Real-time and reversible light-actuated microfluidic channel squeezing in dye-doped PDMS

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Angelo Angelini[a]*, Ubirajara Agero[b], Federico Ferrarese Lupi[a], Matteo Fretto[a], Candido Fabrizio Pirri[c], Francesca Frascella[c*]

[a]*Nanoscience and Materials Division, Istituto Nazionale di Ricerca Metrologica, Strada delle Cacce 91, 10135 Torino, Italy*
[b]*Departamento de Física, Universidade Federal de Minas Gerais- 31270-901 Belo Horizonte, MG, Brazil*
[c]*Department of Applied Science and Technology, Politecnico di Torino, Corso Duca degli Abruzzi 24, 10129 Torino, Italy*

[*]Corresponding authors*: angelo.angelini@polito.it, francesca.frascella@polito.it*

Abstract

Azobenzene chromophore is used as functional dye for the development of smart microfluidic devices. A single layer microfluidic channel is produced, exploiting the potential of a dye doped PDMS formulation. The key advantage of this approach is the possibility to control the fluid flow, by means of a simple light stimulus. Furthermore, the deformation can be controlled in time, space and intensity, giving rise to several degrees of freedom in the actuation of the channel squeezing. Future perspective will be the implementation of the microfluidic platform with structured light, to have the possibility to control the flow in a parallel and reversible manner in several points, modifying the pattern in real time.

Keywords: microfluidic, azo-dyes, stimuli responsive material.

Introduction

Microfluidic devices have been extensively developed and characterized in the last decade, as they offer a valuable tool, especially in the medical and biological field [1-2]. Commercially available devices allow to perform point-of-care diagnosis of different fluids that can be easily analyzed in few minutes [3-4].

Despite the high level of integration and miniaturization achieved in microfluidic systems, some of the main components, such as valves or pumps, still require external macroscopic devices for the actuation and complex fabrication processes [5-7]. In this work we focus on a key functionality in the control of microfluidic flows: the real-time and reversible channel squeezing. Currently the main strategies adopted to control the fluid flow are based on mechanical deformation of the microfluidic channel actuated by an external stimulus, such as pressure variation in a control channel (Quake's valves) [8-15] or a solid object pushing the channel walls [16].

The above-mentioned strategies typically involve multiple steps of fabrication including precise alignment and bonding of the control layers above the functional layer, and an external pump for controlling the flow in the control channels. A single PDMS layer microvalve has been proposed by Sundararajan et al. in 2005 [17], where different devices working with a deformable membrane have been demonstrated.

Despite the easiness of fabrication, the valves obtained offer lower control over the fluid flow, and still require an external circuit to change the pressure in the control channels. Other mechanism to actively control the flow in microfluidic channels include the fabrication of membranes closing channels, that degrade when illuminated [18], with the drawback given by the irreversibility of the process.

