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Synergistic effects induced by oxy-fluorination of carbon preforms to improve the mechanical and thermal properties of carbon–carbon composites

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ABSTRACT

Oxy-fluorination of carbon preforms with various F_2:O_2 gas mixtures were examined to improve the mechanical and thermal properties of carbon fiber-reinforced carbon composites (C/C composites). The oxy-fluorination of the preforms introduced functional groups onto the preform surface, which improved their thermal properties. Oxy-fluorination also improved the interfacial adhesion of the C/C composites, resulting in increased flexural strength and anti-oxidation. Two synergistic effects of oxy-fluorination on the carbon preform are suggested. One optimizes interfacial adhesion by forming hard chemical bonds and soft electrophilic bonds between the surface functional groups of the oxy-fluorinated carbon preforms and the functional groups of the carbon precursors. The other improves anti-oxidation of the C/C composites by improving the thermal properties of the carbon preform itself and interfacial adhesion which resulted in reducing pores, voids, and interfacial cracks.

1. Introduction

In recent years, carbon fiber-reinforced carbon composites (C/C composites) have attracted much attention due to their potential use as materials for extreme environment applications, such as nozzles for solid rocket motors, heat shields for re-entry vehicles, and brake discs for advanced aircraft [1,2]. Important properties of C/C composites for these applications are high thermal conductivity, excellent thermal stability, a low coefficient of thermal expansion, low density, a high strength-to-weight ratio, excellent friction properties, and good resistance to fatigue, as well as a retention of these properties at high temperatures [3–5].

There are various factors on which the mechanical properties of C/C composites depend, such as heat treatment temperature, type and volume of fiber used, fiber architecture, matrix density, and the fiber–matrix interface [6]. Of these factors, the load transfer at the interface is one of the most important factors affecting the mechanical properties [7]. In C/C composites, when the interface adhesion of the composite is too weak, the composite integrity is impaired, whereas when the adhesion is too strong, fiber damage is induced during heat treatment [5]. Therefore, control of the interfacial adhesion between the carbon matrix and the carbon fiber surface in C/C composites is one of the most important issues in preparing C/C composites.

Surface modification of carbon fibers has been reported as a method to improve the interfacial adhesion of composites. Various studies have reported that acid treatment of carbon fibers increases the interfacial adhesion of C/C composites, but the mechanical properties of these composites deteriorate, due to the bonds being too strong in the fiber–matrix (F–M) interface [7–10]. Considering the disadvantages of conventional fiber surface treatment, direct fluorination appears to be a good alternative for improving F–M interfacial adhesion. Direct fluorination is a soft surface modification method, maintaining the bulk properties of the modified material. Based on the gas mixture utilized, fluorination can be classified into two categories, ordinary direct fluorination and oxy-fluorination. Ordinary direct fluorination uses a mixture of fluorine and inert gas, such as nitrogen or argon, whereas oxy-fluorination uses a mixture of fluorine and oxygen. The oxy-fluorination of carbon materials enhances the surface wettability due to the introduction of polar groups such as $>C{=}O$, –OH, and –COOH [11–13]. Therefore, oxy-fluorinated carbon surfaces are expected to have good adhesive properties with free –OH groups of phenol resin, which is a popular carbon matrix precursor for C/C composites. Moreover, oxygen-containing surface groups, introduced to the carbon fibers by oxy-fluorination, may improve the thermal properties of the fiber itself and those of the C/C composites because the carbon surface is already partially oxidized.

In this study, the effects of oxy-fluorination of needle-punched carbon preforms on the mechanical and thermal properties of C/C composites were investigated under various oxy-fluorination conditions. Changes in the surface chemical compositions of the

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carbon fibers and microstructures of the C/C composites were also investigated.

2. Results and discussion

2.1. Surface chemical composition of oxy-fluorinated carbon preforms

Since XPS analysis was very useful to characterize surface chemical structures of the fluorinated carbon fibers in the previous studies [14–16], even only a few studies reported oxy-fluorination of carbon fibers [17], characterization of the surface chemical composition of the oxy-fluorinated carbon performs was carried out using XPS analysis. The resulting survey spectra are shown in Fig. 1 and Table 1. The major surface components of the as-received carbon preform were carbon and oxygen. This result corresponded to all the other previous studies which reported that the untreated PAN-based carbon fiber itself contained oxygen on the surface of the carbon fibers and the sources could be the precursor and sizing agents [14–19]. The major surface components of the oxy-fluorinated carbon preforms were carbon, oxygen, and fluorine, as shown in the XPS spectra of Fig. 1, with three distinctive peaks. The results of a quantitative peak analysis, given in Table 1, show that the surface carbon concentration decreased as the amount of F_2 in the gas mixture increased. It was also found that the oxygen content of the untreated carbon preform (CFPO00) was almost similar to that of the fluorinated carbon preform (CFPO100) which treated with only F_2 gas. This was attributed to oxygen and fluorine being introduced onto the carbon fiber surface of the carbon preforms via oxy-fluorination, indicating that F_2 could provide reaction sites for O_2.

