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A polymer microdevice for tensiometry of insoluble components

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Abstract

A semi-flexible SU-8 polymer microdevice was designed to deflect in response to a change in surface tension at an air-water interface. The suspended microtensiometer encloses a clean water-air interface, while externally the device is surrounded by an interface containing an insoluble component. The difference in surface tension between the inside and outside of the device, called the surface pressure, causes the device to deflect. Finite element simulations were performed to predict device behavior prior to fabrication. Finished devices were tested in a Langmuir trough during multiple compression and expansion cycles, using a platinum Wilhelmy plate for an independent surface pressure measurement. A resolution of 0.16 mN/m was achieved.

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Keywords: MEMS; SU-8; interfacial rheology; surface pressure

1. Introduction

The surface tension of a fluid-fluid interface is crucial to the production and processing of many multiphase materials, which are found in a variety of industrial, engineering and medicinal applications. Often one is interested in the difference in the surface tension of an interface between the interface populated by a surface component and the clean interface. This difference is termed the surface pressure, and it increases as the concentration of surface-active component increases. One conventional technique for measuring surface pressure is to change the surface concentration by compression and measuring the surface tension with a Wilhelmy plate connected to an

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electromagnetic balance [1]. For insoluble monolayers a Langmuir trough [2] is often used to carry out this compression and to control the surface concentration of surface-active substances, such as long-chain surfactants, proteins, fatty acids, phospholipids or even colloidal particles. The setup is somewhat cumbersome to use and requires significant amounts of liquid subphase as well as surface-active components, and may suffer from temperature fluctuations, evaporation and contamination.

In this work, a semi-rigid, semi-flexible polymer structure based on existing microtensiometer devices [3, 4] was redesigned to measure the surface pressure locally, with an embedded read-out system and increased sensitivity. This may be a start for spatially resolved measurements or for miniaturization.

2. Design and simulation

The device consists of two parallel rigid beams, connected by millimeter-scale springs. Together, these components enclose a small amount of clean water-air interface. The interface external to the device contains insoluble surfactant, with a surface tension that is different from the clean interface. Hence, it results in a surface pressure, which causes the device to compress through the flexible springs while the side beams do not noticeably bend.

The beam length l_{beam} is chosen to be 3 mm, comparable to those used in earlier work [3]. The dimensioning of the springs is based on the maximum surface pressure the device should be able to endure and thus the maximum deflection it should be able to undergo without any of the edges touching each other. The stiffness K of the device is a function of the Young modulus E of the structural material, which is around 2.3 GPa for SU-8 [5], and the thickness t. Simulations were performed for several values of t, around the desired thickness of 10 µm. The results are shown in Fig. 1.

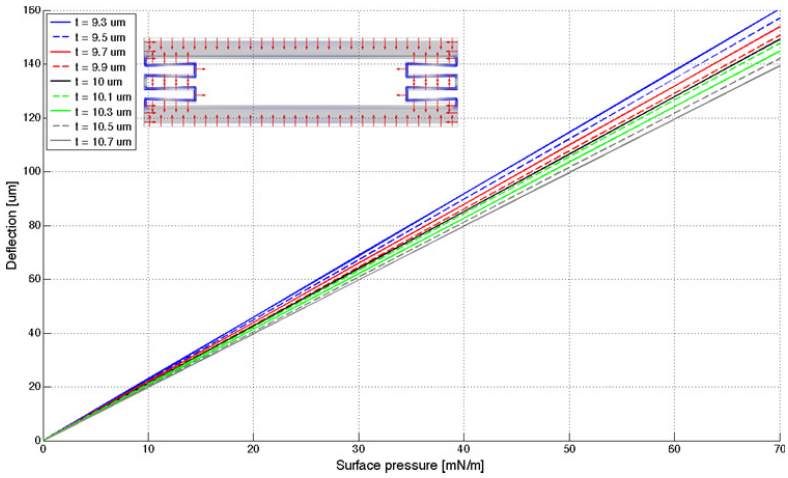

Fig. 1. Simulated deflection of an 850 µm by 3 mm device for various thicknesses. An acting surface pressure is simulated as a uniformly distributed external load on all device boundaries.

