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Mechanical interfacial adhesion of carbon fibers-reinforced polarized-polypropylene matrix composites: effects of silane coupling agents

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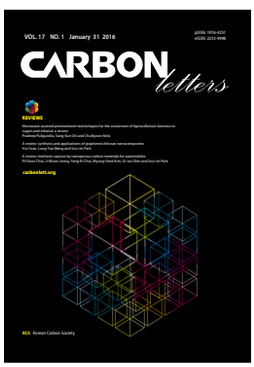

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Thermoplastic composites are used in a variety of applications such as mass transit, automotive parts, and military structures [1-6]. Their appeal compared to conventional materials such as aluminum, steel, and thermoset composites for these applications lies in their high specific strength, corrosion resistance, superior impact resistance, high toughness, and ease of recycling [1-7].

In recent years, carbon fibers (CF) have been widely used as reinforcing materials in high performance composites. CF present several advantages, including high modulus and strength, good stiffness, and creep resistance [8-10]. Despite these advantages, the CF/thermoplastic composites have unsatisfactory mechanical properties because CF have poor interfacial adhesion with most thermoplastic polymers due to their non-polar surface.

The interfacial adhesion between reinforcing fibers and polymer matrices in composites is the controlling factor in obtaining optimum mechanical properties of composites [11-13]. To achieve good interfacial adhesion between CF and thermoplastic matrices, it is necessary to increase the surface polarity. In this light, increasing the surface polarity for van der Waals forces and hydrogen bonding can improve the interfacial adhesion.

In efforts to increase the surface polarity of CF, various surface treatment techniques have been applied, including plasma treatment, anodic oxidation, metal plating, and coupling treatment [14,15]. Among the various treatments, coupling treatment is known to be a very effective method for improving the interfacial adhesion between the fibers and the thermoplastic matrices [16].

The role of coupling agents in improving the interfacial adhesion between polymers and inorganic surfaces has been widely documented [17,18]. However, the effects of different coupling agents on interfacial adhesion between CF and polar-modified thermoplastic matrices have yet to be explicitly identified.

Therefore, the first objective of this study is to evaluate the influence of sizing treatments by silane-based coupling agents on the interfacial adhesion properties between the fibers and the thermoplastic matrix. Another objective is to investigate the effects of the presence of surface functional groups containing silane-oxygen groups on the mechanical properties of carbon fiber-reinforced maleic anhydride-grafted polypropylene.

The CF used in this work were polyacrylonitrile based high strength fibers, T-700SC-13000 (12K unidirectional fabric; Toray, Tokyo, Japan). The average diameter of these CF was around 7 μm. A highly polarized polypropylene sheet prepared by maleic anhydride grafting (Homan Petrochemical Co, Seoul, Korea) was used as a matrix.

The reagents used for the coupling treatments were vinyltriethoxy silane (VTES), 3-amino propyl triethoxy silane (APS), and 3-methacryloxy propyl trimethoxy silane (MPTS; Aldrich, St. Louis, MO, USA), denoted as VTES, APS, and MPTS, respectively. The acid and silane treatments of the CF were carried out by the following procedure. For the acid

treatments, the CFs were immersed in acid solution (3 mol of HNO_3) at room temperature and refluxed for 30 min in order to introduce hydroxyl groups on the CFs.

The silane solution (2 wt%) was prepared by adding the silanes to a mixture of ethanol or methanol (95%), and distilled water (5%). The pH of the solutions was adjusted to 4.0-5.0 by addition of acetic acid. Acetic acid initiates the formation of silanol structures. The solutions were stirred for 1 h to allow for hydrolysis and silanol formation. The acid treated CF were then immersed into each sizing solution for 40 min. In this step, the hydroxyl groups on the CFs can react with the hydrolyzed silanes, resulting in the formation of silanol groups on the CFs. The silane-treated CF were then rinsed with distilled water to eliminate the unreacted silane agents and dried at 80°C for 12 h.

