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Effect of carbon fiber surface treatment with HNO3 and KOH on the interfacial bonding of PMMA resin composite

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This work studied the surface, interface state and physicochemical properties of HNO3-treated and KOH-treated carbon fiber. Poly(methyl methacrylate) (PMMA) composites were prepared by the autoclave molding process using surface-treated carbon fiber as reinforcements. The physical and chemical states of the carbon fiber surfaces and the micro-interface properties and interlaminar shear properties of the composites were studied. The results show that the surface of the HNO3-treated carbon fiber has more groove structure and higher surface roughness and thus forms a better physical bond with the resin matrix. Although the oxygen-containing functional groups of the two carbon fibers are equivalent, the surface oxygen of the HNO3-treated carbon fiber is relatively high, which is beneficial to form a better chemical bond with the matrix resin, and the interfacial shear strength is about 14% higher than that of the KOH-treated carbon fiber composite.

Keywords: *Carbon fiber, PMMA, Surface treatment, Interfacial shear strength, chemical bond*

1. Introduction

Due to its high specific modulus and specific strength [1], good electrical conductivity, corrosion resistance [2], and low linear thermal expansion coefficient, carbon fiber is widely used as a reinforcing phase in polymer composites [3–5]. Especially in the aerospace and transportation sectors, carbon fibers are increasingly used instead of metals in order to reduce the mass of aircraft and vehicles and improve their fuel efficiency and dynamic performance [6–9]. However, due to the few active carbon atoms and low surface energy at the edge of the surface, carbon fibers are inert [10, 11], and have poor wettability with the resin matrix [12]. the production of carbon fiber composites generates internal pores. This leads to discontinuity at the interface between the fiber and the matrix, which adversely affects the mechanical properties of the composite, especially for interfacial shear, interlaminar shear, and impact resistance [13, 14]. Therefore, improving the combination degree of carbon fiber and matrix and giving full play to the excellent performance of carbon fiber is of great significance for improving the performance of composite materials [15].

The interface properties between the fiber and the matrix can be effectively improved by modifying the surface of the carbon fiber. There are three main mechanisms of modification. The first is to etch the surface of the carbon fiber and make it rough, which increases the fiber's surface area. The second is to form acidic functional groups on the surface of the carbon fiber to promote the formation of chemical bonds with the polymer matrix and improve the surface activity and wettability of the fiber [17]. The third is to remove the weak interface layer on the fiber surface and reduce the defect of carbon fiber materials [18]. The first two modification mechanisms often occur simultaneously [13]. Although the bonding ability between the fiber and the matrix can be improved through surface treatment, which is beneficial to improving the strength of the composite material, some surface treatment methods will also weaken the strength of the carbon fiber body, especially the etching effect on the fiber caused by excessive surface treatment [19]. Therefore, the final performance of the composite material depends on the result of the joint action of the two. Carbon fiber sur-

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face treatment can be divided into oxidation treatment (liquid phase oxidation, gas phase oxidation, and catalytic oxidation) and non-oxidation treatment (high-efficiency whiskering, pyrolytic carbon deposition, and plasma polymerization), according to whether it is oxidized [20].

