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Measurement of trace moisture using ball SAW sensor
ボール SAW センサを用いた微量水分の測定

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1. Introduction

Although chilled-mirror hygrometer which can measure trace moisture less than 1 ppm [1] is used to evaluate the performance of barrier films for organic electroluminescence displays and highly pure semiconductor process gases, it is slow and expensive. Fast and inexpensive surface acoustic wave (SAW) sensor can measure only % level moisture [2]. In this situation, the ball sensor [3] capable of detecting the velocity and the attenuation sensitively using multiple roundtrip propagation of naturally collimated beam is expected to realize fast and sensitive trace moisture sensor. However, it was not realized because of the difficulty in generating the trace moisture gases with constant concentration needed to evaluate the sensor performance.

In this study, we generate the trace moisture gas using the diffusion tube method [4] providing a humidity standard, and evaluate the performance of two types of ball SAW sensors for trace moisture mesurement.

2. Measurement implementation

Schematic diagram of a measurement setup using diffusion tube method [4] is shown in **Fig.1**. The number of water molecules evaporated from the diffusion tube in unit time is expressed by

$$n_w = \frac{DSe_w}{LRT} \qquad (1)$$

where D is the diffusivity of Fick's first law, S is a crosssectional area of the diffusion tube, e_w is the vapor pressure at the bottom of the tube, L is the length of the tube, R is the gas constant, and T is absolute temperature, respectively. We used pure nitrogen gas of grade 1, where H_2O concentration is regulated less than 1 ppm, for dry gas. Wet gas with a 5 ppm moisture were always produced at 50 ccm using a mass flow controller. The dry and wet gases were alternatively introduced into a sensor cell by a rotary valve.

In order to compensate the temperature effect of ϕ1mm quartz sensors, the phase response at 240 MHz was subtracted from that at 160 MHz. Also, in ϕ3.3mm langasite (LGS) sensor operated at 150

MHz, the response of a sensor without film was subtracted from that of a sensor coated by sodium dodecylbenzenesulfonate (SDBS) film, a stationary phase material of gas chromatograph for the separation of polar gases.
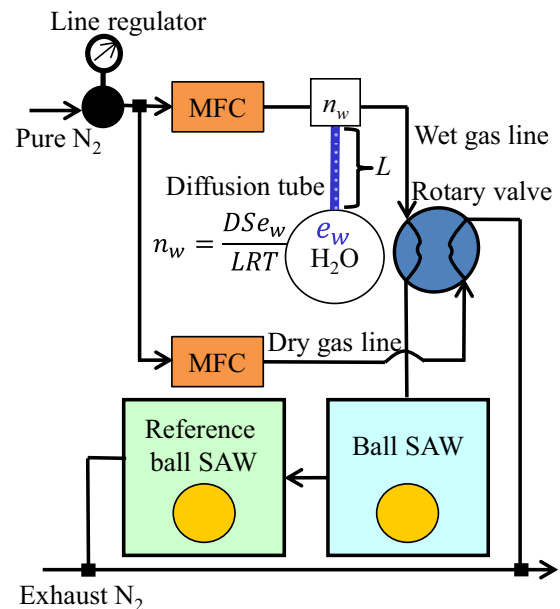

Fig.1 Schematics of measurement implementation.

