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Online measurement of mass density and viscosity of pL fluid samples with suspended microchannel resonator

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ABSTRACT

Physical characterization of viscous samples is crucial in chemical, pharma and petroleum industry. For example, in the refining industry of petroleum, water percentage is verified by measuring the density of a sample. In this article we present a suspended microchannel resonator (SMR) which uses 5 pL of a fluid sample and measures its density with a resolution of 0.01 kg/m^3 and a sensitivity of 16 Hz/kg/m^3. The resonator can also simultaneously measure viscosity of the solutions with an accuracy of 0.025 mPa s. The SMR is part of a system which contains packaging and tubing to deliver samples to the resonator. The system can easily handle multiple viscous fluids to measure their densities and viscosities. The SMR is transparent, facilitating visual inspection of the microchannel content.

1. Introduction

Density and viscosity are known as important physical properties of fluidic samples in pharmaceutical, chemical and petroleum industries. For example, in pharmaceutical plants the concentration of a solution is determined by its mass density. Since drug samples are generally quite expensive, only small amounts are available for characterization. In petroleum refineries as well as in the automotive industry, oil analysis requires very accurate density measurements to ensure required lubrication and water percentage. For characterization purpose, oil samples are available in large quantities but they are viscous and difficult to handle. Microfluidics is already finding applications in analysis of crude oil at microscale [1,2].

For a variety of applications, density sensors have been reported, dealing with fluids like; alcohols [3,4], water/propanol mixtures [5], sucrose/water solution [6], glycerol [7], olive oil [8], slurries [9], various alkane compounds [10], sulphuric acid [11] and single biological cells [12]. All of these works present sensors that either; need large volumes of a sample, cannot handle a variety of samples or require to be fully immersed in a fluid to measure its density or viscosity. Immersing for example a cantilever sensor into a fluid highly degrades the performance of the device.

Along with the density, knowledge of viscosity is also of great interest for applications such as lubricating oils [13], rheological behaviour of paint [14], analysis of polymers [15] and milk analysis [16]. Some of the reported micro viscosity sensors are; AFM cantilevers [17], double clamped beams [18] and plain cantilevers [19,20]. Recently Livak-Dahl et al. reported a nanoliter viscometer [21] where the viscosity of a sample is measured by observing a flow rate of a sample. In this approach, prior information like surface tension and miscibility of a sample are required.

Burg et al. have reported a suspended microchannel resonator (SMR), which is basically a cantilever resonator with an integrated microchannel, and used it as a density [22] and viscosity sensor [23]. However, in this earlier work, the SMR was only used to characterize a single type of viscous liquid and the microchannels were opaque impeding visual inspection. Using SMR, the density of liquid is extracted from a change in resonant frequency of the SMR. This principle of measuring density has already been demonstrated in the form of resonating U-tubes [24] and suspended resonator plates [25]. The resonating tubes are also currently being used in commercial instruments for density measurement [26]. Macro sized U-tubes provide very precise density results. But they might not be economical in industries where even small quantities of samples are relatively expensive. The SMR and suspended resonator plates have presented promising results but their complicated fabrication makes them expensive at a commercial scale. Also, in most cases a new sensor needs to be used for every viscous sample, since it is not possible to empty the sensor between measurements. This might introduce errors due to small variations in sensor performances.

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Here we report a SMR with a transparent microfluidic channel designed to handle multiple viscous samples. The SMR has a structural mass of 12 ng and a sensitivity of 16 Hz/kg/m^3. We have observed that the transparency of the microchannel provides an advantage of visual inspection for the presence of air bubbles, blocked channels and remains of previous samples in the channel. The SMR can provide a cost effective solution for repetitive measurements by employing a single chip to be used for multiple samples.

In our experiments we evaluated the ability of a single SMR to handle viscous sticky samples of crude oil which are often tricky to handle in microfluidic devices. To measure density and viscosity, four samples of interest were identified; Isopar-L, Isopar-V, Oil-SA and Oil-D [27]. The later two samples were completely unknown with no prior information available on their physical properties. In addition, we tested water, isopropanol and ethanol. With a volume of 5 pL, the SMR measured the mass density of the loaded liquid with a resolution of 0.01 kg/m^3. It was fabricated using MEMS technology and made of silicon nitride. A measurement chip containing a single SMR, was packaged in such a way that the sample of interest was introduced from the bottom of the chip while vacuum and optical access were provided from the top. This technique allowed a quick exchange of chips and fast pumping of an integrated small vacuum chamber.

2. Device design, fabrication and packaging

A schematic drawing of the sensor chip is shown in Fig. 1a. The SMR is located in the center of the chip. The cantilever resonator is 20 μm wide and 200 μm long with fluidic-channel dimensions of 4 μm × 3 μm. It is connected by larger sample delivery channels (SDC) to access-through holes connecting the backside of the chip to macro sized fluidic tubing. Each SDC is 150 μm wide and approximately 2300 μm long.

