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Letter to the Editor

Experiment and theoretical analysis of relative humidity sensor based on film bulk acoustic-wave resonator

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ABSTRACT

Relative humidity (RH) was measured with ZnO based film bulk acoustic-wave resonator (FBAR). The resonant frequency of the FBAR decreased linearly in a two-stage manner as the RH increased in the environment. For low RH (RH < 50%), a frequency downshift of 2.2 kHz per 1% RH change was observed. For high RH (RH > 50%), a frequency downshift of 8.5 kHz per 1% RH change was obtained. It was demonstrated that the two-stage response of the FBAR can be interpreted using the power law theory for semiconductor gas sensors and the mass loading effect.

Humidity is a dynamic parameter that is essential for various fields of industry as well as human activities. There is a substantial interest in the development of relative humidity (RH) sensors for applications in monitoring moisture level at home, in clean rooms, cryogenic processes, medical and food science, and so on. Humidity sensors based on changes in the capacitance [1] or resistance [2] of the sensing element from absorption of water vapor have been investigated extensively. Alternatively, surface acoustic-wave (SAW) resonant sensors have also been developed with polymer films deposited on top as the sensing layer [3].

In this study, a novel RH sensing device using ZnO based film bulk acoustic-wave resonator (FBAR) was introduced. FBAR has been well developed both as filters [4] and as high sensitivity mass sensors [5] in recent years. The schematic structure of the FBAR RH sensor is shown in Fig. 1. The FBAR was fabricated on top of a SiN diaphragm (0.6 μm thick). A sputtered ZnO film (1.2 μm) acted both as the RH sensitive layer and the piezoelectric actuation layer for the FBAR sensor. The ZnO film was characterized by X-ray diffraction (XRD) using Cu Kα radiation (Fig. 1). Only the Bragg reflection corresponding to (0 0 2) planes was observed, indicating that the film had preferred orientation along the wurtzite *C* axis, normal to the silicon substrate. The top and bottom electrodes were made of Au (0.2 μm) and Al (0.2 μm), respectively. The fabrication process of the FBAR RH sensor was as follows. In the first step, a SiN layer was deposited on a Si wafer (1 0 0) with low-pressure chemical vapor deposition (LPCVD). Then the Si wafer was etched from the backside anisotropically in potassium hydroxide (KOH) to form the cavity. Next, the bottom Al electrode was deposited by electron-beam (e-beam) evaporation and patterned on top of the SiN film. ZnO was radio-frequency (RF) sputtered and etched to form the desired pattern. The last step was the e-beam deposition and patterning of top Au electrode by lift-off.

The sensor was encapsulated in a chamber, in which RH was controlled by an ultrasonic humidifier and measured by a HH314A humidity temperature meter. The resonant frequency of the FBAR was monitored with an Agilent E5071C network analyzer and recorded by a LabVIEW program. It had a noise floor of 0.7 ppm at 1.4 GHz, which was suitable for integration with a wireless sensor network. The quality factor (Q) of the FBAR was between 530 and 550.

The RH response of the FBAR sensor at room temperature is shown in Fig. 2. A two-stage process can be identified based on the different slope values. At low RH (RH < 50%), the resonant frequency decreased linearly with the RH and a frequency downshift of 2.2 kHz per 1% RH change was observed. With the current noise floor, the detection limit was around 0.45% RH. The RH response in this range was due to the replacement of adsorbed oxygen with water molecules on the ZnO surface [6]. Therefore, the density of the ZnO film increased. The resonant frequency of the FBAR can be determined from the following two equations: $v = (E/\rho)^{1/2}$ and $f = v/2d$, where E, ρ and d are the elastic constant, density and thickness of the ZnO film, respectively. v is the acoustic velocity within the ZnO film and f is the resonant frequency of the FBAR [7]. As the density of the ZnO film increased with RH, the acoustic velocity decreased, resulting in the decrease of the resonant frequency.

It has been known that the electric resistance of a semiconductor gas sensor exposed to a target gas (partial pressure P) is proportional to P^n where n is a constant fairly specific to the kind of target gas (power law). Yamazoe and Shimanoe [8] established a theoretical basis to the power law combining a depletion theory of the semiconductor, which deals with the distribution of electrons between surface state and bulk, with the dynamics of adsorption and/or reactions of gases on the surface, which is responsible for accumulation or reduction of surface charges. By extending their work to address the density change of the semiconductor gas sensor, a theoretical model for the RH response of the ZnO film based FBAR was developed for the low RH region (RH < 50%).

