SOI Processing of a Ring Electrokinetic Chaotic Micromixer

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ABSTRACT

Micromixing has been an active research area in the past decade due to the rapid expanding application of the lab-on-a-chip system in life science. Using the silicon bulk micromachining technology, a ring electroosmotic micromixer, which uses a novel arrangement of electrodes and flow obstacles to induce chaotic mixing, has been designed and fabricated. The design idea uses heavily doped silicon as the fabrication material, and SOI wafer to ensure the electrical isolation of the device from the substrate. The isolation between the electrode and the non-conducting structures is achieved by thermally growing SiO_2 to fill the gaps between the two areas. The SOI processing and the later Focused Ion Beam (FIB) modification method produces integrated electrodes to the full depth of the microfluidic system. Other components of the lab-on-a-chip system can be fabricated following the same process flow. A 25-micron wide, 50-micron deep mixer has been fabricated and assembled for later testing.

Keywords: Silicon bulk micromachining, SOI, FIB, chaotic mixer, electroosmotic flow

1 INTRODUCTION

The technology of micro total analysis systems, or "lab-on-a-chip", has achieved a rapid development in the past ten years due to its wide application in life science and chemistry, such as DNA hybridization, PCR diagnostics, and drug discovery [1-4]. As an important component of the lab-on-a-chip system, micromixing has been given significant attention since most of the reagents need to be mixed with other reagents before the final analysis stage.

Most of the micromixers in the literature can be categorized as either passive mixers that use geometrical stirring [5-8], or active mixers that use movable parts or external forces such as pressure or electrical field [9-14] to achieve mixing. Current passive mixers still require mixing channels of considerable length, and the complicated asymmetric structure makes CFD simulation a challenging task. Active mixing is another approach for rapid mixing with either pressure disturbance [9], electrokinetic [11], magnetic [12] actuation or acoustic vibration [13].

The ring electroosmotic micromixer, which we present here, is a new type of electrokinetic micromixer. Four symmetrically located microelectrodes integrated at the wall of the central circular loop are used to apply a sinusoidal or other waveform electrical field to the fluid, thus inducing an oscillating electromosmotic flow. A two-dimensional FEMLAB simulation is performed to illustrate the exponential separation of the two neighboring particles , providing a proof of the chaotic nature of mixing.

2 PHYSICAL MODEL & DEVICE DESIGNING

The basic idea is to use electroosmotic force, which functions efficiently near a surface in a ring chamber, to induce chaotic mixing. Necessary conditions for chaotic motion require that (a) the system has three independent dynamic variables, and (b) the equations of motion contain a nonlinear term that couples several of the variables [15]. Although the Stokes equation for low Reynolds number flow is linear, the governing equation for the motion of the particle trajectories is nonlinear. Chaotic mixing can be achieved in laminar flows [16, 17]. Chaotic mixing in microchannels has been studied in a number of papers (Refer to the special issue of Proc. Roy. Soc. on microscale transport), such as using a 3-D serpentine pipe [5], side-channel pressure disturbance [9] [14], and ridged-floor with staggered herringbone stirring [7].

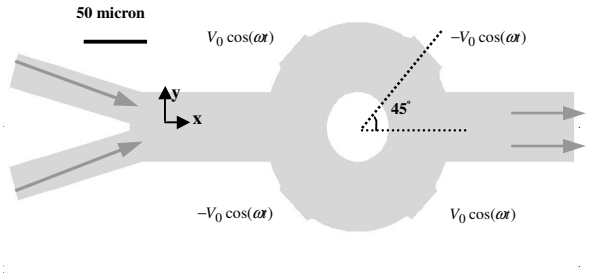

Fig. 1: Schematic of the new mixer with four symmetric electrodes equally distributed at the wall of the mixing chamber. Time-dependent electrical signals are given as shown in the picture.

