Surface Micromachined Nanoparticles Separation Devices

C.Shen, H.T.M.Pham, A.T. Nguyen, P.M.Sarro

Abstract— **A novel concept for a particle separation device consisting of microsieves and a transparent holder for fluidic experiments is reported here. In the microsieve design, “walls” with lateral micro-channels are used to replace conventional membrane with pores. The main advantages of this concept are the fabrication of sieves with pores sizes of 100 nm without the need of nanolithography and the use of an IC compatible fabrication process.**

Index Terms— **Particle separators, Surface micromachining**

I. INTRODUCTION

Micromachined devices to separate small particles in liquids are of great interest for purification and separation processes in industrial, medical and pharmaceutical applications. For the diary industry, effectively separating fat, casein, protein and the removal of unwanted bacteria from milk can help increase the yield and quality of their products. For beverage companies, filtration devices offers a low cost option for purifying rough beer by removing yeast cells and other colloidal particles in rough beer [1]. For biological applications, cells or bacteria can be identified from blood sample so diseases can be determined in a quick and accurate way [2].

Different types of particle separators, including cross-linked porous structure with polymer or glass [3-4], micromachined Brownian ratchets [5-8] have been reported. Separators using electronic fields and micromachined microsieves with mechanical separation are currently being employed or investigated. Among them, microsieves turn out to be the most suitable for nano particle separation. They don’t trap particles inside, are not sensitive to external conditions and work on both charged and neutral particles.

For a successful and large scale implementation of these devices an easy to fabricate design should be employed. Most of the microsieves are of the membrane type with thin horizontal membranes and micro pores on top [1]. Patterning pores is the most critical step in the fabrication process. Using advanced lithography equipments like deep ultraviolet (DUV) or Extreme ultraviolet (EUV) scanners to pattern nano sized pores would be too expensive for many MEMS applications. Non-conventional technologies like e-beam [2], laser interference [1] and UV-interference [3] are either slow in fabrication, still expensive or high resolutions cannot be achieved.

II. DESIGN AND FABRICATIOPN OF A SURFACE MICROMACHINED 100NM SEPARATOR

A. Design

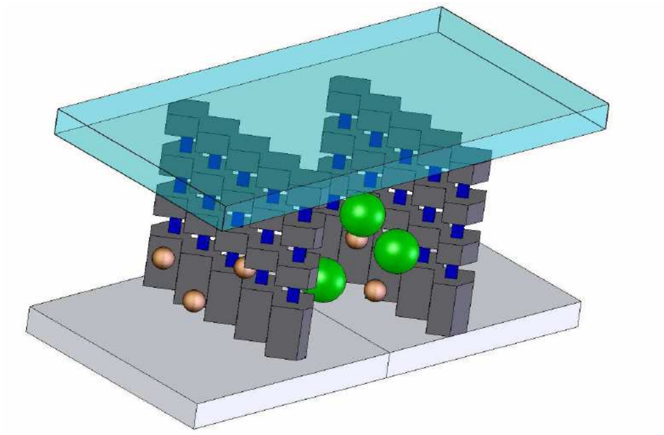

Figure 1: Illustration of a vertical-wall microsieve composed of two vertical walls and sealed with a transparent layer.

In this paper, we present a novel concept of microsieve fabricated with conventional IC and MEMS technologies which addresses two main requirements: definition of nano structures (100 nm) without using advance lithography or non-conventional equipment, and low fabrication complexity. Fig. 1 gives a general illustration of the proposed microsieve.

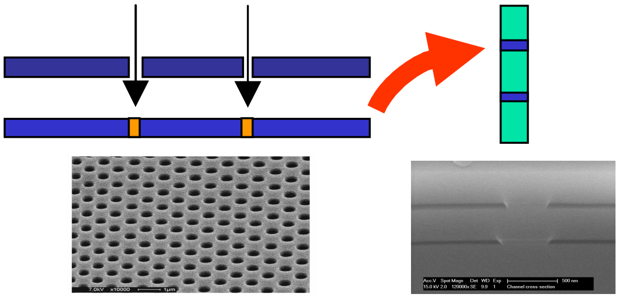

Figure 2: The fabrication of nano pores on a horizontal membrane requires advanced lithography and dry etching. By rotating the horizontal membrane into vertical walls, nano channels can be easily defined by controlling layer thickness and realized by wet etching.

