

Liquid transport detection using single FET devices

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ABSTRACT

Observation and monitoring liquid transport and transport in liquids is a major task for lab on a chip applications and research. We use novel thin film FET devices based on silicon-on-insulator (SOI) substrates as planar sensors for the detection of liquid transport in capillaries to overcome the drawbacks of optical flow observation techniques the same time providing chemical composition analysis capabilities of ISFET technologies. Based on a newly developed theoretical model for Field-Effect liquid movement detection we demonstrate fundamental applications like the detection of capillary filling speed and level. To proof compatibility with ISFET technologies we demonstrate chemical composition analysis capabilities pH-detection and sensitivity to ionic strength. Furthermore we show how the sensitivity of the devices can be tuned as a unique feature provided by the use of silicon-on-insulator substrates.

Keywords: fluid transport , flow, electrochemical sensing, micro fluidic devices, nano fluidic devices

1 INTRODUCTION

Observation and monitoring liquid transport and transport in liquids is a major task for lab on a chip applications and research. In most cases optical techniques like video or fluorescence microscopy are used for this purpose [1]. To achieve high spatial resolution in the micrometer regime the area under investigation must be

comparatively small. Furthermore marker objects or substances often have to be applied which may influence the liquid itself or the objects in the liquid (e.g. influence of marker attachment to the conformation and thus functionality of proteins). Besides few alternative techniques are reported like the measurement of dynamic pressure [2], thermal conductivity [3] and an ion-emitter-detector setup [4]. These techniques require a detailed knowledge of the liquid properties, change locally the liquid composition or disturb flow. We use novel thin film FET (Field Effect Transistor) devices based on silicon-on-insulator (SOI) substrates (**Fig. 1**) as planar sensors for the detection of liquid transport in capillaries to overcome the drawbacks mentioned above the same time providing chemical composition analysis capabilities of ISFET (Ion-Selective Field Effect Transistor) technologies [5].

2 EXPERIMENTAL SECTION

The μm to mm -scale devices are fabricated by using three UV-circuit board lithography steps: After the first step the 55nm thick top silicon layer on top of the 95nm thick buried oxide of the SOI substrate is patterned by wet chemical etching. At the end of the second step 200nm Au on top of 10nm Ti are deposited by thermal metal evaporation as metal contact pads forming the source and the drain of the FET. In the third lithography step $3\mu\text{m}$ thick photoresist is patterned in such away that the metal contacts are insulated from the electrolyte to prevent leakage currents. The so-prepared devices are integrated into a microfluidic cell (**Fig.2a**): The sensor chip (2) is

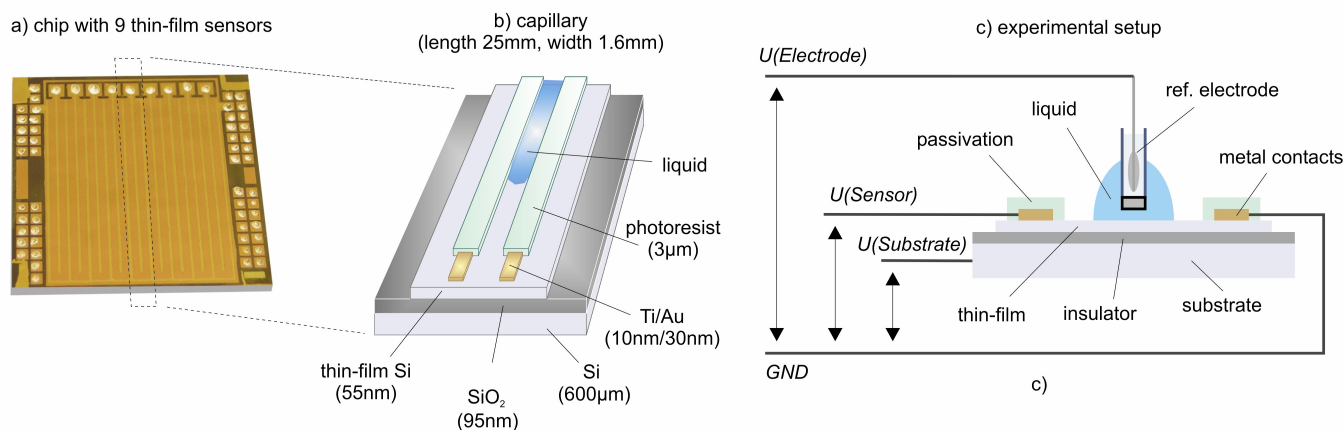


Figure 1: [8] The novel thin film FET Sensor. a) Image of a sensor chip with 9 integrated thin film transistor sensing elements without photoresist passivation. b) Scheme of a sensing element with materials used. c) Cross-sectional view of a sensing element indicating functional parts and electrical connections.