The composites were prepared by a well-known laminate method. Unidirectional carbon fabric laminates were prepared using commercial unidirectional fabric and polarized polypropylene sheets, with 50 vol% of CF. The composite manufacturing process by hot compression molding was carried out in a steel mold with dimensions of 50 × 150 mm. The composite laminates were heated to 150°C at 5°C/min, held for 10 min, and pressed under 200 kg_f/cm^2. They were subsequently heated to 210°C at 5°C /min, pressed under 100 kg_f/cm^2, and held for 10 min.

The surface properties of CF, before and after silane treatments, were characterized by several techniques. Fourier transform infrared spectroscopy (FT-IR) spectra of treated CF were recorded on a FT-IR spectrophotometer (Nicolet-is10; Thermo Fisher Scientific, Waltham, MA, USA). The scan range was from 400 to 4500 cm^{-1}. X-ray photoelectron spectroscopy (XPS) was employed as it is a useful analytical technique to evaluate the surface composition of the fibers [19-21]. An XPS experiment was performed with a VG Scientific ESCA LAB MK-II spectrometer equipped with an Mg-Kα X-ray source. The base pressure in the sample chamber was controlled in a range of 5 × 10^{-8} torr.

The contact angle of the fibers is generally recognized as an important parameter to determine the wetting performance and surface free energy of treated CF [22,23]. Contact angle measurements of treated fibers were performed using a Krüss Processor Tension meter K-100. Two wetting liquids were used for calculating the surface free energy by the modified Washburn equation (1):

$$\frac{m^2}{t} = \frac{c \times \rho^2 \times \gamma_L \times \cos\theta}{\eta} \quad (1)$$

where m is the mass of the wetting liquid; t is the flow time; ρ is the density of measuring liquid; η is the viscosity of wetting liquid; γ_L is the surface free energy of wetting liquid; θ is the contact angle; and c is the packing factor [24]. The wetting liquids used were ethylene glycol (γ_s: 47.70 mN/m, γ_s^d: 31.00 mN/m, γ_s^p: 16.70 mN/m), deionized water (γ_s: 72.80 mN/m, γ_s^d: 21.80 mN/m, γ_s^p: 50.00 mN/m), and diiodomethane (γ_s: 50.80 mN/m, γ_s^d: 50.42 mN/m, γ_s^p: 0.38 mN/m). Contact angles were measured 10 times for the calculation of the surface energetics with a standard deviation of less than 1°.

Interlaminar shear strength (ILSS) was measured by the three-point short beam bending test method to evaluate the interfacial adhesion between the CF and the polypropylene matrix. The three-point bending test was performed with an Instron Model Lloyd LR-5K mechanical tester according to the ASTM D-2344. The span-to-depth ratio and cross-head speed used were 4:1 and 2.0 mm/min, respectively. The ILSS of the composites was calculated by the following equation:

$$\mathrm{ILSS} = \frac{3F}{4bd} \quad (2)$$

where F is the load (N) at the moment of break, b the width (m) of the specimen, and d the thickness (m) of the specimen.

