Visible light emitting Si rich Si_3N_4 μ-disk resonators for sensoristic applications.

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***Abstract*—We demonstrate the high potential of an optical integrated sensor which monitors the changes of the effective refractive index of the resonant optical modes induced by variations of the refractive index of the surrounding material. The detection system is a CMOS compatible structure based on a visible light emitting Si-rich Si_3N_4 μ-disk coupled to a passive Si_3N_4 waveguide placed underneath. We present a complete optical characterization of the active material in the isolated (μ-disk) and combined (plus coupled waveguide) photonic systems. The material has been optimized to obtain bright cavities with high quality factors. As a final result, we demonstrate that the sensor can achieve a sensitivity of 36 nm/RIU for small refractive index changes ($\Delta n = 0.002$) and a minimum detection limit of 1.6 × 10^{-3} RIU. This structure can be used as building block for detection systems with increased complexity, in which demultiplexion and detection could be readily integrated on the same chip.**

***Index Terms*— Active sensor, cavity resonators, optical sensors, optical losses, microcavity, microdisk, photoluminescence, whispering gallery mode (WGM).**

I. INTRODUCTION

In the last decade a large variety of integrated photonic elements found application in the sensoristic field [1]-[2]. Indeed, photonics plays a principal role in the realization of miniaturized, versatile and inexpensive detection systems. Different approaches of direct detection have already been reported such as Mach-Zehnder interferometers [3]-[4]surface plasmon resonator (SPR) [5] and optical waveguide based sensors. [6] Most of them can guarantee very high performances in terms of detection limit (DL) from 10^{-5} to 10^{-8} refractive index unit (RIU), but require a relatively large interaction length with the analyte, or a bulky light coupling system, decreasing the compactness of the device. Sensing photonic structures based on integrated optical resonators such as rings/disks or 2-D photonic crystals [7]-[8]allows robust and compact on-chip integration suitable for high volume production and field use. In particular, circular μ-resonators cavities, such as disks or rings, present reasonably high DL (up to 10^{-4} RIU for Si-based devices [9]-[10] and 10^{-7} RIU for polymeric μ-resonators [11]) and sensitivities (S) of 10^2 nm/RIU [10], while keeping a good tolerance to the fabrication accuracy.

One of the main issues concerning passive μ-resonators is the need of a broadband light source (broadband lamp or tunable laser, normally in the near infrared region) that has to be externally coupled into a bus waveguide. This is usually accomplished by means of grating couplers and it cannot be easily achieved in a hand-held device. In addition, the critical coupling condition is almost mandatory for the waveguide-cavity relative position since it is in this condition where the cavity is charged more efficiently through the waveguide.

An interesting alternative to lessen these conditions is the use of an efficient light emitting material (active material) within the μ-resonator, which can be top-pumped externally by optical or electrical means in a relaxed configuration. As an active photonic material, Silicon-rich Si_3N_4 (SRSN) provides several appealing properties for fabricating compact and efficient emitting devices: complementary metal-oxide-semiconductor (CMOS) compatibility, high refractive index ($n > 2$), efficient photoluminescence (PL) emission in the visible range, fast recombination rates [12] and good characteristics for achieving efficient electrical injection due to the relatively low Si-Si_3N_4 band offsets [13]. In particular the emission in the VIS region of the SRSN allows the use of Si-based detectors, easily integrable using a very mature CMOS standard technology.

In this work, we propose and characterize a basic sensing photonic structure consisting on a μ-disk cavity made of a SRSN material coupled to a passive stoichiometric Si_3N_4 passive waveguide placed underneath. Even though the potential sensitivity would be larger for μ-rings, we have studied μ-disks owing to the possibility of an electrical excitation without affecting the sensitive surface of the cavity.

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The whole device is composed by Si-based materials fabricated using CMOS compatible technological processes. Since it is possible to merge photonics and electronics in the same chip, we foresee this new approach as a promising starting point for low cost advanced sensor systems showing high sensitivity and extremely small footprint, allowing very small quantities of analyte to be measured.

