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Ding Zhenwen

B. Ziaie

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## A pH-tunable hydrogel microlens array with temperature-actuated light-switching capability

Zhenwen Ding<sup>1,2</sup> and Babak Ziaie<sup>3,2,a)</sup>

<sup>1</sup>Department of Physics, Purdue University, West Lafayette, Indiana 47907, USA

<sup>2</sup>Birck Nanotechnology Center, Purdue University, West Lafayette, Indiana 47907, USA

<sup>3</sup>School of Electrical and Computer Engineering, Purdue University, West Lafayette, Indiana 47907, USA

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In this letter, we demonstrate a two step casting process to fabricate a bifunctional hydrogel-based microlens array, which responds to both temperature (becomes opaque above certain temperature) and pH (changes its focal length at different pH levels), and can be operated in air for an extended period of time. Each lens in the array is 1 mm in diameter and its focal length changes from 4.5 to 55 mm when the environmental pH is varied between 2.0 and 5.0. The light-switching capability is measured to be  $\sim 92\%$  when temperature increases from 25 to 35 °C. © 2009 American Institute of Physics. [DOI: 10.1063/1.3089689]

Tunable microlenses have attracted considerable attention for their potential applications in consumer electronics (e.g., cell phones) and microsystem platforms.<sup>1</sup> The majority of the ones demonstrated to date are based on electrical actuation of surface tension between two different liquid media, although there have also been reports on pneumatically and hydraulically actuated ones.<sup>2-9</sup> One can also fabricate arrays of tunable microlenses for applications in imaging, optical data interconnects, and biomimetics (insect eye).<sup>10-13</sup> More recently, several groups have used environmentally sensitive hydrogels to modulate the focal length. This has been accomplished by either varying the curvature of a liquid-liquid interface<sup>14</sup> or encapsulating temperature-sensitive hydrogel in an artificial shell (polystyrene or thin silicone membrane) and providing an external physical actuation source.<sup>15,16</sup> The hydrogel-based tunable microlens presented so far often requires complicated packaging methods in order to seal the liquid and provide actuation inlets. In addition, such arrays are chemically unifunctional, i.e., focal length variability is accomplished through physical methods. In this paper, we demonstrate a simple two step casting process to fabricate a bifunctional hydrogel-based microlens array that responds to both temperature (becomes opaque above certain temperature) and pH (changes its focal length at different pH levels) and can operate in air for an extended period of time.

Figure 1 shows a schematic illustration of the bifunctional hydrogel microlens array. It consists of a glass base with hemispherical array of etched cavities and a bilayer of thermosensitive (poly(*N*-isopropylacrylamide [poly(NIPAM)]) and pH-sensitive (poly(methacrylic acid-*co*-acrylamide [poly(mAA-*co*-AAM)]) hydrogel. In this hydrogel bilayer, poly(NIPAM) is the light-switching layer, which turns opaque and reduces light transmission once its temperature is raised above 32 °C, while poly(mAA-*co*-AAM) is the tunable layer and has a volume response with environmental pH, thus changing the microlens surface curvature and focal length. Figure 2 shows schematic of the fabrication process. It starts with a glass wafer (500  $\mu\text{m}$  thickness) coated with a uniform layer of 1  $\mu\text{m}$  thick parylene C [Fig.

2(a)]. Subsequently, the parylene layer is patterned using oxygen plasma (50 SCCM O<sub>2</sub>, 100 W, 100 mTorr) (SCCM denotes cubic centimeter per minute at STP) and glass wafer is etched down to 100  $\mu\text{m}$  in 10:1 (vol/vol) HF/HCl solution [Figs. 2(b) and 2(c)]. In order to fix the hydrogel boundary in etched cavities, glass is treated with an adhesion promoter  $\gamma$ -methacryloxypropyl trimethoxysilane ( $\gamma$ -MPS):acetone = 1:10 (vol/vol) for 1 h. pH-sensitive hydrogel [poly(mAA-*co*-AAM)] is then loaded in the cavities by tight clamping [Fig. 2(d)]. After waiting 4 h for complete polymerization, a layer of 50  $\mu\text{m}$  thick poly(NIPAM) temperature-sensitive hydrogel (volume transition temperature of 32 °C) is cast on the top [Fig. 2(e)]. Due to the isotropic nature of swelling process, once the bilayer is immersed in phosphate buffered saline solution, the hydrogel in the concaved-etched areas swell more, creating a biconvex lens structure [Fig. 2(f)].

