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Humidity-sensing properties of a ZnO nanowire film as measured with a QCM

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ABSTRACT

The humidity-sensing properties of ZnO nanowires synthesized by carbothermal catalyst-free vapor solid (VS) technique were studied. The morphology and the crystal structure were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively. The humidity adsorption and desorption kinetics of the synthesized ZnO nanowires were investigated via quartz crystal microbalance (QCM) measurements. The observed positive frequency shift of ZnO nanowires when loaded on the QCM crystal under varying relative humidity conditions can be explained in terms of visco-elastic variations in their mechanical stiffness.

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1. Introduction

Humidity sensing is very important for its control in many manufacturing environments such as those of the food, automotive, electronics, and agriculture industries. Therefore, reliable, cheap, sensitive, and small-sized humidity sensors that operate at low temperature are indispensable [1-3]. A wide variety of humidity sensors based on ceramic, polymeric, and composite materials have been fabricated; but each of them has some drawbacks. Therefore, research on both new sensor concepts as well as the use of new materials continues. Ceramic sensors fabricated from a variety of semiconducting oxides and porous materials need an integrated heater to recover their humidity-sensitive characteristics. Polymeric sensors do not need a heating process, but they have been found to be less sensitive compared to ceramics [4]. In recent years, nanostructure materials have attracted great attention as materials for sensing humidity due to their superior features, such as their very high surface to volume ratio, lower cost, and the ease with which they can be fabricated as compared to bulk or thin film counterparts. Among them, ZnO is one of the most promising materials for sensor applications, because it can be grown in different morphologies without much effort and it has chemically active defect sites which enable high sensitivity quite regardless of the chemical environment in which they are employed [5]. The performance of the sensors strongly depends on operation temperature. There are few published works that investigate the

performance of humidity sensors based on ZnO nanostructures for room temperature applications. Zhang et al. [6] showed that resistance changes of more than four of magnitude can be realized when ZnO nanowire devices were exposed to a moisture pulse of 97% relative humidity at room temperature. Zhou et al. [7] presented a wireless relative humidity sensor prototype based on quartz crystal microbalance coated with nanostructured ZnO as the sensing element. Wang et al. [8] developed a humidity sensor that combined a quartz crystal as a transducer with ZnO tetrapods as a sensing element. ZnO tetrapods allowed humidity measurements at maximum sensitivities of nearly 100 Hz/% relative humidity for experimental environments having relative humidity in the range of 40-80%. Zhou et al. [9] described an application of quartz tuning forks coated with nanocrystalline ZnO films as a relative humidity sensor. Erol et al. [10] investigated the humidity adsorption and desorption kinetics of ZnO nanoparticles via QCM measurements at room temperature, observing nearly 40 Hz/% sensitivity for environments of 45-88% relative humidity.

Here we report the investigation of the humidity-sensing capabilities of ZnO nanowires synthesized by the process of carbothermal catalyst-free VS technique. The morphology and crystal structure of synthesized nanostructures were investigated by SEM, and XRD measurements, respectively. The humidity-sensing capability of ZnO nanowires was evaluated using QCM measurements. Under various relative humidity conditions, a positive QCM frequency shift was observed for ZnO nanowire films coated on the quartz crystal. This unusual QCM frequency change due to the ZnO nanowires films can be attributed to visco-elastic changes in the mechanical stiffness of the ZnO nanowire film on the QCM elec-

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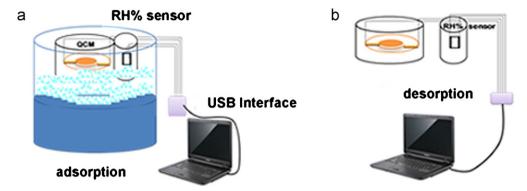


Fig. 1. The experimental setup used to measure the adsorption (a) and desorption (b) kinetics of ZnO nanowires film under accumulated vapor above water level inside a half filled open container using hybrid system of QCM electrodes and a commercial humidity sensor.

trodes during the adsorption process which was performed in a high humid environment.

