

ON THE IMPROVEMENT OF IMPREGNATION EFFICIENCY IN THE FABRICATION OF CARBON-CARBON COMPOSITES

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INTRODUCTION

In the fabrication of carbon-carbon composite materials, a liquid-phase impregnation method has been widely used to fill the pores in the matrix formed by repetitive carbonization processes and thus to increase the density of the composite. This is ascribed to easy handleability, efficiency and some economic reasons of the method in comparison with a vapor-phase impregnation method[1]. The repetitive re-carbonization/impregnation processes to produce high density carbon-carbon composite may take a long duration of several months[2]. During the impregnation steps between the carbonization processes, one may bring up a problem that parts of the impregnated resin flow backward from the pores in the composite to the surface of the composite as the applied pressure or vacuum is released to atmosphere for curing of the impregnant. It may cause the decrease in the impregnation efficiency, which is dependent of the selected impregnation method. Such a problem may be more pronounced in the first carbonized material containing most macro- and micropores inside of the composite.

Consequently, the aim of the present work is to improve the impregnation efficiency in the early stage of the fabrication of carbon-carbon composite, especially after the first carbonization step. Here, the effects of different liquid-phase impregnation techniques and the impregnant added with graphite powders on the impregnation efficiency are discussed.

EXPERIMENTAL

PAN-based carbon fabrics with 12K filaments and 8 H/S texture were used as a reinforcement for carbon-carbon composites. Resol-type phenolic resin with a solid content of 60% was used as a precursor for carbon matrix. The cured resin has a carbon yield of about 70% at 1100°C in N₂. Furfuryl alcohols in the absence and the presence of synthetic graphite powders of 1~2 μm size heat-treated at 2000°C were used as impregnants. The amount of the graphite powder addition was varied from 0 to 35 wt% during the impregnation work. The carbonization and re-carbonization were performed in N₂ up to 1000°C with 20°C/h, isothermally holding for 2 h at the final temperature. The specimens used in methods A, B and C were additionally heat-treated at 2000°C.

In the present study, four different liquid-phase impregnation methods were used to densify the carbon-

ized composites as follows. 1) Ultrasonic technique (Method A): an ultrasonic bath was used. 2) Instantaneous pressure/heat technique (Method B): a pressure vessel used was designed to be upside down to separate the impregnated composite from the partially cured impregnant. 3) Vacuum technique (Method C): a vacuum oven was used without control of temperature. 4) Instantaneous vacuum/heat technique (Method D): a vacuum oven was used with control of temperature and the partially cured impregnant remaining on the surface of the composite was carefully removed by cutting. Each specimen was prepared to be 12mm×12mm×17mm in size. Two impregnation/cure steps were done before re-carbonization. All the specimens after individual performance of the impregnation were completely cured after each impregnation step. The changes in the weight, bulk density, apparent porosity and impregnation efficiency of the composite according to each processing step and graphite powder content were investigated and compared among four different impregnation methods.

RESULTS AND DISCUSSION

In the impregnation methods used here, the methods A and C were experimentally easy to be exposed to atmosphere, but the methods B and D were not. In the figures G/B, 1C/C, 2C/C and 3C/C stand for carbon-phenolic greenbody, the first, second and third carbonized composites, respectively. 1IM and 2IM stand for the first and second impregnated composites after each carbonization step, respectively. Fig.1 shows the weight change at each processing step for four different impregnation methods. Both in the absence and the presence of 10% graphite powders in furfuryl alcohol, the method B exhibits the largest increase in weight by both the 1IM and 2IM in comparison with any other method. The next is followed by the method D. The impregnation efficiency, which is defined as {(weight of re-carbonized composite - weight of first carbonized composite)/ weight of first carbonized composite}, as a function of graphite powder content is representatively shown for the 2C/C in Fig.2. In all the methods here, the impregnation efficiency is not significantly dependent of the content of graphite powder with exception of the 10% addition in the methods B and D. This indicates that the applied pressure or vacuum without returning to atmosphere helps the impregnant with a smaller amount of graphite powders infiltrate into

the pores in the carbonized composite. Fig.3 compares the impregnation efficiency after each carbonization step among the methods. The improvement of impregnation efficiency is also greater in the methods B and D than in the methods A and C. Fig.4 shows the change in the bulk density and the apparent porosity of the composites at each processing step. The increments in the density and the porosity at each step by the individual methods with reference to those of greenbody are tabulated in Table 1. It may be concluded from the present study that the impregnation efficiency to densify carbon-carbon composite is significantly dependent of the impregnation methods. The addition of fine graphite powders to furfuryl alcohol as a modified impregnant dose not influence strongly the efficiency but a small amount of about 10% may increase the value somewhat, especially in the methods B and D.