The fabrication of the SMR was done by a combination of surface and bulk micromachining of a silicon wafer. Low-stress silicon-rich silicon nitride was used as a structural material while polysilicon was used as a sacrificial material to create the hollow microchannel. Both materials were deposited by chemical vapour deposition, patterned by contact photolithography, and structured by dry and wet etching. Most of the fabrication is explained in our previous work [28]. In the current devices, the etching of the access holes and sacrificial material was done simultaneously by an 18 h KOH-etch. The release of the SMR, etching of SDCs and chip boundary was achieved in a single step of KOH etching. A single 4-in. wafer contains 52 sensor chips with a size of 10 mm × 10 mm. Fig. 1b shows the topview of a finished sensor chip. In Fig. 1c a fully etched silicon nitride microchannel is depicted.

The packaging consisted of a polyetheretherketone (PEEK) fixture at the bottom and an aluminium lid at the top of the chip. A schematic of the packaging is shown in Fig. 2. The PEEK fixture provided microfluidic connections, a piezo-actuator, and a temperature sensor. The aluminium top lid provided vacuum and contained a window for an optical readout. The sealing between the PEEK fixture and the sensor chip was provided by a 300 μm thick polydimethylsiloxane layer (PDMS). The PDMS-seal was prepared by a 10:1 ratio of elastomer and curing agent from "Sylgard 184 Silicone Elastomer Kit". Afterwards, a CO^2-laser was used to pattern the PDMS-seal into square pieces equal to the size of the sensor chip. The patterning by laser created a lot of particles which got stuck on the seal. Therefore, the PDMS-seal was sonicated in an ethanol solution for 30 min to remove particles. Later it was dried for 48 h at room temperature. The fluidic samples were provided to the SMR chip by Teflon tubing (inner diameter: 1 mm). The sealing between the chip and the aluminium lid is provided by an o-ring

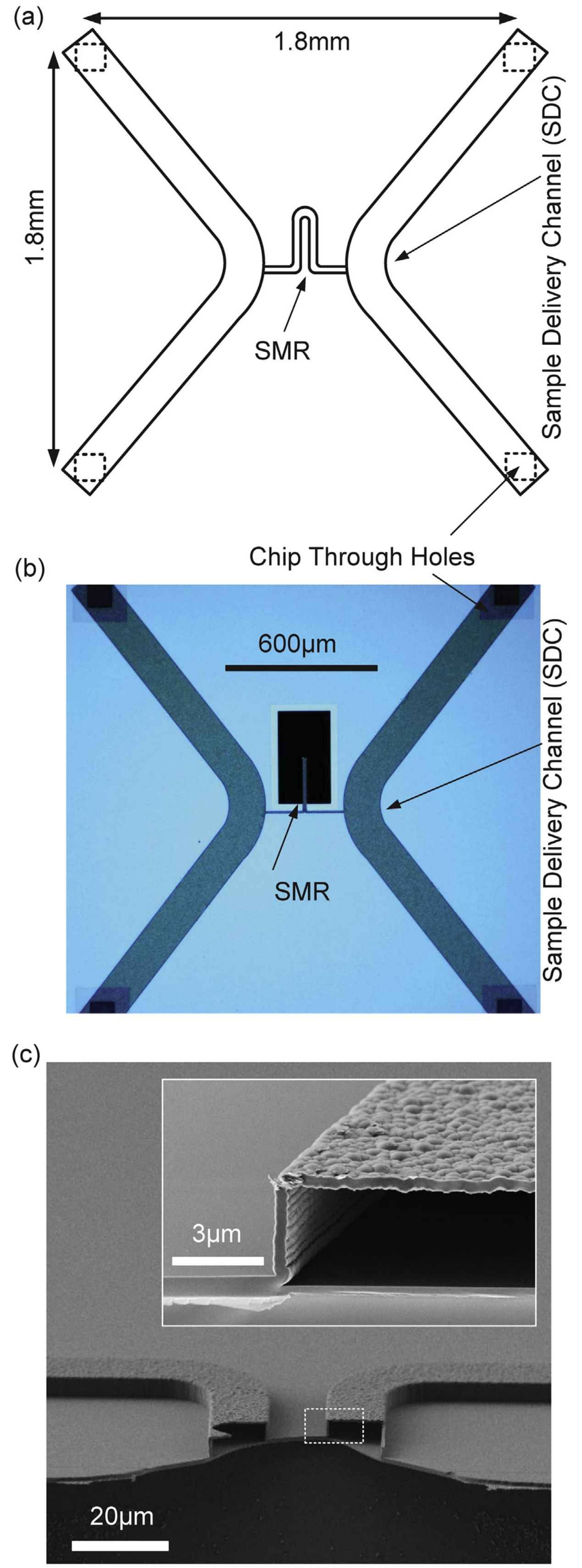

Fig. 1. (a) Schematic drawing of a chip with the suspended microchannel resonator in the center. (b) Optical microscope view from the top of sensor chip. (c) Scanning electron microscope image of microchannel cross section.