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Low Profile ZnO Growth on Si Wafer Using Hydrothermal Method for Sensor Applications

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ABSTRACT

ZnO was grown on a silicon wafer (1 cm x 1 cm) via hydrothermal method in this study. Initially, the zinc oxide colloid solution was spin-coated on the Si wafer at 400 rpm for 30 s for the seeding process prior to the ZnO growth. The presence of ZnO from the growing process was confirmed with the X-ray diffraction (XRD). The crystallite size was calculated to be 6.34 nm using Scherer equation. In addition, an energy dispersive X-ray (EDX) characterization was carried out to further verify the presence of the ZnO. Meanwhile, the scanning electron microscopy (SEM) characterization was done to observe the surface morphology of the sample. It is found that the ZnO structure has a coral-like shape, which is full of asperities and sharp edges. The sample was able to generate a voltage in the range of 180 mV to 240 mV peak to peak under random pressure.

Key words: Zinc Oxide (ZnO), hydrothermal, growth, surface structure, vibration sensor.

1. INTRODUCTION

Zinc oxide (ZnO) is a functional and versatile inorganic material in the II-VI material groups which has been used as early as in ancient Egypt and Rome for adverse skin treatment [1]. In our modern society, it is widely used for paints, as an additive for plastics and rubber as well as a coating material for paper. As for the electronics industry, it is a type of semiconductor with a direct wide bandgap at 3.37 eV (room temperature) and large exciton binding energy (60 meV) [2].

ZnO nanoparticles have been reported to exist in a number of surface morphologies. For instance, they have existed in many forms like nanoflowers, nanotrees, nanobridges, nanonails and nanotbelts [3, 4]. Furthermore, through Yamada and Tobisawa work, they reported more ZnO morphology varieties like plates, columns, pyramids, stellar shape crystal, spheres, whiskers and dendrites under the converging shockwave with an explosive charge [5].

ZnO powder is manufactured industrially by pyrometallurgical or hydrometallurgical processes. Metallurgical methods for the processing of ZnO by oxidation began in Germany in the 1700s [6]. In the 19th century, three large-scale methods, named the direct method (American process), the indirect method (French process) and the hydrometallurgical system, were created to manufacture ZnO pigment [6]. Recently, lots of chemical methods like sol-gel [7], hydrothermal, microemulsions, direct precipitation and microwave-assisted synthesis have been used for the ZnO nanoparticle processing [4, 6]. In addition, other method like radio frequency (RF) sputtering [8] was previously reported. In this context, hydrothermal method is one of the most popular and widely used methods to grow ZnO due to its relatively low-temperature process, environmentally friendly and lost cost [9]. In this paper, ZnO was grown by the hydrothermal method on Si wafer. It aims to understand the feasibility of this method for the ZnO growth on silicon (Si) wafer and to observe the effect on the morphology structure of the grown ZnO.

2. METHODOLOGY

Starting materials: Table 1 summarized all the materials used in this research. They are used without further purification.

Table 1: Starting materials for the ZnO growth

Materials	Provider
Zinc acetate dehydrate ($Zn(CH_3COO)_2.2H_2O$)	Merck
Zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$)	Sigma Aldrich
Hexamethylenetetramine ($C_6H_{12}N_4$)	R & M Chemical
Sodium Hydroxide (NaOH)	Sigma Aldrich
Acetone	Merck
2-Propanol	Merck

Substrate preparation: n-type Si wafer (1 cm x 1 cm) with a thickness of ≈ 0.525 mm was used as the substrate in this study. The substrate was immersed in deionized water and was cleaned in an ultrasonic bath for 10 minutes. Then, it was immersed in acetone and 2-propanol and was clean in an ultrasonic bath for another 10 minutes each. Finally, the substrate was dried on a hot plate at 70 ^oC.

