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Enhanced interfacial, electrical, and flexural properties of polyphenylene sulfide composites filled with carbon fibers modified by electrophoretic surface deposition of multi-walled carbon nanotubes

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ABSTRACT

Electrophoresis can be an effective approach for depositing carbon nanotubes (CNTs) on the surface of carbon fiber (CF). Nevertheless, it has been rarely reported on polyphenylene sulfide (PPS) composites filled with CFs surface-modified by CNTs based on electrophoresis. In this study, we investigated the electrophoresis process conditions that can completely coat CF with multi-walled CNTs (MWCNTs) using self-manufactured electrophoresis equipment, and the enhancement of interfacial, electrical and flexural properties of PPS composites by introducing CFs coated with MWCNTs based on electrophoresis. In particular, interfacial shear strength (IFSS) of the PPS composites was measured by microbond tests and improved by about 41.7% due to the MWCNTs introduced on the surface of CFs. These enhancements were theoretically explained by an interface-modified CF-based micromechanical model. Introducing MWCNTs on the CF surface based on electrophoresis was demonstrated to be an effective method for improving the interfacial, electrical and flexural properties of PPS composites.

1. Introduction

Polyphenylene sulfide (PPS) is one of the most widely used engineering plastics in the aerospace and automotive industries due to its excellent heat resistance, flame retardancy, chemical resistance, dimensional stability and processability [1,2]. However, PPS resins, which are a type of semi-crystalline polymer, have been otherwise limited in application by their relatively low mechanical strength and fragile characteristics [1,2]. The mechanical properties of PPS resin can be improved by incorporating fiber and/or filler [1,3]. In particular, carbon fiber (CF) has been employed as a reinforcing fiber material in PPS resin because it provides excellent reinforcing effect and heat characteristics [1,3]. As a result, CF reinforced PPS composites are considered high-performance engineering materials, not only because they exhibit beneficial physical properties, but also excellent processability [1,3].

The physical properties of CF reinforced composite materials are known to depend on the properties of the matrix and filler, the shape and volume fraction of the fibers, and the chemical and physical interactions between the fibers and the matrix [2]. In order to fabricate a CF reinforced PPS composite with excellent mechanical properties, good interfacial adhesion between the fiber and the matrix is required [4–9]. CF is a highly crystallized graphite based material with a non-polar surface and an inert structure, and interacts poorly with most polymer resins [2,10]. Therefore, it is necessary to control the interface between CF and PPS to improve mechanical properties.

A method has been proposed for growing or adhering a nanoscale whisker, which is characterized by a high aspect ratio and surface to volume ratio, on the surface of CFs to improve fiber-matrix interactions and interfacial properties [11]. The nanoscale whiskers have been demonstrated to significantly increase the load transfer area between the fibers and the matrix, thus effectively enhancing fiber-matrix interactions and interfacial properties [12]. Also, Chen et al. reported that graphene or carbon nanotubes (CNTs) based interface layers could improve load transfer from the matrix to the filler and reduce interfacial stress concentration [13,14]. Several studies have been proposed over the last few years to develop effective CNT/CF hybridization methods. These can be broadly divided into two categories [11]: the first involves the direct growth of CNTs on a CF surface by chemical vapor deposition (CVD) and the second is accomplished by coating CNTs on the CF surface, by spraying or electrophoresis.

Electrophoresis can be an effective approach to coat CNTs on a CF

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surface, and the technique can be easily automated and utilized in industrial applications [15]. Nevertheless, it has been rarely reported on polyphenylene sulfide (PPS) composites filled with CFs surface-modified by CNTs based on electrophoresis. In this study, we investigated the electrophoresis process conditions that can completely coat CF with multiwall CNTs (MWCNTs) using self-manufactured electrophoresis equipment. PPS composites filled with CNTs-coated CF were fabricated to evaluate the interfacial, electrical and flexural properties of the composites. In particular, interfacial shear strength (IFSS) of the fabricated composites was evaluated based on the microbond test and a modeling method based on control of the interfacial properties was proposed to theoretically understand the properties of the composites.

2. Experimental

2.1. Materials

PPS powders (W316 grade) supplied by Kureha Chemical Industry Co., Ltd (Tokyo, Japan) were used as the matrix. The density and molecular weight of the material were 1.35 g/cm^3 and 35,000 g/mol, respectively. CF (Granoc XN-80-A2S) was supplied by Nippon Graphite Fiber Corporation (Tokyo, Japan). The diameter and length of the CF was 10 μm and 6 mm, respectively. Desizing of the CFs was performed by immersing them in 40% nitric acid for 2 h because the sizing agents on the CF surface can act as an insulating gap that prevents the movement of electrons during electrophoresis. MWCNTs (Hanwha Nanotech Ltd., Seoul, Korea) manufactured by the CVD method were used for electrophoresis.

