Preparation of Gold Nanoparticles Inside Titania Nanotubes *via* Wet Chemistry

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Nanostructured titania materials show high photocatalytic activity and are widely used as a catalyst and carrier of catalysts where the tuning of the pore size and overall morphology are crucial. The hollow titania nanotubes with large surface area can offer a prospect to provide highly active hosts for catalytic reactions and for fabrication of high efficient solar cells. Their unique tubular structure (4.5 nm inner diameter) has prompted us to investigate the physical and chemical properties of nanoparticles confined inside their inner nanospaces. Recently, gold nanoparticles supported on titania have been shown to be active catalysts for a number of catalytic reactions (e.g. low-temperature oxidation of CO and selective oxidation of propene). Studies show that less than 5 nm gold particles supported on titania optimize the effectiveness of catalytic oxidation of carbon monoxide [1]. Therefore, the preparation of such gold nanoparticles in a highly dispersed and stable form is now becoming a very challenging area of catalyst research. We report here the synthesis of gold nanoparticles inside titania nanotubes.

Uniform TiO_2 nanotubes were prepared from the assembly of TiO_2 nanocrystals in NaOH aqueous solution at 125 "C for 70 h [2, 3]. TEM examination showed that the such titania consist of open nanotubes with 4 – 5 nm in diameter and 50-400 nm in length. The corresponding specific area was up to 500 m^2/g. Wet chemistry techniques were used to fill the titania nanotubes with gold nanoparticles. First, these nanotubes were dispersed in water by sonication, followed by treating the nanotubes with a suitable amount of $HAuCl_4 \cdot 3H_2O$ in an aqueous solution. The gold precursors were loaded into the nanotubes by adjusting various solution pH values (acidic, neutral, and basic conditions) or using different ligands such as 1,2-ethanediamine ($H_2NCH_2CH_2NH_2$) and thiourea [$SC(NH_2)_2$]. After separation by centrifugation, washing with deionized water, and drying under vacuum, the reduction was performed by heat treatment at 150 – 300 "C. All these products were characterized by TEM. These series of experimental results showed that we have succeeded in preparing the uniform titania nanotubes filled with either gold nanorods or nanoparticles.

References

[1] M. Haruta, *Catal. Today* 36, 153 (1997).
[2] T. Kasuga, M. Hiramatsu, and **A.** Hoson, *Langmuir* 14, 3160 (1998).
[3] G. H. Gu, Q. Chen, R. C. Che, Z. Y. Yuan, and L. M. Peng, ***Appl.*** *Phys. Lett.* 79, 3702 (2001).

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