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Development of a Portable Ball SAW Gas Chromatograph Using Three-layered Metal MEMS Columns

三層構造のメタル MEMS カラムを用いた可搬型ボール SAW ガスクロマトグラフの開発

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1. Introduction

In solar system exploration, a portable gas chromatograph (GC) is an important instument for the on-site analysis of the surface materials on the moon, planets, and small bodies. It is also usuful to monitor the cabin atmosphere in the spacecraft and the International Space Station (ISS) for the human exploration. We have developed a GC system equiped with a ball surface acoustic wave (SAW) sensor[1,2) : a ball SAW GC[3-5)]. The key features of our ball SAW GC are the multiple roundtrips of the SAW on a spherical crystal and a metal micro-electro-mechanical-system (MEMS) column coated with a stationary phase in a 3 m long microchannel formed by diffusion bonding of etched stainless-steel plates.

In this study, we further fabricated a three-layered microchannel with the length of 10 m to improve gas separation performance and developed two types of metal MEMS columns using the channels. In addition, we succeeded to minimize the size of the ball SAW GC to a 10 cm cube including these metal MEMS columns. The performance of this system was examined by analyzing multiple volatile gases.

2. Three-layered metal MEMS column

Fig.1(a) shows the fabrication process of the three-layered microchannel. First, channels with 330 µm width and 0.185 µm depth were formed by wet etching on both sides of a stainless-steel plate (SUS304, 0.5 mm thick). Next, a through hole of φ0.25 mm was drilled to connect channels of both sides. Finally, the plate was sandwiched between two stainless-steel plates (0.2 mm thick), each of which with through holes for connecting the outside and the channel. and the three plates were joined by using diffusion bonding. Fig.1(b) shows a photo of the column. A 10 m channel was formed in the area of 71 x 43 mm and 0.9 mm thick. The inlet of the channel is at the center of the protrusion part, and the outlet is at the same position on the back side. Consequently, the channel length can be increased by stacking the columns via O-rings.

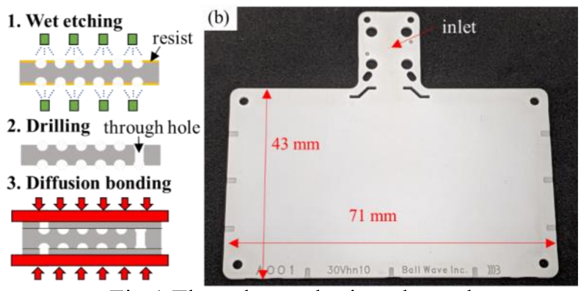


Fig.1 Three-layered microchannel.
(a) fabrication process. (b) photo.

To evaluate the fabricated channel, we performed the nondestructive testing using an acoustic microscope operated at 200 MHz. **Fig.2(a)** shows an acoustic image of the channel focused at bonding interface. Fig.2(c) shows the reflected waveforms at three points in the enlarged image of Fig.2(b). Position 1 is located on the bonding area and 2 is located on the channel. The large amplitude echo S at 6.95 µs and echo C at 7.02 µs represent reflection

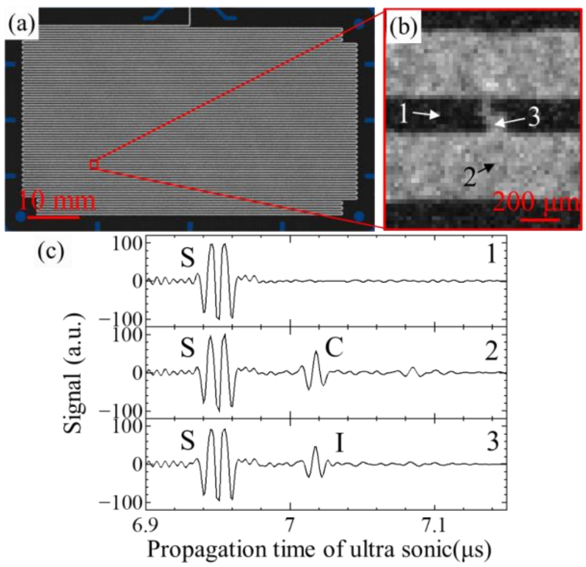


Fig.2 Nondestructive test of a channel by an acoustic microscope. (a) Full image. (b) Enlarged image. (c) Waveforms at point 1, 2 and 3.

from the column surface and interface of the channel, respectively. Position 3 is in the bonding area, but echo I with the same propagation time as echo C was confirmed, suggesting a defect with a risk of connecting neighboring channels. A possible cause of this defect is an excessive etching by delamination of the resist, or inclusion of foreign matters during the bonding. Such defects might branch off the flow path leading to unintended peaks in the chromatogram or cause inhomogeneous thickness of the stationary phase reducing the gas separation performance.

