MICRO-STRUCTURAL STUDY OF COMMERCIAL NEEDLE COKES

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1. INTRODUCTION

The properties of needle coke(NC) are intimately related to its macroscopic as well as microscopic structures, which are defined by its origin and heat treatment conditions[1]. Generally, macroscopic structures contain the anisotropic textures and the macro-pores and cracks, which are determined by the observation of low magnification, using polarized optical microscopy. In contrast, microscopic structures contain the micro-pores and cracks as well as molecular stackings of needle coke. Such microstructures containing nano-scale structures are not still fully clarified[1].

In this study, the present authors examined the microstructures of needle cokes and their changes by heat treatment. Three commercial needle cokes(NCs) were characterized using X-ray diffractometer, polarized optical microscope(OM), scanning electron microscope(SEM), and high resolution scanning electron microscope(FE-SEM).

2. EXPERIMENTAL

Three commercial needle cokes supplied by Japan Energy Co. and their heat treated ones at various temperatures(1000 - 3000℃) were used.

Various sections of as received and heat treated needle cokes were observed by OM, SEM, FE-SEM. X-ray diffraction of needle cokes was carried out according to the method defined by Gakushin method(JSPS), using a X-ray diffractometer(Rigaku Geigerflex; CuK α, 0.15406 nm, 40 kV, 30 mA).

3. RESULTS

Figure 1 shows the OM, SEM, and FE-SEM images of the heat treated NC-D at 3000℃. In the OM and SEM images, the isochromatic regions were well matched with the alignments of domain units. Macro-cracks developed by shrinkage of the assemble units of domains were observed. In the magnified SEM image, the domain units which were composed of micro-domains were clearly observed. Boundaries of domains were sectioned by needle shaped micro-cracks which were developed by the shrinkage of domain units. In the most magnified FE-SEM image, the micro-domain unit which has a the thickness of ca. 3 - 5 nm, were observed. Such micro-domains were assembled to form a domain unit of ca. 300nm thick.

Table 1 summarizes some properties of needle cokes used in this study. NC-D showed the best thermal expansion coefficient among three. **Figures 2** and **3** show the changes of d_{002} and Lc(002) of heat treated needle cokes. NC-E and NC-F exhibited almost same values of d_{002}, while those of NC-D showed a little larger values. Lc(002) of NC-D and NC-F were the same values until the heat treatment of 2500℃. After further heat treatment to 3000℃, NC-D showed larger value than that of NC-F. NC-E always showed larger values of Lc(002) than those of NC-D and NC-F. The graphitizability of three needle cokes increased in the order of NC-F, NC-D, and NC-E.

4. DISCUSSION

Microscopy and X-ray refractometry of commercial needle cokes were carried out for clarifying their micro-structures and relations to properties. The micro-domains of 3-5 nm thick which were observed as the assembly of the carbon planes formed the domains of ca. 300 nm thick as basic units of needle cokes. The alignment of domains determined the size and the direction of the isochromatic regions.

Micro-domains observed in this study is considered to be basic graphitic units in the needle cokes. The thickness of micro-domain is defined as Lc(002) in the X-ray deffractometry. The thickness of micro-domains of various needle cokes were much smaller than the value of Lc(002). More study should be needed to clarify this difference.

REFERENCES

1. I. Mochida, K. Fujimoto, and T. Oyama, "***Chemistry and Physics of Carbon***", Ed. by P.A. Thrower, Marcel Dekker, N.Y., **24**, p. 130, 1994.

Table 1 Some properties of needle cokes used in this study

	NC-F	NC-E	NC-D
Thermal Expansion Coefficient (x 10-6/℃), (1000℃)	NA	0.80	0.67
Density(g/cc)	2.059	2.144	2.139
Volatile matters(wt%)	0.11	0.09	0.08
Vibrated density(g/cc) (8 - 14 mesh)	0.901	0.897	0.943
Sulfur(wt%)	1.03	NA	0.30
Nitrogen(wt%)	NA	NA	0.22
Ash(wt%)	0.28	NA	0.05
Grade for application	C-grade	B-grade	A-grade

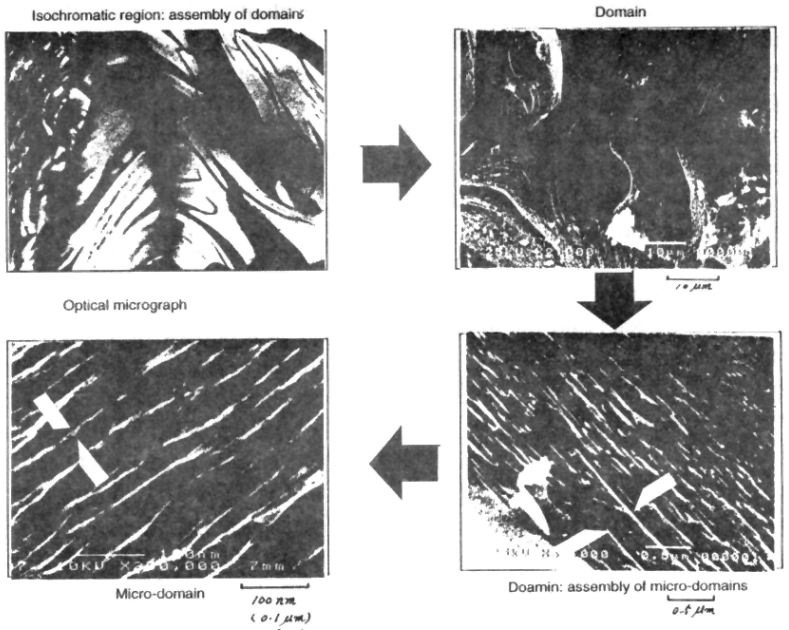

Fig. 1 OM, SEM, and FE-SEM images of heat treated NC-D at 3000℃

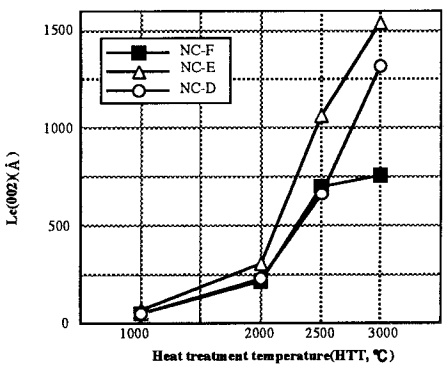

Fig.2 Change of Lc(002) with increasing HTT

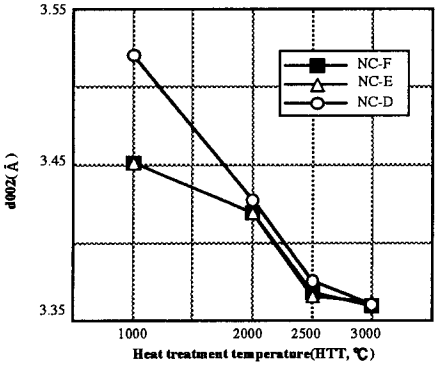

Fig.3 Change of d002 with increasing HTT