Microstructure Analysis of Mica Particles Coated with Metal Oxide by Transmission Electron Microscopy

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Intensive work to prepare oxide layers uniform in thickness, size, and composition on particle substrate has been initiated for the purpose of producing advanced materials in the field of the automobile industry. One of the most important applications of mica is pearlescent pigments in the field of the automobile industry[1,2], which consist of mica powders coated with a thin layer of metal oxide, mostly TiO_2. However, one difficulty which arises from the hydrolysis process is the pealization of uniform thickness and fine grain of the oxide layer on the mica. In order to obtain optimum pearlescent pigment performance, thickness control and a fine structure of the metal oxide are necessary. Mica particles coated with TiO_2 metal oxide were produced by concentrated homogeneous hydrolysis of metal sulphates with urea in the presence of mica particles. By XRD, the crystal structure was identified as anatase, close inspection of the internal structure of these mica particles coated with TiO_2 metal oxide seems to be needed for a complete understanding of their characteristic growth mechanism.

For this purpose, we have attempted direct observation of microstructure. This article is a short report on a study by SEM and TEM coupled with a sample preparation technique with ion milling of the microstructure of mica particles coated with TiO_2 metal oxide prepared from a condensed homogeneous hydrolysis system.

Fig. 1 shows a SEM image of the top surface of a muscovite mica particle. The TiO_2 layers are composed of small particles, approximately 50nm in diameter, and bound by rough surfaces with small bumps. It can be seen that the layer of anatase consists of well-distinguished homogeneous nano particles. Fig. 2(a) shows a cross-sectional bright-field image of the mica coated with metal TiO_2 oxide. The thicknesses of the mica particles and TiO_2 metal oxide layers were estimated to be ~60/50 nm, respectively. The muscovite particle bears a uniform oxide layer and the TiO_2 metal oxide surface layer can be distinguished form the mica substrate. The inset of Fig. 2(a) is a corresponding selected area diffraction pattern obtained from the interface region of the oxide layer and mica with the incident beam parallel to the $[11\underline{2}0]_{mica}$ direction. Fig. 2(b) shows EDX spectra obtained from the thin sections of TiO_2 layer, it was found that the atomic ratio of Ti to O was about 1:2, although there were some variations in the ratio depending on the analyzed positions in the sections. Fig. 2(c) shows high-resolution TEM images of a thin section. From the lattice images of Fig. 2(c), the TiO_2 layers are identified as anatase, in agreement with the results from X-ray diffractometry. This result supports the contention that the optical interference effect of the pigments derived from the homogeneous hydrolysis method was dependent on the thickness of the oxide layer and subsequent thermal treatment.

Acknowledgement

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References

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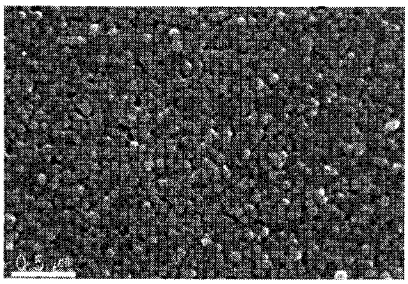

Fig. 1. A SEM image of the top surface of a muscovite mica particle.

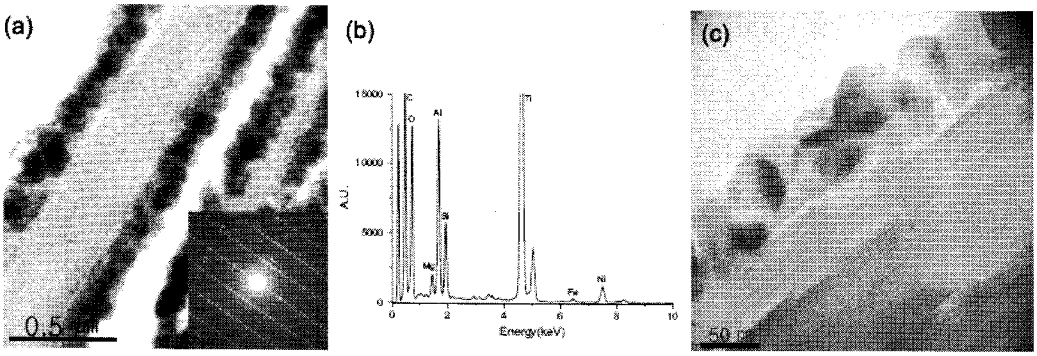

Fig. 2. (a) Cross-sectional TEM image of the mica coated with TiO_2 metal oxide and diffraction pattern. (b) EDX spectra obtained from sections of the TiO_2 metal oxide layer. (c) HREM image of the mica coated with TiO_2 metal oxide.