As mentioned above, when environment temperature is above 32 °C, poly(NIPAM) layer shrinks and becomes opaque, hence blocking the light transmission through the array. Figure 3 shows light transmission through the array at 25 and 35 °C. The photographs show a qualitative view with the letters underneath the microlens array being clear at 25 °C and unreadable at 35 °C. In order to quantify the opacity, a 635 nm and 3.63 mW laser diode was set up as the incident light source, and transmitted light through the microlens array was collected by a photodetector (Thorlabs Inc., DET110). The top panel in Fig. 3 shows the photodetector outputs at two above mentioned temperatures. At room temperature (25 °C), the photodetector output was 2.45 V, which was almost similar to the value obtained by shining

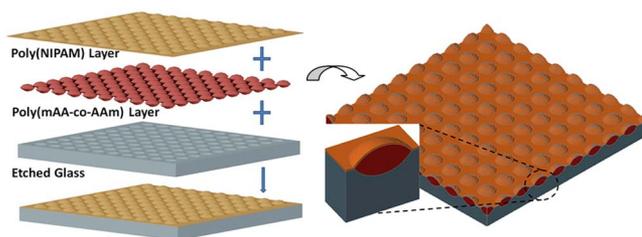


FIG. 1. (Color online) Schematic illustration of the bilayer hydrogel microlens array.

<sup>a)</sup>Electronic mail: bziaie@purdue.edu. Tel.: +1 765-494-0725.

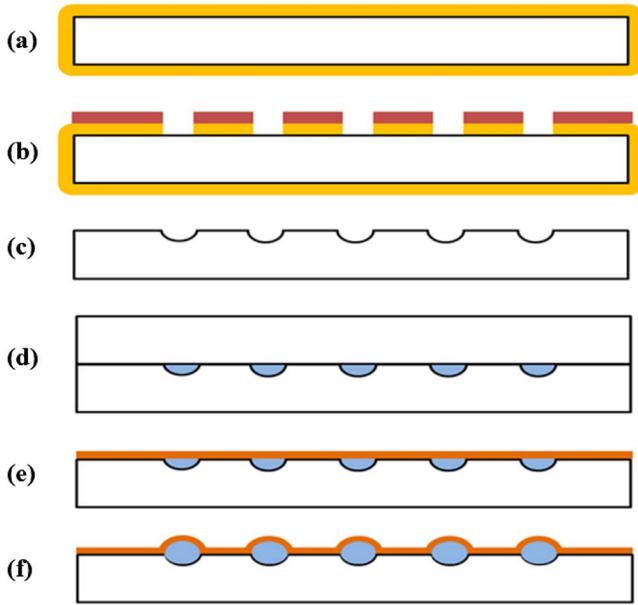


FIG. 2. (Color online) Hydrogel microlens fabrication process.

light directly onto the detector. At 35 °C, when the microlens array became opaque, the output was reduced to 0.18 V (~92% reduction). We also increased the poly(NIPAM) layer thickness to 100 μm and measured the opacity. In that case the detected signal dropped further to 0.043 V (~98% reduction).

