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Development of high precision metal MEMS column for portable SAW gas chromatograph

可搬型SAWガスクロマトグラフのための高精細メタルMEMSカラムの開発

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1. Introduction

For realizing safe and secure society, it is necessary to develop portable gas chromatograph (GC) which can analyze multiple gases. Column, the device for GC used for the separation of gases, can be downsized using micro-electromechanical system (MEMS) technologies[1,2]. However, the ordinary MEMS column is fragile and expensive because it is made from silicon and glass. In this study, we developed a robust and inexpensive high precision metal MEMS column using the wet etching and diffusion bonding of metal plates. Then, we evaluated it with nondestructive test using acoustic microscopy and analysis of multiple gases using portable ball SAW GC.

2. Fabrication and nondestructive testing of metal MEMS column

Fig.1(a) shows a concept of GC and an implementation shown in (b). Sample gas is injected into a column with carrier gas. Different gas components are separated because of different retention times based on adsorption to the column stationary phase. The separated gases are detected by the sensor to give a chromatogram.

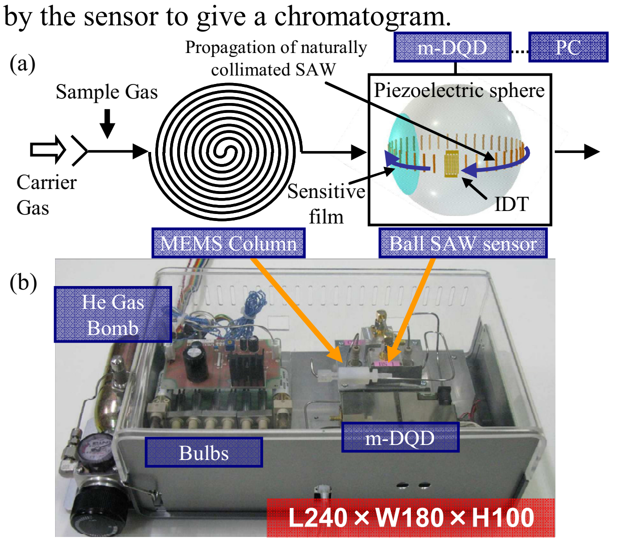

Fig. 1 Portable GC (a) Concept (b) An implementation (Ball SAW GC)

Fig.2(a) shows the fabrication process of a metal MEMS column. It was fabricated by diffusion bonding of two stainless steel plates (SUS304, 0.5 mm thickness) with 240 μm width, 75 μm deep, and 2.8 m long channel formed by the wet etching. Fig.2(b) shows a photo of the column.

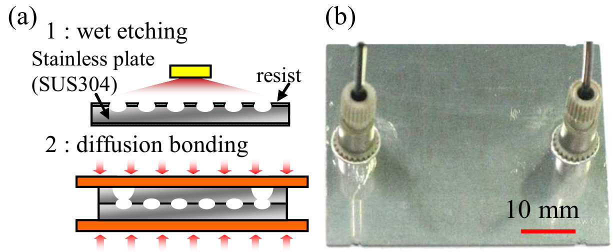

Fig. 2 Metal MEMS column (a) fabrication process (b) photo

Since the bonding interface can not be optically observed, we used an acoustic microscope operated at 180 MHz. Fig.3(a) shows reflection amplitude image with a forcus on the interface. Fig.3(c) shows the reflected waveforms at the three points in the magnified iamge in Fig.3(b). The large amplitude echo **S** at 6.20 μs and **C** at 6.35 μs on a point 1 located on the the channel represent refrection from the column surface and the interface, respectivery. Point 2 is located on the bonded area. The acoustic wave is transmitted through the interface as shown in Fig.4 and the back surface echo **B** is observed.

Since waveform 3 shows an echo **I** at 6.37 μs from the interface, the non uniform area parallel to the channel may be a surface formed by misalignment in the diffusion bonding. Then we cut the column, observed the cross section and confirmed a misalignment of bonding as shown in Fig.4. Here, the red arrows represent propagation paths of acoustic waves.

As shown in Fig.3(d), there are many white spots. Fig.3(e) shows reflected waveform at one of the spots. The horizontal axis represents delay time from column surface reflection. The large amplitude echo **D** at 0.17 μs shows a reflection from the interface coused by the bonding defects like a void. Many defects can cause leakage between the neighboring channels.

Then we selected correctly aligned column without many defects and coated 5% Phenyl- 95% Methylpolysiloxane as stationary phase on the channel wall.