The equation describing the linear device deflection Δy as a function of the surface pressure Π is

$$K(E,t)\Delta y = 2\Pi l_{beam} \tag{1}$$

For the aforementioned Young modulus of 2.3 GPa and a verified device thickness t of 10.5 µm, the simulated stiffness $K(E, t)$ is 2.95 N/m, i.e. a deflection of 2.03 µm/(mN/m).

3. Fabrication

For the fabrication of the microtensiometers, a clean silicon substrate is coated with 2 μm of LOR10B from Chimie Tech Services, which is patterned to form sacrificial islands (Fig. 2a). After a HF dip to ensure a hydrophobic silicon surface, 10 μm of SU-8 2010 from micro resist technology GmbH is spin coated at 3000 rpm. A soft bake of 7 minutes at 95°C precedes the selective exposure of SU-8 to 125 mJ/cm^2 of UV light. The post-exposure bake is performed at 95°C and is followed by a 90-second development in propylene glycol monomethyl ether acetate (PGMEA). A one-hour hard bake at 150°C is necessary to complete the curing process. The final SU-8 structures consist of a tensiometer, a triangular suspension and a square anchor on the substrate (Fig. 2b).

The sacrificial layer is removed in an alkaline solution and a critical point drying with CO_2 is performed to achieve a stictionless release (Fig. 2c). To detach a tensiometer from the substrate, a needle with a droplet of glue (Permabond 102 Industrial Cyanoacrylate Adhesive) is lowered onto the triangular suspension. Once the glue is dry, the connection to the square anchor is severed and the tensiometer is lifted off the substrate.

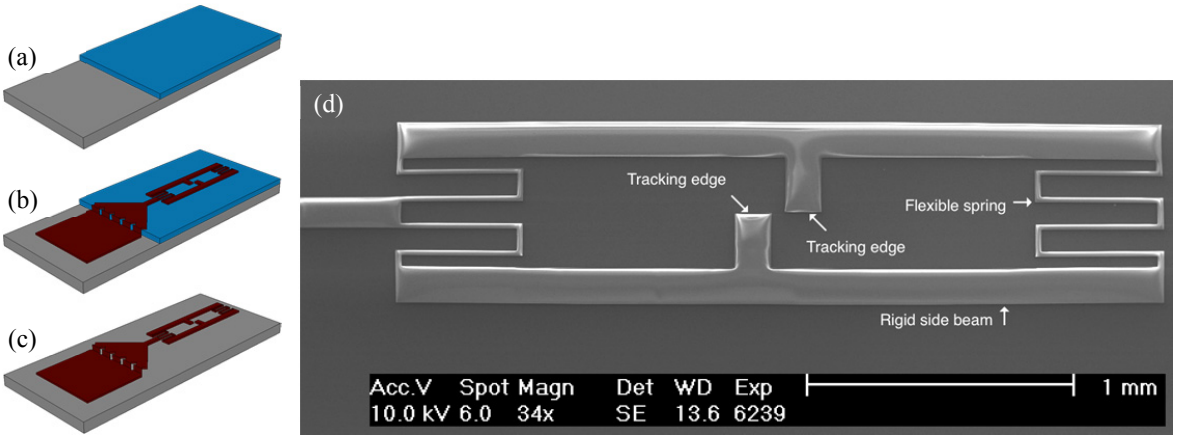

Fig. 2. (a) Sacrificial layer. (b) Patterned SU-8. (c) Released structure. (d) SEM image of an unreleased SU-8 tensiometer with embedded read-out mechanism, consisting of two beams. To the left, the suspension beam that connects to the triangular anchor is visble.

