In-line absorption sensor based on coiled optical microfiber

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We fabricated and tested an evanescent-wave absorption sensor consisting of an optical microfiber coil resonator embedded in fluidic channel walls. Low concentrations of flowing analyte show optical losses in agreement with a modified Beer–Lambert law. Higher concentration causes a limit value of the measured optical losses arising from adsorption mechanisms. © *2011 American Institute of Physics*. [doi:10.1063/1.3582924]

Optical fibers play a prominent role in sensor technology, mostly due to portability, immunity to electromagnetic interference, possibility to work in remote and distributed networks, robustness, and compactness. Among the numerous geometries and devices proposed in the past years, microfibers and microfiber coil resonators represent an innovative approach with regard both to their properties and applications.[1–3] The main advantage of such systems relies in the evanescent-wave (EW) propagating outside the physical boundaries of the waveguide and interacting with the surrounding environment. Moreover, coiled microfibers enable light tunneling between adjacent turns thus forming optical resonator with Q-factors greater than 10^5.[4]

Silica microfiber sensors[3] have been tested over a variety of external stimuli such as refractive index,[5,6] humidity,[7] temperature,[8] strain,[9] and electric current.[10] Polymer microwires have also been proposed because of their porosity to many gases.[11] Yet, the reported devices involve the immersion of the active portion of the fiber in the fluid to be analyzed;[11–13] thus limiting potential applications in microfluidic and lab-on-a-chip technology.

In this letter we report an in-line absorption sensor, where the analyte flows in a fluidic channel whose walls are made of absorption-responsive coiled microfibers. The device fabrication includes four steps; (1) tapering a standard optical fiber by the modified flame brush technique (Corning SMF-28, final diameter $\phi=2.5$ μm, length of uniform waist region $L=9$ mm), (2) coiling of the tapered fiber onto an expendable support rod ($\phi=1$ mm, 2 mm length) of polymethylmethacrylate (PMMA), which connects two Teflon tubes ($\phi_{Int}=1$ mm, $\phi_{Ext}=1.2$ mm), (3) embedding the PMMA rod and the coiled tapered fiber with a Teflon solution (DuPont 601S1-100-6) and curing at 80 °C for 20 min, and (4) dissolving the support rod by dipping the device in acetone for one day. Details on the tapering and coiling processes adopted in steps 1 and 2 are described in Refs. 2–4. Fabrication strategies peculiar of the present letter (i.e., steps from 2 to 4) are depicted in Fig. 1. In order to improve mechanical stability, the device is anchored on a microscope glass slide and covered with an UV-curable acrylate polymer (Luvantix PC-373) as low-loss protective glue. The fluidic channel ends are connected by a pair of heat-shrink tubing to Teflon tubes ($\phi_{Int}=1.5$ mm), finally the device is completed with two valves and the liquid to be analyzed is inserted with a syringe.

Transmission spectra of a 2-turns device before PMMA rod removal and that of the finished device are reported in Fig. 2, using a broadband amplified spontaneous emission as light source and an optical spectra analyzer (OSA) as detector.

The embedding/dissolving process of the optical resonator retains the initial free spectral range of 0.47 nm;[4] nevertheless, the oscillation strengths are significantly weakened. This indicates that the optical path length of a single turn is not modified during the fabrication process; hence the fiber remains anchored to the fluidic channel walls. However, optical losses are introduced during the various fabrication steps and, possibly, the distance between adjacent turns can suffer slight changes, thus decreasing light coupling and resonator performances.

The device was tested measuring the optical absorption of aqueous solutions of Brilliant Blue FCF ($\varepsilon=1.3 \times 10^5$ M^{-1} cm^{-1} at $\lambda=630$ nm) (Ref. 14) with concentration ranging from 6 μM up to 0.18 mM. Optical spectra were recorded using a supercontinuum generator as light source and an OSA as detector. Figure 3 shows the comparison of solution absorption spectra, after baseline correction, recorded with the in-line absorption sensor (0.03 mM, solid black line) with respect to a reference spectra collected with a spectrophotometer (6 μM, dashed red line). Although measure is strongly affected by lamp noise fluctuations, the result is in reasonable accordance with the reference spectrum.
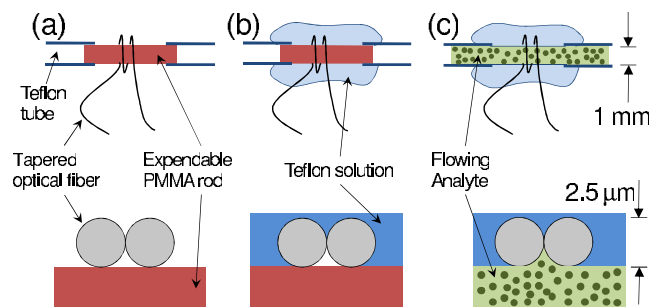

FIG. 1. (Color online) Schematic of the fabrication process; (a) coiling a tapered fiber onto an expendable PMMA rod connecting Teflon tubes, (b) covering the coil with a Teflon solution, and (c) after removing of the PMMA rod by dipping the device in acetone for one day, the analyte may freely move in the channel.