2.2. Preparation of MWCNT suspension for electrophoresis

1 g of MWCNTs and 200 ml of an acid mixture of nitric acid and sulfuric acid in a ratio of 1:3 were mixed, and the surfaces of the MWCNTs were modified at a low rotation speed of 200 rpm. After the surface modification, the MWCNTs were washed with distilled water of 2–3 MΩ to remove residual acid solution, until pH 7. The washed MWCNTs were then dried at 80 °C for 24 h. To overcome the phase separation, aggregation and low dispersion of the MWCNTs, cetyltrimethylammonium bromide (CTAB, Merck, Darmstadt, Germany), a cationic surfactant, was used to alter the electrical properties of the surface of the MWNTs. CTAB (20 times as much as MWCNTs) and the prepared MWCNTs were added to 300 ml of deionized water and dispersed by sonication for 5 min to prepare a positively charged MWCNTs solution.

2.3. Electrophoresis

Electrophoresis is a process in which very small particles or colloids dispersed in a liquid move under electric field in one direction toward a pole. In this study, the positively charged MWCNTs are moved toward the negatively charged CFs by the electrostatic attraction by the electric field, and are deposited. A device was constructed to transfer charge from the prepared suspension to the desized CFs using a copper film, which served as an electrode connected to an external direct current (DC) power source as shown in Fig. 1. Designed to provide continuous treatment to CFs, the system consisted of two power supplies capable of voltage regulation, a motor to control the speed of movement of the CFs, and an acrylic case for electrical safety. The electrophoresis conditions were controlled by adjusting the voltage of the power supply and deposition time.

2.4. Composite fabrication

PPS composites filled with desized CFs or with CFs coated with MWCNTs were fabricated with their respective target compositions as listed in Table 1, using a twin-screw extruder (TK-40, BauTech, Seoul, Korea). The screw rotation speed of the compounder was 300 rpm, and the temperature of the barrel was set at 295, 320, 320, 320, and 290 °C from the inlet of the raw material. The fabricated mixture was pelletized, and specimens for property measurements were prepared at 320 °C using a programmable hydraulic press (MTP-14, Tetrahedron Associates, Inc., San Diego, USA).

2.5. Characterization

2.5.1. Morphology

The morphology of the desized CFs and CF with MWCNTs introduced based on electrophoresis was observed, using a field emission scanning electron microscopy (FE-SEM, Nova NanoSEM 450, FEI Corp., OR, USA) and a transmission electron microscope (TEM, Tecnai F20, FEI Corp., OR, USA). The CF specimens for FE-SEM measurement were coated with platinum in a vacuum for 200 s using a sputter coating machine (Ion Sputter E-1030, Hitachi High Technologies Corp., Japan). The FE-SEM measurements were carried out at 15.0 kV. For the TEM sample preparation, the CFs coated with MWCNTs were dispersed in ethanol and sonicated for 5 min, and subsequently the dispersion was dropped onto a lacey-carbon coated film on a Cu grid. The TEM measurements were performed at 120 kV.

2.5.2. Microbond tests to measure IFSS

To investigate the interfacial properties between the fiber and matrix, the IFSS of the PPS composites filled with desized CFs or with CFs coated with MWCNTs was measured based on a microbond test, as shown in Fig. 2(a). The free fiber length of the prepared specimens in the microbond test was approximately 20 mm. Molten PPS resin droplets were placed on the CF monofilament and cooled, as shown in Fig. 2(b). The microbond test was performed using an interfacial microbond evaluation instrument (Model HM410) fabricated by TOHEI SANYOU CO. LTD (Tokyo, Japan). The prepared CF fiber with PPS droplets was loaded at a speed of 2 mm/min while the force was recorded against the displacement. The interfacial shear strength, τ, was determined using the following equation [16].

$$\tau = \frac{F_{max}}{\pi D L_e} \quad (1)$$

where F_{max} is the force at the moment when the interfacial debonding or sliding occurs, D is the CF diameter, and L_e is the embedded length of the CF in the PPS resin droplet.

