Fabrication of 10 nm enclosed nanofluidic channels

Han Cao[a)] and Zhaoning Yu
Nanostructure Laboratory, Department of Electrical Engineering, Princeton University, Princeton, New Jersey 08544

Jian Wang
Nanonex Corp., Princeton, New Jersey 08540

Jonas O. Tegenfeldt and Robert H. Austin
Department of Physics, Princeton University, Princeton, New Jersey 08544

Erli Chen
NanoOpto Corp., Somerset, New Jersey 08873

Wei Wu and Stephen Y. Chou
Nanostructure Laboratory, Department of Electrical Engineering, Princeton University, Princeton, New Jersey 08544

(Received 27 March 2002; accepted for publication 30 April 2002)

We made uniform arrays of nanometer scale structures using nanoimprint lithography over large areas (100 mm wafers). The nanofluidic channels were further narrowed and sealed by techniques that are based on nonuniform deposition. The resulting sealed channels have a cross section as small as 10 nm by 50 nm, of great importance for confining biological molecules into ultrasmall spaces. These techniques can be valuable fabrication tools for Nanoelectromechanical Systems and Micro/Nano Total Analysis Systems. [DOI: 10.1063/1.1489102]

In the newly emerging field of bionanotechnology, extremely small nanofluidic structures need to be fabricated and used as matrices for the manipulation and analysis of biomolecules such as DNA and proteins at single molecule resolution.[1–4] While nanostructures allow one to observe single molecules, it is also important to construct many millions of these in parallel. Such large arrays of single biopolymers can reveal information about sample heterogeneity that would otherwise be averaged out in traditional population based assays. In this paper we present simple techniques for fabricating high-density small sealed nanofluidic structures over large areas using nanoimprint lithography (NIL) and techniques to further reduce the dimensions of the prefabricated nanofluidic structures. Millions of enclosed nanofluidic channels with dimensions smaller than 10 nm have been fabricated on a 100 mm wafer.

In creating ultrasmall nanofluidic structures for single biomolecule analysis, we face two challenges: reduction of size and creation of sealed fluidic channels. Although the traditional electron beam lithography (EBL) and more recently developed focused ion beam (FIB) milling techniques have high resolution in generating nanoscale structures, both technologies have the disadvantages of low throughput and being expensive. In contrast, NIL is a parallel high-throughput technique that makes it possible to create nanometer-scale features over large substrate surface areas at low cost.[5,6] Current sealing techniques such as wafer bonding[7] and soft elastomer sealing[8] are suitable for relatively large planar surfaces and provide an effective seal. Wafer bonding requires an absolutely defect free and flat surface, and elastomer sealing suffers from clogging due to soft material intrusion into the channels. Within extremely small confining structures, biological samples are also much more sensitive to issues such as hydrophobicity and the homogeneity of the material constructing the fluidic structure. Recently developed techniques using ‘‘place-holding’’ sacrificial materials such as polysilicon,[9] polynorbornene[10] have gained popularity to create sealed small hollow fluidic structures. However, steps needed in removing the sacrificial materials such as heating the substrate up to 200–400 °C or wet etching might not be compatible with downstream fabrication process or limit use of certain materials.

In our fabrication, high-density arrays of nanofluidic channels were first fabricated using NIL. The NIL mold was generated by interferometric lithography (IL) and has 200 nm period gratings over a 100-mm-diameter wafer. A detailed description of the procedure can be found in the literature.[5,6] The minimum feature size of the nanoscale channels on the mold generated directly by IL is limited by the wavelength of the light used for exposure to about 100 nm. Recently, Yu *et al.*[11] have reported reducing the trench width to 50 nm over a large area using NIL combined with a simple edge defining technique.

After NIL and etching, we used a nonuniform deposition[12,13] to, in a single step, both reduce the cross section of the nanochannels made by NIL and reactive ion etching, and if desired, seal the channels. Two approaches for nonuniform deposition have been explored: (1) *e*-beam evaporation with a tilted sample wafer at various angles, and (2) sputter deposition using a large source target.