The chemical bond changes of the carbon preforms after oxy-fluorination were investigated by C1s deconvolution. The results are depicted in Fig. 2. The components of C are shown in Table 2, along with the binding energies and assignments of the chemical bonds. The components observed for CPFO00 were C(1), C(2), C(3), C(4), C(5) and C(6) at 284.50, 285.5, 286.1, 287.1, 288.1, and 289.1 eV, as shown in Fig. 2(a), corresponding to sp^2 carbon, sp^3 carbon, C–OH/C–O–C, C=O, COOR, and COOH, respectively. After oxy-fluorination, C(7) and C(8) components were observed in CPFO37, CPFO55, CPFO73, and CPFO100 at 289.5 and 290.3 eV, as shown in Fig. 2(b)–(e), corresponding to semi-covalent and covalent C–F. All of the results were in good agreement with other studies [14–21]. The concentration of C(3), C(4), C(5), C(6), C(7), C(8) increased with an increasing F_2 to O_2 ratio of the gas mixture, whereas only those of C(7) and C(8) significantly increased when only F_2 gas was used. Based on the C1s deconvolution, it was found that the functional groups, such as –OH, –COOH, –COOR, and –F, were introduced onto the carbon preform surface [17,18,22] and were expected to have good adhesion to the phenol resin carbon matrix precursors, affecting the microstructure and mechanical and thermal properties of the C/C composites. The effect of the functional groups discussed in this section on the composite structure and properties will be further discussed in the later sections.

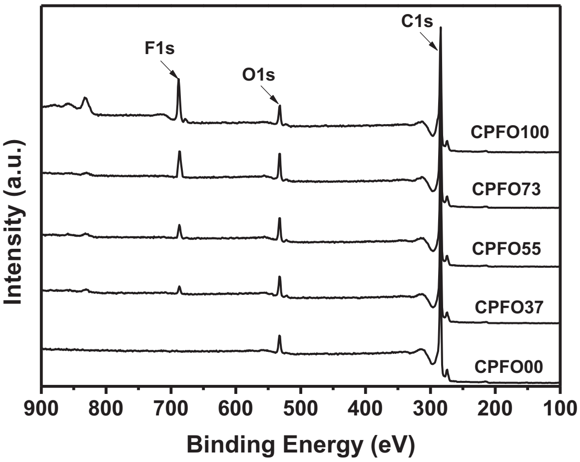

Fig. 1. XPS survey spectra of the as-received and oxy-fluorinated preforms.

Table 1
Surface chemical compositions of oxy-fluorinated carbon performs with various oxy-fluorination conditions obtained by XPS survey spectra.

Sample	Atomic percent (at%)			O1s/C1s	F1s/C1s
	C1s	O1s	F1s		
CPFO00	94.8	5.2	0.0	0.055	0
CPFO37	92.3	6.7	1.0	0.073	0.011
CPFO55	88.3	10.0	1.7	0.113	0.019
CPFO73	85.1	11.6	3.3	0.136	0.039
CPFO100	87.9	5.3	6.8	0.061	0.077

2.2. Microstructures of the prepared C/C composites

The microstructures of the as-received carbon preform and the prepared C/C composites are shown in Fig. 3. Comparing Fig. 3(a) and the others, it can be observed that the voids between fibers and spaces between fiber bundles were filled with carbon matrix. Interfacial cracks and unfilled spaces can be attributed to the low affinity of the phenol carbon precursor to carbon preform. In the image of CPFO00-C (Fig. 3(b)), pores, voids, and interfacial cracks between the carbon fiber and matrix were observed. The pores and voids are attributed to the low affinity of the phenol carbon precursor to carbon preform, resulting in poor impregnation. Interfacial cracks can be attributed to the low interfacial adhesion between the carbon matrix and carbon preform. In the image of CPFO37-C (Fig. 3(c)), pores, voids, and interfacial cracks can also be observed. Therefore, it seems that the interfacial adhesion of CPFO37-C is similar to that of CPFO00-C. In the images of CPFO55-C and CPFO73-C (Fig. 3(d) and (e)), fewer pores, voids, and interfacial cracks were observed than in CPFO00-C and CPFO37-C, with fewer in CPFO73-C than in CPFO55-C. In the image of CPFO-100 (Fig. 3(f)), some interfacial cracks can be observed, but there are fewer than in CPFO00-C and CPFO37-C and more than in CPFO55-C and CPFO73-C, suggesting interfacial adhesion between them.

