

A Solid Electrolyte Valve for Micro/Nano-fluidics

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ABSTRACT

This paper presents a nanovalve that uses electrodeposited nanostructures on a solid-state Ag-Ge-Se electrolyte to regulate liquid flow in micro/nano-fluidic channels. The valve uses the extremely rough surface morphology of electrodeposits to manipulate hydrophobicity, thereby closing and opening the nanovalves. Electrodeposited nanostructures can be grown and retracted on the electrolyte surface by applying voltages between 5V and -20V at room temperature. The nanovalve successfully closes and opens de-ionized water (DI) flow in a 20 μ m tall and 500 μ m wide microfluidic channel. Closing and opening a single nanovalve takes 5.3 min and 3 min, respectively, with a power consumption of 0.08 mW and 0.17mW, respectively. A triple nanovalve takes 3.8min and 2.5min to close and open, representing a 28% and 17% decrease in valving time as compared to a single nanovalve.

Keywords: Nanovalve, MEMS, solid-state electrolyte,

1 INTRODUCTION

Advances in micro-electromechanical system (MEMS) technology and biotechnology have resulted in the success of widely-applied integrated microfluidics (e.g. Lab-on-Chip or micro-Total Analysis Systems (μ TAS)) in biology development, biomedical research, and drug delivery systems [1,2]. The miniaturization of microfluidic systems decreases chemical reaction time and decreases the required volume of expensive chemicals [3]. In addition, miniaturized microfluidic systems reduce the complexity of labor-intensive processing, enhancing task replication and reliability.

From a practical implementation standpoint, however, Lab-on-a-Chip or μ TAS systems pose a challenge because they demand precise fluid control, delivery and regulation of sample fluids in micro/nano-scale channels. For typical fluidic systems, three microfluidic components control fluid motion: micropumps, microvalves, and micromixers. Among these components, a microvalve is essential to fulfil the requirements of precise chemical delivery, dose control, reaction regulation, and more [4]. Generally, microvalves can be divided into two categories: passive and active valves. A passive valve controls the flow in one direction without an actuation mechanism and it is difficult to maintain the valve state. By contrast, an active valve regulates flow in both directions through different actuation

methods, including pneumatic [5], magnetic [6], piezoelectric [7], thermal [8], or electric [9,10] techniques. These valves generally require high voltage or high power consumption (10-100 mW) [11]. Also, reliability, time, and economics are major issues for practical applications of these devices. We present a bistable microvalve with nanometer scale surface roughness which has low operational power consumption and shows potential for scaling down to the nanometer regime. The microvalve is actuated electronically and manipulates the hydrophobicity via surface morphology. In the following sections, we present the material characteristics and microvalve design, and describe the process flow and test setup.

2 NANOVALVE WORKING PRINCIPLE

The working principle of this nanovalve is very similar to electrodeposition using liquid electrolytes; instead of liquid electrolyte, however, we use a solid-state Ag-doped chalcogenide glass ($\text{Ge}_{30}\text{Se}_{70}$). Ge-Se glass becomes a solid-state electrolyte when it is photodoped with silver [12]. Photodoping is the light-simulated dissolution and diffusion of metals like silver and copper into a chalcogenide film [12]. When a positive voltage is applied between the silver (Ag) anode and nickel (Ni) cathode on the solid-state Ag-Ge-Se electrolyte, Ag is oxidized and Ag^+ ions move toward the Ni cathode through the solid-state Ag-Ge-Se as shown in Figure 1(a). Ag^+ ions are reduced at the Ni (cathode) and start forming silver nano-electrodeposits toward the Ag anode. These silver nano-electrodeposits shown in Figure.1(b) have rough hydrophobic surfaces and are 200 to 300 nm in height.

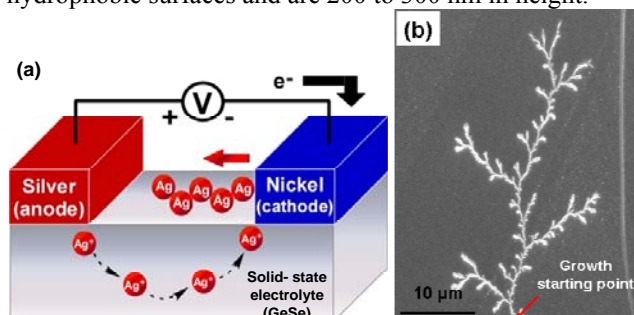


Figure 1: (a) Silver nanodeposits working principle and (b) SEM picture of grown silver nanodeposits.

