CHEMISTRY LETTERS, pp. 1869-1872, 1987. 

# Synthesis of $Al_2O_3$-coated $TiO_2$ Composite Powder by Homogeneous Precipitation Method

Byung-Kwan KIM* and Itaru YASUI

Institute of Industrial Science, The University of Tokyo, Roppongi, Minato-ku, Tokyo 106

$Al(OH)_3$-coated $TiO_2$ composite powder was prepared from the aqueous solution of $Al_2(SO_4)_3$ and rutile powder by the homogeneous precipitation method using urea, then heat treated at 1200 °C for 1 h in order to convert it to $\alpha$-$Al_2O_3$-coated $TiO_2$ powder.

It is important to develop a new class of ceramic powders, such as powders coated with other kinds of substances. For example, $Al_2O_3$-coated $TiO_2$ composite powder is of use as a starting material of aluminum titanate ($Al_2TiO_5$) which is well-known for its apparent low thermal expansion and high melting point.[1)] To obtain improved, reliable composite ceramic powders, coating process is to be optimized. Aluminum titanate is ordinarily prepared from a stoichiometric mixture of alumina ($Al_2O_3$) and titanate ($TiO_2$) powders by conventional processing methods such as ball milling and isostatic pressing.[2)] These methods, however, often yield powders mixed inhomogeneously on a microscopic scale, and do not allow control of particle-size distribution and particle shape in a sintered body. The homogeneous precipitation method is one of the best methods to control pH and to form pure and dense precipitates, so that various ceramic powders such as $TiO_2$, $Al_2O_3$, $MgAl_2O_4$, $Fe_3O_4$, $Fe(OH)_2$, and hydrated $Al_2O_3$ and $Al_2O_3$-coated $Cr_2O_3$ composite powders were synthesized by this method using urea.[3-10)] On the other hand, $TiO_2$-coated $Al_2O_3$ composite powder was prepared from titanium alkoxide precursor and alumina powder by Okamura et al.[11)] This letter deals with the synthesis of $Al_2O_3$-coated $TiO_2$ composite powder using the homogeneous precipitation method as a coating process.

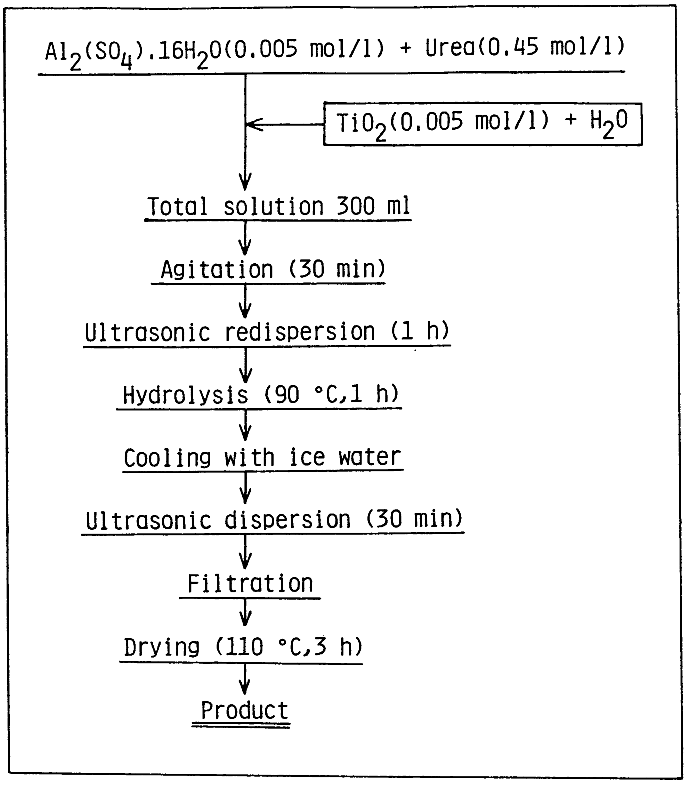



Fig. 1. Flow chart for preparation of hydrated $Al_2O_3$-coated $TiO_2$ composite powder.

