Application of SEM to detect the structure of mesocarbon microbeads

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The typical structure of mesocarbon microbeads (MCMB) prepared from pitches is usually regarded as Brooks-Taylor-type [1, 2] with the polyaromatic molecules approximately parallel to one another and perpendicular to the surface of the sphere. To determine the structure of spherical carbonaceous mesophase, optical microscopy is a traditional method, which is used by detecting the changes of extinction contours or the interference colors under polarized light when they are still in the isotropic matrix of heat-treated pitch [1–5]. Recently, much work was focused on the MCMB formed through heterogeneous nucleation [6, 7] and some results indicated that the quinoline insolubles (QI) or other solid additives in the parent pitches would transform the structures of MCMB into different types from the Brooks-Taylor-type [6, 7]. It seems that optical microscopy is unsuitable when being used to determine the complicated structures of those micron spheres. In this letter, a method for making samples for observation of MCMB structures using scanning electron microscopy (SEM) was developed and its validity proved by comparison with structures examined using optical microscopy. With this method, the complicated structures of MCMB prepared from pitch with more QI were also examined successfully and thus discussed the structure difference between two kinds of MCMB prepared from coal tar pitches with different QI concentrations.

Two kinds of MCMB, MCMB1 and MCMB2, are used as our samples for analyses. They were prepared from two coal tar pitches with different QI concentrations, 0.3 and 4.7 wt%, respectively. Since QI matter in the pitches could disorder the arrangement of mesophase molecules and thus influence the structure of as-obtained MCMB, MCMB1 and MCMB2 are expected to have different microtextures. Before making samples for SEM observation, MCMB samples should be carbonized at a high temperature in advance to eliminate the light components in them and make clear their carbon layers under SEM. In the present experiment, carbonization at 1000 °C for 1 hr was adopted. The carbonized MCMB samples were then embedded in a thermosetting resin to obtain MCMB/resin bulks. Subsequently, the as-received bulks were cut quickly and gently with a sharp scalpel on their surfaces. The formed carbon fractions on the treated bulks were washed away with the help of ultrasonic vibration before observating on a PHILIPS SEM (XL30).

For comparison of the structures observed under SEM with those under optical microscopy, mesophase pitches containing above MCMB, MP1 and MP2, respectively, were embedded in epoxy resin and the as obtained bulks were then ground and polished according to the usual method. The investigation of the obtained pitch/resin bulks were carried out on a Nikon E600 POL polarized light microscope and micrographs were captured using an *in situ* Nikon DXM1200 digital camera.

Fig. 1 shows the optical microscopy micrographs of MP1 and Fig. 1b shows the extinction contour changes of one sphere of MCMB1, pointed out by the arrow in Fig. 1a, by rotating the stage of polarized light microscope per 15° anticlockwise. The regular extinction contour changes indicate that the formed structures of MCMB1 are Brooks-Taylor-type [1, 2] with the model structure shown in Fig. 1c.

According to above-mentioned sample-making method, the microtextures of MCMB1 were disclosed and some typical cross-sections of them are shown in Fig. 2. As can be seen, most of the microtextural layers of MCMB1 (as shown by the dotted white circles in Fig. 2) approximately parallel to one another and perpendicular to the surface of the sphere. Therefore, MCMB1 have Brooks-Taylor-type microstructure as deducted from their extinction contours mentioned above, indicating that the sample-making method for SEM observation of MCMB structures is valid and could be adopted to analyze the structure of this kind of micron spheres.

In Fig. 3, MCMB2, contained in mesophase pitch (MP2), is shown under polarized light. It could be seen that MCMB2 have irregular extinction contours, which means they have complicated microstructures different from MCMB1. When rotating the stage of optical microscope, irregular changes of extinction contours of MCMB2 also indicate their complicated structures, but the definite structures are difficult or even impossible to be deduced from them. Whereas, from the SEM micrographs (as shown in Fig. 3) obtained with the help of

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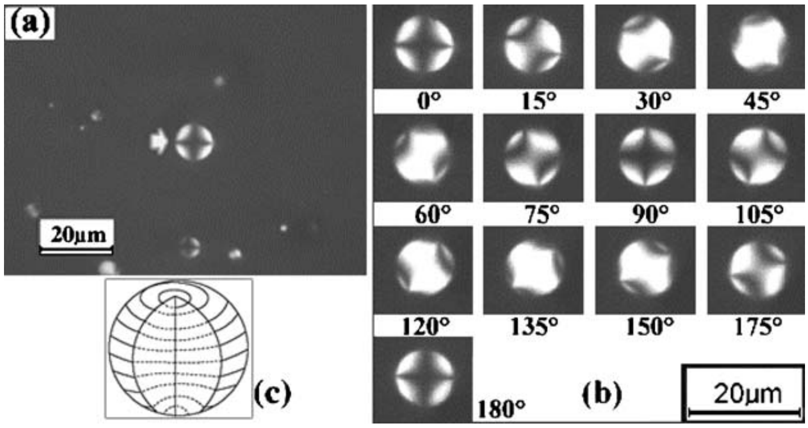

Figure 1 Optical characteristics of MCMB1, which are still in MP1, under polarized light: (a) a typical micrograph, (b) regular extinction contour changes of the sphere pointed out in a by the arrow when rotating the stage per 15° anticlockwise, and (c) model structure of Brooks-Taylor-type MCMB.