The physical characteristics of the silver electrodeposits are determined by changes in surface morphology resulting

from their growth and retraction. The nanovalve closes as silver electrodeposits grow under positive applied voltage, and opens as silver electrodeposits retract under negative applied voltage.

3 DESIGN AND FABRICATION

3.1 Nanovalve Design

The nanovalve design is shown in Figure 2.

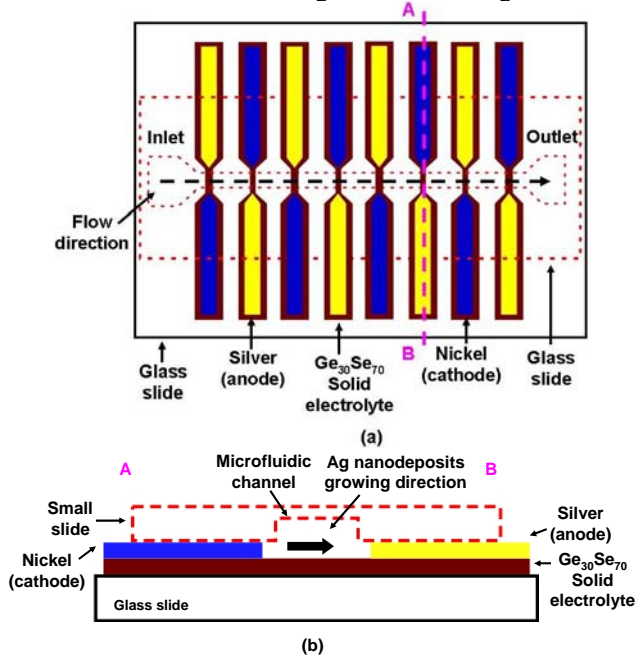


Figure 2: Microvalve and microfluidic channel design, (a) top view, and (b) cross-section view.

Since the specific growth pattern of silver electrodeposits is dendritic and therefore somewhat unpredictable, we designed a narrow line of solid electrolyte between the Ag (anode) and Ni (cathode) to provide a guide path. This narrow line of solid electrolyte is placed precisely underneath and perpendicular to the microfluidic channel to facilitate its opening and closing. The Ag anode and Ni cathode each have a sharp tip aligned to the narrow path and are positioned directly facing each other to create the highest possible electric field between them. The nickel cathode of each nanovalve is placed in a zigzag pattern on the solid electrolyte to create an alternating growth origin for silver electrodeposits.

3.2 Fabrication Process Flow

The nanovalve and microfluidic channels are fabricated on glass slides. Two different sizes of slide are used; $75 \times 40 \times 10 \text{ mm}^3$ and $75 \times 15 \times 10 \text{ mm}^3$, length, width, and thickness, respectively. The large and small slides are used to fabricate the nanovalve and the microfluidic channel, respectively. Fabrication processes are shown in Figure 3, beginning with nanovalve fabrication.

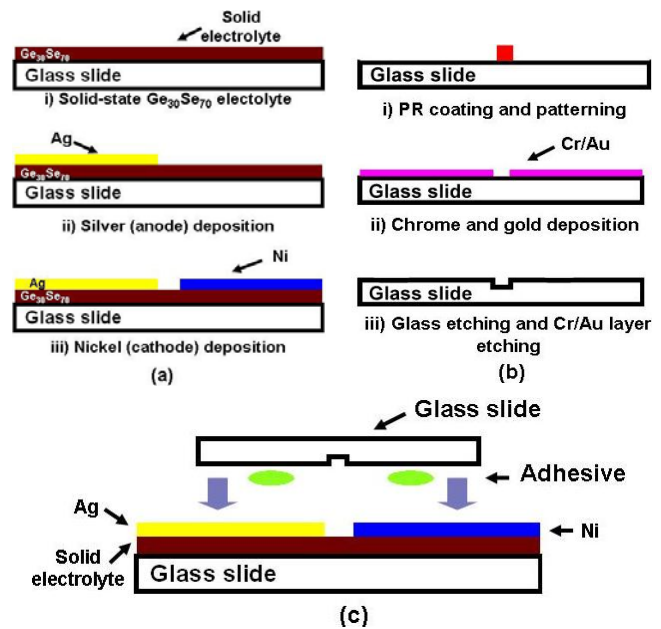


Figure 3: Process flow, (a) nanovalve fabrication, (b) microfluidic channel fabrication, and (c) bonding nanovalve and microfluidic channel.