ZnO seed: 0.1 M of ($Zn(CH_3COO)_2.2H_2O$) was dissolved in 2.5 ml of deionized water and 2.5 ml of NaOH. Then the mixture was stir for 30 mins using a magnetic stirrer at a temperature of 60 ^oC. Then the solution was left for 24 hours in a dry box. After 24 hours, the prepared solution was spin-coated on the cleaned Si wafer for 30 s at a speed of 400 rpm. Then the coated Si wafer was heated at 60 ^oC for 10 min. Then, the Si wafer was re-coated with the similar method for a total of 3 times. Finally, the coated sampled was annealed at 300 ^oC for 1 hour.

ZnO growth: The ZnO growth was carried out in the zinc precursor solution via the hydrothermal method. 0.04 M of $Zn(NO_3)_2.6H_2O$ was added with 0.04 M of $C_6H_{12}N_4$ and 20 ml of deionized water. The prepared Si wafer with the ZnO seed layer was immersed in the precursor solutions in a beaker. Then, the beaker was sealed and heated to 95 ^oC and left for 30 minutes. After that, the prepared Si wafer was cleaned with distilled water for a few times and left to dry.

Characterization: The phase structure of the prepared sample was characterized by X-ray diffraction (Rigaku Miniflex II) with CuKα radiation. To further verified the presence of the elements, energy dispersive X-ray (EDX) analysis was carried out (Oxford Instruments Ultim Max 40). Meanwhile, for the surface morphology structure of the sample was observed using a scanning electron microscope (SEM; JEOL JSM-6350LA).

In addition, the sample was tested for electrical validation caused by applied stress. The electrical response of a ZnO contained cantilever can suggests the sensitivity of the sample as an effective sensor. Higher sensitivity implies better sensing performance. The sensitivity can be detected by both applying a load or without a load. The latter is called open load mode. As such, the testing of the sample was performed in open load mode, i.e., no resistive load was attached. A dual-channel digital oscilloscope was used to detect the induced voltage from the sample. In order to create an inductive surface on the sample, aluminium thin sheet was used which collects the induced effect from the sample. The top and bottom of the sample were connected with the oscilloscope probes to detect the voltage variations. The collected voltage are presented in the following section.

3. RESULTS AND DISCUSSION

Figure 1 shows the XRD spectrum of the ZnO that was growth on the silicon wafer. From the figure, only two peaks are observed and these two peaks are corresponding to ZnO (PDF No. 65-2880) at 33.6 ^{o} and 70.5^o as well as Si (PDF No. 40-932) at 33.2 ^{o} and 68.6^o. The full width at half maximum (FWHM) and the grain size of the ZnO (100) peak at 33.6^o were calculated as 1.3673 and 6.34 nm, respectively. The crystallite size value is connected to the crystallinity process, where the nucleation rate involves in determining the crystal quality and orientations of ZnO particles [10]. In addition, Aneesh et al. reported that the crystallite size of the ZnO nanoparticle increases when the hydrothermal growth temperature increases [11]. From their work, they reported that the crystallite size was ranged from 7 nm to 16 nm for the temperature range of 100 ^oC to 200 ^oC.

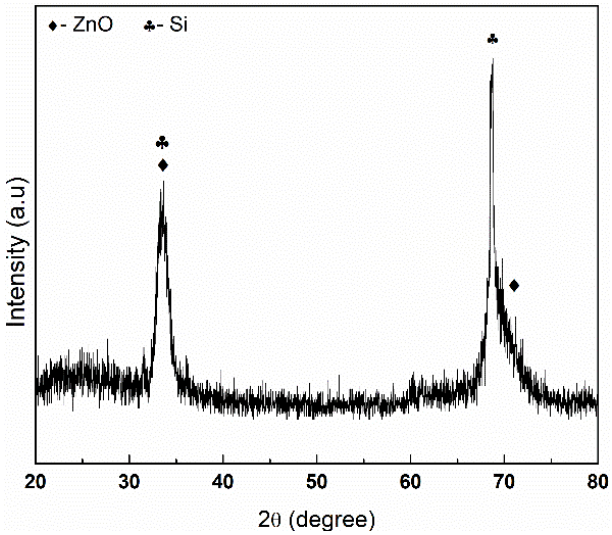

Figure 1: XRD spectrum of the growth ZnO

Since these two peaks representing 2 elements, further characterization was carried out using EDX to verify the presence of elements. The EDX characterization of the sample was done at 505 times of magnification with 5 kV of power. Through this characterization, the presence of Zn and Si in the sample is verified (Figure 2). The ZnO was proved to be grown homogenously on the Si substrate which can be deduced from colour distribution on the EDX image of the sample. Thus, it shows that the ZnO was grown successfully on the Si wafer via the hydrothermal method.