The surface oxidation theethod is to place the carbothe fiber in an oxidant to undergo an oxidation reaction. According to the state of the oxidant, the surface oxidation treatment is divided into two types: gas phase oxidation and liquid phase oxidation. Gas-phase oxidation usually uses O2, O3, CO2, and F2 as oxidants to treat the surface of carbon fibers under heating conditions, and generates oxygen-containing polar groups (such as —COOH, —OH, —C, —Othe.). The polar bonds formed between these polar groups and the polymer can effectively improve the interfacial bonding performance between carbon fibers and the matrix [21]. Liquid-phase oxidation mainly uses solutions such as HNO3, H3PO4, and KMnO4, or mixed solutions as oxidants to treat the surface of carbon fibers. The oxidation treatment of carbon fiber not only changes the elemental composition of the fiber surface, but also increases the number of oxygen-containing functional groups. The concentration of oxidizing medium, treatment time and temperature, and the properties of the fiber itself will all affect the treatment effect of the fiber. Compared with the gas-phase oxidation method, which produces a difficult-to-controloxidation reaction, the liquid-phase oxidation method is milder and more effective, and is currently the preferred method for carbon fiber surface treatment [13]. Dzul-Cervantes et al. [22] measured the contact angle of carbon fiber and epoxy matrix to reflect the wettability of the two, and found that the average contact angle of untreated carbon fiber and epoxy was 43°. The main reason for the decline is that the oxygen-rich groups on the surface of the carbon fiber reacted polarly with the O=S=O groups on the surface of the matrix, and under the joint action of nitric acid oxidation and silane coupling agent chemical adsorption on the carbon fiber surface. The thermoplastic resin poly(methyl methacrylate) (PMMA) and recycled carbon fibers were obtained by pulverizing carbon fiber reinforced plastics (CFRP). PMMA particles were adsorbed on the carbon fiber surfaces via electrostatic interactions, to promote the interfacial adhesion between the carbon fibers and the PMMA resin and thereby improve the dispersion of the fibers in the resin [23].

This work aimed at the surface, interface state, and physicochemical properties of HNO3-treated and KOH-treated carbon fiber. Through the study of fiber micromorphology and interface mechanical properties, the performances of two fibers and resin composites are compared and analyzed. It is of great significance to understand the influence of the microscopic properties of carbon fiber on the macroscopic mechanical properties of composites.

2. Materials and methods

2.1. Materials

The carbon fiber was Polyacrylonitrile (PAN) based 3 K twill woven carbon fiber (CF) of filament diameter 7 μm coated with the epoxy compatible sizing was purchased from Hindoostan Composites Solutions, Mumbai, India. Polymethyl methacrylate (PMMA) was sourced from Shouguang Fine Chemical Co., Ltd. Tetrahydrophthalic anhydride was the curing agent and sourced from Wenzhou Qingming Chemical Co., Ltd. The carbon fiber was repeatedly washed using deionized (DI) water and subsequently dried at 80°C in a vacuum oven for 24 h.

The carbon fiber was placed in a three-port flask containing acetone solution, which was refluxed at 80°C for 24 h, then washed with ethanol and dried at 60°C for 24 h in a vacuum. After drying, the CF was added to a three-port flask containing concentrated HNO3, refluxed at 100°C for 2 h, then washed fully with deionized water until ph = 7 and then vacuum dried at 80° C for 24 h. The CF was added to KOH solution, sonicated at 70°C for 50 min, then left for 12 h. The fibers were taken out, reacted in an 80°C oven for 1 h, and then washed with deionized water and dried at 80°C for 12 h.

2.2. Composite preparation

CF/PMMA composites were prepared by autoclave method. The volume fraction of carbon

fibers was 60%, and the curing process system was: vacuum at room temperature, vacuum degree 0.005MPa, pressurized to 0.6MPa, heated to 185 ~200°C, heat preservation 6 h (heating rate $\leq$ 1.5°C/min), naturally cooled to below 60°C out of the tank.

2.3. Test methods

The surface morphology of the carbon fibers was observed using an Apollo 300 scanning electron microscope (the Apollo 300 is a highly versatile Field Emission Scanning Electron Microscope design which provides a true depth of field capability with a resounding quality of build that provides excellent performance at low beam energies). The surface morphology of the carbon fiber was analyzed by a Solver P47 atomic force microscope (NT-MDT, Russia), and the surface roughness was analyzed. The scanning range was 5 μm × 5 μm. The surface composition of carbon fibers was analyzed using an ESCALAB 250 X-ray photoelectron spectrometer (Thermo Ltd, USA). The fibers were mounted laterally across a sample bar and attached to the side rails of the bar using strips of adhesive tape. In this way, there was no supporting material beneath the analysis area. Wide scan spectra were recorded for elemental quantification, and high energy resolution spectra for chemical state determination were recorded through the C 1s, O 1s, N 1s, and S 2p photoelectron regions. Curve fitting was carried out on the high energy resolution spectra from the C 1s and O 1s photoelectron regions to resolve overlapping peaks, and the data was corrected for charging effects by reference to the graphitic carbon peak at 284.6 eV binding energy.

