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Study to Increase the Sensitivity of QCM Gas-Sensor Coated with Plasma Polymerization Film

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Fundamental and overtone modes of quartz resonators with high frequencies have been studied to increase the sensitivity of QCM gas-sensor. Polystyrene film was plasma-polymerized onto the electrode surface of an AT-cut quartz resonator and then used as a molecular recognition membrane to detect gas-phase analytes. In this study, handmade oscillators with superior stability were used to vibrate each mode of AT-cut quartz resonators. Deriving the theoretical mass sensitivity of an AT-cut quartz resonator vibrating at the overtone mode, confirmed the fact that the theoretical value was the same as the result obtained from the experiments.

Keywords QCM gas-sensor; plasma polymerization; overtone mode

INTRODUCTION

Recently, various types of gas-sensors using quartz crystal microbalance (QCM) method have been developed ^[1,2]. Unfortunately present sensors lack the sensitivity to detect the extremely low concentrations of gas-phase analytes. Therefore this paper proposes a new method to increase the sensitivity of the QCM gas-sensor.

Plasma polymerization is a widely used technique to form ultra-thin films on a variety of sensors and immunoassays in various applications ^[3,4]. The advantages of the plasma-polymerized (pp-) films have already been introduced in a different paper ^[5]. Due to those advantages, the pp-film is expected to be useful as a

molecular recognition film, immobilized rigidly on the QCM.

In the case of an AT-cut quartz resonator vibrating at a specific overtone mode, the relationship between acoustic wavelength λ and quartz plate thickness t_q is $t_q = (\lambda/2) \cdot N$ (N : overtone number). From this equation and sauerbrey's derivation [6], the theoretical equation between ΔF , the resonance frequency change, and Δm , the mass change on the electrode surface, is presented as follows:

$$\Delta F = \frac{2F_o^2}{(\mu_q \rho_q)^{1/2}} \cdot \frac{\Delta m}{A} \cdot \frac{1}{N} \tag{1}$$

Where F_o is the frequency of the quartz resonator prior to the mass change, A , the piezoelectrically active area, μ_q , the shear modulus and ρ_q , the density of quartz. The mass sensitivity per area of each quartz resonator was calculated from equation (1). In addition, the sensitivity ratios of the quartz resonators compared to the mass sensitivity of the 9 MHz quartz resonator were calculated. Both data are presented in Table 1.

TABLE 1 The mass sensitivity per area of each quartz resonator and the sensitivity ratio compared to the 9 MHz quartz resonator.

Mode	Sensitivity [ng/Hz·cm ²]	Ratio
Fundamental 9 MHz	5.458	1
Fundamental 50 MHz	0.177	30.9
3 rd overtone 100 MHz	0.133	41.2
5 th overtone 100 MHz	0.221	24.7

EXPERIMENTAL

Plasma Polymerization

The QCMs were placed on the lower electrode of the plasma polymerization apparatus. Styrene was vaporized and polymerized onto the QCM in the plasma, 100 Watt RF & 100 Pa of vapor pressure, for 240 sec. The amount of pp-polystyrene on the electrode surface of each quartz resonator was 30 ~ 35 ng/cm².

Measurement of Frequency Change

Figure 1 shows the schematic diagram of our experimental setup. Temperature was controlled at 20 °C by the incubator during the experiment. N₂ was purged through the chamber until the resonance frequency of the quartz resonator was stable. 20 ml

of gas-phase analyte was injected by a glass syringe, after 5 minutes N_2 was purged into the chamber again until the resonance frequency of the quartz resonator recovered to the initial frequency. The results are presented in Figure 2.

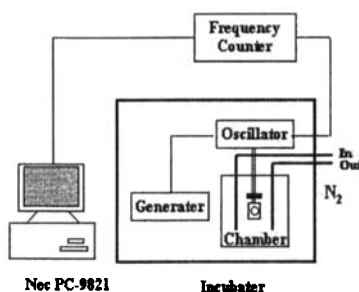


FIGURE 1 Experimental setup

RESULTS AND DISCUSSION

Figure 2(a) shows the frequency response for each mode of the pp-film coated quartz resonators when 20 ml of acetone gas is injected into the chamber. From Figure 2(a), it is clarified that the 3rd overtone mode of the 100 MHz quartz resonator shows the greatest frequency change for acetone gas, followed by the 50 MHz, the 5th overtone mode of the 100 MHz and finally the 9 MHz. This gives that the 3rd overtone mode of the 100 MHz quartz resonator has the best mass sensitivity of the quartz resonators compared. In addition, the frequency change for the 3rd overtone mode of the 100 MHz quartz resonator is approximately 40 times that of the fundamental mode of the 9 MHz, this value is almost consistent with the theoretically calculated sensitivity ratio. The same correlations can be seen for the other QCM's as well (see Table 1). This result was verified through repetitive experiments.

Figure 2(b) shows the frequency response for each mode of quartz resonators when 20 ml of diethyl-ether gas is injected. The sensitivity ratios as well as the order of the frequency change for each mode of quartz resonators were almost the same as the results for the acetone gas. Comparing Figure 2(a) and 2(b), it can be learned that the shape of the frequency response curve varies with the different gases and not with the mode of the quartz resonator. The responses of chloroform and ethanol are not shown here. Gases can be identified from the shapes of the frequency response of four different gas-phase analytes: acetone, chloroform, diethyl ether and ethanol.

CONCLUSION

The sensitivity of the QCM gas-sensor using this method can be drastically increased compared with that of the general 9 MHz QCM gas-sensor.

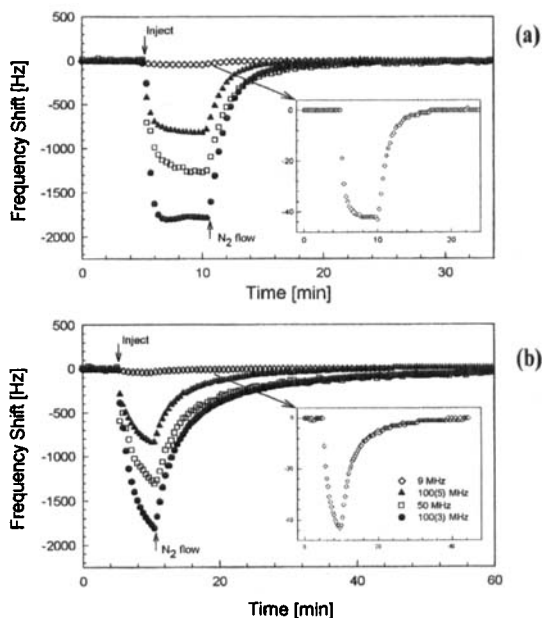


FIGURE 2 The frequency response of each quartz resonator for 20 ml of acetone gas (a) and diethyl-ether gas (b).

Acknowledgments

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