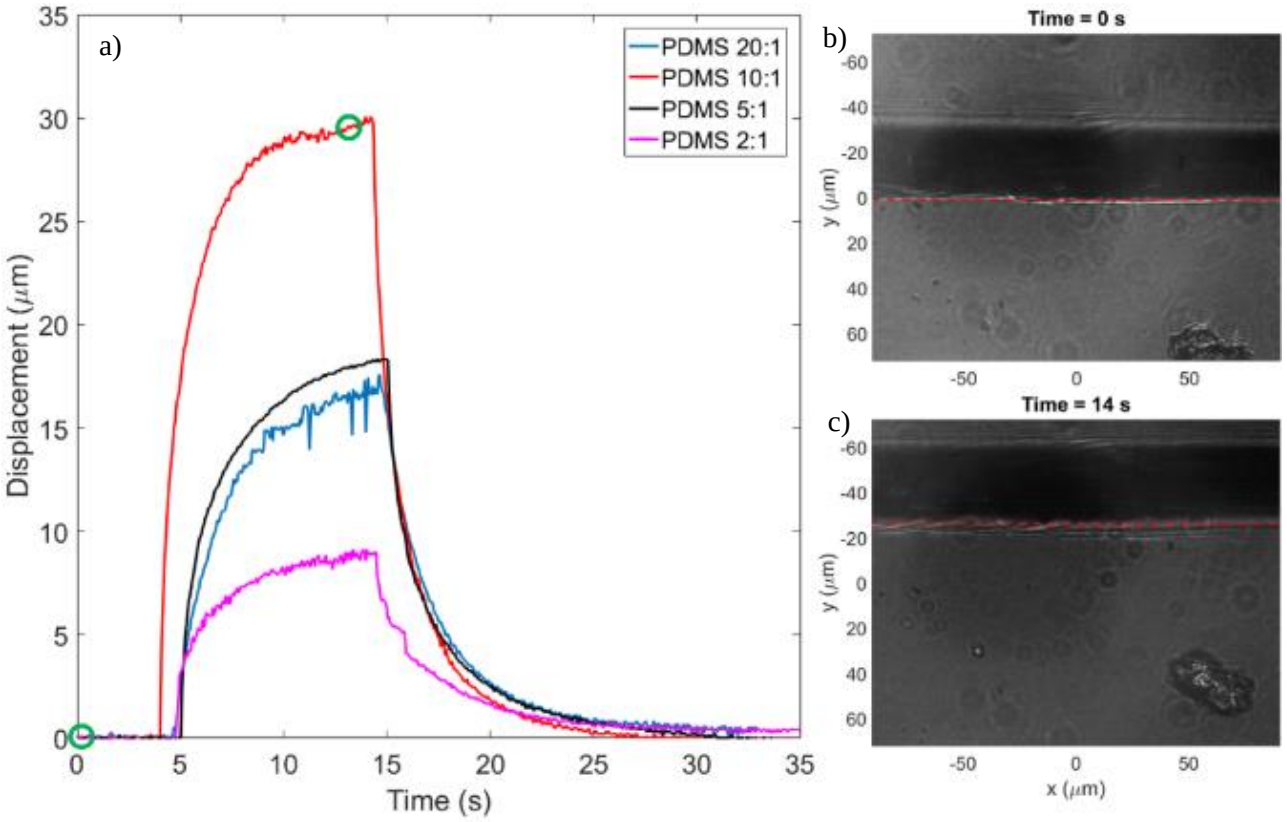


Figure 1 a) Maximum displacement (m) of 4 different PDMS formulation due to light-induced stimulus. b) Real time imaging of PDMS 20:1 membrane displacement from its initial position recorded in time during the light stimulus. The images correspond in time to the point highlighted by the green circle in graph (a).

Herein, we propose a single layer microfluidic channel, where the fluid flow is controlled by a light stimulus. The working principle is the reversible deformation of the channels' walls induced by light absorption, due to the presence of an azo dye [19-20]. When light is focused, the stimulus can be delivered in whichever point of the microfluidic system, providing space and time degree of freedom in the actuation of the channel squeezing.

An intrinsic advantage of this strategy with respect to the above-mentioned ones, relies on the possibility of delivering the light stimulus in any point of the microfluidic device, that can be changed in real-time.

Moreover, by tuning the light intensity the magnitude of the deformation can be deeply controlled, providing an additional degree of freedom.

Experimental Section

2.1 PDMS-azo doped membrane

The elastomeric compound is obtained by mixing the polydimethylsiloxane (PDMS, from Dow Corning, Sylgard 184) at different prepolymer and curing agent ratio, then adding a proper amount of Dispersed Red 1 methacrylate material (DR1M – Sigma Aldrich) solution in toluene (≥99.8%, Sigma-Aldrich), as already described in a previous paper [21].

To test the best PDMS formulation with respect to the displacement behavior, 4 different elastomer solutions were prepared. In particular the PDMS samples were obtained by mixing the prepolymer solution and the curing agent in a ratio ranging from 20:1 to 2:1, to have an increasing tough behavior. To these mixtures a solution a DR1M at 2% wt in toluene was added. The resulting blend solution was dropped on a glass substrate, to obtain a thin and controlled membrane. Then the sample was thermally cured by placing it in an oven at 60 ° C for 2 h.

Finally, a transparent and flexible PDMS-azo doped membrane (thickness around 100 μm) was obtained [22-23]. The reddish

color is due to the DR1M absorption band, which enables the sample to be light stimulated by a green laser.

