TEXTURAL ANALYSIS OF CARBON MATERIALS BY OPTICAL MICROSCOPY AND IMAGE PROCESSING

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Introduction

Optical microscopy (OM) has been used for textural analysis of carbon materials [1,2]. The strength of materials is sensitive to the texture. The orientation of texture of carbon materials can be observed macroscopically by OM, however the OM is qualitative. In order to make the OM observation of carbon materials quantitative, color image processing was introduced. In this study, we report examination results of color image analysis method of texture of carbon materials combined with OM.

Experimental

Anisotropic pitch based carbons were heat treated from 300 to 1000 °C at 4 °C/min heating rate in a tubular furnace with the N_2 gas flow. One pellet shaped raw material was put on a slide glass and fixed on it with pure water, then put into the tubular furnace and heat treated up to 700 °C. Over 700 °C the pellet shaped samples were put in a quartz boat and heat treated. The sample in the quartz tube was pulled out from the furnace and quenched in the N_2 gas flow for preventing reversible reaction.

The free surfaces of the samples were observed by optical microscopy (OM) with a polarizer and an analyzer which were arranged in cross nicol and a λ plate. The observed color images were imputed in to computer by CCD camera. The signals of three primary colors of light (red (R), green (G) and blue (B)) of the images were transformed into color specification system of CIELAB. Hue, chroma saturation and brightness were calculated from the color specification system and the color information was analyzed.

The samples were put in the resin after the surface observation. Cross sections vertical to the surfaces were mirror polished and observed by OM in the same way of the surface observation.

Results and Discussion

Optical microscope images of surface of anisotropic pitch based carbon heat treated at 400°C are shown in Fig.1. The images of Fig.1 (b), (c) and (d) are observed by rotating 45°, 90° and 135° counterclockwise respectively on the basis of image (a) of 0° of the stage of OM.

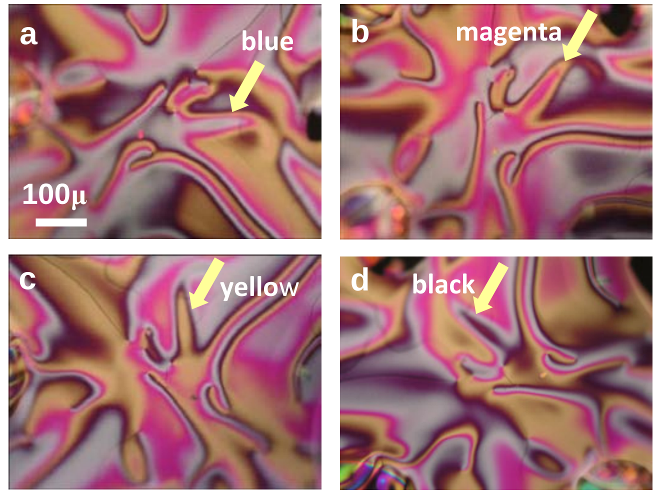


Fig. 1 Optical microscope images of surface of anisotropic pitch based carbon heat treated at 400°C. These four images are the same place of the surface. The images (b), (c) and (d) are rotated 45°, 90° and 135° counterclockwise, respectively, on the basis of image (a) of 0°. The part of blue color of the arrowed shown in (a) changes to magenta (b), yellow (c) and black (d) with rotations.

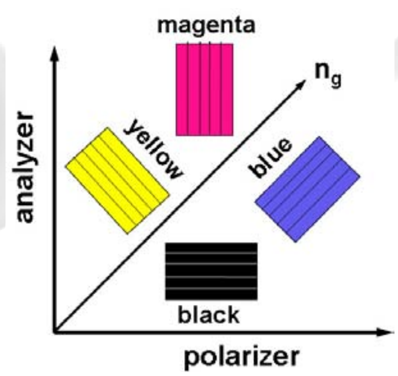


Fig. 2 The relation between colors and the orientations of carbon on optical microscopy.

The relation between colors and the orientations of carbon on optical microscopy is shown in Fig.2. The color observed by OM is blue when the hexagonal carbon layers are parallel to the n_g direction which is the angle of 45° from the polarizer and analyzer (see Fig.2), and vertical to the observed surface corresponding to arrowed blue part shown in Fig.1 (a). The color changes to magenta, yellow, and black every 45°counterclockwise rotation as shown in Fig.1 (b), (c) and (d). This means that the hexagonal carbon layers are perpendicular to the surface (edge on) at the parts arrowed in Fig.1. Many of other parts of the surface of the sample look like edge on too, however quantitative analysis is difficult.