To avoid being caught in a so-called microscopy trap, the bulk densities of the prepared C/C composites were also measured and shown in Fig. 4. The bulk densities of CPFO00-C, CPFO37-C, CPFO55-C, CPFO73-C, and CPFO100-C were 1.1, 1.1, 1.2, 1.3, and 1.3 g/cm^3, respectively. If there are more pores, voids, and interfacial cracks in a C/C composite, its bulk density will be lower. CPFO00-C and CPFO37-C had similar densities, suggesting that the surface chemical structural changes induced by oxy-fluorination were not sufficient to improve the impregnation of the carbon precursor, resulting in no significant increase in the bulk density of CPFO37-C as compared to that of CPFO00-C. CPFO55-C had a higher density than CPFO00-C and CPFO37-C, whereas CPFO73-C and CPFO100-C had a density even higher than that of CPFO55-C. It seems that by increasing the number of –OH, –COOH, and –F functional groups, the impregnation of the phenol carbon precursor improved due to the enhanced affinity of the treated carbon preform surface to phenol carbon precursor. Thus, the density of the C/C composite increased. Therefore, it is suggested that the phenol carbon precursor impregnation is enhanced depending on the oxy-fluorination condition.

As shown by SEM, CPFO73-C and CPFO100-C had fewer pores, voids, and interfacial cracks than the others. Therefore, the density data demonstrates that the observed SEM images are reliable. Due to the differences in the C/C composite microstructures along with their surface chemical structures, the mechanical and thermal