2.5.3. Physical properties of the composites

Electrical conductivity measurements were performed on the PPS composite specimens, which had a length of 80 mm, width of 19.5 mm and thickness of 2.5 mm. The volume electrical resistivity of the prepared composite specimens was measured by standard four electrode method, based on the previous study [17]. A standard electrode assembly for the volume resistivity measurement based on the four electrode method was used with a distance between the knife-edged electrodes of 15 mm and a distance between the potential electrode and the current electrode of 20 mm, respectively. The flexural strength and modulus of the prepared PPS composite specimens were measured according to ASTM D790.

3. Theoretical model for composites containing interface-modified CF

A micromechanics-based theoretical model was adopted to theoretically calculate the electrical and mechanical behaviors of the interface-modified CF embedded composites. The characteristics of the interface was selected as a key parameter to describe the physical and chemical transformations in the complex material system. The theoretical scheme of the present model is represented in Fig. 3. To reflect the actual properties of the composite, the CF was assumed to be 3D

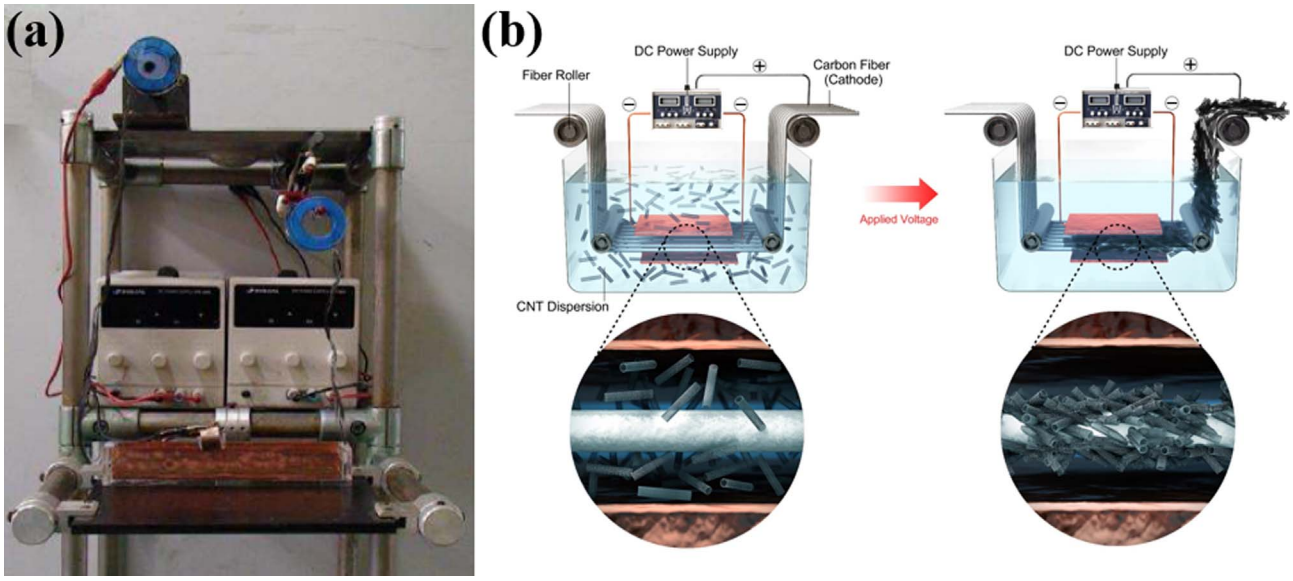

Fig. 1. (a) Self-manufactured electrophoresis equipment and (b) schematic of the designed and fabricated electrophoresis system to provide continuous treatment to CFs. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 1
Composition of fabricated PPS composites.

Sample	PPS (wt%)	CF (wt%)	MWCNT (wt%)
PPS/CF1	99	1	0
PPS/CF5	95	5	0
PPS/CF10	90	10	0
PPS/CF20	80	20	0
PPS/CF30	70	30	0
PPS/CF40	60	40	0
PPS/CF50	50	50	0
PPS/CF60	40	60	0
PPS/CF-MWCNT1	99	0.9	0.1
PPS/CF-MWCNT5	95	4.5	0.5
PPS/CF-MWCNT10	90	9	1
PPS/CF-MWCNT20	80	18	2
PPS/CF-MWCNT30	70	27	3
PPS/CF-MWCNT40	60	36	4
PPS/CF-MWCNT50	50	45	5
PPS/CF-MWCNT60	40	54	6

randomly oriented and uniformly distributed in the matrix. In addition, the CF interface decorated with MWCNTs was modeled as a thinly coated interfacial resistivity and an elastic spring to illustrate the effective electrical and mechanical properties, respectively.