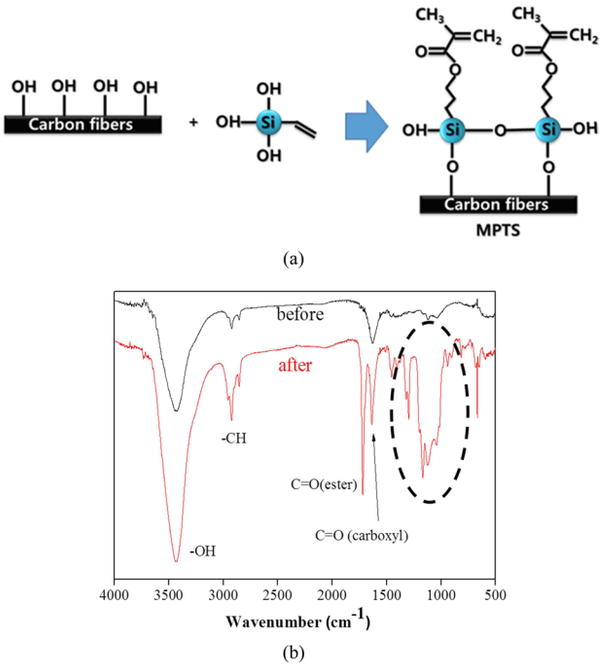

Fig. 1. (a) A schematic diagram of the silane coupling treatment mechanism for methacryloxy propyl trimethoxy silane (MPTS) on acid-treated carbon fibers and (b) Fourier transforms infrared spectroscopy spectra of carbon fibers before (unsized) and after silane coupling treatment (MPTS).

Silane coupling treatments are mainly used for inorganic fillers in composites to improve the adhesion strength between the fillers and the polymer matrix. The silane coupling agent undergoes chemical transformation during hydrolysis and drying. During hydrolysis of the silane, the Si-O-R groups are changed to active Si-OH. Normally, the silanol group of coupling agents reacts with the hydroxyl groups of carbon fiber surfaces and forms a covalent bond, and untreated silanol groups are condensed by a self-reaction with silanol from other silane coupling agents. The silane treatment mechanism is schematically illustrated in Fig. 1a.

The FT-IR transmittance spectra of the CF before and after silane coupling treatments are shown in Fig. 1b. The oxygen functional group (-COOH, C=O) at 1600-1800 cm^{-1}, the hydroxyl group (-OH) at 3450 cm^{-1}, and the hydrocarbon group (-CH) at 2980 cm^{-1} are increased after silane coupling treatments. Meanwhile, organic groups of silane (Si-O-C) at 1380 cm^{-1} and Si-O-Si at 1073 cm^{-1}) increased predominantly as the oxygen functional groups are increased. This demonstrates that the silane coupling treatment efficiently stimulated the oxygen functional

