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Development of Forward Flush Method for Ball SAW Gas Chromatograph
ボール SAW ガスクロマトグラフのためのガス直進法の開発

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1. Introduction

In the fields of energy exploitation or the safety and security, handy multiple-gas sensors are essential. The gas chromatograph (GC) is frequently used as a multiple-gas sensor but it is not handy. We have proposed the ball SAW GC, using the ball surface acoustic wave (SAW) sensor where SAW makes multiple roundtrips without diffusing by the diffraction[1, 2] and the micro separation column fabricated by MEMS(Micro Electro Mechanical System) technology[3].

On the analysis of dangerous gases and natural gases, fast analysis is needed. As a means of fast analysis, the GC with back flush (BF) method was developed[4,5]. However, it needs a complicated valve system and a double injection to increase the number of analysis gases [5]. In addition, gases analyzed once cannot be reused because the detector is mounted at last of analysis line. This is the case also in GCs using a SAW sensor [6, 7]. On the other hand, the ball SAW sensor has a nondestructive property where the gases analyzed once by the sensor are not degraded and can be reused. In this study, using the nondestructive property, we propose a forward flush (FF) method that can simplify valve systems and analyze wide variety of gases by single injection by mounting ball SAW sensors at mid and last of analysis line.

2. Development of Forward flush method

First, for comparison with existing methods, I explain the advantage and problem of the BF method. In general, retention time of large molecular weight is very long using a column that can separate small molecule weight gases. A BF method separates weak and strong retention force gases with a weak retention column 1(Fig. 1). Next, carrier gas is flew back in a column 1 by switching flow paths, molecular weight gases are first detected. After sample gases are injected again, gases are back flushed using column 1 and 2, and detect small weight molecular gases. Thus, detection time is shortened. However, it needs many valves, and thus it is complicated and large.

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Furthermore, this system needs explosion protection because of using a thermal conductivity detector (TCD) requiring high temperature operation [4]. Then, double injection BF method GC [5] shown by Fig. 1 can increase the number of analysis gases. However, detection time is increased because of the double injection.

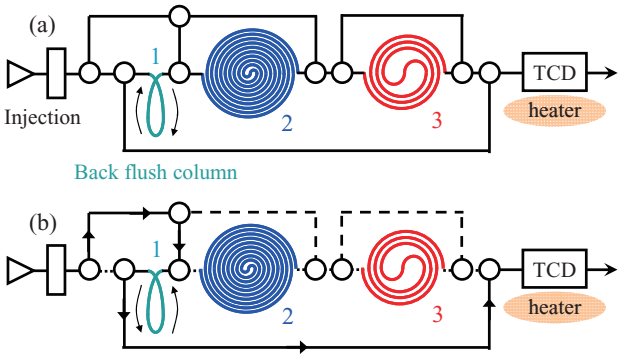

Fig.1 schematic diagram of the back flush method (a) configuration diagram of back flush method (b) after flow path switching

On a FF method shown by Fig. 2, we use a weak retention force column (CL1), strong retention force column (CL2) and two nondestructive sensors (here, ball SAW sensors). At the time of sample gas injection, flow paths are like Fig. 2 (a). First, gases A~G are flow in CL1. Large molecular weight gases (D~G) are retained and separated by CL1, however, small molecular weight gases (A~C) are not retained and pass through a CL1 and first ball SAW sensor (BS1). Then, peak of A-C shown by Fig. 2 (a) is detected. At this time, a ball SAW sensor detect gases nondestructively, so gas property is stationary. At the point, sample gases don't come at a second ball SAW sensor (BS2).

Next, flow paths is put into Fig. 2 (b) by switching valves shortly after gases detected as an overlapping peak by BS1 flow in CL2. Afterward, D~G retained by CL1 are separated and detected peak of D, E, F, and G by BS1 (Fig. 2(b)). A~C not separated by CL1 are separated by CL2 and detected peak of A, B and C by BS2 (Fig. 2 (b)).

As will be appreciated from the above explanation, the FF method GC can downsize the GC because of fewer valves, and detection time is

short because of the single injection. It is small and light, because of no explosion protection by using the ball SAW sensor available at room temperature, as the common advantage of ball SAW GC [3]. In this paper, a FF method using two type of columns is proposed, but it is not limited to two columns. By using three or more columns with different retention forces, we can analyze more gases.