The focal length of the microlens can be calculated by knowing the lens geometry and refractive indices using<sup>17</sup>

$$f = \frac{n_a}{(n_L - n_a)} \frac{(D/2)^2 + h^2}{2h}, \quad (1)$$

where  $D$  is the aperture of the microlens,  $h$  is the protruded thickness of the hydrogel,  $n_a$  is the refractive index of surrounding medium (air, in our case), and  $n_L$  is the refractive index of the lens material. Based on the structure of our microlens,  $n_L$  should be approximated to the average of the refractive indices of glass (1.4–1.5) and hydrogel (~1.33).

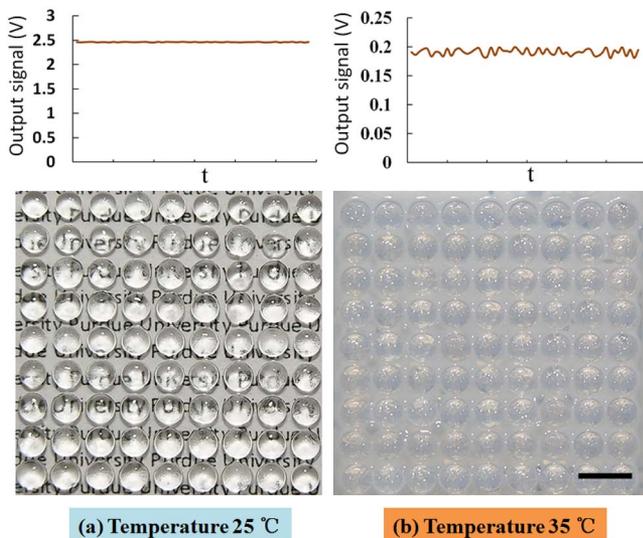


FIG. 3. (Color online) Testing the transparency of the hydrogel microlens array at different temperatures (a) at 25 °C and (b) at 35 °C. Scale bar, 3 mm.

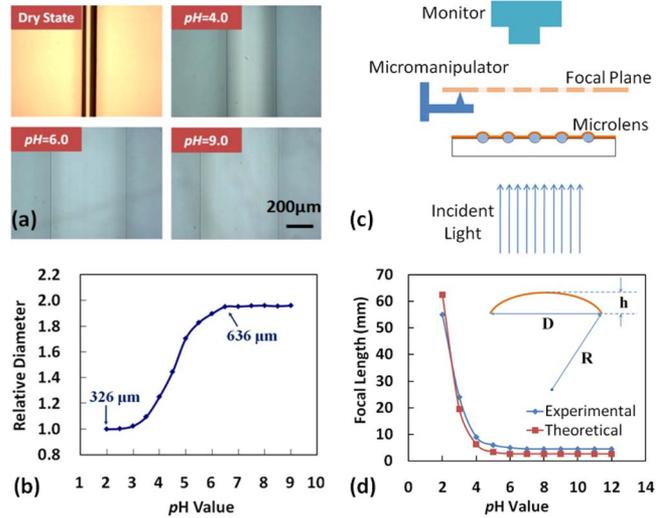


FIG. 4. (Color online) (a) Micrographs of a cylindrical pH sensitive hydrogel at different pH levels. (b) Free-swelling diameter of pH sensitive hydrogel vs pH. (c) Focal length measurement setup. (d) Measured focal length vs pH values and comparison with theoretical calculations.

Poly(mAA-co-AAm) is a well known pH-sensitive hydrogel, which has the free-swelling properties shown in Fig. 4(a), with the micrographs showing increase in the diameter of a cylindrical hydrogel rod at different pH values (150 μm in dry state, 470 μm at pH=4, 618 μm at pH=6, and 638 μm at pH=9). The curve summarizes the hydrogel free-swelling behavior showing a sharp volume transition between pH of 3.0 and 6.0 [Fig. 4(b)]. The variable focal length corresponding to various pH values was measured using the setup shown in Fig. 4(c). A low power laser beam with spot size of 2 mm was used as the incident light source. Using a micromanipulator, the focal length was measured by moving the focal plane and identifying the smallest and brightest spot under microscope. The focal length has a sharp transition (4.5–55 mm) when pH is between 2.0 and 5.0 while showing almost no variations when pH is above 7.0 [Fig. 4(d)]. This is expected since, as shown before, the volume transition occurs between pH of 3.0 and 6.0. Figure 4(d) also shows the theoretical curve closely matching the measured results [curve was obtained by inserting experimental

