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Quartz Tuning Fork as Humidity Sensor

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Abstract

This paper highlight, a novel and simple solution growth method to develop the deposition of nanocrystalline ZnO films on QTF to act as sensor, integrating merits of both SILAR and CBD techniques. The obtained film is characterized by high deposition rate, high film and used for the humidity sensing. And this chapter ends with the experiment done for measuring the frequency shift using PSoC and LabVIEW, which measures the shift in resonance frequency of QTFs due to the additional mass loading.

Keywords: PSOC; Lab view; SEM; XRD; Quartz tuning fork

Introduction

QTF coated with nanocrystalline ZnO thin film is used as humidity sensor and their humidity sensitivity characteristics have been investigated. It is experimentally demonstrated that QTF coated with nanocrystalline ZnO films shows the promising application for humidity sensor. The nanocrystalline ZnO thin films were deposited on the QTF by SILAR method followed by Chemical Bath Deposition. The film was characterized by X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) to obtain the information on the structural and morphological properties. And the humidity sensing characteristics of the sensors have been investigated. Sensing experiments were examined by measuring the shift in resonance frequency of QTF due to the additional mass loading. The results showed that the sensors had high humidity sensitivity, good stability, fast response time and well reproducibility.

Experimental Details

First nanocrystalline ZnO Seed layer is coated on thin film on QTF by SILAR method [1]. This thin film serves as a seed layer for the subsequent growth of nanorods in aqueous solutions at low temperature. The resulting films of highly ordered nanorods have the reproducibility and uniformity in large areas, which can potentially be employed in the development of electronics device like Humidity sensor (Figure 1) among several solutions for growth technologies, SILAR and CBD are two methods widely used to prepare ZnO layer from aqueous solution.

Sensor configuration

Preparation of seed layer using SILAR method: Quartz Tuning Fork, with resonance frequency of 32,678 Hz is used for this application as substrate. QTF is obtained by removing the metal lids and the tuning forks beam was exposed.

First, QTF has to cleaned to free from contamination, it is cleaned by placing it into acetone for 30 min and then washed by anhydrous ethanol and dried in a baking box. ZnO thin films were prepared by SILAR method followed by CBD to form the thin film on the QTF. As precursor zinc acetate dihydrates is dissolved into 100 ml of absolute ethanol at 60°C. After stirring for 1 hour, a clear and homogeneous solution was obtained, which acted as the precursor solution for QTF. Then after this by SILAR technique is followed. QTF is dipped for about 10 seconds. And then the coating was dried in the electronic furnace for 40°C to evaporate the ethanol solvent primarily [2]. After evaporation the ethanol solvent, the QTF have been subjected to annealing in the air in the electronics furnace at 350°C for 30 minutes. Then the white films were found on the surface of the QTF, these white films being the ZnO thin film.

Growth process in the SILAR method: Analytical reagents of zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$) were dissolved in diluted ammonia to get zinc ammonium complex solution, which was then served as zinc cation precursor. Double distilled (DD) water kept at 80°C was used as anionic precursor. The SILAR growth involves in the subsequent immersion of cleaned QTF in cationic and anionic solution along with rinsing the QTF in between in DD water kept at room temperature. In the first step, the QTF was immersed in a beaker containing Zinc ammonium complex, where Zn^{2+} with ammonia formed zinc ammonia complex ($[Zn(NH_3)4]^{2+}$). In the first immersion process, zinc ammonia was adsorbed onto the QTF. In the second step, the zinc ammonia adsorbed QTF was immersed into the beaker containing DD water, where the a adsorbed zinc ammonia complex was converted into zinc hydroxide ($Zn(OH)_2$). As a third step, the $Zn(OH)_2$ coated substrate was subjected to ultrasonic agitation to remove loosely bonded zinc hydroxide ($Zn(OH)_2$) molecules. Finally, the zinc hydroxide coated substrate was immersed into the DD water bath which was maintained at 80°C, where $Zn(OH)_2$ was converted in to solid ZnO film. Each immersion step took a time period of 40, 20, 70 and 20 s respectively [3]. A drying period of 15 s was maintained before the start of another deposition cycle. Figure 2 schematically shows the detailed SILAR procedure for the deposition of ZnO nanostructured seed layer thin films. In this present work, ZnO seed layer were deposited for 25 cycles.