By illuminating the sample with a collimated beam (beam waist about 1 mm), the absorbed light causes an expansion in all directions. By imaging the membrane border, we can measure the linear expansion of the material. In Figure 1a, we report on the dynamic of expansion of the fabricated membranes. We observe that the maximum expansion is obtained when the prepolymer-curing agent ratio is 10:1, and the expansion / relaxation occurs in a time scale of seconds. Figure 1 b and c show the images employed for the measurement corresponding to the green circles in Figure 1 a.

The experimental results suggest that there should be a trade-off between stiffness and motility of the PDMS-azo compound. An increasing stiffness of the matrix allows for a more efficient transfer a local expansion

2.2 PDMS-azo doped microfluidic chip

The microfluidic chip is fabricated by soft lithography. SU8 3010 negative photoresist from MicroChem® is spin-coated on a Si wafer at 1000 rpm per 60 s. After soft bake at 95° for 5 mins on a hot plate the resist is exposed to UV light in a Heidelberg PG101 Laser Writer. A Y-shaped master is obtained after exposing the photoresist. We used the following writing parameters: beam waist 2 m, power 63 mW. After a post-bake at 110° for 2 mins, the sample was developed in SU8-developer for 90 s and then rinsed in Isopropanol for 30 s.

A 10:1 ratio in a weight mixture of a PDMS prepolymer solution and curing agent was deeply mixed and degassed for 30 min, then the mixture free of bubbles, was poured into the master and cured in an oven at 60°C for 4hours. After this, the PDMS was peeled off from the mold and inlet and outlet were fabricated by means of a biopsy punch. The PDMS Y-shaped microfluidic was irreversibly bonded to a glass substrate through an O2 plasma activation treatment. Before the treatment, all the components were cleaned in acetone with the aid of an ultrasonic bath for 5

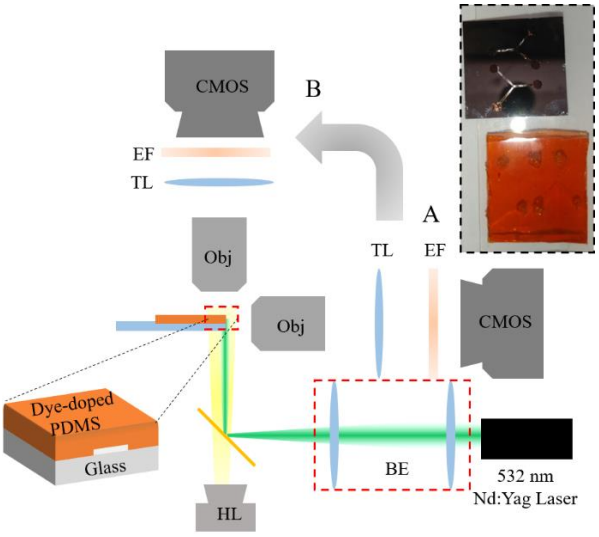


Figure 2 Schematic view of the experimental setup. The imaging arm can be switched from configuration A (transversal position for cross-sectional imaging) to configuration B (collinear to the white light). In the inset, a picture of the SU8 on silicon master and the PDMS microfluidic chip.

min, then rinsed with dry nitrogen and further cleaned in Isopropanol for 5 min. Then, an optimized oxygen plasma activation process was performed for 40s (RF power 50W, O_2 flow= 16.5 sccm), leading to an enhanced PDMS contact angle value (more hydrophilic) and increased surface energy. The PDMS was bonded to the glass substrates immediately after oxygen plasma activation. Careful attention was placed to air bubbles trapped between PDMS and glass, so to obtain a uniform and irreversible bonding in the interconnection area.

The inlet of the microfluidic chip is connected to an external syringe that can be actuated by a motorized stage where flow speed can be programmed (Syringe Pump Harvard Apparatus 11 Plus). The outlets are connected to a reservoir through drainpipes.

2.3 Experimental setups

The microfluidic device has been characterized under two different setups obtained by customizing an optical microscope (Fig. 2). The configuration is like wide field white light microscope equipped with a laser light source (Nd:Yag doubled CW laser, wavelength: 532 nm, max power 200 mW). The imaging arm can be either transversal with respect to the

illumination arm (configuration A) or collinear with the illumination path (configuration B).

