

# Multifunctional Microvalves Control by Optical Illumination on Nanoheaters and Its Application in Centrifugal Microfluidic Devices

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## ABSTRACT

The valve is made of nanocomposite materials in which 10 nm-sized ferrooxide nanoparticles are dispersed in paraffin wax and used as nanoheaters when excited by the laser irradiation. Laser light of relatively weak intensity was able to melt the paraffin wax with the embedded ferrooxide nanoparticles, whereas even a very intense laser beam does not melt wax alone. The microvalves are leak-free up to  $403.0 \pm 7.6$  kPa and the response time to operate both normally closed and normally opened microvalves are less than 0.5 sec. Furthermore, a sequential operation of multiple microvalves on a centrifugal microfluidic device using single laser diode was demonstrated.

**Keywords:** microvalves, laser irradiation, ferrooxide nanoparticle, centrifugal microfluidics, lab-on-a-chip

## 1 INTRODUCTION

One of the attractive microvalves that has been implemented in lab-on-a-chip devices is phase change based microvalve. Various kinds of materials such as hydrogel, sol-gel, paraffin, and ice that can undergo a phase transition from solid to liquid in response to changes in temperature have been investigated. For example, Kellogg *et al.* used wax valve on a CD platform [1]. Liu *et al.* [2]. developed an integrated device for pathogen detection using paraffin microvalves, taking advantages of its simplicity and biocompatibility.

In the proposed novel microvalve concept of Laser Irradiated Ferrowax Microvalves (LIFM) as shown in Figure 1A, magnetic nanoparticles are dispersed in paraffin wax and used as nanoheaters when excited by the laser irradiation. Figure 1B and Figure 1C shows the basic concept to open the Normally Closed LIFM (NC-LIFM)

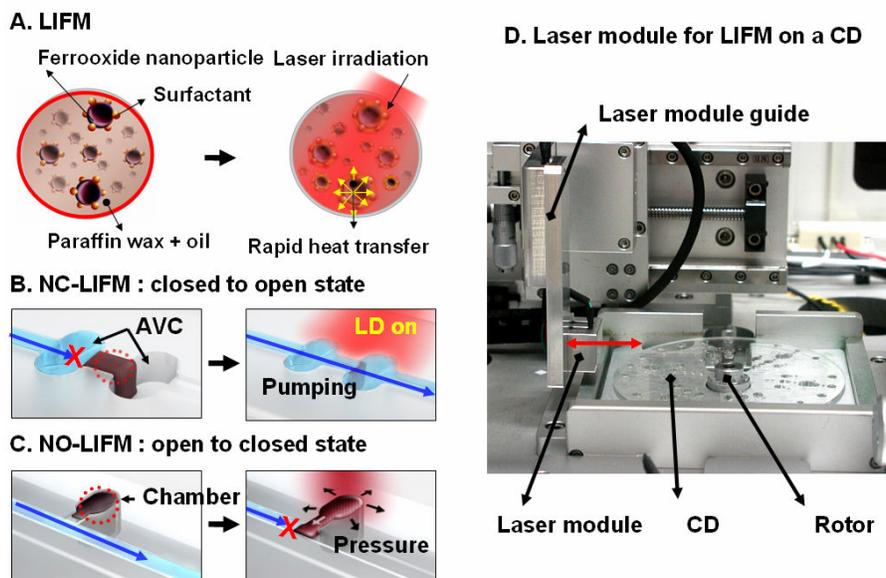


Figure 1 : Schematic diagram of LIFM operation. (A) The principle of LIFM. Highly heated nanoparticles by laser irradiation transferred heat energy to paraffin wax around the nanoparticles, resulting in accelerated melting. (B) Ferrowax plug for NC-LIFM is formed in the capillary between two AVC. As laser power is applied, the molten ferrowax flows to the AVC, solidifies, and resulting in the main channel being opened. (C) In order to block the fluid passage, ferrowax chamber located adjacent to the main channel is preloaded with ferrowax. As the ferrowax is heated by laser irradiation, the molten ferrowax burst into the main channel and immediately solidified and blocks the channel. (D) A photograph showing the experimental set-up for the centrifugal microfluidics and the laser diode motion control unit.

and to close the Normally Opened LIFM (NO-LIFM), respectively. To open NC-LIFM, laser beam is focused at the valve location and the molten ferrowax flows to the Assistant Valve Chamber (AVC) and solidifies, and results in the main channel being opened as shown in Figure 1B. To block the channel, the laser beam is focused at the pre-loaded ferrowax chamber that located adjacent to the main channel. Upon laser irradiation, the pressure of pre-loaded ferrowax increases and the molten ferrowax burst into the main microchannel and blocks the channel as shown in Figure 1C. As a result, rapid operation of many LIFM is possible using only single laser diode without needs of fabricating many embedded microheaters. Figure 1D shows the centrifugal microfluidics experimental set-up for the laser-diode motion control unit and the rotor.