Photoresist (PR) AZ 4330 from Clariant Corp is coated and lithographically patterned to define the solid electrolyte areas. $\text{Ge}_{30}\text{Se}_{70}$ and silver are thermally deposited 120nm and 40nm, respectively, and then silver is photo-dissolved into the $\text{Ge}_{30}\text{Se}_{70}$ film to form a solid Ag-Ge-Se electrolyte film which is then patterned by a lift-off process. The Ag (anode) and Ni (cathode) are lithographically patterned and deposited by an e-beam evaporator. The microfluidic channel, 20 μm high by 500 μm wide by 45 mm long, is etched by hydrofluoric acid using a chrome/gold (20nm/200nm) mask on the smaller glass slide as shown in Figure 3(b). After the smaller glass slide is etched to 20 μm , the chrome/gold mask is removed and we drill a hole on the inlet and outlet reservoirs. A nanoport from Upchurch is then attached on top of the inlet and outlet holes, and the two glass slides are bonded using adhesive. The fabricated nanovalve and SEM cross-sectional picture are shown in Figure 4.

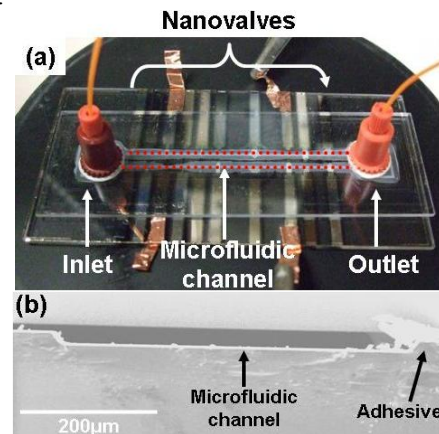


Figure 4: Photo of the fabricated sample: (a) top view and (b) channel cross-section.

3.3 Test Setup

The entire test setup is shown in Figure 5.

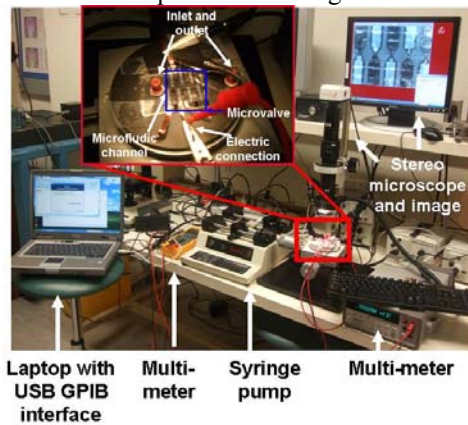


Figure 5: Experimental setup.

A syringe pump, Harvard Apparatus pump 33, delivers de-ionized (DI) water to the microfluidic channel with a 2 $\mu\text{L}/\text{min}$ flow rate. A 2.3mm-diameter glass syringe (Harvard Apparatus) with 250 μL total volume is used because of its high applied pressure. The outlet of the microfluidic channel is connected to a Honeywell flow meter (X115673-AW Liquid Flow Sensor). The flow meter's output voltage is recorded directly to a laptop via infra red/USB connection to a multi-meter (Fluke 189). A power supply from Agilent provides the 14V required to grow silver nano-electrodeposits and close the microfluidic channel, and the -20V required to retract the silver nano-electrodeposits and open the microfluidic channel between two electrodes in the nanovalve. The current between two electrodes is measured by a current meter from Agilent (34401A) connected by GPIB/USB, which allows the data to be automatically saved to a laptop.

4 RESULTS

4.1 Flow Meter Calibration

The flow meter characterization shows the relationship between the output voltage of the flow meter and flow rate in the microfluidic channel. The flow meter can measure flow rate ranging from 5nl/min to 5 $\mu\text{L}/\text{min}$, corresponding to output voltage from 1V to 5V.

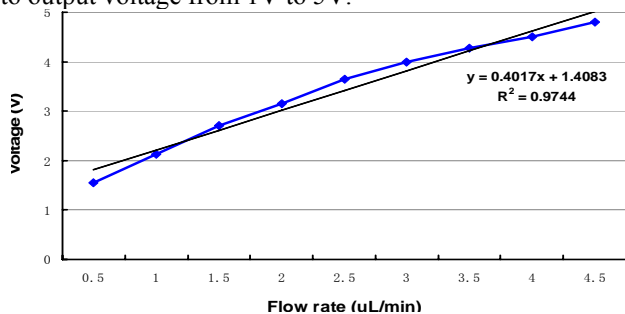


Figure 6: Calibration result of the flow meter, showing flow rate vs. output voltage.