The operational principle of these structures is based on the detection of the induced changes in the effective refractive index of the cavity supported modes. The PL spectrum emitted by the active material is modulated by the Whispering Gallery Mode (WGM) spectrum, which is characteristic of the cavity. The spectral position of the resonances depends both on the geometrical structure of the μ-resonator and on the effective refractive index of the media where the supported modes travel. Variations in the refractive index of the surrounding material will therefore shift the position of the resonances in a way that can be calibrated. The μ-disk is bottom-coupled with a passive waveguide placed underneath, which allows extracting the emitted PL from the resonator and driving it to the detection system. In this kind of configuration only the active cavity remains in contact with the external medium.

In this manuscript we present an optical and structural characterization of the bulk active SRSN, active waveguides and isolated active μ-disks. We have optimized the active material in terms of PL signal intensity and propagation losses to obtain efficient, bright and high quality μ-disks. Furthermore, we present an optical characterization of the coupled photonic system (active μ-disk and passive waveguide) and an evaluation of its sensoristic performances. We demonstrate that the proposed structures emit few nW in a single resonance and show competitive sensitivities (about 36 nm/RIU) and a detection limit about 2 × 10^{-3} RIU.

II. SAMPLE FABRICATION AND EXPERIMENTAL SETUP

The samples under analysis have been fabricated by standard CMOS processes. In the case of the isolated structures, as a first step, 2 μm thick SiO_2 layer has been thermally grown on top of a crystalline silicon wafer, acting as an optical cladding for the active photonic structures. A layer of 0.3 μm thick of stoichiometric Si_3N_4 material has been subsequently deposited using the Low Pressure Chemical Vapour Deposition (LPCVD) technique. The thickness of this layer has been chosen to ensure monomodal behaviour in the vertical direction. Finally, a double ion implantation of Si at 90 and 150 keV has been performed, followed by an annealing procedure in N_2 atmosphere at 950ºC. The ion energies and doses of the double implantation were chosen to achieve a flat Si excess profile and to optimise the overlap with the vertical distribution of the fundamental optical mode. We have produced a set of samples where the implantation doses were varied, covering a range of Si excesses from 2.5% to 12%. EFTEM (Energy-resolved Transmission Electron Microscopy) analysis revealed the absence of Si crystalline nanostructures inside the active layer, even in the case of the highest Si excess. The photonic structures, consisting of active μ-disks and active waveguides, have been finally defined by means of standard photolithographic techniques and Reactive Ion Etching (RIE). In Figure 1 we show an AFM (Atomic Force Microscopy) image of one example of the fabricated structures, a μ-disk of 7.5 μm radius. An average top and lateral surface roughness lower than 1 nm has been determined. Even though we have fabricated μ-disks with radii ranging between 2.5 and 10 μm, within the present work we will mainly focus on the experimental results of the 7.5 μm radius disks, which are representative of the whole set.

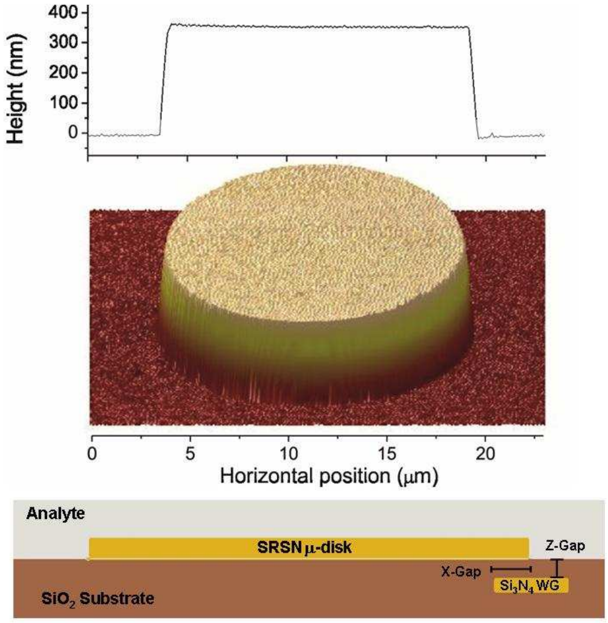

Fig 1. AFM image of an isolated 7.5 μm radius μ-disk (top and central panels) and a scheme of the cross section of the coupled structure (bottom panel). The Z-Gap is defined as the vertical distance between the bottom of the μ-disk and the top of the WG, while the X-Gap represent the center of the WG and the most external point of the μ-resonator circumference.

