SILICON RICH SILICON NITRIDE MICROCHANNELS TO DETERMINE FLUID COMPOSITION BY NEAR INFRARED ABSORBANCE

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ABSTRACT

The aim of multiparameter microfluidic sensors is to determine multiple physical and chemical properties of liquids and gases at a microscale on a same chip platform. Silicon-rich silicon nitride (SiRN) microfluidic channels fabricated with MEMS surface channel technology (SCT) are currently used in multiparameter sensors to detect physical parameters like mass flow rate, density, and pressure with high sensitivity. However, the current platform has limited information on sensing chemical parameters like fluid composition for mixtures of more than two different fluids when using traditional physical methods. The growing interest in integrating optics to microfluidic devices for such measurements has led to an attempt to integrate optics in the current multiparameter sensing platform. Here, we demonstrate absorbance measurements using the Beer-Lambert law for four different methanol in water mixtures in a microfluidic device fabricated with SiRN microchannels using coupled multimode optical fibers. We compare the measured transmission spectral response with the theoretical response of methanol-water mixtures. The absorbance measurements show the feasibility of integrating optical methods at near infrared wavelengths in the state-of-the-art multiparameter flow sensor technology platform.

KEYWORDS

SiRN, surface channel technology, microfluidic channel, near infrared, absorbance, multiparameter sensor, spectral response, flow sensors, optical fibers

INTRODUCTION

Existing multiparameter microfluidic flow sensors, such as micro-Coriolis mass flow meters [1], fabricated using Surface Channel Technology (SCT) with silicon-rich silicon nitride (SiRN) channel walls, have been used to measure physical parameters of the fluid, such as density, mass flow rate, and pressure by capacitive and resistive methods. These measurements are made possible by allowing mechanical moment and deformation of suspended microfluidic channels, see Fig. 1 [1, 2]. To extend the possibilities for fluid composition detection there is a need to integrate additional measurement methods.

Optics-integrated microfluidics systems can provide fast, label-free, and non-invasive concentration measurements. In many cases, absorbance measurements are used to quantify the molecules in a solution which absorb or transmit light at specific wavelengths [3]. This operating principle is realized by either integrating optical fibers or by optical waveguides [3-5]. Incorporating optical fibers into a microfluidic chip is a well-known approach to minimize the number of required optical components [4]. The most common microfluidic devices are based on polymeric substrates, e.g., polydimethylsiloxane (PDMS), cyclic olefin copolymer (COC), and polymethylmethacrylate (PMMA), due to their durability and the high flexibility in terms of design and fabrication [3-6]. However, these cannot be integrated with existing devices fabricated in SiRN surface channel technology.

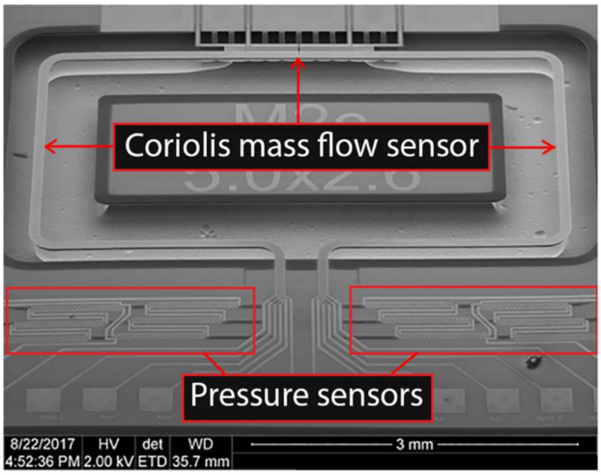

Figure 1: State of the art multiparameter Coriolis mass flow sensor with integrated pressure sensor fabricated in SiRN surface channel technology [1, 2].

In this paper, we present the basic structure of a fabricated SiRN microfluidic device and the applied absorbance principle is explained. The theoretical transmission spectral response (TSR) is estimated based on the absorption of different mixtures of methanol and water inside the microchannel. The process flow to fabricate SiRN microchannels with integrated optical fibers using surface channel technology is described. The experimental test bench using optical fibers to investigate the absorbance is described and measurement results are discussed.

