

EFFECTS OF REACTION PARAMETERS ON THE PROPERTIES OF SiC/C FIBER COMPOSITES

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Introduction

Porous SiC/C fiber composites have been produced by reacting carbon fiber composites (CFC) of various BET areas with SiO vapor. Under certain reaction conditions the resultant SiC composites showed high BET areas useful for catalyst support. In this type of gas-solid reaction, important reaction parameters may be reaction temperature and time as well as specific surface areas of the CFC. This work summarizes the effects of such reaction parameters on the properties of SiC/C fiber composites with high BET area. Effects of incorporation of cerium nitrate into the activated CFC are also presented.

Experimental

Disc shaped samples, 20 mm in diameter and 10 mm thick, and 35-1150 m²/g in BET area were obtained by activating CFC consisting of carbonized phenolic resin bounded PAN based carbon fibers with flowing CO for various time. Some of the samples were impregnated with cerium nitrate solution to investigate effects on the properties of SiC/C fiber composites. The activated CFC samples were reacted with SiO vapor to convert carbon to SiC at temperatures 1200-1500 °C for 10-120 minutes. The compositions and BET areas of the obtained SiC/C fiber composites were analyzed by wet chemical method and multi-point BET method [1].

Results and Discussion

The chemical analysis results for the SiC/C fiber composites after conversion process are shown in Tables 1-2 and Figs. 1-2. They indicate that both BET area of the CFC and reaction temperature are the primary reaction parameters for the conversion of carbon to silicon carbide. Addition more than of 7wt% cerium to the activated CFC decreased the SiC content of the converted sample as shown in Fig. 2. X-ray diffraction patterns of the converted samples loaded with cerium nitrate revealed amorphous phase and many intermediate crystalline phases composed of CeN, Ce₁₀(SiO_{3.61}N_{0.39})₆O_{1.83}, Ce₅Si₃O₁₂N at temperature below 1400 °C. These intermediate phases seems to hinder conversion of carbon to SiC.

In Tables 1-2 and Figs. 3-4, the BET results for the SiC/C fiber composites after conversion process are presented. Fig.3 shows the effect of reaction time on the BET area of the resultant SiC/C fiber composites. These results are surprising since it was expected that a decrease of unreacted carbon with high BET area decreased the BET area of the converted composites. It is attributed to the amorphous silica condensed to CFC, which can seal micropores of CFC effectively. As the SiO-C reaction proceeded, the amorphous silica would be removed by carbothermal reduction with CO. Addition of cerium nitrate into the carbon increased the BET area of converted samples as known in literature. Up to addition of 7wt% cerium, an increase of the BET areas with increasing Ce doping resulted from the reduction of SiC crystallite sizes due to the intermediate compounds of Ce, Si, O and N. On the contrary, high BET areas of SiC/C fiber composites obtained by reacting 10wt% Ce-doped CFC with SiO vapor resulted from unreacted carbon with high BET area as shown in Fig. 4.

Conclusions

Both specific surface area of carbon fiber and reaction temperatures are the primary reaction parameters for the production of high surface area SiC fiber composites. It has been found that an excess generation of SiO vapor during conversion process can hinder the reaction of carbon with SiO vapor. Addition of cerium nitrate increased the specific surface area of converted carbon fiber composites due to the intermediate compounds of Ce, N, O and Si.

References

1. Lee JC, Park MJ, Choi YJ, Lee JS, Hong MS, Park SH. Properties of silicon carbide fiber composites prepared from activated carbon fiber composites. Extend abstracts, International symposium on Carbon, Tokyo, The Carbon Society of Japan, 1998:578-579.

Acknowledgments

Financial support from the Ministry of Environment, Project G-7, is gratefully acknowledged.

Table 1. Compositions and BET areas of converted CFC without Ce (1400 °C, 2hr).

BET area before conversion (m ² /g)	Composition (wt%)			BET area after conversion (m ² /g)
	SiC	Silica	carbon	
35	27	10	63	6.73
497	57	18	25	21.2
692	71	15	14	40.6
1150	90	7	3	48.2

Table 2. Compositions and BET area of converted CFC without Ce at various reaction temperatures.

