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SciVerse ScienceDirect

Procedia Engineering 25 (2011) 856 – 859

Procedia Engineering

www.elsevier.com/locate/procedia

Proc. Eurosensors XXV, September 4-7, 2011, Athens, Greece

Micropatterning and dynamic swelling of photo-crosslinkable electroactive Pluronic hydrogel

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Abstract

This paper presents the controlled swelling of a novel combination of materials for microsystems: a photopatternable electroactive polymer gel. It is very promising as an actuator material for e.g. biomedical or microfluidic applications as it shows a volume swelling of over 50% upon application of very modest voltages in a liquid environment. We present the synthesis of the novel material, the development of a MEMS compatible fabrication process and the measurements on fabricated test structures.

Keywords: micropatterning; photo-crosslinkable; electroactive; Pluronic; hydrogel

1. Introduction

Stimuli sensitive hydrogels can undergo unparalleled volume changes when certain environmental parameters alter. Based upon the absorption and release of an aqueous solution accompanied by considerable swelling forces, the volume changes can exceed a hundredfold.

These “smart” hydrogels have attracted particular attention due to their responsiveness to external stimuli such as temperature, pH, light, electric field, solvent composition, ionic strength, etc. [1, 2]. These active functionalities make stimuli sensitive hydrogels very promising materials for a broad range of applications in the field of sensors and actuators like microvalves, micropumps, pH sensors, glucose

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1877-7058
doi:10.1016/j.proeng.2011.12.210

sensors etc. [3]. Among these possibilities, we are especially interested in the sensitive polymer gels activated by an external electric stimulus.

Hydrogels show promising potentials as an active material for new types of biomedical applications. Their high water content contributes to biocompatibility and their rubbery nature provides minimal mechanical irritation to surrounding tissues. In our work, Pluronic F127 (PF127) is chosen as the hydrogel matrix due to its low cytotoxicity, since it is widely investigated for cell encapsulation, tissue engineering and drug delivery [4]. Furthermore, designing the gel to allow an electric field to be used as an external stimulus has the additional advantage of more precise control. In this paper, we report the synthesis, fabrication and measurement of a novel customized photo-crosslinkable electroactive Pluronic hydrogel.

2. Materials

ABA diblock copolymer Pluronic F127 (A: polyethylene oxide; B: poly(propylene oxide)) was combined with a crosslinkable group methacrylate. The end product, PF127-bis methacrylate (PF127-BMA), was further altered to become electroactive by introducing charged monomers. Dimethylaminoethyl methacrylate (DMAEMA) was chosen as it was reported to show a certain electric responsiveness [5]. After mixing with Irgacure 2959, a photoinitiator for free radical polymerization, this PF127-BMA-DMAEMA hydrogel can be patterned by photolithography.

3. Fabrication Processes

Integrating hydrogels with MEMS fabrication processes has proven to be challenging. The hydrogel can be applied on a D263t glass wafer by spin coating and UV patterning. Silanization was required to improve the adhesion of hydrogels to the substrates [6]. A substrate with chromium contact electrodes was immersed in a surface adhesion promoter, hexamethyldisilazane (HMDS), and heated at 100 °C for 3 minutes. The PF127-BMA-DMAEMA hydrogel (30 wt% PF127-BMA, 6wt% DMAEMA, 2 mol% Irgacure 2959) was then spin-coated and UV exposed at 3000 mJ/cm^2 through a mask. The uncrosslinked hydrogel was developed away with acetone.

Fig. 1 shows a schematic drawing of the fabricated test structures: one hydrogel pillar stands on top of two Cr electrodes. The Cr electrodes can be biased through bondpads, which provides the electric field to actuate the hydrogel. The hydrogel can swell/deswell towards three different equilibrium states: shrunken state without any presence of liquid, swollen state when immersed in liquid, and electroactivated state by applying an electric field in the liquid.