2. Experimental

2.1. Preparation and structural characterization of ZnO nanowires

The synthesis of ZnO nanowires was carried out in a horizontal alumina tube. A mixture of pure ZnO powder (Aldrich, 99.999%) and graphite powder (Fluka, $\sim\!300$ mesh, 99.9%) in a 1:1 molar ratio was used as the starting material and placed in the central region of the alumina tube (66 cm in length and 2 cm in inner diameter) in an alumina boat (40 cm in length). Si (100) substrates were placed in the low temperature zone (400 °C) of the alumina tube. The ZnO and graphite mixture was heated to approximately 1100 °C under a flow of high purity (99.999%) argon carrier gas at a rate of 300 sccm. After 30 min of process time, a layer of white colored cotton-like material was found deposited on the Si substrates.

The growth process is based on a non-catalyst vapor-solid (VS) mechanism, instead of the more commonly employed vapor-liquid-solid (VLS) mechanism that necessitates the use of a metal catalyst such as Au and Cu [11–15]. In this process, graphite powder is used as a reducing agent. Between 800 and 1100 °C, graphite reduces ZnO to form Zn and CO/CO₂ vapors. As a result of the reaction between Zn and CO/CO₂, ZnO nanocrystals can be synthesized. Advantageous to this method is graphite's ability to significantly lower the decomposition temperature of ZnO. Moreover in the catalyst-free VS process, the nanostructures are produced via their condensation directly from vapor phase, therefore preventing the incorporation of metal impurities at the tips of the nanowires, which in contrast can more commonly occur in the catalyst-assisted VLS technique.

SEM and XRD (with Cu $K_{\alpha 1}$ line, λ = 1.5418 Å) were used to determine the morphology and crystal structure of the synthesized ZnO nanostructures, respectively.

2.2. Monitoring of the humidity-sensing capabilities of ZnO nanowires

The QCM is very useful for evaluating the humidity-sensing capability of an investigated material in a chemical environment (via studying adsorption–desorption kinetics) before the actual fabricating stage of a sensor is implemented. The technique relies on the measurement of the frequency changes of the QCM quartz crystal after it has been coated with the humidity-sensing material at the environmental conditions at which the sensor will be ultimately used. The frequency shift is related to both the amount of adsorbed

mass and the physical properties of the film that has been coated onto the quartz crystal [16–18].

The QCM was used to measure the change in the resonance frequency of the quartz crystal between gold electrodes (using a USB interface connected to a computer). In order to prepare the QCM electrodes, gold-coated quartz crystals were placed in ethanol and ultrasonically cleaned and then rinsed with de-ionized water. After drying with under a low flow of argon gas, the quartz crystal was placed into Teflon housing and the reference frequency was recorded. ZnO nanowires were dissolved in ethanol using an ultrasonic cleaner and drop-casted onto the gold-coated quartz crystal. The changes in the QCM frequency were monitored during evaporation of the ethanol under an argon gas flow until the ethanol was completely volatilized allowing the frequency to stabilize.

Fig. 1 shows the experimental set-up used to measure the adsorption (a) and desorption (b) kinetics of ZnO nanowires inside a half filled open container using a hybrid system of QCM electrodes and a commercial humidity sensor. Signals coming from both the QCM and the commercial RH sensor were simultaneously measured during the adsorption process. Then, ZnO nanowires as coated on the QCM electrode surface and the commercial RH sensor were exposed to ambient air conditions (\sim 23 °C and \sim 48% RH) during the desorption process. Both RH and temperature were recorded during the experiments. A EI-1050 selectable digital relative humidity and temperature probe with a response time of 4s and a resolution of 0.03% RH was used as the commercial humidity sensor with a USB controlled LabJack U12 ADC system combined with a single chip sensor module (SHT11) manufactured by Sensirion (Staefa, Switzerland).

During adsorption and desorption processes, the relative humidity value was measured by the commercial RH humidity sensor. The open container was used to maintain the water vapor pressure on the ZnO surface at around 1 atm. This technique is also useful to keep the temperature constant (around 23 °C) during the measurements. For the desorption process, the RH was changed quickly from 88% RH (above the water level) to 48% RH (ambient conditions).

3. Results and discussion

3.1. Structural characterization of ZnO nanowires

Fig. 2 presents SEM images of the ZnO nanowires on the Si substrate when placed in the low temperature ($400\,^{\circ}$ C) zone. As seen from the figure, the morphology of the ZnO products is nanowire-like with diameters varying between 20 and 50 nm and having lengths of up to several tens of micrometers.