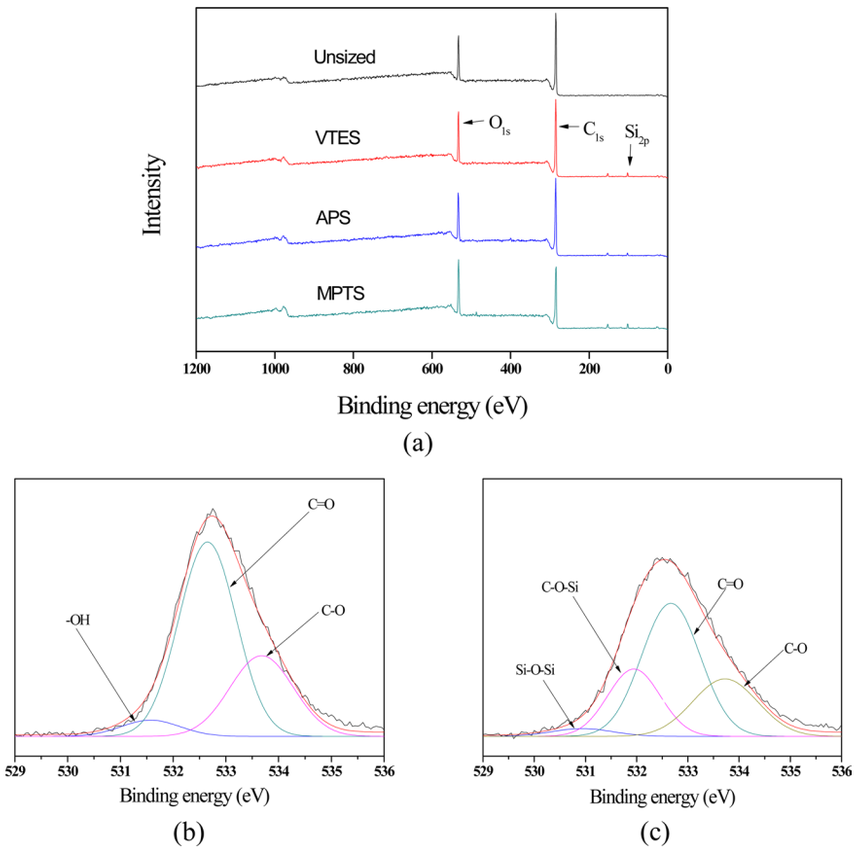

Fig. 2. X-ray photoelectron spectroscopy (XPS) results of carbon fibers (CF) before and after silane coupling treatments; (a) XPS spectra of CF, (b) High resolution of the O_{1S} peak of the unsized CF and (c) MPTS-CF. VTES, vinyltriethoxy silane; APS, amino propyl triethoxy silane; MPTS, methacryloxy propyl trimethoxy silane.

Table 1. Chemical composition of carbon fibers as a function of silane coupling agents

	Element composition (%)					Element ratio		
	O_{1S}	C_{1S}	Si_{2P}	R-O_{1S}[a)]	R-Si_{2P}[b)]	R-O_{1S}/R-Si_{2P}	O_{1S}/C_{1S}	R-O_{1S}/C_{1S}
Unsized[c)]	15.92	83.71	0.37	-	-	-	0.19	-
VTES	17.57	80.40	2.03	1.65	1.66	0.99	0.21	0.020
APS	17.82	79.70	2.48	1.90	2.11	0.90	0.22	0.023
MPTS	22.17	74.39	3.44	6.25	3.07	2.04	0.29	0.084

VTES, vinyltriethoxy silane; APS, amino propyl triethoxy silane; MPTS, methacryloxy propyl trimethoxy silane.
[a)]Real increased content of O_{1S} after silane coupling treatments.
[b)]Real increased content of Si_{2P} after silane coupling treatments.
[c)]Acid-treated carbon fiber before silane coupling treatments.

group formation on the carbon fiber surfaces.

The XPS wide-scan spectra of silane-treated and untreated CF are shown in Fig. 2a, and their chemical information is listed in Table 1. The spectra show distinct oxygen, carbon, and silicon peaks of the samples. It was found that the O_{1S} (BE = 532.8 eV) and Si_{2P} (BE = 102.6 eV) peaks of the silane-treated CF were higher than those of the untreated CF. In contrast, the intensity of C_{1S} (BE = 284.5 eV) was diminished. The enhancement of the O_{1S} and Si_{2P} peaks on the carbon fiber surface is attributed to the effect of the silane coupling treatments on the chemical composition and surface structures, stemming from the introduction of oxygen containing functional groups (C=O, COOH, and C-O) and organic groups of silane (Si-O) on the CF.

Table 1 shows the chemical composition of silane-treated CF. It is found that the surface composition of the CF changed substantially after silane treatments. The carbon content on the surface of CF was significantly decreased after the silane coupling treatments. Meanwhile, the oxygen and silicon content in-

Table 2. Surface energetics and thermodynamic W_A of carbon fibers with P-PP before and after sizing reatments

CF samples	γ_S (mN/m)	γ_S^d (mN/m)	γ_S^p (mN/m)	Composite description	W_A	W_A^L	W_A^{SP}
Unsized-CF	28.7	26.6	2.1	Unsized-CF/P-PP	63.62	56.12	7.50
VTES-CF	33.3	29.9	3.4	VTES-CF/P-PP	69.05	59.50	9.55
APS-CF	35.7	32.1	3.6	APS-CF/P-PP	71.47	61.65	9.82
MPTS-CF	38.3	33.8	4.5	MPTS-CF/P-PP	74.24	63.26	10.98

W_A, work of adhesion; P-PP, polarized polypropylene; CF, carbon fibers; VTES, vinyltriethoxy silane; APS, amino propyl triethoxy silane; MPTS, methacryloxy propyl trimethoxy silane.

