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SYNTHESIS OF ZINC OXIDE POWDER BY HYDROLYSIS OF BIS(ACETYLACETONATO)-ZINC(II) IN AQUEOUS SOLUTION

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Pure and fine powder of zinc oxide was prepared by hydrolysis of bis(acetylacetonato)zinc(II) in methanol-water mixture. A significant difference in the particle shape was observed by TEM between ZnO powders prepared at 25 and 80 °C.

The recent development of ceramic industry needs fine metal oxide powders. Zinc oxide powder is widely utilized for the functional devices (sensor, varistor, etc), pigment, electrography, medical materials and so on. We have recently reported the novel synthetic methods of zinc oxide powder or film by gas-phase reaction between bis(acetylacetonato)zinc(II) and water. Minute zinc oxide particles of uniform size (D = 21-36 nm) were obtained in the temperature range of 100-800 °C.[1] Orientated films of zinc oxide were also deposited at 90 °C or above.[2] The fact that the reaction can be effected at low temperature below 100 °C prompted us to examine the hydrolysis reaction of $Zn(acac)_2$ in aqueous solution. Metal alkoxides have so far been mainly used for the preparation of metal oxides from transition metal complexes in liquid phase.[3,4]

Bis(acetylacetonato)zinc(II) was prepared by the method in the literature.[5] Methanol solution (100 cm^3) of $Zn(acac)_2$ (0.02 mol) was added in a thin stream to deionized water (400 cm^3) with vigorous stirring. On mixing two solutions white precipitate immediately appeared. The resulting suspension was kept for 1 h with stirring. Then the precipitate was filtered off and washed with methanol and finally dried in an vacuum oven at 50 °C. The above precipitation was carried out at 25 and 80 °C; hereinafter the zinc oxide powder prepared at 25 °C is referred as ZnO-25 and at 80 °C as ZnO-80.

X-Ray diffraction patterns of ZnO-

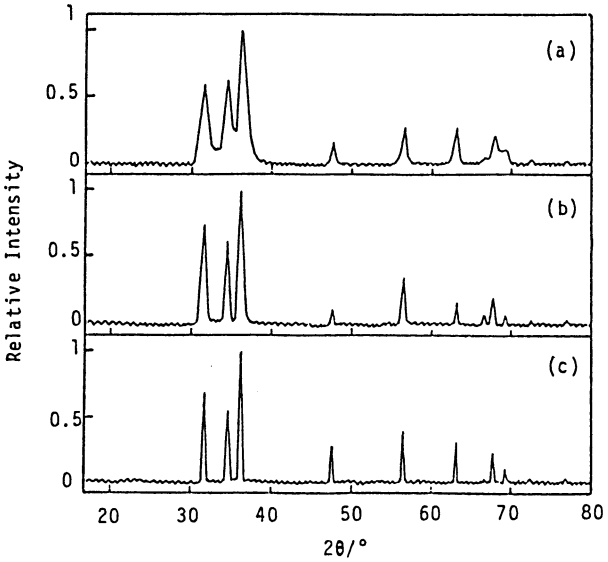

Fig.1. X-Ray powder diffraction patterns of ZnO-25(a), ZnO-80(b) and authentic ZnO(c).

25 and ZnO-80 are shown in Fig. 1 together with that of authentic ZnO.[5] The d-spacings of both ZnO powder are identical with that of the authentic one while the peak width for ZnO-80 is fairly narrow than that for ZnO-25. These results indicate that the products are pure crystalline zinc oxide with no contamination of zinc hydroxide and that the crystallite size of ZnO-25 powder is smaller than that of ZnO-80 powder. The photographs by a transmission electron microscope (TEM) shown in Fig. 2 indicate that ZnO-80 powder is composed of well-shaped rod-like crystals while ZnO-25 powder is irregular in shape.

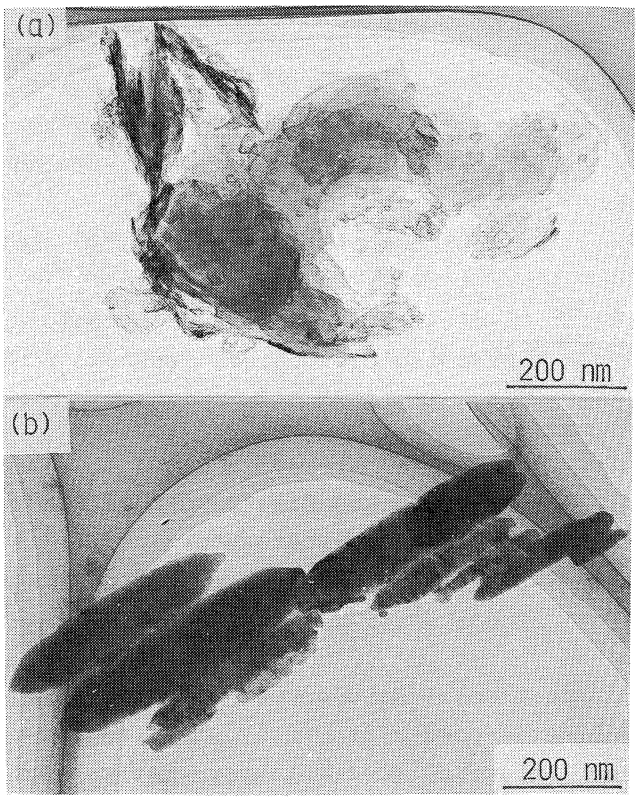

Fig.2. TEM photographs for ZnO-25(a) and ZnO-80(b) powders.

The hydrolysis of metal alkoxides has been reported to yield pure and minute powders of metal oxides or hydroxides.[4] However, the synthesis of zinc alkoxides as starting materials is not so easy and the alkoxides are considered to suffer from the contamination of the alkali metal ions.[6] On the other hand, zinc acetylacetonate is readily prepared from zinc hydroxide and acetylacetone and the hydrolysis products free from the contamination of any other metal ions.[1] Therefore, this method seems to be more favorable than the " alkoxide method " with respect to the preparation of ZnO powder.[7] Details will be reported in the near future.

References

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4) Y. Bando, "Kinousei Ceramics no Sekkei," ed by Nihon Kagaku Kai, Gakkai Shuppan, Tokyo (1982), Chap. 4.
5) A. S. T. M., 5-0664.
6) Y. Ozaki and T. Mitati, 57th Annual Meeting of the Ceramic Society of Japan, Kyoto, May 1982,Abstr., No. C-3.
7) $Zn(acac)_2$ has already been used to a metal ion source in the preparation of $(Mn,Zn)Fe_2O_4$; S. Naka, Y. Suwa, T. Tsutsuki, and S. Hirano, ibid., No. A-37.

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