3.1. Effective electrical conductivity

Let us first consider a two-phase composite consisting of a polymer matrix (phase 0) with electrical conductivity L_0, and a spheroidal CF (phase 1) with electrical conductivity L_1. On the basis of the Ponte Castañeda-Willis (PCW) approach [18], the effective electrical conductivity of the CF-reinforced polymer composite can be derived as [19]

$$L^* = L_0 + [\mathbf{I}-\phi_1\mathbf{T}L_0^{-1}]^{-1}(\phi_1\mathbf{T}) = L_0 + \frac{3L_0\mathbf{T}}{3L_0-\mathbf{T}} \tag{2}$$

with

$$\mathbf{T} = \frac{\phi_1(L_1-L_0)}{3}\left(\frac{1}{L_0 + S_{11}(L_1-L_0)} + \frac{1}{L_0 + S_{22}(L_1-L_0)} + \frac{1}{L_0 + S_{33}(L_1-L_0)}\right) \tag{3}$$

where ϕ_q (q = 0, 1) is the volume fraction of the q-phase; $S_{11}, \ldots, S_{33}$ are the aspect ratio-dependent Eshelby's tensor, which can be defined as

$$S_{11} = S_{22} = \frac{\alpha}{2(\alpha^2-1)^{3/2}}[\alpha(\alpha^2-1)^{1/2}-\cosh^{-1}\alpha], \quad S_{33} = 1-2S_{11} \tag{4}$$

Here, the aspect ratio is $\alpha = L/d$, where L and d are the length and diameter of the CF, respectively. The Hashin-Shtrikman (HS) bounds are also coupled with Eq. (2) to prevent an unrealistic prediction which exceeds the theoretical limitations, and the equation for the HS bounds are given in Pan et al. [19].

In the present study, the consideration of interface effects is inevitable, and to describe those characteristics, we assumed that the CF was surround by a thin layer of electrical resistivity with uniform thickness. From previous studies [19–21], the concept of a thin interface layer of electrical resistivity (ρ) is effective for the nanoscale interphase between fillers and the matrix. A more precise prediction can also be made with tunneling activity (γ) effect using Cauchy's probabilistic model [20]. The abovementioned factors were incorporated into the present model, and a detailed description of the model parameters (ρ and γ) can be found in [20,21], respectively.

3.2. Effective mechanical constitutive relations

The prepared composite used to predict mechanical properties was assumed to be comprised of a matrix (phase 0) with elastic modulus E_0 and Poisson's ratio ν_0, and spheroidal-shaped CFs (phase 1) with elastic modulus E_1 and Poisson's ratio ν_1. Based on the governing equation for linear elastic composites [22], the effective elastic stiffness for the composites filled with interface modified CF can be derived as

$$\mathbf{C}^* = \mathbf{C}_0\cdot[\mathbf{I} + \phi_1\{(\mathbf{C}_1 + \mathbf{C}_0)\cdot\mathbf{C}_0 + \mathbf{S}_1^m\}\cdot[\mathbf{I}-\phi_1\mathbf{S}_1^m\cdot\{(\mathbf{C}_1 + \mathbf{C}_0)\cdot\mathbf{C}_0 + \mathbf{S}_1^m\}^{-1}]^{-1}] \tag{5}$$

with

$$(\mathbf{S}_1^m)_{ijkl} = \frac{1}{4(1-\nu_0)}[\mathbf{S}_{IK}^{(1)}\delta_{ij}\delta_{kl} + \mathbf{S}_{IJ}^{(2)}(\delta_{ik}\delta_{jl} + \delta_{il}\delta_{jk})] \tag{6}$$

where $\mathbf{C}_q$ (q = 0, 1) denotes the stiffness tensor of the q-phase, and $\mathbf{I}$ signifies the fourth-rank identity tensor, respectively. $\mathbf{S}_1^m$ is the modified Eshelby's tensor for a spheroidal inclusion with imperfect interface, and the components can be found in Yang et al. [23]. With the help of the modified Eshelby's tensor and the orientation averaging techniques, the effective stiffness tensor for the present composite is explicitly