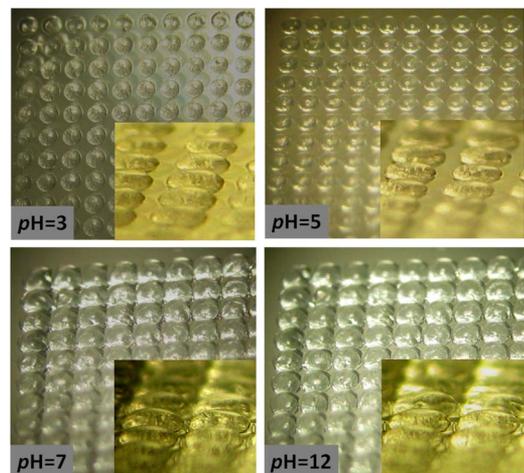


FIG. 5. (Color online) Micrographs of microlens array in different pH environments. Insets show close-up tilted images.

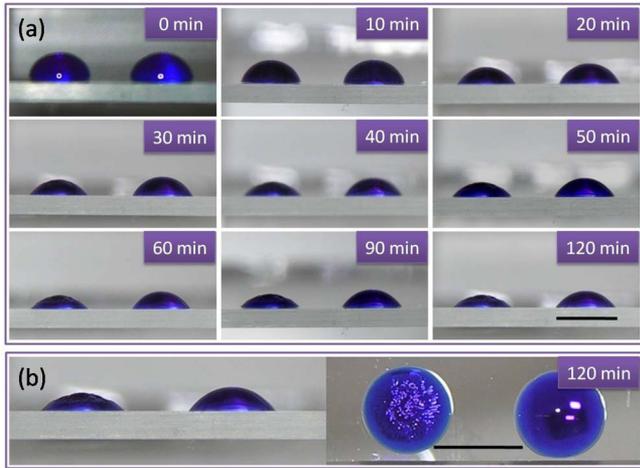


FIG. 6. (Color online) Dry out experiment comparing the hydrogel with (right) and without (left) glycerol at room temperature. Dye has been added to provide contrast. Scale bar, 2 mm.

values into Eq. (1)]. Figure 5 shows the micrograph images of microlens array under different  $pH$  conditions. The corresponding insets show close-up tilted views, clearly showing the curvature variations in the protruded hydrogel lens corresponding to  $pH$  values.

A severe limitation of hydrogel lenses is the requirement for continuous operation in a liquid environment. This is due to the fast evaporation of water from hydrogel matrix in air environment. Addition of glycerol to the hydrogel can delay the water evaporation while still preserving its chemical sensitivity. This allows the microlens array to be used in dry environment for several hours. Figure 6(a) shows the dry out test results for hydrogel samples without glycerol (left) and with 20% (vol) glycerol (right) at room temperature. The sample without glycerol shows a noticeable shrinkage after 10 min and starts wrinkling after 40 min, whereas the sample with glycerol shows a much slower evaporation rate and can still be used after 2 h. Figure 6(b) shows the top and side views of the above mentioned samples after 120 min where

surface wrinkles can be easily seen on the sample without glycerol.

In conclusion, we reported on the fabrication and measurement results of a bifunctional hydrogel microlens array having both  $pH$ -tunability and temperature-activated light switchability. The structure was fabricated through a double casting process onto hemispherically etched glass cavities. Addition of 20% glycerol preserved the hydrogel microlens structural and functional integrity for up to 2 h expanding the applications of such lenses beyond what has been reported so far. Applications in analytical microsystems and hydrogel physical characterization platforms (e.g., determination of elastic constants of bilayer microstructures through simple optical measurements) are envisioned.

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