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DESIGN AND FABRICATION OF A RING ELECTROKINETIC CHAOTIC MICROMIXER (RECM) WITH INTEGRATED ELECTRODES

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ABSTRACT

Micromixing has been an active research area in the past decade due to the rapid expanding applications of lab-on-a-chip system in life science and chemistry. Using the silicon bulk micromachining technology, a ring electroosmotic-driven micromixer which uses a novel arrangement of electrodes and flow obstacles to induce chaotic mixing has been designed and fabricated. The design idea is to use heavily doped silicon as the fabrication material, and SOI wafer to promise the good isolation of the device from the substrate. The isolation between the electrode and the non-conducting structures is achieved by thermally growing SiO₂ to fill the gaps between the two areas. This method could fabricate integrated electrodes to the full depth of the microfluidic system, thus offers high-aspect ratio channels that minimize the floor effects on flow profiles. And the whole process is compatible with conventional IC fabrication process, in addition, other components of the lab-on-a-chip system also can be fabricated following the same process flow, thus this fabrication process is highly integrative. A 25 micron wide, 50 micron high mixer has been fabricated and assembled to later testing. Two-dimensional FEMLAB simulation illustrates the exponentially separation of the two neighboring particles---a proof of the chaotic mixing. And a 50 micron wide, 50 micron high mixer is ready for the length scale effects study.

INTRODUCTION

The technology of micro total analysis systems or "lab-on-a-chip" has viewed a rapid development in the past ten years due to its wide applications in life science and chemistry, such as DNA hybridization, PCR diagnostics, and drug discovery [1-4]. Thus micromixer, an important component of the lab-on-chip system, also drew a lot of researchers' attention since most of the reagents needs to be mixed with other reagents before the final analyses stage. The uniqueness of micromixing lies in its low Reynolds number flow nature. Since the rapid mixing

produced by the turbulence flow is usually not achievable at the microscale, mixing is mainly dependent on diffusion. Some devices with deep, narrow geometries are designed to promote mixing by incorporating the diffusive scale into their design [5]. However, the diffusion coefficient of the macromolecules is much lower than the most of the fluids, which increases the difficulty of rapid homogenous mixing. That is to say flows with low Reynolds number but high Peclet generally mix poor.

Most of the micromixers in the literature can be categorized into passive mixers which use geometrical stirring [6-10] and active mixers which either has movable parts or external forces such as pressure and electrical field [11-16]. Current passive mixers still need mixing channels of considerable length, and the complicated asymmetric structure make CFD simulation a challenging work. Active mixer is another approach for rapid mixing with either pressure disturbance [11], electrokinetic [13], magnetic [14] actuation or acoustic vibration [15].

The ring electroosmotically-driven micromixer, which we presented here, is a new type of electrokinetic micromixer. With four symmetrically located microelectrodes integrated at the wall of the central circular loop, a sinusoidal or other waveform electrical field is applied to the fluid. Electrokinetic instability is achieved through oscillating electroosmotic flow at the chamber boundary. Numerical simulations show that particle paths have sensitive dependence on initial conditions.

PHYSICAL MODEL & DEVICE DESIGNING

Our 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 motions include (a) the system has three independent dynamic variables, and (b) the equations of motion contain a nonlinear term that couples several of the variables [17]. Although the Stokes equation for the low Reynolds number flow is linear, the governing equation for the motion of the particle trajectories is

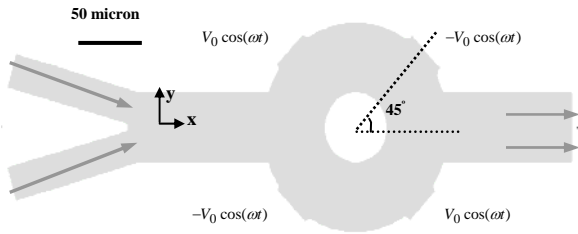


Fig.1 Schematic showing the new mixer with four symmetrical electrodes equally distributed at the wall of the mixing chamber (each electrode recesses into the wall about 4 microns due to the fabrication process which will be explained later)

not linear in general. Ottino demonstrated that chaotic mixing can be achieved in laminar flows [18]. The chaotic mixing in microchannels has been studied in a number of papers, such as a 3-D serpentine pipe [6], side-channels pressure disturbance [11] [16], and ridged-floor with staggered herringbone stirring [9].