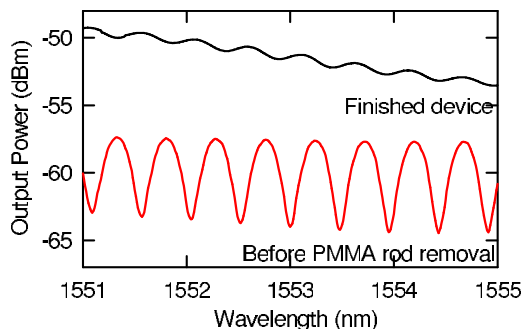a)Electronic mail: roberto.lorenzi@mater.unimib.it.

FIG. 2. (Color online) Transmission spectra of the tapered microfiber coil resonator before and after removal of the PMMA expendable rod.

In order to estimate optical losses we will refer to the Gupta model of EW absorption in optical fibers.[15] The transmitted power P of a tapered fiber of length L in contact with an absorbing medium is related to the transmitted power P_0 in absence of the absorbing fluid by a simple exponential law

$$P = P_0 \exp(-\gamma_{eff}L),\tag{1}$$

where γ_{eff} is the effective EW absorption coefficient. Assuming a uniform core fiber with a constant radius ρ, the expression of γ_{eff} is given by

$$\gamma_{eff} = \frac{\varepsilon C\lambda n_1}{\log_{10}(e)} \frac{f(\varphi_2) - f(\varphi_1)}{\sin^2(\varphi_2) - \sin^2(\varphi_1)},$$

$$f(\varphi) = \frac{(2n_{1,2}^2 - 1)\log[2\sqrt{q^2-1}+2q] - q\sqrt{q^2-1}}{2n_{1,2}^3},$$

$$q = n_{1,2}\sin(\varphi), \quad n_{1,2} = n_1/n_2,$$

$$\varphi_1 = \arcsin(n_{1,2}), \quad \varphi_2 = \pi/2,\tag{2}$$

where C is the analyte molarity, n_1 and n_2 are the refractive index of the fiber and the surrounding medium, respectively. Equation (2) yields to

$$\gamma_{eff} = \frac{\varepsilon C\lambda}{\rho}\eta,$$

$$\eta = \frac{n_2}{\log_{10}(e)2\pi(n_1^2-n_2^2)^2} \times \{n_1 n_2\sqrt{n_{1,2}^2-1} + (n_2^2 - 2n_1^2)\log[\sqrt{n_{1,2}^2-1}+n_{1,2}]\}.\tag{3}$$

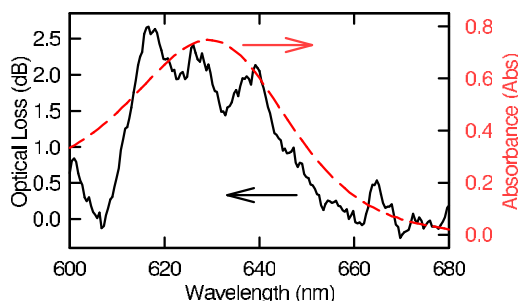Figure 4 reports the parameter η, calculated from Eq. (3), as

FIG. 3. (Color online) Comparison between absorption spectra of Brilliant Blue FCF aqueous solution measured with the proposed device (M =0.03 mM, solid black line) and reference spectrum recorded with a spectrophotometer (M=6 μM, red dashed line).
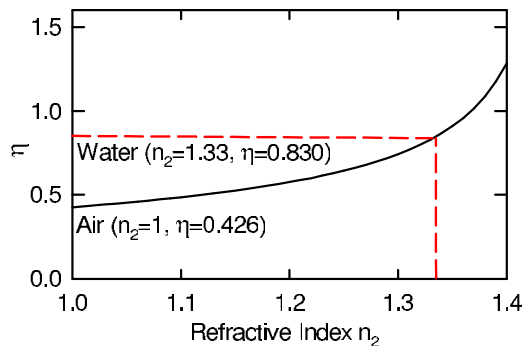

FIG. 4. (Color online) Dependence of η as a function of the refractive index of the surrounding medium for a silica tapered microfiber (n_1=1.45).

a function of the refractive index n_2 of the surrounding medium (n_1=1.45). Since the extent of the EW widens as n_2 approaches n_1; we observe that, for a fixed n_1, η increases monotonically for increasing n_2—the device responsivity is higher for analyte dispersed in higher refractive index fluid as a consequence of a larger probed volume, as an example, $\gamma_{eff}^{Air}=(\varepsilon C\lambda/\rho)0.426$ while $\gamma_{eff}^{Water}=(\varepsilon C\lambda/\rho)0.830$.

