THERMAL CHARACTERIZATION OF PHENOL FORMALDEHYDE/NEEDLE PUNCHED CARBON COMPOSITES BY OXY-FLUORINATION TREATMENT

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Introduction

Carbon fiber reinforced carbon (C/C) composites have high thermal conductivity, excellent thermal stability, low coefficient of thermal expansion, resistance to thermal shock and ablation, and low density, high strength-to-weight ratio [1-3]. These excellent properties enable C/C composites to be considered as potential materials for nozzles of solid rocket motor, heat shields for reentry vehicles, brake discs for advanced aircraft, and aerospace applications in leading edges and nose tips [4, 5]. When using C/C composites for these applications, increasing interfacial adhesion of a carbon precursor on carbon fiber surface in C/C composites is one of the methods to enhance thermal and mechanical properties of the composite.

In this study, oxy-fluorination treatment of a carbon preform was carried out to improve the interfacial adhesion between a carbon precursor and a carbon preform and the effect of the treatment condition on thermal and mechanical properties of C/C composites were investigated.

Experimental

Phenol resin was provided by Kolon Chemical (HM2). Needle-punched carbon perform was provided by DACC. F_2 gas with 99.8% purity and O_2 gas with 99.99% purity were used for oxy-fluorination treatment.

Oxy-fluorination treatment was performed with different rations (3:7, 5:5, 10:0) of F_2:O_2 gas mixtures at room temperature for 20 min.

A needle punched carbon preform was impregnated to the illite/phenol resin mixture at 0.2 bar for 10 min. The impregnated preform was heated to 130 $^{\circ}$C and the temperature was maintained for 5 h to cure phenol resin. Then, it was carbonized at 1000°C for 2h.

X-ray photoelectron spectroscopy (XPS) was carried out to investigate chemical structural changes of the C/C composite surfaces, depending on the F_2 to O_2 ratio for oxy-fluorination of a preform. Thermogravimetric analysis (TGA) was performed to evaluate thermal stability and antioxidation of the prepared samples with heating rate of 5 $^{\circ}$C/min.

Results and Discussion

To characterize chemical structures of the oxy-fluorinated surface, XPS analysis was carried out and the spectra were shown in Fig.1. As the F_2 to O_2 ratio for oxy-fluorination increased, F1s peak intensity increased, whereas, O1s peak intensity decreased. Ideally, when F_2:O_2 (10:0) gas was used, O1s peak should not appear. However, O1s peak appeared, because there was moisture absorbed in the preform itself and that moisture reacted with F_2 gas and then the preform surface, resulting in O1s peak on the surface.

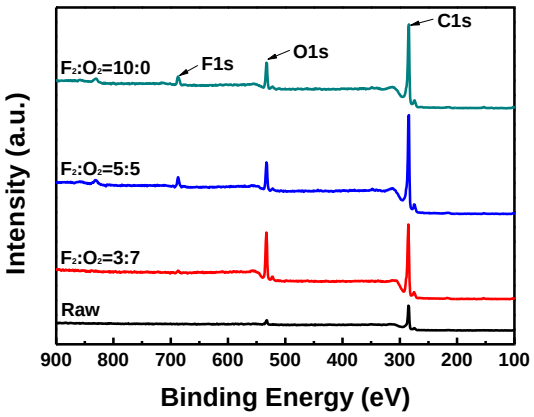
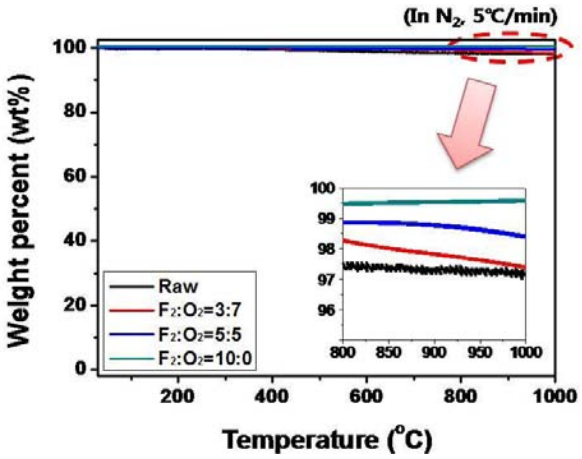


