REACTION OF NAPTHALENE MESOPHASE PITCH WITH P-BENZOQUINONE AND ITS CARBONIZATION

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INTRODUCTION

Importance of carbon-carbon composite has been recognized in various industrial field. The complex procedure and hence the high cost for the high performance should be overcome for their broad application. The required properties of the matrix are low thermosetting temperature (lower than 250 °C), low enough viscosity to be processed, high cross-link density, high carbon yield after carbonization.

In the present study, the feasibility of the naphthalene mesophase pitch (NP) to be used as thermosetting resin was investigated on the basis of the fact of high reactivity of the pitch with p-benzoquinone (BQ) at relatively low temperature 150 °C (1). Investigations were performed to understand thermosetting behavior of NP/BQ and carbonization behavior of the reaction products. The behaviors were examined through the observations of swelling behavior, carbon yield and the texture under a polarized light microscope when the reaction conditions were varied. The reaction conditions chosen were BQ/NP mixed powder, using DMSO as a solvent and dispersion of the NP/BQ in THF.

EXPERIMENTAL

Naphthalene mesophase pitch (Mitsubishi Gas Chemical Co. Inc., softening point, 273 °C) was used. The NP/BQ was mixed by powder and dispersing in THF. Reactions were performed at various ratios of NP/BQ at 180 °C for 1 hour stirring at 400 rpm. Some reactions were performed in DMSO solvent to avoid heterogeneous reactions. The products were washed with methanol and then heat treated at 300 °C for 30 min to remove unreacted BQ in the product.

Ca. 2 g of samples were carbonized in the glass tubes of 2.7 cm diameter (ID) at 600 °C for one hour at the heating rate of 2 °C. Further carbonization was performed at 800 and 1000 °C also at the same heating rate as the previous carbonization. And then, some carbonized samples were graphitized at 2,500 °C. The carbonization yield was calculated by taking ratios between the weight of the sample after carbonization and original weight.

The carbonization behaviors were investigated through the observations of elemental analysis, optical texture, X-ray diffraction, FT-IR spectra.

RESULTS AND DISCUSSION

When the reaction product was carbonized, the swelling height of the sample showed maximum at 10/1 mixture and decreased drastically to 10/8 weight ratio (Fig.1). At 10/10 weight ratio, the reaction product remained as powder with no indication of fusion even after carbonization at 600 °C. Relatively good dimensional stability was observed from the disk molded from NP/BQ (10/10) mixture after carbonization at 600 °C though the BQ concentration is relatively high. The texture of the carbonized samples was changed from the anisotropic fluid to mosaic and to isotropic with a increase in the concentration of BQ.

It was verified by data of the X-ray diffraction (Fig. 2) that the stacks of the pitch molecules delaminated through the reactions with BQ and remained in the carbonization process.

The carbonization yield increased from 87 % (NP) to 92% (10/4). From the elemental analysis, most of oxygen was removed below 600 °C.

REFERENCE

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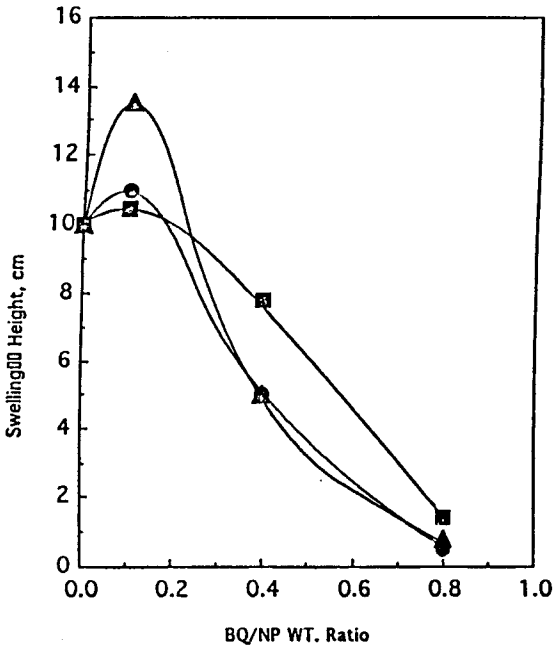

Fig. 1. Swelling height dependence on the BQ/NP weight ratio : heat treated at 300°C; carbonized at 600°C. ●, powder mixing; ■, DMSO solvent; ▲,THF dispersion.

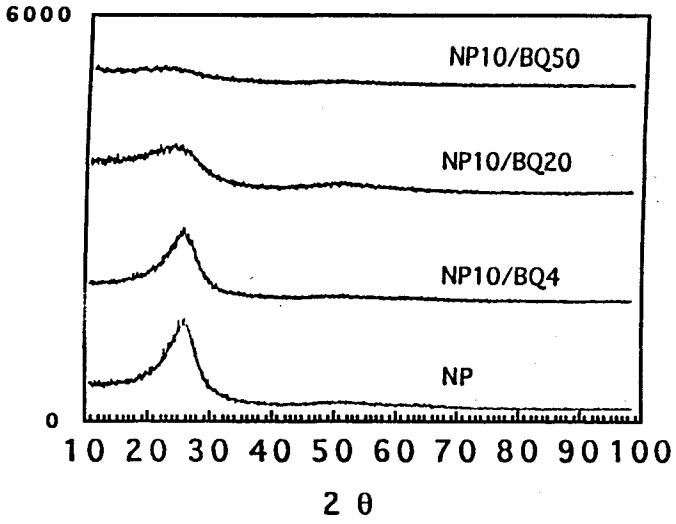

Fig. 2. X-ray difractions of the carbonized samples at various BQ weight ratio ; carbonized at 600 °C.