In the case of the coupled structures, a passive stoichiometric 2 μm wide and 150 nm thick Si_3N_4 layer was deposited on the top of the SiO_2 layer. Subsequently the rib waveguide has been defined by standard photolithographic processes and then covered by another SiO_2 layer, creating the vertical gap (Z-Gap) between μ-resonator and the waveguide. Since there is an unavoidable photomask alignment mismatch impact on the lateral relative position of the coupled structures, we designed a set of 20 coupled structures for a given combination of disk radius and waveguide width (see Figure 1) each one with a different ideal lateral displacement. This ensures finding a waveguide that is_horizontally placed in a way that the on plane energy distribution of the supported mode well overlaps with the radial energy distribution of the fundamental mode of the μ-disk. Their optical losses have been independently optimized down to less than 0.8 dB/cm at 780 nm (about the minimum sensitivity of our setup). A further separation layer of SiO_2 has been also deposited by PECVD (Plasma Enhanced Chemical Vapor Deposition) prior to the fabrication of the μ-disk. Its effect on the extracted intensities and quality factors

(*Q*) of the cavities has been also studied and optimized as a function of the gap dimension.

The optical measurements were performed in a standard μ-PL setup [14]-[15], where the detection is done on the plane of the μ-disks. A 370 nm solid state laser has been used as the excitation source and has been focused onto a single μ-disk by using a long working distance objective (pumping spot of approximately 5 μm of radius). The measurements of the coupled structures were made by pumping the μ-disks from the top and detecting the PL signal coming out from the passive waveguide. The proof-concept sensoristic measurement reported on section V was made by pouring a droplet of the liquids to be sensed with a μ-pipette on the top of the μ-disks.

Shifting Excitation Spot (SES) measurements [16] were carried out on the active rib waveguides in order to extract the propagation losses along the emission spectrum of the active material using the same μ-PL setup described above. This technique consists in varying the relative position between the excitation spot and the edge of the sample while the on plane PL spectrum guided by the active waveguide is collected from its edge. As a consequence of the propagation losses, the light intensity travelling inside the waveguide decays with an exponential relation, following the Beer-Lambert law, from which the losses coefficient can be extracted. Optical losses measurements using the cut-back technique were also performed at 780 nm.

III. ACTIVE MATERIAL CHARACTERIZATION: BULK MATERIAL AND WAVEGUIDES

A first step of this study was to characterise the active material in terms of the emitted PL intensity of the bulk material and propagation losses on strip waveguides.

The black squared curve of Figure 2 shows that the PL intensity scaled with the Si excess. It also scaled with pumping flux and did not show any sign of saturation even for the highest fluxes applied to the system [17].

On the contrary, the analysis of the results obtained from the cut-back measurements revealed that the propagation losses at 780 nm (red dots of Figure 2) dramatically increase with Si excess, which would degrade the performance of an eventual μ-cavity made out of those high Si excess materials. It is therefore clear that a sort of balance has to be obtained in order to obtain bright high *Q* cavities.

We have gone further on the analysis of the propagation losses present in the material and analyse their wavelength dependence. The SES technique was used to extract this information along the spectral range of the emission band. On Figure 3 we report those results in dB/cm for the case of a strip waveguide with 2.5% Si excess. Increasing the Si excess did not modify the spectral behaviour of the losses but only scaled their values similarly to that previously showed for the cut-back experiments.