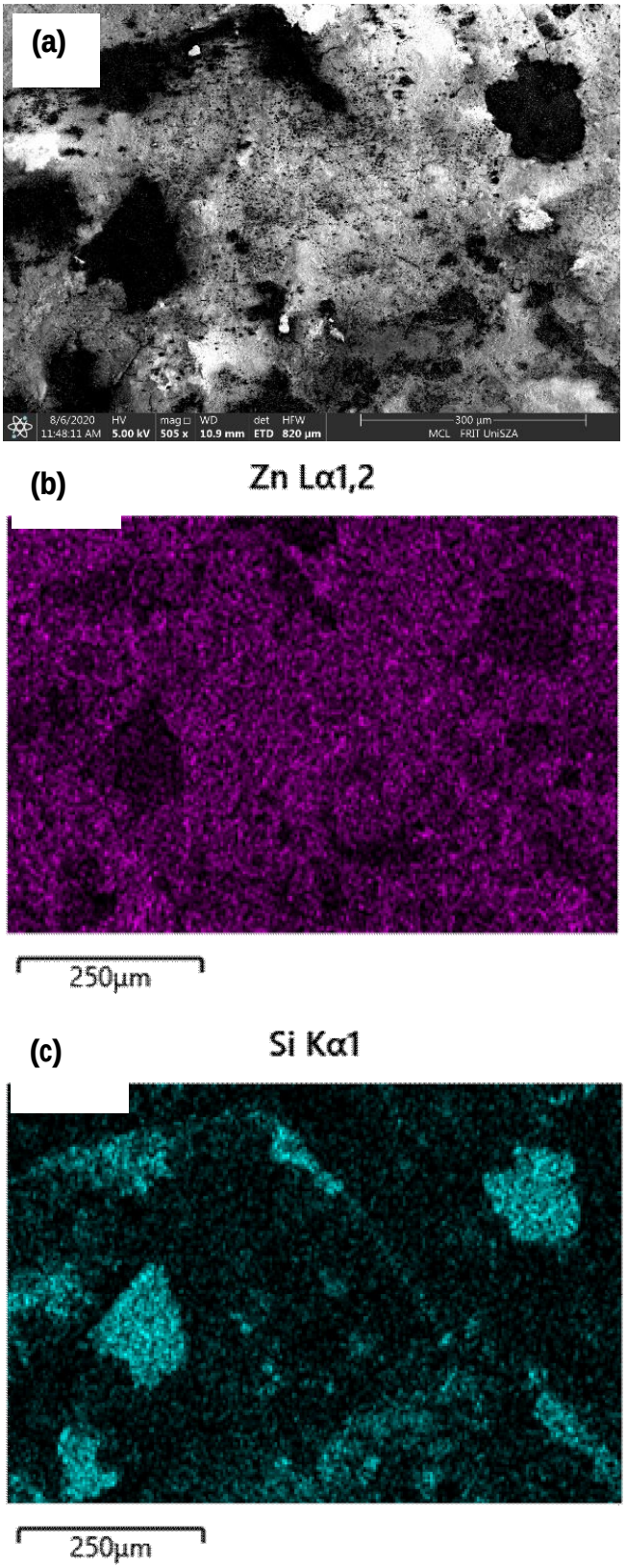

Figure 2: EDX image of the growth sample: (a) actual SEM image, (b) Zn element and (c) Si element

SEM characterization was carried out to study the surface structure of the ZnO growth on the Si wafer. The surface structure was examined at 5000 and 10000 times of magnification (15 kV) as shown in Figure 3(a) and (b). Along with them, Figure 3(c) presents the layers formed due to the deposition. From the figures, interestingly, the ZnO structure was found to be a coral-like shape, which is full of asperities and sharp edges. The particle shape may occur due to a relatively low synthesis temperature and growth period. For instance, Fawcet and Poinern [12] reported that they found that the ZnO had hexagonal rods shape, flower-like shapes and fern-like shapes in a similar sample after 60 minutes of ZnO growth period on silicon chip wafers.