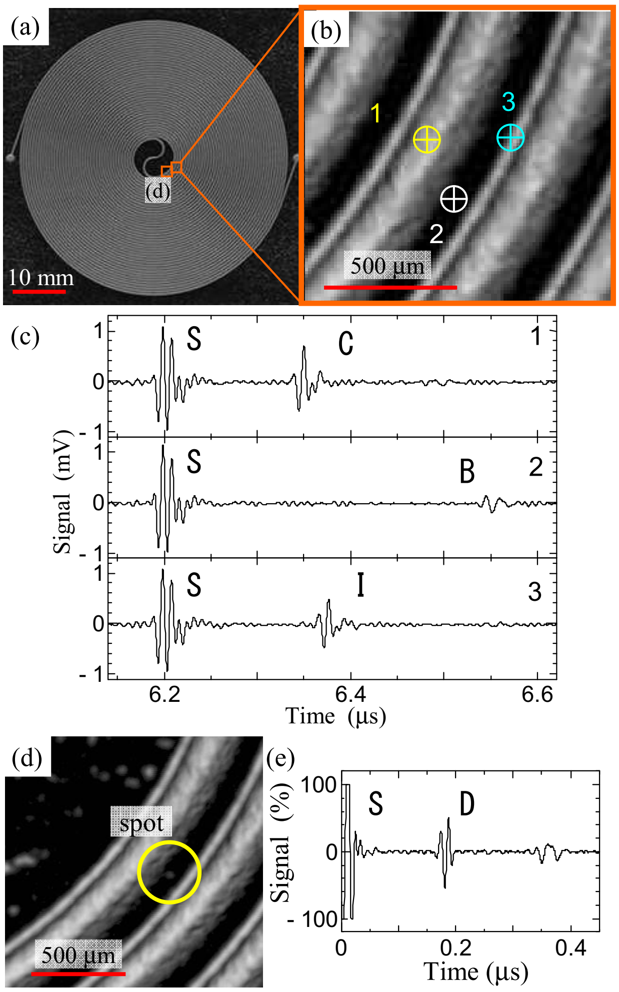

Fig. 3 Nondestructive test by acoustic microscopy (a) Full image (b) Magnified image (c) Waveform at point 1, 2 and 3 (d) Spots (e) Waveform at a spot

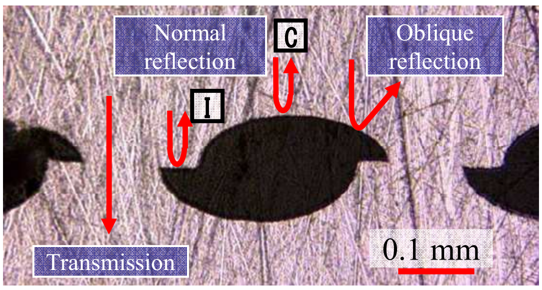

Fig. 4 Cross section of a misaligned column

3. Separation measurement of mixed gases using portable GC

We evaluated the metal MEMS column in a ball SAW GC shown in Fig.1(b), using the ball SAW sensor where SAW makes multiple roundtrips[3]. Then, each gas component is detected by the delay time and amplitude change of ball SAW sensor.

Fig.5 shows a chromatogram of arbitrarily mixed nonpolar volatile organic compounds (VOCs) coated with a ball SAW sensor coated by dimethyl polysiloxiane (PDMS) sensitive film. We succeeded in separating and detecting each component at room temperature. We also succeeded in analyzing mixed polar VOCs using a ball SAW sensor coated with Siponate DS-10 (Fig.6).

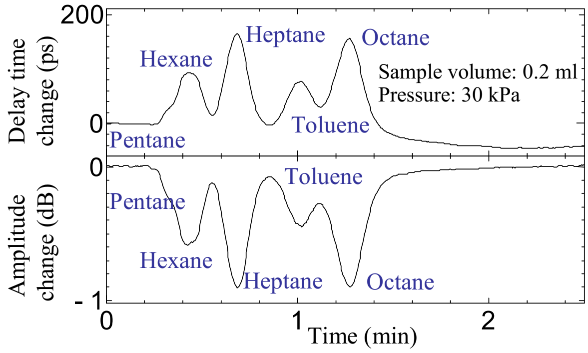

Fig. 5 Separation of nonpolar VOCs by ball SAW GC

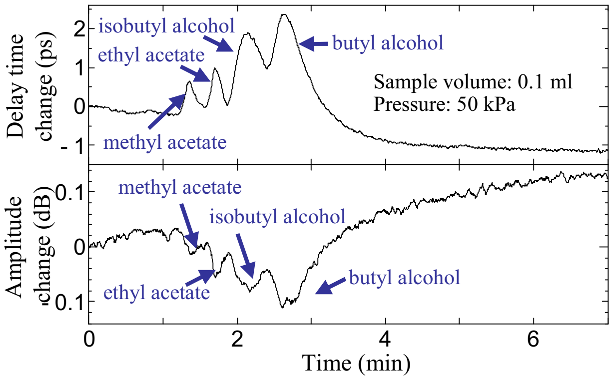

Fig. 6 Separation of polar VOCs by ball SAW GC

4. Conclusion

We developed a metal MEMS column using wet etching and diffusion bonding of stainless steel plate, and tested it using acoustic microscopy. Then, we evaluated it in portable ball SAW GC, and succeeded in separating and detection of mixed gases at room temperature. It is more robust and inexpensive than silicon MEMS column. It is more suitable for transfer, compact and handy than metal capillary column, since the channel is formed into a flat plate. It is expected to be useful in portable GC.

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