2D CARBON/CARBON COMPOSITES PRODUCED WITH MESOPHASE PITCH USING HIGH PRESSURE

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Introduction

Carbon/Carbon composites (C/C composites) have received considerable interest as a material for aerospace applications due to their high heat ablation, small thermal expansion, and good strength retention at high temperature, as well as high thermal shock resistance. Porosity of the C/C composites is due to the density changes that occurs during transformation of the matrix precursor into matrix carbon[1]. Thus, the low carbon yields and density changes lead to a significant pore volume after carbonization. For this reason, the mesophase pitch is good candidate materials to produce dense carbon matrix. However, the main problem in using the mesophase as the matrix precursor is the high viscosity, which may limit the complete impregnation of the fiber preform in the vacuum[1]. This study is concerned with the production of C/C composites with polyaromatic mesophase as matrix precursor.

Experimental

The coal-tar pitch manufactured from the Jung Woo Coal Chemical Co. in Korea was used. Under N_2 gas flow of 800 cc/min, two hundred grams of pitch were heated to 250 ℃ with a heating rate of 2 ℃/min, and then for hold 2 hours to remove the lower molecular weight compounds. The pitch was continuously heated to 430 ℃ with a heating rate of 3 ℃/min and was also held 2 hours. The mesophase pitch prepared was followed by measuring the softening point, coke yield at 1000 ℃ and amount of anisotropy. The softening point was measured using Mettler FP-80. The coke yield was measured under nitrogen at ambient pressure using TGA (Dupont 951). A temperature ramp rate for TGA was 10 ℃/min.. The resultant mesophases were carbonized at 1000 ℃ to form a coke in the nitrogen atmosphere. The powder diffraction of XRD (Shimadzu, XD-D1) was used to analyze of the change of interplanar distance.

Disk shaped two dimensionally reinforced composites were prepared by the hand lay-up technique, thermal press solidification and then, carbonization. The composites had to be carbonized (1st carbonization) in a steel cage to prevent swelling. The carbonized composites were impregnated by vacuum with molten mesophase pitch. The composites were pressed in HIP included excess mesophase pitch with 970 atm at 700 ℃. Fig. 1 shows the profile of HIP process. After the HIP treatment, C/C composites were characterized by density, porosity, optical microscopy and XRD to monitor the influence of high pressure and temperature.

Results and Discussion

Fig 2 show the spherical type mesophase after the heat treatment. The mesophase pitch consists of isotropic and anisotopic pitch. The amount of anisotropic was about 40-50 wt.% in the pitch. Table 1 compares the physical properties of as-received coal-tar pitch (JWCTPAR) and mesophase pitch. The mesophase pitch has shown higher softening point and coke yield than JWCTPAR. This means that the mesophase pitch was composed higher weight molecular fraction than JWCTPAR. Dimension and physical properties of 1st carbonized C/C composites were shown in Table 2. The density and porosity of 1st carbonized C/C composites were 1.1 g/cm3 and 38%, respectively. After HIP process, the density was increased upto 39% and also, the porosity was reduced upto 54%. This result mentioned the mesophase which has exhibited the full property to impregnate for the C/C composite in spite of high viscosity.

Conclusions

The mesophase pitch consists of isotropic pitch with 40-50 wt.% of mesophase. The mesophase pitch exhibit softening point of 175 ℃ and cokes yield of 58.1% at 1000 ℃, and it is easily infiltrated into 1st carbonized C/C composites with 970 atm at 700 ℃. Although further studies are necessary, the matrix precursor used in this study looks promising.

References

1. K. Christ and K. J. Huttinger, Carbon 31, P. 731 (1993).
2. V. Liedtke and K.J. Huttinger, Carbon '95, 22nd Biennial Conference on Carbon, P. 66 (1995).