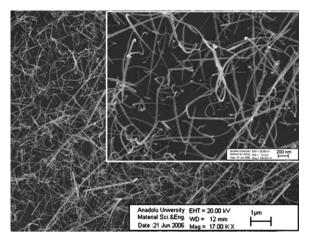


Fig. 2. SEM images at different magnifications of ZnO nanowires deposited onto Si substrates at 400 $^{\circ}$ C.

Fig. 3 shows the XRD pattern of the synthesized ZnO nanowires. Diffraction peaks in the XRD pattern can be indexed as hexagonal wurtzite-structured ZnO (JCPDS card no 36-1451, $a=b=0.33\,\mathrm{nm}$, $c=0.52\,\mathrm{nm}$). In addition to ZnO diffraction peaks, only the Si substrate-related diffraction peak was observed.

3.2. Monitoring of humidity-sensing capabilities of ZnO nanowires using the QCM

Fig. 4 shows the transient frequency responses of an empty and loaded QCM with drop-casted ZnO nanowires for four adsorption—desorption cycles under relative humidity values varied between 48% and 88% RH. The RH values plotted in the figure were measured with a commercial humidity sensor. During the adsorption process, the oscillating resonance frequency of the QCM electrodes increased sharply with increasing RH value while there was no change in that of the empty QCM. On the other hand, during the desorption process, the RH value decreased to the ambient

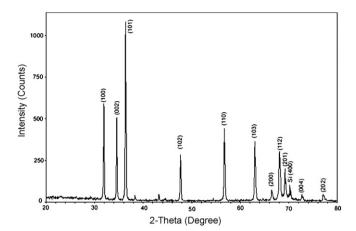


Fig. 3. XRD pattern of ZnO nanowires.

RH value of 48%, and the QCM was able to return to its initial resonance frequency value. The QCM results show that the ZnO nanowires were sensitive to the humidity of the environment and gave reproducible adsorption and desorption kinetic behavior to humidity changes for short time periods. By comparing commercial humidity sensors and ZnO nanowires as coated onto the QCM, it is obvious that the ZnO nanowires on the QCM respond in similar characteristic to the sensing materials of a commercial RH sensor.

To observe the equilibrium conditions of the ZnO nanowires under varying relative humidity, the QCM frequency shift was recorded until a saturation value was reached. Under equilibrium conditions, the frequency response curve of the ZnO nanowire with respect to the relative humidity pulse between 48% and 88% is shown in Fig. 5 and a sensitivity of nearly 3 Hz/% RH for experimental conditions of within the range of 48–88% RH were obtained. The frequency shift increased up to 125 Hz in 600 s and then became constant. The desorption process was investigated by decreasing the RH to 48% by removing the source of water vapor. In this case the frequency shift decreased quickly to 22 Hz in 200 s and then slowly

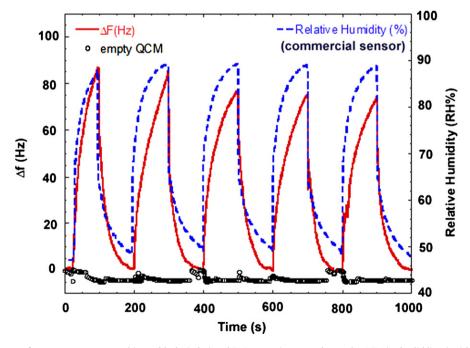


Fig. 4. The frequency responses of an empty quartz crystal (open black circles) and ZnO nanowires coated onto the QCM (red solid lines) with relative humidity values as measured by a commercial sensor (dashed lines) for four humidity adsorption—desorption cycles between 48% and 88% RH. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

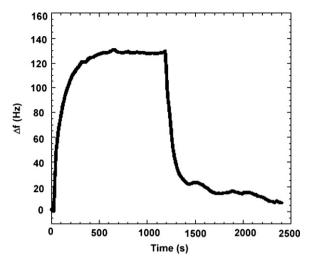


Fig. 5. Long term exposure. Frequency response of ZnO nanowires to varying RH values between 48% and 88%.

reached slowly its initial value. The adsorption process is slower than the desorption process as seen in Fig. 5. The reason for this kind of different behavior between the adsorption and desorption processes is that the rate of relative humidity change between the low-to-high cycle (adsorption) was slower than that of the high-to-low cycle (desorption) as seen from %RH measurements taken by with a commercial humidity detector (compare with Fig. 4). Therefore it is not realistic to determine real response and recovery times from the data of Fig. 5 using our experimental conditions. Under these experimental conditions, the response and recovery times of the ZnO nanowires-coated QCM were determined to be 104 s and 125 s, respectively.

