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Influence of surface modification of carbon fiber based on magnetron sputtering technology on mechanical properties of carbon fiber composites

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Abstract

To improve the interfacial performance of carbon fiber (CF) and epoxy resin, the surface of CF was modified using magnetron sputtering technology, and a CF epoxy resin (CFER) composite was prepared using injection molding technology. The influence of magnetron sputtering technology on the surface properties of CF was investigated by scanning electron microscopy (SEM), atomic force microscopy (AFM), and dynamic contact angle analysis (DCAA). The influence of the surface modification of the CFs by magnetron sputtering on the mechanical and interfacial properties of CF composites was analyzed by testing the tensile and bending properties of the CFER composites. The results indicated that the surface morphology of CF can be modified by magnetron sputtering, and a nano sized carbon film was deposited on the surface of the CFs. The morphology of the carbon film on the surface of the CFs was different from that on the silicon pellet. The surface roughness of the CF increased after it was modified by magnetron sputtering. The surface wettability of the CFs may be improved by increasing the surface free energy of the fiber owing to the deposition of the carbon film. Tests of the tensile and bending properties of the CFER composites showed that the surface modification of CFs by magnetron sputtering can effectively improve the mechanical properties of the CFER composites, which not only improves the tensile strength and bending strength, but also increases the tensile modulus and bending modulus. The SEM images showed that the interfacial adhesion between the modified CF and the epoxy resin was significantly improved. The stress–strain curves showed that the failure mode of the CFER composite modified by magnetron sputtering CF surface changed, and a stress yield phenomenon was observed.

1. Introduction

Owing to the low density, high axial specific strength, high specific modulus, and high-temperature resistance of carbon fiber (CF), it is considered an important composite reinforcement material. CF-reinforced composites are widely applied in aerospace, wind turbine blades, automobile manufacturing, sports and other leisure fields, as well as for many military applications [1–3]. However, untreated CF has a smooth surface and a low specific surface area and surface chemical polarity. CF hardly forms a good interface with resin matrix and this affects the mechanical properties and the excellent performance of CF-reinforced composites [4, 5]. To improve the interfacial properties of CF and resin matrix, the surface modification of CFs was conducted by physical or chemical methods.

Interfacial modification is hot topic in the composite materials research field, and researchers have enhanced the properties of composite materials. Using various reinforcement methods to improve the properties of fiber/resin matrix interface. At present, the surface modification methods are divided into three types: dry modification, wet chemical modification, and multi-scale modification [5]. The interfacial bonding properties...
of the fiber and resin matrix can be improved by increasing the specific surface area of the fiber, introducing reactive functional groups, improving the wettability of the resin matrix with respect to the fiber, and forming an interface transition zone between the fiber and the resin matrix [7–11].

Among them, modifying a fiber reinforced composite by introducing a nanoscale filling phase based on a traditional fiber reinforced composites material [12–14], including the use of carbon nanotubes (CNTs) and graphene oxide, has become an important research method for improving the properties of a composite material [15–17]. A nanoscale filler phase can be introduced in two ways: by modifying the matrix and through fiber surface modification. For example, graphene modified epoxy resin can improve the mechanical properties of f-GFs/epoxy laminated composites [18]. To compensate for the performance defects of CFs and to improve the interfacial bonding properties between the CFs and the resin matrix, researchers have increasingly coated the surface of CFs with CNTs or graphene oxide by chemical vapor deposition, chemical grafting, and electrophoretic deposition to form new multi-scale interfacial structures [19–21]. These modification methods can improve the interfacial bonding strength of CFs and resin matrices. However, these modification methods have some disadvantages including damage to the microstructure. In addition, the process is complicated, its operation is difficult, it causes environmental pollution, and requires high processing cost.