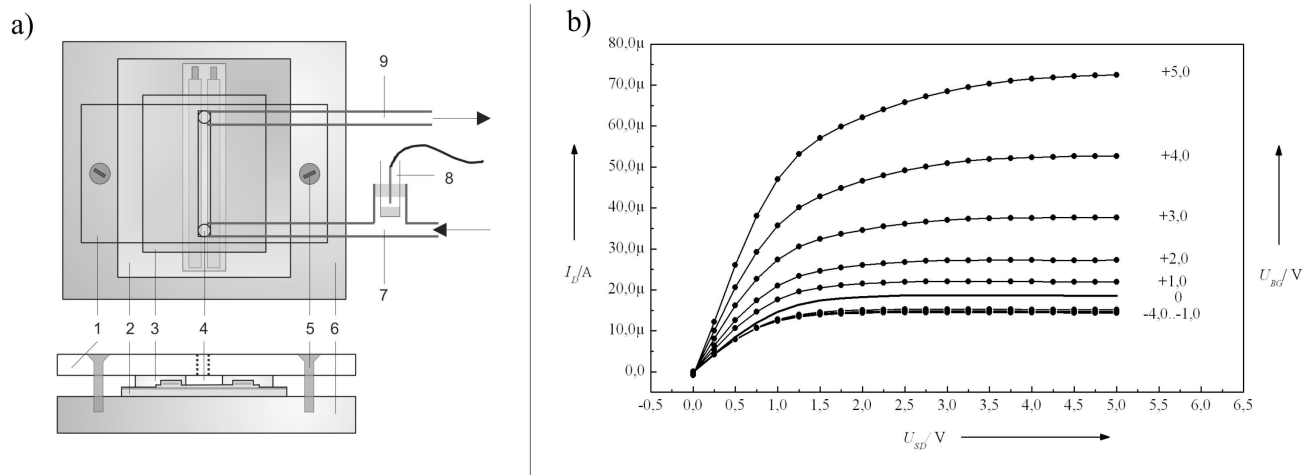


Figure: 2 a) [8] Microfluidic cell. polycarbonate panel (1), sensor chip (2), silicone rubber sheet (3), liquid channel (4), screws (5), sample stage (6), inlet (7), reference electrode (8), outlet (9). b) FET Characteristics. The drain current I_D is recorded as a function of the source-drain-voltage U_{SD} at various backgate (substrate) voltages U_{BG} .

placed onto a copper sample stage (6). Onto the sensor chip (2) a silicone rubber sheet with a gap (3) is placed which is covered by a transparent polycarbonate panel as the liquid interface (1). The liquid channel (4) consists of the sensor surface as the bottom, the liquid media interface with in- and outlets (1) as the lid and silicone rubber sidewalls (3). For appropriate sealing, the silicone rubber sheet is pressed by the liquid interface (1) using screws (5) against the sample stage (6). The liquid is introduced via a tube (7), passes a reference electrode (8), fills the capillary (4) and proceeds to the outlet (9). The devices we obtain are normally-on n-channel FET devices with currents in the sub-milliampere regime (**Fig.2b**). To prevent from leakage currents through the electrolyte via the ultra-thin native oxide the devices have to be operated at comparatively low voltages.

3 RESULTS AND DISCUSSION

Based on a newly developed theoretical model for Field-Effect liquid movement detection [8] we demonstrate fundamental applications like the detection of capillary filling speed and level. The model is based on the assumption that the specific resistance of the silicon thin film is changed from ρ_{Air} when exposed to air to ρ_{Liquid} when exposed to liquid. For the source-drain-channel of a FET device this is true if the gate voltage is large compared to the source-drain-voltage. This requirement is fulfilled with the gate voltage either being the liquid potential or the surface potential at the native oxide-air interface and a low source drain voltage of typically 10mV. The basic idea of flow detection with FET is that the sensor current changes with time as the liquid wets the gate or sensitive area formed by the native oxide. The model prediction that the drain-current as the sensor signal of the device is directly proportional to the capillary filling level is proven by our