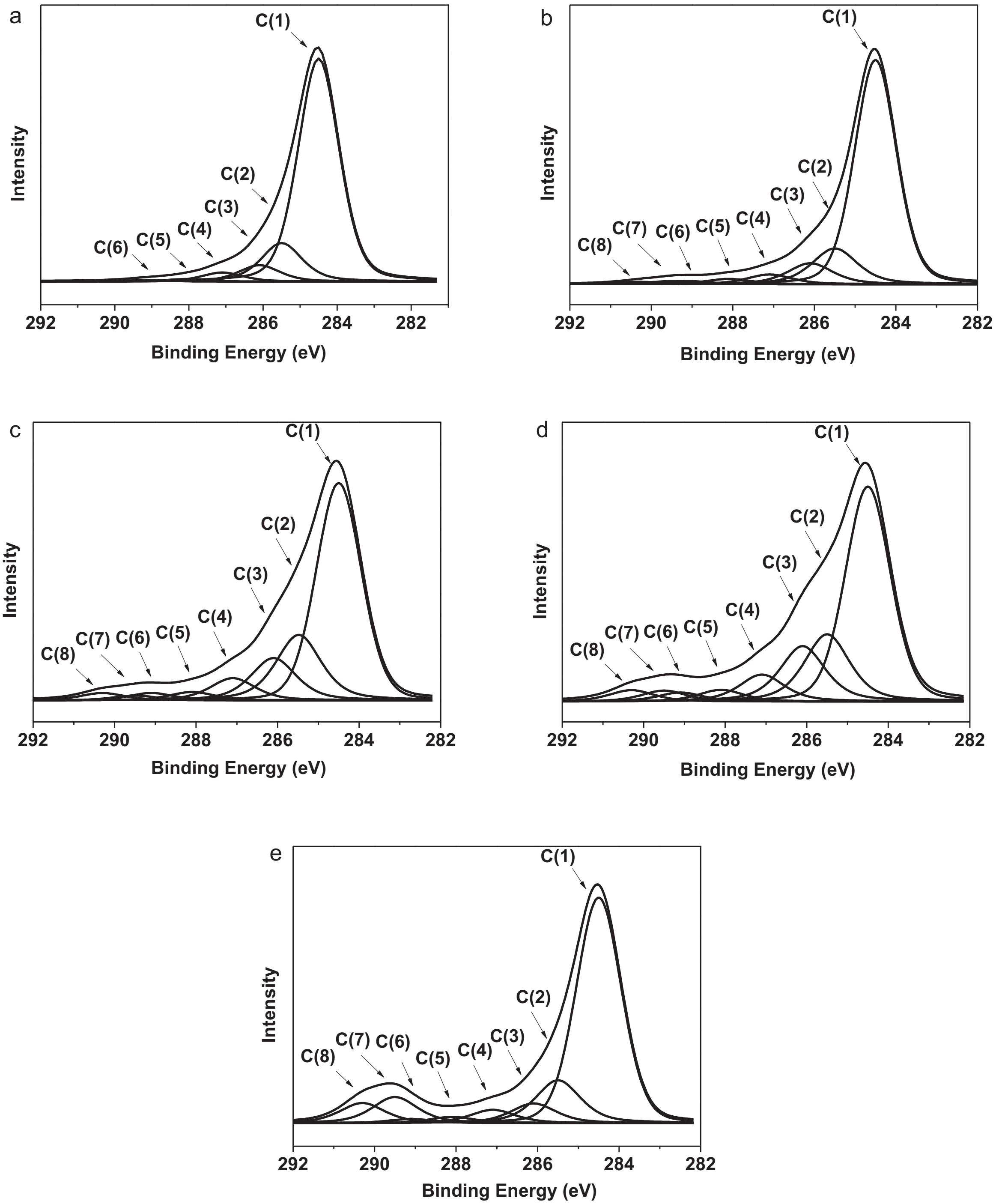

Fig. 2. C1s deconvolution of the an-received and oxy-fluorinated carbon preforms: (a) CPFO00, (b) CPFO37, (c) CPFO55, (d) CPFO73, and (e) CFPO100.

Table 2
C1s spectra and assignment of the oxy-fluorinated preforms.

Component	Binding energy (eV)	Assignment	Concentration (%) of each sample				
			CPFO00	CPFO37	CPFO55	CPFO73	CPFO100
C(1)	284.5	C–C (sp^2)	74.6	69.5	54.3	49.5	59.6
C(2)	285.5	C–C (sp^3)	14.3	13.6	18.9	17.5	13.1
C(3)	286.1	C–OH, C–O–C	6.1	8.0	12.2	14.7	6.0
C(4)	287.1	C=O	3.4	3.7	6.4	7.2	4.1
C(5)	288.1	COOR	0.9	2.0	2.4	3.1	1.9
C(6)	289.1	COOH	0.6	1.2	2.1	2.3	1.3
C(7)	289.5	Semi-covalent C–F	–	1.2	1.4	2.8	8.0
C(8)	290.3	Covalent C–F	–	0.9	2.1	2.9	6.1