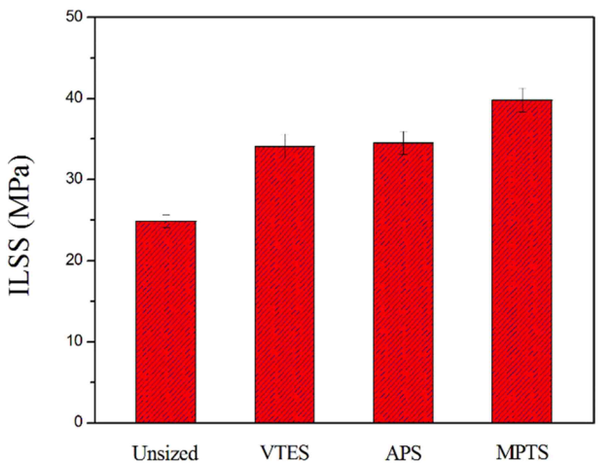

Fig. 3. Interlaminar shear strength (ILSS) of carbon fiber/polarized polypropylene matrix composites before and after various silane coupling treatments. VTES, vinyltriethoxy silane; APS, amino propyl triethoxy silane; MPTS, methacryloxy propyl trimethoxy silane.

creased. Most notably, the oxygen content of the MPTS sample showed predominant enhancement. In order to confirm the actual increase in the amounts of oxygen and silicon content, R-O_{1S} and R-Si_{2p}, the real increased content of each element was calculated by subtracting the values of unsized CFs from those of silane-treated CFs. It was found that R-O_{1s} and R-Si_{2P} of MPTS-treated CFs were dramatically enhanced compared to those of other silane-treated CFs. As confirmed by the R-O_{1S}/R-Si_{2P} ratio, one silane atom introduced two oxygen atoms to MPTS-treated CFs whereas contributed only one oxygen atom. In the case of the R-C_{1S}/R-Si_{2P} ratio, the value of MPTS-treated CFs was three or four times higher than those of other CFs. This indicates that polar surface groups on MPTS-treated CFs was actively formed, resulting in high surface polarity and functionality of the carbon fiber surfaces.

Fig. 2 shows the high resolution O_{1S} curve fit spectra for untreated (Fig. 2b) and silane-treated CF (MPTS) (Fig. 2c). Each functional group gives rise to a signal in the XPS spectrum with a particular range of binding energy. The O_{1S} spectra of the non-treated CF reveal the presence of three peaks. Peak 1 corresponds to the hydroxyl groups (-OH), peak 2 corresponds to the carbonyl groups (C=O), and peak 3 corresponds to the C-O group. The O_{1S} spectra of the silane treated CF reveal the presence of four peaks corresponding to the Si-O-Si group (BE = 530.7 eV), C-Si-O groups (BE = 531.9 eV), C=O groups (BE = 532.7 eV), and C-O groups (BE = 533.6 eV).

It is interesting that two peaks (Si-O-Si and C-O-Si) were newly formed and the hydroxyl group peak was diminished or significantly weakened. This indicates that hydroxyl groups were weakened or mixed with Si-O-Si peaks, due to the condensation reactions between Si-OH and the hydroxyl group of CF.

The surface free energy (γ_S) of a solid can be calculated by the sum of two components: a London dispersive component(γ_S^L) of nonpolar interaction and a specific component(γ_S^{SP}) describing all other type of interactions, such as Debye, Keesom, hydrogen bonding, and other polar effects; that is [25,26],

$$\gamma_S = \gamma_S^L + \gamma_S^{SP} \tag{3}$$

The results of surface free energy measurements of the CF are summarized in Table 2. It is observed that the polar components of fiber surfaces are highly increased by silane coupling treatments. On the other hand, the dispersive component was slightly enhanced. This shows that the silane efficiently increased the surface free energy due to the enhancement of polar and dispersive components. From the surface energetic point of view, it is predicted that an increase of the surface free energy plays a crucial role in improving the degree of interfacial adhesion strength between the polarized polypropylene matrix and the silane treated CF.