4. Experimental details and results

Testing is performed in a Langmuir trough that allows for the modification of the surface concentration of hexadecanol, an insoluble surfactant, via barrier movement. An independent measurement of surface pressure is performed with a Wilhelmy plate suspended as close to the tensiometer as possible. The tensiometer itself - glued on a needle tip - is lowered onto the clean air-water interface and positioned over an inverted microscope via a micropositioner in order to observe the integrated read-out system (see Fig. 2d). After introducing the hexadecanol solution (1 mg/ml in isopropyl alcohol) in the trough and waiting for half an hour to allow the surface concentrations to equilibrate, the barriers are displaced at a speed of 10 mm/min, thereby varying the mean molecular area of the hexadecanol from 47.5 to 20.4 Å^2/molecule. This cycle is repeated three times.

Optical micrographs of the embedded read-out system of the tensiometer are processed using ImageJ and MATLAB. An edge-tracking algorithm is used to measure the motion of two edges inside of the tensiometer (indicated in Fig. 2d) in units of pixels. The deflection of the tensiometer is calculated with the appropriate pixel-to-micrometer conversion factor, which depends on the specific objective and camera in the optical setup. The conversion factor for the work presented herein is 0.315 μm/pixel.

Fig. 3 shows data gathered by the Wilhelmy plate along with the deflection of the microtensiometer as extracted from the image analysis. Micrographs and Wilhelmy plate data were both collected at a frequency of 1 Hz. A very strong correlation between both measurements is immediately clear from the graph. The uncertainty of the tensiometer measurement is defined as the error produced by a single-pixel read-out error and is calculated by taking

into account the device stiffness and the image accuracy. The device described herein has an uncertainty of ±0.16 mN/m, which is comparable to the Wilhelmy plate (±0.1 mN/m) and significantly better than the uncertainty of the devices presented in [3] (±0.7 mN/m).

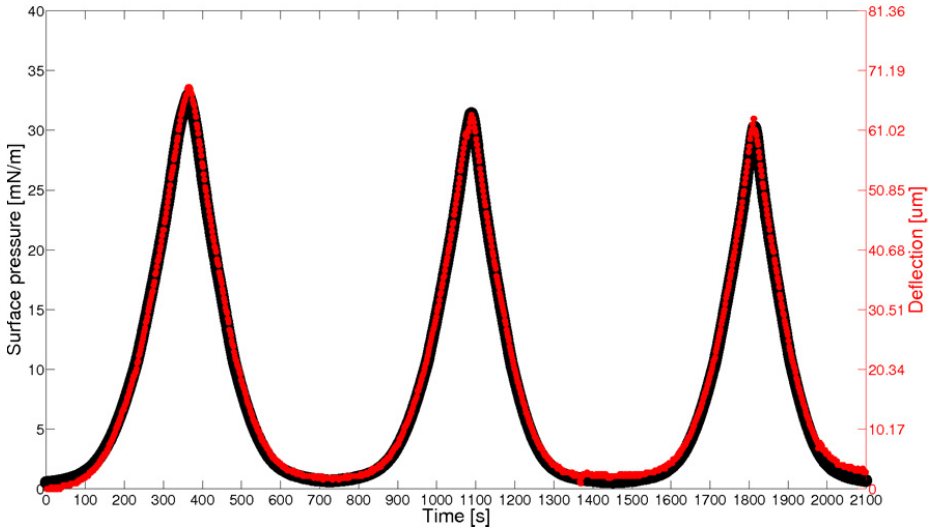

Fig. 3. Measurement results showing the surface pressure measured by a Wilhelmy plate (black) and the tensiometer deflection (red) during three compressions/expansions in a Langmuir trough. As the right axis ticks were obtained from the left axis using the simulated conversion factor of 2.03 μm/(mN/m) it can be concluded that the simulations predicted the device behavior relatively well.

5. Conclusion

Semi-rigid, semi-flexible microtensiometers were designed and tested to measure local surface pressure variations of an insoluble model surfactant. The device encloses a clean air-water interface, while its outer perimeter is exposed to an interface containing an insoluble component. This difference in surface tension results in a linear deflection of the device, which is very strongly correlated with an independent measurement using a Wilhelmy plate. The geometry of the microtensiometers, along with the image processing, allows a maximum resolution of 0.16 mN/m.

Acknowledgements

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