E-beam evaporation creates a point source of material. With the sample far away from the source compared to the size of the sample, the angular distribution is very narrow. To achieve a nonuniform deposition the wafer is tilted at a spe-

[a)]Author to whom correspondence should be addressed; electronic mail: han@ee.princeton.edu

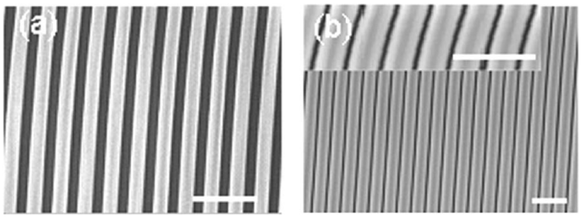

FIG. 1. (a) Top view scanning electron microscopy (SEM) image of nanofluidic channels with a trench width of 85 nm. The dark area is the trench. (b) The channels were narrowed down to less than 20 nm by controlled *e*-beam deposition. The inset shows the channel width at a higher magnification. The scale bars are all 500 nm.

cific angle. The sidewalls of any trench will shadow the evaporation and most of the material will be deposited on the sidewalls at the top of the trench. Beyond a critical depth no deposition will occur. Figure 1 shows a nanochannel grating with original trench width of 85 nm narrowed down to 20 nm by this technique, continuing the deposition further results in the complete sealing of the nanochannels (data not shown). Although the process gives good control over the deposition parameters, it requires multiple depositions with different angles. This method is preferred if a specific geometry of narrowing of the channels is desired.

In sputtering, the second approach, the deposited material impinges onto the wafer at a wide distribution of angles. The surface topology can cause local shadowing effects, leading to non-uniform deposition that can reduce the original size of the channel and seal them off on the top, as shown in Fig. 2. Our sputtering system has a 200-mm-diameter SiO_2 target source chosen to achieve high surface covering uniformity across the device wafer. We experimented with 100–340 nm of SiO_2 deposited onto nanochannels. Effective sealing was achieved with the various deposition conditions we tested. At gas pressure of 30 mTorr, RF power of ~900 W, and DC bias of 1400 V, we have a deposition rate of ~9 nm/min. At lower pressure of 5 mTorr, the sputtered atoms may reach the substrate with less particle collision, the deposition rate increased to an estimated 17 nm/min. All samples shown in this letter were sputtered at 5 mTorr. This method is preferred if a large area needs to be sealed uniformly in a single step.

Figure 3 shows nanochannel structures with different aspect ratios before and after sealing by the sputtering process. Channels with 85 nm trench widths were narrowed down to nearly 55 nm; channels with 65 nm trench widths were narrowed down to less than 17 nm; channels with 55 nm trench widths were narrowed down to less than 10 nm. We were able to seal two-dimensional arrays made using a two-step process with the grating mold rotated 90° between the imprinting steps (Fig. 4). We used the same sputtering process to seal the pillar array structures.

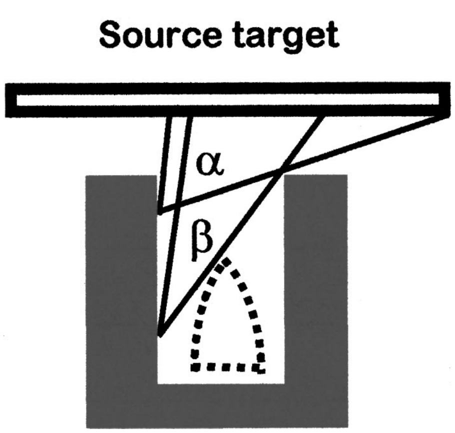

FIG. 2. A schematic illustration of the sputtering deposition process.