BASIC STRUCTURE AND OPERATING PRINCIPLE

The basic structure of a SiRN microfluidic channel with inlets, outlets, and integrated optical fibers is illustrated in Fig. 2. Two multimode optical fibers with diameter of 60 µm are aligned at each end of the microchannel in the direction of the flow. Using optical fibers has been the most common technique to measure in-plane absorbance in microfluidics devices [6]. The optical properties of 2 µm thick SiRN layer on a Si substrate wafer, measured with ellipsometry are shown in Fig. 3. This shows that the SiRN channel wall material has negligible absorption at near infrared wavelengths. For realizing the absorbance principle, an optical fiber with an input

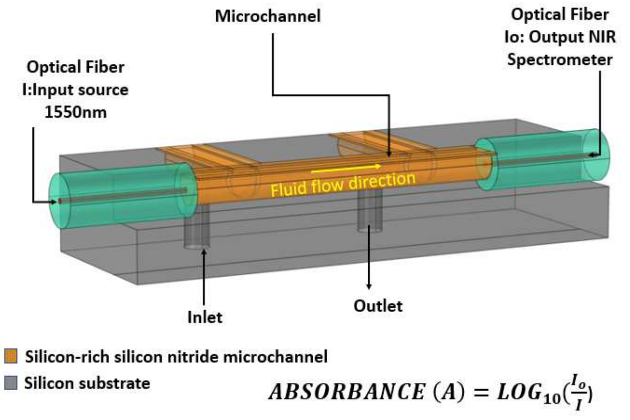

Figure 2: The basic structure of SiRN microchannel for absorbance measurements.

intensity (*I*) at near infrared wavelength is aligned to one end of the microchannel and the other end is aligned to an output optical fiber. The spectrum intensity *(Io)* in the output fiber is measured using a spectrometer for different fluid mixtures flowing in the SiRN microchannel. The absorbance at a given wavelength is calculated by the Beer-Lambert law:

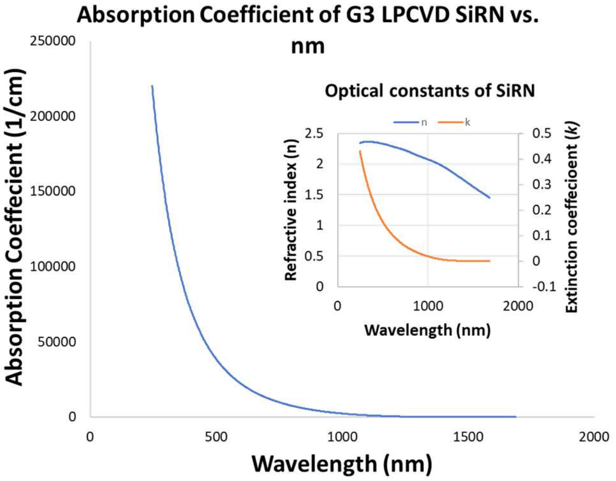

Figure 3: Absorption coefficient of SiRN, Refractive index (n) and Extinction coefficient (k) using ellipsometry.

$$Absorbance = Log_{10}\frac{I_0}{I} \tag{1}$$

The attenuation of the transmitted light due to fluid absorption is calculated at each considered wavelength [8]:

$$T_{(sample\ or\ reference\ )}(\lambda) = e^{-\alpha(\lambda)x} \tag{2}$$

where, *x* is the path length, or the distance travelled by the light in the absorbing medium at that wavelength and α(λ) is absorption coefficient, expressed as,

$$\alpha(\lambda) = \frac{4 * \pi * k(\lambda)}{\lambda} \tag{3}$$

where *k* is the imaginary part of the refractive index of the fluid to be determined. Its behavior as a function of the wavelength is a characteristic 'fingerprint' of each molecule [3-5].

For this paper, we determine the absorbance of methanol concentrations in water at 1540 nm wavelength in order to investigate the suitability of SiRN microchannels for this principle. The choice of wavelength comes from the absorption band of the methanol-water mixture, as the O-H stretching overtone has a higher absorbance close to 1540 nm [7]. Calculated concentrations of the methanol and water mixture can be determined from the refractive index database. The transmission spectral response (TSR) of the output light is given by:

$$TSR(\lambda) = \frac{T_{sample}(\lambda)}{T_{reference}(\lambda)} = \frac{P_{(mixture)}(\lambda)}{P_{(100\%\ Methanol)}(\lambda)} \tag{4}$$

which is the ratio of the detected power for the methanol and water mixture to the detected power for 100 % methanol. The latter is used as reference since the transmission of light through methanol is higher than that of DI-water at 1540 nm. The theoretical transmission spectral response (TSR) of the mixture at near infrared wavelengths through the SiRN microchannel is plotted in Fig. 4.