A similar design for a pressure-driven micromixer appeared in [18]. However, the reported mixing rate was algebraic, compared to the exponential mixing rates achieved here. As shown in Fig. 1, two different fluid solutions from two individual reservoirs flow parallel to

each other, then merge into one channel under a steady pressure gradient, and then enter the mixing chamber. Inside the mixing chamber, four electrodes are symmetrically positioned at the wall of the circular loop, so that a time-periodic electrical force can be applied transversally to the main stream. The middle circular island is designed to limit the transverse mixing length as the inlet mainstream transverse length.

Once the reagents enter the mixing chamber, the electrical double layer (EDL) or Debye layer is formed within a few nanometers from the SiO_2 surface, due to the spontaneous separation of charge generated at a liquid/solid interface. Net charge inside this EDL will migrate due to the Coulomb force. This leads to a flow profile that is a plug flow away from the Debye Layer. Voltage is applied as shown in Fig. 1, causing the complex instantaneous streamline pattern as shown in Fig. 2. The voltage is oscillated in time to provide breaks of separating surfaces in the streamline path. Continuing cycling of the voltage, flow at the rear of the mixer is separated into smaller fluid segments, thereby mixing the fluids.

3 NUMERICAL SIMULATION

The physical intuition, which led to the design of this device, has been verified by numerical simulation using the multiphysics modeling software FEMLAB (Comsol Inc.). The quasi-static electrical field is solved by the conductive media DC model with time-varying electrical potential at the 6-micron concave electrodes surface. The incompressible Navier-Stokes model is probed to determine the fluid velocity field. The two models are coupled together with slip boundary conditions [19] at the non-electrode area of the inner channel wall

$$u_{slip} = -\frac{\varepsilon \zeta E x}{\mu}, \qquad v_{slip} = -\frac{\varepsilon \zeta E_y}{\mu}$$

where ε is the permittivity of the liquid medium, μ is the dynamic viscosity, ζ is the local zeta potential with a typical value of -0.1 V, E_x and E_y are the x, y components of the electrical field independently.

Snapshots of the electrical field and the fluid streamlines are given in Fig. 2 with maximum electrical potential on the electrodes surface of 1Volt. The medium fluid is water with conductivity 0.11845 S/m and relative permittivity 80.2. The inlet velocity is given as 0.1mm/s, and the mainstream channel width is 50 microns. Fig. 2 illustrates that the flow splits into domains with eddy rotation separated by separating streamlines. The time-dependent actuation causes the separatrix breakup and the associated chaotic advection. More detailed simulation analysis can be found in [20].

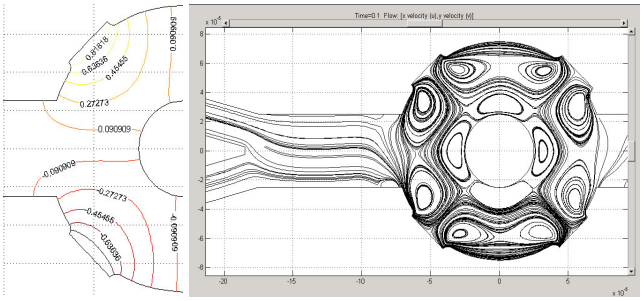

Fig. 2: Electrical potential contours (left) and fluid streamlines (right) distribution by 2-D FEMLAB simulation at time equals to 0.1s with v_0 1V and frequency 10Hz.

4 FABRICATION

RECM is a test device for micro/nano scale computational infrastructure. To efficiently compare 2-D simulation with experiments, it is required that the aspect ratio (channel depth over channel width) of the experimental device be as large as possible in order to minimize the floor effect on the flow profiles.

4.1 Principle

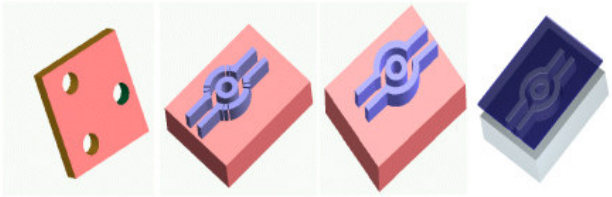

Fig. 3: Main processing steps for device fabrication. In the processing, three reservoirs are etched-through after the first layer photolithography. Then the mixer and electrode networks are patterned and etched as the second layer.