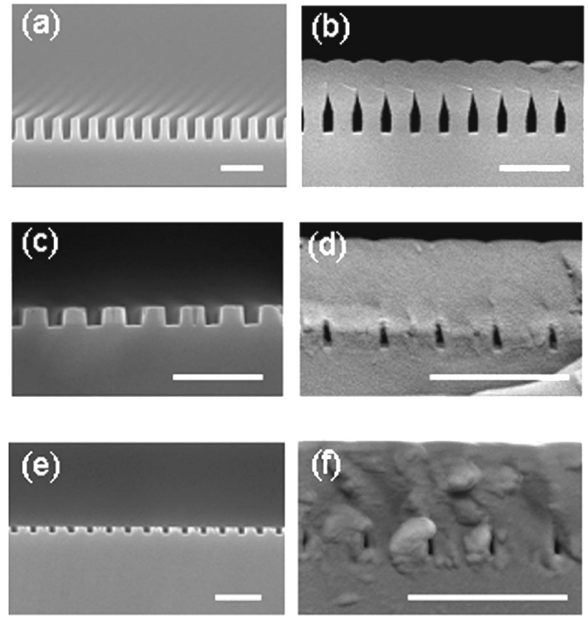

FIG. 3. (a), (b) Nanofluidic channels with trench width of 85 nm were sealed by a single step SiO_2 sputtering, the sealed channel was reduced to nearly 55 nm after the sealing process. The experiment was repeated (c), (d) for 65 nm channels which were reduced to less than 17 nm after sealing. And (e), (f) for 55 nm wide channels which were reduced to less than 10 nm width. The scale bars are all 500 nm.

Figures 4(a)–4(d) shows pillar array structures before, during, and after the sealing process. The transparent nature of the SiO_2 sealing material allows spectroscopic detection of fluorescently labeled biomolecules. This technique also provides an ultrathin capping layer critical for near-field nanofluidic devices.[2,14]

To further confirm the exact size of the nanofluidic channels that we fabricated, FIB milling was used to remove parts of the roof to reveal that the arrays of hollow nanochannels

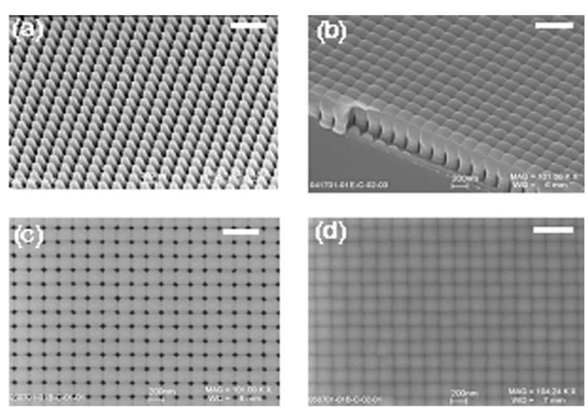

FIG. 4. (a), (b) Nanofluidic pillar array structures before and after sealing by the sputtering process. (c), (d) Top view images of half-sealed pillar array structures with 200 nm of SiO_2 deposited and a completely sealed structure after 400 nm of SiO_2 was deposited. The scale bars are all 500 nm.

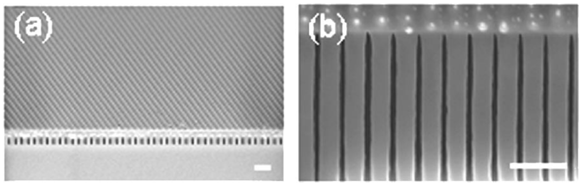

FIG. 5. (a) SEM image shows the "roof" and profile of the nanochannels sealed by the SiO_2 sputtering process. (b) Buried nanochannels under the sealing roof were shown to be perfectly intact after sealing. The scale bars are all 500 nm.

with width as small as 45 nm are intact underneath the deposited SiO_2, noticing that the sidewalls of the channels are smooth (Fig. 5). Fluorescently stained long genomic DNA molecules were effectively stretched in the nanochannels. Figure 6 shows aligned DNA array images acquired by high-definition charge-coupled-device (CCD) videomicroscopy.