Figure 6 shows the calibration result of the flow meter, output voltage versus flow rate. Based on the calibration result, we can check the nanovalve functionality. In this experiment, 2 $\mu\text{L}/\text{min}$ flow rate corresponds to 3V output voltage.

4.2 Single Nanovalve

Figure 7 shows that one nanovalve successfully closes and opens a 2 $\mu\text{L}/\text{min}$ flow rate of a DI water stream in the 500 μm width of a microfluidic channel.

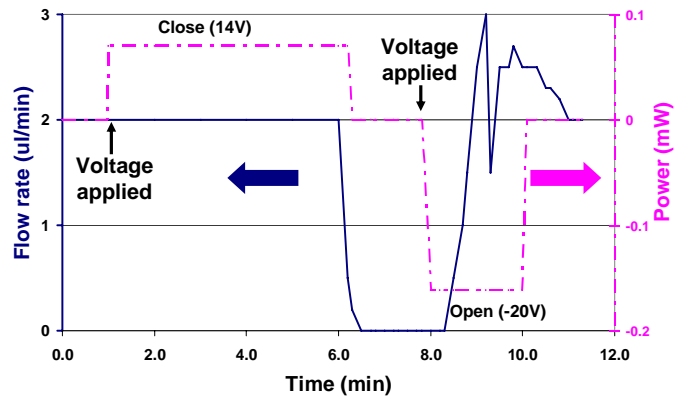


Figure 7: Experimental results for the single nanovalve.

The left and right Y axes in Figure 7 represent flow rate and power consumption, respectively. Applying 14V to the nanovalve, it takes 5.3 min to close the microfluidic channel so that it remains closed. Applying -20V retracts the nanovalve's silver nano-electrodeposits, opening the channel within 3 min. The single nanovalve's power consumption is 0.08mW when closing, and 0.17mW when opening.

4.3 Triple Nanovalves

Figure 8 shows the results of triple nanovalve operation.

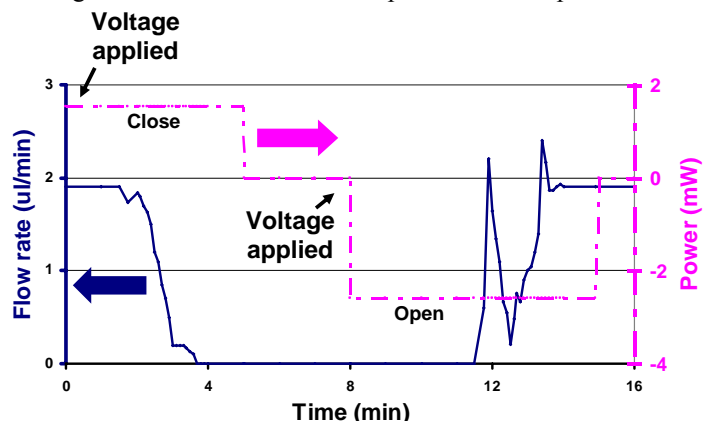


Figure 8: Experimental results for the triple nanovalve.

The triple nanovalves take 3.8 min to close the microfluidic channel, and 2.5 min to open it. The power consumption of triple nanovalves is 1.8mW when closing the microfluidic channel, and 2.8mW when opening it. The results for the triple nanovalve shows that its valving time to close and open is 28% and 17% shorter, respectively, than the single nanovalve counterpart. The triple valve does, however, have significantly greater power consumption than its single valve counterpart.

	Closing time (min)	Opening time (min)
Single nanovalve	5.3	3.0
Triple nanovalves	3.8	2.5
Decrease rate	28%	17%

Table.1 comparison of single and triple nanovalves

5 CONCLUSION

We present a nanovalve that uses silver nano-electrodeposits to manipulate the flow of microfluids. The nanovalves are electrically actuated by applying voltage between silver (anode) and nickel (cathode) on top of a solid-state Ag-Ge-Se electrolyte, which is made of chalcogenide glass (Ge₃₀Se₇₀) with photodoped silver. The results of this experiment show that the nanovalve successfully closes and opens a microfluidic channel 500µm wide and 20µm deep using silver nano-electrodeposits. The single nanovalve closes and opens the microfluidic channel in 5.3 min and 2 min, respectively, consuming power for close and open with 0.08mW and 0.17mW, respectively. Triple nanovalves decreased that closing and opening time by 28% (to 2.5 min) and 17% (to 3.8 min), respectively. The backpressure of the nanovalve is approximately 71 psi when the valve is closed.

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