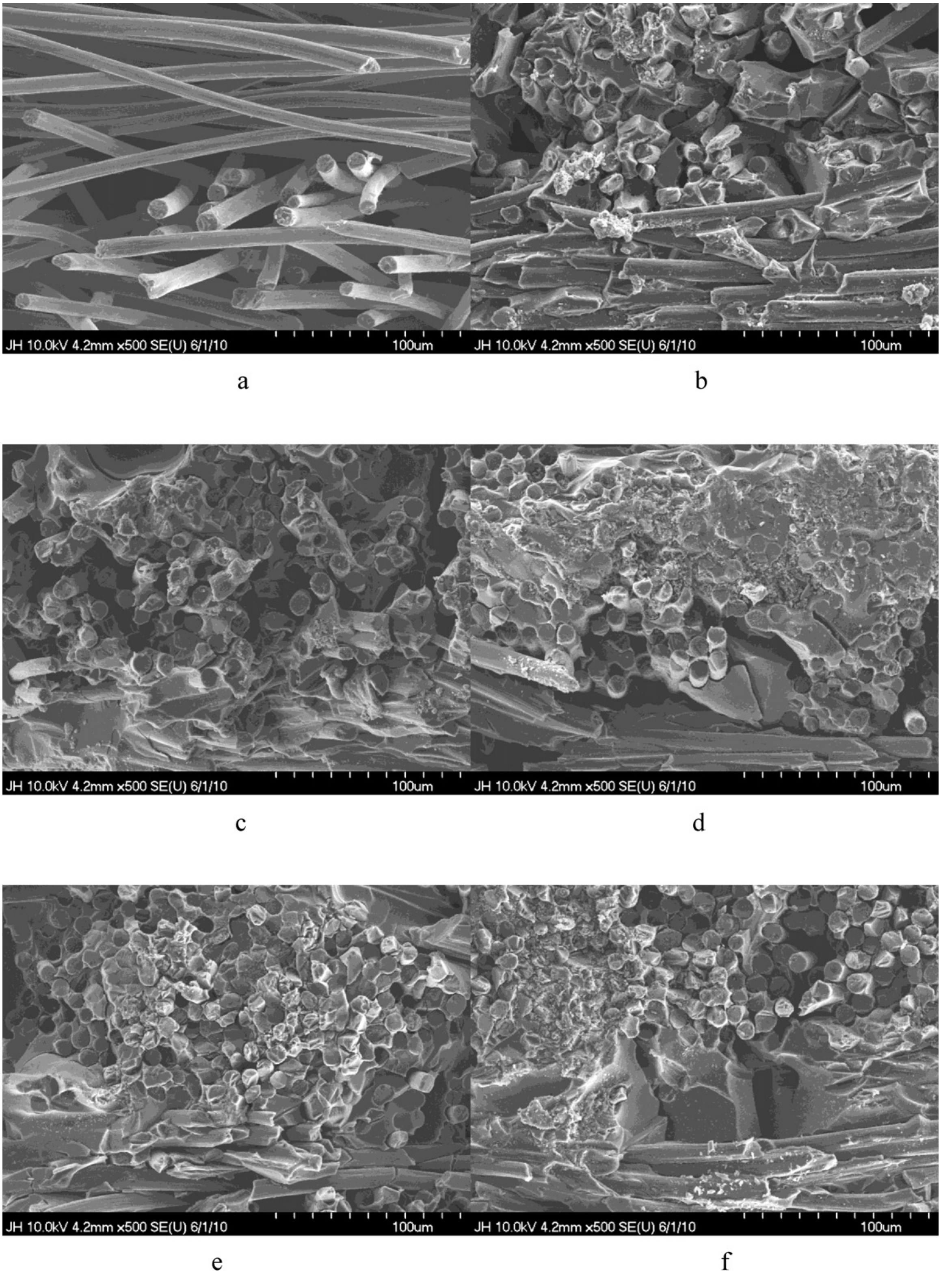

Fig. 3. Microstructures of the prepared C/C composites: (a) CPFO00 (as-received carbon preform), (b) CPFO00-C, (c) CPFO37-C, (d) CPFO55-C, (e) CPFO73-C, and (f) CFPO100-C.

properties of the prepared C/C composites could also be different. A detailed description of this is given in later sections.

2.3. Mechanical properties of the prepared C/C composites

The results of a flexural strength test on the prepared C/C composites are shown in Fig. 5. The flexural strength of the C/C composites increased as the F_2 to O_2 ratio of the mixture gas increased, when both F_2 and O_2 gas were used for oxy-fluorination. The flexural strength of CPFO73-C increased almost 150%, as compared to that of CPFO00-C. There was no significant change in the flexural strength of CPFO37-C as compared to that of CPFO00-C, even though CPFO37-C was prepared after oxy-fluorination of the carbon preform. It is also noteworthy that the flexural strength of

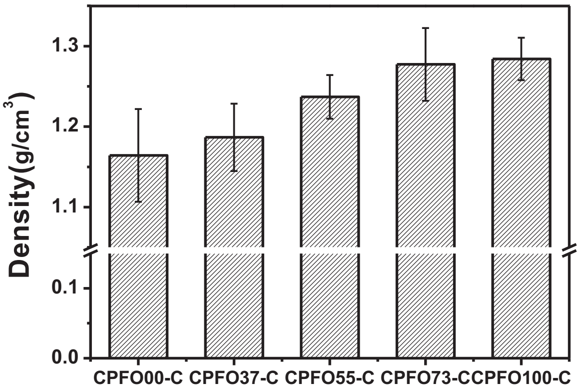

Fig. 4. Bulk densities of the prepared C/C composites.

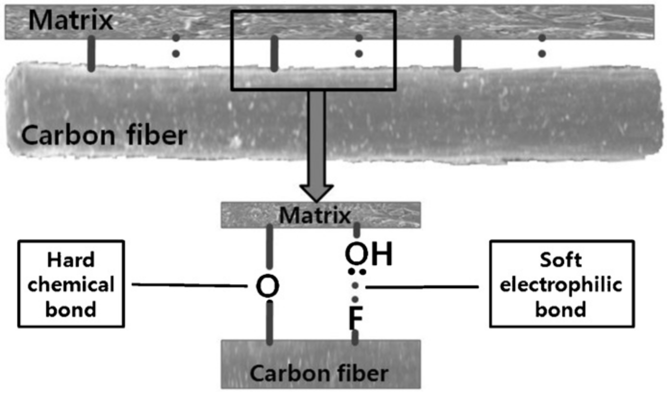

Fig. 6. . Suggested chemical bonds formed between oxy-fluorinated carbon preform and phenol carbon matrix precursor.

