



Research paper

Effects of different silanization followed via the sol-gel growing of silica nanoparticles onto carbon fiber on interfacial strength of silicone resin composites

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ABSTRACT

Effects of different silanization followed via the sol-gel growing of silica nanoparticles (SiO_2) onto carbon fibers (CFs) on interfacial strength of silicone resin composites were investigated systematically. CFs were grafted with 3-aminopropyltriethoxysilane (APS) at different conditions to introduce amino groups (CF-NH₂) or ethoxy groups (CF-Siloxane) for improving interfacial interaction between CFs and SiO_2 . Experimental results indicated that SiO_2 was grown onto different functionalized CFs successfully, which enhanced the wettability owing to the increases of fiber polarity and surface roughness. Interlaminar shear strength (ILSS) and interfacial shear strength (IFSS) of SiO_2 modified CFs composites enhanced significantly, especially for CF-Siloxane grafted with SiO_2 (CF-Siloxane- SiO_2), increasing 46.79% in ILSS and 39.61% in IFSS. Additionally, the introduced Si—O—C bonds increased hydrothermal aging resistance of the resulting composites greatly.

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1. Introduction

CFs are considered promising reinforcements for polymer composites because of their inherent advantages such as strong strength, light weight, good corrosion resistance and outstanding thermal stability [1–3]. CFs reinforced polymer composites show a wide range of structural applications in the fields of the automotive, marine and aerospace industries [4,5]. It is well accepted that the properties of composites depend highly on the quality of fiber-matrix interface, which controls the stress transfer efficiency between CFs and matrix resin [6,7]. However, the development and application of CFs reinforced polymer composites are seriously limited owing to non-polar and smooth graphitic surface of CFs, which cannot provide desired interfacial interactions with matrix resin [8]. Hence, many modification techniques are reported to change smooth and chemical inert surface to the active and rough one for enhancing interfacial strength of composites [8–12].

Recently, the introduction of different active nanoparticles (e.g., graphene oxide, carbon nanotubes, and polyhedral oligomeric silsesquioxane) onto the fiber surface was demonstrated for improving interfacial properties of composites [13–15]. A hybrid reinforcement has been reported by grafting octaglycidyl dimethylsilyl

polyhedral oligomeric silsesquioxane (POSS) onto CFs by bridging poly(amido amine), leading to significant enhancement of interfacial properties [16]. Size-controllable graphene oxide sheets (GO) via poly(oxypropylene) diamines as the bridging agent was introduced into the interface for high-performance composite materials [17]. All the hierarchical reinforcements with many active groups and special structures could enhance interfacial strength and properties of composites. However, chemical grafting has a fatal limitation that vigorous acid corrosion and tedious modification process damage inevitably fiber tensile strength [18]. The physical routes usually refer to chemical vapor deposition, the sizing process and the sol-gel method, which in suit introduce the reinforcing agents onto the fiber surface. It can heal fiber surface flaws and protect fiber surface, which are suit for practical applications and large-scale industrial production.

Noteworthy, SiO_2 has become a research hot spot because of its unique spherical structure and superior physicochemical properties, which is widely used to reinforce polymer composites [19–21]. Enormous silanol groups of SiO_2 surface not only show a good wettability and interfacial interactions with matrix resin, but also provide the reactive sites for further grafting. Moreover, Compared to other nanoparticles as mentioned above, SiO_2 is easily obtained via the sol-gel method and has distinct price advantage, which helps to adopt at large-scale for practical applications [22]. Considerable efforts have been made to introduce SiO_2 coated onto the surface of CFs or incorporated into the polymer system

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resulting in the significant increases of interfacial adhesion and properties of composites [23–25]. In fact, the interfacial structure between SiO₂ and CFs gains a key role for its applications. If the interfacial interaction between SiO₂ and CFs is relatively poor, SiO₂ may easily form serious aggregation onto the fiber surface, which leads to stress concentrations and thus affects the degree of interfacial improvement. However, the non-polar, and smooth graphitic surface of untreated fiber makes it difficult to provide desired interactions between SiO₂ and CFs. Thus, sufficient active functional groups on the fiber surface play important roles in enhancing interfacial compatibility between SiO₂ and CFs and controlling the surface morphologies of SiO₂ grafted CFs. That is to say, it is highly important to study the interfacial interaction between SiO₂ and CFs and further reveal the interfacial mechanisms that enhance mechanical properties of the hybrids composites.

In this study, a novel and effective method was reported to enhance interfacial compatibility between SiO₂ and CFs for the introduction of SiO₂ onto different CFs surface, and studied effects of fiber surface treatment on interfacial properties of composites. CF-NH₂ and CF-Siloxane were well prepared, and then in suit grown of SiO₂ onto different CFs (Untreated fiber, CF-NH₂ and CF-Siloxane) by the hydrolysis of tetraethoxysilane (TEOS). Surface morphologies and chemical composition were examined by scanning electron microscopy (SEM), atomic force microscopy (AFM), fourier transform infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy (XPS). Surface energy and wettability of different CFs were characterized by dynamic contact angle analysis (DCA). The different SiO₂ modified CFs with respect to ILSS, IFSS and hydrothermal aging resistance of CF/methylphenylsilicone resin (MPSR) composites could be discussed in more detail, and composites interfacial reinforcing mechanisms were also studied and compared.

