RESEARCH ON MICRO-FABRICATED GAS CHROMATOGRAPHIC COLUMNS WITH EMBEDDED ELLIPITIC CYLINDRICAL POSTS

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ABSTRACT

This paper reports a newly developed micro-fabricated gas chromatographic (μGC) column with embedded elliptic cylindrical posts (ECP) in a silicon-glass architecture, which has higher surface area and less quasi-zero velocity zone compared to the state of the art. By coating polydimethylsiloxane as the stationary phase, the 2 m and 4 m columns can separate methane and ethane with 0.66 and 1.02 resolution.

INTRODUCTION

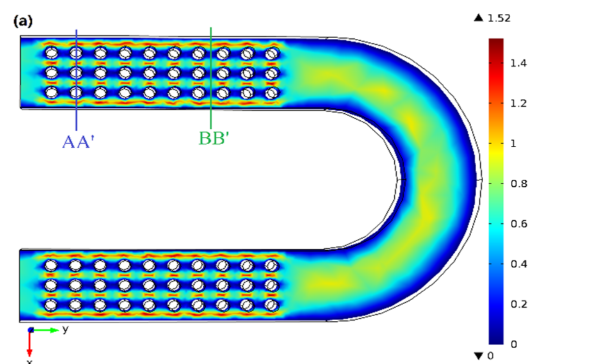
Gas chromatograph based on MEMS (Micro-electro-mechanical system) technology have attracted many researchers' great interest due to its lower cost, shorter analysis time and decreased device dimensions for field testing. A typical GC system includes injector, separation column, and gas detector. Among these components, separation column, which is the core part of the whole GC setup, plays a vital role in the final separation performance and has been intensively investigated [1,2]. Until now, apart from applying different stationary phase [3-6], various micro channel configurations have been developed to improve the resolution. This work is on the basis of a concept of semi-packed serpentine column, which has been introduced previously [7-9]. Herein, we will present a comprehensive design, fabrication and evaluation of a newly developed μGC column. Meanwhile, finite element simulations have been performed so that guidelines for structure optimizing can be provided. The new structure demonstrated in this paper differs from previous work in the geometry of micro posts, by which, larger surface area and less quasi-zero velocity zone can be provided. As a result, methane and ethane can be separated by 2 m and 4m μGC column with ECP structure.