CPFO100-C increased by about 60% as compared to that of CPFO00-C. In regard to the surface chemical structures of the oxy-fluorinated carbon preform and microstructures of the prepared C/C composites, this difference in flexural strength of the prepared composites was attributed to the structural changes induced by oxy-fluorination. Based on the results of XPS analysis, it was found that CPFO73 had the most –OH, –COOH, and –F groups in total with the second most –F groups on the surface, whereas CPFO100 had the second most –OH, –COOH, and –F groups in total with the most –F groups on the surface. It was also found that CPFO37 had more –OH, –COOH, and –F groups than CPFO00, but had a similar flexural strength, compared to CPFO00-C. In addition, as mentioned earlier, based on the microstructures and bulk densities of the prepared composites, the interfacial adhesion between the carbon preform and carbon matrix was relatively strong in CPFO73-C, CPFO55-C, and CPFO100-C, but relatively weak in CPFO37-C and CPFO00-C. Therefore, it seems that the surface functional groups introduced by oxy-fluorination were not sufficient to increase the interfacial adhesion and improve the flexural strength of CPFO37-C. However, increased adhesion of CPFO55-C and CPFO 73-C was observed with increasing –OH, –COOH and –F content, resulting in significantly improved flexural strength of CPFO55-C and CPFO 73-C as compared to CPFO00-C. However, increased adhesion as compared to CPFO00-C and CPFO37-C and decreased adhesion as compared to CPFO55-C and CPFO73-C was observed in CPFO100-C with a significantly increased –F content, resulting in the flexural strength between CPFO00-C/CPFO37-C and CPFO73-C/CPFO100-C. These results can be interpreted by starting from the suggested bonds present in interface of carbon fiber and phenol carbon matrix precursor as shown in Fig. 6. The –OH groups in the form of –OH and –COOH were reported to react with free –OH groups of the phenol resin during curing. In addition, it has also been reported that –F functional groups can form an electrophilic bond with the lone pair electrons of O and N [23]. The surface –F groups introduced by oxy-fluorination of the carbon preform may also form this electrophilic bond with O in phenol resin. Hence, overall, after oxy-fluorination of the carbon preforms, hard chemical bonds and soft electrophilic bonds form at the interface between the carbon preform and the phenol carbon matrix precursor, increasing the interfacial adhesion. The hard chemical bonds formed (C–O–C) remained even after carbonization at 1000 °C [24–26]. Therefore, the chemical bonds formed at the interface increase interfacial adhesion between carbon fibers of the carbon preform and the phenol-based carbon matrix. However, there are two possible reaction pathways for the soft electrophilic bonds during carbonization. One is that C–F and HO–C of the soft electrophilic bonds react to produce more C–O–C bonds during carbonization and increase the interfacial adhesion. The other is that C–F bonds are broken by heat and produce no chemical bond. In this case, C–F bonds just work for improved impregnation of the phenol carbon matrix precursor due to the improved affinity of carbon fiber to the phenol, resulting in the densified C/C composites after carbonization. It is generally believed that the C–F bonds start to be broken above 300 °C [27]. And after C–F bond breakage, all of the two reactions can occur. If all the C–F bonds were involved in forming more C–O–C bonds, the flexural strength of CPFO100-C would be higher than CPFO55-C, because CPFO100 has more O- and F-related functional groups in total than CPFO55 and CPFO100-C has higher bulk density than CPFO55-C. If all the C–F bonds were just broken without forming any C–O–C bonds, interfacial cracks, originated from the contraction of the phenol during carbonization, would similar to those CPFO00-C. Therefore, it seems that not all of C–F bonds react with HO–C to form C–O–C and part of C–F bonds react to form C–O–C bonds in the interfacial area, maximizing the mechanical properties of the C/C composites. It is also important that too strong interfacial adhesion impairs the mechanical properties of composites, due to damage incurred during carbonization [7–10]. However, oxy-fluorination of the carbon preform resulted in hard chemical bonds and soft electrophilic bonds, thus achieving improved flexural strength. Hard chemical bonds between the carbon preform and the matrix precursors can withstand carbonization, whereas only part of soft electrophilic bonds withstand and the others are broken due to heat-induced defluorination during carbonization [21]. This suggests that the interfacial adhesion was strong but still flexible enough to not induce any damage during carbonization. The relatively unique change produced in the

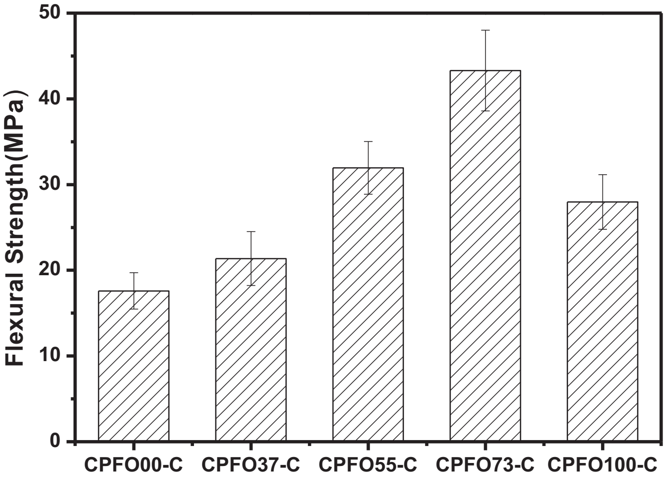

Fig. 5. Flexural strength of the C/C prepared composites.