Magnetron sputtering is a physical vapor deposition method. It ionizes inert gases using direct current or high-frequency electric fields, sputtering out the target atoms or molecules due to the bombardment of high-speed electrons and ions, and then depositing them on the substrate to form thin films. Compared to other film-forming techniques, magnetron sputtering technology has the advantages of fast sputtering speed, uniform film formation, excellent process controllability, and good adhesion, while doing little damage to the substrate [22]. Presently, magnetron sputtering technology has been used to prepare carbon particle films. Diamond-like carbon (DLC) films treated by magnetron sputtering have excellent wear resistance, high-temperature oxidation resistance, and improved optical and electrical properties of the material surface [23, 24]. It requires the early unbalanced magnetron sputtering technology to explore and understand the wear resistance of DLC film materials as well as the influence of magnetron sputtering technology on the morphology and structure of the carbon film [25–28]. However, few researchers have applied magnetron sputtering technology to modify the interface of CF composites. In this study, we focused on the influence of magnetron sputtering technology on the interface of CF-reinforced composites. The surface roughness and surface morphologies of the CFs and carbon film were examined using scanning electron microscopy (SEM) and atomic force microscopy (AFM). The surface free energy and contact angle of the CFs and carbon films were characterized using dynamic contact angle analysis (DCAA).

Epoxy is an excellent thermosetting resin. Due to its excellent physical, electrical insulation, chemical corrosion resistance, heat resistance and adhesion properties, it has become an irreplaceable matrix material in the field of fiber reinforced composites. To date, many researchers have modified epoxy resin to improve its properties [29–31]. Therefore, in this paper, carbon fiber epoxy resin matrix composites were prepared with epoxy resin as the matrix. The effects of surface modifying the CFs by magnetron sputtering technology on the mechanical and interfacial properties of the CF composites were analyzed by examining the mechanical properties of the CF composites before and after the modification process.

2. Experiment

2.1. Materials

CF (T700, 12K and diameter approximately 7 μm) was purchased from Toray Industries, Japan. The carbon target (purity 99.9%, Ø60 mm, and thickness of 2 mm) was purchased from Hefei Co., Ltd, China. Epoxy resin (JL-235) and epoxy curing agent (JH-242) were provided by Changshu Jiafa Chemical Co., Ltd of China.

2.2. Deposition of the film

Owing to the small diameter of the CF, it is difficult to carry out in situ characterization of the carbon film on the surface of the CF. Therefore, to more objectively determine the morphology and performance of the carbon film, carbon particle film was also deposited on silicon pellets using the same magnetron sputtering process in addition to carbon film deposition on the CF surface. First, the CF tow was refluxed in acetone at 80 °C for 72 h, after which it was repeatedly washed with deionized water. The resulting CF tow was dried under vacuum at 80 °C for 3 h to remove the sizing agent. The silicon pellet was cleaned in deionized water, acetone, and absolute ethanol for 20 min in an ultrasonic cleaner to remove impurities and oil contamination adsorbed on its surfaces during production and transportation. To prevent the volatilization of acetone, the open end of the beaker containing the acetone was wrapped with a fresh-keeping film which was cleaned several times with distilled water until no acetone was perceived, the silicon pellet was then removed and dried in a vacuum drying chamber at 100 °C. Next, carbon particles were deposited on the CFs and silicon pellet using a multifunctional
high-vacuum magnetron sputtering equipment (JGP-450A, Shenyang Technology Development Co., Ltd of China). The CFs and silicon treated using magnetron sputtering are denoted as CF-CF (carbon film on CF) and Si-CF (carbon film on silicon pellet), respectively. The machining process is shown in figure 1. The carbon target was installed on the cathode plate, and CF and silicon pellets were used as substrates. The CF bundles were cut to smaller sizes (8 cm), and they were flattened and fixed on a cardboard. The substrates were placed under the substrate bracket. The substrates were treated according to the determined magnetron sputtering process, and the sputtering conditions were as follows: the sputtering power was 450 W, sputtering time was 30 min, sputtering pressure was 1.0 Pa, vacuum pressure of the back bottom was $2 \times 10^{-3}$ Pa, flow rate of the sputtering gas (argon, 99.99% purity) was 80 ml min$^{-1}$, substrate bracket rotation speed was 30 r min$^{-1}$, and target-to-substrate distance was 40 mm.