Moreover, inserting the expression for γ_{eff} in Eq. (1) one obtains a Beer–Lambert law modified by the system efficiency. The optical loss, expressed in logarithmic scale, is linearly related to fiber length, molar concentration of the absorbing analyte, and its molar extinction coefficient. Loss signal for a given wavelength varies inversely with the fiber diameter and vice versa. This latter evidence indicates, once more, that the extent of the EW governs the device sensitivity. We stress that the Gupta model is strictly valid only for tapered fibers completely immersed in a fluid. On the contrary, in our system, less than one fourth of the EW overlaps with the analyte (see Fig. 1) while the rest of the light propagates in the Teflon layer. Thus, the exponential argument in Eq. (1) should contain a "filling factor" Φ that is a measure of the ratio between the surface area of the fiber facing the channel and total active area, giving

$$P = P_0 \exp\left(-\Phi\frac{\varepsilon C\lambda}{\rho}\gamma_{eff}^{Water}L\right).\tag{4}$$

Figure 5 shows the optical loss as a function of analyte concentration. Dashed blue line represents the expected optical loss calculated from Eq. (4) for a microfiber with dimensions
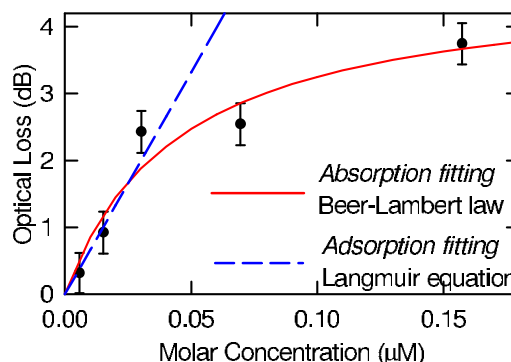

FIG. 5. (Color online) Dependence of the measured optical losses at 630 nm on the analyte molar concentration. Dashed blue line represents the fitting curve calculated by the Gupta model of EW absorption in optical fibers. Solid red line represents the fitting result assuming adsorption mechanism and applying the Langmuir equation.

consistent with our tapered fiber (length of uniform waist L =9 mm and radius ρ=1.25 μm), operating at λ=630 nm, and assuming a filling factor Φ=0.25. The linear relationship is a good approximation only for low concentrations.

Let us analyze the origin of linearity deviations observed for higher concentrations. Optical losses are much less than expected and the signal does not vary significantly with concentration. This trend is a quite common behavior in surface science and involves adsorption processes,[16] that is, the adhesion of liquids or gases to a solid surface. Such a process has been already observed and employed in EW absorption sensor made of subwavelength tapered silica fiber.[17] On one hand, the tapering process is expected to alter the silica surface by formation of defect sites and hydroxyl groups; these species enhance the interaction between analyte and substrate because of attractive forces such as van der Waals, hydrogen bonds and dipole–dipole interactions. On the other hand, light absorption takes place primarily in the vicinity of the fiber surface with the result that the adsorbed layer is the main responsible for optical losses.

At low concentrations, the optical absorption will be proportional to the number of molecules effectively dispersed in the solution; since the number of molecules attached onto the fiber surface is too small to give any contribution. On the contrary, as the molarity increases, more and more molecules attach onto the fiber surface and the resulting optical absorption is dominated by these molecules. However, the number of surface sites able to anchoring the flowing analyte is finite. Consequently, the number of adsorbed molecules will reach a critical value above which any further increment of solution concentration does not change the amount of adsorbed species. In the following, we assume optical losses originated from the fraction of adsorbed molecules rather than the total number of dispersed ones. We recall that the surface coverage Θ can be calculated by the Langmuir equation $\Theta = K\alpha C/(1+\alpha C)$, where the constant α is proportional to the binding energy of the specific adsorbate-surface pair and K is a normalization constant. The solid red line in Fig. 5 represents data fitting of $\Theta(C)$. It can

be seen that this model predicts optical losses over the entire investigated concentration.

In conclusion, we have fabricated an EW absorption sensor. The device comprises a coiled microfiber embedded in a Teflon fluidic channel, so that measurement occurs while the analyte flows in the tube. The presence of optical resonances before and after the support rod removal ensures that the original coil geometry is preserved during the fabrication process. The Brilliant Blue FCF absorption spectrum registered with the proposed device is in accordance with the reference one. Measurements as a function of analyte concentration have been interpreted by two models—a modified Beer–Lambert law (low molarity region) and Langmuir adsorption law (molarity interval). The proposed device may find applications as absorption responsive tube when small volumes are needed, as required for lab-on-a-chip applications.

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