DEVELOPMENT OF A HIGHLY-RELIABLE METAL MICROCHANNEL PLATE APPICABLE TO SEPARATION COLUMN OF GAS CHROMATOGRAPHY

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ABSTRACT

We report a microchannel plate made of stainless-steel, which is applicable to separation column of gas chromatography(GC). To realize robust connection between the microchannel and outer tubing, we developed two types of interface structure. In both structure, high-temperature resistance of 400^{o}C and high-pressure tightness of 1MPa were confirmed. Using a fabricated column plate, gas chromatography analysis was demonstrated, and theoretical plates of 57,000 is obtained.

KEYWORDS

gas chromatography, column, interface, stainless steel

INTRODUCTION

In the late 1970s, the earliest work of a silicon microfabricated GC column was reported by Stanford University group[1], and recently several research groups are developing miniature GC systems[2–4]. The MEMS technology is desirable for GC development, which allows downsizing of GC whole systems, reducing thermal mass of a heating unit, integration of detectors, and other benefits. Our group also has developed a miniature GC systems using a silicon microfabricated column[5]. However, we come up against critical issues for practical uses of the silicon microfabricated column. One of the issues is fragility of the silicon column and the other is connection between the column and outer tubing. In particular, the tubing connection of GC column is difficult problem, which requires high-pressure tightness of 1MPa, high-temperature resistance of 400^{o}C, its thermal-cycle resistance, and small dead-volume in the interface. In the previous researches, to make small dead-volume connection, connection tubing or tube fitting connector was bonded with the microfabricated column by glue. However these connections could not meet the demand of temperature resistant and tightness.

In this paper, we report the improvements to eliminate the issues as mentioned above. To improve structure robustness of the microfabricated column, we developed a GC column on a metal microchannel plate. On the other hands, to realize highly-reliable connection between the column plate and outer tubing, we propose two types of interface structure.

METAL MICROCHANNEL PLATE

Figure 1 shows structure of the developed column plate made of stainless-steel 316L. The microchannel of 200µm wide, 100µm deep and 14m long is arranged in a 100mm squared plate. The microchannel was defined by photolithography and formed by chemical etching on the mirror polished stainless-steel plate. The cover plate and the channel plate were bonded by diffusion bonding. A cross-sectional observation of Fig.1(c) confirms that metal grain grew beyond the boundary of the both plate by diffusion bonding, which proves robust bonding.

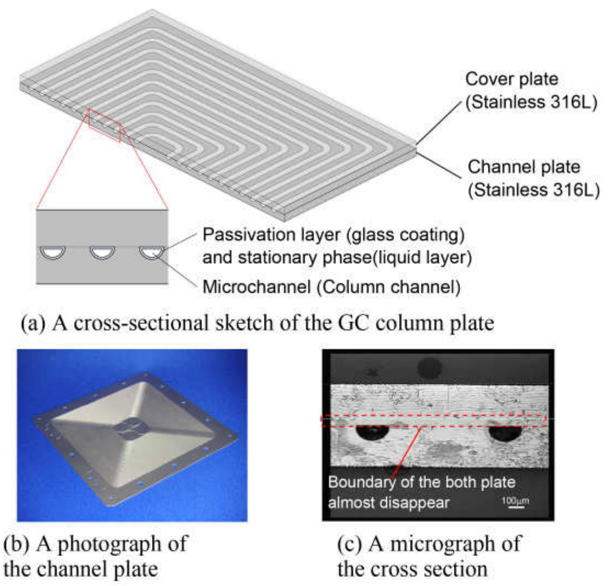


Fig.1 Microchannel structure of the GC column plate

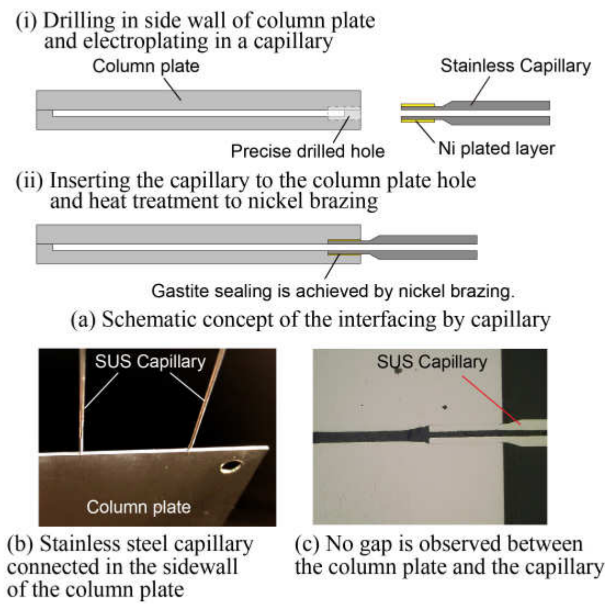


Fig.2 Interfacing with stainless steel capillary

OUTER TUBING CONNECTION

In addition to the requirements for connection of GC column as mentioned above, it is desirable that the interface structure have insignificant influence on temperature uniformity of the column plate. Therefore, the interface structure is preferable to be isolated thermally from the column plate. In order to ensure the demand, we developed two types of interface structure. In the case of Fig.2, the column plate is coupled with a stainless capillary directly. A nickel plated capillary is inserted into drilled hole in the side of the column plate. By heat treatment under vacuum, brazing bonding between the column plate and the capillary was achieved. Fig.2(c) shows a cross-sectional micrograph of the connection, which confirmed that fine bonding and extremely small dead-volume in the connection were obtained.

