DOI: 10.1017/S1431927603441688
Microsc Microanal 9(Suppl 2), 2003

High Resolution Electron Microscopy and Spectroscopy Characterization of Tungsten Oxide Nanowires

Jun Jiao, Lifeng Dong, Daniel Carter, and Tatyana Gushtyuk
Physics Department, Portland State University, P.O. Box 751, Portland, Oregon 97207-0751

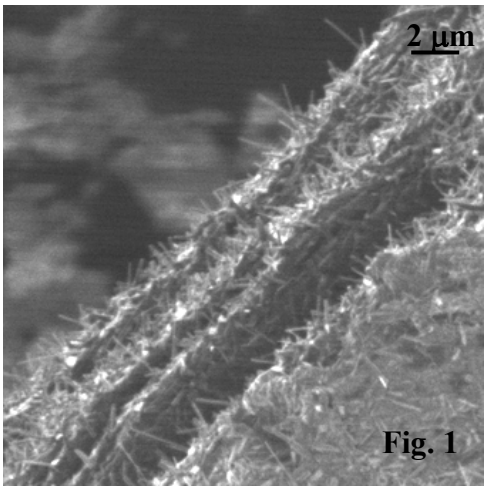
Metallic oxide and semiconductor nanowires have attracted much research interest in recent years due to their potential as building blocks for nanoscale devices and systems [1-2]. Tungsten and tungsten oxide have well-known chemical, physical, electrical, and mechanical properties that make them popular materials in various applications. In an effort to grow tungsten oxide nanowires and to study their electron field emission, we have produced tungsten oxide nanowires by a thermal evaporation method.

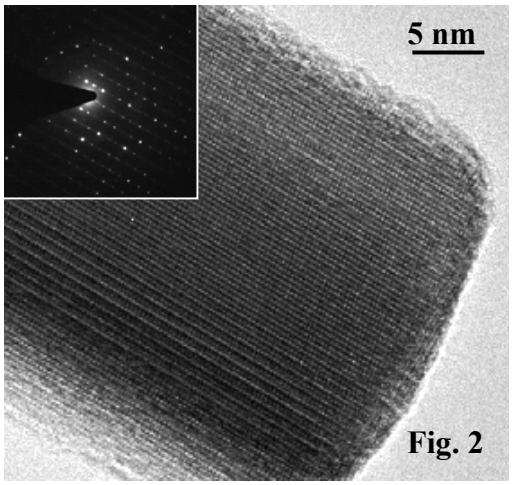
During synthesis, two different growth conditions were tested. First, a piece of tungsten (W) plate was polished and coated with a gold (Au) thin film. The plate was then heated at 900 °C for 30 minutes in an open horizontal quartz tube furnace at atmospheric pressure under a continuing flow of Ar gas. After this process, a layer of gray-colored powder was formed on the surface of the W plate. Secondly, the quartz tube containing a polished W plate without Au coating was sealed and evacuated to 10^{-3} Torr. The chamber was then heated at 1000 °C for 60 minutes with a flow of Ar gas and the reaction pressure was kept at 150 Torr. Again, a thin layer of gray-colored powder was obtained on the surface of the W plate. An FEI 611 focused ion beam (FIB) microscope operated in secondary electron mode was used to characterize the morphologies of the products, while an FEI Tecnai F-20 field emission high-resolution transmission electron microscope (HRTEM), equipped with scanning transmission electron microscopy (STEM) capability and an energy dispersive x-ray spectrometer (EDS), was employed to investigate their internal structures and chemical compositions. TEM specimens were prepared by carefully scratching off the gray-colored powder onto holey carbon TEM grids.

