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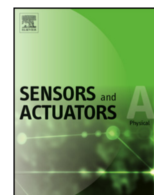

Langmuir monolayer characterization via polymer microtensiometers

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ABSTRACT

A semi-rigid, semi-flexible SU-8 polymer microdevice was designed to measure changes in interfacial tension at an air–water interface. The suspended microtensiometer enclosed a clean air–water interface, with an insoluble surfactant on the exterior. The difference in surface tension between the inside and the outside of the device, called the surface pressure, caused the 850 μm by 3 mm 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 large area changes and slow compression speeds. Shorter experiments subjecting the interface to rapid local monolayer concentration variations were also performed. A platinum Wilhelmy plate was used as an independent surface pressure measurement. The microtensiometer had a theoretical resolution of 0.02 mN m^{-1}.

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1. Introduction

The surface tension of a fluid plays a key role in the formation of droplets, insects walking on water, inkjet printing, tears in a wine glass [1] and the Cheerios effect [2]. Moreover, the mechanical and rheological properties of a fluid–fluid interface (of which surface tension is one aspect) are crucial to the production and processing of myriad multiphase materials, which are found in a variety of industrial, engineering and medicinal applications [3–5]. Often one is interested in the surface tension of an interface populated by a surface-active component as compared to when that interface is clean (i.e. no surfactant). The difference is termed the surface pressure and it increases as the concentration of surface-active component increases (i.e. its mean molecular area decreases). One well-established technique for measuring the surface pressure is to change the surface concentration by compression of the fluid surface and measuring the surface tension with a Wilhelmy plate [6] connected to an electromagnetic balance. For insoluble monolayers, a Langmuir trough [7,8] is often used to compress the interface and to control the surface concentration of surface-active substances [9]. The latter include 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, effects of evaporation, and the large open area makes it prone to contamination. Moreover, the accuracy of the Wilhelmy plate is limited (±0.1 mN m^{-1}) and its response time is typically longer because of the time constants of the feedback loop of its measurement system. Besides the Wilhelmy plate, other surface or interfacial tension measurement methodologies are available such as the Du Noüy ring method [10] and the pendant or spinning drop method [11]. Drawbacks of the former technique include the need for a correction factor and the fact that the measurement is performed in a non-equilibrium state of the interface. The latter method is very accurate for low surface tensions (<10^{-2} mN m^{-1}) but is unsuitable for high values of the interfacial tension. Miniaturised setups for tensiometry measurements are not entirely new [12] and in this work, we build on earlier ideas by Zell et al. [13] in which a microtensiometer had been proposed, made of a semi-flexible polymer structure. The device described by Zell et al. was a rectangular structure, with thin walls which deflected under the action of surface pressure. The deflection of

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the walls was modeled with the beam equation. In the present work, we develop a device with an embedded read-out system and with the goal to obtain an increased sensitivity. The device described herein is a further step towards routinely measuring the local surface pressure on the microscale, which would greatly benefit the characterization of complex fluid–fluid interfaces. The first section of this article explains the design choices that were made and provides some theoretical background. This is followed by a detailed overview of the microfabrication process for the devices. Before discussing the results in a final section, the experiments and equipment are described.

2. Theoretical framework and design

2.1. Operating principle

The device consists of two parallel rigid beams, connected by two millimeter-scale springs. Furthermore, there are two shorter beams in the center of the structure to measure the deflection, as will be discussed in Section 4.1.2. A 2D model of the structure in its resting state is presented in Fig. 1(a). The device can be placed at a fluid–fluid interface so that the inner part of the sensor contains a pristine interface. For all the simulations and experiments discussed in this article, this was a water–air interface. The possibilities of using an oil–water interface are to be explored in future work. Insoluble, surface-active materials can be deposited on the outside of the device, their concentration being varied by dosage or compression, thus creating a surface tension that is different from that of the clean interface. Hence, it results in a surface pressure which causes the device to be compressed through the flexible springs, while the main beams do not noticeably bend. This approach differs from the original design by Zell et al. [13] who relied on the bending of the main beams. The thickness of those beams, combined with the method to measure their deformation, set the sensitivity and the dynamic range of the device. As in the present design the constants of the flexible springs can be altered by both the shape and thickness of the springs, a higher sensitivity can be reached, with the same microfabrication techniques.

