PREPARATION OF MESOPHASE PITCH-BASED CARBON FIBER FROM FCC-DO

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1. Introduction

The key for the preparation of mesophase(MP) pitch is how to remove non-mesogen component to be large enough molecules sustaining the mobility of the mesogens anisotropic property avoiding excess polymerization with non-thermoplasticity. Because the sources of the pitches consist of a large variety of species with various reactivities, condensation of the species with low reactivitiy inevitably leads to the excess condensation of the more reactive species producing infusible solids[1, 2].

In this study, biphasic(BP) pitch precursor with appropriate softening point was prepared from FCC-DO through condensation reaction in the presence of Br_2. The BP precursor pitch was melt-spun smoothly, stabilized, carbonized. The morphological structure and mechanical properties of the fibers were examined and explained on the basis of analytical results of the sample from the preparation process.

2. Experimental

The FCC-DO(SKC Co. Korea) was oligomerized in the presence Br_2 (Junsei Chemical Co. Ltd., first-grade) at a feeding rate of 0.1 *ml*/min in a reactor for 2 hours at 110 . A subsquent thermal ageing was performed for 9 hours at 420 stirring at 600 ± 50 rpm. Precursor pitch was melt-spun by using pressurized nitrogen through a mono-hole spinneret (L/D = 1, D = 0.3mm) at 335 and 1 kgf/cm^2. The as-spun fibers were oxidatively stabilized in air at 310 for 2 hours at a heating rate 2 min^{-1}. The stabilized fibers were carbonized at 700, 1000 and 1200 for 1 hour under Ar atmosphere at a heating rate of 5 /min. The fiber samples were fractured in liquid nitrogen and loaded on graphite paste to examine fracture surface by using SEM (Hitachi, S-4700, Japan). The crystalline sizes(Lc002) and interlayer spaces of carbon planes(d_{002}) of all fibers were calculated on the basis data of Brown Ladner and Bragg equation respectively from X-ray diffraction (Dmax 1200, Rigaku; CuK radiation, 40 Kv and 30 mA). Tensile strength of 2.5 cm sample was loaded and measured by using lab-scale tensile tester (Nano Technics Co, Korea) with load cell of 150g at a cross head speed of 2.5 mm/min and the average data were taken from the at least 5 tests for each sample according to JIS R 7601 method.

3. Results and discussion

Table 1. summarizes some properties of BP precursor pitches. The pitch was characterized of softening point of 279 , MP content of 55 vol.%. Fig. 1. shows the X-ray parametes calculated XRD curves. The stack height Lc(002) was increased by spinning, minimum at 700 and increased again from that temperature in the carbonization process. The interlayer spacing (d_{002}) was behaved in a opposite direction. Fig. 2. shows the tensile strengthes of as-spun fiber, stabilized fiber, and carbonized fibers at 700 , 1000 , and 1200 . The tensile strengths increased with an increase heat treatment temperature. The highest value was 30 kgf/mm^2 at carbonized temperature at 1200 . Fig. 3. shows the fracture surface of cross-sectional area of the carbon fiber showing remained BP structure.

References

[1] Marsh H, Martinez-Escandell M, Rodriguez-Reinso F, Carbon 1999;37(3): 363-390.

[2] Mochida I, Korai Y, Ku CH, Watanabe F, Sakai Y, Carbon 2000;38:305-328.

Table 1. Some properties of mesophase precursor pitches.

Sample ID	SP[a] (°C)	Yield (wt.%)	AC[b] (vol.%)	Solubilities(wt.%)			
				HI	BS	BI-PS	PI[c]
F420-9	297	22	55	97.6	16.3	5.9	77.8

[a]Softening point measured by mettler, [b]Anisotropic contents, [c]HI. Hexane insolubles: BS. Benzene solubles: BI-PS. Benzene insolubles and pyridine solubles: PI. Pyridine insolubles.

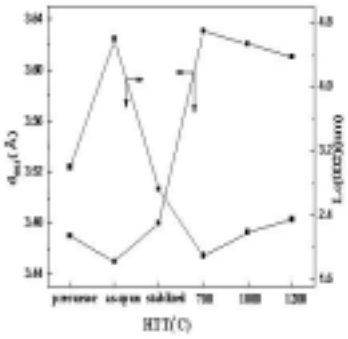

Fig.1. The X-ray parameters calculated from XRD curves.

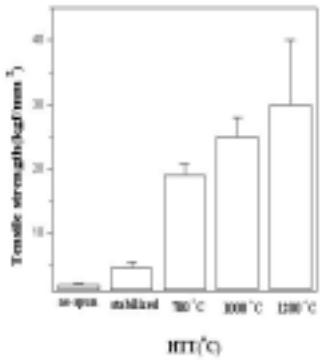

Fig. 2. The tensile strengthes of different conditions.

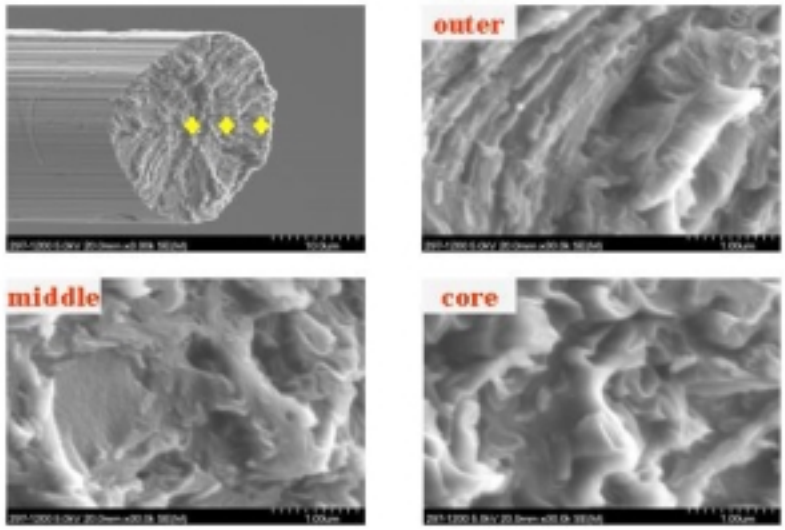

Fig. 3. SEM microphotographs of the carbonized fiber at 1200 °C.