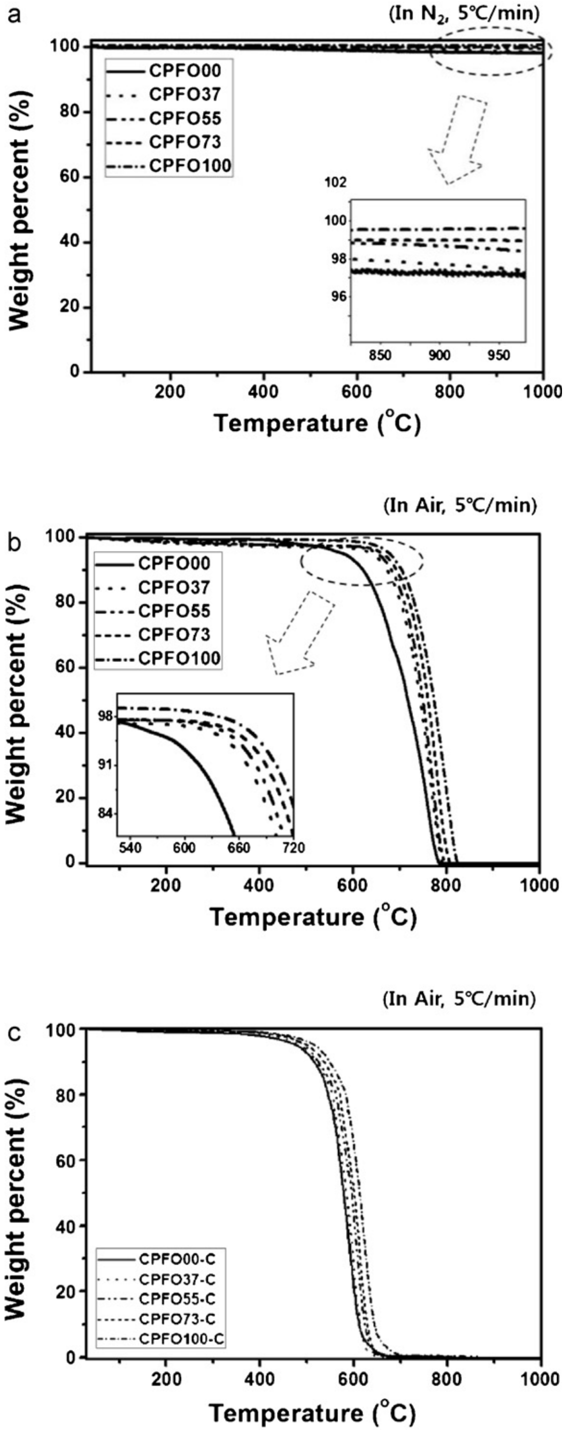

Fig. 7. TGA results of oxy-fluorinated preforms (a) under N_2 and (b) under air and the prepared C/C composites (c) under air.

flexural strength of CPFO100-C was attributed to the dominant soft electrophilic bonds as compared to the hard chemical bonds. In addition, the functional groups introduced by oxy-fluorination, such as –OH, –COOH, and –F, also increased the affinity of carbon preforms to phenol resin, promoting impregnation of the phenol resin to the carbon preforms as shown in bulk densities and SEM images of the prepared C/C composites. This also enhanced the interfacial adhesion between the carbon preform and carbon matrix.

2.4. Thermal properties of the oxy-fluorinated carbon preforms and the prepared C/C composites

Thermal property changes after oxy-fluorination of the carbon preforms were analyzed using TGA, and the results are shown in Fig. 7. Fig. 7(a) shows that the thermal stability of the preform improved very slightly in the order of CPFO00, CPFO37, CPFO55, CPFO73, and CPFO100. Fig. 7(b) shows that the anti-oxidation of the preform improved slightly in the order of CPFO00, CPFO37, CPFO55, CPFO73, and CPFO100. In both Fig. 7(a) and (b), CPFO00 and CPFO37 showed similar thermal behaviors with similar TGA curves. As we recall the XPS analysis results of the oxy-fluorinated preforms, changes in surface chemical compositions of CPFO37 was not significant compared to the others. CPFO55 and CPFO73 had higher oxygen concentrations on their surfaces than the others, whereas their fluorine concentrations were higher than in CPFO37 and CPFO00 and lower than in CPFO100. CPFO100 had much more fluorine on the surface, with an oxygen concentration similar to that of CPFO00 and CPFO37. As shown earlier in the XPS survey spectra of the preforms given in Section 2.1, the binding energies of C–F were higher than any of the others. These higher binding energies may contribute to the improved anti-oxidation of the oxy-fluorinated carbon preforms. In addition, the oxy-fluorination also has a surface etching effect, removing active sites (thermally less stable parts) on the surface [18,28]. Therefore, the slight improvement in thermal stability of the oxy-fluorinated carbon preform was observed by removing active sites on the surface which is weak to thermal decomposition.