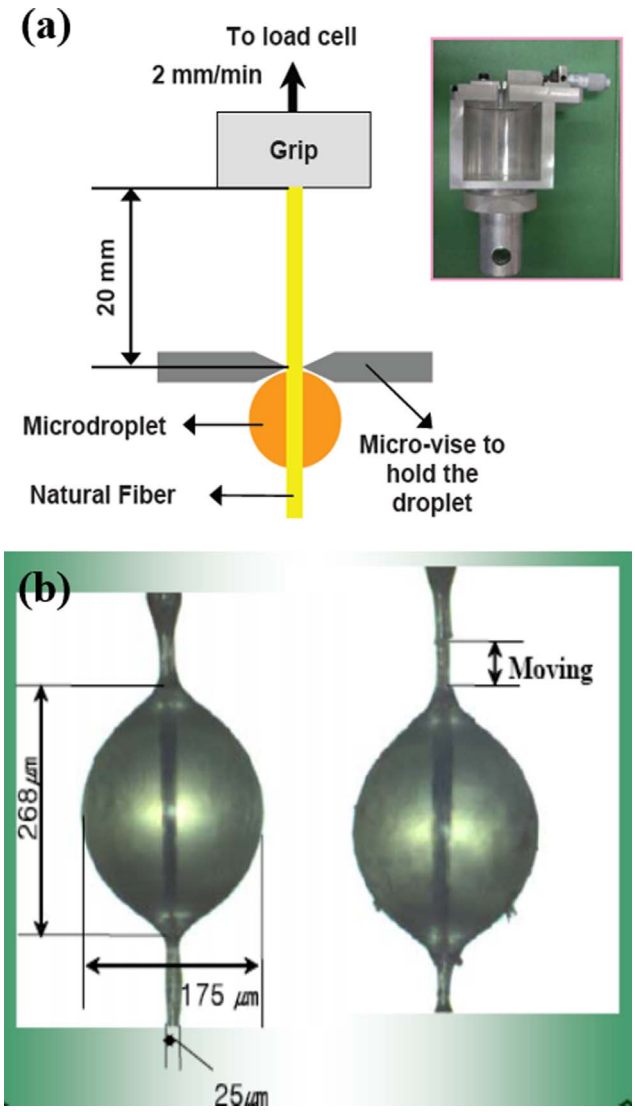

Fig. 2. (a) Schematic of the microbond test and (b) photograph of the prepared microbond test specimen. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

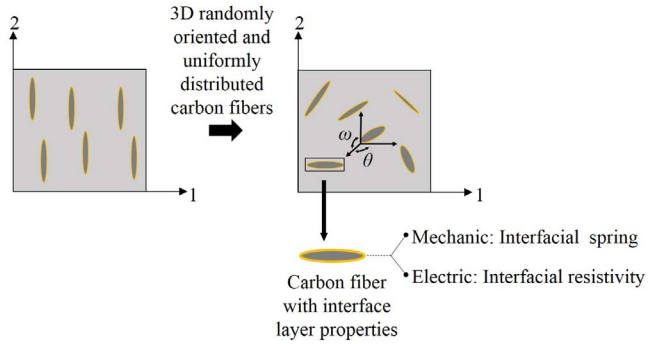

Fig. 3. A theoretical schematic representation of the present composite system with interfacial characteristics. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

expressed as

$$\mathbf{C}^* = \lambda^* \delta_{ij}\delta_{kl} + \mu^*(\delta_{ik}\delta_{jl} + \delta_{il}\delta_{jk}) \tag{7}$$

with

$$\lambda^* = \frac{1}{15}[C_{11}^{(1)} + 4C_{12}^{(1)} + 4C_{21}^{(1)} + 6C_{22}^{(1)} + 2C_{11}^{(2)} - 4C_{12}^{(2)} + 2C_{22}^{(2)}]$$
$$\mu^* = \frac{1}{15}[C_{11}^{(1)} - C_{12}^{(1)} - C_{21}^{(1)} + C_{22}^{(1)} + 2C_{11}^{(2)} + 6C_{12}^{(2)} + 7C_{22}^{(2)}] \tag{8}$$

where the components $C_{11}{}^{(1)}, \ldots, C_{22}{}^{(2)}$ are given in Eq. (4) in Yang et al. [23].

4. Results and discussion

4.1. Electrophoresis conditions

The amount of MWCNTs introduced on the CF surface was investigated according to important process parameters of the electrophoresis system constructed in this study such as voltage and deposition time. Thus, electrophoresis experiments were carried out while adjusting the voltage between 10 and 30 V and deposition time between 5 and 10 min. As a result, it was found that when the voltage was larger and the deposition time was longer, a greater number of MWCNTs were introduced onto the CF surface, as shown in Fig. 4. In particular, it was confirmed that the external DC voltage had the greatest influence on the amount of MWCNTs introduced. As shown in Fig. 4(c), when a voltage of 30 V was applied for 5 min, it can be seen that the MWCNTs were adsorbed on the CF surface as much as they can be coated. Lee et al. reported that excessive amount of the CNTs may cause negative effect on the mechanical properties of the composites because of the wetting of resin on the fiber surface and the adhesion between CNTs and CFs [11]. Therefore, in this study, the electrophoresis conditions used for coating MWCNTs on CFs were determined as a voltage of 30 V and a deposition time of 5 min, and the weight fraction of CF and MWCNT was controlled to 9–1 under the conditions.