2.3. Preparation of carbon fiber-reinforced composites

The CF epoxy resin (CFER) composite was prepared according to the ISO527-3 standard [32]. The self-made mold for the polytetrafluoroethylene film plate was 1.0 mm thick. The CFs deposited using magnetron sputtering were placed between two molds, being at the middle area in the thickness direction. Injection molding technology was used to prepare the CF-reinforced epoxy resin composites with epoxy resin as the matrix. The fiber content was 5% of the mass content of the composite, and ratio of the epoxy resin to that of the curing agent was 100:27, which was cured at room temperature for 48 h. The composite sample and size are shown in figure 2.

2.4. Characterization of the carbon fibers and carbon films on the silicon pellets

2.4.1. Scanning electron microscopy

The surface morphologies of the CFs and carbon films on the silicon pellets were characterized using a scanning electron microscope at 5 KV (SEM, SU1510, Hitachi Corporation, Japan) with gold paint. To investigate the effect of the surface modification of the CFs by magnetron sputtering on the interfacial properties of the CF composites, the surface morphology of the tensile fracture surface and the adhesion between the fiber and the resin in the composites were observed. An electronic balance with an accuracy of 0.1 mg was used to measure the change in weight of the CF after magnetron sputtering.
2.4.2. Atomic force microscopy
The surface roughness and surface morphologies of the CFs and carbon films on the silicon pellets were characterized by atomic force microscopy (AFM, Agilent 5500, Agilent Corporation, USA). All AFM images of the CF and carbon films using the tapping mode were obtained at a scan area of 3 μm × 3 μm. The surface roughness of the CFs and carbon films were expressed by Ra and Rq, where Ra is the root mean square roughness and Rq is the arithmetic mean roughness. At least 20 valid dates were applied for each specimen.

2.4.3. Surface wettability
The dynamic contact angle tests were performed using a dynamic contact angle meter (DSA100, Kruss Corporation, Germany). Deionized water (γd = 21.8 m Nm⁻¹, γp = 51.0 m Nm⁻¹, and γ = 72.8 m Nm⁻¹) and glycol (γd = 29.3 m Nm⁻¹, γp = 19 m Nm⁻¹, and γ = 48.3 m Nm⁻¹) were used as test liquids and their surface free energy as well as their dispersive and polar components can be derived using the following equations [33]:

\[
\gamma(1 + \cos \theta) = \frac{4\gamma_d \cdot \gamma}{\gamma_d + \gamma} + \frac{4\gamma_p \cdot \gamma}{\gamma_d + \gamma} \tag{1}
\]

\[
\gamma_{Total} = \gamma_d + \gamma_p \tag{2}
\]

where θ is the dynamic contact angle, γd, γp, and γ are the surface tension of the testing liquid, its dispersive and polar components; each measurement was performed at least ten times.

2.4.4. Mechanical performance test
The tensile properties of the CFER composites were tested following ASTM D3039 on a CSS-881 universal testing machine (Changchun Research Institute for Mechanical Science Co., Ltd) [34]. The bending properties of the CFER composites were tested following ASTM D790 on a CSS-881 universal testing machine [35]; the loading speed was 2 mm min⁻¹. The tests were carried out at 20 °C and relative humidity of 50%.

3. Results and discussion

3.1. Morphology study and surface roughness
The surface morphologies of the CFs and Si-CF are shown in figure 3. Initially, it was observed that the untreated CF had a smooth surface (figure 3(a)); however, after magnetron sputtering treatment, the surface of CF was covered with films (figure 3(b)), and the accumulation of particles on the surface of the CF-CF showed a typical columnar structure (figure 3(c)). The carbon particles deposited on the silicon pellet had an evident membrane structure, and cracks were observed on the surface of the carbon film which were not observed on the surface of the CF-cf. However, the cross-sectional image of Si-CF is similar to that of the CF-CF which also showed a typical columnar structure. This may be due to the planar nature of the
silicon pellet, and carbon particles can be completely collected by the silicon pellet during magnetron sputtering. However, the single CF is fine and the surface of the fiber is curved. At the same time, the CF sample exists in the form of bundles, and there are some arranged pores between the fibers. During the sputtering process, the carbon particles penetrated through the pore between the fibers, and these particles were deflected and transferred owing to the curved surface of the fibers, which makes it impossible to observe a similar morphology for the deposited carbon film on the surface of a single CF and the silicon pellet.