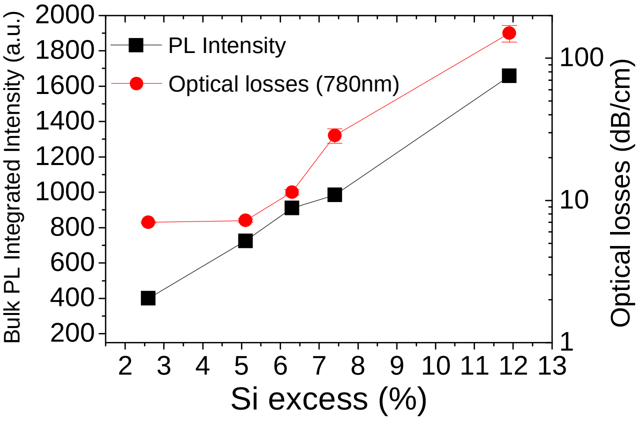

Fig. 2. Integrated PL intensity (black squares) and waveguide optical losses at 780nm (red dots) as a function of the Si excess present on the active material.

On the inset of Figure 3 we report the normalised spectrum resulting from three different positions of the excitation spot. The red-shift of the transmitted signal, when increasing the distance to the edge is related to an increasing of the losses for shorter wavelengths. This result is quantified in the main panel, where we identify Rayleigh scattering from Si nanoclusters present in the matrix as the main contribution to the losses at long wavelengths (red continuous curve). At shorter wavelengths there is a clear deviation from the $1/\lambda^4$ behaviour and the contribution of direct absorption losses starts to play a non negligible role.

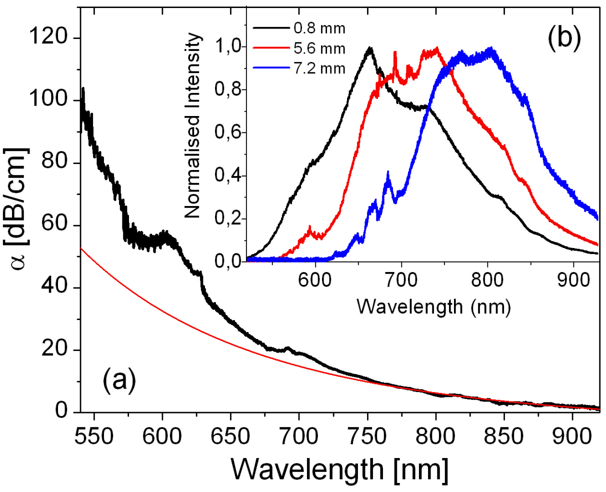

Fig. 3 (a.) Spectral dependence of the optical losses of an active strip waveguide with 2.5% Si excess, as extracted from the SES measurements. The fit using a Rayleigh type scattering dependence is also shown. (b) Guided spectrum collected at the output of the waveguide for three different positions of the pumping spot.

IV. SINGLE μ-DISKS CHARACTERIZATION

In view of the realization of a low detection limit system, one of the most important parameters is the *Q* of the μ-cavity. Such a parameter represents the fraction between the total energy contained in the cavity and the energy lost in a round trip. It is inversely related to the optical losses (α) and directly proportional to the group refractive index of the propagating mode (n_g). For a circular resonator it can be defined as the ratio between the wavelength of the maximum of a resonance peak and the Full Width at Half Maximum (FWHM) of the aforesaid resonance.

$$Q=2\pi n_g/\lambda\alpha=\lambda/FWHM \qquad (1)$$

In Figure 4 we represent the two main contributions to the total Q of the μ-disk. The black curve is associated to the material-related propagation losses as extracted from the experimental data of Figure 3. The scattered data represent the Q associated to the radiative losses of the geometrical structure, as extracted from FDTD simulations, when different μ-disk radii are considered. It is worth noting that we have neglected the contribution of the surface scattering losses motivated by the AFM low roughness results.

It is clear from the picture that the material losses dominate through all the emission spectrum if the radius is large enough, which is the case for a 7.5 μm radius μ-disk. We have indeed observed that for small μ–disks with high Si excess, Q as a function of wavelength starts rising, while beyond a critical value it starts to decrease, dominated then by the geometrical losses. The direct correlation between material losses and experimental Q has been reported by us in a recent work [14].

On figure 4, we have also represented the maximum resolution that our detection system is able to achieve, which is slightly above 10^4 within the spectral range of interest and below the expected Q for the case of 2.5% Si excess.

