Optofluidic microvalve-on-a-chip with a surface plasmon-enhanced fiber optic microheater

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We present an optofluidic microvalve utilizing an embedded, surface plasmon-enhanced fiber optic microheater. The fiber optic microheater is formed by depositing a titanium thin film on the roughened end-face of a silica optical fiber that serves as a waveguide to deliver laser light to the titanium film. The nanoscale roughness at the titanium-silica interface enables strong light absorption enhancement in the titanium film through excitation of localized surface plasmons as well as facilitates bubble nucleation. Our experimental results show that due to the unique design of the fiber optic heater, the threshold laser power required to generate a bubble is greatly reduced and the bubble growth rate is significantly increased. By using the microvalve, stable vapor bubble generation in the microchannel is demonstrated, which does not require complex optical focusing and alignment. The generated vapor bubble is shown to successfully block a liquid flow channel with a size of $125\ \mu\text{m} \times 125\ \mu\text{m}$ and a flow rate of $\sim 10\ \mu\text{l}/\text{min}$ at $\sim 120\ \text{mW}$ laser power.
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I. INTRODUCTION

Microscale vapor bubbles have received increasing attention in microfluidic applications. These vapor bubbles can be generated in a controlled manner at a designated location with an adjustable shape, size, and duration.^{1–4} The controlled vapor bubbles have great potential to be used as micro-actuators, which have no mechanical moving parts, thus allow for simple actuator structure design and easy fabrication process. The vapor bubble-based micro-actuators have been widely utilized as microvalves,^{5,6} micropumps,^{7–9} micromixers,^{10,11} bio-particle manipulators,¹² and droplet generators.¹³

In microfluidic systems, vapor bubbles are usually created by utilizing resistive or laser-based microheaters. Resistive heating makes use of surface-micromachined metal or polysilicon film resistors to enable a localized nucleate boiling of liquids for generating thermal bubbles on the surface of the microheaters.^{14–16} As a non-electrical approach, laser-induced bubble generation creates vapor bubbles by irradiation of liquids or light absorption in liquids. Lasers used for generating bubble can be either pulsed lasers or continuous-wave (CW) lasers. By focusing a high intensity pulsed-laser beam inside liquids, cavitation bubbles can be created through thermal confinement (i.e., energy deposition from heating) and/or stress confinement (i.e., pressure reduction induced by thermoelastic acoustic waves).¹⁷ In this case, liquids are often dyed to increase the light absorption and thus lower the threshold laser power required for cavitations.⁸ However, dyes are potential contaminants to sample fluids. Therefore, an additional structure is usually required to separate sample fluids from dyes.¹⁸ On the other hand, low-power CW laser irradiation of light absorbing mediums can be used to induce local heating of the mediums. The laser beam, in contact with liquids, generates thermal bubbles on the heated surface of the medium by localized nucleate boiling. Metal films or nanoparticles are

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typically used as mediums in microfluidic chips for converting optical energy to thermal energy.^{3,19} However, the large optical power loss due to the light transmission through the optical path to the light absorbing medium is inevitable. Furthermore, the unwanted exposure of laser to the sample fluids poses another potential limitation, which can be especially harmful for biological samples. To address these limitations, metal- or nanoparticle-coated optical fibers have been studied for creating thermal bubbles. For example, Taylor *et al.* coated a platinum thin film on the fiber end-face.²⁰ Xing *et al.* deposited graphene oxide nanosheets on the fiber side-wall,²¹ and Dominguez *et al.* coated carbon nanotubes and silver nanoparticle on the fiber end-face.²² In particular, metal films, compared to nanoparticles, can be deposited on optical fibers with good uniformity and controllable thickness due to the well-established micro-electro-mechanical systems (MEMS) fabrication techniques. However, the previously reported metal-coated optical fiber microheater employs a microscale tapered fiber tip that requires an elaborated etching process to fabricate.²⁰ It should also be noted that all the previous studies on optical fiber based bubble generation were carried out in open environment and a fully functional optical fiber based optofluidic microvalve has never been demonstrated.

