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Photothermal Microfluidic Chemical Sensor Made by Integrated Optics on Glass

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Abstract—In this article, we describe the realization of a photothermal integrated optical sensor for chemical analysis, based on a Young interferometer made by Ag/Na ion-exchange on glass. An excitation beam is used to generate a phase shift proportional to the analyte concentration. Tests were conducted with a solution of cobalt in ethanol. The detection limit was evaluated to be 4×10^{-3} M with a probed volume of (33.5 ± 3.5) pl.

Chemical analysis, Photothermal sensors, integrated optics on glass, ion exchange, nuclear fuel reprocessing, borosilicate glass, wafer bonding, microfluidics.

I. INTRODUCTION

The reduction of the volume of chemical samples or chemical wastes required for analysis is an important feature to be addressed in sensor design. This is particularly important when the chemical or radiological hazards connected to the nature of the fluids are of major concern, such as in the nuclear fuel reprocessing cycle. For this reason, we discuss here a photothermal chemical sensor [1] fabricated by co-integrating microfluidics with waveguides made by ion-exchange on glass [2].

This paper is organized as follows. At first, a brief description of the sensor working principle is given, followed by the description of its realization. Then, the sensor is tested using solutions of cobalt dissolved in ethanol. A discussion of the detection limit is given at the end of the article.

II. DESCRIPTION OF THE SENSOR

Fig. 1 schematically depicts the proposed sensor. The working principle is based on a Young interferometer built around an integrated Y-junction. It is made with single mode optical waveguides on a Borofloat 33 glass substrate by Ag/Na ion-exchange [3, 4]. At the two outputs of the Y-junction, the light passes through a microchannel containing the solution to be analyzed. The probe wavelength used in the interferometer is $\lambda_p=980$ nm, which is neither absorbed by cobalt nor by ethanol. A pulsed excitation beam at $\lambda_e=532$ nm is brought by an optical fiber vertically in front of the interferometer probing arm. The excitation wavelength, on the other hand, is chosen to coincide with an absorption line of cobalt. The coherent light at 980 nm coming from the two arms of the interferometer creates an interference pattern. When absorption is weak, it can be

shown that the excitation beam absorption leads to fluid heating which is proportional to the analyte concentration. Through the photothermal effect, the fluid temperature increase determines a change in the fluid refractive index, which leads to a fringe phase shift at the output of the interferometer. After each pulse, a synchronized camera acquires the image of the optical fringes just after an adjustable delay (typically 1 μ s). This approach has the advantage of separating the spectrometric signal from thermal diffusion, 2 or 3 orders of magnitude slower than the spectrometric signal. In spite of its simplicity, this system has several advantages, such as the fact that the measurement is intrinsically differential. This feature increases the robustness to variable and potentially uncontrollable fluid properties. A second advantage is that the structure is compatible with the measurement of a continuous flow, as long as the fluid movement is slow with regard to the acquisition time.

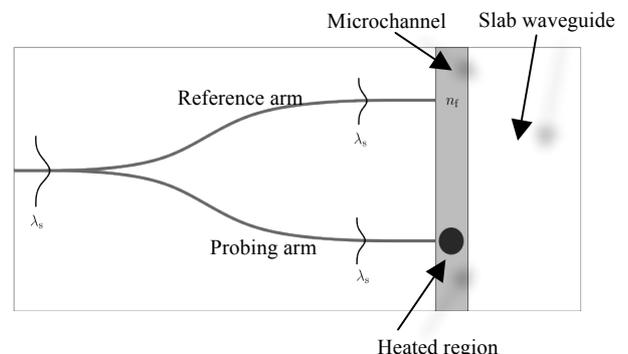


Figure 1. A scheme of the proposed integrated Young interferometer. The fringes produced at the right facet are collected by a microscope objective and focused on an infrared camera.

III. DEVICE FABRICATION AND CHARACTERIZATION

The presented device was fabricated with a two-step ion-exchange process on a Schott Borofloat 33 (BF33) substrate. This glass was chosen for its chemical robustness, its low cost and its compatibility with the Ag/Na ion-exchange process. However, the low sodium content of the glass limits the maximum refractive index increase to 0.02, which is sufficient for the fabrication of waveguides. The optical chip was

fabricated by a $\text{AgNO}_3/\text{NaNO}_3$ ion-exchange at a molar ratio of 0.8 and at a temperature of 353°C for 14.5 min. A second step was done by applying a 360 kV/m electric field for 100 min, while the sample was kept at 260°C in a $\text{Mg}(\text{NO}_3)_2$ and NaNO_3 mix. This allows burying the waveguides inside the substrate at a depth of $(4.9 \pm 0.9) \mu\text{m}$ from the surface. The microchannel was then fabricated using a micro dicing tool. The obtained depth and width were respectively $180 \mu\text{m}$ and $235 \mu\text{m}$. A BF33 cover was then bounded onto the substrate by molecular adhesion. A groove aligned to the channel was fabricated in the cover, to the vertical fiber $200 \mu\text{m}$ above the channel.