Usually, metal sputtering or evaporating is used to deposit metal on the silicon chip as an electrode. But because of the difficulty in sidewall deposition (50 micron deep for our mixer) using current surface-based microfabrication technology, this method does not provide an electrode with a sufficiently high aspect ratio. To solve this problem, we use heavily boron-doped silicon with resistivity of 0.02ohm·cm (Ultrasil Corp.) as the fabrication material, and SOI (Silicon On Insulator) wafer to obtain isolation between the device and the substrate bulk material. The isolation between the electrode and the non-conducting structures is achieved by thermally growing SiO_2 to fill the gaps between the two areas. The 3 micron wide, 50 micron deep gaps are formed after the second photolithography.

4.2 SOI Processing

A 25-micron wide, 50-micron deep mixer (see Fig. 4) has been fabricated at our new MEMS/NEMS cleanroom and UCSB's nanotech research cleanroom. We also designed another two sizes (1 micron and 50 micron chamber size) to investigate length-scale dependence. Two photolithography steps are required to fabricate the mixer. One mask is used to create the fluid reservoirs and the second one is for patterning the mixer.

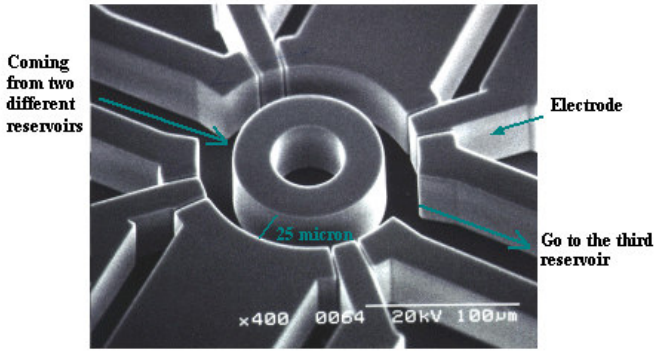

Fig. 4: SEM picture of the ring mixer (25micron wide, 50 micron deep) at the step 8 (before FIB characterization).

First, a 7.5-micron thick photoresist is spun (SPR220-7.0 spin at 3000 rpm for 30 seconds) onto the 4-inch SOI wafer (Step 1). This step is followed by photolithography patterning three reservoirs on the wafer by a GCA 6300 i-line wafer stepper (Step 2). Using a Bosch etcher (Plasma-Therm 770 SLR), the wafer is etched down 50 microns to expose the buried one-micron SiO2 layer (Step 3).

Then the buried SiO_2 layer in the reservoir area is removed. Li. et al [21] reported a 7 min dipping in saturated HF (49%) to remove the buried oxide. We developed a safer technology. After 15 seconds plasma O_2 cleaning, the wafer is immersed in improved buffer HF for 10 minutes, and then the procedure is repeated. RIE etching (O2 and CHF3) follows to totally strip the buried SiO2 layer inside the reservoir area (Step 4). Next, DRIE is performed to etch through the reservoirs (Step 5). By this step the first layer is done.

For the second layer, we use AZ-P4110 positive photoresist (spin at 4000 rpm for 30s, 1.1 micron in thickness). The pattern of the mixer (including mixer chamber, channels, integrated electrodes and contact pad) is defined (Step 6) and etched with DRIE down to 50 microns (Step 7). After cleaning (Nanostrip at 60 $^{\circ}C$ for 20 minutes), the wafer is loaded into the furnace (Tystar 8" oxidation furnace) to grow a 6-micron thick SiO2 layer (wet oxidation, 1050 $^{\circ}C$ for 96 hours) to enclose the gap between the electrode and non-electrodes area (Step 8). Although the gap is only 3 micron wide, since every 1micron growth of SiO2 consumes 0.44 micron silicon, we need to grow 6 microns of SiO2 to close the gap. RIE (Panasonic E640 etcher), at the etch rate of 2 microns per 10 minutes, is used to remove the surface oxides so that the silicon core of the contact pad is exposed for later probe testing (Step 9).

