbon fibers (3×10^3 single filaments per tow; diameter, 7 μm; tensile strength 3500 MPa; tensile modulus 230 GPa; provided by Toray industries Inc., Tokyo, Japan	Carboxyl functionalized holloysite nanotubes (HNTs) grafted to CF surface using 3-aminopropyl triethoxy silane (APS)	Water contact angle decreased from 78.5 to 42.95°; surface energy increased from 35.87 to 62.13 mNm^{-1}; anti hydrothermal aging behavior of the modified CF/methyl phenyl silicone resin (MPSR) composite improved; ILSS retention ratio enhanced by 93.61 % upon hydrothermal aging;	[175]
Carbon fiber (12 K, 1.80 g/cm^3), diameter, 7 μm; provided by Shenying carbon fiber co Ltd., China	Graphene oxide (GO) modified with hexamethylene diisocyanate (HDI) tripolymer coupling agent and grafting the modified GO to the oxidized CF surface	IFSS enhanced by 40.2 %	[176]
Mesophase pitch-based CFs (Mitsubish Chemical Corp) used in the form of brush	Surface coating of CF with poly amine (PANI)/reduced graphene oxide (rGO)	Power density of microbial fuel cell increased by 1.21 times when used as anode electro catalyst;	[177]

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Unsized T300 PAN-based 12 K tow fibers; provided by Jilin petro chemicals, China	Oxidation with conc. HNO_3 followed by treatment with ferrous oxalate; deposition of magnetite (Fe_3O_4) nanoparticles on CF surface	Removal efficiency of chemical oxygen demand, ammonia nitrogen and total phosphorous increased by 7.18, 10.30, and 9.40 %	[178]
Carbon fibers, diameter 7 µm; tensile strength, 3500 MPa; purchased from Toray industries;	Grafting 3-aminopropyl triethoxy silane (APS) to CF; CF-Silaxone was further functionalized with SiO_2 NPs (through TEOS hydrolysis)	Presence of Si-O-C bonds at the interface increased hydrothermal aging resistance; ILSS and IFSS increased by 46.79 and 39.61 %	[179]
Carbon fibers, 3 mm length and 7 µm diameter	CNT grafted to oxidized CF surface through 3-amino propyl tri ethoxysilane (KH550) coupling agent	Flexural strength of cement pastes at 3, 7 and 25 days enhanced by 48.5, 42.2 and 45.5 %	[180]
Carbon fibers, 12K A-42 procured from Dow Aksa, Turkey	Coating of porous carbon with Fe (small particles); Aqueous desized CFs were soaked in a solution of glucose and $FeSO_4$ $7H_2O$ followed by drying and high temperature carbonization 950 °C in Ar/H_2 for 6 h)	Modified CF find application in textile composites	[181]
CF continuous biwoven 0°/90° PAN-based, supplied by MIS zoltek corporation Inc.,	Electroless coating of Ni (nano to µm size) on CF surface (20 min);	70 % enhancement in storage modulus; flexural strength and ILSS enhanced by 20 and 69 %	[182]
T-700 PAN CFs provided by Japan, Tenax Co.,	In situ growth of CNTs on CF surface using Fe nanoparticles as catalyst by CVD	Flexural strength of the epoxy composite enhanced by 28.1 %	[183]
PAN based CF (length, 25-56 µm; diameter, 7 µm); provided by Nanjing fiber glass R and D Institute, China	Coating CF surface with SiO_2 NPs (500 nm) by treatment with TEOS in the presence of NH_3	Hardness and elastic modulus of polyimide composites enhanced by 22 and 12.2 % respectively; wear resistance enhanced by 75 %	[184]

Carbon fibers (diameter, 6-8 µm; length, 3-5 mm) provided by Nanjing Weida composite material Co Ltd., China	Growth of ZnO nanowires on the CF surface via hydrothermal synthesis	Paper based friction materials; greater wear resistance; stable dynamic friction coefficient; excellent tribological properties; promising wet paper-based friction material	[185]
Carbon cloth fibers	Hydrothermal and chemical bath deposition were used to grow Co_3O_4 nanowires on carbon cloth fibers; and further modified with NiO and MnO_2 to produce core shell structure;	Cell configuration of Co_3O_4@NiO (1:2)/cc(a|c)Co_3O_4@MnO_2 (1:2)/cc , where cc is the carbon cloth support used, exhibited maximum power density of 33.8 $mWcm^{-2}$	[186]
Carbon fiber	PdCo alloy NPs were supported on CFs by impregnation and reduction process and PdCo/CF was used as anode electro catalyst for the electrolysis of coal for hydrogen production	16.9 % enhancement in the anode electrocatalyst performance for hydrogen production from coal electrolysis	[187]
Carbon fiber T 300 (length, 1 mm; diameter 7 µm; strength, 4900 MPa; tensile modulus, 240 GPa) provided by the Japanese company Toho	Oxidized CF (with oxygen functionality; carboxyl, hydroxyl, carbonyl) was produced by treatment with HNO_3; oxygen functionality reduced to hydroxyl groups by treatment with $LiAlH_4$ and $NaBH_4$; hydroxyl groups were further treated with silane coupling agent (vinyl triethoxy silane) to obtain silanized CF which were subsequently functionalized with Ag (treatment with $AgNO_3$ and reduction with $NaBH_4$) nanoparticles followed by grafting with acrylate	Silanized CF-silver-acrylate composite exhibited improved performance of electrical conductivity and antibacterial activity with allocation in medicine;	[188]
High strength PAN-based 232 twill weave 3K carbon fabric provided by CNME international, China	Functionalized (hydroxyl carbonyl and ether type) multi walled CNTs were coated on CF surface by electrophoretic deposition (EPD);	Flexural strength and ILSS of modified CF epoxy composites increased by 15 and 18 % respectively	[189]

