STRUCTURAL AND PROPERTIES OF HIGH COMPRESSIVE CARBON FIBER FROM HEAT-TREATED ISOTROPIC PITCHES

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1.INTRODUCTION

Mesophase pitch based carbon fiber is still strongly expected to obtain a high compressive strength for its broads applications. For this purpose, its transverse texture is to be designed by a high degree of orientation along fiber axis. The present authors have reported that an isotropic pitch containing mesophase spheres prepared from a synthetic naphthalene pitch was spun smoothly into a thin fiber and was stabilized, carbonized and graphitized into a carbon fiber of optical anisotropy.

In the present study, heat-treated isotropic pitches with various mesophase content (0 to 100%) were prepared from the synthetic naphthalene and methyl naphthalene isotropic pitch and were spun, stabilized, carbonized and graphitized into graphitized fiber to design their structure for better performance.

2.EXPERIMENTAL

Synthetic isotropic pitches were prepared from naphthalene with aid of HF/BF_3 as a catalyst by Mitsubishi Gas Chemical. The properties were summarized at Table1. Isotropic pitches were heat-treated at 300-360 for 15-72h under bubbling of nitrogen to develop mesophase in the matrix.

Heat-treated pitches were spun into fiber at 280-320°C. The spinning extrusion rate was all fixed at 50mg/min. The pitch fibers were stabilized at 270°C for 60min, heating rate of 0.5°C /min. These fibers were carbonized and graphitized under argon atmosphere at 2500°C.

The pitch and fiber were observed by optical and high resolution scanning electron microscope (HR-SEM). The mechanical properties of graphitized fibers were measured.

3.RESULTS AND DISCUSSION

Fig.1 shows the microphotographs of pitches heat-treated for 15 to 72h. Mesophase developed according to the heat-treatment time. There was no visible mesophase portions in heat-treated pitch for 15h by OM observation. Mesophase spheres appeared when the pitch was heat-treated longer than 24h. Bulk mesophase in the whole pitch heat-treated the heat-treated for 72h. Fig.2 shows the transverse texture of graphitized fibers from heat-treated pitch and mesophase pitch. The size of micro units in fibers were very different. Microdomain in the fiber from heat-treated pitch grew according to the heat-treatment time. And that of mesophase pitch-based fiber was very smaller and curved. The structure of fibers may be concerned with the microdomains in each pitches.

The degree of preferred orientation increased according to the heat-treatment time (Table1). The graphitized fiber from heat-treated pitch for 15h, which contains no mesophase portions had much higher orientation comparing with isotropic pitch-based fiber. The graphitized fiber from heat-treated pitch for 72h showed almost same value to mesophase pitch-based fiber. Thus, graphitized fibers from

heat-treated pitch had high degree of preferred orientation regardless of the heat-treatment time. These facts may indicate that longer heat-treatment made the molecules in isotropic pitch larger one which was easy to be graphite. Fig3 shows the relation of compressive strength and Young's modulus of heat-treated pitch-based fibers. Compressive strength decreased with the heat-treatment time because of higher mesophased content. However, compressive strength of graphitized fiber from heat-treated pitch was much higher those that of typical pitch-based fiber. Especially, the compressive strength of the fiber from heat-treated pitch for 72h, which has high value of Young's modulus, was almost two times as strong as that of typical pitch-based fiber. These higher compressive strength may be concerned with the size and shape of microdomains.

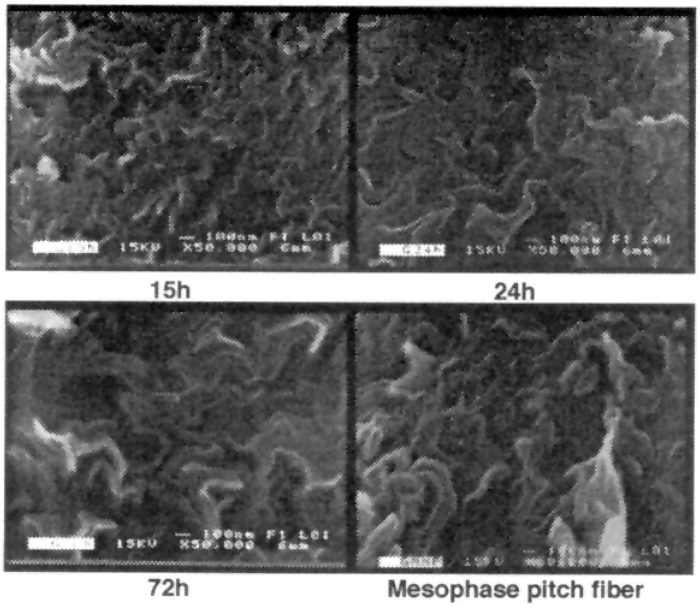

Fig. 1 OM photographs of the heat-treated pitches

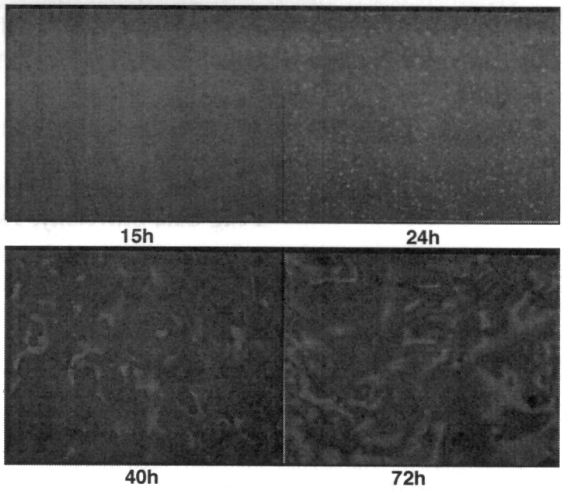

Fig. 2 Transverse textures of graphitized fibers from heat-treated pitches at 360°C

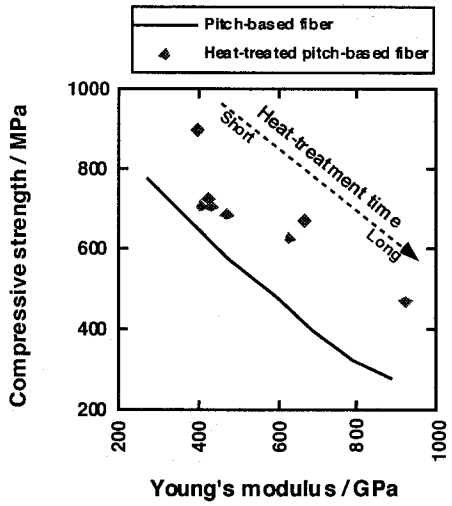

Fig. 3 Compressive strength and Young's modulus of heat-treated pitch-based fiber

Table1 The degree of preferred orientation of fibers

Heat-treated time/h	Degree of preferred orientation/%	
	as-spun	graphitized
Isotropic pitch	56.7	62.5
15	63.5	90.9
18	64.2	91.8
24	65.6	92.6
30	69.2	93.1
40	69.4	95.1
72	72.2	95.3
Mesophase pitch	78.0	95.5

Heat-treatment temperature is 360°C
Graphitization temperature is 2500°C