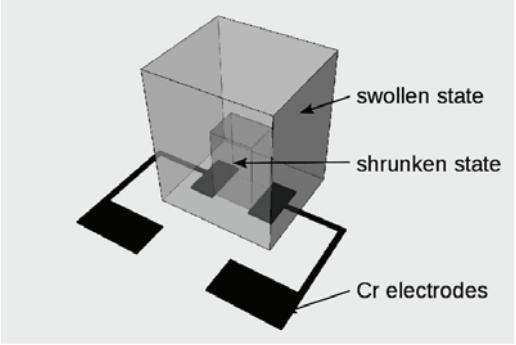
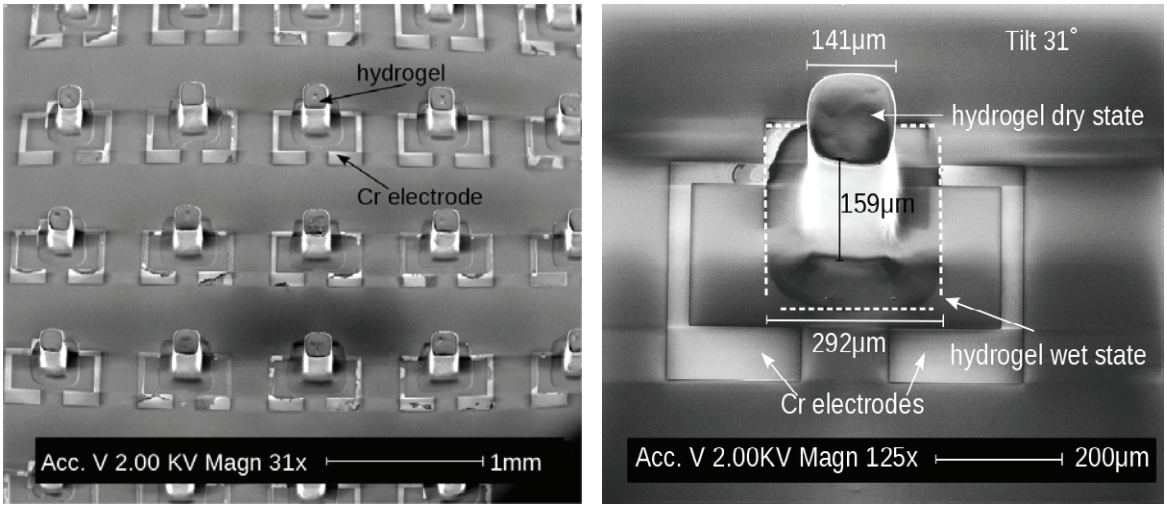


Fig. 1. Schematic drawing of a hydrogel pillar on top of Cr electrodes, both swollen and shrunken states are depicted.

In Fig. 2 (a), the hydrogel pillars are in their dry state. Despite of the 6-fold volume change in the wetted state, the bottom of the hydrogel pillars remains anchored on the substrate. Fig. 2 (b) was taken at 31° tilt. It shows one single hydrogel pillar with an aspect ratio of 2.3.



(a) (b)

Fig. 2. (a) SEM picture of an array of hydrogel pillars with Cr electrodes beneath. The hydrogel is at shrunken state; (b) SEM picture of one single hydrogel pillar. The extent of the wet state gel is clearly visible in the footing of the gel pillar.