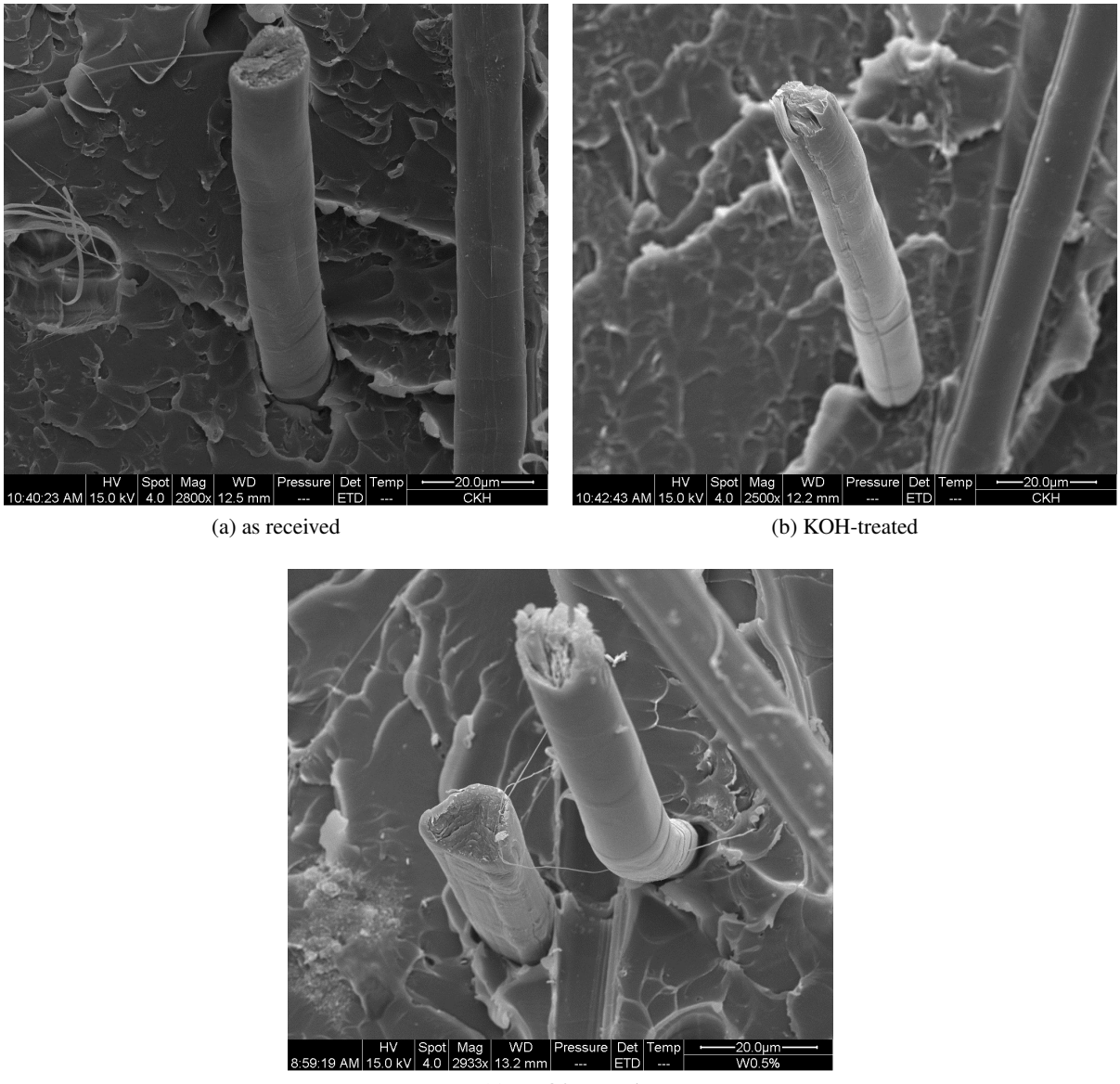
The surface energy of carbon fiber was tested by DCAT21 dynamic contact angle analyzer (DataPhysics Ltd, USA). The depth of the carbon fiber inserted into the immersion medium was 3 mm, the surface infiltration rate was 0.1 mg/s, and the advancement and retreat rate was 8 μm/s. The media are deionized water, ethylene glycol and diiodomethane, respectively. The interfacial shear strength between the two fibers and the resin matrix was tested using a FA-640 micro-debonding tester. The bending test was used to study the interfacial properties between CFs and thermoplastic resins. The measurement was taken at a speed of 0.1 mm/min. Five specimens were tested for each sample. Errors were included based on a 95% confidence limit.

