Microelectronic Engineering 97 (2012) 173–176

Contents lists available at SciVerse ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Fabrication of bottle-shaped nanochannels in fused silica using a self-closing effect

Mariusz Graczyk, Martina Balaz, Heiner Linke, Ivan Maximov *

Division of Solid State Physics/Nanometer Structure Consortium, Lund University, Box 118, S-211 00 Lund, Sweden

ARTICLE INFO

Article history:
Available online 8 February 2012

Keywords:
Nanochannels
Electron beam lithography
Oxidation
Fused silica

ABSTRACT

The spatial control of molecular motor function, using nanostructured surfaces, is of great interest for the development of commercial devices for diagnostics and high-throughput drug screening with molecular motors as targets. In the present study we have fabricated 100–300 nm wide nanochannels, completely subsurfaced on fused silica chips, with the aim to interface them with a microfluidic system. Such a system will allow for changes in the chemical environment surrounding molecular motors, with minimal influence on their directional motion. This will be achieved by changing the chemical environment in a perpendicular direction to the motor motion and allowing the chemical substances to diffuse in and out of the nanochannels via a small slit (5–10 nm) on the top of the nanochannels. To create this slit, and to control its width, we here demonstrate the use of a self-closing effect based on the volume increase (2.27 times) during oxidation of silicon. The details of the fabrication steps (EBL, RIE and oxidation) are discussed.

1. Introduction

Molecular motors are nano-scale biological machines that are responsible for most forms of motion in living cells. Molecular motor proteins, such as kinesin and myosin, participate in many different intracellular cargo transport systems, and they therefore also play significant roles in the pathogenesis of a variety of diseases [1]. Thus, it is of great interest to develop chip-based devices for diagnostics [2] and high throughput drug screening that target molecular motors [3].

Most chip-based devices for molecular motors use the geometry of the *in vitro* motility gliding assay, where filaments, such as actin or microtubules, are propelled by motors attached to a surface. One of the main challenges in developing chip-based devices for such assays lies in the ability to obtain spatial control, and to guide the filaments along predetermined paths. Efforts in the field during the last 15 years have produced several approaches to guiding the filaments, including (i) spatially selective chemistry, (ii) mechanical guiding by nanochannels, and (iii) combinations of chemical and mechanical approaches [4].

Generally, substrates of silicon [5] or glass [4] have been used to fabricate tracks for molecular motors. There are several advantages of fabricating nanochannels on glass or fused silica, compared to Si: (i) because of its excellent transparency and low background emission, fused silica has the advantage of giving a high signal to noise ratio during fluorescence microscopy observations. (ii) Use of a transparent surface for nanochannel fabrication enables imaging of samples from the bottom while a microfluidic system may be integrated on top of the device. The main advantage of integrating a microfluidic system on a chip-based device is that it allows for fast switching of the chemical environment surrounding the motors. For example, this could enable the testing of many different drug candidates that target molecular motors on the same device during a short time period.

Our goal is to fabricate a device with nanochannels on fused silica chips that can be integrated with a microfluidic system to allow for real time changes in the chemical environment surrounding the molecular motors, with minimal influence on their directional motion. This will be achieved by allowing the chemical substances to diffuse in and out of the nanochannels via a very narrow slit (5–10 nm) on the top of the channels. Due to the shape and surface chemistry of our silica nanochannels, it will likely be possible for the molecular motors to be adsorbed all around the channel surface. Thus, it is important that we design the slit on top of the nanochannels to be narrow enough to prevent actin filaments ($\varnothing \sim 9$ nm) [6] or microtubules ($\varnothing \sim 25$ nm) [7] from being propelled out of the channels.

In the present work have fabricated 100–300 nm wide nanochannels on fused silica chips. These nanochannels differ from previous designs as they are completely subsurfaced on fused silica and have narrow slits on top.

2. Experimental details

Fig. 1 shows the details of the fabrication process. $1'' \times 1''$ fused silica substrates with a thickness of 500 μm were used as a starting material. Prior to thermal evaporation of 30–50 nm thick Si onto

---

* Corresponding author.
E-mail address: ivan.maximov@ftf.lth.se (I. Maximov).

0167-9317/$ - see front matter © 2012 Elsevier B.V. All rights reserved.
doi:10.1016/j.mee.2012.01.010

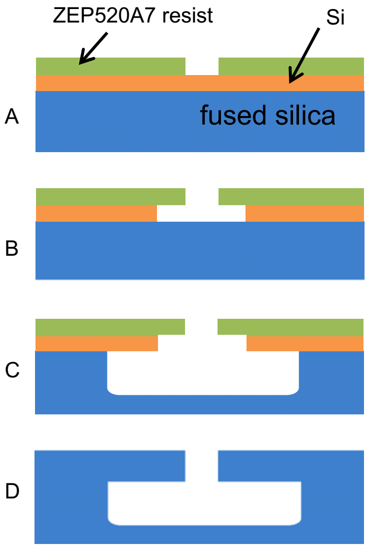

Fig. 1. Schematic illustration of the fabrication process: (A) evaporated Si layer with EBL-exposed and developed resist. (B) Selective RIE of Si layer. (C) Wet etching of fused silica. (D) Oxidation of the Si layer to SiO_2 to form the final nanochannel.

