AMTC Letters
Vol. 2 (2010)


# TEM observation of structure and growth of the tungsten oxide nanorods

[1]Tomoharu Tokunaga, [2]Tadashi Kawamoto, [1]Kenta Tanaka,
[3]Yasuhiko Hayashi, [1]Katsuhiro Sasaki, [1]Kotaro Kuroda

[1] Department of Engineering, Nagoya University, Furocho Chikusa-ku Nagoya Aichi 464-8603 Japan
[2] Department of Quantum Engineering, Nagoya University, Furocho Chikusa-ku Nagoya Aichi 464-8603 Japan,
[3] Department of Frontier Materials, Nagoya Institute of Technology, Gokiso Showa Nagoya Aichi 466-8555 Japan

The metal oxide nanorods are widely studied, as the characteristics of the metal oxide nanorods are different from the characteristics of bulk metal oxide. Tungsten oxide nanorods are one of the metal oxide semiconductors can be easily made. Therefore considering its semiconducting properties it is applied for electrical devices [1]. But the growth mechanism of the tungsten oxide nanorods is not clarified yet, and growth control of tungsten oxide nanorods is not succeeded. Then, the tungsten oxide nanorods growth process was observed by in-situ transmission electron microscope (TEM) observation, and the growth mechanism was examined.

The pure tungsten wire of the purity 99.99 % was used as a wire-heating holder, which is able to introduce the gas into TEM, and the holder was inserted in TEM. The tungsten oxide nanorods has been grown up, after the current was flowing through the tungsten wire. The pressure of specimen chamber was regulated by introducing oxygen in TEM through the wire heating holder.

A Hitachi H-9000NAR TEM operated at an accelerating voltage of 300kV, equipped with a Gatan GIF 1000 and a Gatan model 627 CCD camera was used. The gas injection nozzle of wire heating holder permits gas flow around the specimen. The pressure of the electron gun chamber could be kept in the range of $10^{-5}$ Pa while the pressure at the specimen chamber was controlled.

The nanorods began to grow when specimen chamber pressure was kept to $7x10^{-4}$ Pa by introducing oxygen in TEM, and the tungsten wire was heated up to 800 $^{o}$C.

The electron energy loss spectroscopy (EELS) mapping of the nanorods was carried out. It is clarified that nanorods are composed of tungsten oxide, as EELS mapping showed oxygen area and tungsten area of nanorods was placed in the same area (Fig.1). Electron diffraction and high resolution TEM images was shown in Fig. 2. These images identified that nanorods has rhombohedral crystal system of $W_{18}O_{23}$. Finally, images of scanning electron microscopy of the grown wire showed the nanorods are grown on the convex shape of the wire surface (Fig. 3). In another condition without introducing oxygen and reduced specimen pressure of $2x10^{-5}$ Pa, the growth of the nanorods was not observed. These results suggested that edge of tungsten was oxidized, and a nanorod was grown on edge of tungsten. Fig. 4 shows in-situ TEM images of tungsten nanorod, which was observed from (a) [100] and (b) [010], in any seconds growth. The black line contrast was observed on (020) and that contrast moved to <001> in Fig. 4(a). Fig. 4(b) confirmed the fringe on (020) was moved to [001]. These results indicated (020) was priority grown to [001]. Gu et. al. anticipated tungsten oxide nanorod growth mechanism was not VLS mechanism and our results supported them [2].

[1] M. Gillet, R. Delamare, E. Gillet, App. Surf. Sci., 254, 270-273 (2007)
[2] G. Gu, B. Zheng, W. Q. Han S. Roth, J. Liu, NANOLETTER, 2, 8, 849-851 (2002)

