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Development of compact, fast, and sensitive ball SAW trace moisture sensor using amorphous SiOx film

非晶質 SiOx 膜を用いた小型・高速・高感度なボール SAW 微量水分計の開発

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1. Introduction

There are some unmet need in the trace moisture measurement of clean gases used in semiconductor and energy industries: namely the sensitivity and the response time [1]. To meet the need, we have explored the possibility of trace moisture measurement using a 3 mm ball SAW sensor incorporating the unique phenomenon of naturaly collimated surface acoustic waves on a sphere[2,3,4]. This paper demonstrates, for the first time, a very fast and sensitive trace moisture sensor (TMS) capable of mesuring the frost point -80°C(concentration 540 ppb) or less within 1 min.

2. Ball SAW TMS

A schematic illustration of TMS is shown in **Fig. 1**. It was composed of burst-waveform under sampling (BUS) circuit [5, 6], a sensor unit mounting the ball SAW sensor, rf cable, and a computer for control and data processing.

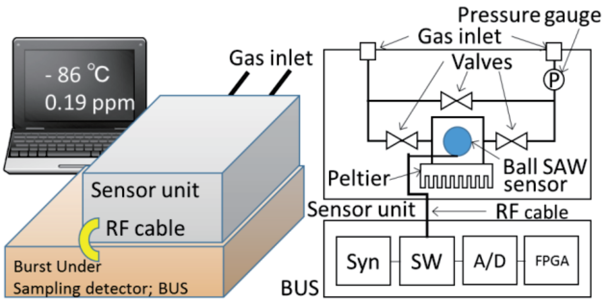

Fig. 1 Schematics of the ball SAW TMS.

The BUS circuit excites SAW with two frequencies of 80 MHz and 240 MHz, and acquires the waveform after narrow band-pass filtering. The sensor unit has a gas inlet port of a VCR connector. Introduced gas goes through over the ball SAW sensor. For monitoring pressure, absolute pressure gauge is mounted behind the sensor.
Since H2O absorption of amorphous SiOx filmdepends on the temperature, the sensor temperature is kept constant by a Peltier element.

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Three valves are installed to avoid contaminating the TMS during transportation. The ball SAW sensor is isolated from the gas flow during transportation or initial purge of the connection. The developed TMS is shown in **Fig. 2.**

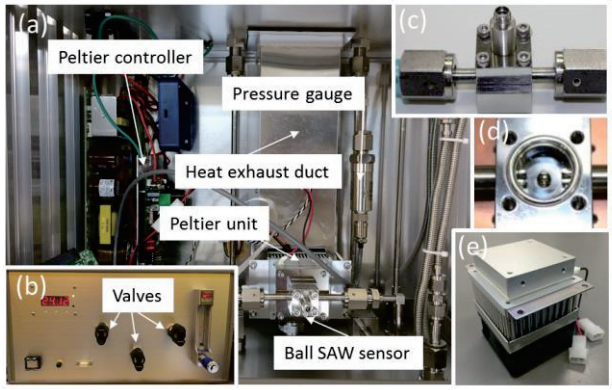

Fig. 2 Prototype of the ball SAW TMS. (a) Sensor unit from the top. (b) Front panel. (c) Ball SAW sensor cell. (d) Installed sensor. (e) Peltier unit.

3. Experiment

A quartz harmonic ball SAW sensor (3 mm diameter, 80 MHz fundamental frequency) coated with amorphous SiOx film prepared by sol-gel method was set in ultra-high vacuum cell [4]. The nitrogen gas flow rate of trace moisture generator [3] was increased to 1 L/min from previous study [3, 4] for simulating practical use.

We measured the delay time by using the wavelet analysis [2]. The temperature drift was compensated by two-frequency measurement [5,6]. The delay time change was converted to H_2O concentration by a calibration with the data of a cavity ring down spectroscopy (CRDS) [1] connected to outlet of the TMS in a preceding measurement.

4. Results

4.1 Response to trace moisture

The responses of the ball SAW TMS and the CRDS to moisture change are shown in **Fig. 3.** Both sensors measured the six steps of change in the trace moisture concentration, and the measured value of the H2O concentration was almost the same. This shows that the ball SAW TMS is as sensitive as CRDS. In the first step from 2.4 ppb to 18 ppb at 1 h, the response of CRDS was delayed more than 30 min (**Fig.3 (b)**). It was probably due to the moisture adsorption to inner wall of a pipework and a laser cavity.