Figure 3 shows the other structure using metal gasket sealing. Gas-tight sealing is obtained in the protruded portion of the column plate using the metal gasket and the retainer nut. Through the retainer nut, the microchannel can connect to a capillary using the Siltite^{TM} ferrule(SGE Analytical Science Ltd). Using test pieces of the proposed interface, gas tightness was investigated in thermal cycle between 50^{o}C and 400^{o}C, as shown in Fig.4. The result shows that mass flow indicated zero except temperature varying period, in which thermal expansion and shrinkage of helium gas occurred in the microchannel. After the thermal cycle test, no leakage from the interface was detected by a leak detector with pressurized helium gas of 1MPa. From these results, it is confirmed that reliable gas-tight connection under GC operating temperature range was obtained. In addition, we investigated the sample peak deformation due to dead-volume in the connection. In case that dead-volume exists in the connection, gas flow eddies in the dead volume, and consequently, the sample peak shape deforms (i.e. peak tailing). To evaluate the peak shape deformation, a microchannel plate was connected by the proposed interface with precolumn. The sample peaks of gaussian profile established in the precolumn pass through the interfaces and the microchannel, and then the sample peaks are detected by GC-detector. Figure 5 shows comparison of the sample peak shape between the cases of with and without the test interface pieces. The result shows that no deformation of the peak shape symmetry was observed at the exit of the test piece.