Then, we selected defect-free channels and coated each column with 5% diphenyl - 95% polydimethylsiloxane (PDMS) and polyethylene glycol (PEG) as the stationary phases were coated.

3. 10cm ball SAW GC

The prototyped portable ball SAW GC that implements the forward flush method[1,2] using two pairs of columns and sensors is shown in **Fig.3**. The carrier gas was supplied from a hydrogen storage canister, and the flow rate was controlled by a pressure regulator. The first column (CL1) was a 10 m long metal MEMS column coated with 5% diphenyl-95% PDMS. The second column (CL2) was 30 m connecting three 10 m long metal MEMS columns coated with PEG. The column temperature was controlled by seat heaters mounted on both sides of the column and cooling fans. The first sensor (BS1) with a PDMS sensitive film was connected to the outlet of CL1, and the second sensor (BS2) with poly-N-vinylpyrrolidone sensitive film was connected to the outlet of CL2. In addition, it included a gas sampler unit equipped with a pre-concentrator[6], a detection circuit for operating a ball SAW sensor, and a control circuit for valves and heaters. This GC was 10 x 10 x 10 cm in size and weighed 762 g.

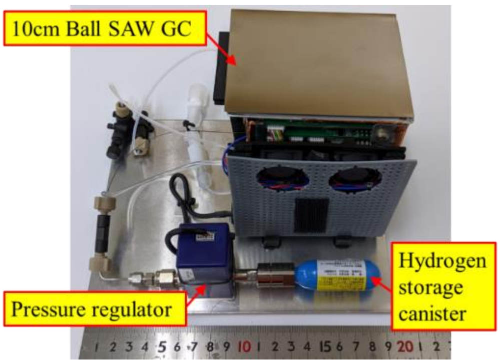
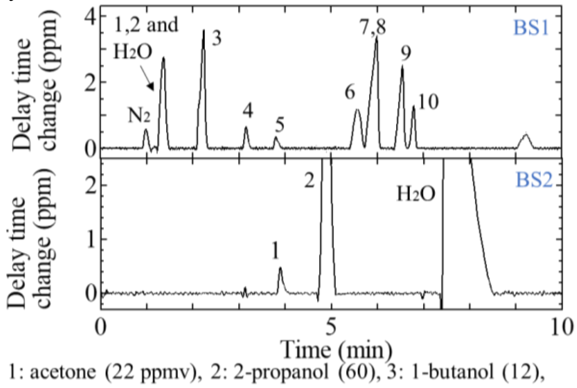


Fig.3 10cm ball SAW GC.

4. Analysis of multiple gases

We mixed 10 kinds of gases selected from a list of airborne contaminants in the spacecraft[7] at the maximum allowable concentrations for a 180-day exposure and analyzed them using the prototype GC. The sample gas was collected at a flow rate of 33 ml/min for 2 minutes. The column temperature was maintained at 40°C for 5 minutes and raised to 140°C at the rate of 10 °C/min. The chromatograms are shown in **Fig.4**. Acetone, 2-propanol and H_2O mixed during the sample preparation were not separated by CL1 and detected as one peak by BS1. They were separated by CL2 and detected by BS2. Other gases were separated by CL1 and detected as clear peaks by BS1.



1: acetone (22 ppmv), 2: 2-propanol (60), 3: 1-butanol (12), 4: toluene (4), 5: n-octane (3), 6: ethylbenzene (12), 7: m-xylene (8.5), 8: p-xylene (8.5), 9: o-xylene(8.5), 10: nonane (3)

Fig.4 Chromatograms by ball SAW GC. The number in parentheses indicates concentration.

5. Conclusion

We have developed two types of three-layered metal MEMS columns with different stationary phases, evaluated by the nondestructive testing using an acoustic microscope. Using this column, we have developed the prototyped ball SAW GC of 10 cm cube and 762 g weight. This GC equipped with the same functions as those of the desktop GC that include the sampler, the pre-concentrator, and the column temperature control. It was succeeded in separating and detecting 10 kinds of gases within 10 minutes measurement and showed the capability of the on-site analysis of multiple gases.

Acknowledgment

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