

RUTILE NANOPOROUS MICROSPHERES FORMED IN WATER SOLUTION

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Fabrication of mineral-like synthetic crystals in a nanosize range is a paramount task of modern materials science because of unusual physical and chemical properties due to micro- and nano- morphology valuable for numerous applications in nanochemistry, photocatalysis, and electrochemistry. There are several polymorph modifications known for TiO₂ such as orthorhombic brookite and tetragonal anatase and rutile. Solution-based techniques designed for TiO₂ precipitation typically leads to anatase structure formation. Titanium (IV) oxide is widely studied functional material due to excellent chemical stability and interesting electronic structure. It has promising application for water-splitting photocatalyst resulting in hydrogen generation, support for heterogeneous catalyst, electrode for dye-sensitized solar cells, nanoelectronic applications. Present study is aimed to inquire into potentials of low-temperature solution way ($T = 100^{\circ}\text{C}$) for obtaining of nanoporous TiO₂ microspheres with high-temperature rutile crystal structure.

Synthesis of TiO₂ microspheres was carried out by the two-stage synthesis. Firstly commercial TiO₂ (99.99 %) was dissolved in the strong ammonia water solution under the stirring and heating at $T = 100^{\circ}\text{C}$. So prepared ammonium titanate water solution was filtered with filter paper. Then the solution with pH = 14 was acidated to pH = 1 with nitric acid under continuous stirring and heating at $T = 100^{\circ}\text{C}$. The final powder-like deposit was washed by distilled water up to pH = 6 of wash water and dried in air at room temperature. The phase composition of the samples was determined by powder x-ray diffraction (XRD) method. The morphology of TiO₂ nanoporous microsphere crystals was examined by scanning electron microscopy (SEM) by a LEO 1430 (CKP Nanostructures) device. As a result, white monosized microspheres with typical diameter $\sim 20 \mu\text{m}$ were fabricated as shown in Fig.1. The SEM images of the microspheres show developed nanoporous structure with radially extending pores. The pore sizes are very uniform with $\sim 10 \text{ nm}$ diameter. Phase composition of the precipitate has been confirmed by XRD analysis as rutile ($P4_2/mnm$, PDF 21-1276).

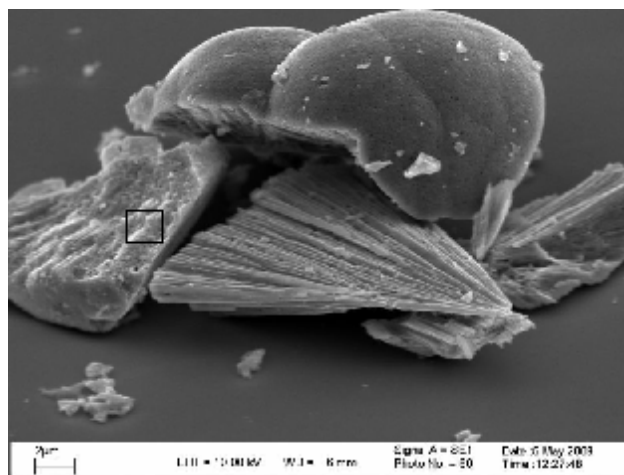


Fig.1. SEM image of splitted TiO₂ microspheres.