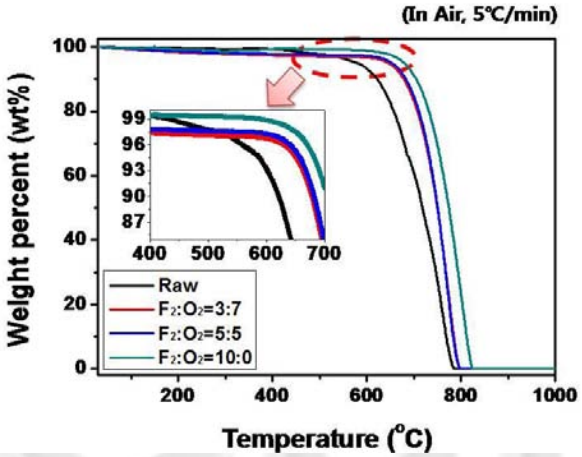
Fig. 1 XPS spectra of oxy-fluorinated performs with different different F_2:O_2 ratios.

Thermal property changes after oxy-fluorination treatment were analyzed using TGA and the results were shown in Figure 2. Fig. 2(a) showed that thermal stability of the preform was enhanced, as the F_2 to O_2 ratio for oxy-fluorination of a preform increased. Fig. 2(b) showed that anti-oxidation of the preform was enhanced, as the F_2 to O_2 ratio for oxy-fluorination of a preform increased. Especially, in both thermal stability and anti-oxidation, when the F_2 to O_2 ratio for oxy-fluorination was 10:0, the thermal properties were best. It could be suggested that the surface functional groups introduced onto the preform surface after oxy-fluorination, such as –OF, C-F, -COOH, contained oxygen in them [6], and prevented thermal decomposition and oxidation of the preform, working as a coating.

Thermal property changes of the C/C composites prepared after oxy-fluorination treatment of the preform were also analyzed using TGA and the results were shown in Figure 3. Unfortunately, it was not clear that anti-oxydation of the C/C composites was enhanced as the the F_2 to O_2 ratio for oxy-fluorination of a preform increased. However, it could be observed that anti-oxidation was obviously enhanced, when the F_2 to O_2 ratio for oxy-fluorination of a preform was 10:0. Therefore, it could be suggested that 10:0 was the best F_2 to O_2 ratio for oxy-fluorination of a preform to enhance thermal properties of both the preform itself and the C/C composite. It could be also suggested that due to the functional groups introduced after aforementioned oxy-fluorination, the interfacial adhesion between the preform and the carbon precursor increased, resulting in enhanced thermal properties.



(a)



(b)

Fig. 2 TGA results of oxy-fluorinated performs with different F_2:O_2 ratios (a) under N2 (b) under air.

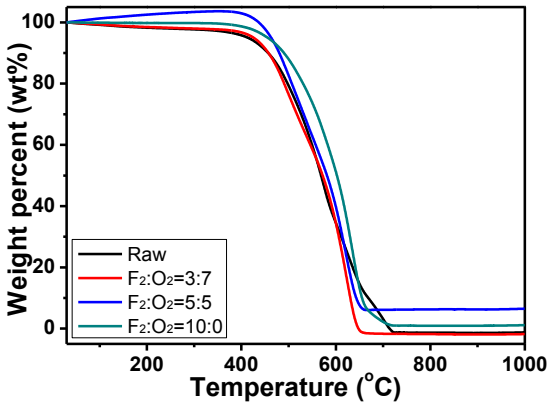


Fig. 3 TGA results of the C/C composites prepared with oxy-fluorinated performs with different F_2:O_2 ratios.

Conclusions

Oxy-fluorination treatment of the needle punched carbon preforms enhanced thermal stability and anti-oxidation of the preforms themselves. In addition, the use of oxy-fluorinated preform to prepare C/C composites improved thermal property of the composites. The improved thermal properties seemed to be caused by enhanced interfacial adhesion between a carbon precursor and a preform after oxy-fluorination treatment which introduced functional groups, such as –OF, C-F, -COOH, onto the preform surface.

Acknowledgment

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References

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