Here, we report a CW laser-induced vapor bubble microvalve-on-a-chip that makes use of a surface plasma-enhanced fiber optic microheater (Fig. 1). The fiber optic microheater is formed by depositing a titanium thin film on the roughened end-face of a silica optical fiber (Fig. 1(a)), which is embedded into a microfluidic chip to achieve an optofluidic microvalve-on-a-chip system (Fig. 1(b)). Titanium is chosen as the light absorbing medium for vapor bubble generation due to several reasons. First, titanium has a high optical absorption (44.3% in an 808 nm wavelength) and is an excellent plasmonic material for excitation of localized surface plasmons (LSPs).²³ Second, titanium has good adhesion to the silica fiber and a high melting point (1668 °C). In addition, titanium is a biocompatible and inexpensive material. The unique design of the fiber optic microheater not only enables the excitation of LSPs for enhancing light absorption in the titanium film but also helps provide improved nucleation boiling conditions for vapor bubble generation. Owing to the rough titanium-silica interface, bubble generation can be achieved with a higher growth rate by heating of the titanium film at a lower threshold laser power. The generated bubble grows into a vapor plug, realizing a valve to effectively block the flow in the microchannel (Fig. 1(c)). The optical fiber based optofluidic microvalve renders an all optical fiber system without any electrical interconnections and free-space optics. By using the optical fiber as a waveguide, although the position of bubble generation is limited

FIG. 1. Schematic of the optofluidic vapor bubble based microvalve-on-a-chip with a fiber optic microheater: (a) cross sectional view of the LSPs-enhanced fiber optic microheater, (b) optofluidic chip with an embedded fiber optic microheater, and (c) vapor bubble generation inside the microchannel and a vapor plug blocking the flow in the microchannel.

to the end-face of embedded optical fibers, the transmission loss of laser power can be minimized and complex optical focusing and alignment systems can be obviated. More importantly, since the fiber guided laser power is mostly absorbed in the titanium film, the laser irradiation to the sample fluids will be minimal, which makes the device a safe choice for the manipulation of biological samples. In addition, unlike other microfluidic devices demonstrated for laser bubble generation,^{3,19} there is no need for metal film deposition on substrates for chip fabrication, which allows for flexible chip design and simple fabrication with only polymer processes.

The rest of the paper is organized as follows. We first describe the fabrication of the LSP-enhanced fiber optic microheater, the integration of the microheater into a microfluidic chip, and the experimental setup for characterizing the bubble generation and the bubble-based microvalves. Then, we show the experimental results and numerical simulations for optimizing the titanium film thickness and surface roughness of the fiber optic microheater. We further characterize the bubble generation inside a microchannel and demonstrate a CW laser-induced microvalve. Finally, concluding remarks are provided.

II. MATERIALS AND METHODS

A. Fabrication of the fiber optic microheater

Multimode silica optical fibers (ThorLabs, Inc) were utilized for the transmission of a laser beam. The diameters of the core and cladding of the optical fibers were 105 μm and 125 μm , respectively. The end-face of the optical fibers was first grinded by lapping films (AngstromLap[®], fiber optic center, Inc) to increase the surface roughness. The arithmetic roughness (R_a) of the fiber end-face was measured by a topology measurement system (TMS-1200, Polytec, Inc). The grinded end-faces of the optical fibers were then cleaned with isopropyl alcohol (IPA) and distilled water using an ultrasonic cleaner followed by nitrogen dry. Finally, a titanium film was deposited on the fiber end-faces by magnetron sputtering. Fig. 2(a) shows the end-face of the fabricated fiber optic microheater.