(Obj) and a tube lens and finally recorded by a CMOS camera (Thorlabs DCC14p35M).

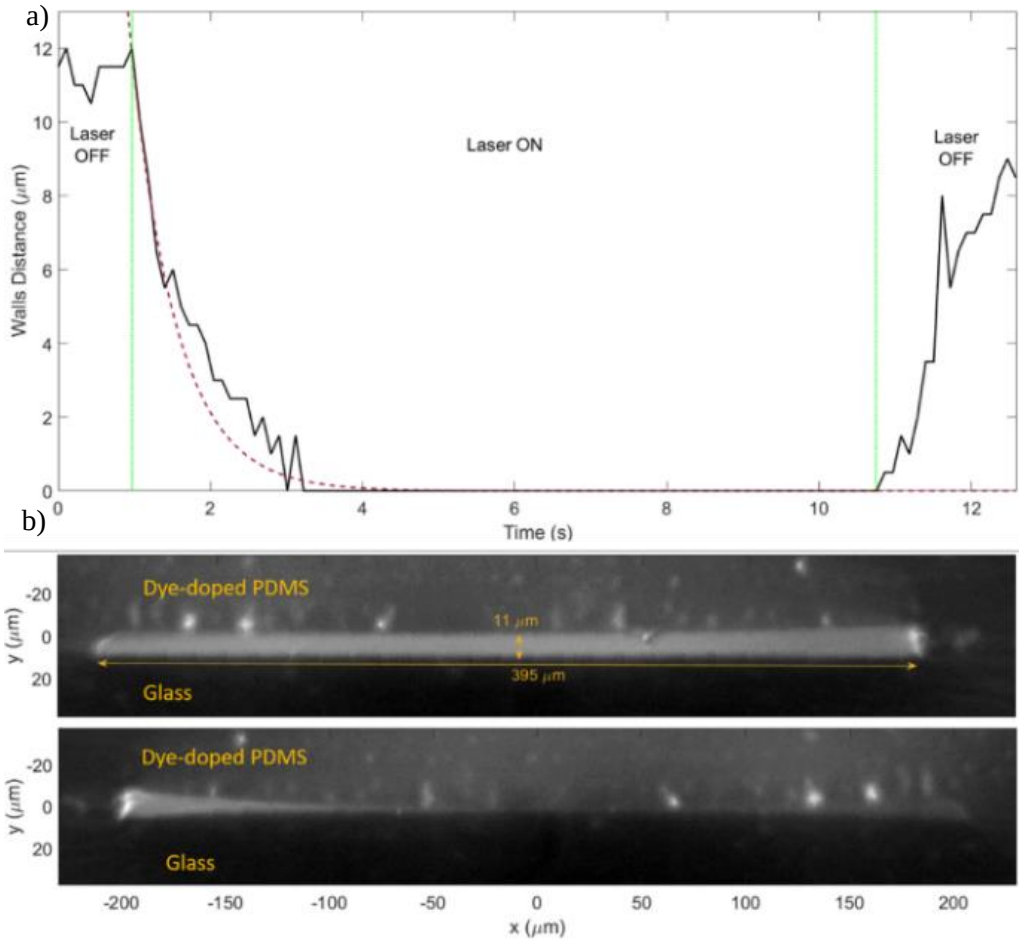


Figure 3 a) Maximum channel height displacement (μm) of the dye-doped PDMS single layer microfluidic channel, due to light-induced stimulus. b) Real time cross section of the dye doped PDMS channel from its initial full opening position, to the maximum expansion one (closed).

When employed in configuration B, the imaging arm is rotated by 90 degrees to observe the cross-section of the sample. In the present work, Configuration A is employed to observe the microfluidic channel cross section during the illumination, in order to directly observe the squeezing of the channel, whilst configuration B is employed to evaluate the effect of the squeezing on a liquid flowing within the channel. In detail, a halogen lamp (HL) illuminates the sample, while the laser beam, weakly focused by a beam expander (BE) stage, is deflected by a dichroic mirror.

