

***In-Situ* TEM Observation of Metal Zn Nanocrystal Growth on ZnO Films**

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The nanocrystal formation has attracted significant attention due to their unique properties and their potential applications to electronic and optoelectronic devices operating with lower currents at higher temperatures [1]. Extensive efforts have been made to fabricate various kinds of nanocrystals on semiconductor substrates. Recently, proximal probes in scanning tunneling microscopy (STM) and atomic force microscopy (AFM) have been suggested as potential tools for the nanocrystal fabrications together with their capabilities of atomic-level imaging and manipulating [2]. One of the most promising techniques is a local anodic-oxidation method using AFM/STM, which can produce a pattern of nanocrystal arrays on surface of semiconductor substrate for fabricating nano-devices [3]. However, the local anodic-oxidation mechanism with elemental rearrangements has not yet been established. Therefore, it is important to investigate the anodic-oxidation mechanism for nanocrystal formation. In this study, the formations of ZnO and Zn nanocrystals on ZnO film through current-AFM (C-AFM) and biasing tungsten-tip were characterized both in *ex-* and *in-situ* transmission electron microscopy (TEM). *In-situ* TEM was performed with a biasing tungsten-tip specimen holder. This *in-situ* approach allows us not only to observe nanocrystal growth in live but also to characterize microstructure rearrangements by electron diffraction followed by elemental analysis.

Figure 1 shows a set of AFM images of (a) as-grown ZnO film on a *n*-Si (001) substrate, ZnO films after applying C-AFM (b) line- and (c) dot-oxidation on the as-grown ZnO film. In addition to the AFM images, a cross-sectional TEM image of the ZnO film after applying C-AFM dot-oxidation is also shown in Fig. 1(d). When a C-AFM conductive tip with a sample voltage of 10 V is positioned on the ZnO film surface, additional oxide layer is formed at the location because OH⁻ and O²⁻ ions may be adsorbed onto the surface due to a strong electric field between the tip and the sample [4]. Furthermore, Zn nanocrystals can be fabricated using biasing tungsten-tip in *in-situ* TEM. In this approach, the size, position and shape of Zn nanocrystals can be controlled. A schematic diagram of the nanocrystal formation by biasing tip is shown in Fig. 2(a). A bright-field TEM image after applying bias to the ZnO film in *in-situ* TEM indicates the Zn nanocrystal formation. Further nanocrystal formation mechanisms based on *in-situ* TEM observation will be discussed [5].

References

- [1] N. J. Craig et al., *Science* 304 (2004) 565.
 [2] D. M. Eigler et al., *Nature (London)* 344 (1990) 524.
 [3] R. H. Kim et al., *Appl. Phys. Lett.* 90 (2007) 172907.
 [4] Y.-R. Ma et al., *Phys. Rev. B* 64 (2001) 195324.
 [5] This work was supported by the National Center for Electron Microscopy, Lawrence Berkeley Lab, which is supported by the U.S. Department of Energy under Contract # DE-AC02-05CH11231.

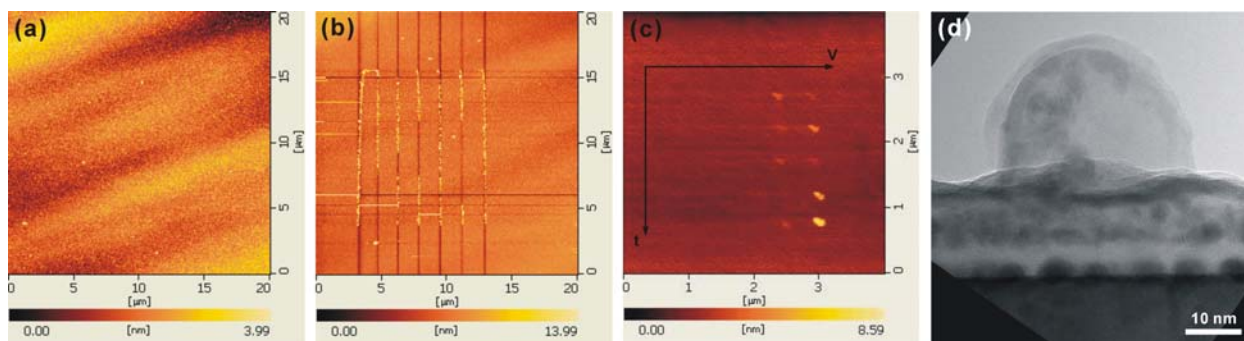


Fig. 1. (a) An AFM image of as-grown ZnO/*n*-Si (001) heterostructures. AFM images of the ZnO film after C-AFM (b) line- and (c) dot-oxidation. (d) A cross-sectional TEM image of dot-oxidation in (c).

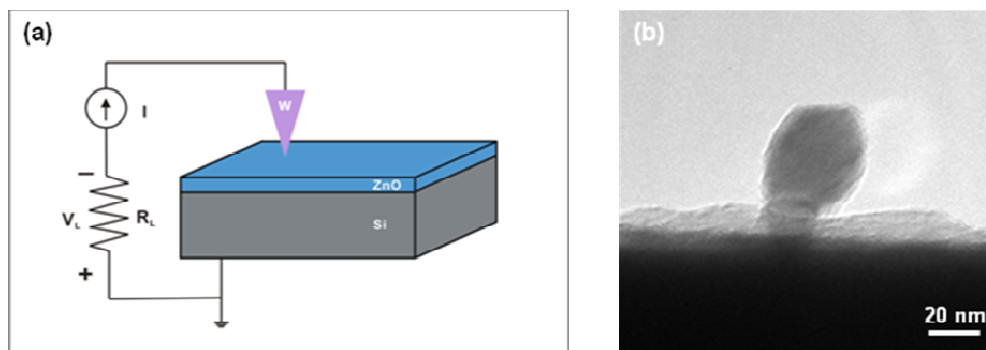


Fig. 2. (a) A schematic diagram of the fabrication Zn nanocrystals using a biasing tungsten-tip. (b) A bright-field TEM image of a Zn nanocrystal fabricated by *in-situ* TEM with the biasing tungsten-tip.