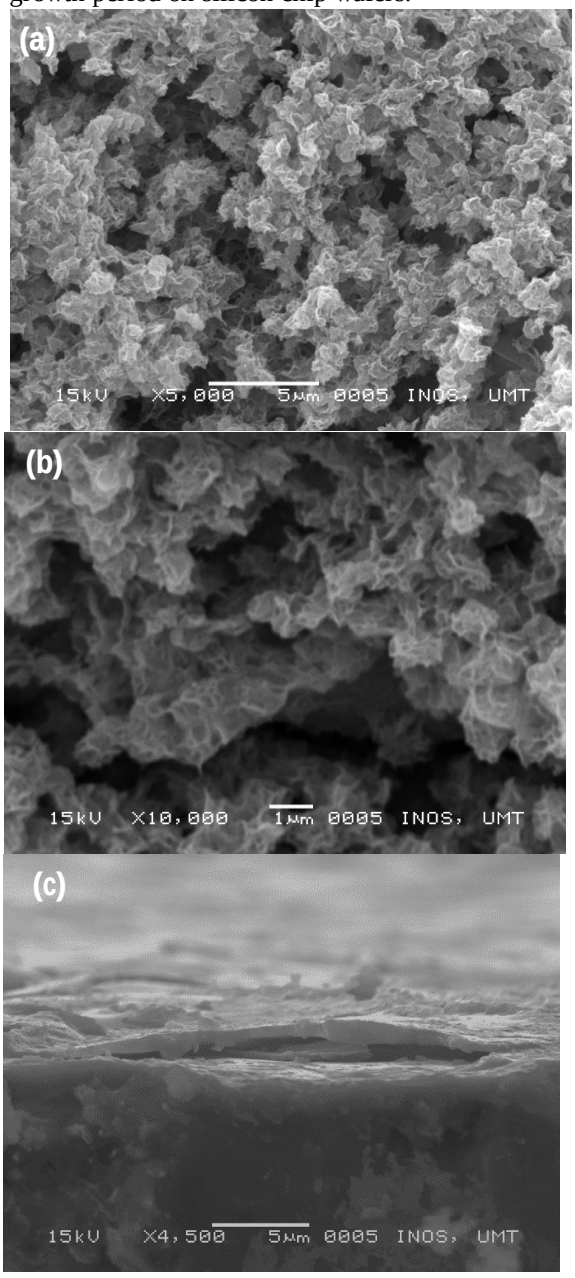

Figure 3: SEM image of the ZnO at (a) 5000 x magnification (b) 10000 x magnification and (c) 4500 x magnification with layered exposure

The fabricated sample was tested under random pressure to verify its electrical properties (in voltage) against applied random pressures. The pressure was produced by simple finger generated impact on the sample. The pressures were increased linearly in the consecutive tests. The observed produced voltages were 180 mV to 240 mV peak to peak from test 1 to test 4. The output voltage implies the effectiveness of the fabricated sample despite the simplicity of the fabrication. In fact, the sample can be treated for comparison in the context of induced voltage [11]. Hence, this low profile cantilever can be used for ultra low frequency vibration sensor. The results are summarized in Table 2.

Table 2: Induced voltages by the sample

Test	Induced Voltage mV pp
Test 1	180
Test 2	196
Test 3	200
Test 4	240

5. CONCLUSION

ZnO was growth on a Si wafer in this study through the hydrothermal method. From the XRD characterization, the presence of ZnO was confirmed. This output was further verified with the EDX characterization that confirms the presence of Zn element that is well distributed in the sample. The crystallite size was calculated to be 6.34 nm. From the SEM analysis, the ZnO was found to has a coral-like shape, which is full of asperities and sharp edges. The sample was able to generate a voltage in the range of 180 mV to 240 mV peak to peak under random pressure.

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