2. Experimental

2.1. Materials

CFs with the diameter of round 7 μm and the tensile strength of about 3500 MPa were purchased by Toray Industries, Inc. MPSR with the molecular weight of 2400 was received by ShangHai Chemicals Co. APS, triethylamine and TEOS were all obtained by Aladdin. All other reagents, like N,N'-Dicyclohexyl carbodiimide (DCC), 4-Dimethylaminopyridine (DMAP), toluene, concentrated nitric acid (HNO₃), tetrahydrofuran (THF), and ammonia were provided from Tianjin Bodi Organic Chemicals Co. Ltd.

2.2. Surface grown of SiO₂ onto CFs

2.2.1. Fiber desizing, oxidation, CF-NH₂ and CF-Siloxane

As-received CFs were firstly added into the mixed solution of supercritical acetone/water at 633 K for 30 min aiming to remove polymer sizing agents for obtaining Untreated CF. Subsequently, Untreated CF was added into a round bottomed flask containing concentrated HNO₃, and then heated and reacted at 353 K for about 4 h to produce massive carboxyl groups (denoted as CF-COOH). After that, a bundle of CF-COOH was bound on a glass frame and then added in the solution of APS (1 mL) and toluene (100 mL), and allowed to react at 333 K for 24 h. After that, the modified fiber (CF-NH₂) was centrifuged and washed with deionized water and toluene each for several times and dried in a vacuum oven before further use. To obtain CF-Siloxane, CF-COOH tied with the glass frame was reacted with the mixture solution of DCC (0.1 g), DMAP (0.01 g), APS (1 mL), and THF (100 mL) at 333 K for 24 h under nitrogen atmosphere. When completed, CF-Siloxane was washed in excess anhydrous THF repeatedly to remove unreacted APS and dried.

2.2.2. Grown of SiO₂ onto different CFs by the hydrolysis of TEOS

The grown of SiO₂ onto different CFs was prepared via the hydrolysis of TEOS [26,27]. Briefly, Untreated CF, CF-NH₂ and CF-Siloxane were separately bound on a glass frame and added into an alcohol-water mixed solution of 82 mL ethanol and 3.4 mL water by stirring separately. Then, 5.6 mL ammonia solution as a catalyst was added and stirred for 10 min. Subsequently, the 0.05 mol/L TEOS was quickly added into the solution and allowed to react for 15 h. After modification, the obtained products were washed repeatedly with deionized water and absolute ethanol each and dried. The obtained products were denoted as CF-SiO₂, CF-NH₂-SiO₂ and CF-Siloxane-SiO₂. Schematic of the growth of SiO₂ on different CFs by TEOS hydrolysis in suit was illustrated in Fig. 1.

2.3. Preparation of CF/MPSR composites

The compression molding method was carried out to prepare unidirectional composites with different CFs and MPSR using the unidirectional fiber prepreg. First, CFs were wrapped around a metal frame with 35 circles, and then placed into the given mold. Subsequently, MPSR was injected into the mold for fully making matrix resin saturate into the fibers, followed by being degassed with a vacuum pump until no bubbles came out of fiber prepreg.

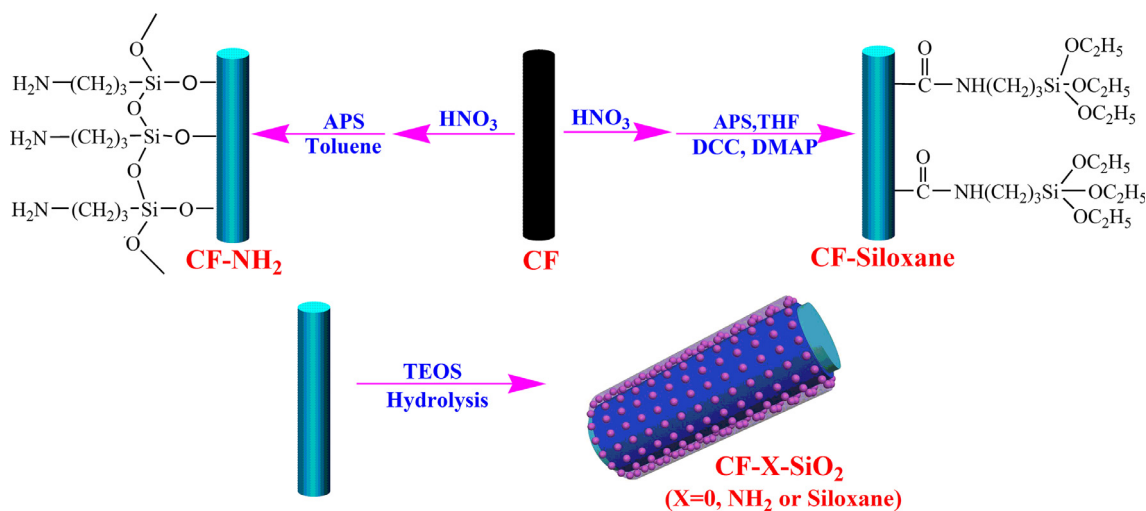


Fig. 1. Schematic of the growth of SiO₂ onto different CFs surface.

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