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Short communication

The effect of a compliant polyimide nanocoating on the tensile properties of a high strength PAN-based carbon fiber

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ABSTRACT

The effect of a compliant polyimide nanocoating on the tensile strength of a polyacrylonitrile-based high tensile strength (T1000GB) carbon fiber was investigated. The pyromellitic dianhydride/4-4′-oxydianiline polyimide nanocoating was deposited by high-temperature vapor deposition polymerization. The thickness of the polyimide coating was about 100 nm. The tensile strength and Weibull modulus of nanocoated and uncoated fiber bundles were evaluated using a polyimide-impregnated bundle-composite. The results clearly demonstrated that the compliant polyimide nanocoating is effective in improving the tensile strength and Weibull modulus of T1000GB carbon fiber.

Carbon fiber has been developed as reinforcement for composite materials due to its high specific strength and modulus. These composites are widely used in aerospace, automotive, and sporting goods industries. A recent trend in the development of carbon fiber has been directed toward improving its tensile strength and ductility. A polyacrylonitrile- (PAN) based carbon fiber with high tensile strength (exceeding 6 GPa) and ductility of over 2% is commercially available. A variety of oxidative and non-oxidative surface modification techniques have been developed to improve the adhesion and bonding between the carbon fiber and matrix [1–3]. Several recent studies have reported that grafting on carbon nanotubes (CNT) [4] and titanium carbide (TiC) coating [5] improve the tensile strength and the Weibull modulus of single carbon fiber. A dense CNT network and TiC coating act to reduce defects in the carbon fiber. However, the TiC coating layer is brittle, and prone to cracking under impact loading. Therefore, a more compliant polymer coating would be better for healing surface defects in the carbon fiber and for improving its ability to absorb impact energy. Similar healing effects of using nanocomposite coatings to improve the tensile strength and its distribution of glass fibers have been reported [6,7].

The carbon fiber has been coated with the polymer using dip coating [8–10], electro-polymerization [11–13], and deposition processes [14,15]. High-temperature vapor deposition polymerization (VDP_H) [16-18] is a promising approach due to its ability to form a relatively uniform and thin layer on three dimensional objects and porous materials. Using the VDP_H approach, a compliant polyimide nanocoating can be deposited directly on each filament within a fiber bundle. In this study, the effect of VDP_H polyimide nanocoating on the tensile properties of a high strength carbon fiber was investigated.

The carbon fiber bundle used in the study was a PAN-based high strength T1000GB carbon fiber supplied by Toray Industries, Inc. The physical properties of the T1000GB fiber bundle are listed in Table 1. The as-received carbon fiber bundle without de-sizing treatment denotes a-CF. A thermoset polyimide, which is synthesized from pyromellitic dianhydride (PMDA) and 4-4′-oxydianiline (ODA), was selected as the compliant coating layer. A VDP_H apparatus (ULVAC Corp., Japan) was used for the nanolayer coating. This VDP_H approach allows the formation of a solventless thin layer on a complex topological substrate—details are described in the literature [16-18]. The monomer sources of PMDA and ODA were heated at 190 °C and 188 °C in separate chambers under vacuum, prior to deposition. The temperature conditions of monomers were decided by the FT-IR spectra of the PMDA/ODA coating on an aluminum sheet, which results mentioned that the PMDA/ODA

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Table 1
Physical properties of T1000GB fiber bundle.

	As-received	Coated
Density[a], ρ/g cm^{-3}	1.80	–
Tow size[a]/yarn	12 k	–
Tex[a], T/g km^{-1}	485	–
Tensile strength, σ_{ft}/GPa	5.31 (0.29)[b]	5.76 (0.25)[c]

() indicate standard deviations.
[a] Producer's data sheet. T1000GB: Catalog for TORAYCA, Toray Industries, Inc. Toray, High performance carbon fiber Torayca in Japanese, 2004.
[b] Data of the a-CF/PI(BTDA/MDA)-matrix bundle-composites.
[c] Data of the a-CF/PI(PMDA/ODA)-coating/PI(BTDA/MDA)-matrix bundle-composites.

coating was formed without excessive monomers and a residual polymetric acid in our preliminary experiment. Thermal imidization of the a-CF/PI(PMDA/ODA)-coating was conducted at 300 °C for 60 min in air, under a vacuum of 10^{-2} Pa.

The precursor solution of aromatic polyimide (SKYBOND® 703, Industrial Summit Technology Corp.) consists of 3,4,3′,4′-benzophenone tetracarboxylic dianhydride (BTDA) and 4,4′-methylendianiline (MDA). Hereafter, PI(PMDA/ODA)-coating and PI(BTDA/MDA)-matrix denote PI_1-coating and PI_2-matrix, respectively. The a-CF/PI_1-coating was impregnated with a liquid polyimide precursor solution, and the a-CF/PI_1-coating/PI_2-matrix cured at 300 °C for 60 min to form a 1 mm diameter rigid bundle-composite.