4.2. Interfacial properties of composites

Fig. 4(f) shows the results of microbond tests of the PPS composites filled with desized CFs or CFs coated with MWCNTs based on electrophoresis, under a voltage of 30 V and a deposition time of 5 min. The IFSS of the PPS composites filled with the CFs coated with MWCNTs and with desized CFs were 47.2 and 33.3 MPa, respectively, indicating that the IFSS of the PPS composites was improved by about 41.7% due to introduction of MWCNTs on the surface of CFs. To derive accurate theoretical predictions, the improved interfacial properties provided by the electrophoresis of MWCNTs were applied to the theoretical calculations.

4.3. Electrical properties of composites

The percolation threshold of the PPS composites filled with the MWCNT coated CFs based on electrophoresis was lower than that of PPS composites filled with desized CFs, as shown in Fig. 5. When the filler content was 60 wt%, the electrical conductivities of the PPS composites were measured to be 320 S/m and 180 S/m, respectively, depending on whether electrophoresis was performed or not. Introducing MWCNTs on the CF surface improved the electrical conductivity of the composites by about 78%. This is probably due to the fact that the electrical conductivity of the CFs was improved by introducing MWCNTs, which are excellent electrically conductive fillers, on the CF surface through electrophoresis.

In addition, comparisons between the experimental data and predictions on the modified CF-reinforced composites were made to illustrate the predictive capability of the theoretical model. The same material properties as those measured in the experiments were adopted for the comparisons: $L_0 = 5E-10$ S/m, $L_1 = 1E3$ S/m, $L = 6$ mm, and $d = 10$ μm. The electrical conductivity of MWCNT along the transverse axis is assumed to be 1000 times smaller than the longitudinal axis. Since the model parameters for the electrical interface could not be experimentally determined in the present study, fitted values were