The ring design that we present is similar to the one presented by Quake [19] for pressure driven flows. The difference is that in our mixer the motion of fluid particles is chaotic. Two different fluid solutions from two individual reservoirs flow parallel to each other in one channel under a steady pressure gradient, then go to the mixing chamber. Inside the mixing chamber, four electrodes are symmetrically positioned at the wall of the circular loop as shown in Fig.1, so that 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. Our mixer is fabricated based on silicon bulk micromachining technology. Heavily doped silicon is used as electrode material, and non-electrode area is covered with thermally growth silicon dioxide. Once the reagents enters the mixing chamber, the electrical double layer (EDL) or Debye layer is formed within a few nanometers from the SiO₂ surface due to the spontaneous separation of charge generated at a liquid/solid interface. Net charge inside this EDL will migrate due to the action of the Coulomb force $F=qE$. This leads to a flow profile that is a plug flow away from the Debye Layer. Voltage is applied as Fig.1 illustrates, causing a complex instantaneous streamline pattern as shown in Fig.2. Voltage is oscillated on time to provide for breaks of separating surfaces in the streamline path. Continuing this cycling of the voltage, flow at the rear of the mixer is to be separated into smaller fluid segments---mixing occurs.

NUMERICAL SIMULATION

Our physical intuition is verified by numerical simulation with a multi-physics software FEMLAB (Comsol company). The conductive DC media model with time-varying electrical potential at the 4-micron concave electrodes surface is used to solve the electrical field, and the incompressible Navier-Stokes model with slip boundary at the mixing chamber non-electrode area provides us the information of velocity field of the fluids. They are coupled together by the slip boundary condition [30] in the N-S model

$$u_{slip} = \frac{\varepsilon \zeta E_x}{\mu}, \quad v_{slip} = -\frac{\varepsilon \zeta E_y}{\mu}$$

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 0.1V, E_x and E_y are the x, y components of the electrical field independently.

A snapshot of the electrical field and the fluid streamlines is given at Fig.2 with maximum electrical potential on the electrodes surface 1V. The medium fluid is water with conductivity 0.11845 S/m and relative permittivity 80.2. Inlet velocity is given as 0.1mm/s, and 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.

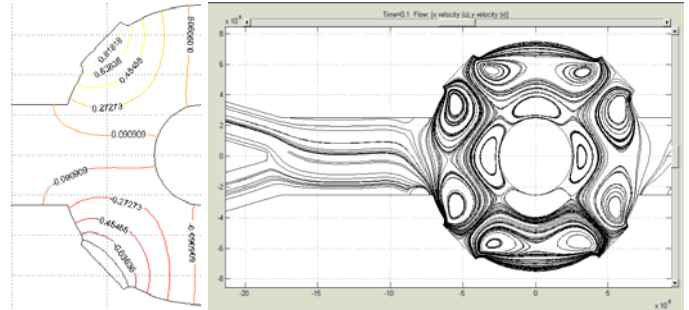


Fig.2 Electrical potential contour (left) and fluid streamlines (right) distribution by 2-D FEMLAB simulation at time equals to 0.1s. Boundary condition is given as Fig.1 illustrated with V_0 1V, frequency 10Hz

A more effective way to prove a chaotic system is to calculate the Lyapunov exponent (LE), which describes the average exponential rates of divergence of initially close orbits in the phase space. Fig. 3 gives the distance σ vs. time relation between two initially nearby particles. The slope is the Lyapunov exponent.

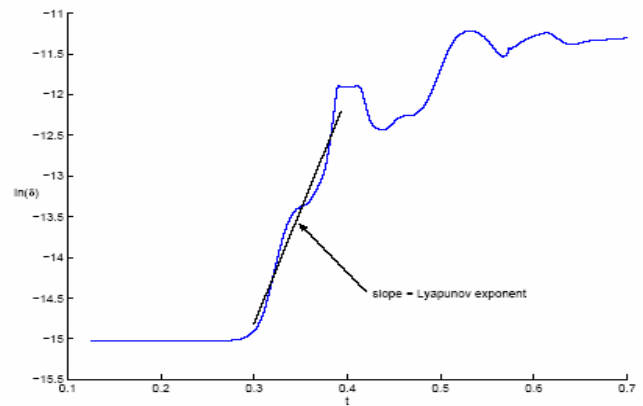


Fig.3. Evolution of the distance between two nearby particles. A positive slope implies an exponential divergence

FABRICATION

RECM is a test device for micro/nano scale computational infrastructure. To efficiently compare 2-D simulation with

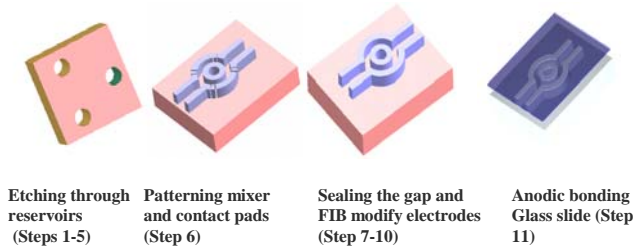


Fig.4 Main processing steps of the fabrication. In the processing, 3 reservoirs are etched through after the first layer photolithography. Then mixer and electrode networks are patterned and etched as the second layer

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. However, this raises a problem for fabrication. Usually metal sputtering or evaporating is used to deposit metal on the silicon chip as an electrode. However, 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 receptivity 0.02ohm²/cm (Ultrasil Company) 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 SiO₂ to fill the gaps between the two areas.

