Preparation of Meso-Carbon Microbeads(MCBs) from AR Isotropic Pitches

Seong-Ho Yoon, Sumihito Ishida, Yong-Gang Wang, Yozo Korai, and Isao Mochida
Institute of Advanced Material Study, Kyushu University

1. INTRODUCTION

Meso-carbon microbeads(MCBs) have been known as an excellent raw materials for high density carbon materials and super active carbons. MCB has been conventionally prepared by solvent extraction of pitches containing anisotropic spheres. However, the low yield of MCB in the conventional manufacture has not been satisfactory and is still a major factor for high price.

In this paper, the present authors prepared the special meso-carbon microbeads(MCBs) by long-time and low-temperature heat treatment of naphthalene derived AR isotropic pitches. Effects of compositions in the isotropic pitches on the formation and homogeneity of MCBs were examined. The formation mechanism of ansiotropic spheres in the carbonization of pitch was discussed based on the microstructure of obtained MCB.

2. EXPERIMENTAL

Some properties of AR isotropic pitches used in this study were summarized in **Table 1**. AR isotropic pitches were prepared from naphthalene with aid of HF/BF_3 as a çatalyst by Mitsubishi Gas Chemical Co.

Isotropic pitches were heat treated for several hours with mild agitation under nitrogen atmosphere. Heat treated pitches were extracted with pyridine by conventional soxhlet extraction. Obtained MCBs were rinsed with toluene and dried under vacuum.

Heat treated pitches were observed using polarized optical microscope(Olympus, B2). MCBs were also observed using scanning electron microscope(JEOL, JSM5400) for determining their size distribution.

3. RESULTS

Fig. 1 shows the polarized optical micrographs of heat treated pitches at 380 C for 4 and 6 hs. Heat treated pitch from EP-2 pitch contained more anisotropic contents and better homogeneity than those from EP-1 pitch by the same heat treatment.

Table 2 summarized the carbonization and PI yields, anisotropic contents, and H/C atomic ratio of carbonized pitches. EP-1 showed over 30% anisotropic contents after the heat treatment at 370 - 380 C for 20 hs. EP-2 showed 30 - 40% ansiotropic contents only 6hs after the same temperature heat treatment. PI fractions of heat treated pitches were around one half of the anisotropic contents in the heat treated pitches. Atomic ratios of H/C of the heat treated pitches were ca. 0.60 which was certainly larger than those of pitches from petroleum and coal tar pitches, reflecting high naphthenic contents of AR pitches.

Fig. 2 shows SEM photographs of the extracted PI. The MCBs exhibited very special assembling structure of smaller anisotropic units. Enlarged SEM images clarified that such a assembling structure was formed by channeling and micelling of basic anisotropic units which was considered as a back-bone unit with the domain of liquid crystal mesophase pitch. Special shaped MCB from AR pitch showed larger specific surface area of ca. 10 m^2/g, which is induced from the channeling shapes.

4. DISCUSSION

Special channeling shaped meso-carbon microbeads(MCBs) were successfully prepared by low temperature and long time heat treatment of AR isotropic pitches. EP-2 pitch containing more AI-BS fraction showed more ansiotropic contents and well distributed homogeneous MCBs than EP-1 under same heating conditions, clarifying AI - BS fractions being very influential for forming anisotropic spheres. Channeling shaped MCB is considered to be introduced by low insoluble yields of PI's, and also to ascribe larger contents of naphthenic contents and high dissolving property of AR pitches.

From these results, it can be clarified that liquid crystal mesophase pitch consists of the assembling of basic domain units and such domain units are formed by the assemble structure of mesogen molecules.

Table 1 Some properties of AR isotropic pitches derived from naphthalene with aid of HF/BF3

Designation	Softening point(°C)	Anisotropic contents(vol%)	Solvent solubilities(wt%)					H/C
			HS	HI-AS	AI-BS	BI-PS	PI	
EP-1	165	0	5	26	36	30	3	0.69
EP-2	169	0	7	17	48	26	2	0.72

Table 2 Carbonization and PI yields of heat treated AR pitches

Raw pitch	Heat treatment conditions Temp.(℃)	Time(h)	Carbonization yield(wt%)	Anisotropic contents(vol%)	H/C of pitches	PI yield(wt%)
	360	10	90	7	0.66	4
	360	20	87	9	0.65	4
	370	20	86	16	0.63	7
	375	20	83	34	0.62	18
EP-1	380	10	85	11	0.64	6
	380	20	79	42	0.62	15
	385	15	82	37	0.61	19
	390	10	85	22	0.61	14
	400	4	85	50	0.60	23
	420	2	75	-	0.58	5
	380	4	92	18	0.58	9
EP-2	380	6	88	30	0.59	14
	400	2	-	70	-	-

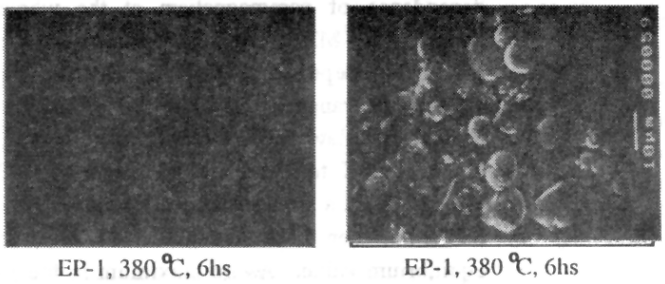

Fig. 1 Polarized optical micrographs and SEM photographs of Heat treated pitches and extracted PI's

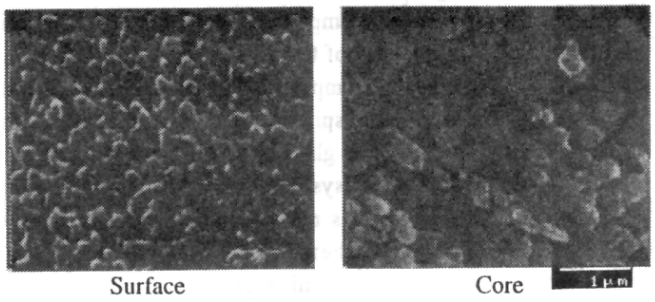

Fig. 2 Enlarged SEM images of prepared MCB