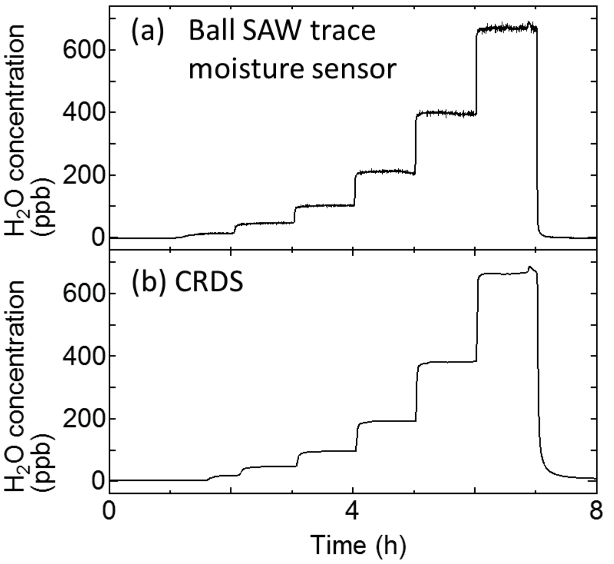

Fig. 3 Comparison of sensor responses to trace moisture. (a) ball SAW TMS and (b) CRDS.

4.2 Response time

To evaluate the response time, the response at the step from 390 ppb to 680 ppb is shown in **Fig.4**.

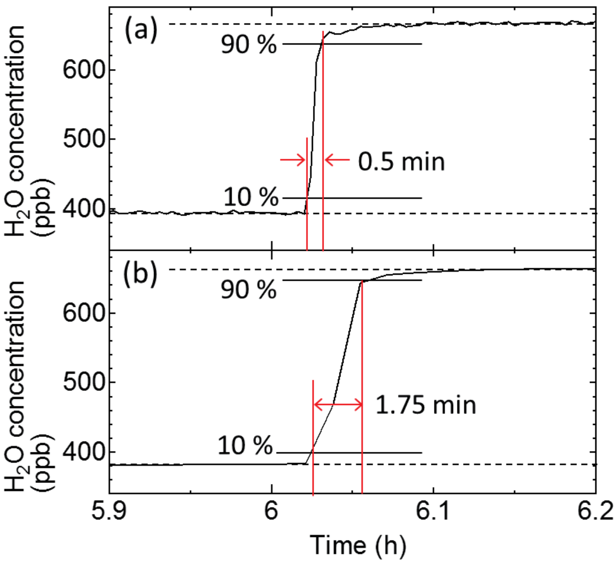

Fig. 4 Comparison of 10%-90% response time. (a) Ball SAW TMS. (b) CRDS

The 10%-90% response time of the ball SAW TMS was 0.5 min, (a), and that of CRDS was 1.75 min, (b). Thus, the ball SAW TMS was 3.5 times faster than the CRDS.

The comparison in other steps is summarized in **Table 1**. In all steps, the response time of the ball SAW TMS was shorter than that of the CRDS. Hence, it was established that the ball SAW TMS can measure the frost point change at frost point below -80°C (H2O concentration 540 ppb) with response time less than one minute.

Table 1 Response time at each step of the H_2O concentration change.

	Ball SAW (min)	CRDS (min)	CRDS/Ball
48→96 ppb	3.9	7.0	1.8
96→190 ppb	1.8	3.1	1.7
190→380 ppb	0.7	1.9	2.7
380→680 ppb	0.5	1.8	3.5

5. Conclusions

We developed the ball SAW TMS consisting of BUS with two-frequency measurement and the sensor unit mounting a 3 mm ball SAW sensor coated with amorphous SiOx film. It has a measurement sensitivity comparable to that of CRDS, and response time faster than the CRDS.

From this result, it is expected that the SAW sensor contributes to the measurement of clean gases used in semiconductor and energy industries.

Acknowledgements

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References

1. H. H. Funke, B. L. Grissom, C. E. McGrew, and M. W. Raynory, Rev. Sci. Instrum.74, 3909 (2003).
2. K. Yamanaka, S. Ishikawa, N. Nakaso, N. Takeda, D. Y. Sim, T. Mihara, A. Mizukami, I. Satoh, S. Akao, and Y. Tsukahara, IEEE Trans. Ultrason. Ferroelectr. Freq. Control 53 (2006) 793.
3. N. Takeda and M. Motozawa: Int. J. Thermophys. 33 (2012) 1642
4. S. Hagihara, T. Tsuji, T. Oizumi, N. Takeda, S. Akao, T. Ohgi, K. Takayanagi, T. Yanagisawa, N. Nakaso, Y. Tsukahara, K. Yamanaka, Jpn. J. Appl. Phys. 53 (2014) 07KD08.
5. T. Nakatsukasa, S. Akao, T. Ohgi, N. Nakaso, T. Abe, and K. Yamanaka, Jpn. J. Appl. Phys. 45 (2006) 4500.
6. T. Tsuji, T. Oizumi, N. Takeda, S. Akao, Y. Tsukahara, K. Yamanaka, Jpn. J. Appl. Phys. 54 (2015) 07HD13.