According to Yamazoe and Shimanoe [8], the density of conduction electrons at the surface $[e]$ can be expressed as:

$$[e] = N_d \exp\left(-\frac{m^2}{2}\right) \tag{1}$$

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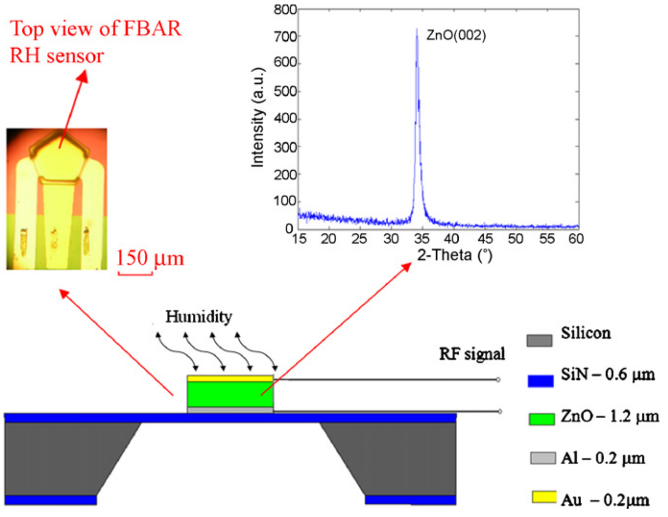


Fig. 1. Schematic cross-sectional structure of the FBAR RH sensor with a photograph of the top view of a fabricated device on the left and an XRD trace of the ZnO film illustrating that it had ⟨0 0 2⟩ crystal orientation on the right.

where N_d is the density of donors and m is the reduced depletion depth. Using the double Schottky barrier model, the amount of adsorption/desorption of oxygen at each grain-boundary is proportional to $[e]$. Thus the relative density change, $\Delta\rho$, induced by adsorption/desorption of oxygen is proportional to $[e]$.

Meanwhile, the frequency shift Δf can be formulated as:

$$\Delta f = \frac{1}{2d}\left(\sqrt{\frac{E}{\rho+\Delta\rho}} - \sqrt{\frac{E}{\rho}}\right) = \frac{\sqrt{E}}{2d}\,\frac{1-\sqrt{1+(\Delta\rho/\rho)}}{\sqrt{\rho+\Delta\rho}} \tag{2}$$

In the equation above, $\rho \gg \Delta\rho$. In this way,

$$\Delta f \approx \frac{\sqrt{E}}{2d}\cdot\frac{1-1-(1/2)(\Delta\rho/\rho)}{\sqrt{\rho}} = -\frac{\sqrt{E}}{4d\rho\sqrt{\rho}}\Delta\rho \tag{3}$$

Therefore the frequency shift Δf is proportional to $\Delta\rho$ and thus, it is also proportional to $[e]$.

At room temperature, oxygen is adsorbed as $O_2{}^-$ on the ZnO surface [9].

$$O_2 + e \underset{k_{-1}}{\overset{k_1}{\rightleftarrows}} O_2{}^- \tag{4}$$

Here e stands for a conduction electron at the surface, and k_1 and k_{-1} are the rate constants of forward and reverse reactions, respectively. The rate of accumulation of $O_2{}^-$ is given by