B. Integration of the fiber optic microheater into a microfluidic chip

Microfluidic chips were fabricated by soft lithography²⁴ as follows. First, a glass substrate was cleaned by IPA and distilled water followed by nitrogen dry. The substrate was then coated with a 125 μm thick ultraviolet (UV) curable polymer layer (OrmoComp[®], micro resist technology GmbH), which was further patterned into a T-junction microchannel by photolithography to form a mold. Next, polydimethylsiloxane (PDMS) (Sylgard 184, Dow Corning) mixed with a 10:1 base-to-curing agent ratio was deposited on the fabricated mold and cured at 60 $^{\circ}\text{C}$ for 2 h. After releasing the PDMS microchannel from the mold, it was bonded to a partially cured PDMS bottom substrate and post-cured at 60 $^{\circ}\text{C}$ for 2 h.²⁵ Fig. 2(b) shows the fabricated

FIG. 2. (a) SEM image of the end-face of the fabricated fiber optic microheater. The inset shows a magnified image of the end-face. Scale bar represents 10 μm . Optical microscopic images of the T-junction microchannel (b) before embedding the optical fiber and (c) after embedding the optical fiber. The scale bar represents 100 μm .

T-junction microchannel. The fabricated fiber optic microheater was inserted into the T-junction microchannel with the fiber end-face well aligned with the junction. The gap between the fiber and microchannel was sealed by using UV curable polymer to prevent the leakage of liquid. Due to the surface tension induced capillary force, the gap was completely filled with the UV polymer,²⁶ and a capillary pressure barrier at the end of the vertical microchannel successfully stopped the polymer flow at the edge of the fiber end-face,^{27,28} as shown in Fig. 2(c). Finally, the UV polymer in the gap was cured with UV light exposure.

C. Experimental setup

A CW 808 nm laser diode (F6-808, Coherent) with a maximum output power of 1 W was utilized as a laser source. For the bubble generation in an open environment, the fiber optic microheater was submerged in a petri-dish filled with distilled water and more than 1 mm of free space was secured around the fiber end-face. For the microvalve experiments, syringes were connected to a motorized linear actuator (VP-25X, Newport) to generate a constant, controllable flow for a microchannel. The liquid used in all the experiments was room-temperature distilled water. To characterize the growth of the vapor bubbles, a charge-coupled device (CCD) camera connected to an inverted microscope was used to record the bubbles at a speed of 20 frames per second. The radius of the bubbles was obtained by analyzing the acquired video by utilizing MATLAB image processing toolbox (MathWorks®).

III. RESULTS AND DISCUSSION

A. Optimization of the titanium film thickness and surface roughness

The rough titanium surface used in the fiber optic microheater enables two enhancement mechanisms for vapor bubble generation. First, this rough surface can help enhance nucleation boiling for bubble formation. In the localized nucleation boiling, vapor bubbles or nuclei are usually generated on a superheated metal surface. In a poorly wetted surface, gas-filled cavities form the typical nuclei. Therefore, on rough surfaces that provide a larger size and shape range of cavities, vapor bubbles can be generated at a lower temperature.^{29,30} Second, the rough titanium film enables the excitation of LSPs for enhancing light absorption, which will help improve the bubble growth rate. Due to the heat flux from the superheated metal surface, the evaporation of liquids surrounding the bubble will happen, and as a result, the generated bubble will grow. Here, metal heating is induced by the light absorption in the rough titanium metal film. Previous studies have shown that the nanoscale surface roughness and thickness of the metal film can greatly affect the light absorption and the excitation of LSPs on roughened metal surfaces can enable strong light absorption enhancement.^{31–34}

Therefore, the effects of the thickness and surface roughness of the titanium film used in the fiber optic microheater on the formation and growth of vapor bubbles should be characterized. We first conducted open environment experiments to investigate the effects of the film thickness in terms of the threshold laser power and bubble growth rate (see Fig. 3). Fig. 3(a) shows the history of vapor bubble generation in the open environment. A temporary vapor bubble was first formed on the superheated fiber end-face and collapsed in a short period of time (~ 0.1 s). Then, a stable vapor bubble started to grow and kept adhering to the fiber end-face during the bubble growth, which is due to the strong thermocapillary effect in the vicinity of the superheated fiber end-face.³⁵ The threshold laser power was measured as the minimum power required for instantaneous bubble formation with a start-time of less than 1 s. Here, the start-up time is defined as the time taken for a bubble to emerge starting from the time when the laser was turned on. The bubble growth rate was determined by analyzing the changes of the bubble radius during the first 4 s after the stable bubble formation at a constant input laser power larger than the threshold power. Since the bubble growth process is governed by the heat dissipation, the bubble radius, r , follows an asymptotic curve; that is, $r \propto t^{1/2}$,¹⁵ where t is the time from ~ 0.1 s. Here, the measured bubble radii at different times were curve-fitted into an