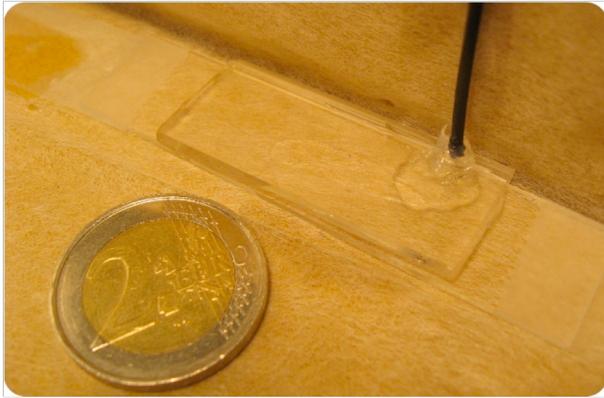


Figure 2. A photograph of the sensor with the excitation fiber glued.

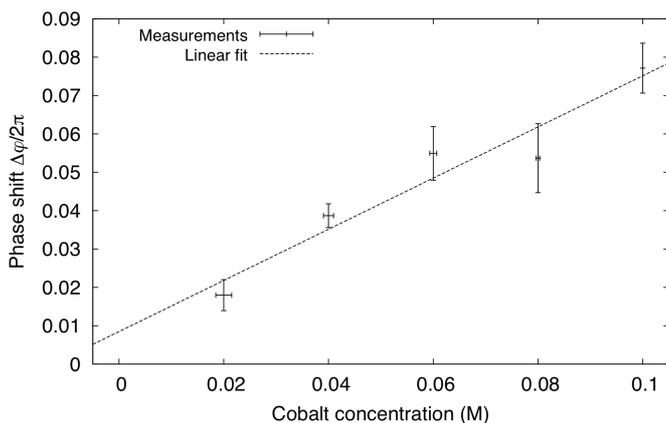


Figure 3. The phase shift versus the molar concentration of cobalt in ethanol (range 0 to 0.1 M). Excitation laser pulses of $(500 \pm 20) \mu\text{J}$.

Fig. 2 shows the sensor with the excitation fiber vertically aligned on one arm of the interferometer and glued into the cover groove. Another pigtailed fiber (not shown) was used to inject the 980 nm probe light. A microscope objective was then used to focus the light coming from the interferometer on a CCD camera. The fringes obtained when the excitation laser is switched on and off are acquired. Then, the phase shift is evaluated by means of a comparison between the FFT calculations of the two fringe images.

Fig. 3 and 4 show the measurement results obtained by varying the molar concentration of cobalt in ethanol, with a Y-junction separation of $100 \mu\text{m}$. During the measurements

shown in Fig. 3, the position of the excitation fiber was optimized and the measurements of Fig. 4 were taken after the glue had been hardened. The error bars on the x-axis represent error margins for the cobalt concentration, estimated during the preparation of the test solutions. For each point, six measurements were made from which we estimated the sample mean x and the standard deviation σ , assuming independent and identically distributed measurements. If we further assume that the samples are normally distributed, we can use Student's t-distribution to compute a confidence interval for the estimated sample mean, in our case, the estimated mean phase shift. The vertical error bars correspond to a 3σ interval. In this configuration, the detection limit was evaluated as $4 \times 10^{-3} \text{ M}$. The probed volume is defined as the intersection between the probing and the excitation beam and was evaluated to be $(33.5 \pm 3.5) \text{ pl}$.

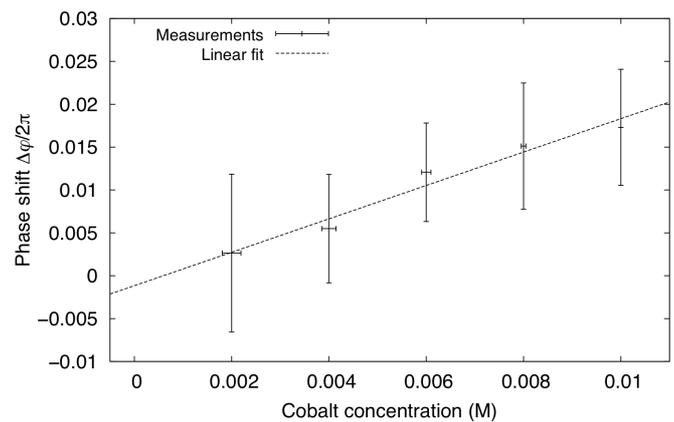


Figure 4. The phase shift versus the molar concentration of cobalt in ethanol (range 0 to 0.01 M). Excitation laser pulses of $(500 \pm 20) \mu\text{J}$

IV. CONCLUSION

In this article, we have presented an integrated sensor for chemical analysis made by combining ion-exchange integrated optics on glass with a microfluidic channel. The sensor is based on an integrated Young interferometer working at 980 nm, where the phase shift is photothermally induced by the absorption of 532 nm excitation laser pulses in a solution of cobalt in ethanol. The detection limit was $4 \times 10^{-3} \text{ M}$ with a probed volume of $(33.5 \pm 3.5) \text{ pl}$.

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