The Preparation and Characterization of Isotropic Carbon Fiber from Chemically Modified Coal-tar Pitch

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Introduction

Air blowing has been applied in preparations of isotropic precursors with high enough softening point. Mochida et al[1-3] obtained coal tar pitch precursor exhibiting softening point of 280℃ (originally from 82℃) and excellent spinnability, containing 1% oxygen through air-blowing. The air-blowing resulted increases in molecular sizes through dealkyration, dehyration and aromatization.

Yang et al [4] reported that a coal tar pitch (CP) was reactively polymerized with p-benzoquinone in the Diels-Alder mechanism at about 150℃, resulting an incresae in molecular size and that lead to suppressing a development of anisotropy.

Experimental

A coal tar pitch(CP; Jung-u Coal Co. Korea; softening point, 85℃) polymerized with 10%wt. benzoquinone(BQ) at 131℃ and successive heat-treatment under nitrogen flow at 380℃ for 3 hours. Such a modified pitch raised the softening of the pitch from 85℃ to 271℃ at the yield of 40%. It was spun through a circular nozzle (D=0.2mm, L=0.4mm) at 285℃ and at 10Kgf/cm^2. The pitch fibers were stabilized stepwise in an oven by convection of air at temperatures of 236℃ and 312℃ for 3 hours each and followed by carbonization at 1000℃(5℃/min) and graphitization at 2400℃(10℃ /min). X-ray diffraction (Rigaku, Japan) was also used to characterize the morphological structures. The tensile properties were measured by Instron(4200 series, full scale load range; 0.250kgf) with a single filament standard method(KS K 0327), gauge length of 25mm, a stretching rate of 0.5mm/min. 15 test average was taken for the results. The surfaces of the samples were investigated by SEM(JSM 5400, Japan).

Results and Discussion

The elemental analysis data in Table 1 shows variations of oxygen content in the stabilized pitch fiber. As the stabilization time and temperature increased, the content of oxygen in pitch fibers increased, and 13.5% oxygen was obtained through the stabilization.

X-ray diffraction data of the fibers with isotropic appearance are shown in Fig. 1. The $d_{(002)}$ values of carbon and graphite fibers were large as 3.86 and 3.49Å and their $Lc_{(002)}$ values were small as 11.24 and 25Å, respectively.

SEM microphotographs of carbon and graphite fiber with diameters of 17㎛ and 15㎛ are shown in Fig. 2. No structural significance of the carbon and graphite fibers was shown.

The tensile properties of the fibers are tabulated in Table 2. Tensile strength of carbon and graphite fibers was equally 570 MPa, and their Young's modules was 39.4 and 43.1 GPa, respectively, which are large enough as a precursor for ACF preparation.

Conclusions

The isotropic carbon and graphite fiber which have good physical properties was prepared, i.e.; tensile strength of 570 MPa, Young's modulus of 39.4 and 43.1GPa and low orientation values of 56.2 and 58.1% respectively.

Reference

1) Maeda, T., Zeng, S. M., Tokumitsu, K., Mondori,

J., and Mochida, I., *Carbon,* 1993, **31**, 407
2) ibid., *Carbon,* 1993, **31**, 421
3) Yang, K. S., Kim, C., Choi, J. H. Kumagai, H., and Sanada, Y., in Carbon '95 (Ext. Abstr. 22nd Biennial Conf. Carbon), San Diego, CA,1995, PP 212-213

Table 1. Elemental analysis of samples during a stabilization.

Temp.(℃)/ Time(hrs)	control	236 / 3	312 / 0	312 / 1.5	312 / 3
C	92.7	89.6	87.7	84.5	82.1
H	3.9	3.4	3.0	2.8	2.6
N	1.1	1.1	1.1	1.2	1.4
S	0.4	0.4	0.5	0.4	0.3
O	1.9	5.5	7.7	11.0	13.6

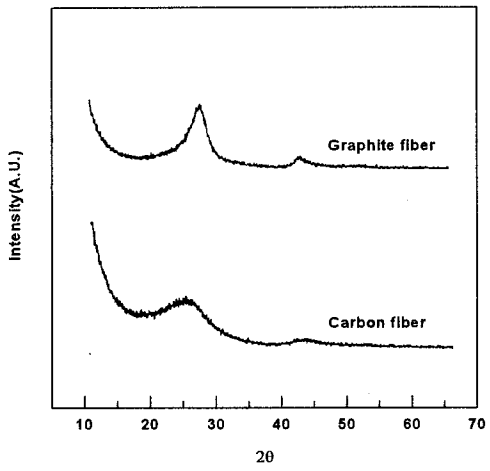

Fig. 1. X-ray diffraction curves of the carbon and graphite fiber.

Table 2. Properties of carbon and graphite fiber.

	Diameter (㎛)	$d_{(002)}$ [1] (Å)	$Lc_{(002)}$ [2] (Å)	DO [3] (%)	TS [4] (MPa)	YM [5] (GPa)	UE [6] (%)
Carbon Fiber	**17**	**3.86**	**11.24**	**56.2**	**567**	**39.4**	**1.44**
Graphite Fiber	**15**	**3.49**	**25**	**58.1**	**567**	**43.1**	**1.32**

(1) $d_{(002)}$ spacing, (2) Apparent crystallite stack height
(3) Degree of preferred orientation, (4) Tensile strength
(5) Young's modulus, (6) Ultimate elongation

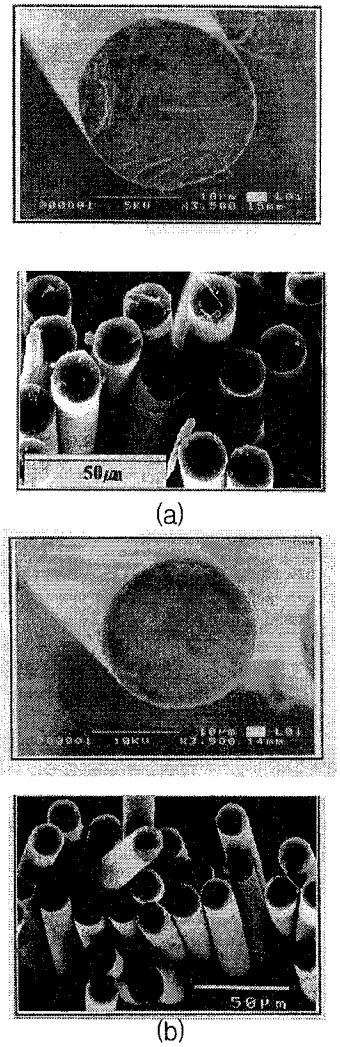

Fig. 2. SEM microphotographs of (a) carbon(1000℃) and (b) graphite fibers(2400℃).