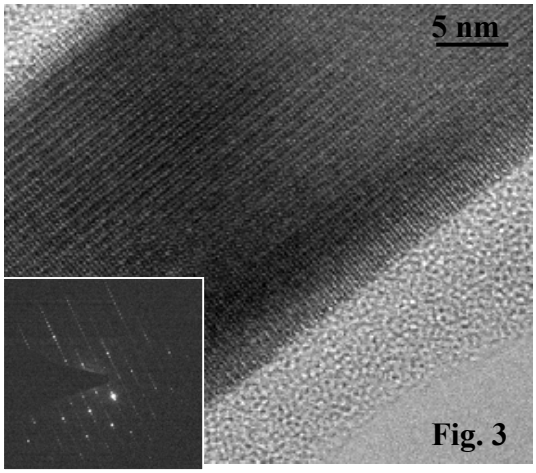
FIB characterization suggests both specimens consist of a high yield of straight nanowires. Fig. 1 shows a secondary electron image of the specimen prepared under low vacuum pressure. The diameter distribution of the nanowires ranges between 15 nm and 100 nm for both specimens. However, a higher percentage of thicker nanowires (50 – 100 nm) was observed in the specimen prepared under atmospheric pressure. Fig. 2 and Fig. 3 show the HRTEM images and diffraction patterns of nanowires obtained from both specimens. The results suggest that the nanowires are single crystalline. The tip of the wire usually terminated in a polyhedral shape (Fig. 2). However, a major difference was that the nanowires produced in the low vacuum pressure were not as stable as those synthesized under atmospheric pressure. During the HRTEM characterization, an electron beam induced amorphous layer was formed on the surface of the low vacuum produced nanowire as shown in Fig. 3. We used the EDS operated in STEM nanoprobe mode to investigate the chemical composition of the nanowires. The qualitative and quantitative analyses suggest that nanowires produced by both preparation conditions are tungsten oxide. Fig. 4 (a) is an STEM image of a tungsten oxide nanowire synthesized on the W plate with the Au coating, while Fig. 4 (b) shows a corresponding line scan spectrum. Note that the Au nanoparticle formed on the side of the body of the tungsten oxide nanowire instead of on the tip of the wire. This growth phenomenon is quite different from the formation of other types of metallic oxide nanowires such as ZnO and SiO_2, in which a Au catalytic particle usually is formed on the tips of the nanowires [3]. The result (Fig. 3) from the second set of preparation conditions also suggests that it is not necessary to use Au as a catalyst to grow tungsten oxide nanowires while keeping the growth in a low vacuum pressure. Although we have not been able to obtain tungsten oxide nanowires without the presence of the Au catalyst under atmospheric pressure, a continuation of this study is underway.

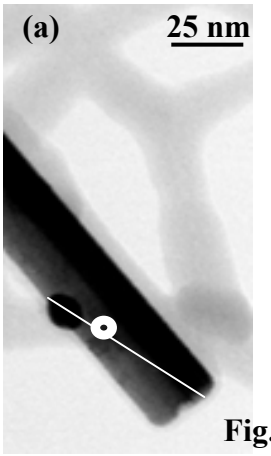
Acknowledgement: Financial support for this research was provided in part by the NSF under awards No. DMR-0097575 and No. ECS-0217061 and Petroleum Research Fund Award No. PRF-38108-G5.

References:
1. T. Ruecks, K. Kim, E. Joselevich, G.Y. Tseng, C. Cheung, C.M. Lieber, Science 289 (2000) 94.
2. M.H. Huang, S. Mao, H. Feick, H. Yan, Y. Wu, H. Kind, E. Weber, R. Russo, P. Yang, Science 292 (2001) 1897.
3. L.F. Dong, J. Jiao, D.W. Tuggle, and J.M. Petty, S.A. Elliff, and M. Coulter, Appl. Phys. Lett. 82 (2003) 1096.









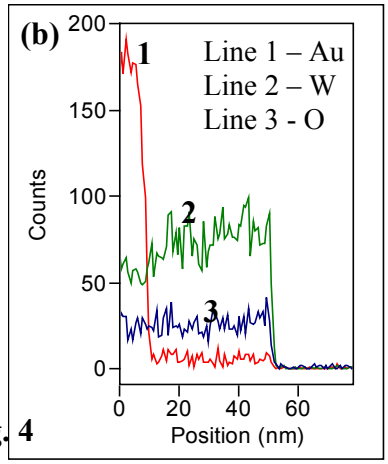


Fig. 1 shows a low magnification secondary electron image of tungsten oxide nanowires produced by evaporating a tungsten plate under low vacuum pressure.

Fig. 2 demonstrates a HRTEM image and its corresponding diffraction pattern of a tungsten oxide nanowire synthesized by evaporating a gold-coated tungsten plate under atmospheric pressure. Note that there is no gold particle on the tip of the wire.

Fig. 3 reveals a similar crystalline structure as the one shown in Fig. 2 from a tungsten oxide nanowire synthesized by evaporating tungsten plate only (without gold coating catalyst) in a low vacuum pressure.

Fig. 4 (a) is a STEM image of a tungsten oxide nanowire, on which EDX line scan analysis was performed. (b) shows the line profiles of each elements collected along the white line in Fig. 4 (a). The white dot represents the nanoprobe of the EDX.

https://doi.org/10.1017/S1431927603441688 Published online by Cambridge University Press