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PAN based carbon fiber tow (unsized/ untreated, JILIN tow-24K precursor) provided by Carbon Nexus at Deakin University	Amino functionalized nano clay (montmorillonite) nano plates were grafted to the glycedyl trimethyl ammonium chloride functionalized CFs by cation exchange process;	Surface roughness, coefficient of friction and BET specific surface area of carbon fibers were enhanced by 61, 10 and 5 % respectively;	[190]
Carbon fibers produced using wet-spun poly acrylonitrile (PAN) as the precursor fiber; unsized carbon fibers provided by carbon Nexus facility at Deakin University	Electrochemical deposition of graphene oxide sheets followed by modification with carbon dots	Improved selectivity and sensitivity (6.5 nA/μM) in the detection of neurotransmitter (dopamine); detection limit 0.02 μM	[191]
Toray T700 carbon fibers, diameter, 7 μm; length, 10 cm;	In-situ growth of SC nanofibers by catalytic chemical vapour deposition at 1000 °C using Ni nanoparticles as catalyst coated on CF surface by electroplating from $NiSO_4$ precursor (15 wt.%)	Enhanced microwave absorbance and oxidative resistance; reflectivity of microwave radiation is less than -10 dB in the frequency range of 9.2 to 11.7 GHz;	[192]
JT-400A-3K, provided by Jilin Shen Zhou carbon fiber Co Ltd.,	Co-grafting of CNT's and graphene oxide on CF surface leading to uniform coating	Polar functional groups and surface energy enhanced; ILSS and IFSS of composites (epoxy matrix) enhanced by 48.12 and 83.39 %	[193]
Carbon fibers (1K T-300™) provided by Toray industries, Japan	MWCNT's oxidized by conc. HNO_3 treatment followed by grafting onto the CF surface by electrophoretic deposition	Tensile strength, failure strain, Young's modulus of the composites (epoxy matrix) increased by 9.86, 44.01, and 12.4 % respectively	[194]
T700, provided by Toray, Japan	Amine functionalized graphene oxide (GO-NH_2) grafted covalently to CF surface	36.4 % enhancement in IFSS	[195]
Carbon fiber cloth	Graphene coating; Glucose oxidase immobilized modified CF used as anode and Bilirubin oxidase immobilized modified CF as cathode in the biofuel cells	Improved performance as electrodes for bio(glucose) fuel cells; power density of the fuel cell enhanced by 85.4 %	[196]

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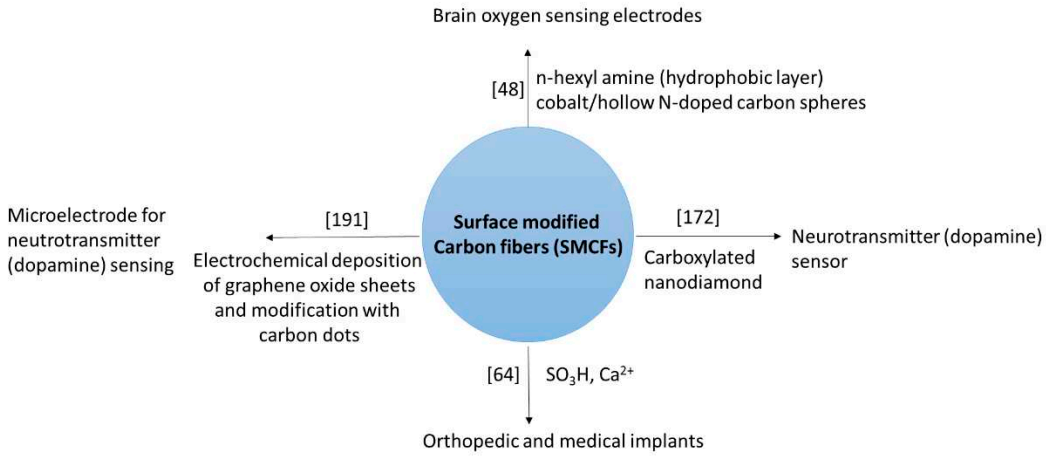
PAN based CF two, T700, 12K; provided by Toray industries, Japan	Graphene oxide deposition; treatment with H_2O_2 and HNO_3 in a electrophoretic deposition process	ILSS of the composite (with epoxy resin matrix) enhanced by 55.6 %	[197]
Carbon fibers provided by Toray industries Inc.,	Grafting of trisilanolphenyl-polyhedral oligomeric silsesquioxanes (trisilanolphenyl-POSS) nanoparticles using toluene – 2, 4-diisocaynate (TDI) as briding agent	ILSS and impact resistance of composite (methyl phenyl silicone resin, MPSR matrix) enhanced by 41.91 and 28.65 % respectively	[198]
Carbon fibers, T700SC-12000-50C, 12K, provided by Toray industries, Inc.,	Ag NPs deposited on CF by electrochemical means in the presence of poly(vinyl pyrrolidone) to control the geometric shape and size of Ag NP's	Tensile strength of Ag-CF enhanced by 52.7 %; IFSS of composite (epoxy matrix) enhanced by 27.2; 2 fold enhancement in electrical conductivity compared to pristine CF observed	[199]