3. Result

3.1 ϕ1mm 160 MHz quartz ball SAW sensor

The delay time change of ϕ1mm quartz sensor is shown in **Fig.1 [5]**. **Fig.1(a)** shows a result of 160 MHz sensor. Though it had no sensitive film, response to the 5 ppm moisture was clearly observed, overlapped with a long period thermal drift due to the temperature variation of the gas. On the other hand, the response to the moisture of 240 MHz sensor was not clear, as shown in **Fig.1(b)**. **Fig.1(c)** showing the differrence between **Figs.1(a)** and **(b)** revealed clear response of 1.5 ppm moisture, as a result of the compensation of the thermal drift. The detection limit at signal to noise ratio (S/N) of 3 was 4.9 ppb moisture corresponding to a dew point of -109℃.
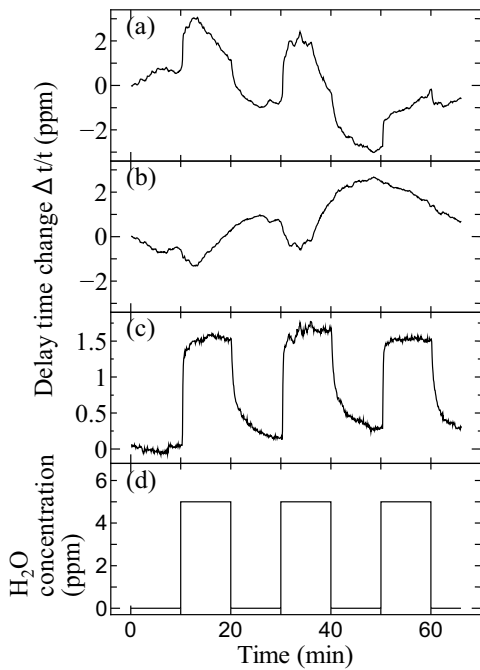
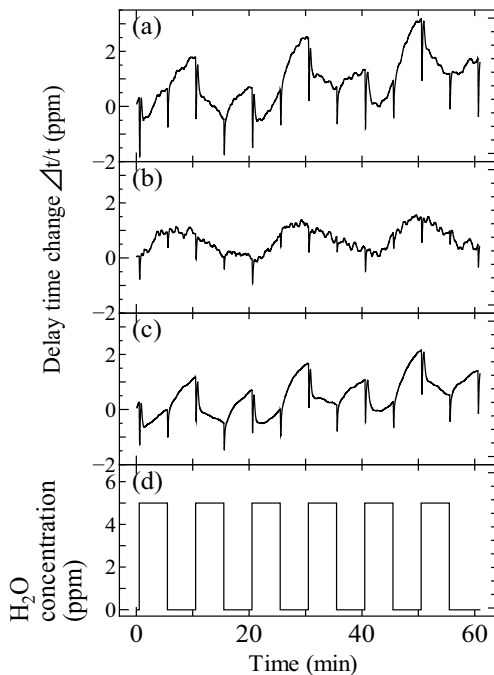Fig.2 Delay time response of φ1mm quartz sensor (a)160 MHz (b)240MHz (c)Difference between (a) and (b) (d)Concentration of H₂O.

Fig.3 Delay time response of φ3.3mm LGS sensor (a)SDBS (b)no film (c)Difference between (a) and (b) (d)Concentration of H₂O.

3.2 φ3.3mm SDBS-coated LGS ball SAW sensor

The delay time response of SDBS-coated LGS sensor to the 5 ppm moisture is shown in **Fig.3**. Similar to **Fig.2**, the differential response of **Fig.3(c)** revealed about 1 ppm response. Insufficient elimination of the thermal drift is probably caused by the delay of the temperature change of the reference sensor because the distance between two sensors (about 5 cm) was further longer than that in the case of φ1mm (about 5 mm).

In contrast to the sensor in 3.1, this sensor was also able to measure the attenuation change (**Fig.4**). Since the 5 ppm moistue caused a change of about 0.15 dB/m, the detection limit at S/N=3 was 0.53 ppm moisture corresponding to the dew point of -85℃.
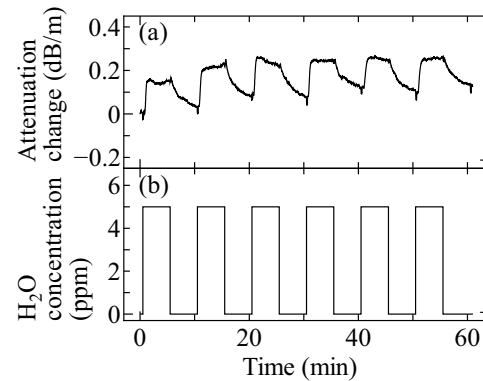

Fig.4 Attenuation change of φ3.3mm LGS sensor (a) attenuation change (b) Concentration of H₂O.

4. Conclusion

We demonstrated that the ball SAW sensor was able to measure trace moisture. In future, we plan to solve problems of the differential measurement in the delay time response, and investigate the performance of φ3.3mm sensor which is practical to the production of devices and the surface treatment for enhancing the sensitivity.

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