To better understand the surfaces of the untreated and modified CF, AFM was conducted. A comparison of AFM images of the untreated CF, the treated CF, and the Si-CF is shown in figure 4. It can be seen that the surface of the CF appears to be neat and smooth. After magnetron sputtering, large areas of protuberances were observed on the surface of the CF and the silicon pellet. The protuberances of the modified CF are small and delicate, while the protuberances on the silicon pellets are relatively larger.

The surface roughness of the CF and the Si-CF was analyzed by AFM along with the software of the instrument. The surface roughness Ra and Rq of the CF and Si-CF are shown in table 1. In the scanning range of 3 μm × 3 μm, the surface roughness values of unmodified CF Ra and Rq were 131 nm and 157 nm, respectively, which is consistent with the values reported by Deng et al [33]. The surface roughness values of Si-CF Ra and Rq were 2.78 nm and 3.56 nm, respectively, and the surface roughness values of CF modified by magnetron sputtering Ra and Rq were 1.72 nm and 2.26 nm, respectively. The surface roughness of the unmodified CF was significantly higher than that of the modified CF and the Si-cf. This may be due to the fineness of the CF and the change in the curved surface structure of the fiber. Therefore, in the test area, the surface roughness of the CF and carbon film was calculated within an area of 0.1 μm × 0.1 μm and 0.2 μm × 0.2 μm, respectively; that is, the surface roughness of CF was characterized assuming that the surface of CF is approximately plane. The results showed that the surface roughness values of the unmodified CF Ra and Rq were only 0.46 nm and 0.583 nm in the 0.1 μm × 0.1 μm region, respectively. The surface roughness values of the modified CF Ra and Rq were 0.963 nm and 1.28 nm, respectively. The results showed that the surface roughness of the CF increased significantly after modification by magnetron sputtering, which enhances the meshing property between the CF and the resin. The surface roughness of Si-CF treated by the same magnetron sputtering was larger than that of CF-CF, and the surface roughness of Si-CF had little influence on the change in the area used for the calculation. The surface roughness of the CFs increases with the increase in the area used for the calculation. This shows once again that the surface morphology of the CF affects the surface roughness of the CF. In the process of magnetron sputtering, the carbon particles were transferred between the CF bundles, and the carbon particles deposited on the surface of a single CF were less than those deposited on the silicon pellets.

The SEM and AFM results showed that the surface morphology of CF became more complex after the surface was modified by magnetron sputtering. The carbon particles that were deposited on the surface of the CF formed a nano-sized film structure, which increased the surface roughness of the CF, this enhanced mechanical interlocking between the fiber and the resin.
3.2. Surface wettability

The effects of magnetron sputtering modification on the surface wettability of CF and Si-CF were characterized using DCAA. The contact angle, the dispersive component of surface free energy, the polar component of surface free energy, and the total surface free energy of the CF, as well as that of Si-CF, are shown in Table 2. The surface energy was obtained by the addition of polar and nonpolar components. The polar component is related to the induction of matter, while the nonpolar component is related to the van der Waals force and the diffusion between materials. After modification by magnetron sputtering, the contact angles of CF with distilled water and glycol decreased from 78.98° and 69.23° to 65.67° and 61.34°, respectively. The surface free energy of CF increased by 22.16%, from 28.20 mJ m\(^{-2}\) to 34.45 mJ m\(^{-2}\). Among the components, the dispersible component increased significantly, from 3.24 mJ m\(^{-2}\) to 9.26 mJ m\(^{-2}\), while the polar component increased only by 0.23 mJ m\(^{-2}\). The contact angle of Si-CF with distilled water and glycol was the lowest, which was 4.87 mJ m\(^{-2}\) and 3.2 mJ m\(^{-2}\) less than that of the modified CF, and the surface free energy increased by 2.27 mJ m\(^{-2}\); the increase in the surface polar free energy was the main reason for this observation. The results showed that surface modification by magnetron sputtering increased the surface free energy of the CF, it also improved the surface wettability of the CF and the distribution of the resin matrix on the surface of the CF during the composite formation process. The reason for these observations is because after the magnetron sputtering treatment, the structure of the surface of the CF was reduced to nano-size, the surface area and the surface roughness of the CF increased, and this improved the surface wettability of the CF.