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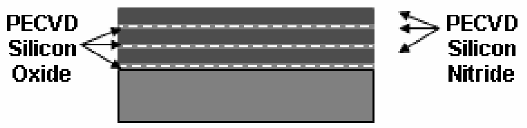
The issue of defining nano structures without advanced lithography is solved by using thin film deposition technology, which can control the film thickness down to several nanometres. When we “rotate” the microsieve membrane into vertical “walls”, the nano pores on membrane become nano channels on the walls. To fabricate 100 nm high channels, we can build a wall with 100 nm thick sacrificial layers inserted in it and then use wet chemical echants to remove those layers (Fig.2).

B. *Fabrication Process*

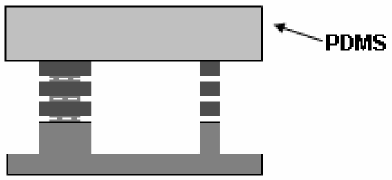
Our vertical microsieve design consists of a 20 µm wide central channel and two 40 µm wide side channels. All channels are 4 mm long and about 20 µm deep. Two sieve walls were used to separate the three channels. For experiment and observation, each channel has its own inlet and outlet port, and the device is sealed by a transparent Poly-dimethyl-siloxane (PDMS) sheet.



a. Stack structure and sacrificial layers

b. Pattern and dry etch to form sieve walls

c. Wet etch to form sieve pores and supporter



d. Sealing microsieve with PDMS

Figure 3: Schematic view of the process flow to fabricate the vertical-wall microsieve

The fabrication process is schematically depicted in Fig.5. The first step is to prepare a sandwich structure that will eventually become the microsieve’s wall. Three 500nm plasma enhanced chemical vapour deposited (PECVD) silicon nitride layers and three 100nm PECVD silicon oxide layers are deposited alternatively on a silicon wafer. The silicon nitride forms the structural layers for the microsieve and the silicon oxide works as sacrificial layers (Fig.3a).

After finishing the sandwich layer, the shape of the sieve walls is patterned and dry etched completely down to the silicon substrate to build the "walls". Part of the silicon substrate is also etched to form a deep trench in order to increase the channel size and thus the flow rate (Fig.3b).

All sacrificial layers are then removed in a single wet chemical etching step using buffered hydro fluoric (BHF) solution. Nanochannels with a precisely controlled height of 100nm are thus formed on the walls. The wall is designed to have a zigzag shape, which make sure that part of the sacrificial oxide will be un-etched and left to support the structure, as Fig.3c shows. A detail of fabricated structure before sealing is shown in Fig.4.

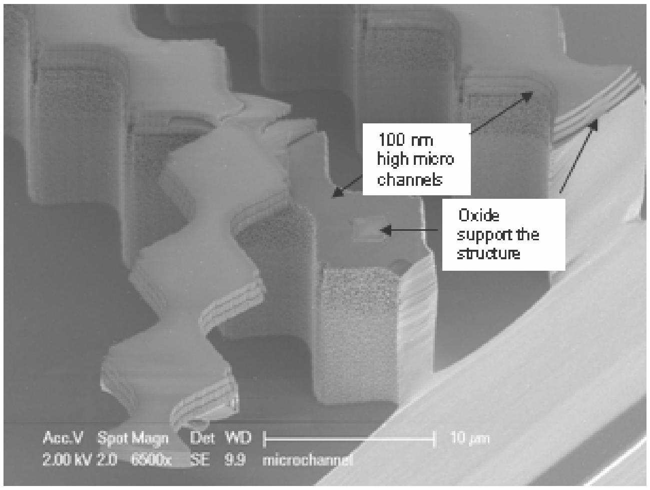


Figure 4: SEM picture of a part of the microsieve before PDMS sealing.

To seal the microsieves a transparent material is preferred to be able to easily observe the particle separation. PDMS sheets about 1 mm thick are used as the cover of the sieve. Dowcorning’s SYLGARD®184 Silicon Elastomer Kit is used here. The base part and curing agent are mixed in a standard 10:1 ratio. Then the mixture is poured on a flat plate and cured at 100ºC on a hot plat for 1 hour. After that, a small piece of PDMS with the same size of the microsieve chip is cut and pressed on the top of the microsieve chip (Fig.3d). Finally both PDMS cover and microsieve chip are treated in oxygen plasma for a good bonding. Before inserting the fluid with particles, the bonded devices are immersed in an ethanol solution for 1 day to reduce surface tension, remove and prevent air trapping in the micro channels, and also inhibit micro-organisms from growing inside the device.