4. Measurements

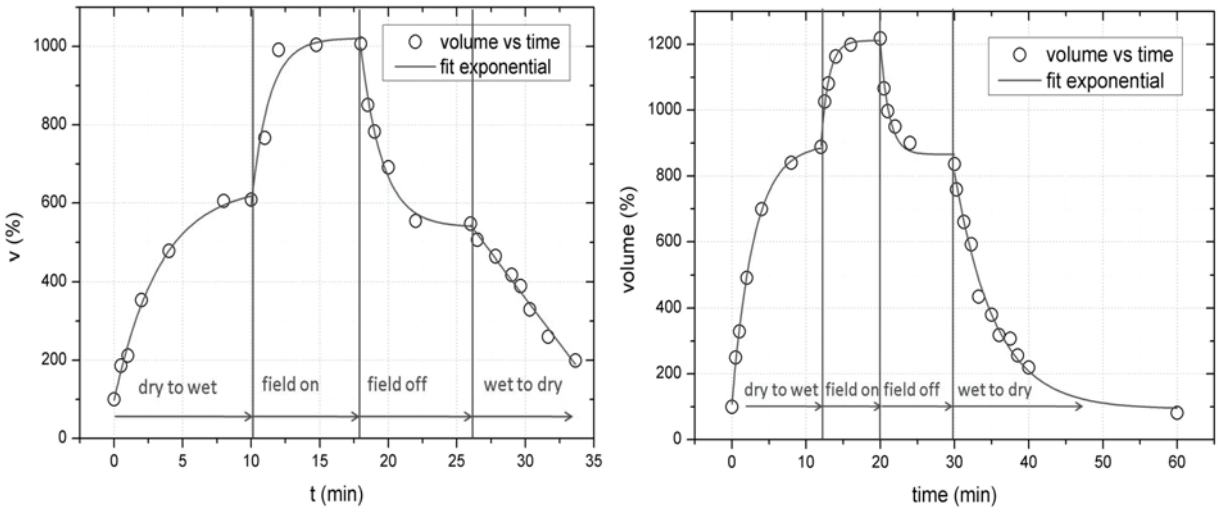
For measurements of the volume changes, a Mitutoyo QuickVision 3D optical microscope was used. Dimension changes in xyz axes were measured simultaneously. By determining the focal plane, the thickness of the hydrogel test structure was acquired. Dry sample measurements could be confirmed using SEM. Comparing to the more commonly utilized interval measurements of weight differences, transportation of hydrogel pieces to an analytical balance is not required.

Fig. 3 (a) shows the measured swelling/shrinking curve of the hydrogel pillar in deionized water. The hydrogel at shrunken state is immersed for a sufficient time to reach its equilibrium swollen state, a 600% volume change. Then, a voltage of 2V is put over the electrodes while the hydrogel is still in the water. Because of its electroactive behavior, the hydrogel undergoes another expansion, until its equilibrium electroactivated state is reached, 1000% of the dry volume. Afterwards, the applied voltage and distilled water are removed sequentially with sufficient time in between to allow the hydrogel to relax back to its original state. The swelling/shrinking effect is shown to be reversible.

In Fig. 3 (b), conductive PBS solution is used instead of deionized water, while maintaining the rest of experiment parameters the same as in Fig. 3 (a). A 20% higher swelling is achieved. The comparison of characteristic time constant τ and swelling ratios is listed in Table 1.

Table 1. Comparison of swelling ratios and characteristic time constant τ.

	swollen state	electro-activated state	τ dry to wet	τ field on	τ field off
deionized water	600%	1000%	3.4	1.3	1.6
PBS	840%	1220%	3	1	1.2



(a) (b)

Fig. 3. (a) Deionized water is used as solvent. Electric field is generated by supplying 2V voltage across the Cr electrodes. The curve is exponentially fitted according to $y \propto e^{x/\tau}$, as the swelling is diffusion dominated. τ is the characteristic time constant.; (b) Phosphate buffered saline (PBS) is used as solvent.

5. Conclusion

We have demonstrated a novel active material for microscale applications in wet environments, entirely compatible with wafer-level lithographic microfabrication tools. With 50% volume change in an aqueous solution upon the application of a low actuation voltage, the material is expected to contribute significantly to fields as biomedical, microfluidics, drug release, etc.

Acknowledgements

This work is sponsored by the IWT SBO (Strategic Basic Research in Flanders) program and supported by the Hercules initiative for large equipment (AKUL034, Cluster for micro and nanolithography).

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