the surfaces, the fused silica substrates were cleaned using RCA1 and RCA2 cleaning procedures. The evaporation (Pfeiffer Classic 500L), was started at a basic pressure below 5×10^{-8} mbar and the chamber pressure was kept below 5×10^{-7} mbar during evaporation, to avoid oxidation of the Si layer. Phosphorus doped silicon with the resistivity of 0.01 Ω cm was used for the evaporation. The Si/fused silica wafers were then spin-coated at 6000 rpm for 60 s with a positive electron beam resist, ZEP 520A7, diluted in anisole. After spin-coating, the samples were baked on a hot plate at 180 °C for 5 min. The resist thickness was evaluated by ellipsometric measurements and gave a thickness of approximately 40 nm. Nanochannels were defined by EBL (Raith 150, Raith GmbH) using an acceleration voltage of 20 kV and current of 20 pA. The widths of the exposed single pixel lines were adjusted by varying the dose from 150 to 360 μC/cm. Following EBL exposure, the resist was developed in hexyl acetate for 45 s at 0 °C and rinsed in MIBK:IPA (1:3) for 30 s [8]. Prior to reactive ion etching (RIE) of Si, a short descum process of the resist residues was performed in oxygen plasma in the same RIE system (Sirus T2, Trion Technology, Inc.) at the RF-power of 30 W and a pressure of 50 mTorr. Alternatively, the sample was ashed in oxygen plasma asher (Plasma Preem, Plasmatic Systems Inc.) at 5 mbar for 10–20 s with a Faraday cage. The RIE in SF_6 or $SF_6/CHF_3/O_2$ [9] mixture of Si was used to pattern the Si mask with subsequent isotropic wet etching of silica by a buffered HF-solution (BOE). RIE was performed for 8–15 s at a pressure of 150 mTorr. The applied RF power was 75 W and the basic flow of SF_6 was 15 sccm. The etch rate in BOE was determined to be 50 nm/min and the etch time was varied based on desired size of the nanocavities. During etching of the fused silica substrate, both Si and resist were used as an etch mask. As a last step before the Si oxidation, the resist was stripped by oxygen ashing (5 mbar, 2 min) and the resulting structure was again cleaned using standard RCA1 and RCA2 processes. The final oxidation of Si was performed in furnace in a dry oxygen atmosphere at a temperature of 1100 °C for 30–60 min. The conversion of the Si layer into SiO_2 results in an increase of its volume, which leads to shrinkage of the slit on top of the nanochannel ("the self-closing effect"). The intermediate and final structures of the nanochannels were imaged using scanning electron microscopy (SEM LEO 1560, Carl Zeiss AG). The SEM inspection of the fused silica samples were generally performed after sputtering of 10 nm Pt.

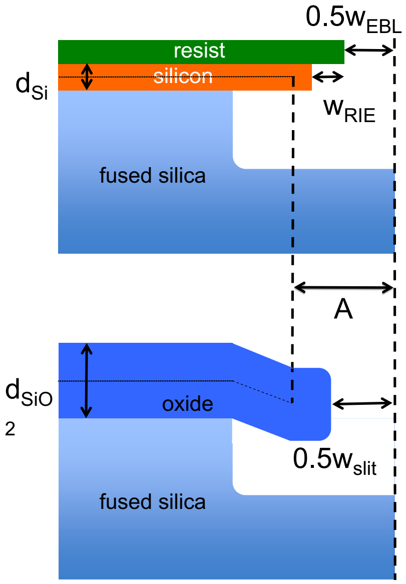

Fig. 2. A simplified model of the "self-closing effect". The model is based on the volume increase of oxidized Si by a factor 2.27 compared to pure Si. The increase of volume after oxidation of the evaporated Si layer leads to lateral extension of the SiO_2 layer to decrease the nanochannel slit width on top. For model details, see the main text.

3. Results and discussion

The proposed method of using a self-closing effect to fabricate nanochannels with a controllable slit width on top is based on the volume difference between the initial Si layer and its oxide (Fig. 2). Assuming that distance A between the centre of nanochannel and the centre of convex (invariable point) [10] in the Si or SiO_2 layer is constant, the resulting slit width may be described by Eq. (1):

$$W_{slit} = W_{EBL} + 2\,W_{RIE} - 1.27\,d_{Si} \tag{1}$$

where, W_{EBL} is the width of the EBL defined channel after resist development, W_{RIE} is an additional widening of the channel after patterning of the Si layer by the RIE process, d_{Si} and d_{SiO_2} are the respective thicknesses of the Si and the final SiO_2 layer, and W_{slit} is the final width of the slit after conversion of Si to SiO_2. Eq. (1) shows a simplified relationship between the width of the EBL-defined line, the lateral widening and thickness of the Si mask. For example, for 20 nm wide EBL defined lines and 20 nm lateral widening of the 30 nm thick Si mask, the calculated slit width will be 2 nm only. By carefully choosing the geometrical parameters men-

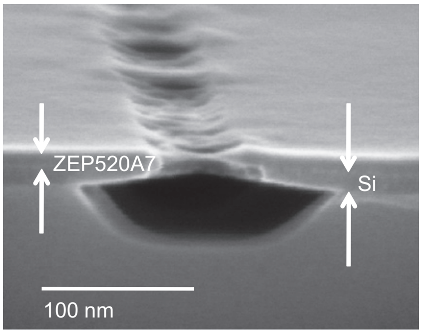

Fig. 3. SEM image of a $Si/SiO_2/Si$ test structure with layers of ZEP520A7 resist and Si mask after BOE etching. The image shows the mechanical stability of the structure.