III. A TRANSPARENT HOLDER FOR EXPERIMENT

To carry out experiments on the microsieve chips, a simple holder is designed and fabricated (Fig.5). This holder provides six inlet/outlet ports to connect from the outside to the chip, a transparent observation window and ensures a good sealing. It allows us to perform fluidic measurements on the microsieves.

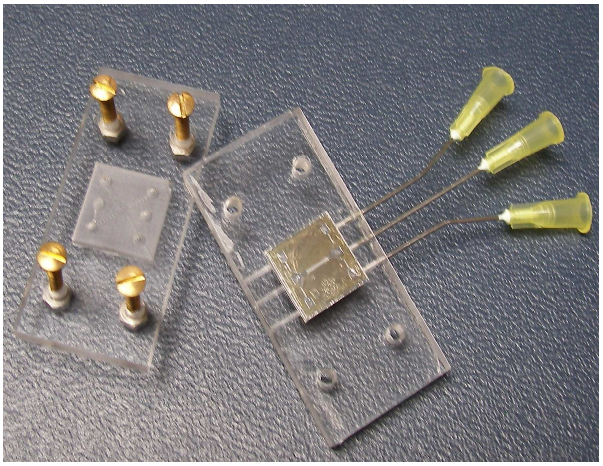


Figure 5: Photo of the transparent holder with a microsieve chip .The holder is composed of two parts and fixed by 4 screws. Three injector nozzles are used to serve as input port.

The main components of the holder are two 600 mm long, 300 mm wide and 5 mm thick Poly methyl methacrylate (PMMA) boards. The top PMMA board works as an interface between the microsieve chip and the outside world, while the bottom board works only as housing for the chip. They are tightened by four screws and bolts with a thin piece of PMMA in between as gasket.

On the center of the top PMMA board, six vertical blind holes, 2 mm in diameter and 3.5 mm in depth, are drilled at the expected location of the microsieve chip's inlets and outlets. Then six laterals through holes, 1mm in diameter, are drilled connecting the blind holes to outside the PMMA board. They are separated into two groups: three inlet channels and three outlet channels (Fig. 6). With these inlet/outlet channels and blind holes, fluid can be injected into the microsieve chip and drained after being sieved (Fig.7). Since PMMA is transparent, the sieving process can be clearly observed through the top board under a microscope.

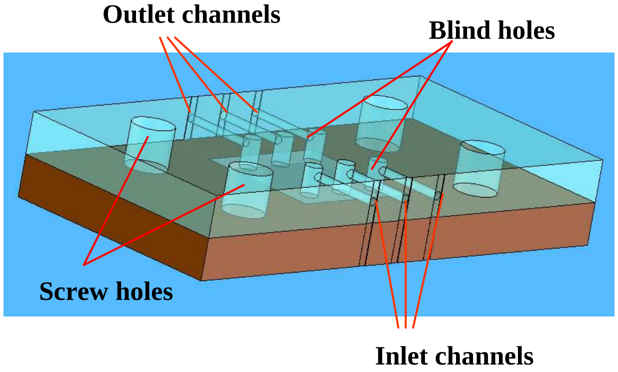


Figure 6: A drawing of the transparent holder.

On the center of the bottom PMMA board, a square cavity, 15 mm wide and 1.5 mm deep, is machined. It is used as housing for the chip.

On both PMMA boards, four vertical through holes with a diameter of 3 mm are drilled. These holes are left for screws and bolts that can tighten both boards together.

To ensure a good sealing after pushing two PMMA boards together, a piece of flat PDMS sheet is placed between the two boards. When they are presses tightly together with screw and bolts, the PDMS sheet will deform and seal the gap between two PMMA boards.

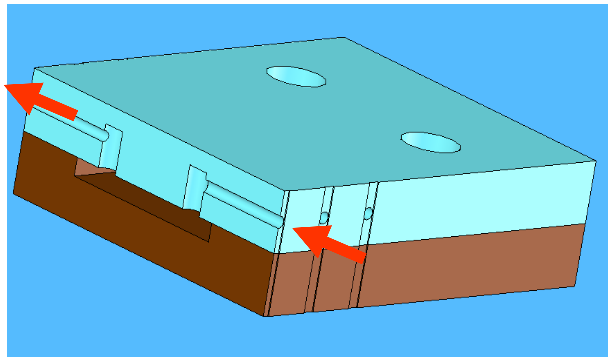


Figure 7: A drawing of the cross section of the transparent holder.