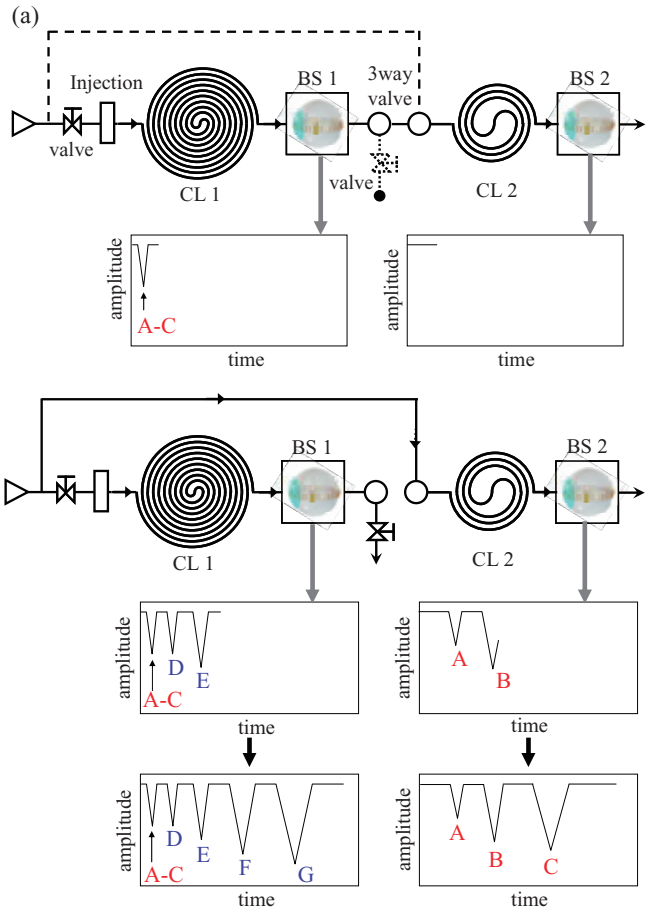

Fig.2 fundamental of the forward flush method
(a) before flow path switching (b) after flow path switching

3. Result and discussion

We separated and detected carbon dioxide, ethane, propane, benzene and toluene by the FF method as a system demonstration experiment. Fig. 3 shows responses of each ball SAW sensors. The ball SAW sensor were a 3.3 mm langasite single-crystal ball, first sensor was coated with polydimethylsiloxane as sensitive film and second was no sensitive film. A column temperature was room temperature. Flow paths are switched at 1.5 minutes. Overlapping peak of carbon dioxide, ethane and propane, benzene and toluene were separated by Open tube MEMS column and detected by first ball SAW sensor. Carbon dioxide, ethane and propane were separated by Packed MEMS column and detected by amplitude response of second ball SAW sensor.

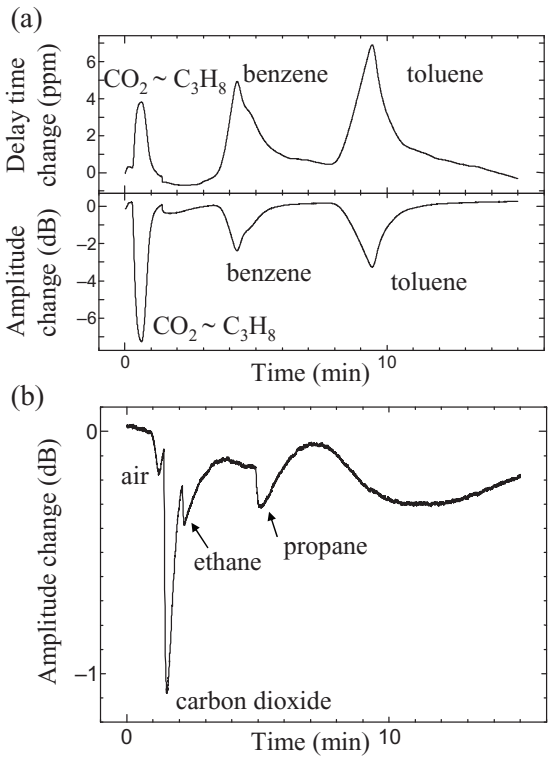

Fig.3 Gas chromatogram of sample gases separated by (a) OT-MEMS column (b) packed MEMS column

4. Conclusion

We developed the FF method to realize fast analyzable handy multiple gas sensor. This method owes to nondestructive property of ball SAW sensor. This system needs no explosion protection, so it is expected downsizing and weight saving. Moreover, sample gas injection is one time.

On a demonstration experiment, we succeeded in separation of carbon dioxide, ethane, propane, benzene and toluene, so we could substantiate the FF method. It is therefore concluded that the no explosion protection multiple gas sensor is feasible.

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