In this report, we also used the phase transition of the ferrowax for valving mechanism; however, only single laser diode was used instead of many microfabricated heaters and magnets. As a result, it becomes very simple to control multiple microvalves. We have demonstrated the operation of multiple microvalves on a centrifugal

microfluidic platform by using single laser diode. In addition, the response time to actuate microvalves were accelerated because the laser beam effectively heat the ferrowax nanoparticles embedded in the paraffin wax matrix. To the best of our knowledge, it is the first to use optical excitation of nanoparticles for the mechanism of microvalve operation. Because it is fast, robust, cost-effective, biocompatible, and simple to implement for the control of multiple microvalves on a chip, it could be a key component to realize fully integrated lab-on-a-chip devices [3].

## 2 MATERIALS AND METHODS

As shown in Figure 2A, the polycarbonate (PC) test chip is composed of a top layer consisting of various inlet holes and a bottom layer consisting of channels and chambers. The channel width was 1 mm and the depth was 100  $\mu\text{m}$ . The depth of the chamber was 3 mm. The inlet holes and channels were produced by conventional CNC, and the two separate layers were bonded by adding

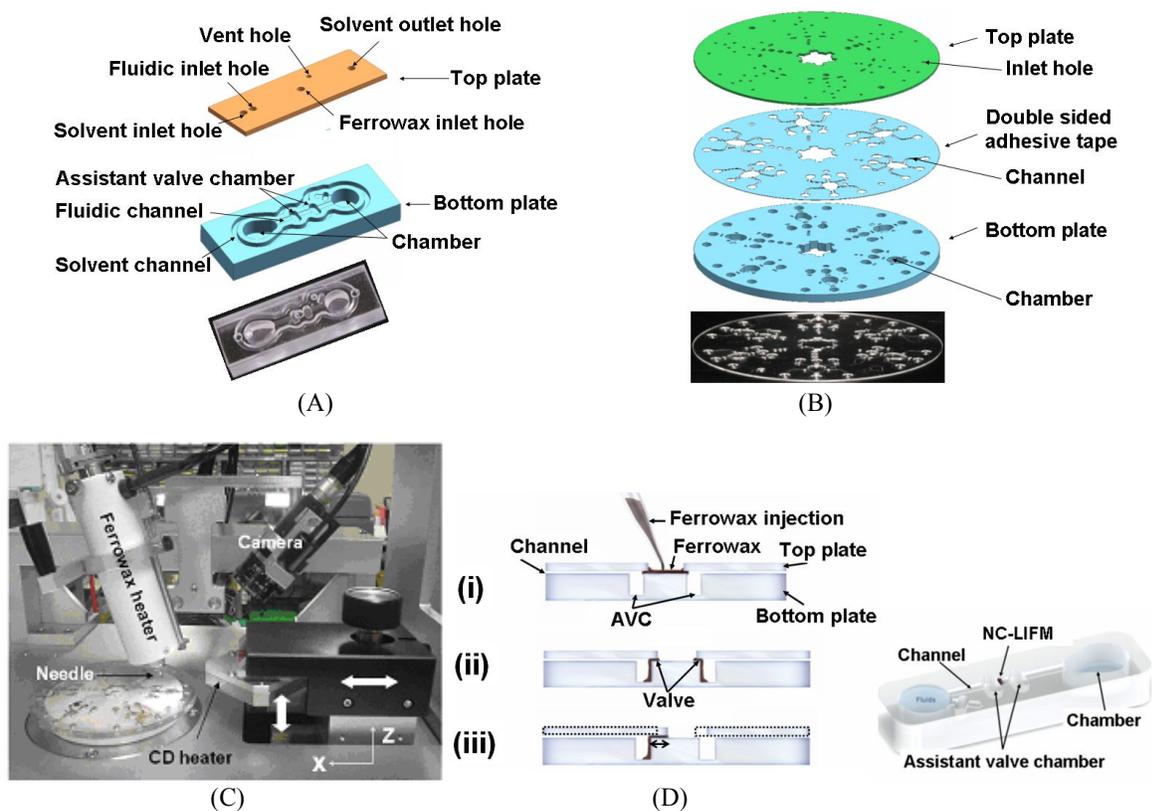


Figure 2 : Schematic diagrams and images of the microfluidic test chip for normally closed valve (A) and test CD for application of LIFM on rotational microfluidic platforms (B). The dispensing ferrowax (C) was automatically controlled by a custom-designed instrument equipped with CD heater, CCD camera, X-Y stage and a syringe installed with a ferrowax heater schematic diagrams for the fabrication of LIFM. Schematic diagrams for the fabrication of NC-LIFM (D). (i) Injection of the molten Ferrowax into the channel of the microfluidic test chip for normally closed valve. (ii) Formation of the LIFM in microchannel and excess amount of the molten Ferrowax flows into the AVC (iii) Desired valve length control using by the Ferrowax injection hole and AVC position.