3.3. Mechanical properties of the CFER composites

The effect of magnetron sputtering modification on the quality of CF was analyzed by testing the quality of the CF before and after magnetron sputtering. The weight of a unit length of the unmodified bundle CF was 8.375 mg cm\(^{-1}\), and the weight of a unit length of the bundle CF after magnetron sputtering treatment was still 8.375 mg cm\(^{-1}\). This shows that the weight of a unit length of the CF was not affected by the magnetron sputtering modification, and the mass ratio of the CF to the resin in the composite was not affected.

Figure 5 shows the tensile stress–strain curves and bending stress–deflection curves of the CFER composite. It was found that the tensile modulus and bending modulus of the CFER composite were improved after modification by magnetron sputtering. The tensile fracture work of the CFER composite also increased significantly, from 33.77 J m\(^{-2}\) to 108.94 J m\(^{-2}\), after the surface modification of the CFs. Furthermore, the CFER composites without fiber surface modification exhibited no stress yield during the loading process, and brittle fracture occurred at the load limit. However, the stress yield stage of the CFER composite is observed after modification by magnetron sputtering during the loading process, and fracture delay occurs at the load limit. According to the bending stress deflection curve in Figure 5(b), the bending deflection of the CFER composite
was increased by approximately 1.18 times after the surface modification by magnetron sputtering. The results indicate that the surface modification of CFs by magnetron sputtering enhanced the tensile properties and toughness of the CFER composite, and altered interfacial structure, thereby changing the mode of load transfer in the composite during the loading process, and effectively delaying the onset of fracture of the composite during the failure process.

The tensile strength and bending strength of the CFER composite are shown in figure 6. The mechanical properties of the CFER composites modified by magnetron sputtering were significantly improved, and the tensile strength and bending strength of CFER composites were increased by 47.6% and 78.36%, respectively. The improvement in the tensile strength of the CFER with carbon nanotubes is similar to that reported by Han et al who showed that this material also undergoes pseudo-plastic fracturing during the tensile process [36].
The SEM images of the tensile section and the fracture surface of the CFER composites are shown in figure 7. It can be seen from figure 7 that there is a notable gap between the CF and the resin at the tensile section (figure 7(a)) of the unmodified CFER composite. It can also be observed from the fracture surface (figure 7(c)) that the surface of the fiber is smooth with little resin remaining on the CF. The adhesive properties of the CF and the resin at the tensile section (figure 7(b)) of the modified CFER composite material was significantly improved, the gap between the CF and the resin was significantly reduced, and the residual resin content on the CF in the fracture surface (figure 7(d)) was significantly increased. The results showed that the adhesion between the CF and the resin improved after the surface was modified by magnetron sputtering, which enhances the mechanical properties of the CFER composite materials.

4. Conclusions

Surface treatment of CFs by magnetron sputtering can change the surface morphology of CFs, it can deposit a nanosized carbon film on the surface of the CFs, which increases the specific surface area of the CFs and enhances the contact area and adhesion between the resin and the CFs. It increased the surface free energy of the CFs by 22.3%, which improves the distribution of the resin on the surface of the CFs and the wettability of the CFs surface. The increase in the surface roughness of the CFs after the modification through magnetron sputtering, enhances the mechanical interlocking performance between the CFs and the resin. By employing the CFs modified by magnetron sputtering, the tensile strength and bending strength of the CFER composites increased by 47.69% and 78.36%, respectively. The surface modification of the CFs not only enhances the tensile and bending properties of the CFER composites, but also improves their toughness. After the surface modification of CFs, the tensile fracture work of the CFER composites increased to 75.17 J m$^{-2}$, and the bending deflection increased by approximately 1.18 times. Further the interface structure of the CFER composites was altered, and a failure delay phenomenon of the composites was observed during the loading process.

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