THE ANTI-OXIDATION CHARACTERIZATION OF ILLITE/PHENOL/CARBON COMPOSITES

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Introduction

Carbon/carbon composites (C/C composites) as advanced engineering materials for space vehicles and automobiles are required to have high mechanical properties [1-3]. However, C/C composites are oxidized to CO_2, CO under O_2 atmosphere over 450 ℃ and lose their high mechanical properties [4]. Therefore, preventing oxidation under high temperature is a key to produce C/C composites.

Illite is a clay material, which consists of more than 50% of clay materials on earth, having SiO_2 and Al_2O_3, stable at high temperature, as main components [5]. Hence, it is expected that using illite as a filler can improve antioxidation of C/C composites.

In this study, illite was mixed with phenolic resin and then, the mixture was impregnated to carbon preform. The preform was then heat-treated at different temperatures. The anti-oxidation of the C/C composites prepared was investigated using TGA and their structures were characterized using XRD.

Experimental

Phenol resin was provided by Kolon Chemical (HM2). Needle-punched carbon perform was provided by DACC. Illite was provided by Yonggoong illite cooperation (1000 mesh).

Illite (1, 3, 5, and 10 wt% to phenol resin) was added to phenol resin and stirred for 2h. Then, 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 °C and the temperature was maintained for 5 h to cure PHENOL resin. Then, it was carbonized at 1000°C for 2h or 1650 °C for 1 h.

X-ray diffraction (XRD) was carried out to investigate structural changes of the C/C composites, especially formation of SiC, depending on the carbonization temperature. Thermogravimetric analysis (TGA) was performed to evaluate antioxidation of the prepared samples with heating rate of 5 °C/min.

Results and Discussion

In addition to high thermal stability of illite itself, it contained a lot of SiO_2 as quartz impurities, which could form β-SiC when carbonized above 1600 ℃. Since it would be complicated to characterize structural changes of the C/C composites prepared with the reaction between the illite and a phenol resin precursor and a carbon fiber preform, the structural changes of illite/phenol resin precursors after carbonization at different temperatures were investigated instead.

XRD patterns of illite/ phenol precursor carbonized at 1000 °C or 1650 °C were shown in Figure 1. The XRD results showed that there was only one significant change which arose from SiO_2 of illite when illite/phenol resin precursors were carbonized at 1000 °C. On the other hand, when illite/ phenol resin precursors were carbonized at 1650 °C, new peaks were observed in the XRD patterns of the carbonized composites. Those new peaks at 35.68, 60.06, and 71.80 ° corresponded to (1 1 1), (2 2 0), and (3 1 1) peaks of β-SiC, respectively and these peak positions were in good agreement with other studies [6]. The formation of β-SiC was expected because there was SiO_2 present in illite itself and some of those SiO_2 could react to produce β-SiC during carbothermal reduction by virtue of the intimate contact to phenol resin in the precursor.

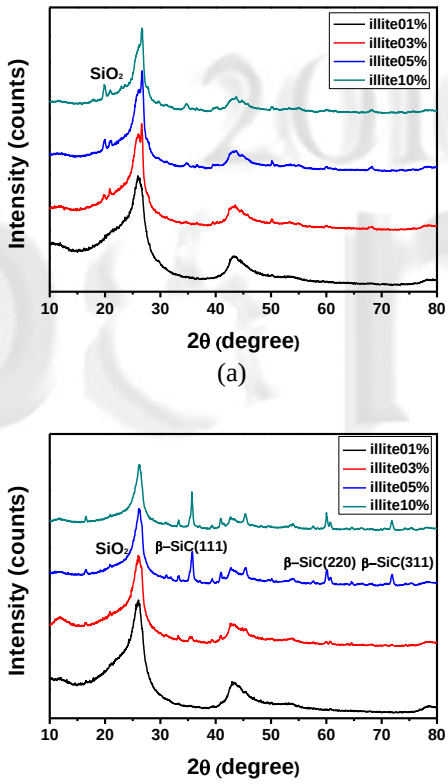
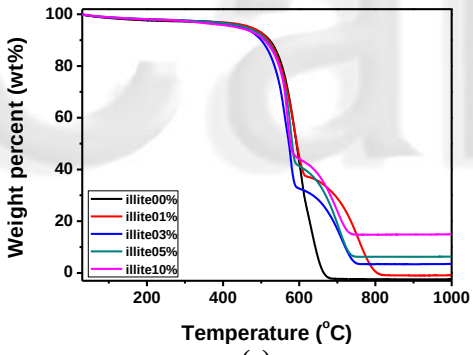


