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### Active micromixer using electrokinetic effects in the micro/nanochannel junction

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HIGHLIGHTS

- ► A novel active micromixer combines a microchannel and nanojunction.
- Nanojunction was created by the electrical breakdown of PDMS.

▶ Mixing is caused by the non-equilibrium electrokinetic effect near the nanojunction.

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## 1. Introduction

#### ABSTRACT

We developed a novel active micromixer that uses a driving electrical voltage and combines a microchannel and nanojunction. The microchannel was fabricated using the general soft lithography process with polydimethylsiloxane (PDMS). The nanojunction was created at a specific position on the microchannel by using the breakdown of PDMS due to the application of high electrical voltage. The proposed micromixer efficiently mixed two fluid streams by the combined effects of bulk electro-osmotic flow and vortical flow created by the non-equilibrium electrokinetic effect near the nanojunction. The proposed micromixer achieved up to 90% mixing of the two fluid streams, and could be easily integrated with other microfluidic components due to its planar structure and simple fabrication process.

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Microfluidic systems used in analyzing chemical and biological samples have seen significant development in recent years [1,2]. These systems can perform various biochemical processes including drug delivery [3,4], clinical diagnostics [5,6], DNA analysis and sequencing [7,8] and biological/chemical agent detection [9,10] in microscale. The systems consist of many microscale components such as micromixers, microvalves, micropumps, and preconcentrators [11–13]. Among these micro-components, the micromixer is one of the most important parts in a microfluidic system that requires rapid and accurate mixing or reactions of two or more sample solutions. Mixing different fluids at the microscale is very slow and inefficient because fluid flows in microchannels are restricted to laminar flow, and mixing is mainly governed by the diffusion of molecules [14–16].

Recently, various micromixers designed for efficient mixing have been studied. In general, micromixers can be classified as

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either active or passive. Passive micromixers do not require external energy sources; they mostly rely on diffusion or chaotic advection using a particular channel geometry that creates vortical flow [17–19].

In contrast, active micromixers rely on moving parts [20] or an external energy source to induce the mixing of fluids. The external energy source could be an electric field [21], a magnetic field [22], a pressure disturbance [23], or an acoustic disturbance [24]. Although active micromixers can provide fast and controllable mixing of fluids, the fabrication processes for the moving parts and external energy sources are complicated and expensive. Recently, Kim et al. [25] developed a simple and effective active micromixer based on non-equilibrium electrokinetics near the micro/nano-channels. However, this mixer requires an expensive deep reactive ion etching (DRIE) process for the nanochannels. Although there are several methods of nanochannel fabrication such as focused ion beam (FIB) [26,27], electron-beam lithography (EBL) [28,29], and nanoimprint lithography [30–32], these also require complicated procedures that are costly and time-consuming.

To overcome the cost and time problems, the dielectric breakdown technique has been used as an alternative for nanochannel fabrication. Whitesides et al. [33] made nanochannels in a



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PDMS-glass microchip by applying a voltage sufficiently above the dielectric strength of PDMS. The size of the nanochannels was determined by the applied voltage and by the ionic strength of a buffer in the channels.

In this study, an active micromixer with a microchannel and nanojunction composed of small nanochannels was fabricated using only soft lithography for the microchannel and electrical breakdown for the nanojunction. The proposed micromixer achieved up to 90% mixing of two fluid streams, quickly and in a controllable manner.

#### 2. Materials and methods

#### 2.1. Microchannel fabrication

The micromixer (2.25 cm horizontal length  $\times$  100 µm width  $\times$  28 µm depth) shown in Fig. 1 was fabricated with polydimethylsiloxane (PDMS). A mold for casting PDMS was produced using the photolithograph method with SU-8 photoresist (SU-8 2025, Microchem, MA, USA) on top of a silicon wafer. Initially, a 6 in.-diameter silicon wafer was spin-coated with SU-8 at 3000 rpm for 30 s to produce a 28 µm-thick layer. The wafer was exposed to 365 nm UV light at 150 mJ/cm<sup>2</sup> (22 s at 6.8 mW/cm<sup>2</sup>) through a high-resolution dark film mask. After a post-bake process, the wafer was developed to remove unexposed SU-8 using SU-8 developer.