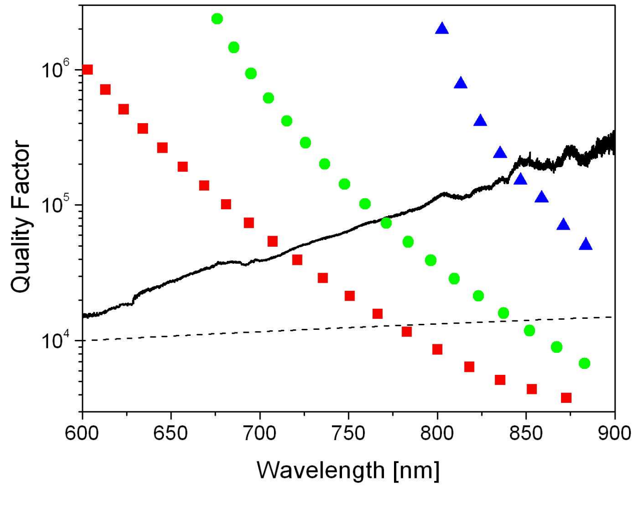

Fig. 4. The black continuous line represents the Q factor related to the material losses reported in Figure 3. The dotted curves (squares, circles, and triangles) are the radiative Q factor of μ-disks of different dimension (3,4 and 5 μm radius) obtained by FDTD simulations. The dashed line represents the maximum Q of the experimental setup.

The experimental μ-PL characterization of the μ-disk of 7.5 μm radius with 2.5% Si excess is reported as a black curve in Figure 5. This sample showed the best results in terms of Q. We have indeed observed a saturation of the experimental Q over the analyzed spectral range up to 1.4 × 10^4 (inset of Figure 5). This is the highest value ever reported for visible light emitting Si based circular resonators and even higher values are expected since we are just experimentally limited by the resolution of our setup. It is also worth noting that when comparing these results with the ones from sample of 12.5% Si excess (gray curve), the Q factors are clearly different while the on-plane PL intensities are of the same order. The latter result reveals that, even though the bulk material of the 12 % Si excess sample was emitting more strongly (Figure 2), the cavity effect and the low losses on the 2.5 % Si excess sample can compensate for that.

We have also quantified the emitted powers within a resonance to be of the order of few nW by using a calibrated Si photodetector. Power efficiencies of 10^{-5} when integrating the signal contained within the whole resonance spectrum were also determined. Those results are obtained without decreasing the Q of the resonances, since carrier absorption losses are practically negligible in this material as a consequence of the very high excited carrier recombination probability. [14] For the same reason, the material never reach enough carrier population depletion on the valence band to induce an increasing of Q at short wavelengths, where the losses were dominated by intraband carrier absorption. The measured power values are well above the minimum sensibility of state-of-the-art visible silicon based integrated photodetectors, which would in principle allow demultiplexing and detecting the emitted signal within the same chip.

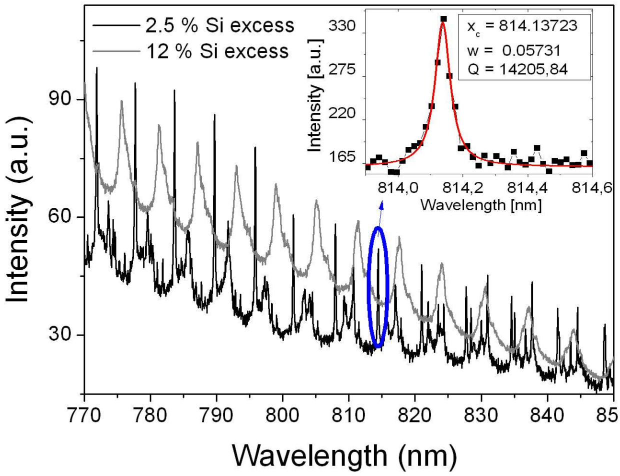

Fig. 5. μ-PL spectrum of a 7.5 μm radius μ-disk with Si excesses of 12% (gray) and 2.5% (black). On the inset we show a particular resonance at 814 nm of the 2.5% Si excess μ-disk, where Q factor of 1.42x10^4 is measured.

