

Pressure-driven flow control system for nanofluidic chemical process[☆]

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Abstract

We developed a novel flow control system for a nanofluidic chemical process. Generally, flow control in nanochannels is difficult because of its high-pressure loss with very small volume flow rate. In our flow control method, liquid pressure in a microchannel connected to the nanochannels is regulated by utilizing a backpressure regulator. The flow control method was verified by using simple structured microchip, which included parallel nanochannels. We found that the observed flow rate was three times lower than the value expected from Hagen–Poiseuille's equation. That implied a size-dependent viscosity change in the nanochannels. Then, we demonstrated mixing of two different fluorescent solutions in a Y-shaped nanochannel and also a proton exchange reaction in the Y-shaped nanochannel. The flow control method will contribute to further integration of nanochemical systems.

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1. Introduction

In recent years, research on integration of chemical systems using microfabricated devices has been the focus of much attention from analytical chemists. The integrated systems are known as miniaturized total analysis systems (μ -TAS) or lab on a chip [1–3]. Various applications, such as organic synthesis [4], DNA analysis [5], cell analysis [6] and so on, have been achieved. We have developed the concepts of micro unit operations (MUOs) and continuous-flow chemical processing (CFCP) to utilize the advantages and characteristics of microfluidic devices [7]. In order to realize various kinds of MUOs, organic solvent as well as aqueous solvent should be handled in microspace. Therefore, a pressure-driven flow system has been used in CFCP because organic solvent can be handled unlike in an electroosmotic flow (EOF) system, which can only handle aqueous solvent.

In micro fluidic devices, diffusion time is very short and the specific interfacial area is very high and effective chemical

processes have been realized by utilizing these characteristics [8–12]. Further integration of chemical systems will enable us to utilize advantages of microfluidic devices even more effectively, which will lead to nanofluidic chemical processing (NFCP). As a typical chemical process, we investigate mixing and reaction in a nanochannel.

Since the specific interface area of a channel becomes much higher for NFCP, interfacial phenomena become very influential. A size-confinement effect in nanospaces has been investigated in porous materials such as porous silica or zeolite [13,14]. In our previous report [15], water in a fused-silica nanochannel was investigated with time-resolved fluorescent spectroscopy.

From an engineering viewpoint, further integration and more effective chemical processes can be achieved in NFCP. From the viewpoint of basic chemistry, it is important to investigate how property changes in the nanospace will affect specific chemical processes. In order to realize NFCP, a flow control technique for nanochannels is indispensable. In previous papers on nanofluidic systems [16,17], EOFs have been demonstrated. However, in EOF systems, only aqueous solutions can be utilized and applications are limited. Therefore, a pressure-driven flow control method is favorable for a general nanofluidic chemical process.

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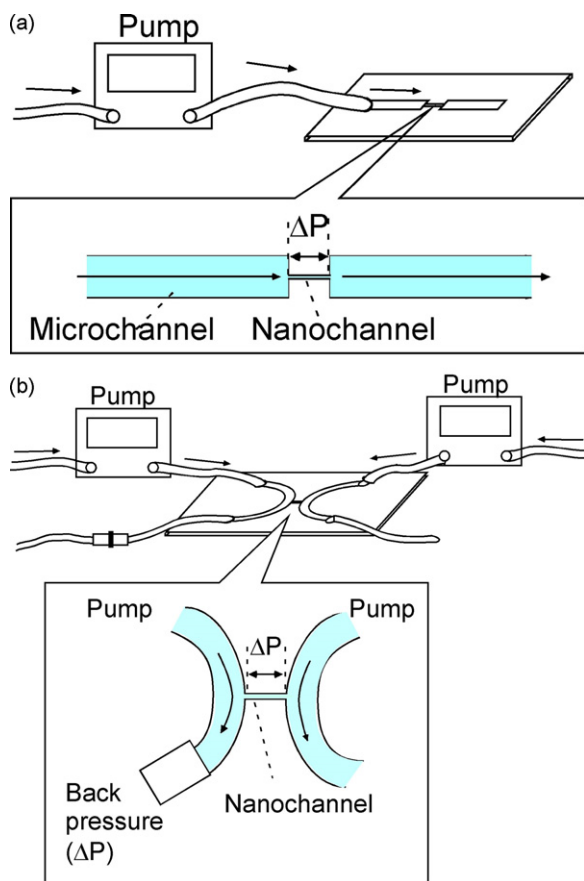


Fig. 1. (a) Schematic of a conventional liquid introduction system. (b) Schematic of a backpressure regulation system.