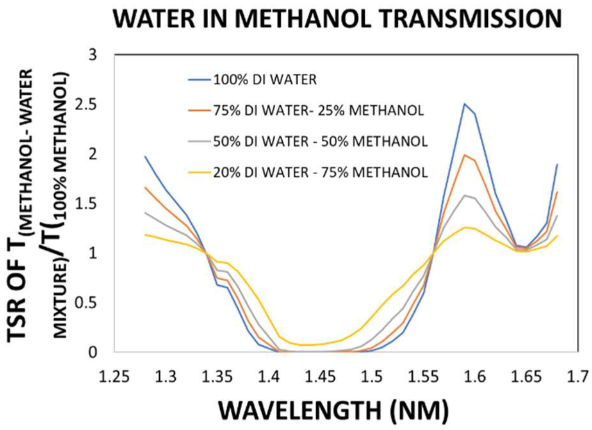

Figure 4: Theoretical transmission spectra of methanol-water mixture concentrations at near infrared wavelength

FABRICATION PROCESS

The fabrication process is based on the existing surface channel technology (SCT) [2]. The simplified process is illustrated in Fig. 5. First, 500 nm thick layers of low-stress silicon-rich silicon nitride (SiRN) and silicon dioxide are deposited by low pressure chemical vapor deposition (LPCVD). The latter serves as hard mask for the etching of rows of slits with dimensions of 5 x 2 µm. All etch processes are plasma based. Next, the microfluidic channel is etched through the slits and the silicon oxide layer is removed, see Fig. 5b,c. Inlets and outlets are etched from the backside of the wafer while the channel wall is temporarily protected by a silicon oxide layer, see Fig. 5d,e. The fabrication of the channels is completed by LPCVD of a second layer of SiRN which closes the slits, see Fig. 5f. In addition to these standard steps, we etched fiber grooves in the Si substrate with SF_6 plasma (isotropic), see Fig. 5g,h, which also exposes the end of the SiRN channel wall. Figure 6 shows photographs of the fabricated chip, which contains SiRN microchannels with several path lengths of 5000 µm, 4000 µm, 3000 µm.

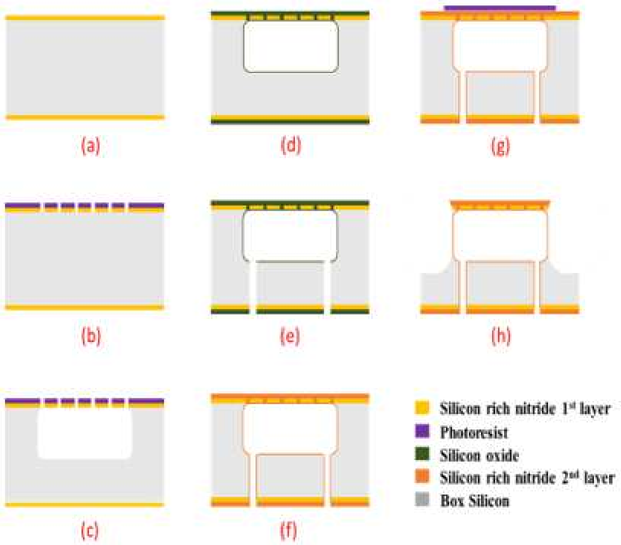

Figure 5: Fabrication process flow of silicon-rich silicon nitride microchannel, (a) LPCVD of SiRN 1st layer, (b) etching of the slits, (c) etching of the microchannel, (d) LPCVD of silicon oxide, (e) backside etching for inlets and outlets, (f) LPCVD of SiRN 2nd layer, (g) photoresist mask layer for release etch, (h) release etch for optical fiber grooves.