Temperature (°C)	Composition (wt%)			BET area after conversion (m ² /g)
	SiC	silica	carbon	
1200	12	52	36	405
1300	68	10	23	50.6
1400	90	7	3	48.2
1500	100	0	0	8.7

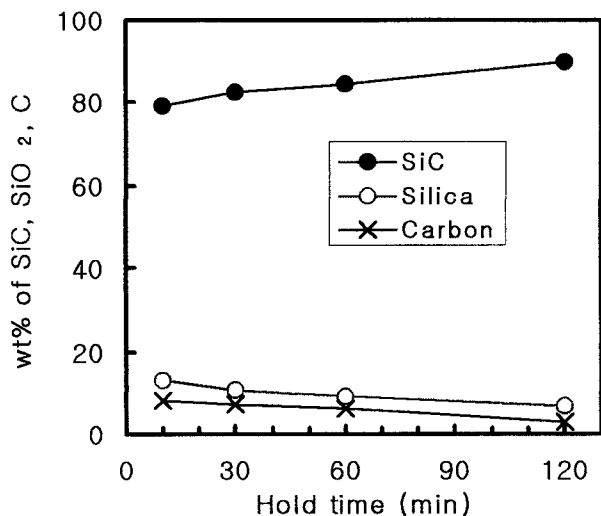


Fig. 1. SiC, C, and SiO₂ contents in the converted CFC without Ce as a function of hold time.

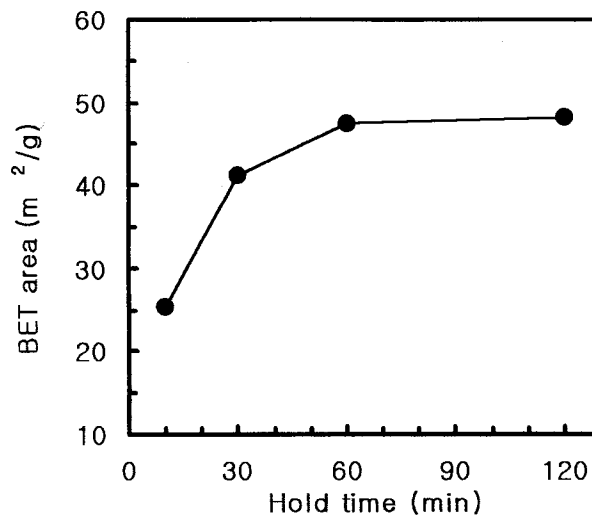


Fig. 3. BET area of converted CFC without Ce as a function of hold time.

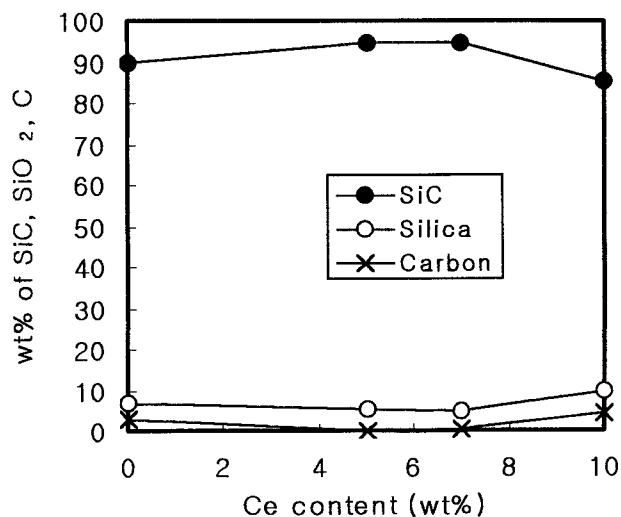


Fig. 2. SiC, C, and SiO₂ contents in the converted CFC as a function of Ce content (1400 °C, 2hr).

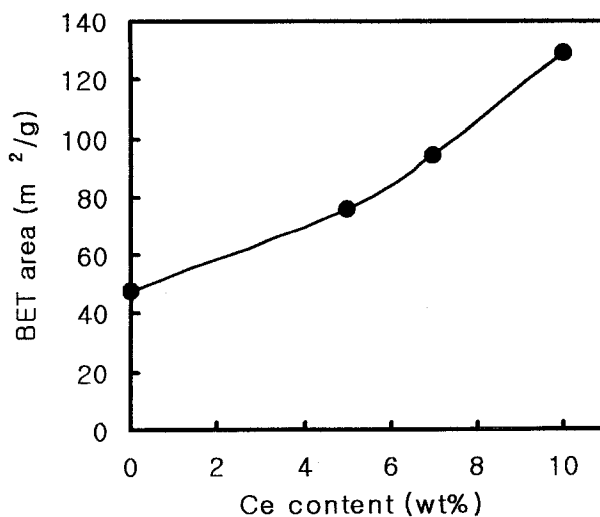


Fig. 4. BET area of converted CFC as a function of Ce content (1400 °C, 2hr).