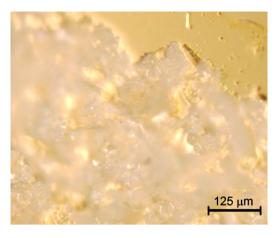
As seen from Figs. 4 and 5, the response of ZnO nanowires to humidity molecules results in a positive frequency shift. However, in the literature, it is observed that the interaction between adsorbed molecules and sensing material generally causes a negative frequency shift according to the well-known relation described by Sauerbrey [19]. The positive frequency can be explained in terms of the visco-elastic changes in the mechanical stiffness of non-rigid ZnO nanowire films.

According to the Sauerbrey equation, QCM measurements rely on calculation of the change in mass (Δm) on the quartz crystal surface via measurement in the change in vibration frequency (Δf) :

$$\Delta f = -\frac{2f_0^2}{A\sqrt{\mu\rho}}\Delta m\tag{1}$$

where f_0 is the fundamental frequency of the unloaded quartz crystal, A the area of the gold electrodes coated onto the QCM crystal (0.205 cm²), ρ the density of the crystal and μ the shear modulus of quartz. The reference crystal has an oscillation frequency of 8 MHz. The density of the QCM quartz crystal is 2.648 g/cm³ and the shear modulus μ is 2.947 × 10¹¹ g/cm s². For 8 MHz crystal, a change in mass of 0.14 ng corresponds to frequency shift of 0.1 Hz.

Eq. (1) is only applicable for the case when the added mass is much less than the mass of the crystal and the mass is firmly attached to the quartz crystal surface, oscillating rigidly together with the crystal. The oscillating solid-deposited film should have the same velocity as the quartz crystal surface throughout. Eq. (1) shows a linear relationship between mass uptake and frequency shift without considering the physical properties of the deposited solid film on the quartz crystal and describes negative frequency shifts occurring with increasing mass uptake into a rigid film on the quartz crystal. However unusual frequency shifts (i.e., positive and/or both positive and negative shifts) have been observed in the literature [20–22]. In fact, nonlinear or positive shifts in the reso-



 $\label{eq:fig.6.} \textbf{Fig. 6.} \ \ \text{Optical microscope image of the drop-casted ZnO nanowires on gold-coated quartz crystal.}$

nance frequency of a quartz crystal arise from some perturbations in the physical and mechanical properties of the deposited film or material on the quartz crystal. Hunt et al. [23–25] derived an equation using the time-dependent perturbation theory to explain the positive shifts in QCM resonance frequency as

$$t\frac{\partial \Delta \omega}{\partial t} + \Delta \omega = \frac{\omega_u h_f}{\pi \sqrt{\rho_q \mu_q}} x \left[-\omega_u \left(\Delta \rho - \frac{\Delta \mu}{V_s^2} \right) + j \left(\frac{\partial \Delta \rho}{\partial t} - \frac{1}{V_s^2} \frac{\partial \Delta \mu}{\partial t} \right) \right]$$
(2)

where h_f is the thickness of the deposited film, ρ_q is the mass density of the quartz crystal, V_s is the acoustic velocity across the deposited film thickness, ρ is the density of the film; μ_q is the shear stiffness of the quartz, μ is the stiffness of the film; Δ is the difference between perturbed and unperturbed quantities, and the subscript u is used for the unperturbed quantities. Assuming that $\Delta\rho$, $\Delta\mu$ and $\Delta\omega$ do not change with time and ignoring kinetic changes, Eq. (2) can be expressed as

$$\Delta f = -\frac{2f_u^2 h_f}{\sqrt{\rho_q \mu_q}} \left(\Delta \rho - \frac{\Delta \mu}{V_s^2} \right) \tag{3}$$