experiments under static [8] and dynamic (**Fig.3a**) conditions. Interestingly direct proportionality is just obtained if liquid flow direction is vertical to electrical current flow direction. Furthermore the detection of the capillary filling speed and the impact of the capillary properties will be discussed [8]: Contrary to intuition we find that the flow speeds measured with the FET sensor are different from flow speed measurements using a reference tube. We propose this is due to the difference of continuous flow in a completely liquid filled system and the filling process of a system. While in the first case the pressure is constant in the system in the second case the pressure changes with time especially at material interfaces which causes delays measured by the FET. Thus the devices might have the capability to probe the wetting properties of the capillary which are influenced by wall roughness and surface chemistry. For such applications it is no longer sufficient to measure the average filling speed but to analyze dv/dt . As FET size has reached submicron dimensions and does not require transparent materials this might be the only way to monitor or probe submicron channels in multilayer systems in situ.

As a unique feature provided by the use of silicon-on-insulator substrates we show how the sensitivity of the devices can be tuned by an additional backgate (**Fig.3b**). For this experiment we change the liquid potential by applying different voltages to a reference electrode introduced into the liquid and record the device current at different backgate voltages. The change of the liquid potential causes a potential change at the native oxide surface which is similar to a potential change caused by a change of the liquid composition or a binding event at the surface. It is clearly visible that device sensitivity increases with increasing negative applied potential and is more sensitive to positive potential changes.

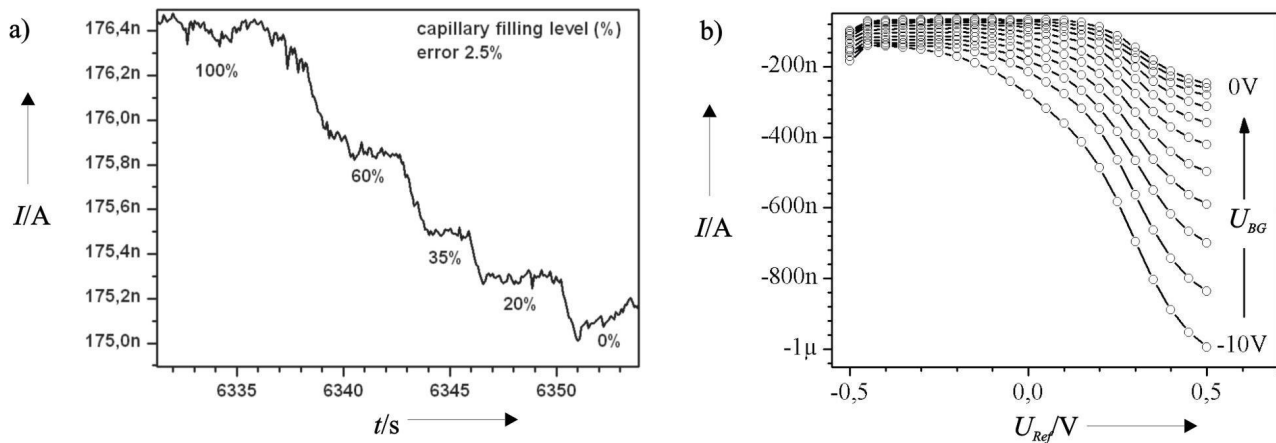


Figure 3: a) [8] Detection of capillary filling level. Device current I versus time t is recorded while a full capillary with an integrated sensor is stepwise emptied, with a pipette as a pump. b) Tunable sensitivity. The more negative the backgate Voltage U_{BG} the stronger the device current I depends on surface potential or reference potential changes U_{Ref} .

