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Study on PDMS Microchannel Structure of Wireless-Electrodeless QCM Sensor and Application to Gas Sensor

無線無電極QCMセンサのPDMS流路構造検討とガスセンサへの応用

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1. Introduction

Quartz crystal microbalance (QCM) is based on mass detection sensor that detects mass loading, when the target substance adsorbs on the surface of the quartz oscillator, as a change in the resonant frequency. The sensitivity of QCM is inversely proportional to the square of the quartz thickness[1)]. Therefore, the thinner the quartz oscillator is, the higher the detection sensitivity is. However, in the commercially available QCM, because the metallic electrodes for exciting the quartz oscillator are formed on the quartz surface, their inertia resistance relatively increases as the quartz oscillator becomes thinner, and the quartz oscillator does not eventually vibrate. To improve these issues, the wireless-electrodeless QCM sensor chip was developed[2,3)]. Due to adopting the structure supporting the quartz oscillator by the micropillars and the wireless driving system using electromagnetic (EM) wave, the QCM sensor with the quartz thickness of 10 μm or less was realized, and its usefulness as a biosensor was shown[1)]. Furthermore, the wireless-electrodeless QCM sensor that can be fabricated at a lower cost and at a low temperature around a room temperature was developed by nanoimprint lithography using the polydimethylsiloxane (PDMS)[4)]. In this study, we estimated the structural damping due to the micropillar arrangement in the wireless-electrodeless QCM sensor by the piezoelectric analysis, and compared the results with the demonstration of the actual device, and investigated a better arrangement of micropillars. In addition, using the manufactured sensor chip, the application to a gas sensor was investigated.

2. Piezoelectric analysis by FEM Simulation

The influence of structural damping due to the micropillars to the quartz oscillator vibrating in shear mode was investigated through piezoelectric analysis using finite element method (FEM) simulation software (Femtet, Murata Software Co., Ltd.). Piezoelectric analysis was performed to a rectangular AT-cut quartz oscillator with a width of 2.5 mm, a length of 1.7 mm, and a thickness of 30 μm. The micropillar is the cylindrical shape with an outer diameter of 200 μm and a height of 50 μm, which does not cause shape defects when fabricated using PDMS and has high reproducibility. The conditions of the micropillar arrangement in the piezoelectric analysis included the case where the pillars were arranged at the four corners of the quartz oscillator and the case where the pillars were arranged near the center were investigated. Figure 1 shows the displacement distribution in the x axis direction near the resonant frequency.

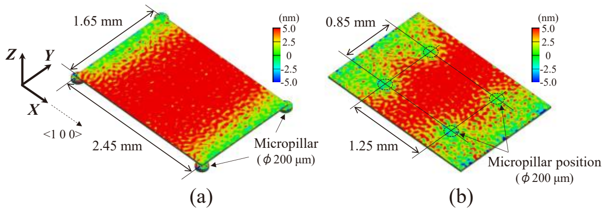

Fig. 1 Displacement distribution in the x axis direction of the AT-cut quartz oscillator: (a)when supporting the four corners, (b) supporting near the center.

3. Evaluation as a hydrogen gas sensor

In order to verify the piezoelectric analysis results, the wireless-electrodeless PDMS QCM sensor chip was actually fabricated and the resonant spectra were evaluated. The AT-cut quartz oscillator with a width of 2.5 mm, a length of 1.7 mm, and a thickness of 25 μm was installed in the PDMS microchannel, and the Q-factor of the resonant spectra in the atmosphere were evaluated. The EM wave is applied from the copper foil antenna attached to the upper side of the sensor chip to excite the quartz oscillator, and the electric charge induced on the quartz surface is received by the other antenna attached to the lower side (Fig.

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2(a)). Figure 2(b) shows the resonance spectra of sensor chips with different micropillar arrangements. The Q-factor of the resonant spectrum was 5500 when the micropillars were placed near the center of the quartz oscillator, while it was 21900 when placed at the four corners.