2.4. Discussion and Summary:

Surface modified carbon fibers (SMCFs) influence almost all spheres of human life by improving the standing of living and alleviating the suffering of mankind. Advent of materials based on SMCFs is envisioned to revolutionize the chemical, energy, environment, health, infrastructure, materials, agricultural, defense and chemical industries in new and incomprehensible ways owing to drastic enhancement in the properties and the resulting multi functionality of these materials. Representative examples of the application of SMCFs revolutionizing various industries are as shown in Schemes 1-5 and are self-explanatory.



Scheme 1. Revolutionary applications of surface modified Carbon fibers (SMCFs) in health

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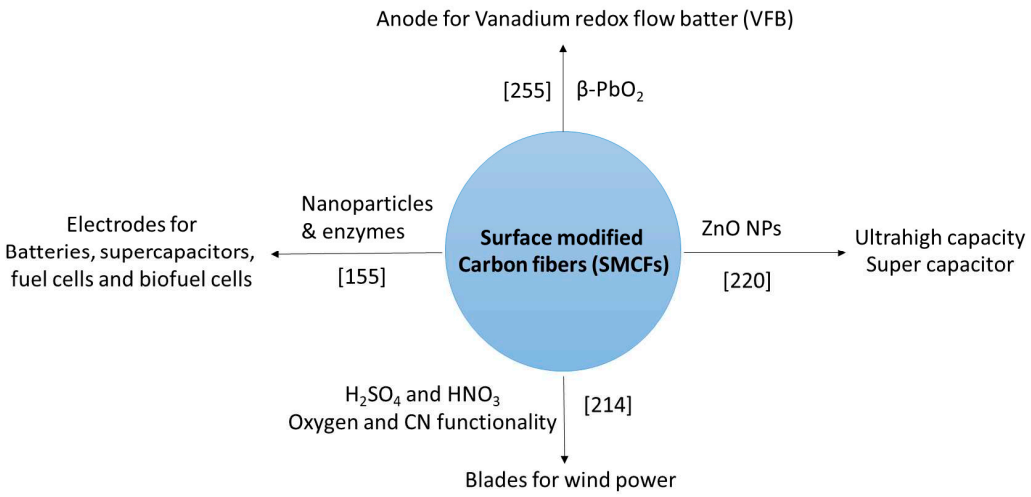
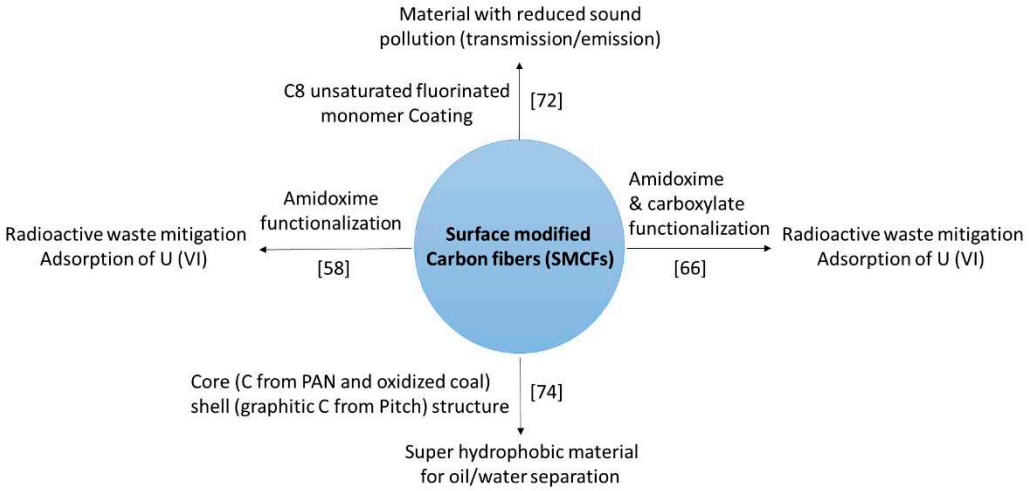
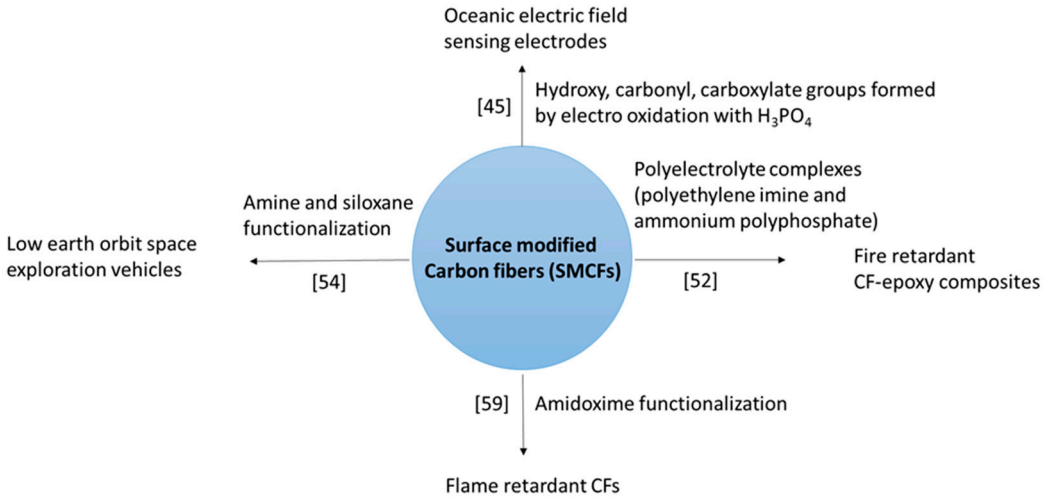