A 10:1 mixture of PDMS prepolymer and a curing agent (Sylgard 184, Dow Corning, Midland, MI, USA) was poured into the SU-8 mold. The mold was placed in a vacuum to evacuate any bubbles created during mixing, and was cured at 70 °C for 2 h. After curing the PDMS, the PDMS replica was detached from the mold, and holes for an inlet and outlet were punched to allow fluid samples to be introduced into the microchannel. The PDMS replica was bonded to a pre-cleaned slide glass after air plasma treatment (CUTE-100LF, FEMTO Science, Korea) for 1 min.



**Fig. 1.** Schematics of the proposed micromixer. (a) Overall micromixer system. (b) Top view of micromixer. The dimensions of the micromixer are 2.25 cm horizontal length, 100  $\mu$ m width (*W*), and 28  $\mu$ m depth. The gap (*J*) where the nanojunction is located is 30  $\mu$ m. The gap between the microchannel segments (*G*) is 150  $\mu$ m. The bent angle (*B*) is 90°.

#### 2.2. Nanojunction formation

After the microchannel was fabricated by PDMS-glass plasma bonding, a nanojunction was created at a specific position in the PDMS membrane within the 30  $\mu$ m gap between the microchannels (See Fig. 1). Formation of the nanojunction in the PDMS membrane was achieved using the electrical breakdown of PDMS without the use of etching techniques [34]. Schematics of the nanojunction formation process are shown in Fig. 2a. The fabricated microchannel was filled with 50 mM potassium chloride (KCl) buffer solution for electrical breakdown. Then, a voltage of 1400 V was applied for 1 s through inlet 1 and 2 with outlet 3 grounded. The applied voltage is above the electrical breakdown voltage of PDMS (21 V/ $\mu$ m) [33].

#### 2.3. Verification of nanojunction formation

The formation of the nanojunction created by the electrical breakdown of the PDMS membrane was verified by measuring the current inside the microchannel filled with 50 mM KCl solution before and after the electrical breakdown as a function of applied voltage. The voltages were applied from 10 to 50 V using a picoammeter (Keithley 6487 picoammeter/voltage source, Keithley Instruments, Inc., USA).

The dimensions of the nanojunction were approximately measured using fluorescent dye imaging and an electrical circuit model. First, we assumed that the nanojunction is a bundle of nanochnnels and then the width and length of the nanojunction were measured by filling the nanojunction with  $100 \mu$ M Rhodamine B dye in deionized (DI) water. An image of the nanojunction, shown in Fig. 3a, was captured using an inverted fluorescence microscope (Ti-U, Nikon, Japan). The width and the length of the nanojunction were measured using the NIS-Elements image processing tool (Nikon, Japan).

The depth of the nanojunction was measured by assuming that the resistance of the KCl solution in the channel is proportional to the length of the channel. The resistances of the channel before and after the electrical breakdown of the PDMS membrane can be expressed as shown in Fig. 2b [35]. The resistance of the KCl solution in the channel is given by

$$R_{\text{before}} = \frac{R_1 R_1}{R_1 + R_1} + R_2 + R_3 + R_4 \tag{1}$$

$$R_{\text{after}} = \frac{R_1 R_1}{R_1 + R_1} + R_2 + \frac{R_3 R_n}{R_3 + R_n} + R_4 \tag{2}$$

The depth of the nanojunction can be calculated as follows:

$$D_n = \rho \frac{L_n}{W_n R_n} \tag{3}$$

where  $\rho$  is the resistivity of the KCl solution,  $D_n$  is the depth of the nanojunction, and  $L_n$  and  $W_n$  are the length and width of the nanojunction, respectively.

#### 2.4. Mixing experiments

Mixing experiments were performed with rhodamine B (Rh. B) fluorescent dye solution to visualize the mixing of the fluids. 1 mM KCl solution and 1 mM KCl solution labeled with 100  $\mu$ M Rh. B were introduced at inlets 1 and 2, respectively, as shown in Fig. 1. In this experiment, we chose 1 mM KCl solution as an electrolyte to make the nanochnnel formed by electrical breakdown of PDMS work as a perm-selective membrane, since the lower concentration electrolyte solution can form a thicker EDL that more easily induces overlapping of the EDL compared to a higher concentration solution [35,36].

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