V. ACTIVE μ-DISKS COUPLED TO PASSIVE SI3N4 WAVEGUIDES AND SENSORISTIC PROOF OF CONCEPT.

In this section we present the results obtained on a photonic structure in which the 2.5% Si excess μ-disks have been bottom-coupled to a passive waveguide made of stoichiometric Si_3N_4, which was optimised to provide loss values down to 0.8 dB/cm at 780 nm.

FDTD simulations of the coupled structure showed that the optimum situation allowing high Q factors and high extracted PL intensities is found when the gap separation between the top surface of the waveguide and the bottom of the µ-disk is between 0.2 and 0.3 µm. Those simulations were realized taking into account the TM polarization, which turns to be slightly more sensible to refractive index changes of the surrounding medium. In such situation, *Q* values slightly lower than 10^4 could be achieved taking into account the experimental results reported in the previous section for the isolated µ-disks. Samples with different gaps within the optimum range (from 205 to 310 nm) and passive waveguide widths of 1µm were realized to verify the simulated predictions. The main results of this study are reported on Figure 6 and confirmed the expected qualitative behavior both for the TM polarized PL intensity measured at the output of the waveguide and the *Q* factors of the resonances. However, the *Q* factor values of the coupled structure, although still competitive, show a reduction of almost an order of magnitude with respect to the expectations. Indeed, a maximum *Q* of 1.48×10^3 at 762 nm has been recorded for the sample with the highest separation gap. Further AFM measurements showed that the observed *Q* factor reduction is associated to a small deformation of the µ-disk due to the presence of the waveguide, which has been only partially attenuated by a mechanical polishing done on the top of the separation SiO_2. In the following we will show results corresponding to the sample with the thickest gap.

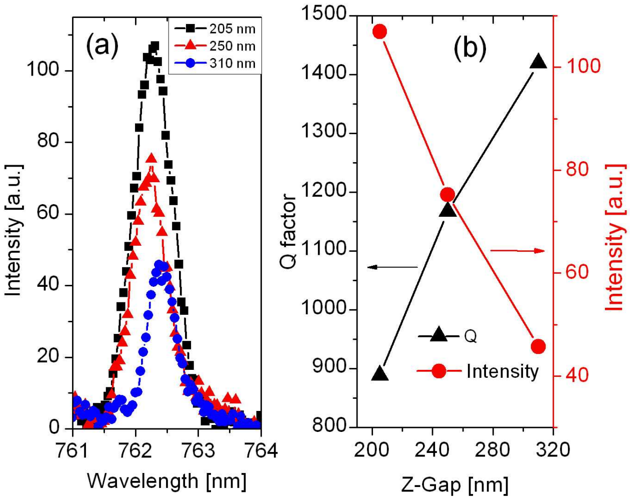

Fig. 6. (a) TM polarized µ-PL intensity of a resonance at about 762 nm for different gap distances between the bus waveguide and the µ-disk. (b) Quality factor (black triangles) and µ-PL resonance intensity (red circles) as a function of the vertical gap distance.

In order to evaluate the sensitivity of the device, we have carried out µ-PL measurements by changing the environment of the µ-disks and following the spectral displacement of a certain resonance. We have made this characterization on a coupled structure with R = 7.5 µm and Z-Gap = 310 nm, which gave the best result in terms of *Q*. It is also worth noting that this sample was providing just TM polarized signal on the output waveguide since TE signal was not coupled out because of the wide gap. On the top of a µ-disk we have poured drops of liquids with different refractive indices: we have varied the refractive index of the analyte as a function of the molar fractions of Methanol and Ethanol in a Methanol-Ethanol solution The results of this measurement are showed in Figure 7a, in which we can appreciate an overall resonance displacement of $\Delta\lambda = 1.37$ nm as a consequence of a change in refractive index of $\Delta n = 0.038$. From the slope of the linear fit of the experimental results we can extract the maximum sensitivity of our device, defined as the resonance wavelength shift for RIU, so that:

$$S = \Delta\lambda / \Delta n = 36.52 \text{ nm/RIU} \qquad (2)$$

This value depends only on the material and the characteristics of the cavity and is only slightly lower than of some state-of-the-art ring resonator sensors [1,10].