Because μ and ρ are constants and the mass uptake can be expressed as $\Delta m = \Delta \rho A h_f$ (A is sensing area), the first term of Eq. (3) is the Sauerbrey equation including static changes in the mechanical stiffness of the deposited film. Eq. (3) explains negative frequency shifts as being due to attached masses on the QCM sensor (first term) and positive frequency shifts being related to the mechanical stiffness of the film (second term). Another effect that leads to a deviation from the Sauerbrey equation may arise as a result of liquid deposits growing by condensation on the coated quartz crystal. In this case, after an initial decrease of the frequency shift, a subsequent increase can be observed due to the liquefaction of the adsorbed product [22]. After considering these issues, the positive frequency shift of our ZnO nanowires under varying relative humidity conditions can reasonably be ascribed to changes in the mechanical stiffness of the ZnO nanowire film on the QCM electrodes as a result of the interaction between water molecules (from the humid vapor conditions) and the ZnO nanowires. We believe that the ZnO nanowires are loosely bound to the goldcoated surface of the QCM electrodes after the drop-casting process. ZnO nanowires stand on the electrodes in cotton-like structure. ZnO nanowires with full of porosity should have more available active sites for adsorption process as seen from optical image given in Fig. 6. Our experimental results show that the Sauerbrey equation must be modified to include both a mass accumulation term and a visco-elastic term to consider the mechanical damping factor for cases in which the coated film on the quartz crystal is not rigidly attached. In our situation only a positive frequency shift was observed; therefore, the second part of Eq. (3) dominates over the mass loading effect quickly due to high a humidity level that may cause the diffusion of water molecules into the ZnO nanowire film and change the visco-elastic properties as well as the mechanical stiffness of the film on the QCM electrodes. In environments of high humidity, condensation of water droplets inside or on the surface of a ZnO nanowire film structure is another possible process, which would effectively cause a positive shift in frequency as seen in Ref. [22].

The frequency shift of the ZnO nanowires with 3 Hz/% RH is relatively low compared to other values found in the literature. The frequency shift is directly related to the amount of available active adsorption sites on the nanowires. The nanostructures have more active adsorption sites with increasing sensitivity due to higher surface/volume ratio compare to bulk and thin film forms Therefore it is expected that nanodots may have higher sensitivity than nanowires as observed in our previous work [10] on ZnO nanoparticles with 10 nm diameter. Another reason for the lower sensitivity of ZnO nanowires may be related with that the observed frequency shift should be less than its real value, since the net observed frequency shift is the result of combining an anomalous negative frequency change due to the mass loading effect (absorption of water) with a positive frequency change due to a decrease in mechanical stiffness of the ZnO nanowire film structure on the OCM electrodes as a result of the diffusion of water into the film.

4. Conclusions

The relative humidity response of ZnO nanowires synthesized by carbothermal catalyst-free vapor solid technique was investigated. A positive frequency response has been observed and explained by considering the variation in the visco-elastic behaviors of ZnO nanowires films (as coated onto QCM electrodes) during their resonance oscillations under varied relative humidity conditions. The sensitivity of the ZnO nanowires was found to be 3 Hz/% RH for experimental conditions of in the range of 48–88% RH. We also discussed the non-Sauerbrey behavior of the ZnO nanowires as coated on a QCM sensor, ultimately considering the variation in mechanical stiffness of the ZnO nanowire coating under conditions of large changes in relative humidity changes as a reasonable cause for this anomalous behavior.

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Ayşe Erol received her Doctor of Philosophy degree in Physics from Istanbul University, Turkey in 2002. Her Ph.D. thesis work focused mainly on electrical and optical characterization of hot electron light emitting devices. During her Ph.D. she also has been at Essex University, UK working on the dilute nitride semiconductors as a visiting fellow. Currently, Ayşe Erol is working at Physics Department of İstanbul University as an Associated Professor. Ayşe Erol's scholarly output includes papers mainly focused on optical and electrical properties of low dimensional III–V group semiconductors, and synthesis of II–VI semiconductor nanostructures as an additional research work, a refereed international conference proceeding, an edited book published by Springer in 2008 and a popular physics book published in 2006. She supervises two M.Sc. theses relating to mainly synthesis of ZnO nanostructures and characterization

Salih Okur received his bachelor's degree in Physics Education from Hacettepe University in Ankara, Turkey in 1989, his master's degree in Physics from Ankara University in Ankara, Turkey in 1992, and master's degree in Physics from Illinois Institute of Technology (IIT) in Chicago, Illinois, USA in 1996, and his Ph.D. in Physics from IIT in 1998. He is currently an Associate Professor in the Department of Physics at Izmir Institute of Technology in Izmir, Turkey. His current research interests include SAM, LB, organic semiconducting thin film interfaces and their application to electronic devices such as organic photovoltaics, OLEDs, OTFTs, nano-dots, nano-wires.

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