The morphology of the polyimide nanocoating on carbon fibers was examined using a field-emission scanning electron microscope (S-4800, Hitachi High-Technologies Co.). The infrared spectra of the a-CF, the a-CF/PI_1-coating, and the a-CF/PI_2-matrix were measured in the 1000–2000 cm^{-1} range using a vacuum Fourier transform infrared spectrometer (V80, Bruker Optics Co.). The bundle composite specimens were trimmed to about 65 mm, and emery paper tabs were bonded to each end of the specimen to act as the gripping region during the tensile testing. Tensile tests of the bundle-composite with a 25 mm gauge length were performed using a universal testing machine (Shimadzu co.) with a 5 kN load cell. The test was conducted using a crosshead speed of 5 mm/min at room temperature. A total of one hundred specimens were tested to evaluate the Weibull statistics.

Fig. 1 shows a SEM micrograph of the typical surface and cross-section of the polyimide nanocoating on the T1000GB fiber. The as-received fiber has a comparatively smooth surface and exhibits a particulate morphology in the cross-section, as shown in Fig. 1a and c. The VDP_H PI (PMDA/ODA) nanocoating was uniformly deposited on each filament, as shown in Fig. 1b and d. The coating thickness on a fiber was ~100 nm.

Fig. 2 shows the FT-IR spectra of (a) the as-received bundle (a-CF), (b) the PMDA/ODA polyimide nanocoated bundles (a-CF/PI_1-coating), and (c) the BTDA/MDA polyimide matrix with the uncoated bundle (a-CF/PI_2-matrix). As shown in Fig. 2b and c after thermal imidization, amid peak at 1660 cm^{-1}, which corresponds to vibration of C=O, disappeared; while strong imide peaks appeared at 1720 and 1380 cm^{-1}, which correspond to the C=O and C–N stretch. The imide absorption peaks confirm the formation of a polyimide coating on the carbon fibers. The PMDA/ODA polyimide coating and the BTDA/MDA polyimide matrix can be distinguished by comparing the spectra having different peaks in the 1000–1300 cm^{-1} range.

Typical cross-sectional image of a-CF/PI_1-coating/PI_2-matrix bundle composite is shown in Fig. 3. It clearly shows that the indi-

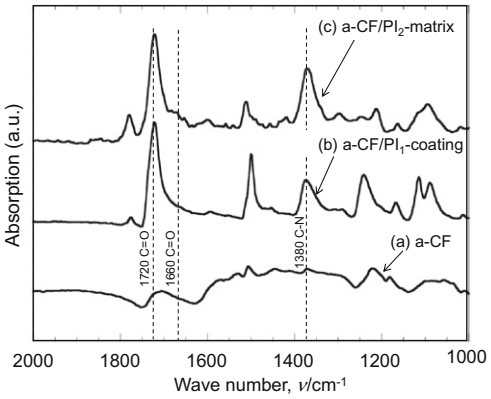

Fig. 2. FT-IR spectra of (a) the as-received bundle (a-CF), (b) the a-CF/PI_1-coating, and (c) the a-CF/PI_2-matrix specimens.

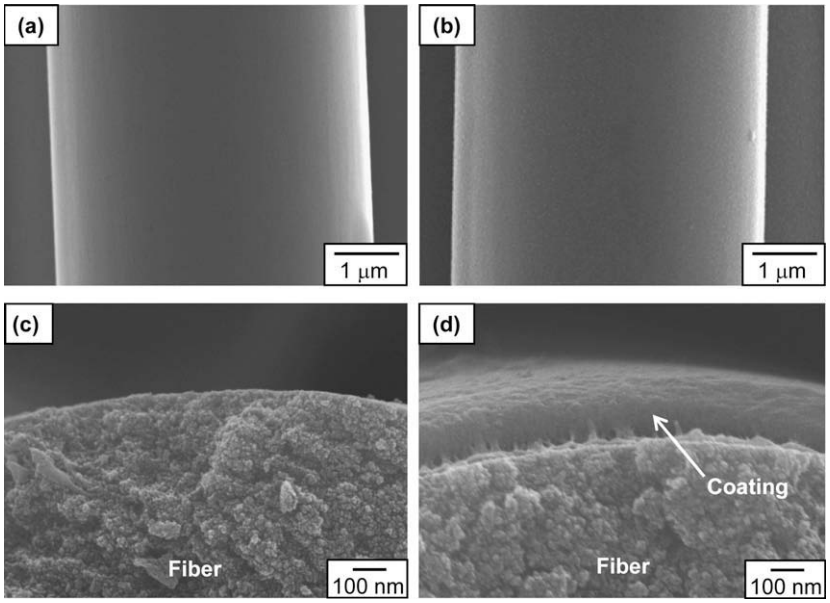

Fig. 1. FE-SEM images of the surface and cross-section of the as-received T1000GB fiber (a-CF: (a) and (c)) and the PMDA/ODA coated on the fiber (a-CF/PI_1-coating: (b) and (d)).