ZnO nanorods growth by CBD process: In a typical synthesis process, ZnO seed layer film was used for the growth of ZnO nanorods by a simple CBD. The chemical bath aqueous solution was prepared by mixing equimolar (0.05 M) aqueous solution of zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$) and hexamethylene tetra ammine (HMT) ($C_6H_{12}N_4$). Then, ammonium hydroxide (28 wt% NH_3 in water) was added to alter the pH of the solution. The prepared ZnO

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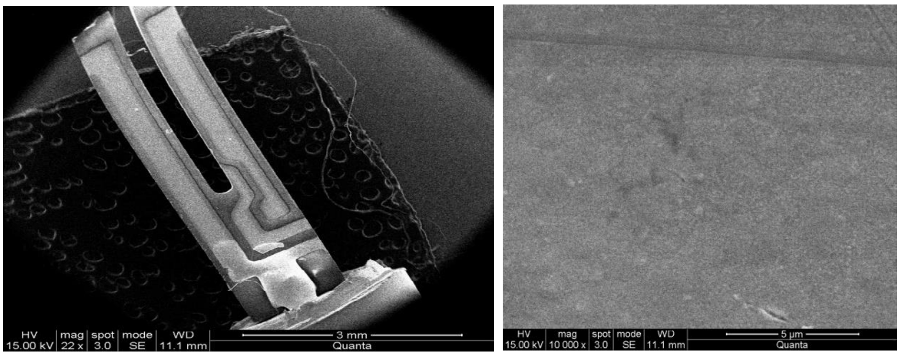

Figure 1: SEM picture of encapsulated QTF and uncoated QTF taken using ICON Analytical (FEI QUANTA 200) Scanning Electron Microscope.

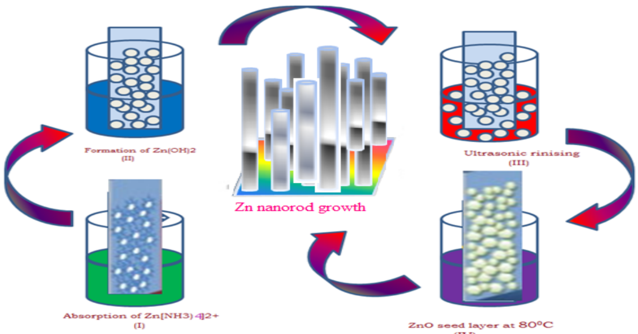

Figure 2: Schematic growth illustrations of ZnO nanorods.

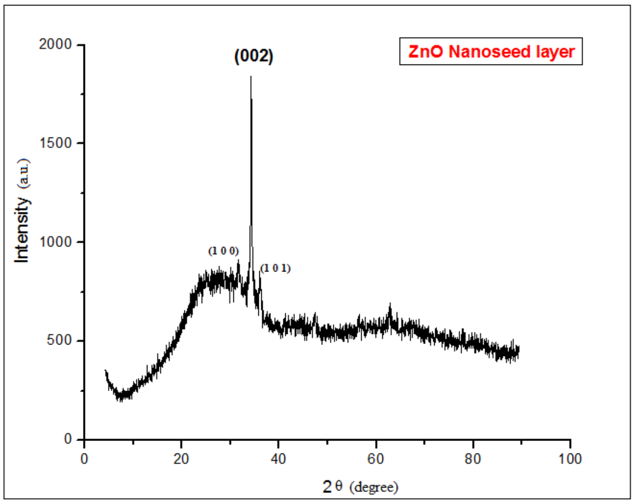

Figure 3: XRD pattern of SILAR grown ZnO nanostructure seed layer taken using PAN alytical (X'PERT-PRO) X-Ray diffractometer.

seed layered substrates were placed vertically inside the chemical bath at a constant temperature of 70°C for 6 h. After the completion of the reaction, the constant temperature bath is allowed to cool down to room temperature. Finally, as-grown CBD films were carefully washed with deionized water and acetone for several times to remove residual salts and then dried at room temperature for further characterization. Finally, ZnO nanorods stability behaviors were subjected to humidity sensing [4-7].

Characterization studies of ZnO coated QTF

The structural, morphological properties of QTF coated with Nanocrystalline ZnO using SILAR followed by CBD were examined by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) [8]. The XRD diffraction analysis is carried out using PANalytical (X'PERT-PRO) X-Ray diffractometer with cu Kα radiation (λ=1.54056 Å). The SEM images were recorded using ICON Analytical (FEI QUANTA 200) Scanning Electron Microscope and particle seem to be uniform distribution of ZnO rods like crystal is formed on the layer of QTF. The humidity sensing is done by means of aqueous solution showered on the QTF which sense the humidity (Figure 3) and readied by coated QTF's shift in resonance frequency are measured using PSoC and Lab VIEW.

Structure morphology analysis

The XRD pattern of SILAR grown ZnO nanostructure seed layer deposited at 25 cycles (Figure 4). The XRD pattern from the deposition cycle has shown the diffraction peaks at 2θ equal to 34.64, which is corresponding to (0 0 2) orientation of wurtzite hexagonal structure of ZnO (JCPD no.36-1451). In particular, it was clearly observed that the intensity of the (0 0 2) peak enhanced with the increase in the number of deposition cycle. In addition to ZnO peak, a weak intensity peak corresponding to $Zn(OH)_2$ phase has higher intensity compared to the higher deposition cycled films.