4.3 FIB Characterization

The FEI DB235 Dual-Beam Focus Ion Beam System is used for image and ion milling.

One measurement is to check the cross sectioning of the silicon-silicon dioxide isolation segment. The left image (Fig. 5) shows that the two silicon dioxide interfaces merge and close the gap after milling away half of the mixing chamber wall at the interface area.

Ion milling is used to modify the electrode surfaces. The design of the RECM requires that the electrodes must contact the fluid, and the remaining parts of the mixer are thermal oxide to achieve a higher slip velocity. We used a FIB with an ion source of $20\,nA$ for 3 minutes to selectively remove silicon dioxide from the electrode surfaces in contact with the fluid (Step 10), as shown in the right image of Fig. 5. Step 10 can be substituted by using a third mask to etch away SiO_2 on the electrodes sidewall.

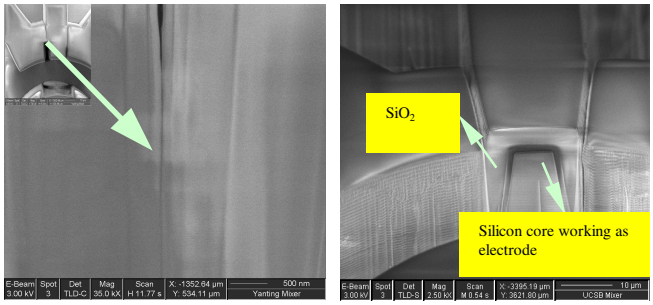

Fig. 5: Left: FIB characterization of the sidewall sealing. Right: FIB milled away 6-micron inner-sidewall silicon dioxide to expose the electrode to fluid.

4.4 Anodic Bonding

A 6 mm square, 170 micron thick Pyrex 7740 (Precision Glass & Optics) is anodically bonded to the chip by a self-made bonding apparatus in the air at 385°C (silicon and glass interface temperature) and –650V for 10 minutes (Step 11). Since our cover slip is quite thin, contrary to the widely believed 1000V voltage, our data shows that as low as 600V enables bonding. A high voltage can easily generate an electrical arc in air and thus damage the sample. Temperature is another key parameter. Bonding a SOI wafer requires a higher temperature than that used to bond a silicon wafer to the Pyrex glass slip when other parameters are kept the same.

5 MIXER TESTING SET-UP

A schematic diagram of the mixer testing is shown in Fig. 6. It is composed of three main parts: fluid supply

system, electrical field supply system and video capture system. Three-micron diameter fluorescent polymer microspheres (Duke Scientific Corp.) are used for micro PIV analysis. Testing results will be presented in future publications.

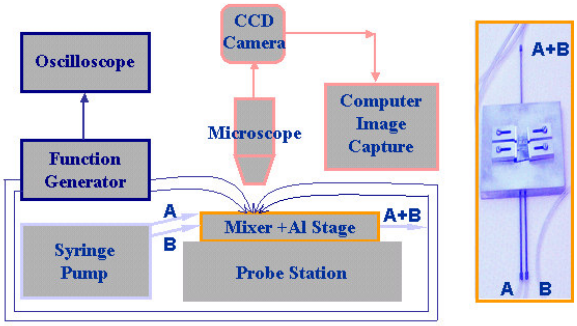

Fig. 6: Left: Schematic picture of the testing setup Right: Mixer on the Aluminum Stage with connecting tubings.

6 CONCLUSIONS

We designed and fabricated a new ring electrokinetic chaotic mixer (RECM) with 3-D integrated electrodes on a heavily doped single crystal silicon wafer. Simulation results indicate that chaotic mixing is possible with this design. The SOI processing and later FIB characterization provides high aspect ratio electrodes that can be fabricated to the full depth of the microfluidic system. Other components of the lab-on-a-chip system can be fabricated following the same process flow.

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