The laser spot, characterized by a gaussian intensity distribution, shows a beam waist of about 600 μm, that is slightly larger than the channel width. The imaging of the sample is performed by a collection objective

In configuration A, the objective employed is a 20x Mitutoyo plan apo infinity corrected Long Working Distance, 0.42 NA. In configuration B, to follow the dynamic of microspheres inside the channel, the objective is a 100x Mitutoyo plan apo infinity corrected Long working distance Objective, 0.70 NA instead.

Results and Discussion

The first step was to demonstrate that the expansion of dye doped PDMS membrane can be effectively used to close a void microfluidic channel. The microfluidic single layer channel is cut perpendicular to the surface, so to have a channel whose cross-section was exposed to the imaging system (configuration A). The laser beam was positioned to illuminate the cut interface (see Fig. 2 configuration A). In Figure

3 the distance between the top wall and the bottom of the channel is recorded versus time.

The starting channel height is 11 μm, then when the laser is switched on at its maximum power, the upper wall approaches the bottom of the channel (closed configuration Figure 3) within a transient time, that can be estimated to be about 1 second. The illumination beam keeps the channel closed until the laser is switched off and the PDMS relaxes to the initial position, with a dynamic that like the expansion one. The full process can be observed in *Movie 1*.

Therefore, having established the possibility to close the channel exploiting a light stimulus, we decided to test the chance to use the channel squeezing to shift floating objects within the microfluidic circuit. To demonstrate an active and reversible control, we filled the microfluidic chip with silica microspheres (mean diameter 2 μm) dispersed in water.

The optical experimental set up, switched to the configuration B, allows to record the position of single microspheres within the channel. We recorded the position versus time at different light intensities, from 60% of the maximum power to 100%, with steps of 10%. Figure 4a reports on the maximum displacement achieved by a microsphere from its own initial position versus light density power. As can be observed, the maximum displacement gradually increases from few microns to 60 μm. The error bar reported in the graph accounts for the Brownian motion and the error in the positioning of the object. To show the reversibility of the process, we selected a laser power density of 0.5 μW/μm (red circle in Fig. 4a) and recorded the full motion of a microsphere versus time (see *Movie 2*). After about 4 seconds, the light is switched on and the squeezing of the channel pushes the microsphere about 22 μm far from its initial position. The position is held until the light is switched off, when the relaxation of the elastomer pulls the microsphere back to its initial position.