3. Results and discussion

3.1. Surface characteristics of CFs

The interface is a very important microstructure of the composite material. It is a bridge and a bond between the reinforcing fiber and the matrix. Good interface bonding ensures that the load is effectively transmitted to the fiber through the matrix, thereby maximizing the bearing capacity of the fiber. Interfacial interactions are generally thought to consist primarily of physical and chemical interactions. The physical action is mechanical meshing. The key to mechanical meshing is that the surface of the bonded object should have a large number of grooves, cavities, and wrinkles. When the adhesive is flowed, squeezed, impregnated, and filled into these grooves, after curing, it tightly combines with the groove to exhibit a higher bond strength. Chemical action is the chemical bonding of the matrix resin to the reactive functional groups on the surface of the reinforcing fibers, or the combination of intermolecular forces, hydrogen bonding, etc. The results of scanning electron microscopy (SEM) of the surface morphology of carbon fiber are shown in Figure 1. The HNO3-treated carbon fiber has more groove structure than the KOH-treated carbon fiber, and these groove structures are parallel.

The fiber penetrates the fiber in the direction of the axial direction. This structure is a typical fiber morphology characterized by a wet spinning process during the solidification process of wet-spun fiber, due to the double expansion between liquid-liquid two phases. The scattered motion forms a surface structure of scattered grooves under the action of diffusion mass transfer; at the same time, the surface layer of the primary fibers is oriented under the action of stretching to produce a structure similar to the original filament; the stretching is enhanced with the spinning process. The aggre-