FIG. 3. (a) Bubble radius as a function of time obtained in open environment experiments (Film thickness: 200 nm). The insets are optical microscopic images showing the bubble generation at 350 mW laser power. The scale bar represents 100 μm . (b) Threshold laser power and bubble growth factor as a function of the thickness of the titanium film. The results were obtained with cleaved fibers without end-face grinding. Five samples were tested for each titanium thickness. The bubble growth factor was measured at 350 mW laser power, which is above the threshold power for all four titanium thicknesses.

equation $r = R + C \cdot t^{1/2}$, where R is the initial bubble radius and C is the bubble growth factor. The bubble growth factor determines the actual bubble growth rate.³⁰

To investigate the influence of the titanium film thickness on the threshold laser power and bubble growth factor, four different thicknesses of the titanium films were deposited on the fiber end-faces without surface grinding: 50 nm, 100 nm, 200 nm, and 300 nm. It can be observed from Fig. 3 that there is a clear difference in both the threshold laser power and the bubble growth factor between the 50-nm thick films and the films of larger thicknesses. For the 50-nm thick film, the threshold laser power was higher (~ 330 mW) and the bubble growth factor ($\sim 30 \mu\text{m}/\text{s}^{0.5}$) was lower than the films of larger thicknesses. This is due to the stronger penetration of the incident light through the 50-nm thick film³, which results in a lower light power absorption for bubble generation. The films with other thicknesses were found to have similar threshold laser power (~ 290 mW) and bubble growth factor ($\sim 31.5 \mu\text{m}/\text{s}^{0.5}$). For the rest of the experiments, we utilized 200-nm thick titanium films to prevent the light penetration.

To characterize the effects of the surface roughness, we first performed numerical simulations based on Rigorous Coupled-Wave Analysis (RCWA) to investigate the light absorptance in the rough titanium-silica interface. The inset of Fig. 4(a) shows the schematic of the titanium-silica interface model used for the RCWA simulations, in which periodic, square shaped nanostructures were used to describe the roughened fiber end-face coated with a titanium film. The roughness was characterized by using the pattern size d , which is the depth and periodicity of the nanostructures. An 808 nm plane wave was used as the propagation light

FIG. 4. (a) Simulated light absorptance in the titanium-silica interface as a function of the pattern size d . The inset shows the schematic of the titanium-silica interface model. (b) Electric field obtained at the titanium-silica interface for $d = 520$ nm. (c) Threshold laser power for the bubble generation and bubble growth factor as a function of R_a . The fiber end-faces were coated with 200-nm thick titanium films. The bubble growth factor was measured at an input laser power of 300 mW, which is above the threshold power for the four different R_a values. Five samples were tested for each R_a value.