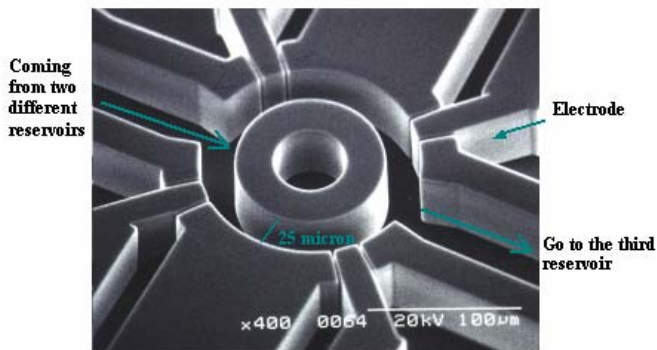


Fig.5 SEM picture of the ring mixer (25micron wide, 50 micron deep). The image shows after 41 hour 1050 °C thermal oxidation but before the FIB characterization.

A 25-micron wide, 50-micron deep mixer (see Fig.5) has been fabricated at our new MEMS/NEMS, Class 100/1000 clean room. We also designed another two sizes (1 micron and 50 micron chamber size) for later length scale study. 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.

First, a 7.5-micron thick photoresist is span (SPR220-7.0 spin at 3000 rpm for 30 seconds) on the 4-inch SOI wafer (Step 1), and then the first photolithography patterned three reservoirs on the wafer by a GCA 6300 i-line wafer stepper (Step 2). Using a deep RIE (Plasma-Therm 770 SLR), the wafer is

etched down 50 microns to expose the buried one-micron SiO₂ layer (Step 3).

Then the buried SiO₂ need to be removed. Li. et al [21] reported a 7min dipping in saturated HF (49%) to remove the buried oxide under movable structures. We developed a safer technology. After 15 seconds plasma O₂ cleaning, the wafer is immersed in improved buffer HF for 10 minutes, then repeat this procedure once. 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). At this step the first layer is done. For the second layer, we use AZ-P4110 positive photoresist (spin at 4000 rpm for 30s, 1.1microns 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 °C for 20 minutes), the wafer is loaded into the furnace (Tystar 8" oxidation furnace) to grow a 4-micron thick SiO₂ layer (wet oxidation, 1050 °C for 41 hours) to enclose the gap between the electrode and non-conducting area (Step 8). Characterization of the isolation structure is done by Focused Ion Beam (FIB) cross sectioning the silicon electrode—silicon dioxide isolation segment, Left image of Fig.6 displays that the two silicon dioxide interfaces merge and close the gap (Fig.4, Step 6). The final steps include using RIE (Panasonic E640 etcher) to remove the surface oxides so that the silicon core of the contact pad could be exposed for later probe testing (Step 9).

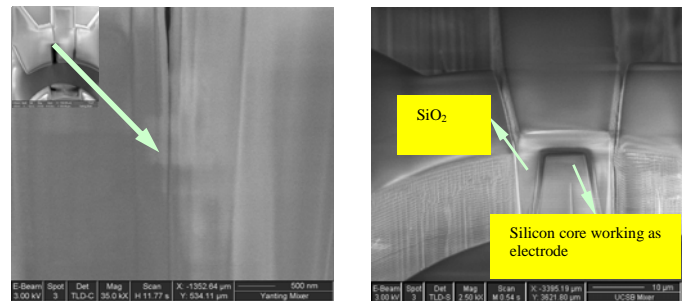


Fig.6 (left: FIB characterization of the sidewall sealing Right: FIB milled away 4 micron silicon dioxide which covers the electrode sidewall to expose the electrode to fluid

The design of the RECM requires that the electrodes must contact the fluid, but the remaining parts of the mixer are thermal oxide to achieve a higher slip velocity. Currently, FIB (FEI DB235 Dual-Beam Focus Ion Beam System) is used at ion source 20,000 µA 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.6. Step 10 can be substituted by using a third mask to etch away SiO₂ on the electrodes sidewall. Finally, a 6mm square, 170 micron thick Pyrex 7740 (Precision Glass & Optics) is anodic bonded to the chip by a self-made bonding apparatus at 420 °C and –1000V for 10minutes (Step 11). Since our cover slip is quite thin, -1200V shorts the circuit and digs a hole through the wafer while -800V is not big enough for forming current.