IV. EXPERIMENT RESULTS

In order to test the fabricated and sealed microsieves and verify their filtration capability, fluid mixed with fluorescence particles is injected into the microsieve and observed under a fluorescence microscope.

Due to the difficulty in observing nano particles smaller than 100 nm, water with Rhodamine dye is used to represent particles smaller than 100 nm. Fluorescent polystyrene particle beads with a diameter of 200 nm are used to represent particles larger than 100 nm. For easier observation, 2 µm polyester beads are added as tracers.

When illuminated, both particle beads and Rhodamine dye will give reddish fluorescent light and their location and movement can be clearly followed.

Fig.8 shows the experimental result. The fluids are supplied from the lower-left channel, pass through two microsieves, and then are drained from the upper-right channel. Water with Rhodamine dye can be seen in both left and right channels. This means that water can flow easily through the two 100 nm size microsieves channels. On the other hand, the 200 nm and 2 µm fluorescent beads are only found in the left channel, which means that the 200 nm or larger particles are not able to penetrate the microsieves as water did. This means that particles larger than 100nm have been successfully blocked by the microsieve.

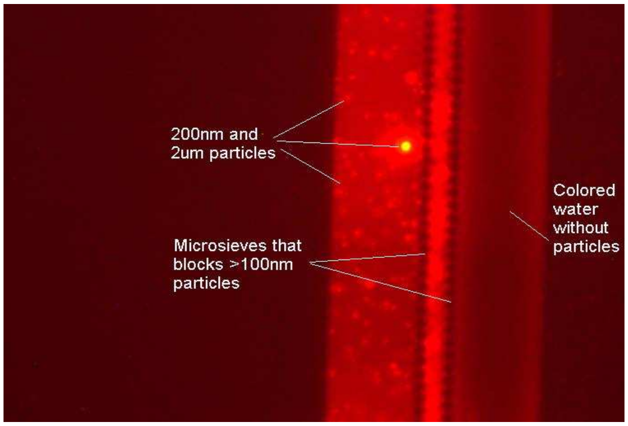


Figure 8: Optical image showing 200nm particles successfully blocked by the microsieve.

V. CONCLUSIONS

To separate nano size particles in fluid, we have designed and fabricated a vertical-wall microsieve with 100 nm high channels. A simple, but functional holder with six inlet/outlet ports and a transparent observation window has been designed and fabricated.

Successful separation of 200 nm particle beads from water has been observed with this microsieve.

In this demonstrator, we choose 100 nm as the height of the channels in the microsieve. But it's possible to change this size while using the same process by only modifying the layers thickness. So it's also possible to make microsieves for even smaller particles.

The fabrication process for this microsieve is simple and the sieve is located on the wafer surface. As the process used is CMOS compatible, this kind of microsieve can be integrated with other devices or electronic functions on the same substrate. This can be very helpful for lab-on-a-chip applications in particle separations.

VI. ACKNOWLEDGEMENT

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REFERENCES

1. S.Kuiper, *Development and Application for Micro sieves*, Ph.D. thesis, Univ.Twente, ISBN 90-36514754
2. http://www.fluxxion.com
3. http://www.spg-techno.co.jp
4. http://www.Millipore.com
5. C.F. Chou et al. *Sorting by diffusion: An asymmetric obstacle course for continuous molecular separation* PNAS Vol. 96(1999), 13762-13765,
6. R. H. Austin et al. *Ratchets: The problems with boundary conditions in insulating fluids* Appl. Phys. A: Mater. Sci. Process, Vol.75 (2002), 279
7. J.W. Dufty et al *Brownian motion in a granular fluid* 2005 New J. Phys. 7 20
8. L.R.Huang et al *Role of Molecular Size in Ratchet Fractionation* Phys. Rev. Lett. 89, 178301 (2002)
9. J. Fu et al *A patterned anisotropic nanofluidic sieving structure for continuous-flow separation of DNA and proteins* Nature Nanotechnology 2(2006), 121 – 128
10. www.ASML.com
11. M.J.Kim, et al *Rapid fabrication of uniformly sized nanopores and nanopore arrays for parallel DNA analysis*, Adv. Mater. 2006, vol. 18, p. 3149
12. Y.K. Yoon, et al *Integrated vertical screen microfilter system using inclined SU-8 structures* Proceeding of MEMS 2003