$$\frac{d[O_2{}^-]}{dt} = k_1 PO_2[e] - k_{-1}[O_2{}^-] \tag{5}$$

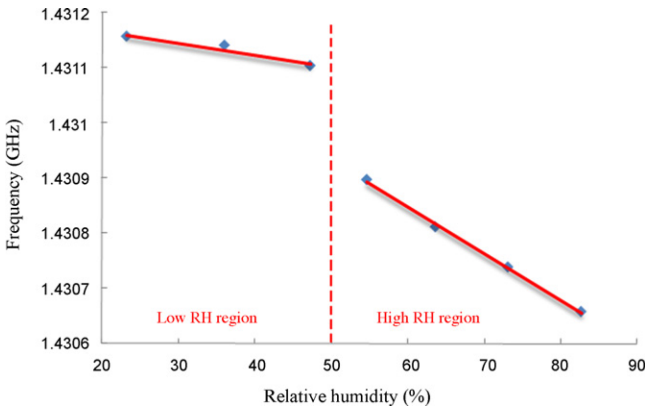


Fig. 2. The RH response of the FBAR sensor: two different linear stages can be identified. At low RH (RH < 50%), a frequency downshift of 2.2 kHz per 1% RH change was observed. At high RH (RH > 50%), a frequency downshift of 8.5 kHz per 1% RH change was obtained.

Here the brackets mean the density of $O_2{}^-$ per unit area or that of electrons per unit volume, t is time, and PO_2 is the partial pressure of oxygen. At equilibrium, the rate is zero, thus,

$$k_{o1} PO_2[e] = [O_2{}^-] \tag{6}$$

$$k_{o1} = \frac{k_1}{k_{-1}} \tag{7}$$

where k_{o1} is the equilibrium constant of oxygen adsorption.

Assuming that there are no electron-trapping sites other than $O_2{}^-$ on the surface, then $O_2{}^-$ ions would be solely responsible for the surface charge density, that is,

$$[O_2{}^-] = N_d\omega \tag{8}$$

Here ω is the depletion depth and $\omega = mL_D$, where L_D is the Debye length [8].

Insertion of (1) and (8) into (6) yields,

$$k_{o1} PO_2 \exp\left(-\frac{m^2}{2}\right) = \omega \tag{9}$$

As mentioned above, water molecules will take the place of the adsorbed oxygen on the ZnO surface [6]. Two water molecules can dissociate and become chemisorbed in the ZnO film [6,10]. At equilibrium,

$$k_{o1} PO_2[e] = [O_2{}^-] + k_{o2} PH_2O[O_2{}^-] \tag{10}$$

$$k_{o2} = \frac{k_2}{k_{-1}} \tag{11}$$

where k_2 is the rate constant of water absorption and PH_2O is the partial pressure of water vapor, which is proportional to RH at a certain temperature.

From the discussion above, the power law exponent n, defined as $d(\log \Delta f)/d(\log PH_2O)$, is obtained,

$$\frac{d(\log \Delta f)}{d(\log PH_2O)} = 1 - \frac{1}{1+m^2} \tag{12}$$

where m is sufficiently large under usual conditions [8]. Thus n is around 1, which can be used to explain the linear relationship between the resonant frequency and the RH in Fig. 2 in the low RH region.

In Fig. 2, at high RH (RH > 50%), the resonant frequency decreased linearly with the RH and a frequency downshift of 8.5 kHz per 1% RH change was obtained, corresponding to a detection limit of around 0.12% RH. With increasing RH, a discrete water layer began to form on the ZnO surface, which acted as a mass loading on the FBAR. Thus the resonant frequency of the FBAR decreased linearly with the mass of the water accumulated on top of the resonator [5]. This can also be observed from the Q response of the FBAR with increasing RH (Fig. 3). At RH higher than 50%, Q decreased quickly with humidity, while at RH lower than 50%, Q changed less than 10%. These results indicated that water layer formed on the ZnO surface when RH was higher than 50%, thus attenuating acoustic-wave, resulting in a lower Q.

The Butterworth–Van Dyke (BVD) equivalent circuit of FBAR with and without liquid loading is shown in Fig. 4, where C_o, L_m, C_m, and R_m are the clamped capacitance between the two FBAR electrodes, motional inductance, motional capacitance, and motional resistance of the resonator, respectively. R_2 and L_2 are associated with acoustic energy loss and mass loading by the liquid, respectively [5]. In the equivalent circuit, the circuit elements are related