The XRD pattern of the ZnO nanostructured films grows using the CBD method (Figure 5). The (0 0 2) diffraction peak exhibited a higher intensity, showing a fact that the CBD grown ZnO is better aligned along (0 0 2) directions indicating that this particular seed layer condition is more suitable for the nanostructured films growth [9]. In addition to the (0 0 2) peak, we also observed other peaks such as (1 0 0), and (1 0 1) which are corresponding to the hexagonal ZnO phase. The diameters of the ZnO nanorods ranged (depending on the growth conditions) from only a few hundred nanometers to about 1 micrometer.

Morphology analysis

Seed layer morphology: The SEM images of the ZnO nanostructured seed layer films prepares at deposition of 25 cycles and has random distribution of spindle like structures, randomly distributed spindle-like (i.e. having a circular cross-section and tapering towards each end) structure with length ranging from 100 µm and has spot of 3.0 with magnification of 446 and formation of seed layer were observed (Figure 5). Further increase in the seed layer deposition cycle to 25 has resulted in random distributing of ZnO nanocrystals have hexagonal shape with uniform coating throughout the QTF.

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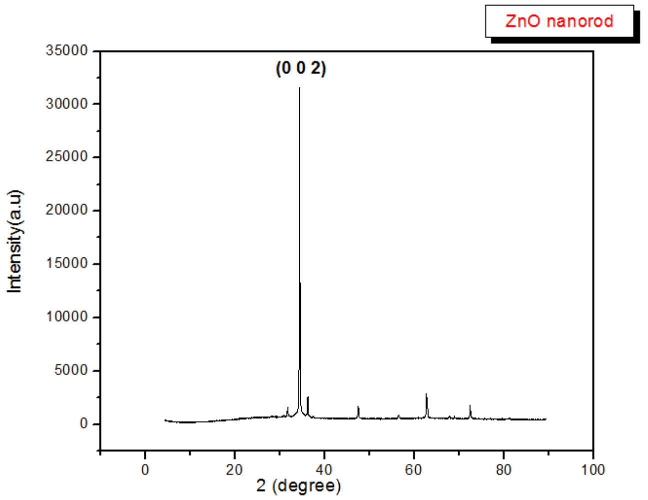

Figure 4: XRD pattern of nanocrytsalline ZnO nanorods deposited on the QTF.

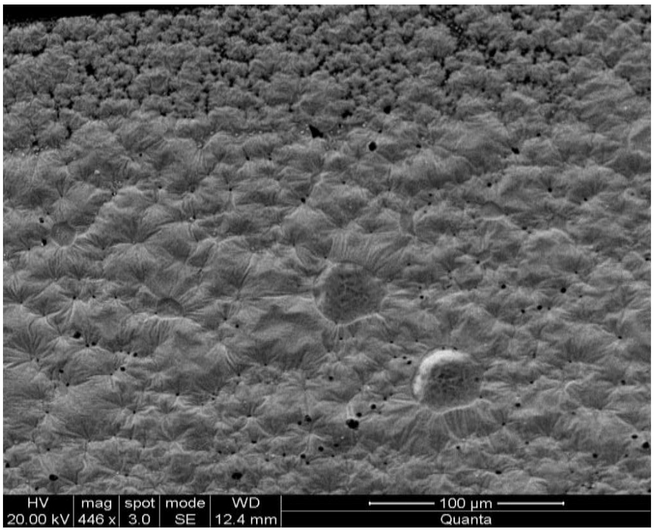

Figure 5: SEM of nanostructure seed layer growth on prepared by SILAR method using ICON Anakytical (FET QUANTA 200) Scanning Electron Microscope.

Nanorods growth morphology: Figure 6 shows the SEM image of CBD grown ZnO nanostructure film over a 25 cycle nanorods layer deposited on QTF. Interestingly the surface morphology shows vertically aligned ZnO which has magnification of 24,000 and spotted with 3.0 of HV of 25,00 kV has the diameter range of 2 µm observed to be uniform nanorods. From this observation we note that the seed layer deposition cycles is a key factor for the vertical growth of ZnO nanorods [10]. In general lower deposition cycle i.e. below 20 the cycle, ZnO seed layer growth is at an initial stage and therefore the orientation along (0 0 2) direction is very weak. But the when the deposition cycle is 25 cycle hexagonal shaped ZnO phase showed relatively strong XRD diffraction intensity along the (0 0 2) direction is proved to be sensitive towards the humidity.