Scheme 2. Revolutionary applications of surface modified Carbon fibers (SMCFs) in energy



Scheme 3. Revolutionary applications of surface modified Carbon fibers (SMCFs) in environment



Scheme 4. Revolutionary applications of surface modified Carbon fibers (SMCFs) in defense

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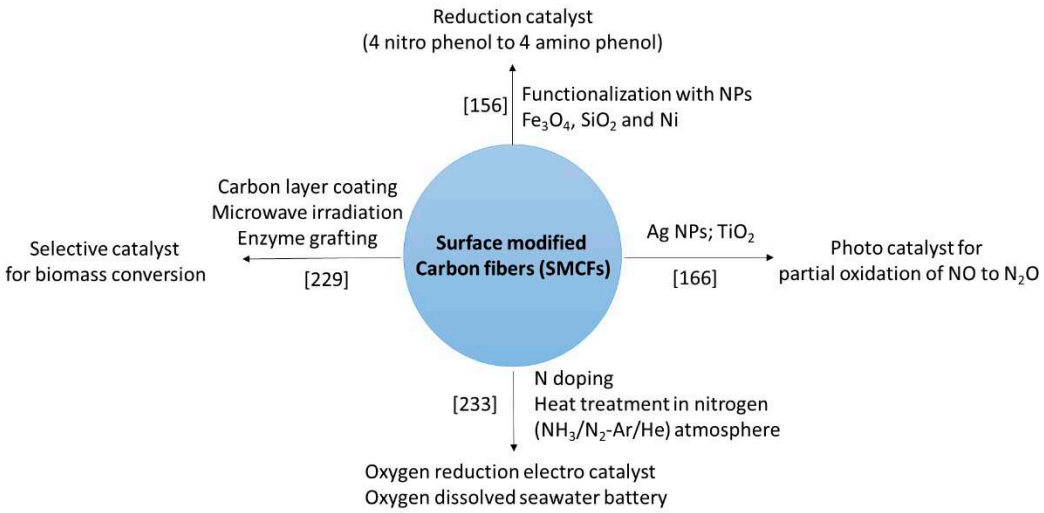
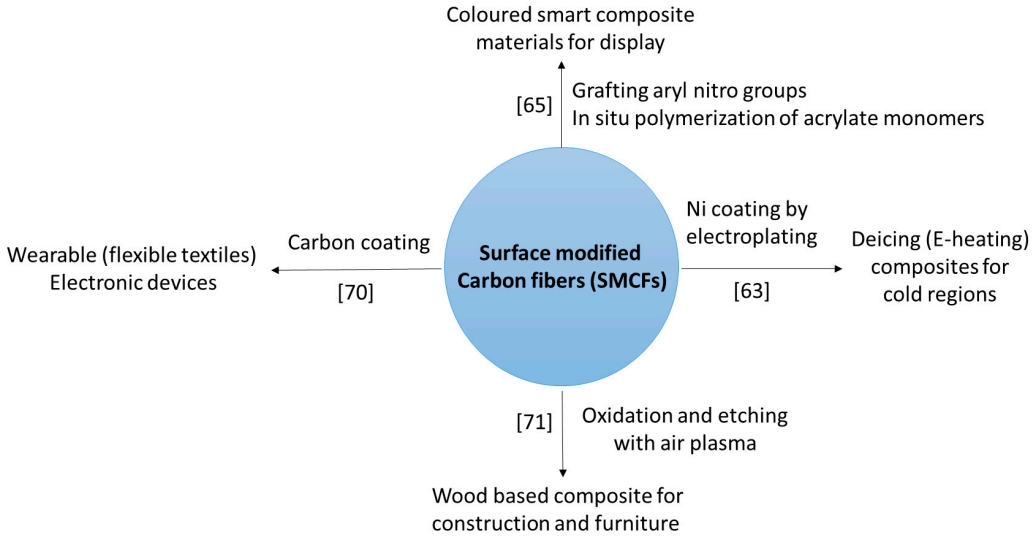