(a) as received

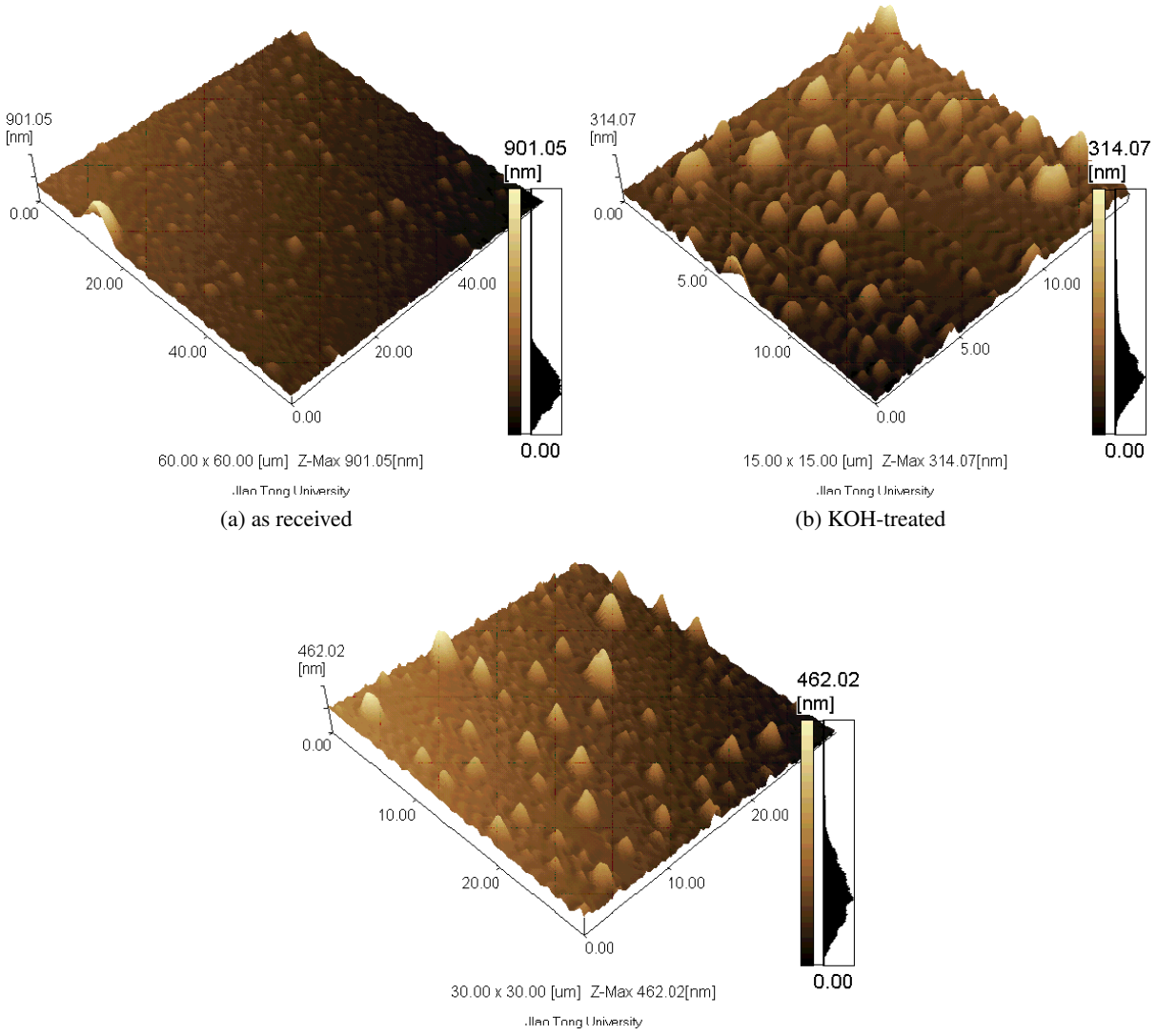
(b) KOH-treated

(c) HNO3-treated

Fig. 1. Surface morphology of carbon fiber

gation state of the polymer changes drastically, the structure of the original filament further develops, and the morphology of the supramolecular structure on the surface of the fiber appears as a groove, and this structure is inherited.

Atomic force microscopy (AFM) further showed the difference in the surface morphology of carbon fiber (Figure 2). Compared with the smooth surface of KOH-treated carbon fiber, the surface of HNO3-treated carbon fiber is rough and dense along the fiber axis. The mechanical meshing of the carbon fiber with the matrix resin helps to improve the interfacial properties of the composite. The average surface roughness (Ra) of HNO3-treated carbon fiber and KOH-treated carbon fiber is about 0.021 μm and 0.012 μm, respectively. The higher surface roughness means that HNO3-treated carbon fiber can produce stronger interface interactions with the resin matrix than KOH-treated carbon fiber.



(a) as received (b) KOH-treated

(c) HNO3-treated

Fig. 2. AFM surface morphology of carbon fiber

3.2. Surface chemical properties of carbon fiber

Semi-quantitative analysis and characterization of the surface composition, surface functional group type, and relative content of carbon fiber were carried out by XPS [24].