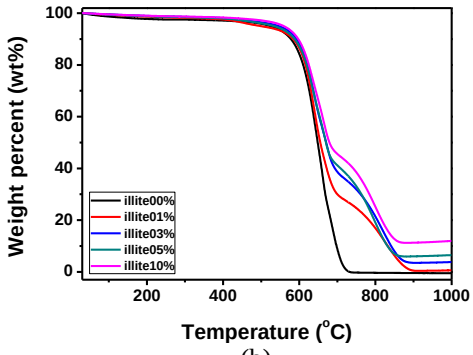
Fig. 1 XRD patterns of illite/phenol composites carbonized at (a) 1000 °C and (b) 1650 °C.

TGA results of the illite/phenol based C/C composites carbonized at 1000 °C or 1650 °C were shown in Figures 2. When the illite/phenol based C/C composites were prepared at 1000 °C, initial decomposition temperature (IDT) of those prepared C/C composites increased as the amount of illite added increased (Figure 2(a)). This could be supported by

IPDT (Integral Procedural Decomposition Temperature) calculations, which was one of the effective TGA data analysis methods [7], shown in Table 1. As shown in Table 1, IPDT of the prepared C/C composites increased as the illite content increased. IPDT of only phenol based C/C composite was only 463.8 °C, whereas that of 10% illite/phenol based C/C composite was 584.2 °C with 26 % increase. In the same way to the illite/phenol based C/C composites prepared at 1000 °C, when the illite/phenol based C/C composites were prepared at 1650 °C, IDT of the prepared C/C composites increased as the amount of illite added increased (Figure 2(b)) and IPDTs of the prepared C/C composites also increased (Table 1). In addition, the IPDTS of the C/C composites prepared at 1650 °C were higher than those of the C/C composites prepared at 1000 °C. Especially in 10% illite added carbon matrices, IPDT of the carbon matrices prepared at 1650 °C was 65.9 °C higher than that of the carbon matrices prepared at 1000 °C. All of these improvements in antioxidation could be explained by SiC formation at 1650 °C. As shown earlier, when the illite/phenol resin impregnated carbon preforms were carbonized at 1650 °C, SiC was formed as a result of the carbothermal reduction by virtue of the intimate contact of illite SiO_2 to phenol resin in the precursor and carbon preform. Therefore, it could be suggested that synergistic effect of illite with good antioxidation and SiC formed enhanced the anitoxidation of the illite/phenol based C/C composites prepared at 1650 °C more than that of the C/C composites prepared at 1000 °C.



(a)



(b)

Fig. 2 TGA results of the C/C composites with various illite contents prepared at (a) 1000 °C and (b) 1650 °C.

Table 1. IPDTs of Illite/phenol based C/C composites prepared at 1000 ℃ or 1650 ℃.

Prep. Temp. (°C)	Illite contents (%)	IPDT (°C)
1000	0	463.8
	1	483.2
	3	474.1
	5	516.5
	10	584.2
1650	0	555.4
	1	567.4
	3	593.2
	5	607.7
	10	650.1

Conclusions

Illite addition to phenol based C/C composites increased their anti-oxidation. In addition, depending on the temperature to prepare the composite, especially at 1650 °C, β-SiC was formed by carbothermal reduction of SiO_2 contained in illite and carbon materials.

Acknowledgment

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