through the silica fiber with a 0° incident angle. The simulation result of the light absorbance as a function of d is provided in Fig. 4(a), which shows a peak absorbance when d is around 520 nm. We further obtained the corresponding light electric field distribution at the titanium-silica interface for $d = 520$ nm, as shown in Fig. 4(b), which exhibits a strong field enhancement in the vicinity of the edges of the nanostructures. The strong field enhancement is believed to be due to the excitation of LSPs on the edges of the nanostructures patterned on a titanium surface.³³ We then investigated the effects of the surface roughness on the threshold laser power and bubble growth rate in open environment experiments. Fiber end-faces with four different roughness R_a values were prepared, including the end-face without grinding and the grinded end-faces by the lapping films with 5 μm , 30 μm , and 60 μm grits. The R_a values of these end-faces were measured to be 61.52 nm, 340.63 nm, 526.18 nm, and 672.89 nm, respectively. Fig. 4(c) shows the bubble growth factor and threshold power as a function of the surface roughness. The bubble growth factor was found to first increase with increasing surface roughness with the peak growth factor obtained at a roughness R_a of 526.18 nm and then decrease. Compared with the bubble growth factor obtained with fibers without end-face grinding (i.e., $R_a = 61.52$ nm), the bubble growth factor at $R_a = 526.18$ nm was increased by 32.6%. Such an increase is believed to result from the increased heat flux from the titanium film, which attributes to the LSP-enhanced light absorption. It is evident that the peak growth factor agrees well with the peak absorbance obtained in the simulations (see Fig. 4(a)). On the other hand, the threshold laser power was found to decrease with increasing surface roughness. In particular, the threshold laser power at $R_a = 526.18$ nm was higher than that at $R_a = 672.89$ nm, even though a higher heat flux was generated at $R_a = 526.18$ nm, which rendered a higher growth factor. This indicates that the formation of bubbles is largely governed by the size and shape range of nucleation cavities on the titanium film, while the bubble growth rate is mainly determined by the heat flux generated from light absorption. For this reason, the lowest threshold power was obtained with the films of the highest roughness (i.e., $R_a = 672.89$ nm). However, the threshold laser power at $R_a = 526.18$ nm was still significantly lower (35.7% lower) than that at $R_a = 61.52$ nm. Since a fast bubble growth is more desirable for quickly forming a vapor plug to block the microchannel, for the following experiments in a microchannel, we chose to utilize the fibers with end-face roughness R_a of 526.18 nm for the fiber optic microheaters.

B. Vapor bubble generation inside a microchannel

We investigated the in-channel bubble generation utilizing a $125\ \mu\text{m} \times 125\ \mu\text{m}$ PDMS microchannel. The threshold laser power for the instantaneous bubble formation inside the microchannel was found to be 40 mW, which was 78% lower than that in the open environment. Such a decrease in the threshold laser power is due to the smaller volume of liquids in the vicinity of the fiber end-face and the slower heat dissipation through the PDMS microchannel, which makes it easier to reach superheats with less optical power.¹⁹ In the experiments, it was found that bubbles could even be generated with less than 40 mW laser power. However, as can be seen from Fig. 5, at a lower power (e.g., 30 mW), the required start-up time for the bubble formation increased dramatically and the bubble formation became less predictable as there was a large variation in start-up times. The insets of Fig. 5 show the in-channel bubble generation at 90 mW of laser power. A bubble was formed on the fiber end-face and grew into a vapor plug, which entirely blocked the microchannel. The vapor plug kept growing toward both ends of the microchannel due to the evaporation of liquids. After the laser was turned off, the evaporation stopped and the vapors in the plug started to condense into liquids, which resulted in the contraction of the vapor plug. However, the bubble was not completely collapsed (see insets of Fig. 5 at 30 s) due to the residual gas, which came from the pre-dissolved gas in liquids and the pre-trapped gas in the walls of the microchannel. It is expected that when degassed water is used, the residual gas plug can be eliminated.

C. Vapor bubble-based optofluidic microvalves

The vapor bubble generated by using the fiber optic microheater can be utilized as a microvalve to control the liquid flow in microchannels. To demonstrate such bubble-based optofluidic

FIG. 5. Start-up time for in-channel bubble generation as a function of the laser power. The insets are optical microscopic images showing the bubble generation inside a microchannel of $125\ \mu\text{m} \times 125\ \mu\text{m}$. The laser power was 90 mW, and it was turned off after 20 s. The scale bar represents $100\ \mu\text{m}$.

