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Effect of Process Parameters on Phase Evolution during Synthesis of TiO_2 Nanopowders by Flame Method

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Introduction

Various kinds of crystalline phases such as anatase, rutile and brookite are found in titanium dioxide (TiO_2). Recently, TiO_2 nanopowder is one of key materials in photocatalytic applications [1]. The TiO_2 nanopowder can be synthesized using either aqueous solution media (sol-gel and hydrolysis) or gaseous precursors (flame method). The TiO_2 nanopowder produced by the flame method is known more effective for photocatalytic applications.

Many works related to flame method have focused on verifying the role of operation parameters on the size and shape of nanopowders [2-4]. In addition, a role of process parameters on the evolution of constituent phase(s) in the TiO_2 nanopowder needs to be verified to improve its photocatalytic property [5]. In spite of several studies to determine the controlling parameters for phase evolution, still there are no agreements generally accepted yet.

In order to clarify the effect of cooling rate of hot flame on the evolution of crystalline phases in the TiO_2 nanopowder, the composition and flow rate of gas mixtures were varied. In particular, the evolution of phases during the formation of TiO_2 particles was investigated from the samples directly collected by in-situ TEM sampler inserted into the interior of flame.

Keywords: Titanium oxide nanopowder, Anatase TiO_2, Rutile TiO_2, Flame method, Flame temperature.

Experimental procedure

A precursor of titanium tetraisopropoxide (TTIP) was used for synthesis of TiO_2 nanopowder. TTIP has melting temperature of 18℃, boiling temperature of 232℃, molar weight of 286.26 g, and density of 0.955 g/cm^3. TTIP was vaporized from the bottle immersed in the oil bath and transported to the burner nozzle by nitrogen as a carrying gas. The amount of TTIP precursor was controlled by the temperature of oil bath and flow rate of nitrogen gas. The flow rates of oxygen (oxidizer) and methane (fuel) were varied separately.

TiO_2 particle forming at each position in the flame was collected with an in-situ TEM sampler by inserting a carbon-coated Cu-grid for 1 sec in the flame. The morphology and size distribution of TiO_2 nanopowder were observed with a field emission scanning electron microscopy (FESEM, Philips XL 30) and transmission electron microscopy (CM30 TEM). The change of TiO_2 nanopowder was examined by selected area diffraction pattern (SADP). The constituent phase of TiO_2 nanopowder was determined with a Philips XRD 1830 using Cu K_α radiation.

Results and Discussion

TiO_2 nanopowder synthesized at the oxygen flow rates of 0.5 slm and 3.0 slm are shown in Fig. 1. The morphology of TiO_2 nanopowder was proved not sensitive to the operating conditions and its size is in the range of 70-120 nm in diameter.

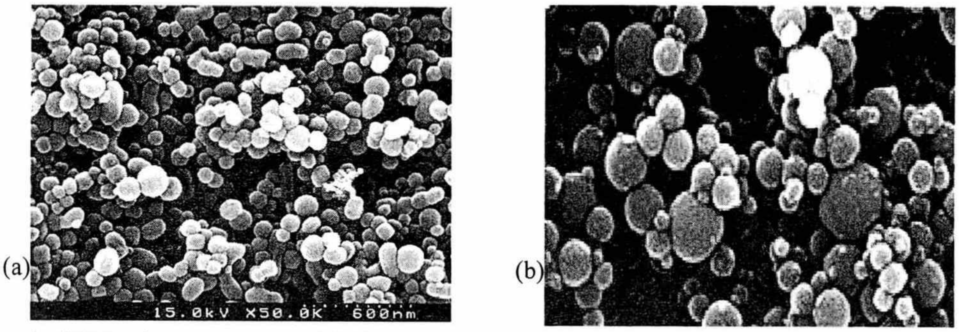

Fig. 1. SEM microstructures of TiO_2 nanopowders synthesized at the flow rates of oxygen/methane/nitrogen =; (a) 0.5/0.3/0.5 and (b) 3.0/0.3/0.5

Contrary to the indifference of morphology and size to the flame condition, the evolution of crystalline phases shows a strong dependancy on it. Fig. 2 shows X-ray diffraction patterns obtained from the TiO_2 nanopowders synthesized at various conditions. Among the constituent gases of oxygen, methane, and nitrogen, the flow rate of oxygen was changed from 0.5 slm to 5 slm keeping the flow rates of methane and nitrogen by 0.3 slm and 0.5 slm, respectively. Note that the peaks at $2\theta_{(111)}$ = 25.3° are of anatase TiO_2 and those at $2\theta_{(111)}$ = 27.4°, rutile TiO_2. At 0.5 slm oxygen condition, the TiO_2 nanopowder of anatase phase comprising about 30% rutile phase was obtained. The anatase phase was evolved predominantly at every flame condition with high oxygen flow rate more than 1.0 slm.

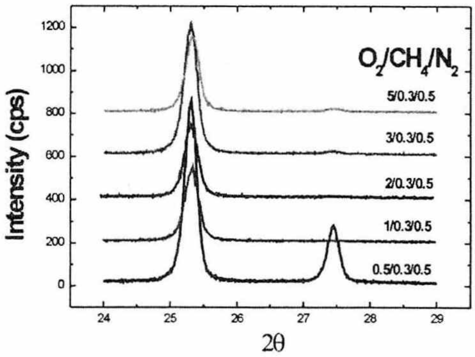

Fig. 2 XRD patterns of TiO_2 nanopowders synthesized at various flow rates of oxygen keeping the flow rate of methane and nitrogen constant.

In this presentation, an explanation on the phase evolution in the TiO_2 nanopowder during synthesis by the flame method will be given through measuring the temperature distribution in the each flame and determining the phase(s) of TiO_2 nanopowders by TEM.

References

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