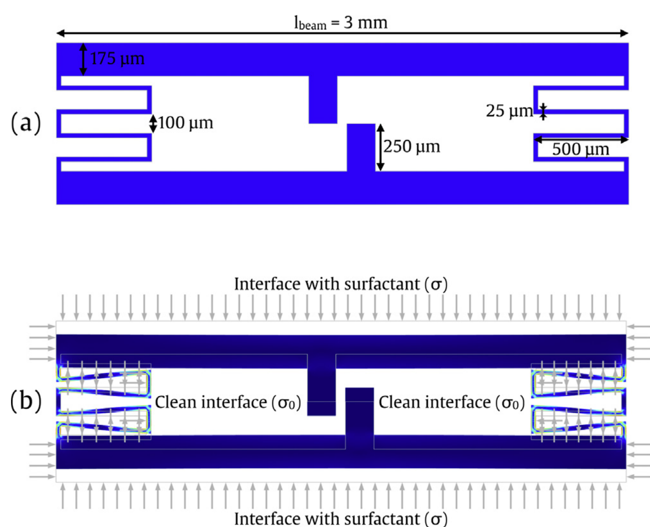

Fig. 1. 2D model of the SU-8 tensiometer. (a) The device in its neutral state with an indication of the main dimensions. (b) A surface pressure deforming the structure is simulated as a uniformly distributed external load, perpendicular to all device boundaries.

The surface pressure Π is defined as the change in surface tension between a clean subphase, indicated by σ_0 and that of a surface-active monolayer, denoted simply by σ. Moreover, the surface tension at the interface after addition of a surface-active component strongly depends on the surface concentration, Γ. Eq. (1) summarizes this definition.

$$\Pi(\Gamma) = \sigma_0 - \sigma(\Gamma) \tag{1}$$

The subphase in this stage of the tensiometer development was water. Thus, the maximum surface pressure the device should be able to endure is equal to the surface tension of water (at 20 °C), i.e. 72.86 mN m^{-1} [14]. This constraint determines the minimum required stiffness of the device, as none of the device (spring) edges are allowed to touch each other, even under maximum surface pressure conditions ($\sigma(\Gamma)$ = 0). To determine the device compliance, which is dominated by the stiffness of the spring structures, finite element simulations were performed using the COMSOL Multiphysics [15]. Fig. 1(b) shows a deformed device under maximum surface pressure conditions.

2.2. Finite element simulations

The beam length l_{beam} and the desired device thickness t were chosen to be 3 mm and 10 µm respectively. These values are comparable to those used in earlier work [13]. The main beam width was fixed at 175 µm to ensure the bending of the main beams would be negligible. The stiffness K of the device – for a specific spring geometry – is a function of the Young modulus E of the structural material and its thickness t. The Young modulus of SU-8 was assumed to be 2.3 GPa [16], which is slightly higher than the 2 GPa mentioned in the data sheet [17], because the processing conditions (see Section 3) were altered [18]. From these design choices, the dimensions of the spring structures were optimized via simulations using the maximum surface pressure constraint described at the end of Section 2.1. All the main in-plane dimensions of the microtensiometer are indicated in Fig. 1(a). To study the impact of process variations – specifically the device thickness – on the device compliance, simulations were performed for several values of t, around the chosen thickness of 10 µm. The results of these simulations are shown in Fig. 2. It is clear that as the device thickness, and with that also $K(E, t)$, increases, the deformation for a given surface pressure is lower. Eq. (2) describes the linear tensiometer deflection Δy as a function of the surface pressure Π.

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

This equation solely contains parameters stemming from the device geometry and the material used for the fabrication. For any observed deflection, the surface pressure Π can be directly deduced from this equation without the use of any adjustable or fitting parameters, on the condition that the Young modulus and thickness are both known. Results obtained using the microtensiometers can thus be compared to other, independent measurement techniques such as the Wilhelmy plate. For the aforementioned Young modulus of 2.3 GPa, spring dimensions as indicated in Fig. 1(a) and a verified device thickness t (post-fabrication, using a Dektak XT profilometer) of 10.5 µm, the simulated stiffness $K(E, t)$ is 2.95 N/m, i.e. a compliance of 2.03 µm/(mN m^{-1}).

3. Fabrication process

The fabrication of the microtensiometers starts with the thorough cleaning of a 3-in. silicon substrate in Piranha, a 3-to-1 mixture of sulphuric acid and hydrogen peroxide. After rinsing the

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