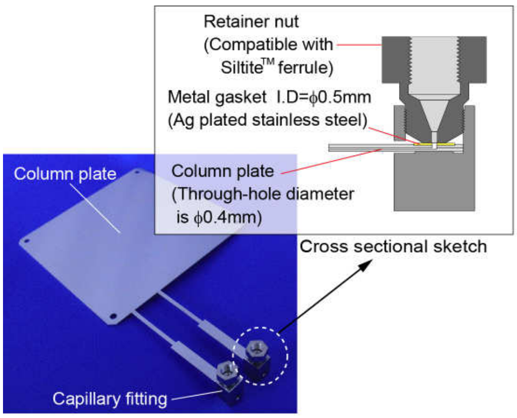


Fig.3 The interfacing using metal gasket sealing

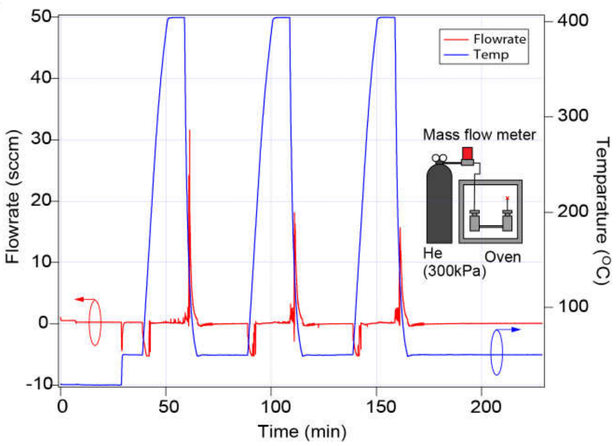


Fig.4 Leak test result in thermal cycle

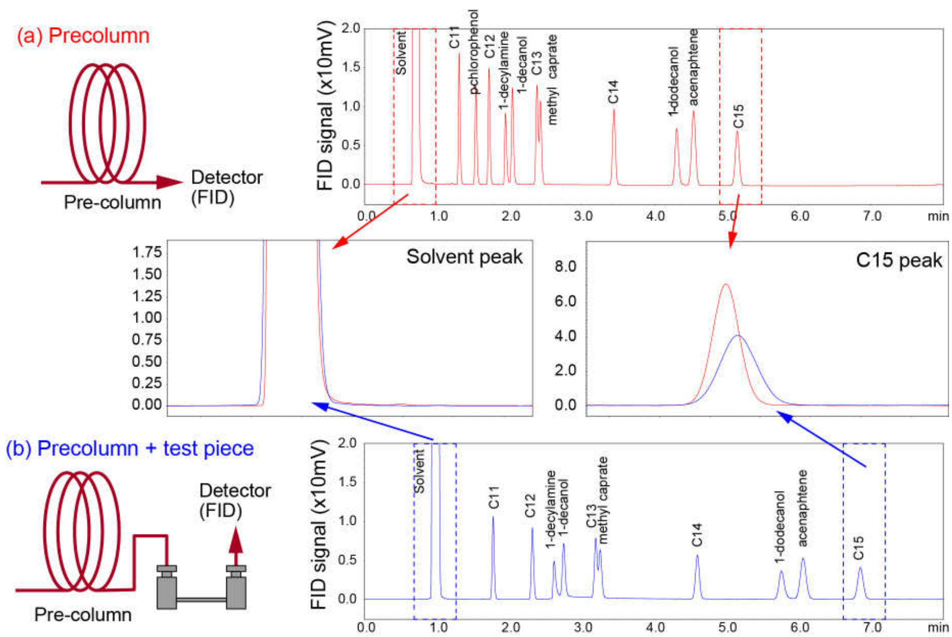


Fig.5 Evaluation results of peak shape deformation due to dead volume

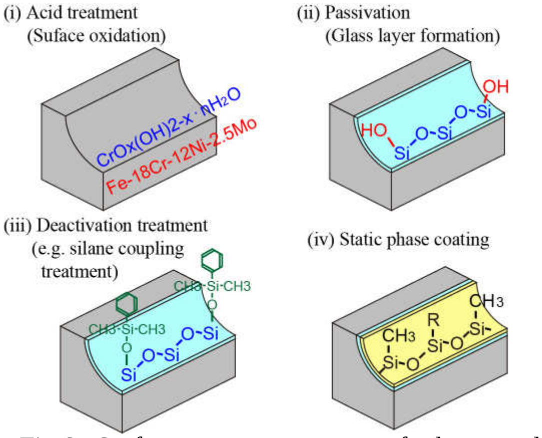


Fig.6 Surface treatment step of the metal column plate

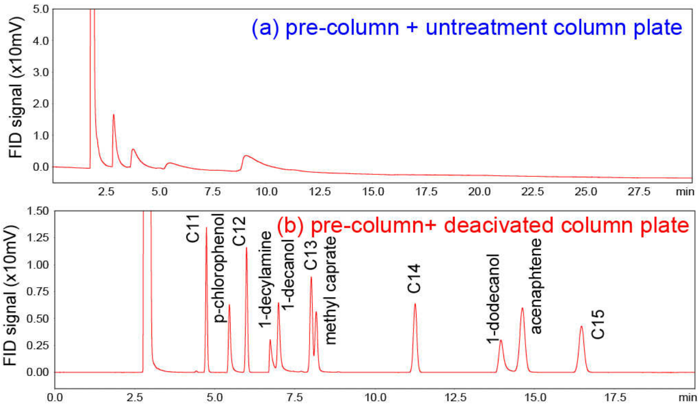
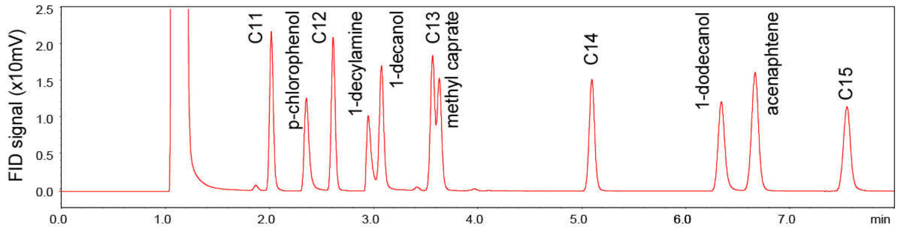


Fig.7 Evaluation results of the sample adsorption



Theoretical plates are calculated to be 57,000 in C15 peak.

Fig.8 Chromatogram obtained by a fabricated column plate

EVALUATION FOR GC SEPARATION COLUMN

In order to evaluate the microchannel plate as a separation column of GC, surface treatment of inner surface was achieved. Figure 6 shows surface treatment step of the column plate. To avoid sample adsorption by metal oxide sites, the metal surface is covered by glass passivation layer. To form the passivation layer, polysilazane was coated and was cross-linked on the channel surface. Subsequently silanol groups on the surface of the passivation layer were terminated by silylating reagent. Finally, static phase layer such as poly-methylsilicone having some functional groups was formed by static-coating method[5]. In Fig.7, we evaluated peak shape deformation due to sample adsorption on the microchannel surface. While sample peak shape deformed severely due to adsorption in the untreated column plate, no peak shape deformation is confirmed in the deactivated column plate. Finally, we show an example of chromatogram obtained by a fabricated column plate. In this chromatogram, theoretical plates are calculated to be 57,000.

CONCLUSION

In this paper, we reported a highly reliable metal microchannel plate and its outer tubing connection applicable to a separation column of GC. For the connection between the microchannel plate and outer tubing, the structure of direct capillary bonding and the interface using metal gasket sealing were proposed. In the structure of direct capillary bonding, robust connection was obtained by nickel brazing, which has extremely small dead-volume. In the interface using metal gasket sealing, gas-tightness in thermal cycle and insignificant influence for sample peak shape were confirmed. Using the fabricated microchannel plate and the interfaces, inner surface treatment was achieved and basic performance was evaluated as separation column of GC. From the results, good performance of 57,000 theoretical plates was obtained.

ACKNOWLEDGEMENT

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