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Development and Evaluation of metal MEMS Column for the Ball SAW Gas Chromatograph

ボール SAW ガスクロマトグラフのための金属 MEMS カラムの開発と評価

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1. Introduction

For the safety and security of society, development of portable gas chromatograph (GC) capable of analyzing multiple hazard and toxic gases is required. We have developed the ball surface acoustic wave (SAW) sensor, where SAW makes multiple roundtrips without diffusing by the diffraction, realizing ultra-high sensitivity of velosity and attenuation change[1]. And we also succeed to downsize the gas separation column by micro electro mechanical system (MEMS)[2,3]. Using them, we have prorosed the ball SAW GC[4].

However, ordinary MEMS column is rather expensive and fragile because it is fabricated from Si and glass substrate. Purpose of this work is to develop a low cost and robust metal MEMS columns by using wet etching and diffusion-bonding and to evaluate it by ultrasonic imaging and SAW sensors.

2. Ball SAW gas chromatograph

Figure 1 shows a concept of ball SAW GC. Sample gas is injected into the MEMS column with carrier gas. Each component of sample gas is separated by retention time based on adsorption to the column stationary phase. Each component is detected by delay time and amplitude change of ball SAW sensor to give a chromatogram. Ball SAW GC is operated at room temperature with low power consumption without a need for heating, and is also suitable for miniaturization for explosion-proof.

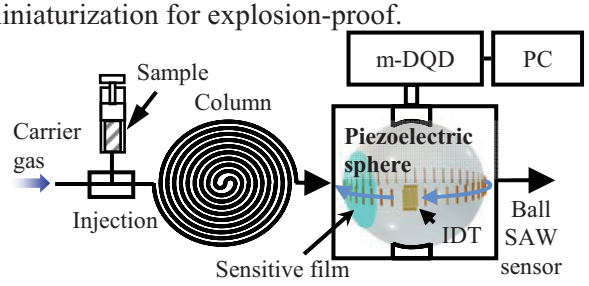

Fig. 1 Concept of ball SAW GC

3. Fabrication of metal MEMS columun

3.1 Formation of channel

Figure 2 shows a fabricaion of metal MEMS column. The through channel of 0.5 mm width was formed into a 0.4 mm thick stainless steel plate by wet etching. Next, it was sandwiched by a plate with gas inlet holes and a raw plate. And they were diffusion bonded.

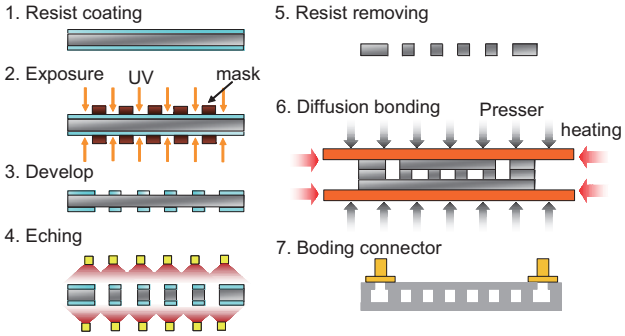

Fig. 2 Fabrication process of metal MEMS column

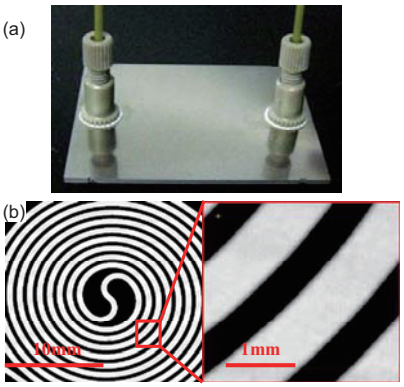

Fig. 3 Metal MEMS column (a) photo
(b)Amplitude image of reflection wave

Figure 3(a) shows a picture of the column. As the bonded interface could not be optically observed, we used ultrasonic imaging at 50 MHz. Fig. 3(b) shows a reflection amplitude image with a focus on the interface. The white area represents area of large amplitude, showing the channel. The area between them shows the channel wall, and if

the bonding is complete, the amplitude is zero. It was evaluated there was no bonding defect.

Next, to separate fuel cell related gases, we packed molecular sieving carbon (MSC) and active carbon (AC) at the filling pressure of 4.0 and 2.0 MPa. To separate natural gases, styrene divinylbenzene (SDB) was packed at 4.0 MPa. There was no damage by the packing as a result of observing the channels by the ultrasonic image similar to Fig.2.