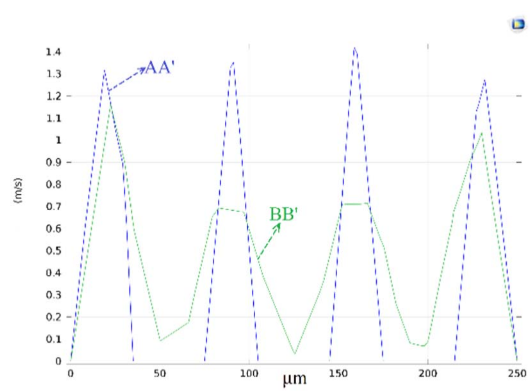
DESIGN AND FABRICATION

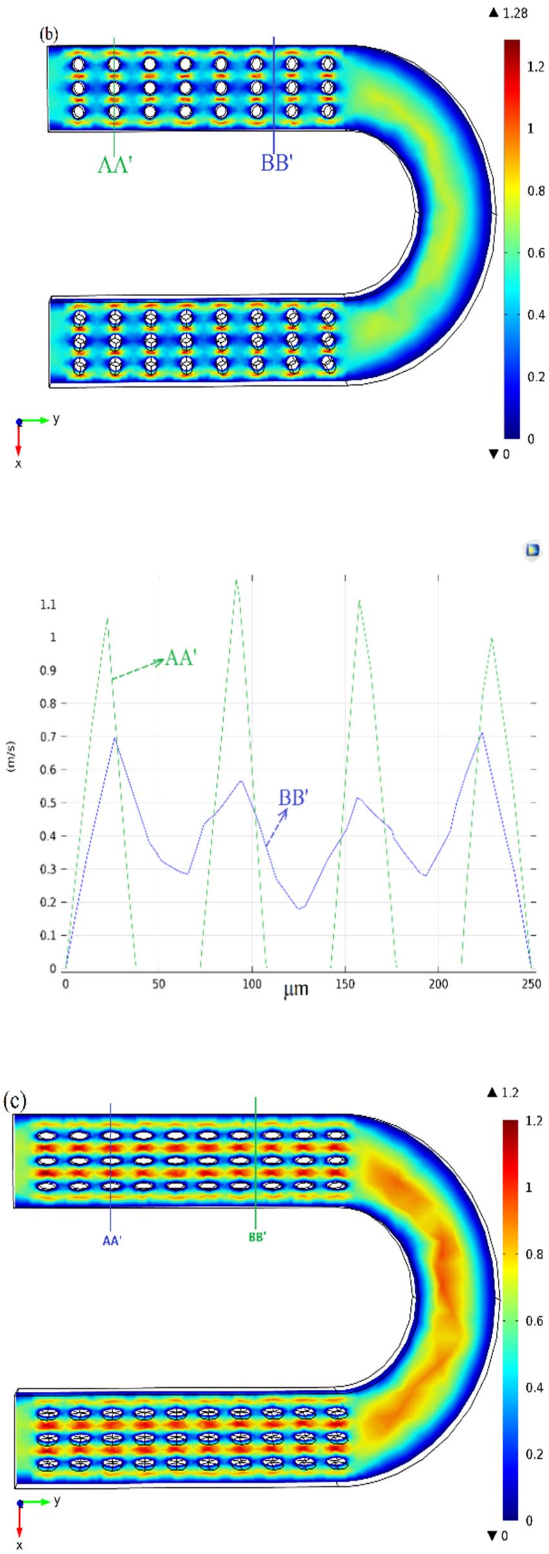
We design elliptic cylindrical posts (ECP) to replace the original cylindrical posts (CP) by theoretical arithmetic and simulations. ECP has 30 μm major axis and 10 μm minor axis, each row has three posts, the posts are spaced 80μm in horizontal orientation (y-axis in Figure 1) and 70μm in vertical orientation (x-axis in Figure 1). The radius of CP1 and CP2 is 20μm, the posts in CP1 structure are spaced 80μm in horizontal orientation, while the spacing is 120μm in CP2.The surface area of μGC column with ECP is larger than 11% of μGC column with CP1 and 211% of μGC column with CP2.

The eddy diffusion and uniformity of gas velocity are other factors that can impact μGC separation performance; we use finite element simulation to analyze these structures. In order to contrast velocity zone in the CP1, CP2 and ECP, we decrease pressure at the entrance of the column embedded with ECP and CP2, and the same gas velocity can be obtained.

According to the simulation results, we can find a lot of quasi-zero velocity zone behind CP1 (Figure 1a), the velocity profile BB' in Figure 1a is not uniform. While, in μGC column with ECP, there are less quasi-zero velocity zone (Figure 1c), and the velocity profile behind posts (BB' in Figure 1c) is more even than CP1 and CP2, which contributes to a more uniform coated stationary phase film [8] .In contrast to virtual wall model [10], the eddy diffusion term of ECP is larger than CP1 (Figure 1a) that spacing between the posts is equal to the dimension of the individual post, and is smaller than CP2 (Figure 1b) that the spacing is twice dimension of the single post. This compromise (Figure 1c) structure can decrease the eddy diffusion and can also get the even carrier gas velocity behind posts.







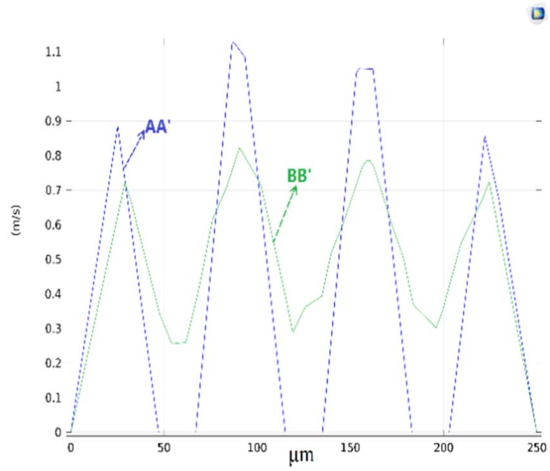


Figure 1: Velocity contour plot and velocity profile along cross sections for µGC columns with CP1, CP2 (a, b) and ECP (c); the posts in CP1 and ECP are spaced 80µm in horizontal orientation and 70µm in vertical orientation; the posts in CP2 are spaced 120µm in horizontal orientation and 70µm in vertical orientation