Table 1 shows the surface element composition and content of two carbon fibers. The surface elements of carbon fiber are mainly composed of carbon, nitrogen, silicon, oxygen, and other elements. The surface activity of carbon fiber can be expressed by oxygen/carbon ratio (O/C). The higher the O/C ratio, the greater the surface activity, and the stronger the chemical bonding force. The O/C ratio of the surface of the HNO3 treated carbon fiber is 0.26, while that of the KOH-treated carbon fiber is 0.22 (Table 1). Figure 3 is an XPS-fitted peak of the C1s element on the surface of carbon fiber. The functional groups (C-OH or C-OR; C=O) contained in the C1s fitting peak of the carbon fiber and their contents are shown in Table 2. According to the binding energy and atomic relative concentration of each peak in the XPS spectrum, the carbon fiber can be known.

Table 1. Surface compositions of carbon fibers

Sample	Cls		Ols		Nls		Si2p		O/C
	Binding Energy /eV	Atom Fraction /%	Binding Energy /eV	Atom Fraction /%	Binding Energy /eV	Atom Fraction /%	Binding Energy /eV	Atom Fraction /%	
HNO3	285.0	74.96	532.4	20.34	400.1	1.76	102.6	3.14	0.26
KOH	285.0	77.24	532.8	17.67	400.4	1.83	102.4	3.29	0.22

Table 2. XPS Cls curve fitting results of carbon fibers

Sample	C-C or C-H (Peak1)		C-OH or C-OR (Peak2)		C-O (Peak3)		COOH (Peak4)		Activated Carbon /%
	Binding Energy /eV	Atom Fraction /%	Binding Energy /eV	Atom Fraction /%	Binding Energy /eV	Atom Fraction /%	Binding Energy /eV	Atom Fraction /%	
HNO3	285.0	61.61	286.3	21.67	287.2	12.13	288.7	4.18	39.54
KOH	285.0	59.58	286.3	24.28	287.2	16.46			40.32

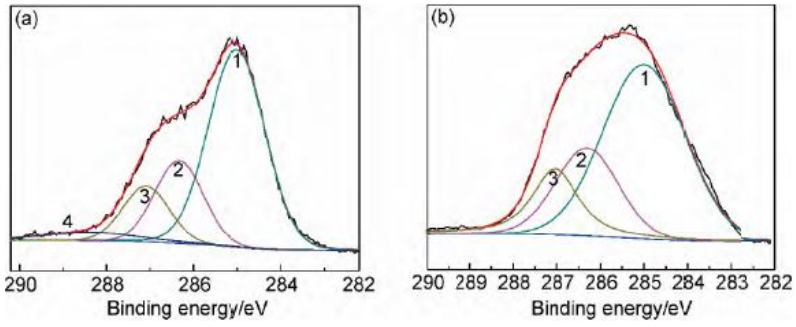


Fig. 3. XPS-fitted peak of the C1s element on the surface of carbon fiber (a: HNO3-treated; b: KOH-treated

3.3. Surface element types and relative content

As can be seen in Table 2, both carbon fiber surfaces contain C-OH or C-OR and C=O reactive functional groups, and the domestic carbon fiber surface also contains carboxyl groups (COOH). The C1s peaks are mainly C-C or C-H bonds. Bind energy key. The active carbon atom fractions of HNO3-treated carbon fiber and KOH-treated carbon fiber are 39.54% and 40.32%, respectively. Because these values are basically the same, the content of activated carbon atoms in each carbon fiber is basically the same. It is estimated that the surface chemical activity of HNO3-treated carbon fiber is similar to that of the KOH-treated carbon fiber. The carbon fiber and the resin form an interaction with the matrix resin through chemical bonding or intermolecular force, thereby improving the interfacial properties of the composite material. Thus, the HNO3-treated carbon fiber and the KOH-treated carbon fiber both have good interface properties with the resin matrix. Although the carbon fiber body may interfere in the activated O ratio, if the interfacial shear strength is greatly affected by the oxygen-containing functional group, as the O/C ratio is larger, the oxygen-containing functional group is greater, and the interfacial shear strength is larger. The adhesion between the fiber and matrix can be improved either by physico-chemical processes, mechanical processes, or both [25]. In physico-chemical process, the adhesion is improved by attaching reactive functional groups on the fiber surface [26].