3.2 Evaluation of ventilation characteristic

For evaluation of the packing, we measured the ventilation characteristic of columns. The flow rate of carrier gas of column is given by

$$V_t P_0 L = \frac{aB_0}{2\mu}\left(P_i^2 - P_0^2\right) \qquad (1)$$

where P_i and P_0 are the inlet and outlet pressures, L is the channel length, a is the channel cross section, μ is the viscosity of carrier gas, and B_0 is the specific transmission coefficient. When the flow resistance due to the packing is large, the coefficient B_0 becomes small.

Figure 4 shows the measured flow rate in the columns packed with MSC, AC and SDB. The result of fitting by eq. (1) is shown by the solid line. The coefficient B_0 of MSC packed column was larger than that of AC and SDB, showing smaller flow resistance.

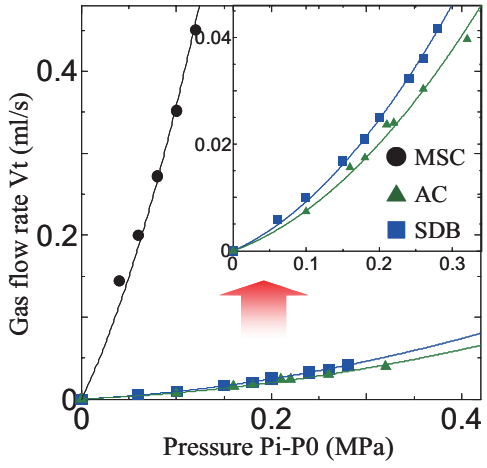

Fig. 4 Relation between pressure and flow rate

4. Separation measurement of mix gases

Figure 5 shows separation of 3% hydrogen (H_2) and N_2 in fuel cell gases by using AC packed column and thermal conductivity detector of a desktop GC. We succeded to separate them. However, we could not separete them by using the MSC packed column with low packing pressure.

Next, we installed a SDB packed column to the ball SAW GC as shown fig. 6 and analyzed the equal concentration mix gas of the methane, ethane and propane.

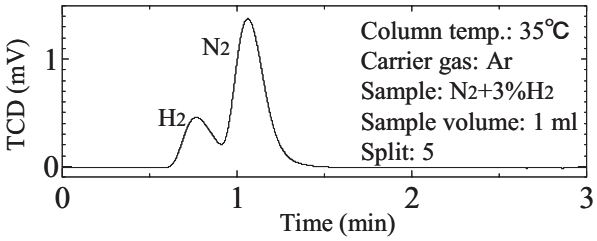

Fig. 5 Separation of H_2 and N_2 in fuel cell gases

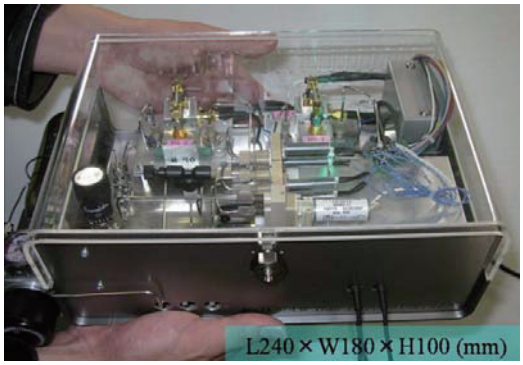

Fig. 6 Ball SAW GC

Figure 7 show a chomatogram, showing success in separation and detection of each component. This performance was comparable to that of silicon MEMS column made before[3].

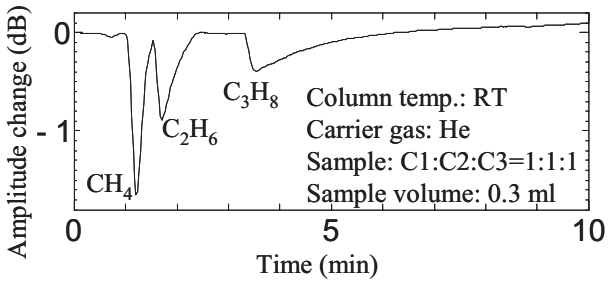

Fig. 7 Separation of natural gas components

5. Conclusion

We developed a metal MEMS column by wet etching and diffusion bonding and evaluated the channel by using ultrasonic imaging and gas separation measurement. It is concluded that low cost and highly robust metal MSMS column was developed useful for portable GC.

Reference

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