The dispersive components in surface free energy can be determined as a result of the molecular volume of the silane coupling agents due to the different chemical structure. The dispersive component can be calculated by the Lorentz-Lorenz-Debye equations [27].

$$\left(\frac{n^2-1}{n^2+2}\right)\frac{M}{\rho} = \frac{N_A\alpha_0}{3\varepsilon_0} \tag{4}$$

Here, n is the refractive index, M is the relative molecular mass, ρ is the molecular density, and ε_0 is the permittivity of the vacuum. The dispersive component (α_0) is a function of M/ρ, which represents the specific molecular volume. Among silane coupling agents, MPTS has a large molecular volume compared to APS and VTES. This result shows that the large molecular volume by silane treatments can be attributed to the high dispersive component, which results in enhancement of the surface free energy of the silane treated CF.

The thermodynamic work of adhesion (W_A) determines the energy required to separate two phases reversibly from their equilibrium when in contact with each other. According to the

Fowkes and Harkins [28]-equation, the W_A can be calculated using the surface free energy obtained from the contact angle. The following equation holds:

$$W_A = W_A^L + W_A^{SP} \quad (5)$$

$$W_A^L = 2\sqrt{\gamma_S^L \cdot \gamma_L^L} \quad (6)$$

$$W_A^{SP} = 2\sqrt{\gamma_S^{SP} \cdot \gamma_L^{SP}} \quad (7)$$

where the superscripts L and SP refer to the London dispersive and specific components, respectively, and the subscripts S and L represent the solid and liquid phases, respectively.

The calculated work of adhesion of CF before and after silane treatments are shown in Table 2. It is observed that the work of adhesion of silane-treated CF showed higher adhesion strengths than that of the non-treated fibers. It was also found that the MPTS-treated carbon fiber sample showed the highest work of adhesion. As seen in Table 2, MPTS-treatments yielded the highest surface free energy due to the highly enhanced polar and dispersive components. The enhanced surface free energy of CF can be attributed to the increase in work of adhesion, which results in increased interfacial adhesion strength between the CF and the polarized polypropylene matrix.

The mechanical interfacial properties of fiber reinforced composites depend strongly on the degree of adhesion between the fibers and the matrices. The degree of adhesion at the interface between a fiber and a matrix can be measured by the ILSS for the mechanical interfacial behaviors.

From the ILSS results shown in Fig. 3, the presence of silane coupling agents leads to improved ILSS values of the carbon fiber reinforced polarized polypropylene composites. Furthermore, the optimum strength was found with MPTS treatment. Notably, the ILSS results support the reliability of the data, since the tendencies in the ILSS values are very similar to those of the XPS data and surface free energy. This shows that the oxygen functional groups and surface free energy values can determine the resulting adhesion strengths of the carbon fiber/polarized polypropylene matrix in this system.

Practically, the well-known ring-opening crosslinking reaction between amino-silane coupling agents (APS) and polypropylene-co-maleic anhydride can dramatically enhance the mechanical interfacial strength [29-34] of a composite. However, we used partially polarized polypropylene ($Ma_{0.2}$-$PP_{99.8}$) as a matrix. It indicates that the maleic anhydride structures of the matrix will not significantly react with the silane-treated CF. This indicates that the effects of the cross-linking reaction between the silane coupling agents and P-PP on the final mechanical interfacial properties of the composites are negligible in this system.

To sum, we evaluated the effects of various silane coupling agents on the mechanical interfacial strength of carbon fiber-reinforced polarized-polypropylene composites. Small effects of cross-linking reactions are ignored, and all mechanisms for the enhancement of mechanical interfacial properties of the composites involve physical interlocking between fillers and matrices.

In this work, silane treatments of CF were carried out to improve the interfacial adhesion between CF and polarized polypropylene. As a result, it was found that the interfacial adhesion of the carbon fiber/polarized polypropylene matrix increased with the polar component of surface free energy, and oxygen functional groups increased after silane treatment, leading to improved mechanical performance of the composites. These results indicate that the silane treatments on the CF surface play an important role in improving the degree of adhesion at the interface in a polarized polypropylene matrix composite system.

Conflict of Interest

No potential conflict of interest relevant to this article was reported.

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