Table 3. Carbon fibers surface energies and contact angles in different liquids

Sample	Contact angle/			γ/(mJ·m^-2)	γ/(mJ·m^-2)	γ/(mJ·m^-2)
	Deionized water	Ethylene glycol	Diiodomethane			
HNO3	57.3±1.2	42.7±0.9	28.1±1.1	29.5±0.7	16.9±0.8	44.6±1.4
KOH	55.4±1.1	43.9±0.8	27.3±1.0	28.4±0.6	17.6±0.5	46.3±1.6
As received	56.2±1.3	43.2±0.7	27.6±1.2	28.9±0.8	17.1±0.7	45.7±1.5

3.4. Carbon fiber surface energy and infiltration with PMMA resin

The contact angle between the carbon fiber and different liquids was measured several times by a dynamic contact angle analyzer. The contact angle between the two carbon fibers and different liquids and the surface energy of the carbon fibers are shown in Table 3. The data showed that the polar surface energy and total surface energy of HNO3-treated carbon fiber are slightly lower than that of KOH-treated carbon fiber. That is to say, from the surface energy theory, the matching performance of HNO3-treated carbon fiber and resin is slightly lower than that of KOH-treated carbon fiber.

The contact angle of HNO3-treated carbon fiber and PMMA resin is 57.3°, the contact angle of KOH-treated carbon fiber and PMMA resin is 55.4°, and the contact angle of KOH-treated carbon fiber and PMMA is slightly smaller than that of the HNO3-treated carbon fiber., This indicates that the wettability of KOH-treated fiber and PMMA resin and the wettability of HNO3-treated carbon fiber and PMMA resin are basically the same, and the PMMA resin and both fibers have good compatibility, which indirectly indicates that the two carbon fibers have good interface matching with the PMMA resin. The increased surface roughness and functional group resulted in increased total surface area and surface free energy and hence improved wettability of the fiber. Generally, the surface of the carbon fiber is highly hydrophobic due to high temperature carbonization and graphitization during manufacturing [27]. Hence, surface modification is needed to decrease the hydrophobic nature of the fiber.

3.5. Interface properties of composite materials

As can be seen from the above, the difference in the wettability of the two carbon fibers is small, but the surface groove of the HNO3-treated carbon fiber is more than that of the KOH-treated carbon fiber, which improves the mechanical meshing action between the HNO3-treated carbon fiber and the matrix resin. Therefore, the interface performance of the domestic carbon fiber composite material is excellent. Therefore, the interfacial shear strength (IFSS) of the composite can also be determined by a micro-debonding test to quantitatively characterize the composite's interfacial properties. The interfacial shear strength is obtained by averaging multiple measurements.

During the bending test, the lower surface of the sample showed tensile damage, the fiber was debonded from the resin, and the fiber was pulled out. The delamination is also serious, indicating that the interface bonding performance of carbon fiber and PI is poor. The IFSS measurements of the HNO3-treated/PMMA and KOH-treated/PMMA are 67.56MPa and 61.64MPa respectively. The IFSS of the HNO3 treated/PMMA is 15% higher than that of the KOH treated/PMMA, which further confirms the test results of carbon fiber SEM, AFM and XPS, indicating that the carbon fiber is KOH-treated. It forms a better interface bond with the resin. It shows better bonding performance between HNO3-treated carbon fiber and PMMA resin.