Humidity measurement: When the saturated aqueous solution of LiCl, K_2CO_3, $Mg(NO_3)_2$,KCl and $CuSO_4$ is used in the closed glass vessel at the temperature of 25°C humidity environment is achieved. Using this environment [11-16], QTF is dropped into the solution. Humidity

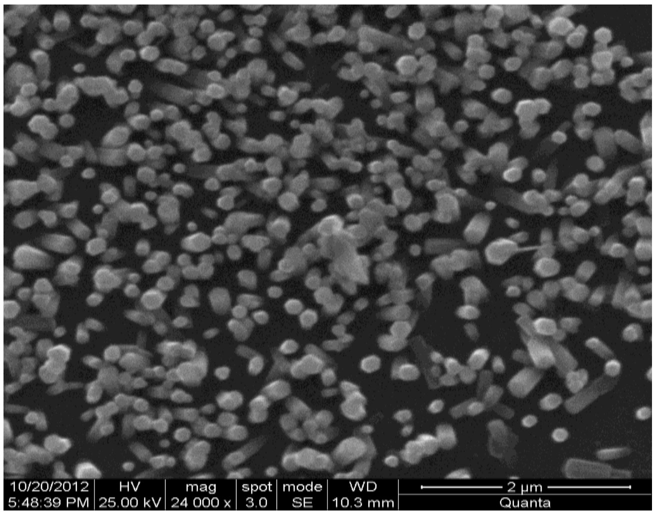

Figure 6: ZnO nanostructure of CBD growth SEM image.

Mean frequency	32765.39 Hz
Standard deviation	1.298187
Minimum	32762 Hz
Maximum	32768 Hz

Table 1: Statistical details of the recorded data (300 reading has been taken).

will act on the coated layer and corresponding shift in frequency occurs. This oscillation is achieved due to the coating of nanocrystalline ZnO on QTF. Finally, it is sensed by PSoC, a menu driven graphical user interface program written in LabVIEW does on-line acquisition of QTF data, on-line plotting, saving the data in user specified file path has been carried out and the reading (Table 1).

User interface in LabVIEW

The values of frequencies measured on the PSoC are fed to a PC. The data is acquired by LabVIEW which offers a graphical user interface. A serial data acquisition module has been used in LabVIEW which collects data from a COM port. The block diagram and front panel of this VI has been illustrated in Figure 7. These values are displayed on the LabVIEW front panel and also a waveform chart is used to plot these data.

In this study QTF sensors were used to measure the response to the relative humidity. And the response of a blank uncoated QTF is also investigated to make sure that the sensing response to relative humidity is actual behavior of the ZnO film.

Result and Discussion

This related LabVIEW and graph can explain by a model based on the absorbed water and the mass increased on the beam of QTF. The deceased in shift in resonance frequency of the QTF with increased relative humidity in the film is related to the absorption of water vapor on the large film surface with capillary pores. The water molecules are absorbed on the grain surface and in pores. The higher surface area provides more suited for water absorption. When the QTF was placed in a humidity environment, the water molecules were absorbed on the surface of the film and in the pores. Then the mass on the QTF increased.ie resonance frequency of the QTF decreases.

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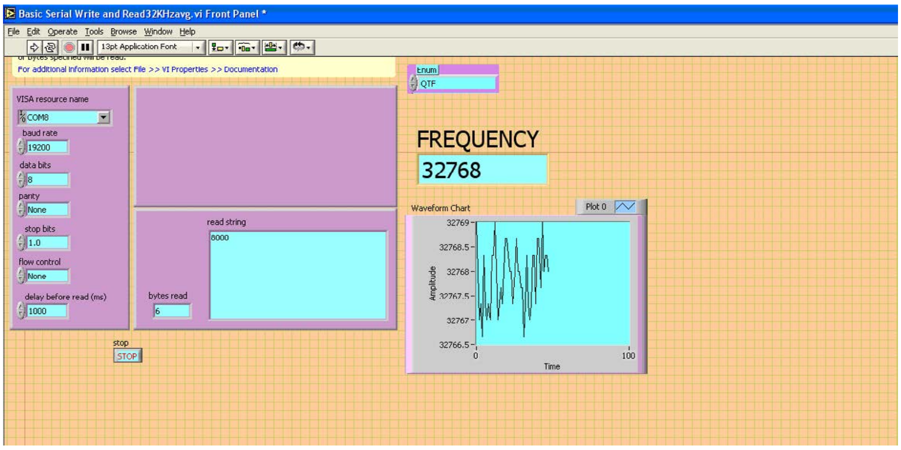

Figure 7: Front Panel of LabVIEW Program of PSoC embedded design for QTF for humidity measurement.

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