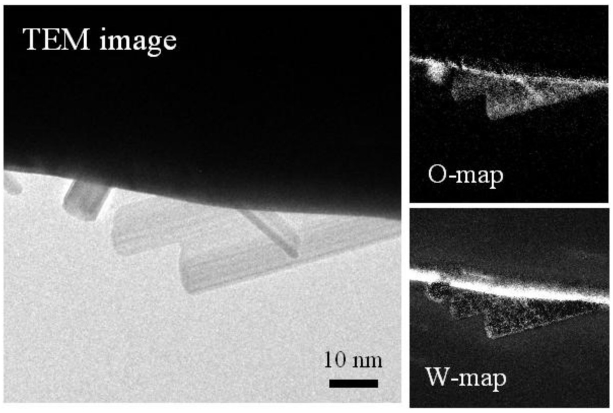



Fig. 1 TEM and mapping image of tungsten oxide nanorod

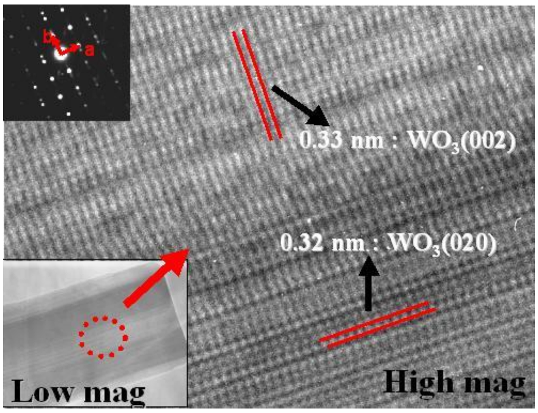



Fig. 2 HRTEM and TEM images of tungsten oxide nanorod

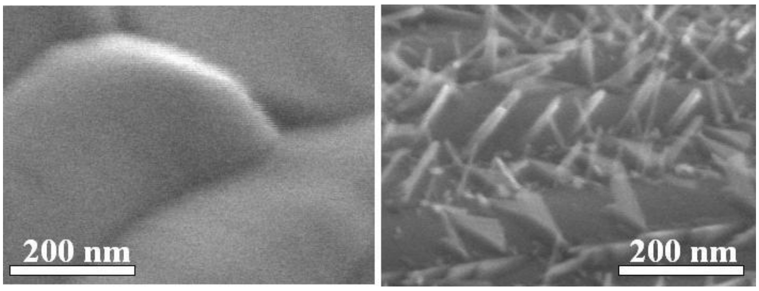
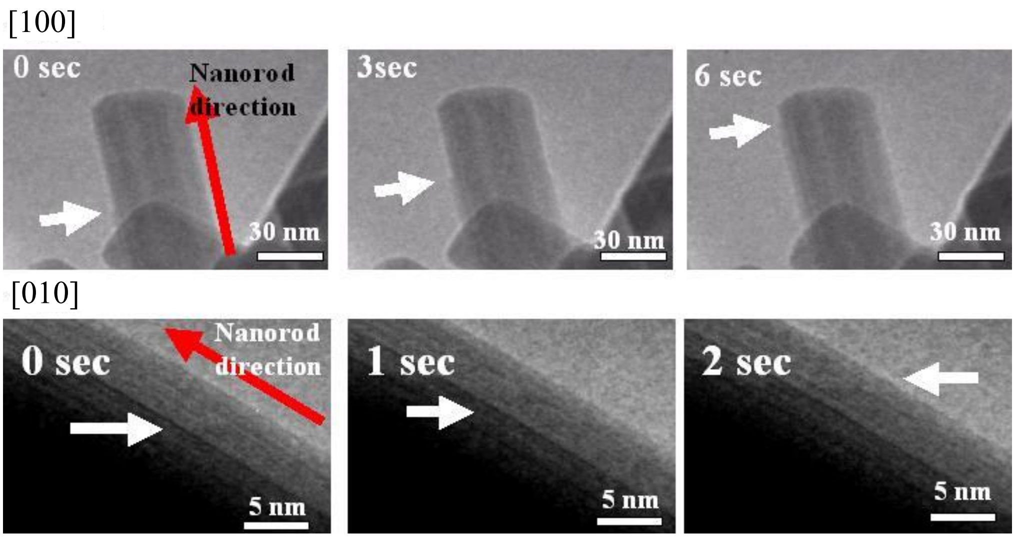



Fig. 3 SEM images of tungsten wire before and after growth




In-situ TEM image of tungsten oxide nanorod from [100] and [010] direction