Detailed fabrication process of the µGC column embedded ECP is illustrated in Figure 2a-d. Firstly, we use LC100A as barrier layer by spin coating process, and thickness of photoresist is 1.7µm. Then, the exposure process is carried out by Karl Suss MA6 for 9 seconds and time of development process is 45 seconds, as shown in Figure 2a. After that, deep reactive-ion etching is used to create serpentine channels with a 250-µm -wide by 300-µm -deep rectangular cross section (Figure 2b), in which each micro elliptic cylindrical post has 30 µm major axis and 10 µm minor axis. Subsequently, a glass wafer is bonded to the silicon wafer, as we can see in Figure 2c. Finally, the wafer is diced and coated with polydimethylsiloxane (OV-1) by static coating procedure [11], as shown in Figure 2d.

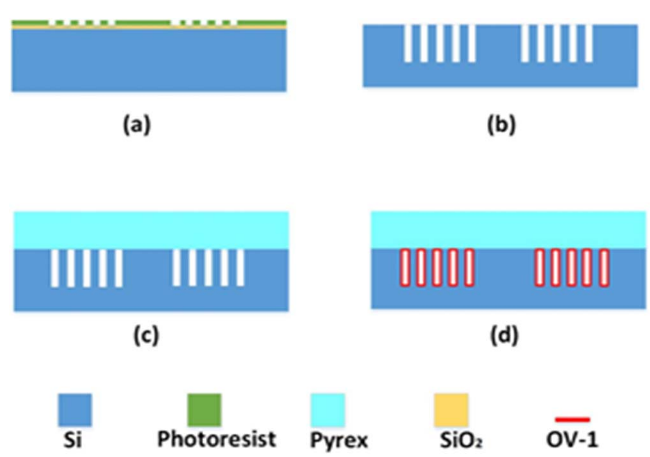
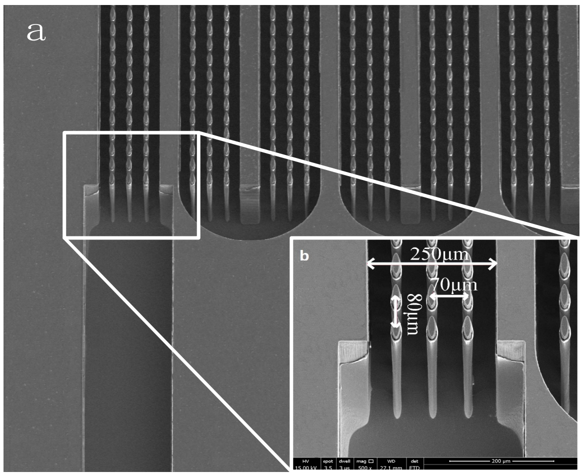


Figure 2: Process flow for the fabricated of µGC column: (a) photolithography ; (b) deep reactive ion etching; (c) anodic bonding; (d) static coating process.

EXPERIMENTAL RESULTS

Figure 3a and 3b shows the structure of μGC columns with ECP, every post is etched into elliptic cylindrical, neatly arranged in the deep channel, each row has three posts and the total amounts of posts are 76494. The picture of μGC column chip with packaged two capillaries is displayed in Figure 3c, the length of μGC column is 2m, the rectangular cross section of inlet and outlet is 360μm width and 370μm depth.