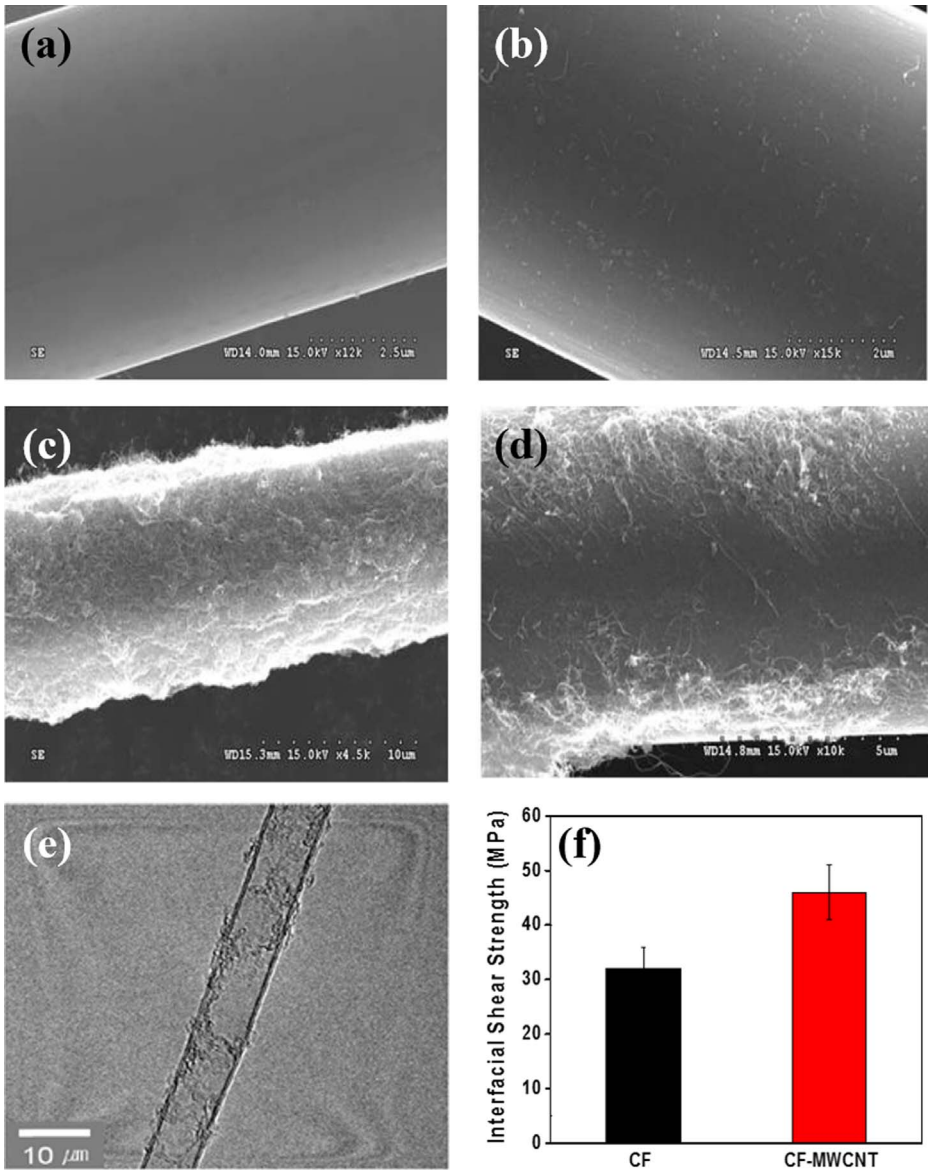

Fig. 4. SEM images of CFs (a) without MWCNTs and coated with MWCNTs based on electrophoresis (b) at 10 V for 10 min, (c) at 30 V for 5 min, (d) at 20 V for 10 min, (e) TEM image of CFs coated with MWCNTs based on electrophoresis at 20 V for 5 min, and (f) IFSS of PPS composites filled with desized CFs and CFs coated with MWCNTs based on electrophoresis at 30 V for 5 min. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

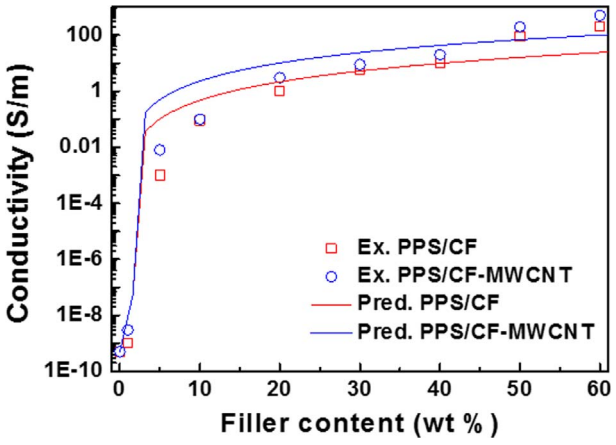

Fig. 5. Electrical conductivity of PPS composites. "Ex." and "Pred." indicate experimental and predicted results, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

applied: $\rho = 5E-5\,m^2/S$ for PPS/CF and $\rho = 1E-5\,m^2/S$ for PPS/CF-MWCNT where ρ denotes the interfacial resistivity. The parameter of the tunneling effect within the composite was assumed to be identical in all cases ($\gamma = 5E-2$).

The calculated electrical conductivity based on the above material and model parameters is shown in Fig. 5. Overall, the predicted values of the composites were in good agreement with the experimental data. As the same trends observed in the experiment, it is predicted that the modification of interfacial properties by the material treatment would greatly influence the effective electrical property of the composite material. Specifically, the electrical conductivity value gradually improved as the interface resistance decreased from $\rho = 5E-5$ to $\rho = 1E-5$.

4.4. Flexural properties of composites

Flexural properties of PPS composites filled with desized CFs or CFs with MWCNTs based on electrophoresis were evaluated as shown in Fig. 6. The flexural strength and the modulus of the PPS composites