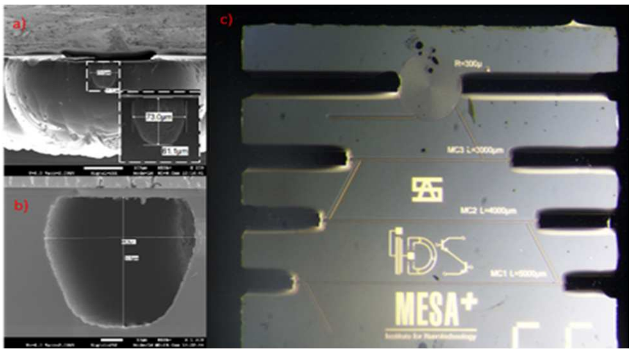

Figure 6: Fabricated device (a) SiRN end wall seen from the fiber groove, (b) cross-section of the SiRN microchannel with a diameter of approximately 65µm, (c) microscopic image of the fabricated device

EXPERIMENTAL SETUP

The schematics of the optical test bench is illustrated in Fig. 7a. The device is mounted to the same driving PCB used to measure the Coriolis mass flow sensor [1]. The optical fibers are aligned at the 4000 µm microchannel in the device, see Fig. 7b. A Thorlabs multichannel 1550 nm single wavelength source with spectral resolution of 15nm with 1 mW output power is used for the input optical fiber (*I*) and an Avantes spectrometer is connected to the output fiber, which measures the spectrum and output intensity (Io). Syringes of 3ml volume are used to prepare solutions of four different methanol and water mixtures with ratios of 0:100, 25:75, 50:50, 75:25, and 100:0. The solutions are supplied to the microchannel through a syringe pump with a constant flow rate of 20 µl/min. Once the fluid enters the channel, the syringe pump is turned off and static measurements are taken at an exposure time of 50 ms of the spectrometer. The exposure time is chosen depending on the sensitivity of the CMOS sensor which is integrated in the spectrometer for a small change in absorbance for DI water (reference measurements). For all measurements, 100% (DI) water is used as a reference to the input intensity (I). Three different data sets (Io) are taken to test the repeatability of the absorbance with respect to the change in concentration under the no-flow condition.

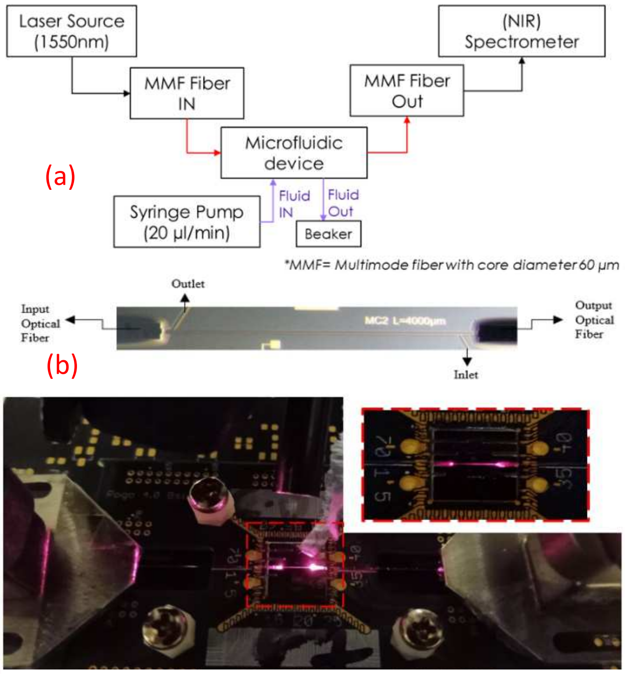

Figure 7: Experimental setup (a) the SiRN microfluidic device for absorbance measurements, (b) optical fibers aligned to the 4000 µm microchannel.