Scheme 5. Revolutionary applications of surface modified Carbon fibers (SMCFs) in catalysis



Scheme 6. Revolutionary applications of surface modified Carbon fibers (SMCFs) in smart materials

Common organic precursors used for the production of CFs include polyacrylonitrile (PAN) and pitch with a market share of 97 and 3 % respectively. Owing to their low price and wide availability PAN based CFs (20 €/kg) CFs are extensively studied and used compared to pitch based fibers (100 €/kg) as evident from the research findings summarized in Tables 3 & 4. However, diverse methods are being developed to produce CFs with varying properties [200]. Effect of surface modification method is substrate (CF) specific and cannot be generalized. This is evident from the specific example of physical activation methods (NH3 plasma, O2 plasma, air plasma, electrolysis) employed with carbon fibers of equivalent (electrode surface and material) properties, namely, HTS40 and CT50. Significant effects of activation were observed in the case of CT50 while the effects of activation were only marginal in HTS40. Compared to CT50, HTS40 showed better electrode performance when used as electrode material for bio electro chemical systems (BES). Carbon fiber properties influenced the bacterial (Shewanella Oneidensis MR-1) current generation properties. Potschke et al., reviewed the selection criteria of CFs for application as electrode material, especially for BES. Commercial CFs namely, HTS40, CT50 and T300 were supplied by Teijin carbon Europe Gmbtt, Japan/Germany, SGL carbon, Germany, and Toray Industries Inc., Japan were studied. Effect of physical activation and desizing on electrode performance were examined. Surface activation was found to reduce the startup time while its impact on maximum current density could not be ascertained [200]. On the contrary, removal of sizing (protective coating) of CFs is very vital

for all the fibers studied for BES application and such desizing enhanced he maximum current density up to 40 fold. Exact composition of the sizing is a subject of intellectual property rights (IPR) of the industries. In the specific example of T300 fabrics, highest desizing (83.3 %) is achieved by pyrolysis in inert atmosphere (N_2, 500 °C, 20 min) resulting in 45 fold enhancement in BES performance. A combination of free filament ends and high degree of graphitization enhanced the electrode performance by 100 %. This is to demonstrate that the selection of CF (substrate) influences the electrode performance.

The method of surface modification of CFs is application specific. No single modification methods is applicable for generating the desired functionality on CFs that suits all the demanding applications. As a result there is scope for further development of surface modification methods as diverse as the target applications, demands, challenges and needs of human societies. However, generalizations can be drawn for the knowledge generated by research and development in the field, especially over the past decade (2010-2010). Even though, a web of Science search with the keywords, namely, "carbon fibers" and surface modification", yields 3044 results, these results could be further screened out to few hundred papers based on major advances made in this field. The summon substance of such screened out results, is that the applicability of CFs can be drastically enhanced in astonishing ways by surface modification. Essentially, surface modification methods impart polarity (oxygen functionality; presence of metal NPs; grating of carbon nanomaterials; coating of functional polymers) to the CF surface which is otherwise inert and non-reactive.

Commercial significance of the surface modification of CFs is evident from the very many patents issued on this subject recently [201–278]. A glimpse of the kind of surface modifications reported in patent literature are highlighted in Table 6.

Table 6. Highlights of the surface modification methods developed in patent literature (2020-2010).

Surface modification method	Reference
UV-ray irradiation for C-C bond scission	[205]
Imidization for molecular assembly of condensed aromatic ring structure	[207]
Oxidation with H_2SO_4	[208,209]
Oxidation with HNO_3 followed by Si-O-Si grafting by treatment with silane coupling agent	[210]
Sizing with thermoplastic comprising of cyclic phenyl sulfide	[211]
Desizing, plasma treatment followed by sizing	[212]
Graphitized spinal nano CF mixed with ethanol and ball milled followed by acid treatment	[213]
Desizing; oxidation with a mixture of H_2SO_4 and HNO_3; CN functionalization followed by applying performance paint for CF blade for wind power application	[214]
CF in the form of woven carbon fiber (WCF) were treated with plasma followed by coating nanoparticles of ZnO	[215]

