Study on Optimum Low Temperature for ZnO Formation via Hot Water Treatment

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Abstract
The effects of surface oxidation of etched metallic Zn foils by hot water treatment for variable times and temperature investigated. ZnO nanostructures were obtained after undergoing hot water treatment at 50°C and 90°C for 2 to 24 hours. The SEM morphology showed ZnO nano-star-like and nanoneedle-like were observed with different hot water treatment temperature. XRD analysis confirmed the existing of ZnO nano-star-like and nanoneedle-like in a hexagonal-wurzite structure. On the basis of the changes in morphology and microstructure induced by hot water treatment, it is concluded that the formation of ZnO are promising at low temperature.

Keywords: ZnO Nanostructure, Hot Water Treatment, Etched Zn Foils, Low Temperature

Introduction
With large excitation energy 60 meV and wide band gap semiconductor, noncentral symmetry piezoelectric and biocompatible for biomedical application are the unique characteristic that attract many researcher’s attention on ZnO nanostructure synthesis and physical properties [1]. Tan et al., (2012) demonstrated HWT process of etched Zn foils [2] and unetched Zn foil [3]. The studies were carried out without stirring during HWT process at 90°C obtained ZnO nanowires, nanorods and nanosheets structures. Formation of ZnO with low temperature is considered as cost effective. Therefore, it is interesting to investigate the formation of ZnO nanostructure at constant stirring HWT process and optimum low temperature.

Experimental method
The 99.95% pure Zn foils with 0.5 mm thick were cut into 2 x 2 cm being used as oxidation specimens. They were then ground using ascending grit sizes SiC paper, polished with alumina powder and cleaned with acetone in an ultrasonic bath. Prior to hot-water treatment, the Zn foils were chemically etched using 5% HCl in ethanol for 5min. The etched Zn foils were then hot-water treated with 400rpm stirring speed at 50°C and 90°C for 2, 4, 6, 12 or 24 h using distilled water before being dried at room temperature prior to characterization. The morphologies of the ZnO nanostructures formed were examined using an X-Ray Diffraction (XRD), Scanning electron microscope (SEM), and atomic force microscope (AFM).

Results and Discussion
XRD patterns of the synthesis sample are shown in Fig. 1. Both sample exhibited 2θ peaks at 31.77°, 34.42° and 63.25° which correspond to (100), (002) and (101) planes of ZnO (ICDD no : 00-036-1451). Growth of peak on (002) direction confirmed that ZnO with wurzite structure oriented along c-axis. Underlying metallic Zn substrate can be observed at 36.29°, 38.99° and 43.99° which correspond to the (002), (100) and (101) planes of Zn (ICDD no 00-004-0831). Overlaps (002) peak of Zn and (101) ZnO could be due to both of Zn layer
and ZnO nanostructure formed on the surface.

Fig. 1: XRD patterns of etched Zn foils hot-water treated at 4 hours for 50°C and 90°C

Surface morphologies of etched Zn foil after hot water treated for 4 hours at 50°C and 90°C are shown in Fig 2.

Fig. 2: Morphologies of ZnO nanostructure after 4 hour HTW: (a) 50°C and (b) 90°C.

As shown in Fig. 2 (a) about 5.04-5.47μm length of ZnO nanostar-like and 877-991nm diameter were formed after hot water treated at 50°C. Meanwhile with different temperature of HWT bunch of ZnO nanoneedle-like were formed with 2.33-2.43μm length at 90°C. The reduction of in the length and diameter of the ZnO nanostructure was due to dissolution of ZnO upon extended temperature HWT exposure.

Formation of nanocrystals acted as a seed layer resulted from kinetic oxidation due to fast adsorption and breakup of the water molecule on the Zn surface. Supersaturated condition occurred at the region of solution near the surface then the dissolved hydroxide was redeposited onto the surface, to form ZnO as shown in Eq. 1-3.

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\begin{align*}
\text{Zn}^{2+} + \text{OH}^- & \leftrightarrow \text{Zn(OH)}^+ \quad (1) \\
\text{Zn(OH)}^+ + \text{OH}^- & \leftrightarrow \text{Zn(OH)}_2 \quad (2) \\
\text{Zn(OH)}_2 & \leftrightarrow \text{ZnO} + \text{H}_2\text{O} \quad (3)
\end{align*}
\]

**Conclusion**

Highly crystallized ZnO nanostructure were formed on etched metallic Zn foils by a hot water treatment technique at the temperature of 50°C and 90°C for 2, 4, 6, 12 and 24 hours of treatment. ZnO nanostar-like and nanoneedle-like structures were observed and formed at 50°C and 90°C temperature respectively. At 90°C a highly crystalline ZnO were formed at plane (002) ZnO. It is noteworthy that the formation of ZnO nanostructure at temperature of 90°C was preferred via Hot Water Treatment compared to 50°C.

**References**