In a conventional pressure-driven flow method, pressure from a pump was directly applied to a nanochannel (Fig. 1a). A normal HPLC pump cannot offer stable flow when the volume flow rate is less than $1 \mu\text{l}/\text{min}$. In a 100-nm-diameter nanochannel, 700 GPa is required for a flow rate of $1 \mu\text{l}/\text{min}$, however, connectors or microchips would be broken under such a high-pressure condition. When the pressure is 1 MPa in the nanochannel, the flow rate is 1 pl/min, which is too low rate for control by a normal HPLC pump. Furthermore, when the flow rate of 1 pl/min is applied, it takes a very long time to replace solution in the reagent microchannel, which has a volume of 1 nl at least.

In this paper, we developed a novel pressure-driven flow method for NFCP. In the method, a U-shaped microchannels for liquid introduction was connected to a nanochannel and the pressure was applied indirectly utilizing a backpressure regulator (Fig. 1b). Since most part of the flowing liquid goes through the backpressure regulator, the volume flow rate up to several $\mu\text{l}/\text{min}$ is allowed and pressure at the entrance of the nanochannel remains constant. In order to evaluate the performance of this method, we investigated dependence of the flow rate in nanochannels on the backpressure. Then, we fabricated a Y-shaped nanochannel, where the three ends of the nanochannel were connected to U-shaped microchannels. First, we demonstrated mixing of two different fluorescent solutions

in the nanochannel. Second, we demonstrated a proton exchange reaction in the Y-shaped nanochannel. Our flow control method offers a very low flow rate with high pressure, which is necessary for NFCP. This flow control system will contribute to the development of NFCP.

2. Experimental

2.1. Fabrication

Each substrate was a 0.7-mm-thick synthetic silica glass rectangle plate with 30-mm and 70-mm sides. Details of the fabrication scheme were described previously [15]. A nanoscale patterning was drawn by an electron-beam lithography system (ELS-7500, Elionix, Hachioji, Japan) on the substrate coated with an electron-beam resist (ZEP-520, Zeon, Tokyo, Japan) and conductive polymer (Espacer 300, Showa Denko, Tokyo, Japan). The pattern was developed by xylene and etched with a plasma etching system (NE-550, Ulvac, Chigasaki, Japan).

Microchannels were fabricated by photolithographic patterning and plasma etching. After the nanochannel fabrication, a chromium layer was sputtered on the substrate. A photoresist layer (OFPR-800, 20 cP) was coated on the chromium layer. A photomask with microchannel pattern was aligned on the substrate with a mask-aligner. After developing the photoresist, the chromium layer was etched. A microscale was also etched using the plasma etching system. Finally, the chromium and photoresist layers were removed. Holes were drilled through the substrate at each end of the microchannels. A vacuum electronic furnace (KDF-900GL, Denken) was used to thermally bond a 0.7-mm-thick silica glass cover plate to the substrate; bonding temperature was 1030°C .

2.2. Experimental setup

A microchip holder for aligning and fixing fluid connectors was made of stainless steel. The connectors consisted of PTFE ferrules and stainless steel nuts. The resisting pressure of this system was about 30 MPa. Backpressure regulators (P-762 to P-765, P-455, Upchurch; regulating pressure 0.51 MPa, 0.69 MPa, 1.72 MPa, 3.45 MPa, 6.90 MPa, respectively) were used for regulating the pressure in the microchannel.

2.3. Chromatography

For chromatographic determination of probe and internal standard molecules in the flow rate measurements, a packed silica-gel column was used. The silica gel has a diameter of $4.0 \mu\text{m}$ and octadecylsilane groups. The column diameter and length were 4.0 mm and 25.0 mm, respectively. For mobile phase of separation, aqueous solution of methanol (50%, v/v) was pumped with a flow rate of 0.5 ml/min. Sample injection volume was set to $20 \mu\text{l}$ and absorbance of the probe and internal standard molecules at a wavelength of 254 nm were recorded.

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