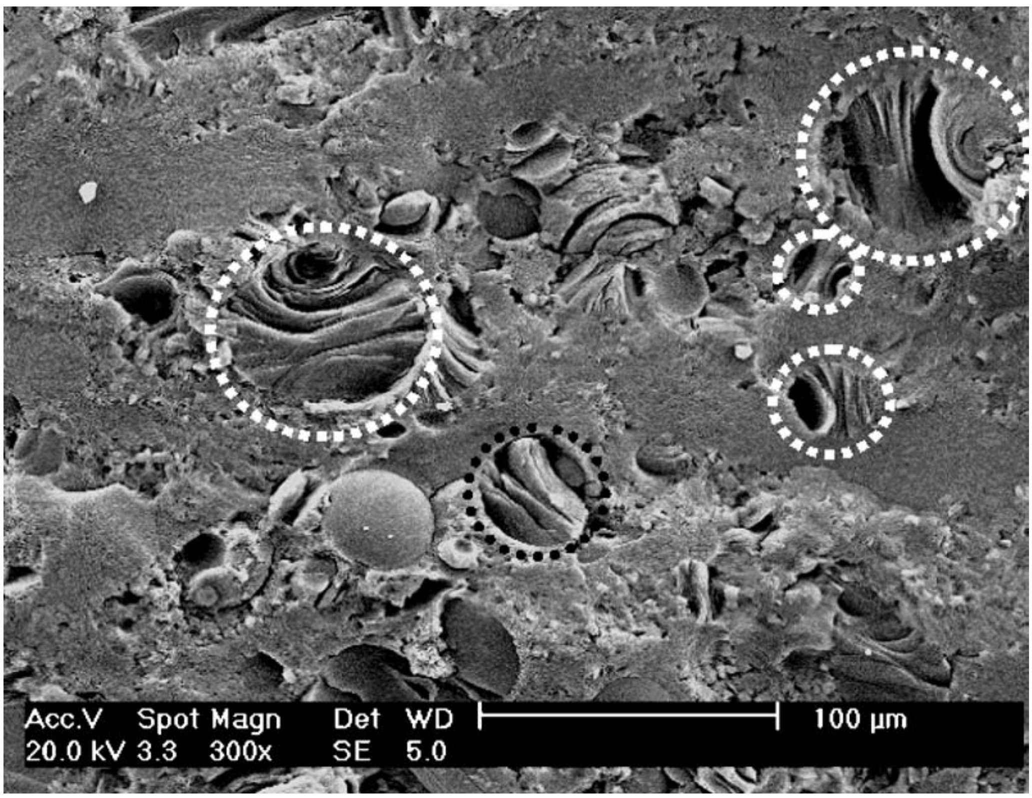

Figure 2 SEM micrographs of the cross-sections of MCMB1.

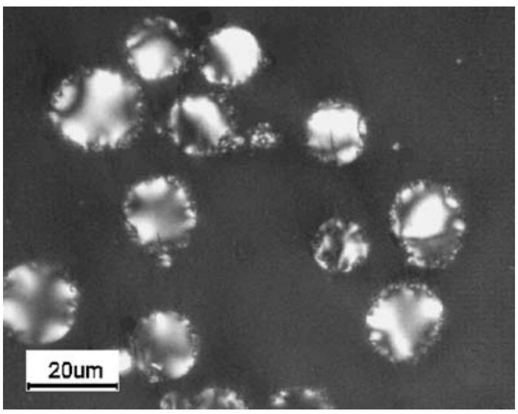

Figure 3 Optical characteristics of MCMB2, which are contained in MP2, under polarized light microscope.

the above-mentioned sample-making method, it can be seen clearly that the microtextures of MCMB2 are complicated. The microtextural layers of MCMB2 in Fig. 4a are almost parallel, which could be regarded as a near Brooks-Taylor-type structure, whereas those of the one in Fig. 4b are deformed into gourd ladle shape and are very different from Brooks-Taylor-type structure. These characteristics of the microstructure of MCMB2 also proved the viewpoint that QI in the parent pitch can influence the natural stacking of mesophase molecules.

From above analyses, it can be considered that the sample-making method for SEM observation is effective for investigation of the microstructures of MCMB, a kind of micron carbon sphere. After comparing the microtextures of MCMB1 with MCMB2, a conclusion

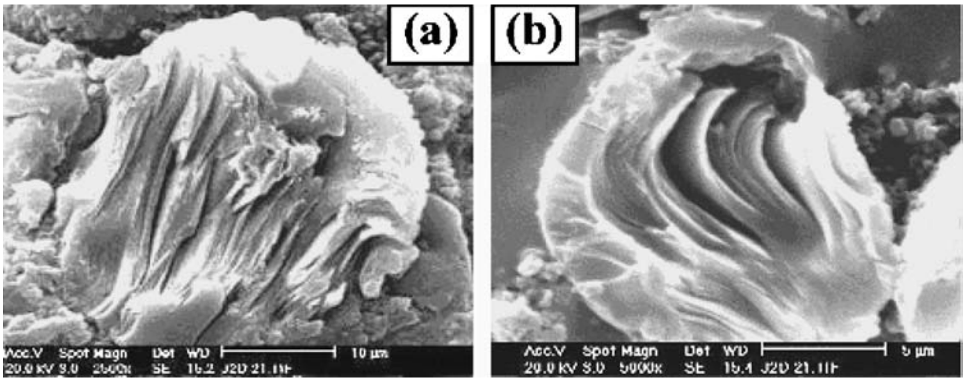

Figure 4 Complicated structures of MCMB2 detected by SEM.

can be drawn that MCMB prepared from coal tar pitches with different QI can have different microtextures. In addition, SEM observation with the help of the sample-making method given here could be further extended to analyze other micron materials including those without optical activity.

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