RAPID PUBLICATION

High-Voltage High-Resolution Electron Microscopy on Thin Films of Monodispersed Pseudocubic and Peanut-Type Hematite Particles

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In order to investigate internal structures of monodispersed hematite ($\alpha\text{-Fe}_2\text{O}_3$) particles of pseudocubic and peanut-type shapes prepared by the gel-sol method (J. Colloid Interface Sci., **152** (1992), 587; Colloids Surf. **70** (1992), 167), their thin sections were prepared by an ultra-microtome and studied by transmission electron microscopy. The surfaces and arrangements of the individual small subcrystals as components of the monodispersed particles were clearly observed on the atomic scale by high-voltage high-resolution electron microscopy. This is direct evidence of the polycrystallinity of these particles. Furthermore, it was found that the subcrystals of the pseudocubic particles had a square shape bounded by sharp $\{01\bar{1}2\}$ surfaces lining up with the same orientation, whereas those of the peanut-type particles had relatively round surfaces and the crystallographic orientations of adjacent subcrystals slightly differed from each other.

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Monodispersed oxide particles, which are uniform in shape, size and composition, have been prepared widely for the purpose of producing various advanced materials⁽¹⁾⁻⁽⁴⁾. Recently using a newly developed technique, the gel-sol method⁽⁵⁾⁽⁶⁾, we have succeeded in obtaining large quantities of micron-size monodispersed hematite particles ($\alpha\text{-Fe}_2\text{O}_3$) of various shapes, such as pseudocubic, ellipsoidal, and peanut-type. Besides, characterization of some hematite particles of various shapes has been carried out⁽⁷⁾. For the platelet-type particles prepared by the gel-sol method, microscopic morphology of the external surfaces and the internal structures were analyzed in detail by high-resolution electron microscopy (HREM) on their sections prepared with an ultra-microtome⁽⁸⁾. In this paper, we will briefly report the internal structure of the pseudocubic and peanut-type hematite particles obtained by the same technique.

The samples of pseudocubic and peanut-type hematite particles were prepared by the standard procedures of the gel-sol method⁽⁵⁾⁽⁶⁾. For the preparation of the peanut-type particles, the concentration of sodium sulfate was 3×10^{-2} mol/dm³. The detailed procedures of the preparation of the sections are given in the references⁽⁸⁾⁽⁹⁾. In addition to conventional electron microscopy at relatively low magnification with a 200 kV electron microscope (JEM-2000EX), an HREM study was carried out by using a high-voltage electron microscope (JEM-

ARM1250) with an accelerating voltage of 1250 kV.

Figures 1(a) and (b) show electron micrographs of sections of pseudocubic and peanut-type particles, respectively. From their sizes and shapes, it is easily seen that the pseudocubic particle was cut almost parallel to the basal plane and the peanut-type particle parallel to the revolutional axis. The pseudocubic and peanut-type monodispersed particles shown in the insets are the groups of the original particles. In the micrograph of the thin section of the pseudocubic particle in Fig. 1(a), there are small subcrystals 10 to 20 nm in size lining up outwards from the center of the particle. In the micrograph of the thin film of the peanut-type particle in Fig. 1(b), the subcrystals are elongated and radially oriented from the revolutional axis of the particle.

Figures 2(a) and (b) show HREM images of thin sections of pseudocubic and peanut-type particles which were on a carbon film of a microgrid. The locations of the HREM images were the surface parts of these particles indicated by the arrows in the insets. In both Figs. 2(a) and (b), the fine fringes correspond to the crystal lattice of the hematite with the corundum structure, and the shapes of the subcrystals and their arrangement are clearly observed on the atomic scale. It is found that the subcrystals of the pseudocubic particle in Fig. 2(a) have a cuboidal shape bounded by sharp $\{01\bar{1}2\}$ surfaces and round edges with the same crystallographic orientation.

Fig. 1 Electron microscope images of the sections of pseudocubic (a) and peanut-type (b) hematite particles. In each inset, the respective original hematite particles without cutting are shown.

Fig. 2 High-voltage HREM images of the parts of the sections of pseudocubic (a) and peanut-type (b) hematite particles. The arrows in the low-magnification images in the insets indicate the regions corresponding to the HREM images. The indices of the crystal planes were assigned based on the hexagonal system.

In contrast to the pseudocubic particle, the subcrystals of the peanut-type particle in Fig. 2(b) have relatively round surfaces and the crystallographic orientations of adjacent subcrystals are slightly tilted from each other, as indicated by A and B. Detailed investigation on the size distribution and arrangement of the subcrystals in the

particles with electron diffraction and HREM is under progress. The results will be published elsewhere.

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