3.6. Interlaminar mechanical properties of composites

The interlaminar mechanical properties of the composites are shown in Figure 4. The interlami-

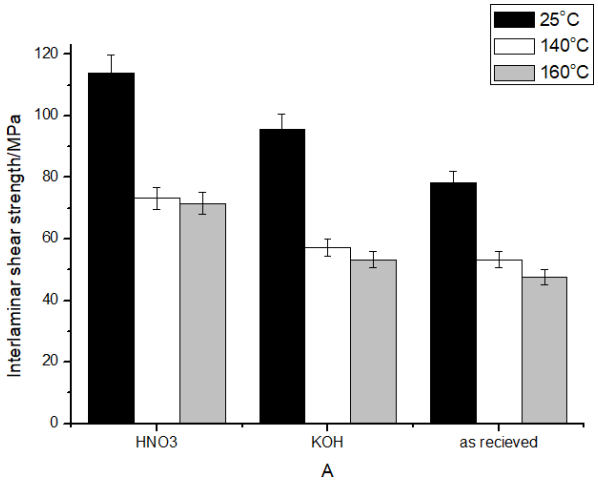


Fig. 4. Interlaminar shear strength of composites

nar shear strength of the HNO3-treated CF/PMMA composites is higher than that of the KOH-treated CF/PMMA composites under various temperatures, and the HNO3-treated composite is dry at room temperature. At 25°C, the interlaminar shear strength (114 MPa) of HNO3-treated CF/PMMA composites is 19% higher than the interlaminar shear strength (95.8MPa) of KOH-treated CF/PMMA composites. The interlaminar shear strength of HNO3-treated CF/PMMA composites is higher than that of KOH-treated CF/PMMA composites with increasing temperature. Polar functional groups and contacts in HNO3-treated carbon fiber and KOH-treated carbon fiber. During the composite molding process, some of the carbon fibers will fall off into the surrounding resin, forming an interface structure different from the fiber and the matrix. For this case, the line scanning functions of force modulation. Under the condition that the angle is basically equivalent, the surface groove of the HNO3-treated carbon fiber surface enhances the mechanical meshing action with the resin matrix and improves the interface performance of the composite material.

These findings indicate that the interfacial properties between the CF and matrix depend upon the treatment parameters, such as time duration to modify the surface of the fiber. Improving the wettability of the CF enhances the mechanical properties of the surface-treated CF composite for the following reasons. Firstly, the unevenness on the CF surface increases, which improves the mechanical interlocking between the CF and the matrix. Secondly, oxygen-containing reactive functional groups functionalize the basal sites, hence enhancing the CF/matrix adhesion. The surface treatment technique delays the crack initiation process at the fiber/matrix interface and favors improved adhesion. Therefore, optimizing surface treatment is essential to improve mechanical performance [28–31]. The surface modification of CF by chemical treatment is a wet technique. The chemical treatment activates the inert surface of the CF by inducing polarity on its surface. Secondly, it corrodes or introduces perforations on the surface of the CF for better mechanical interlocking of the CF with the epoxy resin.

4. Conclusions

1. The HNO3-treated carbon fiber has a large groove structure on the surface and a high surface roughness, which helps to form a better physical meshing action with the resin matrix. Good interface bonding can ensure that the load is effectively transmitted to the fiber through the matrix, thereby maximizing the bearing capacity of the fiber.
2. Although the oxygen-containing functional groups of the two carbon fibers are equivalent, the surface oxygen carbon of the HNO3-treated carbon fiber is relatively high, which is favorable for forming a better chemical bond between the carbon fiber the matrix resin. The O/C ratio of the surface of the HNO3-treated carbon fiber is higher than that of the KOH-treated carbon fiber.
3. The surface energy of HNO3-treated carbon fiber is slightly lower than that of KOH-treated carbon fiber, but the interfacial performance results of composite materials show that the interfacial shear strength of HNO3-treated CF/PMMA composites is about 14% higher than that of KOH-treated CF/PMMA composites. The interlaminar

shear strength of HNO3-treated CF/PMMA composites is 114 MPa, 19% higher than the interlaminar shear strength of KOH-treated CF/PMMA composites (95.8 MPa). The interlaminar shear strength of HNO3-treated CF/PMMA composites is higher than that of KOH treated CF/PMMA composites with increasing temperature. HNO3-treated CF/PMMA composites can be widely utilized in structural applications, due to their high specific strength to conventional metallic materials.

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