RESULTS AND DISCUSSION

The measured NIR spectrum without flow, shown in Fig. 8, clearly shows the dependence of the output spectrum on the fluid concentration. From the results, it can be seen that water has higher absorbance than methanol at 1540 nm, whereas methanol has shown higher transmission when compared to water at 1540 nm. Measurements were repeated for three times for each concentration and the respective syringes containing the fluids were changed for

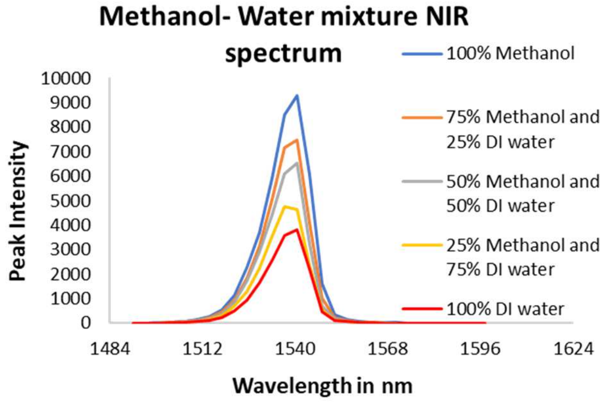

Figure 8: NIR spectrum of dataset 3 under static/no flow at 1540nm absorbance of methanol-water mixture concentration

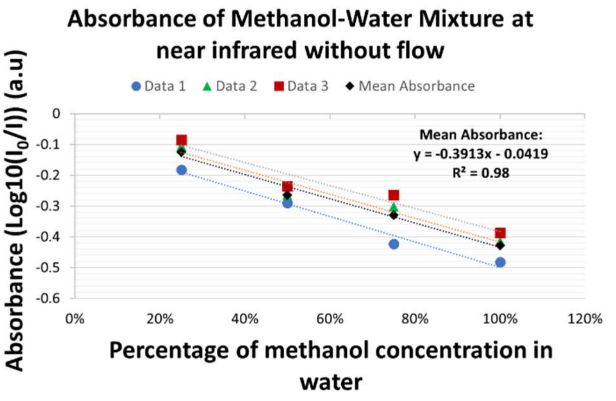

Figure 9: Absorbance of three data sets under static/no flow conditions at 1540nm of methanol-water mixture concentrations

every data set. The absorbance of three data sets with different concentrations of methanol in water are plotted in Fig. 9. The deviation in the results can be due to misalignment of the optical fibers or due to the scattering of light at the source end of SiRN microchannel wall. The mean of the data sets have shown a linear fit of $R^2=0.98$. The reason we see the absorbance in a negative value, is mainly due to referencing the measurements with DI-water. The spectral response *TSR* is also calculated from the measured (mean) NIR spectrum and is compared with the theoretical *TSR* values, see Fig. 10.

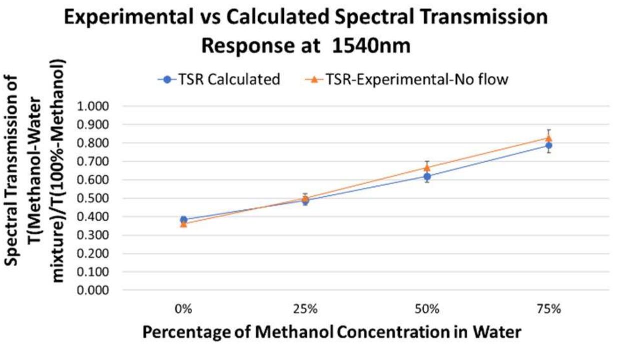

Figure 10: Transmission spectral response (TSR) of methanol concentration in water. Calculated vs experimental (mean) response without flow.

CONCLUSION

From the results it can be concluded that fabricated SiRN microchannels coupled with optical fibers are a feasible option for integrating optics in a multiparameter sensor platform and for measuring the fluid concentration using the absorbance principle. The proposed surface channel fabrication technology allows the optical coupling of fibers to the microchannel, allowing one to measure the transmitted light through the liquid flowing in the channel at near infrared wavelengths. On the other hand, limitations exist for using optics with SiRN microchannels in the UV or visible spectrum for absorbance measurements, since the light will be strongly absorbed by the material itself.

We plan to improve the alignment of the fibers to the microchannel either with epoxy or UV glue to avoid drift during measurements. Furthermore, the design needs to be extended for composition detection of mixtures of three or more liquids with a broad NIR spectrum range under flow conditions. Ideally the fabrication process should be adapted so that both the existing SCT based microfluidic sensor devices and the microchannels for concentration measurements using optics can be integrated on the same chip for multiparameter sensing applications.

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