Acknowledgment

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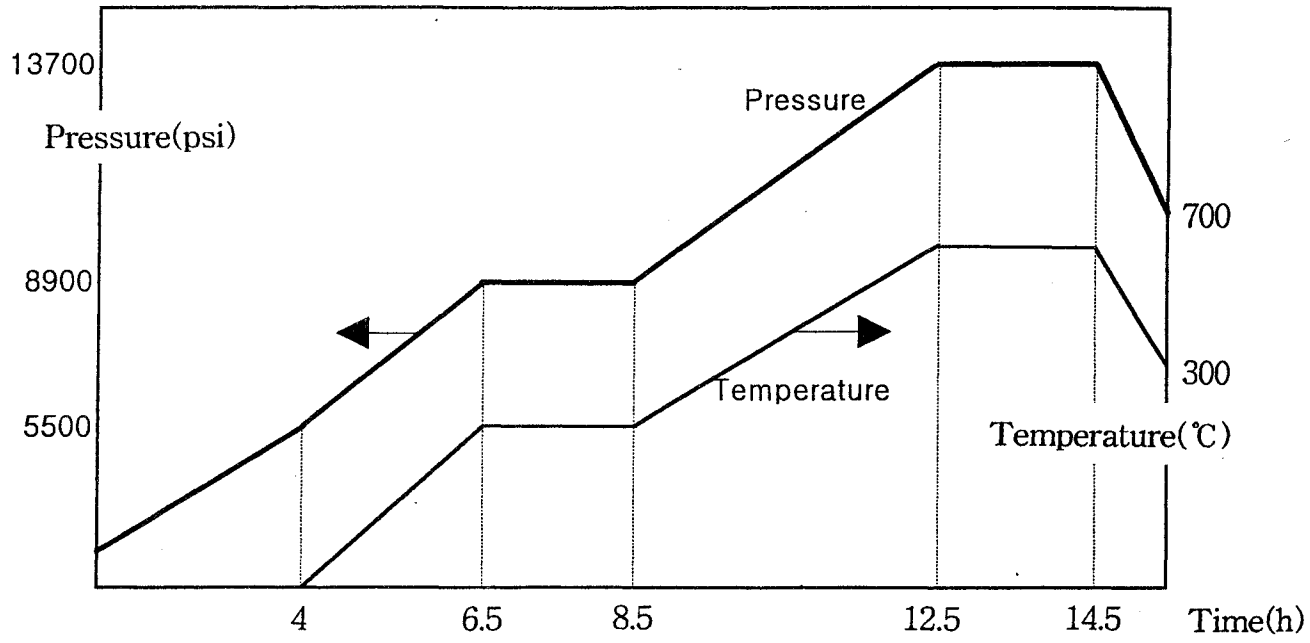

Fig. 1. Profile of HIP process

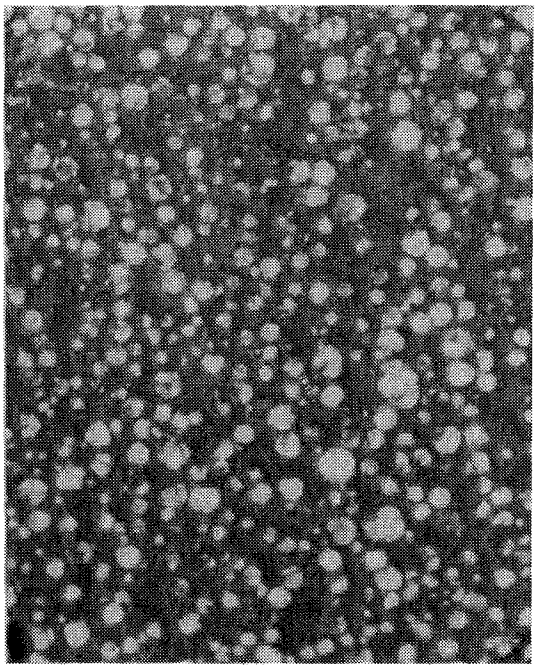

Fig. 2. Anisotropy after the heat treatment

Table 1. Characterization of Mesophase Pitch.

	JWCTPAR	Mesophase
Softening Point (℃)	116	175
Cokes yield (%)	42.8	58.1
$d_{(002)}$ (Å)	3.581	3.483

Table 2. Dimension and Physical Properties of 1st Carbonized C/C composites

	Before HIP treatment
Φ_L (mm)	80.1
Φ_S (mm)	6.0
Thickness (mm)	21.2
Density (g/cm3)	1.12
Porosity (%)	38

Table 3. Dimension and Properties of HIPed C/C Composites.

	After HIP Treatment
Density (g/cm3)	1.56
Porosity (%)	17.3
Interplanar Distance, d002 (Å)	3.370