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Sizing with thermosetting resin; -COOH groups generated on the CF surface	[216]
CNT functionalized CF; ILSS increased by 15 %	[217]
Sizing with polycarbonate followed by silane agent KH-550 to generate Si-O-Si functionality	[218]
Desizing; plasma treatment; coating with thermoplastic type resin oil agent (oil agent is of a poly urethane, polyethylene, polypropylene or acrylic type); interfacial bonding in composites enhanced	[219]
ZnO NP functionalized CF for ultrahigh capacity super capacitor application	[220]
Graphene oxide grafting to silane functionalized CF's by electrophoretic deposition; IFSS of the composite enhanced	[221]
Nano silica (20-100 nm) functionalized spiral nano carbon fiber	[222]
Electro oxidation of CF (obtained from heating asphalt and PAN at 1200-3000 °C) using composite electrolyte containing nitrogen (urea) and –OH group (glycerol) containing compound; oxygen and nitrogen content enhanced; adhesion of CF to rein matrix improved	[223]
Acryl chloride based CF grafted with branched tannic acid; IFSS increased from 49.5 to 93.2 Mpa	[224]
CF modified with double sizing agent; treatment with silane agent (generates Si-O-Si groups) followed by grafting amino functionalized MWCNTs; cutting performance of composite enhanced by 24 %; tensile property improved by 29 %;	[225]
Carbon nanofiber surface modified by treatment with silane, titanate and aluminate coupling agents; impact strength increased by 30 %	[226]
Electron beam (50-1000 kgy; 1-3 mev) irradiation of acid treated CFs; multifunctional groups formed; mechanical strength improved	[227]
Polypyrrole coating on CF's by in situ polymerization of aqueous solution of pyrrole in the presence of oxidizing agent; good adsorption efficiency for sewage pollutants	[228]
Enzymes (xylanase, invertase, alphaarabinose, glycosidase and lignin peroxidase) grafting on to organic agent (succinic acid, lactic acid in the presence of H_3PO_4) modified by microwave irradiation (15 min; 915-2450 MHz); potential catalyst for biomass conversion;	[229]
CF oxidized (with $KMnO_4$, H_2O_2, potassium perchlorate, potassium ferrate) and reduced (with HCl, HBr, oxalic acid, oxalate) followed by grafting biological species; used as biological bomb and biomedical material	[230]
Double phase CNTs grafted to CF surface for power source devices	[231]

CF is surface grafted with acryl amide; improvement in shear strength and mechanical property	[232]
Nitrogen doping on CF surface by heat treatment in nitrogen containing atmosphere (NH_3 gas; N_2 gas); application as oxygen reduction electro catalyst (cathode) in oxygen dissolved seawater battery	[233]
Electro oxidation, carbonization and activation resulted in acid-base active sites; enhanced hydrophobicity and pollutant adsorption from sewage waste;	[234]
SiC NPs coating on CF followed by ball milling; toughness and wear resistance of material enhanced	[235]
CF functionalized with titanium carbide and titanium diboride NPs, CF/TiC-TiB_2; wear resistance, corrosion resistance and fatigue resistance enhanced; material is applicable for the fabrication of ship, automobile and air craft components;	[236]
Ultrasound assisted electro chemical oxidation with KMnO4 in the presence of ammonium salts like ammonium carbonate; tensile strength increased;	[237]
Graphene (mono/multilayer) coating followed by plasma (methane, ethane, ethane, ethyne, benzene, and ethanol) treatment	[238]
Desizing, oxidation (with H_2SO_4 and HNO_3) followed by grafting of thermosetting resin (unsaturated polyester resin, epoxy or phenolic resin); minimal performance loss;	[239]
Oxidation with H_2SO_4; reduction with $LiBH_4$; deposition of TiO_2 NPs from tert butyltitanate precursor; wettability, roughness of the modified CF enhanced; mechanical properties of the composite increased;	[240]
Treatment with silane coupling agent KH560 (RTM: 3-glycidoxy propyl trimethoxy silane); dispersion of the CF by treatment in diethyl ether and $LiBH_4$ under ultrasound irradiation	[241]
TiO_2 NPs deposited on CF surface by treatment with titanium acid ester coupling agent; followed by thermal treatment in air at 600 °C; ILSS improved to 124.6 MPa;	[242]
Pt NPs and Al_2O_3 NPs (using trimethyl aluminum precursor) were deposited on CF surface; heterogeneous catalyst with reduced hydrogenation activity;	[243]
Oxidation (treatment with H_2SO_4 and HNO_3) followed by treatment with fluorinated organosilane in DMSO; used as filter screen filler with pore size 2-100 μm	[244]