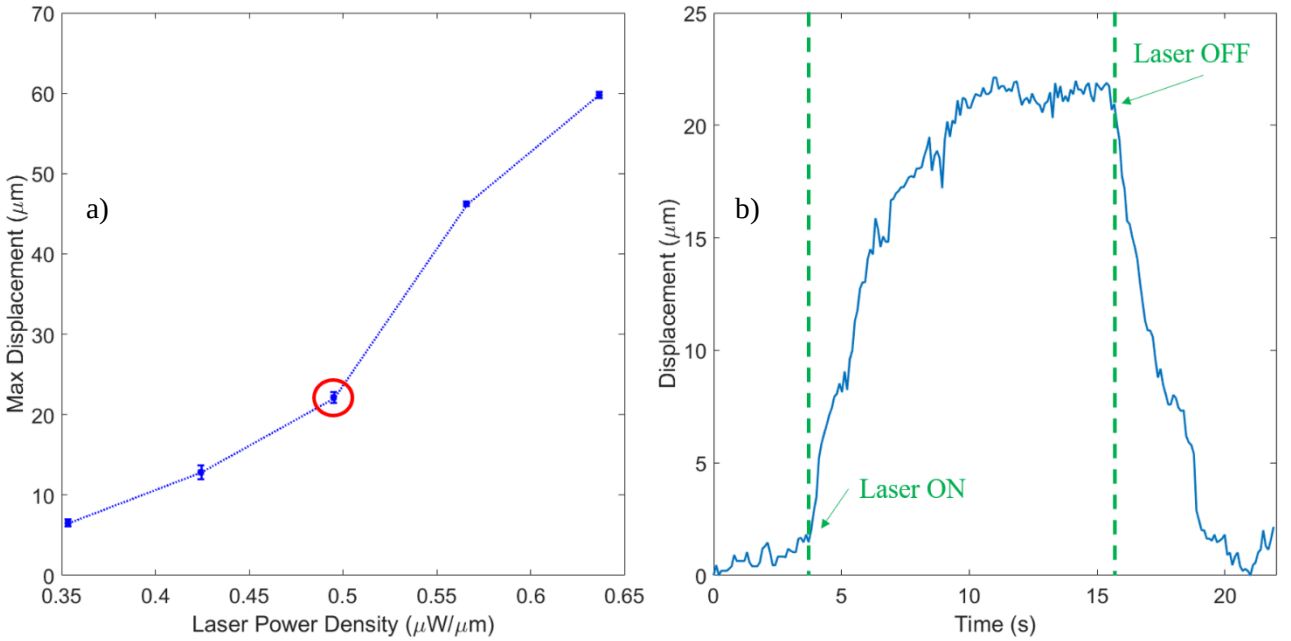
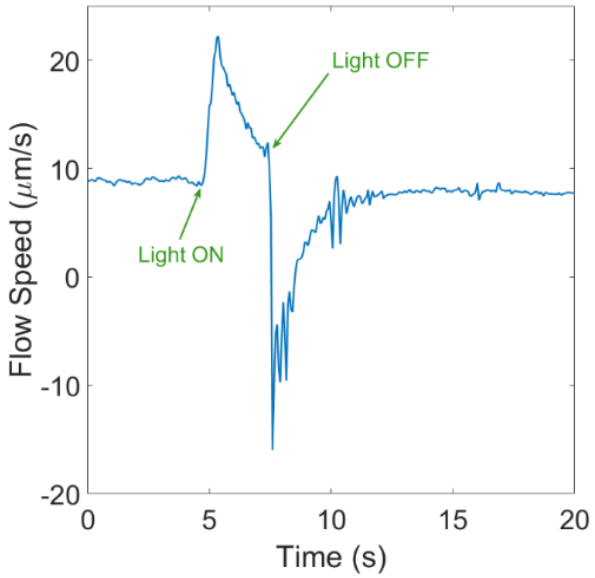


Figure 4 a) Maximum displacement of silica microspheres floating in water due to light-induced channel squeezing at different light intensities. b) Microsphere displacement from its initial position recorded in time corresponding to the point highlighted by the red circle in (a) when the laser is turned on at 4 s and off at 16 s.

Given the experimental conditions, the microsphere takes about 5 seconds to reach the maximum distance from its initial condition, and approximately the same time to go back when the light is turned off. Reducing the channel cross-section can be also exploited to control a continuous flow.

Channel squeezing can be also exploited to control a continuous flow within the channel.

To demonstrate it we monitored the flow of silica microspheres pushed by a motorized syringe pump. Figure 5 shows the average flow speed within the microchannel. The laser spot is placed upstream the monitored section of the channel, slightly out of the field of view. At first, the speed is kept constant by the syringe pressure at about 9 μm/s. Switching on the laser, we observe a sudden increase of the flow speed due to the squeezing that pushes on the



fluid. After that, by keeping the light on, the fluid flow relaxes toward the initial speed that is

Figure 5 Graph showing the average flow speed in the microfluidic channel with respect during time. Light on and light off indicate the presence or absence of the light stimulus

given by the syringe pump. Releasing the channel by switching off the light results in a pressure drop that pulls back the fluid causing the fluid to reverse its flow. After the transient, the flow gets back to the initial condition.

Conclusion

In conclusion, formulations containing azo-chromophore (DR1M) PDMS showed excellent displacement behavior and chemical stability after the light stimulus, making them highly suitable for smart microfluidic device. A single layer channel, in which DR1M was used as dye and functional agent, showed the possibility to control the fluid flows, under proper laser irradiation, in a reversible a real-time manner. An interesting aspect pointed out was that the deformation analysis showed the possibility to completely squeeze the channel and a full recovery of it. Future perspective will be the development of the system with structured light, to have the possibility to control the flow in a parallel and reversible manner in several points, modifying the pattern in real time.

Author Contribution

Acknowledgments

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Supplementary

1. Video chiusura canale in cross section
2. Video movimentazione particelle