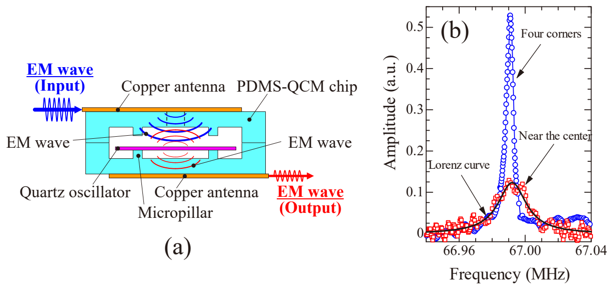

Fig. 2 (a)Wireless driving principle of sensor chip, (b)reosonant spectra by different supporting conditions.

We investigated the application of the wireless-electrodeless PDMS QCM sensor chip to the hydrogen gas sensor because a high Q-factor was obtained in the atmosphere. Palladium (Pd) is widely known as a hydrogen storage material. Therefore, we fabricated the wireless-electrodeless PDMS QCM sensor chip with the built-in quartz oscillator (width 2.5 mm, length 1.7 mm, and thickness 30 μm) of which the Pd thin film of about 10 nm was formed on one side. Figure 3 shows the constitution of the hydrogen gas sensor evaluation system.

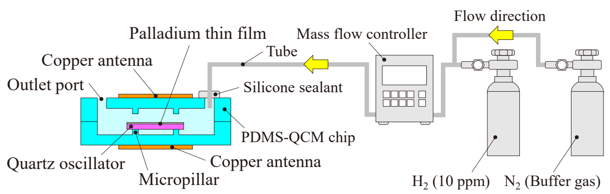

Fig. 3 Experimetal setup of hydrogen gas sensor.

At the first, using a mass flow controller, a buffer gas (pure nitrogen gas) was fed at a flow rate of 50 sccm to obtain a baseline. After that, the valve of the buffer gas was closed and the hydrogen gas with a concentration of 10 ppm was fed. When the hydrogen gas was fed in the sensor, a frequency shift of about 1.2 ppm was obtained (Fig. 4).

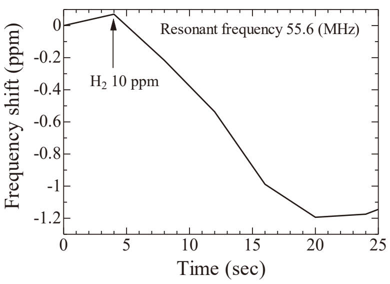

Fig. 4 Detection of hydrogen gas via Pd thin film.

4. Results and Discussion

Investigating the influence of the micropillar arrangement on the thickness shear vibration of the quartz oscillator through the piezoelectric analysis, when the micropillars are placed near the center of the quartz oscillator, the influence of structural damping is significant, and when placed at the four corners, it was found that there was no structural damping. As a result, the sensor chip with micropillars arranged at the four corners of the quartz oscillator had high Q-factor which was about four times higher than arranged near the center. These results show the same tendency as the result obtained by the FEM. The wireless-electrodeless PDMS QCM hydrogen gas sensor chip was manufactured using the quartz oscillator on which the Pd thin film was formed, and it was possible to detect the frequency shift caused by the gas storage of the hydrogen gas with an extremely low concentration of 10 ppm. When the storage amount of the Pd thin film was calculated from the frequency shift, it was 0.41 ng, which was an unrealistic value. Therefore, it is considered that the frequency shift is occurred by the bending of the quartz oscillator caused by the expansion of the Pd thin film due to the storage of the hydrogen gas.

5. Conclusion

Through piezoelectric analysis, a better arrangement for supporting the rectangular AT-cut quartz oscillator with the micropillars was investigated. As a result, it was found that it is desirable to arrange the micropillars at both ends of the quartz oscillator in the longitudinal direction. When the micropillars were arranged at the four corners of the quartz oscillator, a high Q-factor was obtained, therefore the application to a gas sensor was evaluated. As a result, we succeeded in detecting the hydrogen gas with an extremely low concentration of 10 ppm.

Acknowledgment

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