In order to determine the ratio of the edge on surface, color image processing was performed. A distinguishable

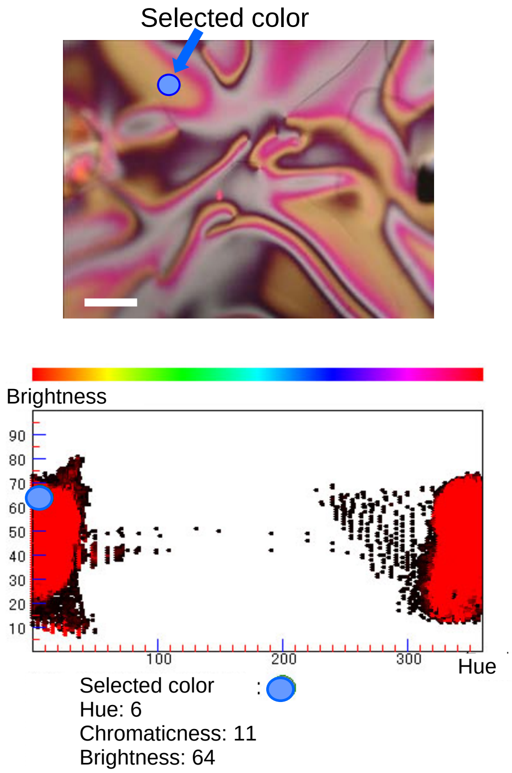
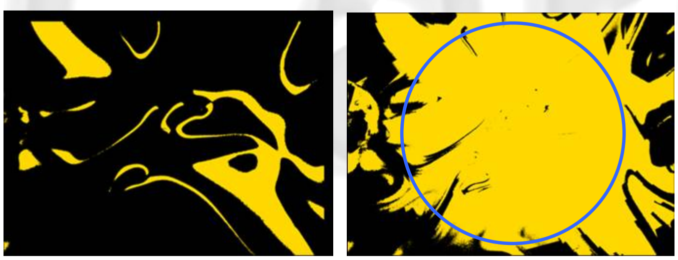


Fig. 3 Selection of representative color and distribution of brightness and hue.



(a) (b)

Fig. 4 Extraction of area of the selected color. The image (a) is obtained from the image of rotation angle 0°. The image (b) is obtained by superposition of images of which rotation angles are from 0 to 180°.

yellowish color was selected from the OM image and the color was transformed into color specification system of CIELAB, and then plotted on the color map of brightness versus hue as shown in Fig.3. The samples were rotated from 0 to 180° on the turning stage and observed every 5 degree by OM. The area within a fixed color difference on the basis of the yellowish color was extracted from the OM images and then transformed to binary images. The binary image of rotation angle 0° is shown in Fig.4 (a).

All the binary images obtained from OM observation images of all rotation angles were superimposed by logic operation OR as shown in Fig.4 (b). The effective area is in

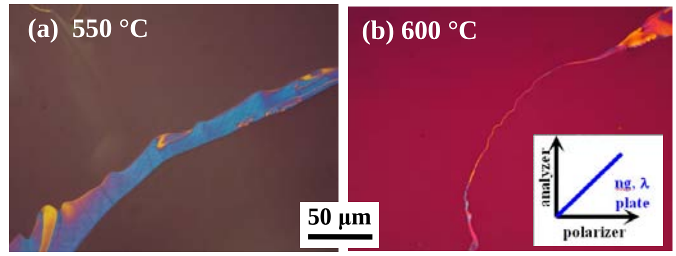


Fig. 5 OM observation images of cross section vertical to the free surface of the samples heat treated at 550 °C (a) and 600 °C (b).

the circle shown in Fig.4 (b) because the images are rotated at the center of them and superimposed one another. The ratio of oriented edge on area estimated 96 % which means edges of hexagonal carbon layers are appear almost all free surface of the sample.

Images of polished cross section vertical to the free surface of the samples heat treated at 550 °C (a) and 600 °C (b) observed by OM are shown in Fig.5. The upper left of both of images is the out side of the material and the lower right is the pores in the samples. The surface of the sample shown in Fig.5 (b) is edge on because the color of the thin pore wall is yellowish which means the hexagonal carbon layers are perpendicular to the n_g direction. There are edges on rims at the surface of the sample as shown in Fig.5 (a). On the other hand, inside of the pore wall is bluish which means the aromatic carbon layers are parallel to the n_g direction.

The edge on surface of the anisotropic pitch seems to be formed by interaction between liquid surface of the sample and N_2 gas atmosphere [3].

Conclusions

We proposed color image analysis method by using color specification system of CIELAB. The color image analysis method can make OM observation quantitatively and detected anisotropic area precisely.

The surface and cross section observations of the samples estimated relation between the surface and inside of the samples.

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References

[1] Lafdi K, Bonnamy S, Obérlin A. Mechanism of formation of the texture and microtexture in as-spun then oxidized anisotropic pitch-based carbon fibers, part I: Radial with wedge type i1. Carbon 1992;30(4):551-567.

[2] Oshida K, Bonnamy. Primary carbonization of an anisotropic ‘mesophase’ pitch compared to conventional isotropic pitch. Carbon 2002;40(14):2699-2711.

[3] Jian K, Shim HS, Tuhus-Dubrow D, Bernstein S, Woodward C, Pfeffer M, Steingart D. Liquid crystal surface anchoring of mesophase pitch. Carbon 2003;41(11):2073-2083.