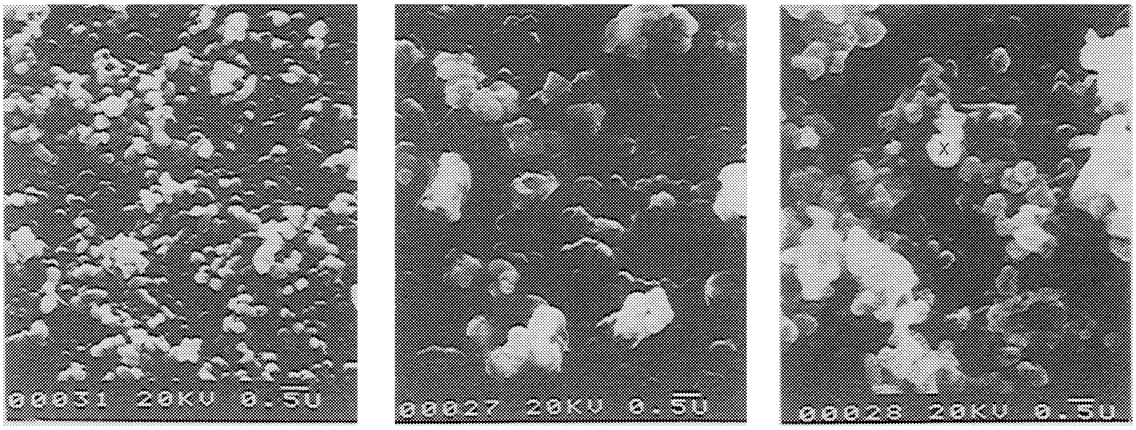
16-hydrated aluminum sulfate and urea used were guaranteed grade (Wako Chemical Co.) and $TiO_2$ powder (rutile form) (Sakai Chemical Co.) consisted of approximately spherical particles with the average particle size of 0.2±0.04 μ (Fig. 2(a)). An aqueous solution of $Al_2(SO_4)_3.16H_2O$ (0.005 mol/l) and urea (0.45 mol/l) was prepared in a three-neck flask with a capacity of 300 ml and $TiO_2$ (0.005 mol/l) was added. From this solution hydrated $Al_2O_3$-coated $TiO_2$ composite powder was prepared according to the procedure shown in Fig. 1. Agitation was required through the hydrolysis in order to disperse $TiO_2$ in the reaction solution.

Urea is hydrolyzed to ammonia and carbon dioxide as follows:

$$CO(NH_2)_2 + H_2O \longrightarrow 2NH_3 + CO_2.$$

Urea is suitable to a hydrolytic process because of its very weak basic properties ($Kb=1.5\times10^{-14}$) and its high solubility into water. It hydrolyzed easily at 80-100 °C and the hydrolysis can be quickly terminated at a desired pH by cooling the reaction mixture to room temperature.

In order to confirm the formation of hydrated $Al_2O_3$-coated $TiO_2$ composite particle, it is preferable to observe the cross section of hydrated $Al_2O_3$-coated $TiO_2$ composite powder. This experiment was nearly impossible because the product was submicron in size. Thus, this was confirmed indirectly by means of




(a) (b) (c)

Fig. 2. SEM photographs of starting $TiO_2$ powder(a), hydrated $Al_2O_3$-coated $TiO_2$ composite powder(b), and $\alpha$-$Al_2O_3$-coated $TiO_2$ composite powder(c).

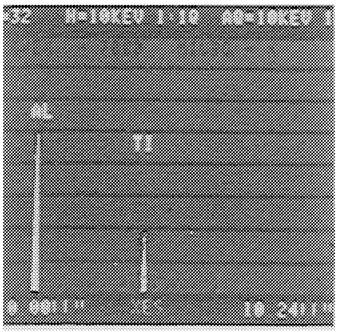



Fig. 3. EDX spectrum of spot analysis at the mark 'x' shown in Fig. 2(c).

SEM (S-700, Hitachi Co.) observation and EDX (kevex-7000) analysis. Hydrated $Al_2O_3$-coated $TiO_2$ composite powder consisted of approximately spherical particles with some agglomeration and its average particle size was 0.29±0.09 μ (Fig. 2(b)). By heating at 1200 °C for 1 h, the product was converted to $\alpha$-$Al_2O_3$-coated $TiO_2$ composite powder with the average particle size of 0.27±0.08 μ (Fig. 2(c)). The change in average particle size between this composite powder and the starting $TiO_2$ powder was an indirect proof of the formation of hydrated $Al_2O_3$-coated $TiO_2$ composite particle. Figure 3 is the EDX spectrum of spot analysis at the mark 'x' shown in Fig. 2(c). X-Ray count ratios of Al/Ti obtained for other particles had almost same value as the one shown in Fig. 3. This was also a proof of the formation of hydrated $Al_2O_3$-coated $TiO_2$ composite particle. It is difficult to describe a formation mechanism of hydrated $Al_2O_3$-coated $TiO_2$ composite powder in detail. In general, $TiO_2$ powder has high dispersibility in an aqueous solution and its surface is more active, compared with other metal oxides such as $SiO_2$, $ZrO_2$, MgO, and ZnO etc.[12] It is supposed that $TiO_2$ particles dispersed in the solution act as seeds or accelerators in the formation of hydrated $Al_2O_3$. Further characterization of this composite powder and application of this coating process to preparing other coated composite powders, are in progress.

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( Received July 20, 1987 )