Preparation of Tungsten(VI) Oxide Flake-Wall Films for Photoelectrochemical Water Oxidation

Ding Li,1 Fumiaki Amano1,2 and Bunsho Ohtani1,2
1Graduate School of Environmental Science and 2Catalysis Research Center, Hokkaido University

Photoelectrochemical water oxidation into oxygen is induced by photogenerated valence-band holes of n-type semiconductor metal oxide electrodes. High incident photon-to-current conversion efficiency has been reported for nanocrystalline films with large interface of semiconductor/liquid. The nanocrystalline films require several micrometers thickness to maximize absorption of incident photon, while increase in the thickness would increase the density of grain boundaries resulting in retardation of electron transfer to the back-contacted conductive substrate. On the other hand, films of plate-like crystallites vertically aligned to the substrates is expected to exhibit photoelectrochemical performance superior to that of films with large number of grain boundaries. Herein, for the first time, tungsten(VI) oxide (WO$_3$) films consisting of crystalline flakes vertically aligned to substrate (flake-wall films) were successfully prepared by wet process and their photoelectrochemical property was investigated.

Tin-oxide coated transparent glass plates were used for conductive substrate and they were coated by nanocrystalline WO$_3$ thin films$^1$ as a base layer for heterogeneous nucleation of crystalline flakes. The WO$_3$ thin film coated glasses were soaked in an ethanol solution of tungsten chloride and heat treated in a Teflon-lined autoclave at 373 K for 20 h. After cooling to room temperature, the substrate was washed, dried, and calcined at 773 K for 2 h in air.

Figure 1 shows scanning electron microscopic (SEM) images of the prepared WO$_3$ flake-wall film. Almost all the flakes were aligned vertically on the substrate. Average thickness and height of flakes were ca. 80 nm and ca. 3 μm, respectively. It was found that WO$_3$ flake-wall films exhibited photoactivity higher than that of films consisting of horizontally-laminated flakes which were prepared using precipitates prepared by the similar reaction conditions for the flake-wall film preparation. Since the crystallinity of flakes was similar between those films, the difference of photoactivities would depend on orientation of plate-like crystallites.

Name: Ding LI

**Position and Affiliation:** Master Course Student of Graduate School of Environmental Science, Hokkaido University

**Postal Address:** North 21, West 10, Kita-ku, Sapporo 001-0021, Japan

**Phone/Facsimile:** +81-11-706-9129/+81-11-706-9129

**Email:** liding@cat.hokudai.ac.jp

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**Biographical Sketch:**

2005 July
Bachelor of Engineering, Xiamen University, China

2006 April-2007 April
Research Student of Catalysis Research Center, Hokkaido University, Japan

2007 April
Enter to Graduate School of Environmental Science, Hokkaido University, Japan