20  $\mu\text{L}$  of acetone to the solvent injection hole and moved via capillary action through the solvent channel. The distribution of acetone through the gaps in the fluidic channel, chamber and the solvent channels resulted in a permanent bonding.

As shown in Figure 2B, CD type microfluidic devices are made of polycarbonate (PC) plates bonded with a double sided adhesive tape. The inlet holes and microfluidic layouts were produced by conventional CNC. The top plate has inlet holes and the bottom plate has chambers with a depth of 3 mm. The microfluidic channels to transfer liquids between reservoirs are formed in the middle layer of adhesive tape. The width of the channels was 1 mm and the depth of the channel was 0.1 mm because the thickness of the tape was 100  $\mu\text{m}$ .

After heating the plastic test chip or CD using a hot plate at 80  $^{\circ}\text{C}$ , the molten ferrowax was placed into each inlet holes located on the top plate of the chip using a custom-designed wax dispensing machine as shown in Figure 2C. Ferrowax consists of paraffin wax and ferrofluid.

The molten ferrowax is oily and has very low surface tension because its major component is the hydrocarbon based oil. Therefore, immediately after making contact with the floor of the channel molten Ferrowax exhibits capillary action and moves easily through the channel Figure 2D-i. Also, as shown in Figure 2D-ii, Excess amount of the molten Ferrowax flows into the AVC and therefore, the effect of this valve occurs only where the two plates overlap. The chip was then removed from the hot plate. The Ferrowax solidified, resulting in microvalves in the fluidic chip. In addition, by varying the exact location in which the Ferrowax is placed, one can control the dimensions (ie. length) of the valve Figure 2D-iii. The Ferrowax access holes were subsequently sealed using optical adhesive tapes.

In order to measure the the response time of the LIFM, a test chip shown in Figure 2A was used. Green color ink was introduced with the flow rate of 100  $\mu\text{Lmin}^{-1}$  using a syringe pump (Harvard PHD2000, USA). Pressure of 46 kPa was maintained using a pressure sensor and a data acquisition system. At the same time, the laser beam (808 nm, 1.5 W) was irradiated at the valve and the total process was captured by using a high speed camera.

The hold-up pressure of LIFM was measured again by using the pressure sensor and the data acquisition system by flowing a dye solution with a flow rate of 100  $\mu\text{Lmin}^{-1}$  using a syringe pump.

### 3 RESULTS AND DISCUSSION

The response time to open the NC-LIFM and to close NO-LIFM was only  $12 \pm 1$  msec and less than 444 msec as shown in Figure 3A and Figure 3B, respectively. Compared to the response time of 2 ~ 10 sec. for the case

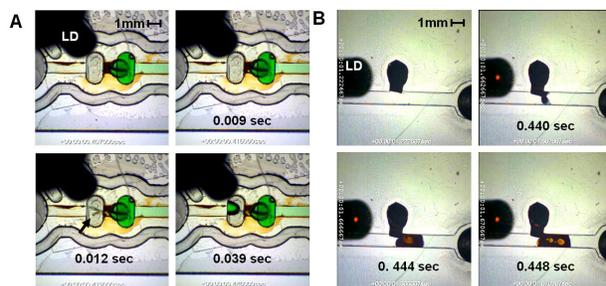


Figure 3 : Photo images of LIFM measured by high speed camera. (A) Upon the laser irradiation (808 nm, 1.5 W), the ferrowax plug was melted at 0.012 sec and the channel was open to flow liquids (arrow) when the pressure of 46 kPa was maintained. (B) Ferrowax in the NO-LIFM chamber burst into the main channel and complete sealing was obtained within 0.444 sec of laser irradiation. The ferrofluid composition was 50%.

of conventional wax valve, it is significant improvement that both NO-LIFM and NC-LIFM could be operated within 0.5 sec. without needs of additional chip space or extra tools such as air pocket or external pump.

We further demonstrated sequential operation of multiple microvalves on a centrifugal microfluidic device using single laser diode as shown in Figure 4. Table 1 shows the spin program and the detail description of the process at each operation step.

Majority of centrifugal microfluidic platforms so far utilized either hydrophobic valves; hydrophobic surface prevents further liquid flow, or capillary valves; liquid stops by a capillary pressure barrier at junctions where the channel diameter suddenly expands. The fabrication and the simultaneous actuation of multiple valves were relatively simple. However, for the robust control of the valving operation, fine tuning of the spin speed as well as the local surface properties or dimension of the microchannels were required. As a result, only a limited number of biological assays that do not require complex fluidic design have been developed on a CD platform and launched to the market.