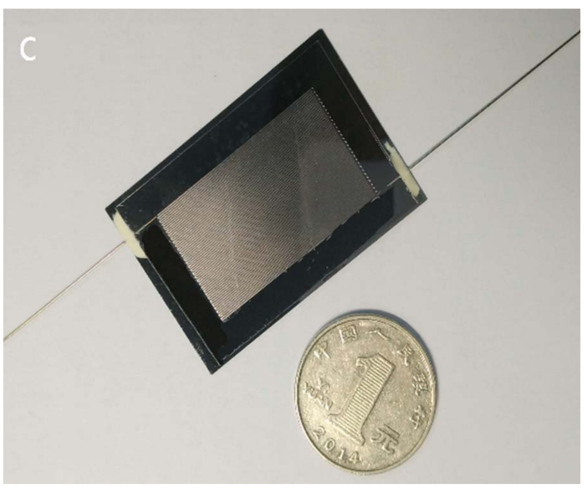
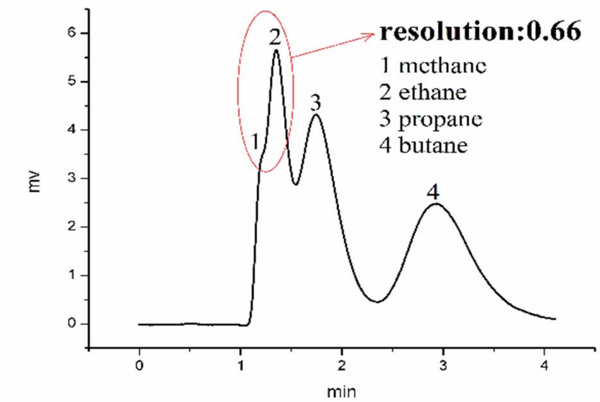
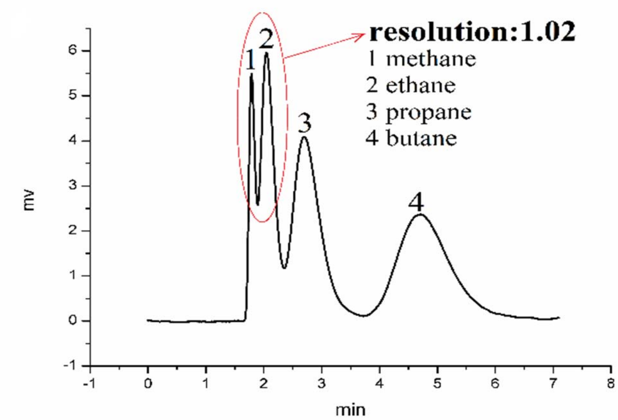


Figure 3: SEM images of (a) μGC column with ECP; (b) the close-up of ECP structure in column's port; (c) photograph of μGC column chip.

In order to evaluate the separation performance of our new μGC column, separation experiments of light alkane (methane, ethane, propane and butane) has been carried out. As demonstrated in Figure 4a, methane and ethane can be identified by using 2m ECP-embedded column with the resolution 0.66. Furthermore, in 4 m columns, the resolution of methane and ethane is 1.02 (ECP-embedded). These results indicate that μGC column with ECP holds good separation performance, which can be rooted from the increased surface area and even stationary phase film. Unfortunately, there is currently no model that can predict the HETP (Height equivalent to a theoretical plate) of semi-packed columns as they have no traditional counterpart [10]. In this new structure, the optimal average carrier gas velocity cannot be obtained by theoretical analysis method. Therefore, we need to do many experiments for finding the optimal average carrier gas velocity. So far we are still trying to analyze and compile the experiment results, in order to seek out relations among the structure parameters, HETP and the optimal average gas velocity about this ECP structure.



(a)



(b)

Figure 4: Separation results of (a) 2 m column with ECP, methane and ethane can be separated, the resolution of methane and ethane is 0.66; (b) 4 m column with ECP, resolution of methane and ethane is 1.02.

CONCLUSION

The work here demonstrates that it is possible to embed ECP in μGC column to separate light alkane. The column embedded with ECP has large surface area and even stationary phase film. Both of these factors can enlarge the interaction zone between mobile and stationary phases, increasing the amount of gaseous analyte in the stationary phase to improve separate resolution.

ACKNOWLEDGEMENTS

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