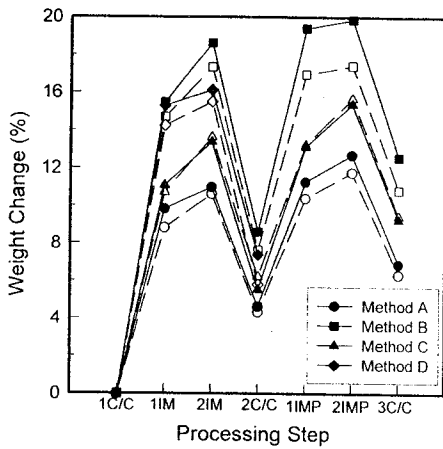


Fig.1 Weight change at each processing step for four different impregnation methods. (In Fig.1 and Fig 3 the broken line indicates the absence of graphite powders and the solid line the presence of 10% graphite powders in furfuryl alcohol.)

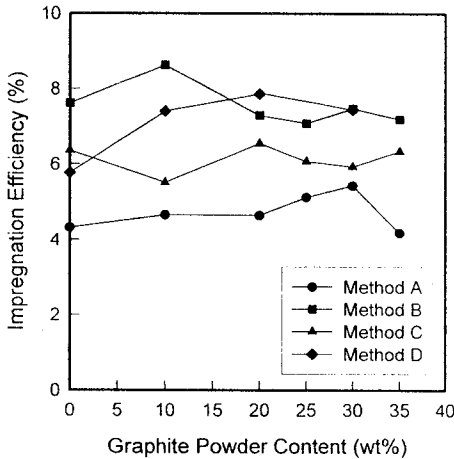


Fig.2 Impregnation efficiency as a function of the content of graphite powders examined for the second carbonized composite (2C/C).

ACKNOWLEDGMENT

This research has been performed with a financial support from the Korea Science and Engineering Foundation (Grand No: 961-0805-037-1).

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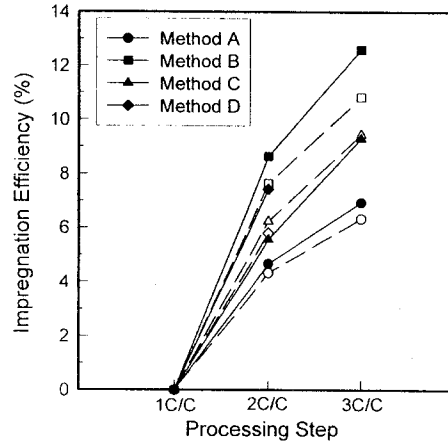


Fig.3 A comparison of impregnation efficiency after each carbonization step.

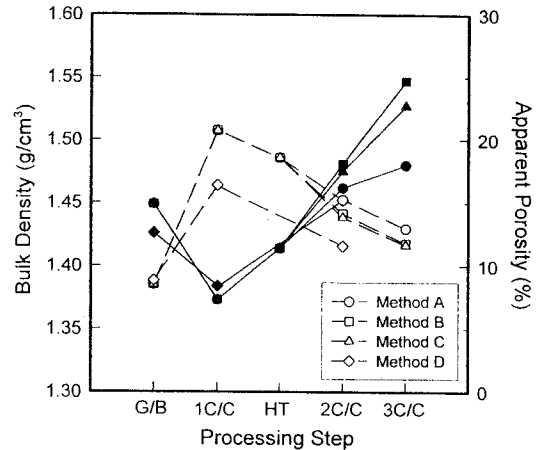


Fig.4 Variations of bulk density (solid line) and apparent porosity (broken line) of the composites at each processing step.

Table 1. Percent changes of the bulk density ($\Delta\rho$) and the apparent porosity (ΔP) shown in Fig.4.

	$\Delta\rho$ (%)				ΔP (%)			
	A	B	C	D	A	B	C	D
G/B	0	0	0	0	0	0	0	0
1C/C	-5.24	-5.24	-5.24	-2.95	143	143	143	85
HT	-2.42	-2.42	-2.42	-	118	118	118	-
2C/C	0.90 (1.31)	2.21 (2.55)	1.79 (1.59)	1.82 (2.88)	79 (79)	66 (64)	63 (66)	31 (23)
3C/C	2.14 (2.55)	6.76 (6.69)	5.38 (5.38)	-	52 (58)	38 (39)	37 (31)	-

The data in the parenthesis indicate the case of the addition of 10wt% graphite powder.