microvalves, we fabricated a Y-junction microchannel, as shown in the inset (1) of Fig. 6(a). The fiber optic microheater was embedded into the microfluidic chip and its end-face was exposed to the sidewall of the lower inlet microchannel. The liquid flow in the upper inlet microchannel was colored by food dyes to visualize the on/off status of the bubble-based microvalve. The inset (2) of Fig. 6(a) shows the bubble generation in the microchannel with a liquid flow rate of $0.5\ \mu\text{l}/\text{min}$. The generated bubble grew into a vapor plug, and the right

FIG. 6. (a) Threshold laser power required to block the liquid flow in the $125\ \mu\text{m} \times 125\ \mu\text{m}$ microchannel as a function of flow rate. (1) Schematic of the Y-junction microchannel embedding the fiber optic microheater. (2) Optical microscopic images of the bubble generation at a flow rate of $0.5\ \mu\text{l}/\text{min}$ inside the $125\ \mu\text{m} \times 125\ \mu\text{m}$ microchannel with a laser power of 40 mW. (b) and (c) Optical microscopic images of the Y-junction during laser OFF/ON. The scale bars represent $100\ \mu\text{m}$. The flow direction in all images is from right to left.

boundary of the vapor plug kept staying near the edge of the fiber end-face such that it blocked the liquid flow, while the left boundary of the vapor plug moved toward the left side of the microchannel due to the pressure gradient. As shown in the Figs. 6(b) and 6(c), at the Y junction of the microchannel, the bubble-based microvalve successfully blocked the liquid flow. When the laser was turned off, the residual vapor plug was flushed away from the microchannel by the liquid flow. In the experiments, it was found that there was a threshold laser power at which the generated vapor bubble could grow into the vapor plug and successfully blocked the microchannel. When the laser power was lower than the threshold, the evaporation of liquids was not fast enough, and as a result, the generated vapor bubble could not completely fill the microchannel. The threshold laser power was measured to be from 40 mW to 120 mW for the flow rate ranging from 0.5 $\mu\text{l}/\text{min}$ to 10 $\mu\text{l}/\text{min}$, as shown in Fig. 6(a), which was found to be linearly proportional to the flow rate. Compared with the previously reported bubble-based microvalve that makes use of a free-space laser¹⁹ (with 250 mW laser power for blocking a 1.7 $\mu\text{l}/\text{min}$ liquid flow in a 500 $\mu\text{m} \times 40 \mu\text{m}$ microchannel), the microvalve based on an optical fiber heater allows for blocking channel with a much higher flow rate at a significant lower laser power.

IV. CONCLUDING REMARKS

We developed an optofluidic microvalve for flow control in a microchannel, which makes use of an LSP-enhanced fiber optic microheater for vapor bubble generation. The fiber optic microheater was formed by depositing a titanium thin film on the roughened end-face of a silica optical fiber, which was embedded into a microfluidic chip by using capillary force-assisted UV polymer sealing. The rough titanium-silica interface can help excite LSPs, rendering a strong light absorption enhancement in the titanium film for improving the bubble growth rate. Furthermore, due to the increased size and shape range of air cavities on the rough titanium film, the bubble nucleation can be easily facilitated, which helps lower the laser power required for bubble formation. With this unique fiber optic microheater, stable vapor bubble generation in an open environment was demonstrated with 35.7% decrease in the threshold laser power and 32.6% increase in the bubble growth factor compared to that obtained with fibers without surface roughening. By using the fiber optic microheater in a microfluidic channel, a vapor bubble-based microvalve was successfully demonstrated with a flow rate up to $\sim 10 \mu\text{l}/\text{min}$ at ~ 120 mW laser power. It should be noted that although only a single microvalve was demonstrated, owing to the multiplexibility of optical fiber based systems, multiple fiber optic heaters can be embedded on an optofluidic chip to achieve a multiplexed microvalve system for controlling multiple microchannels. In addition, the fiber based optofluidic microvalve can help eliminate the optical focusing and alignment systems and avoid the unwanted laser exposure to sample fluids and human. Therefore, it is expected that the fiber-based optofluidic microvalves will have great potential to be used for manipulation of biological samples in many applications.

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