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Oxidation with conc. HNO_3 followed by polyimide coating; improvement in mechanical property;	[245]
Desizing, oxidation (conc HNO_3 treatment), amination (by treatment with 3-amino propyl triethoxy silane) grafting graphene oxide; wettability and surface roughness enhanced; strength enhanced by 20-50 %; toughness enhanced by 35-40 %	[246]
Coating silicone layer (< 1μm); modified CF should excellent stability; enhanced strength and rigidity of the CFRP	[247]
Grafting of methyl methacrylate (MMA) monomer to Ni (submicron) coated CF; application in high temperature plastics with electromagnetic shielding potential	[248]
Hydroxy functionalized CF (treatment with EtOH followed by ball milling) were grafted with pyrrole monomers followed by oxidation with H_2SO_4; modified CF/poly pyrrole composite exhibited excellent electromagnetic wave absorption performance in 7-17 GHz microwave band	[249]
Anode electro oxidation in the presence of ammonium bicarbonate; graphite plate used as cathode in the anodized surface treatment	[250]
SiO_2 NPs functionalized CFs; thermal stability, wettability and roughness of CF are improved; in the composite material stress concentration is alleviated	[251]
Hydroxy functionalized CF (by treatment with ethanol) and grafting acrylate monomer followed by oxidation with inert gas plasma; modified CF-polycarbonate composite find application in electric, aerospace, military and chemical industries	[252]
Desizing, oxidation, acylation and grafting bis (3-aminophenyl) phenyl phosphine oxide	[253]
Carbon fiber in power form is oxidized with conc. H_2SO_4; oxygen functionality generated ; modified CF find application in aviation vehicles, buildings and chemical industries	[254]
Carbon fiber fabric modified with β-PbO_2 by linear sweep voltammetry; used as anode material for vanadium redox flow battery (VFB) ; energy conversion efficiency and power density increased	[255]

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Removal of styling agent; acidification ; in situ polymerization of amine monomer under microwave and sonication with sodium nitrite as initiator leading to polyamide amine grafting; specific desired functionality generated depending on the modifying chemical agent; wettability of modified CF improved; strength of the composite enhanced;	[256]
Oxidation with conc. HNO_3	[257]
Oxygen and nitrogen co-doped CF (PAN based) through electrochemical oxidation; excellent pseudo capacitance property	[258]
Treatment with modified with hydroxyl group containing compound and poly amide; modified CF/polyamide composite is used for transportation, sports, medical and civil construction equipment	[259]
Aniline functionalized CF; enhanced roughness and IFSS	[260]
Mesoporous inter leaving multilayer graphene modified CF by coating with active metal (Li, Na, K, Cs) layer, carbon fiber felt, high temperature activation in inert atmosphere followed by removal of active metal and generating mesoporosity and graphitic carbon	[261]
Oxidation, grafting of hexachlorocyclotriphosphazene phosphine followed by reacting with graphite to coat graphene on CF surface	[262]
Cryogenic treatment of CF with liquid nitrogen; surface roughness of CF enhanced	[263]
Grafting catechol type structure (treatment with epinephrine) on CF surface by polymerization; useful in CFRP, luminescent and antibacterial materials	[264]
Electro oxidation with strong electrolyte (alkali metal hydroxide) followed by electro oxidation with week electrolyte (aq. NH3); modified CF/composite material useful in aerospace, defense and civil fields;	[265]
Oxidation with potassium persulfate ($K_2S_2O_4$) and $AgNO_3$; mechanical performance enhanced;	[266]
Plasma treatment under inert atmosphere for high temperature carbonization of CF (PAN based) sized with E-51; grafting liquid modifiers (maleic	[267]

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anhydride/acrylic acid) by atomizing spray nozzle, wettability, surface energy increased; ILSS enhanced	
Electrochemical deposition of graphene oxide followed by thermal treatment (80-600 °C; 10 sec – 60 min) in air/oxygen/ozone/nitrogen/argon/ammonia gas;	[268]
Coating of phenolic (o-cresol, m-cresol) monomer and aldehyde (formaldehyde; n-propionaldehyde) in the presence of alkali metal hydroxide catalyst followed polymerization and carbonization; Shear strength increased by 12-53 %; adsorption capacity of the fiber enhanced;	[269]
Oxidation with HNO_3 (45-85 mass %) followed by hydrothermal treatment (120-180 °C; 126 MPa, 3-6 h) followed by thermal activation (800-950 °C; 2-4 h; N_2/CO_2); useful for the treatment of Sewage sludge;	[270]
Surface etching by high energy particles beam (γ ray beam/plasma beam/X-ray beam; of energy 25-120 eV for 80-150 min) followed by cleaning with solvent under ultrasonic irradiation to generate micro nano groves on the surface; interfacial bonding strength enhanced;	[271]
Oxidation by treatment with a mixture of H_2SO_4 and $KMnO_4$; oxygen content enhanced by 22.7 %; strength improved; useful in aerospace, automobile, transportation; construction and chemical industries;	[272]
Ultrasonic treatment (20-100 kHz; 200-2000 W; 20-70 °C; 15-40 min); treatment with silane coupling agent followed by oxidation with $K_2Cr_2O_7$/sodium hypochlorite/H_2O_2/potassium persulfate; ultrasound pretreatment enhanced surface chemical modification; mechanical property of the CF enhanced;	[273]
Nano graphene coating followed by plasma treatment; mechanical, conductivity and heat resistance properties enhanced;	[274]
Electro chemical treatment in the presence of salts like ammonium bicarbonate, ammonium chloride, ammonium sulfate and ammonium oxalate (0.1-3 mA/cm^2; 10-180 sec; 10-60 °C); tensile strength increased; shear strength enhanced;	[275]
CNTs (single and multiwalled) oxidation with H_2SO_4 and HNO_3; oxidized CNT's were electrochemically grafted to CF's	[276]
Grafting with hexachlorocyclotriphosphazene followed by caprolactum monomers; surface reactivity of CF enhanced;	[277]

