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EFFECT OF DISSOLVED OXYGEN ON ZnO NANOSTRUCTURES GROWN BY WET OXIDATION OF Zn FOIL

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ABSTRACT

This study presents the growth of ZnO nanostructures using wet oxidation of zinc (Zn) foil in water. The effect of dissolved oxygen on the resulting nanostructured ZnO was investigated using scanning electron microscope (SEM) and X-ray diffraction (XRD). Zn foil (99.95 %, 15x15x0.5 mm) was used as the starting material. The foil was ground and polished first and cleaned with acetone for 10 min in an ultrasonic bath. Before oxidation, the foil was etched in 5 wt% hydrochloric acid in ethanol for 3 min. Then the sample was immersed in water at 90°C for 4h. To determine the role of dissolved oxygen, the solution was slowly purged with nitrogen (N₂) or oxygen (O₂) gas.

![Figure 1. SEM images of the ZnO nanostructures formed on Zn foil after oxidation in water for 4 h at 90 °C: (a) ambient air (without purging), bubbled with (b) N₂ gas and (c) O₂ gas. Images labeled with (i) are taken at higher magnification.](image)

Large hemispherical structures consisting of hexagonal flat-topped nanorods developed without purging. The diameter of the nanorods increases outwards with an outermost mean diameter of 440 nm. Diffraction peaks at 31.47°, 34.18° and 36.02° corresponds to the 100, 002, and 101 peaks of hexagonal (wurtzite) ZnO, respectively. Also, the diffraction peaks from the underlying Zn substrate can be observed. Here, throughout the wet oxidation of Zn foil in water, Zn²⁺ ions diffused from the ridges and grain boundaries produced by etching to the Zn surface. At
the same time, the reduction of dissolved oxygen (O₂) and hydrogen (H₂) gas evolution provides hydroxyl ions (OH⁻) resulting to an increase of the local pH near the vicinity of the Zn foil. This process triggers the growth of ZnO nanostructures through the hydrolysis of Zn²⁺ ions. Additionally, the hexagonal-shaped of the nanorods can be attributed to the inherent wurtzite crystal structure of ZnO. It is well known that wurtzite ZnO has polar ± (0001) and non-polar {2110} {0110} planes. Polar Zn-terminated (0001) planes are metastable and chemically active, while the non-polar planes have lower energies and inert [1]. Thus, anions such as OH⁻ ions would be adsorbed specifically onto the polar Zn-terminate positive plane creating 1-D ZnO nanostructures like the nanorods. When the solution is bubbled with N₂ gas, smaller hemispherical structures can be observed comprising of coarse particles. This can be attributed to the reduction in the concentration of OH⁻ ions which resulted from the decreased amount of dissolved oxygen upon purging of N₂ gas. On the other hand, the mean diameter of the nanorods increased dramatically to 750 nm due to the increased OH⁻ ions present in the solution when purged with O₂ gas. It can be seen that in both purged solutions, the resulting nanostructures have rough or coarse surfaces. This could be attributed to the collision of the gas molecules with the precursor materials during the formation of the nanostructures.

![Figure 2. XRD pattern of the etched Zn foil after oxidation in water at 90 °C for 4h without purging.](image)

**Keywords:** Dissolved Oxygen, Wet Oxidation, ZnO nanostructures

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**Reference**