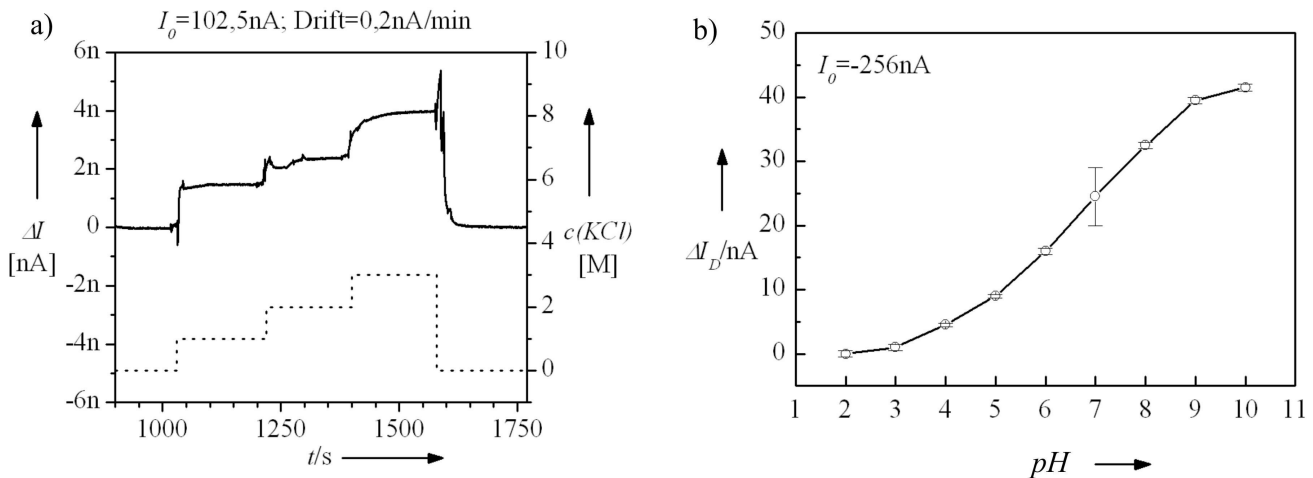


Figure 4: a) [8] Ionic Strength. The device current I versus time t is recorded while the concentration of KCl is changed. b) pH sensitivity. Device current I versus as a function of pH. The maximum sensitivity is achieved from pH 5 to pH 9.

To proof compatibility with ISFET technologies we demonstrate chemical composition analysis capabilities like pH-detection and sensitivity to ionic strength. The sensitivity to ionic strength is determined by recording device signal as a function of KCL concentration from 0M to 3M in phosphate buffered saline solution (Fig.4a). In this concentration regime the concentration is measured with a resolution of 0.3M. The native oxide surface in the buffer solution becomes charged by the dissociation of the OH-groups at the native oxide surface and by adsorption (binding) of alkali ions. Next to the surface the diffuse electric double layer builds up due to mutual repulsion similar to the accumulation of mobile charges on the surface of any charged conductor. The surface potential at the solid-liquid interface not only depends on the surface charge but as well on the nature of the electric double layer which depends on the ionic strength. This relationship is described in the GCSG (Gouy, Chapman, Stern, Grahame)

model [6]. pH changes can be measured from pH = 2 to pH = 10 with a resolution of $\Delta\text{pH} = 0.2$ (Fig.4b). The sensitivity to pH changes is described by the site dissociation theory [7] where the dissociation constants of the surface groups (OH, O⁻, OH²⁺) are used to calculate the surface charge as a function of pH which is included into a linearized version of the GCSG model. The pH sensitivity of our device is reduced compared to the theoretical value due to device drift and noise.

4 CONCLUSION

In conclusion we developed a sensor that can be used to study the influence of geometry, surface topography and chemical composition on the liquid movement in capillaries—a major topic of fundamental research in the field of micro- and nanofluidics. In the future, arrays of such devices may also contribute to the fundamental

research on the movement of liquid fronts on large scales with high spatial resolution. In lab on a chip systems, the new sensor devices are ideal candidates to monitor liquid transport, as well as analysing the liquid composition. Droplet based lab on a chip systems seem to be especially suitable for applications of the new sensor, since the droplets themselves can be used as tracking objects and moving liquid at the same time, so that even in full capillaries the detection principle is applicable. Furthermore, the new sensor is reusable because gold covered metal contacts and silicon dioxide surfaces combined with appropriate passivation layers allow intensive cleaning procedures. Compared with one-dimensional arrays of ISFETs which might be suitable for comparable tasks the single thin film FET provides the following advantages:

1. The spatial resolution is high and not limited by the size of the device.
2. The device integration is simple as just two electrical contacts and corresponding wires are required.
3. The device sensitivity is improved since the aspect ratio makes lower current densities sufficient.
4. Liquid flow is less influenced by channel wall heterogeneities caused by the integration of individual sensing elements into one channel.
5. The device is more flexible in lateral shapes.
6. Due to a reduced number of sensors, data processing speeds up and time resolution becomes higher.

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