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CNT's were grown on CF surfaces by chemical vapour deposition with Ni catalyst [278]

Research and development, especially on the surface modification of carbon fibers, is intensifying as evident from the increasing trend in the publications in this field with time. A web of Science search with the keywords, "carbon fibers" and "surface modification" for the years from 2010 to 2021 shows growing interest in field of "Carbon fiber surface modification" as shown in Figure 7.

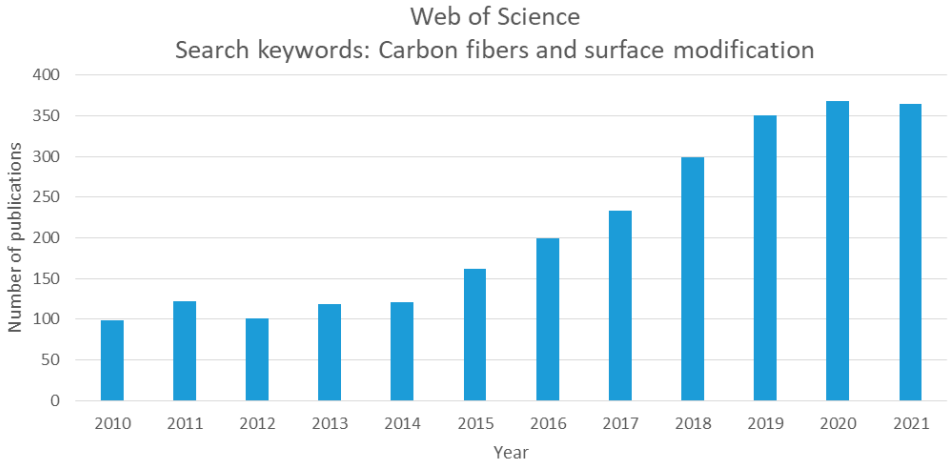

Figure 7. Growing interest in the research field of "Surface modification of carbon fibers" during 2010-2021.

For further knowledge on the surface modification of carbon fibers by physical (plasma, microwave, electron and gamma ray, laser irradiation), chemical (sizing, oxidation with mineral acids, nanoparticle deposition, grafting of polymers, amine and oxygen functionalization), electrochemical (oxidation, in situ polymerization, grafting) methods, leading to the generation of surface polarity, improving surface roughness, energy and wettability and inducing multifunctionality, and enhancing the mechanical properties of the CF reinforced composites by improving the interfacial adhesion, the readers are directed to the following literature published during 2015-2010 [279–365]. Moreover, a bibliography of the latest updates in the field of surface modification of carbon fibers during the past two years (2021-2022) has also been provided [365–459].

Conclusion:

Literature on the subject of "surface modification of carbon fibers" over the past decade (2020-2010) is critically reviewed. Grafting was pointed out be some reviewers on this subject as the ideal surface modification method for CFs. However, recent evolution in this field, during the past decade, clearly demonstrate that hybrid surface modification methods are the future, meaning, a combination of two or more methods like oxidation, followed by grafting; grafting followed by coating; oxidation followed by electro polymerization should be adopted. Moreover, the method of modification is application specific. Depending on the problem at hand and the need, the functionality (modification method) need to be tuned. The 459 research findings reviewed in this paper provide the needed guidance to researchers and industrial practitioners working with carbon fiber based materials, for fine tuning of surface modification methods depending on the application. The method of surface modification of CFs is application specific. No single modification method is applicable for generating the desired functionality on CFs that suits all the demanding applications. As a result there is scope for further development of surface modification methods as diverse as the target applications, demands, challenges and needs of human societies. However, generalizations can be drawn for the knowledge generated by research and development in the field, especially over the

past decade (2010-2020). Even though, a web of Science search with the keywords, namely, "carbon fibers" and surface modification", yields 3044 results, these results could be further screened out to few hundred papers based on major advances made in this field. The summon substance of such screened out results, is that the applicability of CFs can be drastically enhanced in astonishing ways by surface modification. Essentially, surface modification methods impart polarity (oxygen functionality; presence of metal NPs; grating of carbon nanomaterials; coating of functional polymers) to the CF surface which is otherwise inert and non-reactive.

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