In summary, we have demonstrated simple deposition techniques to create roofs over prefabricated structures as well as narrowing of nanofluidic channels with precise control of the location and the thickness of the sealing. Many micro/nanofluidic structures created using currently available fabrication technologies such as nanoimprinting, EBL, FIB milling, or photolithography, could be sealed with this process, and more importantly, this process allows us go beyond what standard patterning technology can do and further minimize existing hollow space in nanostructures making it a valuable processing method in fabrication of future integrated nanoscale devices.

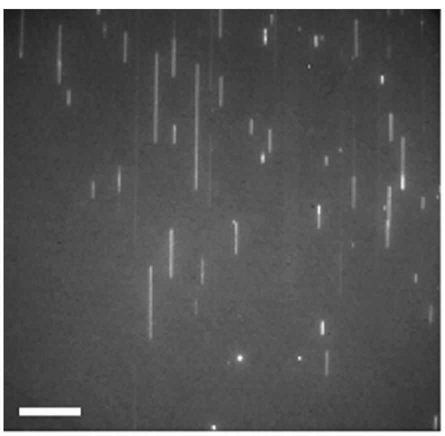

FIG. 6. CCD image of λ-phage DNA concatemers stretched in nanofluidic channels. The scale bar is 30 μm.

This work was supported in part by DARPA, NSF, and NIH. The authors wish to thank Dr. L. Kong and B. Cui for helping with the sputtering system, Dr. Lei Chen for developing the imprinting resist, Dr. H. Ge for mold preparation, and P. Deshpande for many valuable discussions.

[1] O. Bakajin, T. A. J. Duke, J. Tegenfeldt, C. F. Chou, S. S. Chan, R. H. Austin, and E. C. Cox, Anal. Chem. **73**, 6053 (2001).
[2] J. O. Tegenfeldt, O. Bakajin, C.-F. Chou, S. Chan, R. H. Austin, E. Chan, T. Duke, and E. C. Cox, Phys. Rev. Lett. **86**, 1378 (2001).
[3] J. Han and H. Craighead, Science **288**, 1026 (2000).
[4] S. R. Quake and A. Scherer, Science **290**, 1536 (2000).
[5] S. Y. Chou, P. R. Krauss, and P. J. Renstrom, Appl. Phys. Lett. **67**, 3114 (1995).
[6] Stephen Y. Chou, Peter R. Krauss, and Preston J. Renstrom, Science **272**, 85 (1996).
[7] M. Stjernstrom and J. Roeraade, J. Micromech. Microeng. **8**, 33 (1998).
[8] H. P. Chou, C. Spence, A. Scherer, and S. Quake, Proc. Natl. Acad. Sci. U.S.A. **96**, 11 (1999).
[9] S. W. Turner, A. M. Perez, A. Lopez, and H. G. Craighead, J. Vac. Sci. Technol. B **16**, 3835 (1998).
[10] D. Bhusari, H. A. Reed, M. Wedlake, A. M. Padovani, S. A. B. Allen, and P. A. Kohl, J. Microelectromech. Syst. **10**, 400 (2001).
[11] Z. N. Yu, W. Wu, L. Chen, and S. Y. Chou, J. Vac. Sci. Technol. B **19**, 2816 (2001).
[12] L. F. Johnson, K. A. Ingersoll, and G. W. Kammlott, Appl. Phys. Lett. **34**, 578 (1979).
[13] N. Tsumita, J. Melngailis, A. M. Hawryluk, and Henry I. Smith, J. Vac. Sci. Technol. **19**, 1211 (1981).
[14] Z. N. Yu, P. Deshpande, W. Wu, J. Wang, and S. Y. Chou, Appl. Phys. Lett. **77**, 927 (2000).