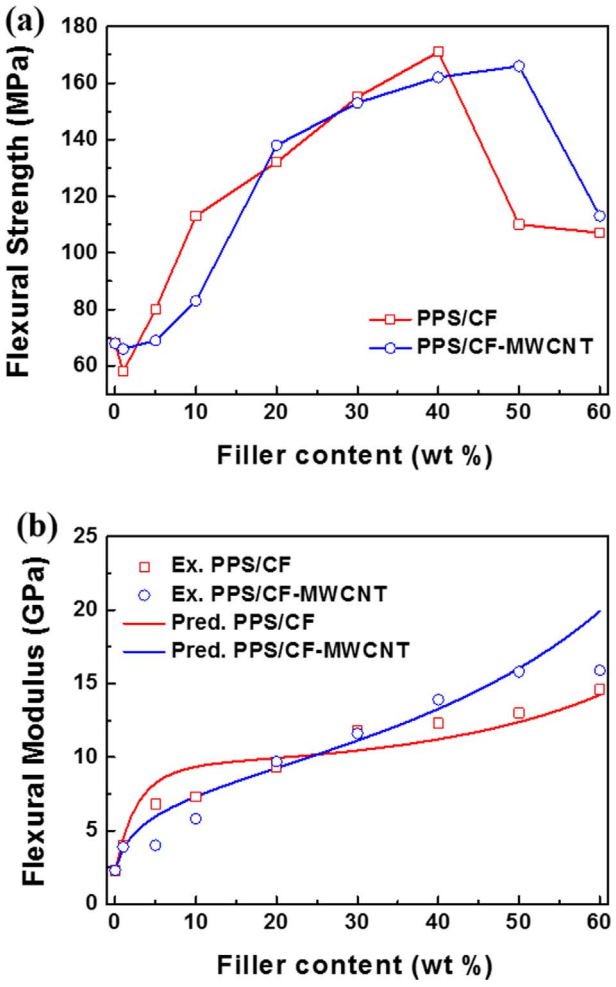

Fig. 6. Flexural (a) strength and (b) modulus of PPS composites. "Ex." and "Pred." indicate experimental and predicted results, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

filled with CFs coated with MWCNTs were improved. In particular, the flexural strength of the composite filled with 50 wt% fillers was significantly improved as shown in Fig. 6(a). These results can be attributed to the fact that the filler and PPS resin form a more complete interface and show an improved IFSS as MWCNTs were introduced on the CF surface based on electrophoresis, as previously mentioned at the IFSS results in Fig. 4(f).

To further demonstrate the effectiveness of the present micromechanical model, the theoretical predictions for the flexural modulus of the composites were compared with the experimental data. We adopted the material properties obtained from the experiment: $E_0 = 2.3$ GPa, $\nu_0 = 0.35$, $E_1 = 235$ GPa, and $\nu_1 = 0.2$. It should be noted that various interface parameters were applied to the experimental comparison, because the interfacial characteristics differ depending on the combination of composites. Hence, the compliance of the interface parameters, β, was estimated by fitting the experimentally obtained flexural modulus: $\beta = 3 \cdot {\phi_1}^{1.3}$/GPa for PPS/CF and $\beta = 2 \cdot {\phi_1}^{0.8}$/GPa for PPS/CF-MWCNT, respectively. The flexural modulus can be calculated via $E^f = \{\mu^*(3\lambda^* + 2\mu^*)\}/(\lambda^* + \mu^*)$, where λ^* and μ^* are given in Eq. (7).

In order to predict the flexural strength, a further plastic theory is required; however, the proposed model only considers the elastic behaviors of composite materials. It was concluded that this consideration of the plasticity is beyond the scope of the present study, and the comparison between the experimental results and the simulation was made only within the elastic region. A good correlation of flexural moduli can be observed with different composite systems, as shown in Fig. 6(b), while a slight deviation was observed from the PPS/CF-MWCNT. These comparisons allowed the effect of the interfacial properties between the matrix and CF, induced by the hybrid with MWNTs, to be theoretically analyzed. They also indicate that the interfacial treatment has a high potential to improve the mechanical behaviors of the composite.

5. Conclusion

Electrophoresis was used to coat CFs with MWCNTs to improve the interfacial, electrical and flexural properties of CF filled PPS composites. To fully coat the CFs with MWCNTs, electrophoresis was performed using self-manufactured electrophoresis equipment under investigated conditions, specifically, at a voltage of 30 V and deposition time of 5 min. The IFSS of the PPS composites was improved by about 41.7% due to the MWCNTs introduced on the surface of CFs based on the electrophoresis. The percolation threshold of the PPS composites filled with CFs coated with MWCNTs was lower than that of CF filled PPS composites without MWCNTs. The introduction of MWCNTs on the CF surface improved the electrical conductivity of the composites and, at 60 wt% hybrid fillers, the conductivity increased by about 78%. This result was explained by a micromechanics-based theoretical model which attributed the improvement in the electrical conductivity of the CFs to the electrophoresis-deposited MWCNTs, which are excellent electrically conductive fillers. The flexural strength and the modulus of the PPS composites filled with MWCNTs-coated CFs were also improved. From the comparisons between experimental and theoretical results, the effect of the interfacial properties between the matrix and CF induced by the hybrid with MWCNTs was theoretically analyzed. The results demonstrated there was great potential for improving mechanical behaviors of the composites through the interfacial treatment.

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