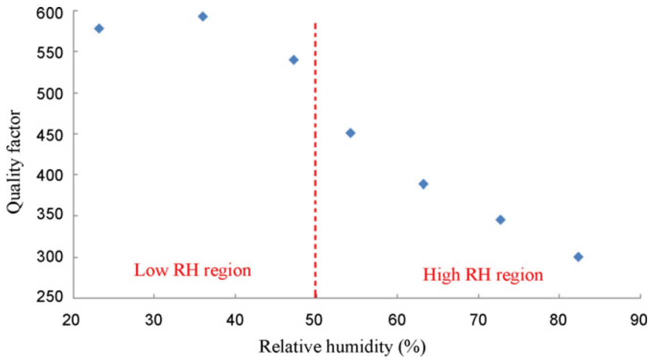
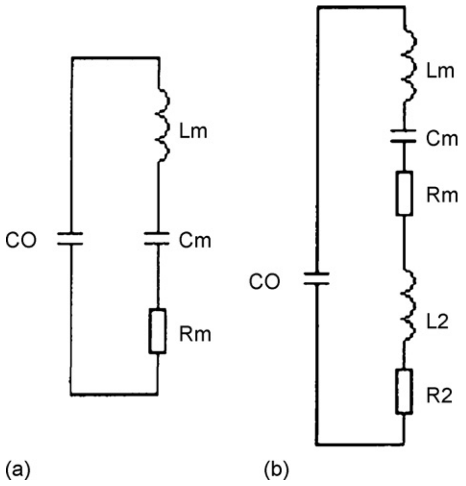


Fig. 3. The response of FBAR's Q vs. RH. Two different stages can be identified. At RH lower than 50%, Q changed less than 10%. However, at RH higher than 50%, Q decreased quickly with humidity.



(a) (b)

Fig. 4. Butterworth–Van Dyke (BVD) equivalent circuits: (a) a FBAR without liquid loading; (b) a FBAR with liquid loading.

to the physical parameters of the resonator and the added liquid by

$$f_s = \frac{1}{2\pi\sqrt{L_m C_m}} \tag{13}$$

$$f_p = \frac{1}{2\pi\sqrt{L_m(C_m C_o/C_m + C_o)}} \tag{14}$$

$$L_m = \frac{1}{4\pi^2 f_p^2 C'} \tag{15}$$

$$C' = \frac{C_m C_o}{C_m + C_o} \tag{16}$$

$$f'_p = \frac{1}{2\pi\sqrt{(L_m + \Delta L)(C_m C_o/C_m + C_o)}} \tag{17}$$

$$\Delta L = L_2 \tag{18}$$

$$L_2 = \frac{2L_m \rho_1 d_1}{\rho_0 d_0} \tag{19}$$

where f_s and f_p are the series and parallel resonant frequency of the FBAR. f'_p is the parallel resonant frequency with the liquid loading. ρ_0, d_0, ρ_1, and d_1 are the density of the piezoelectric layer, the thickness of the piezoelectric layer, the density of the added liquid and the thickness of the added liquid, respectively [5].

From the equations above (similar results can be obtained for f_s),

$$\Delta L = \frac{1}{4\pi^2 C'}\frac{f_p^2 - f'^2_p}{f_p^2 f'^2_p} \approx \frac{1}{4\pi^2 C'}\frac{2\Delta f_p}{f_p f'^2_p} \tag{20}$$

$$\Delta f_p = f_p - f'_p \tag{21}$$

$$\frac{\Delta f_p}{f_p} = \frac{\Delta L}{2}\frac{1}{L_m + \Delta L} \approx \frac{\Delta L}{2L_m} = \frac{\rho_1 d_1}{\rho_0 d_0} \tag{22}$$

Eq. (22) indicates that the resonant frequency shift of an acoustic resonator is linearly related to the mass of a material added on the top of the resonator. Assuming the mass of the water accumulated on the FBAR is proportional to the RH, a linear response of the FBAR can be expected in the high RH region, which is in agreement with the experiment results.

In summary, a RH sensor was developed with ZnO based FBAR. The resonant frequency of the FBAR decreased linearly in a two-stage manner as the RH increased in the environment. In low RH region (RH < 50%), a frequency downshift of 2.2 kHz per 1% RH change was observed. This effect was attributed to water molecules replacing the adsorbed oxygen on the ZnO surface, thus increasing the density of the film. In high RH region (RH > 50%), a frequency downshift of 8.5 kHz per 1% RH change was obtained, which was due to the mass loading effect of the water layers formed on the ZnO surface. This study has demonstrated the feasibility of measuring RH using ZnO film based FBAR.

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