The proposed laser irradiated microvalves are not sensitive to spin speed or surface properties. Furthermore, it functions as both to open and to block the channel using the same actuation mechanism. As demonstrated in Figure 4, various microfluidic functions such as metering, valving, and mixing was possible using the innovative LIFM together with the centrifugal microfluidic pumping.

### 4 CONCLUSION

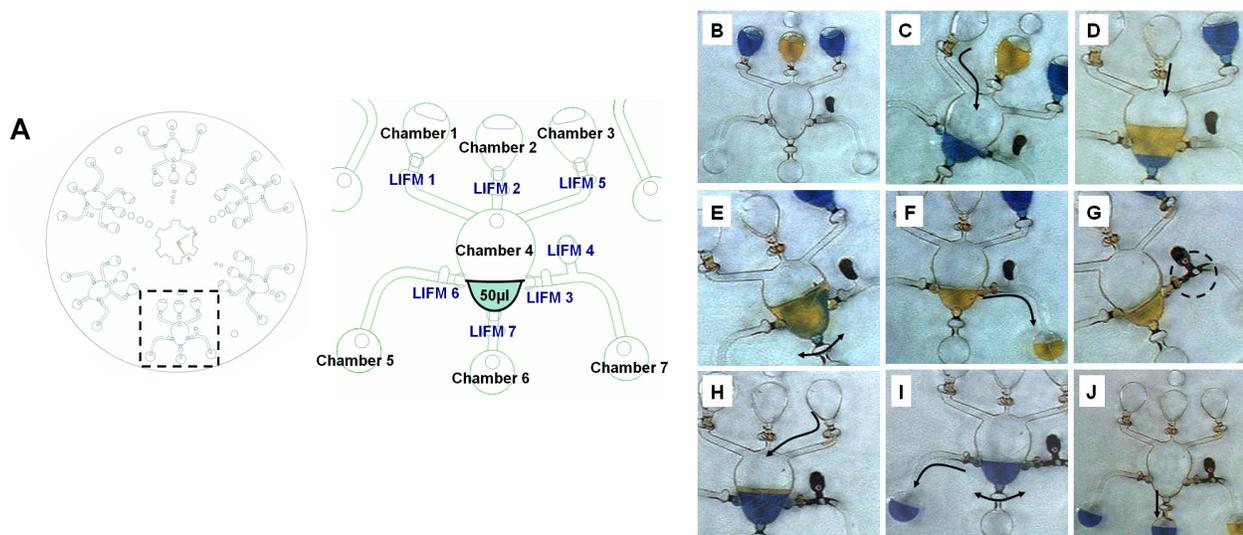


Figure 4 : (A) Schematic diagram of the microfluidic layout on a disc. (B ~ J) Photo images captured during the operation of the microfluidics on CD.

Table 1 : A spin program for the microfluidics on a CD

Spin No.	Spin speed (Hz)	Time (sec)	Operation
-	-	-	Input 0.1 N NaOH solution (blue) 50 µL to chamber 1 (Figure 4B). Input 0.1 N HCl (yellow) 50 µL to chamber 2 (Figure 4B). Input 0.1N NaOH (blue) 50 µL to chamber 3 (Figure 4B).
1	30	5	LIFM 1 opened, blue solution transfer to chamber 4 from chamber 1 (Figure 4C).
2	30	5	LIFM 2 opened, yellow solution transfer to chamber 4 from chamber 2 (Figure 4D).
3	+9 ~ -9	12	Mixing, Color changes from blue to yellow by neutralization (Figure 4E).
4	30	5	LIFM 3 opened, defined-volume of 50 µL in chamber 4 is transferred into chamber 7 (Figure 4F).
5	-	-	LIFM 4 closed (Figure 4G).
6	30	5	LIFM 5 opened, blue solution in chamber 3 is transferred into chamber 4 (Figure 4H).
7	+9 ~ -9	12	Mixing, Color changes from yellow to blue (Figure 4H).
8	30	5	LIFM 6 opened, defined-volume 50 µL in chamber 4 is transferred into chamber 5 (Figure 4I).
9	30	5	LIFM 7 opened, defined-volume 50 µL in chamber 4 is transferred into chamber 6 (Figure 4J).

A novel microvalve actuated by laser irradiation is developed. The volume of the LIFM is less than 100 nL and the multiple microvalves could be operated by single laser diode (808nm, 1.5W). The microvalves are leak-free up to  $403.0 \pm 7.6$  kPa and the response time to operate both normally closed and normally opened microvalves are less than 0.5 sec. We also implemented the LIFM on centrifugal microfluidic platforms and demonstrated various microfluidic functions such as valving, metering, mixing, and distribution.

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