Since there were only slight differences in the thermal stabilities of the oxy-fluorinated carbon preforms, only anti-oxidation of the prepared C/C composites was investigated. The thermal properties of the prepared C/C composites are shown in Fig. 7(c). As observed in the oxy-fluorinated carbon preforms, their thermal properties improved in the order of CPFO00-C, CPFO37-C, CPFO55-C, CPFO73-C, and CPFO100-C. These results can be attributed to the thermal properties of the base carbon preforms of the composites, because the order of the stability is the same to that of the carbon preforms. In addition, there are studies that have reported that the anti-oxidation of composites depends on the voids, pores, and interfacial cracks of the C/C composites [4,29–31]. The improved anti-oxidation could be attributed to improved adhesion, which results in fewer pores, voids, and interfacial cracks, because the strong interfacial adhesion prevents the diffusion of oxygen gas inward in the C/C composites. Therefore, the improved anti-oxidation of the prepared C/C composites seems to be due to the oxy-fluorination-induced synergistic effect of improved thermal properties of the carbon preform itself and interfacial adhesion between the carbon preform and the carbon matrix.

3. Conclusions

The oxy-fluorination of carbon preforms was shown to improve the thermal properties of the preforms by introducing surface functional groups. The introduced surface functional groups also increase the interfacial adhesion between the carbon preforms and the carbon matrix, resulting in improved mechanical properties of the C/C composites. The improved interfacial adhesion between the carbon preform and carbon precursor was achieved by hard chemical bonds between –OH and –COOH groups on the surface of the oxy-fluorinated preforms and free –OH groups of the phenol carbon matrix precursor as well as soft electrophilic bonds between –F groups on the surface of the oxy-fluorinated preforms and lone pair electrons of free –OH groups of the phenol carbon matrix precursor. This leads to improved adhesion between the carbon preform and carbon precursor after carbonization. The improved thermal properties of carbon preform itself and interfacial adhesion of the C/C composites were also improved by the oxy-fluorination-induced synergistic effect, which resulted in fewer voids, pores, and interfacial cracks of the C/C composites.

4. Materials and methods

4.1. Materials

Resol-type phenol-formaldehyde resin (HM2) was provided by the Kolon Chemical Cooperation. PAN-based needle-punched carbon preform was provided by the DACC Cooperation and dried at 120 °C for 24 h in oven before surface treatment to remove moisture in the preform.

4.2. C/C composite preparation

Oxy-fluorination treatment on the carbon performs was performed with different ratios (3:7, 5:5, 7:3, 10:0) of F_2:O_2 gas at 1 bar of total pressure and 25 °C for 20 min. The oxy-fluorinated samples were named as CPFO37, CPFO55, CPFO73, and CPFO100, corresponding to F_2:O_2 gas ratios and as-received preform was named as CPFO00. The detailed experimental procedure employed herein has been reported in a previous study by the authors' research group [32,33]. A needle-punched carbon preform was impregnated in the phenol resin at 0.2 bar for 10 min. The impregnated preform was heated to 130 °C, and the temperature was maintained for 5 h to cure the phenol resin. Then, the phenol-impregnated carbon preform was carbonized at 1000 °C for 2 h. The C/C composites prepared with the oxy-fluorinated preforms were termed as CPFO00-C, CPFO37-C, CPFO55-C, CPFO73-C, and CPFO100-C, corresponding to the carbon preforms used.

4.3. Sample characterization

X-ray photoelectron spectroscopy (XPS, Thermo Electron Cooperation, MultiLab 2000 Spectrometer, England) was carried out to investigate chemical structural changes in the carbon preform surfaces after oxy-fluorination. Al Kα (1485.6 eV) X-rays were used with a 14.9 keV anode voltage, a 4.6 A filament current, and a 20 mA emission current. All samples were treated at 10^{-12} bar to remove impurities. The survey spectra were obtained with a 50 eV pass energy and a 0.5 eV step size. Core level spectra were obtained at a 20 eV pass energy with a 0.05 eV step size. FWHM of each component was 1.3 eV, because it exhibited very well in the other studies about fluorination of carbon materials [34]. Scanning electron microscopy (SEM, JEOL, JSM-7000F, Japan) was used to investigate chemical structural and microstructural changes in the C/C composite surfaces. The bulk densities and flexural strength of the prepared C/C composites were measured using ASTM C20 and ASTM C1161, respectively. The ASTM C20 method recommends two replications by two different workers, but in this study, 20 replications were carried out. First, dry weight (D) was measured by placing the specimen in the oven until there was no weight change. Then, suspended weight (S) was measured after saturating the specimen with water by boiling for 6 h and the specimen was weighed in water. After wiping the surface water of the specimen, saturated weight (W) was measured. Finally, bulk density of the specimen was calculated using the equation, $D/(W-S)$, and the result was recorded by one decimal place. Thermogravimetric analysis (TGA, Mettler-Toledo, TGA/SDTA851, Switzerland) was performed to evaluate the thermal properties of the prepared samples with a heating rate of 5 °C/min.

Acknowledgments

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