Temperature is another key effect here, which must be over 400 °C to enable the SOI bonding. Finally in order to improve the probing, a 200nm Aluminum thin film is evaporated on the contact pads area to enable Ohmic contact between the probe tip and the sample surface (Step 12).

DISCUSSIONS

Optimize Mixer design

We also varied some parameters such as the electrode size, the chamber size and the existence of the middle circular island to optimize the mixer design. Streamlines of the fluids are shown in Fig.7 with the same boundary conditions as above. The results show that electrode size does not influence the working mechanism of the mixing (Fig.7 (b)). The difference is electrical field is more concentrated at the electrode area when using small electrode, thus causing high density streamlines near the electrodes. It is the chamber size that plays an important role in the mixer design. Half the chamber width limits the fluid flow area (Fig.7(c)), separated surfaces in the streamline path does not form; while no middle obstacles causes no mixing occurs in the chamber middle area (Fig.7 (d)). So if each branch channel has a width of W , the best design parameter should be mixing chamber width $2W$ and middle island width $2W$.

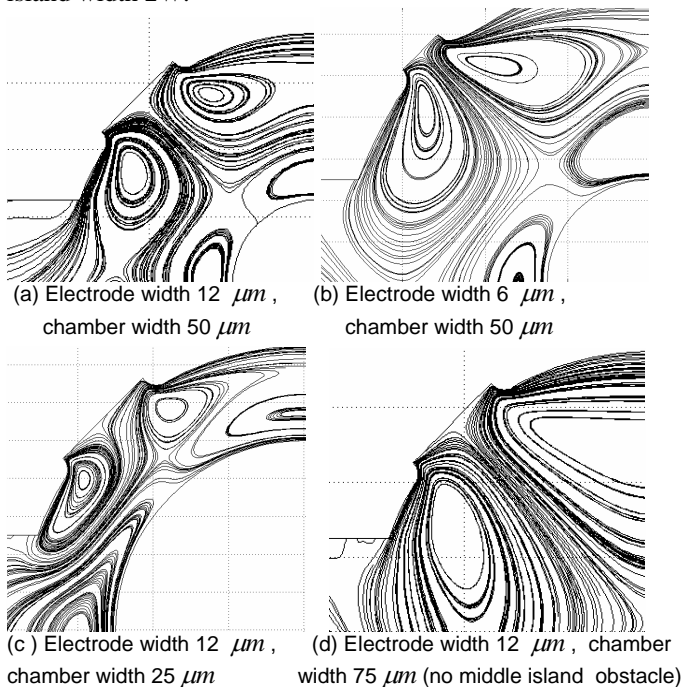


Fig.7 Streamlines of fluid field at various geometries of the mixer chamber under the maximum electrical potential 1V

Mixer testing

A mixing testing experiment is undergoing. Schematic setup picture is shown in Fig.8.

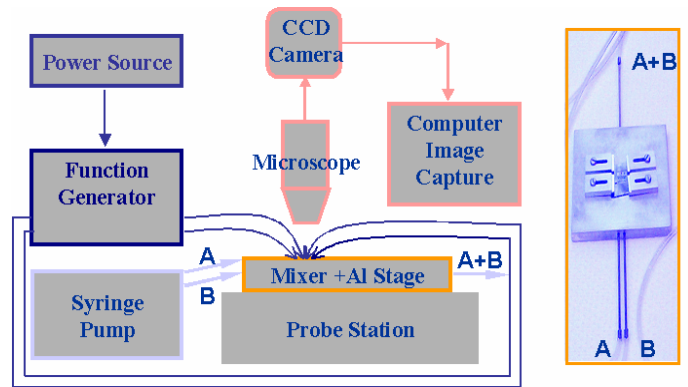


Fig.8 Left: Schematic picture of the testing setup

Right: Mixer on the Aluminum Stage with connecting tubings

Since our mixer has electrodes, redox reactions may bring electrode fouling and gas generation problem. In some cases, electron transfers do not modify the surface of the electrode, and in other cases it causes corrosion [22]. Most of the problems happen to metal electrodes. Our mixer uses heavily doped silicon as electrode materials which potentially will reduce fouling. And we use lower voltages which reduce electrolysis but not eliminate it.

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. It provides high aspect ratio electrodes that can be fabricated to the full depth of the microfluidic system. Simulation results indicate chaotic mixing is possible with this design. A mixing testing experiment is in progress, simulation data will be compared with the experiment data for the next step.

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