It is also important to establish a definition for the sensor DL, in terms of minimum measurable refractive index variation (Δn_{min}). On reference [18], this limit is defined as the subjective ability to distinguish a displacement of a single resonance. The principal issue deriving from this definition is the tight dependence on the resolution limit of the experimental setup, normally given by the excitation source in the case of a tunable laser or the detection system in the case of a monochromator. By using this definition, we believe that, taking into account our S value and the resolution and noise conditions of our measurements, we can distinguish down to a $\delta\lambda = 0.06$ nm shift, so that the detection limit of our measurement is:

$$DL = \Delta n_{min} = \delta\lambda / S = 1.6 \times 10^{-3} \text{ RIU} \qquad (3)$$

However, the previous definition of DL does not allow to reliably comparing our results with others reported elsewhere. In fact, this result is only slightly related to the *Q* factor of the devices. Therefore, it is useful to also provide the minimum refractive index change that provides a shifting equal to the FWHM of the resonance, so that it increases by an order of magnitude, i.e. $DL_{\mathrm{FWHM}} = 1.7 \times 10^{-2}$. The described results are slightly lower than that reported the state of the art CMOS compatibles devices [10], but obtained with the previously described advantages given by the active SRSN material. It is worth to note that the reported S value can be greatly improved by optimizing the polishing process on the SiO_2 separation layer. Indeed, it has been recently demonstrated a wafer-scale integration of a monolithic planar microresonator/waveguide vertically coupled system on a silicon chip demonstrating *Q* factors above 20000 on the IR region. [19]Another important improvement could be given by the use of µ-ring resonators, which potentially own a better sensitivity respect to µ-disks due to the greater surface in contact with the volume in which the WGM is developed.

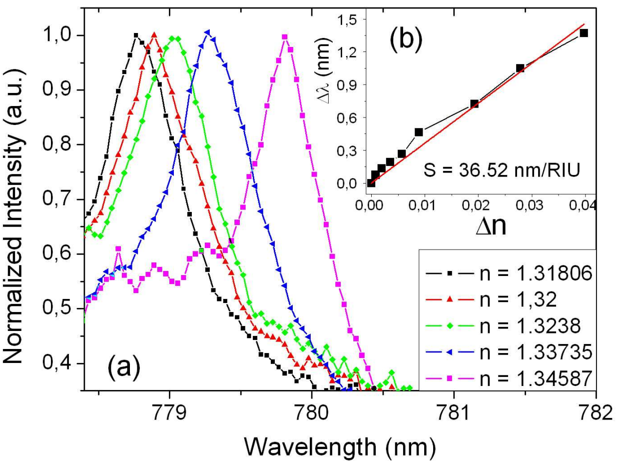

Fig. 7. (a) Spectral displacement of a resonance for five analytes with different n. (b) Linear behavior of the Δλ as a function of Δn for all the analytes prepared. The slope of the linear fit is 36.52 nm/RIU.

VI. CONCLUSION

We have presented a thorough study on the optical properties of SRSN μ-disks, in an isolated configuration and when coupled to a passive waveguide placed underneath. As a result of a careful optimization of the active material in terms of PL intensities and optical losses, we have been able to produce bright and high Q isolated μ-disks, achieving maximum values about 1.4×10^4 in a wide spectral range in the VIS and emitting up to few nW on a single resonance. The reported Q values are the best ever reported in circular Si-based light emitting μ-cavities and are just limited by the spectral resolution of our experimental setup. The coupled structures demonstrated Q values up to 1.48×10^3, which are susceptible to be greatly improved through optimization of the fabrication process. We have demonstrated that these structures are very sensible to the surrounding material and are able to detect refractive index changes with sensitivities of 36.52 nm/RIU and minimum measured refractive index change of 1.6×10^{-3} RIU. On the basis of these results, we believe that SRSN μ-disks have great potentiality to become building blocks of a photonic platform for sensing where demultiplexion and detection can be integrated on the same chip.

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