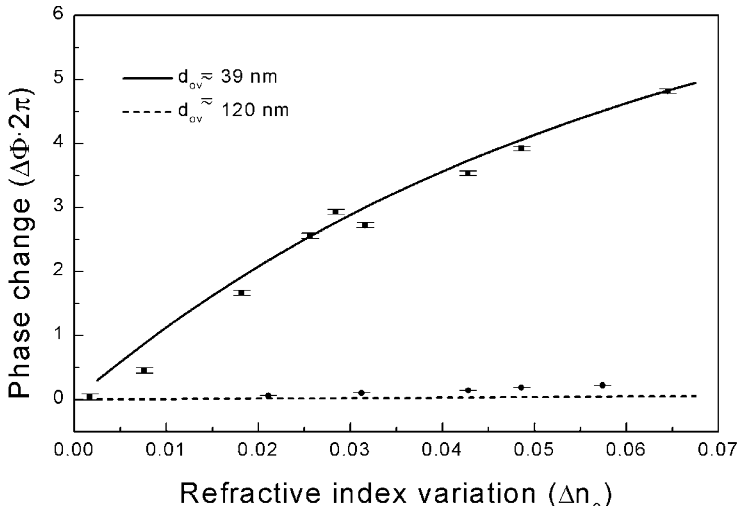


Fig. 5.19: Response of the MZI sensor as a function of the refractive index variation. The lines correspond to the theoretical simulation of the phase change: - (solid line) d_{ov} ~ 39 nm; -- (dashed line) d_{ov} ~ 120 nm.

Thence, optimizing the silicon nitride layer over the core so as to enhance the evanescent field propagation causes an increase of the sensitivity by an order of magnitude. However, it is still an order of magnitude above the required refractive index variation detection.

In chapter 2, it was observed that as the core thickness was reduced, an increase of the losses was straightforward. However, for values up to 3µm, losses could be acceptable if a significant increase of the device properties were to be obtained. In this case, since a minor core means a higher evanescent field, MZI with waveguide cores of 3µm should have better sensitivity. Experimental results shown in **[3]** proved that ARROW-A structures with a core of 3µm, sensing layer of 37nm and sensing region of 15mm were able to detect refractive index variations of $\Delta N=4.10^{-7}$. Thus, with this configuration it could be possible to detect bio-molecular reactions. It has to be noted that its sensitivity is still a factor 3 below these of a silicon nitride TIR Mach-Zehnder Interferometer **[5]**, nevertheless, due to the fact that the sensing region of ARROW MZI is 3 times larger, minimum refractive index variation that could be detected with the latter devices is an order of magnitude higher. Moreover, it has to be taken into account the previously described advantages that ARROW waveguides have as compared to TIR, as could be the much lower insertion losses and the align easiness.

As a direct application for this highly sensitive interferometers, it could be used as a refractometer. In fig 5.20 it can be observed that the signal obtained at the sensor output strongly varies as a function of the refractive index and, perhaps what is more important, it is observed that the signal value obtained for a given refractive index is recovered after measuring a different sample, that is measurements done with the MZI are reversible.

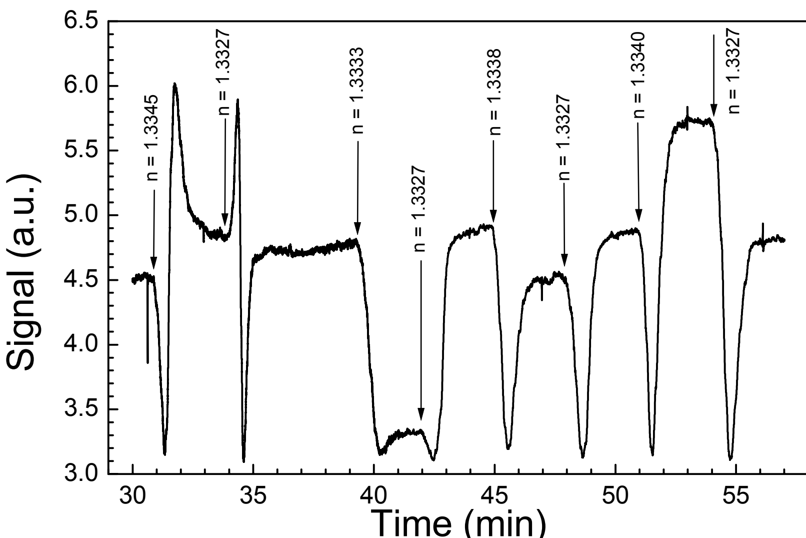


Fig. 5.20: Response of the MZI sensor for glucose solutions with different refractive indexes.

As an example of biosensing application, the response to the physical adsorption of $6.7 \cdot 10^{-6}$ M anti human serum albumin (α-hSA) is presented in fig. 5.21. Measurements have been performed in phosphate buffered saline (PBS), with a pH of 7.35, at room temperature and maintaining a low flow rate (approximately 30 μl/min). PBS has demonstrated to provide with the optimum pH and salinity conditions so as to keep the proteinic structure of the compounds unaltered. Antibodies form a monolayer with a thickness of around 10 nm by physical binding (electrostatic forces) to the waveguide surface. Causing a extremely small, however detectable, change in the signal at the device output.

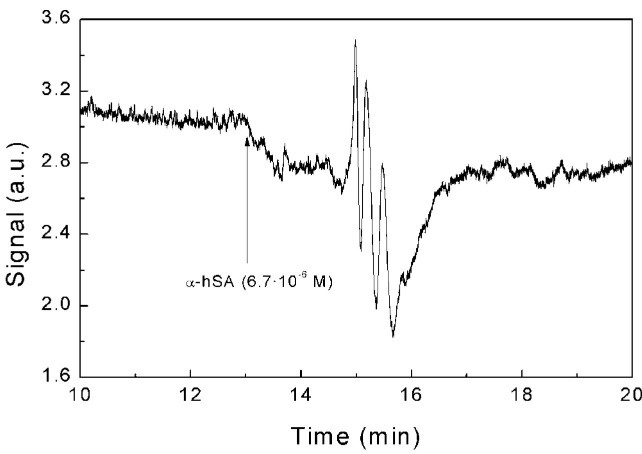


Fig. 5.21: Signal response of the MZI sensor to the physical adsorption of an antibody monolayer (TE polarisation).

5.7 Absorption Sensor

The technological steps that allow obtaining absorption sensors are exactly equal as those presented for the MZI. The first mask would define the waveguide width, while the second would allow opening the passivation in the sensing region. This 1st generation of optochemical absorption sensors were successfully fabricated and tested **[1]**, showing a sensitivity of 10^{-4}M for K^+ detection. The main drawback on that design was that input and output fiber optics were placed at both sides of the device. As can be seen in fig. 5.22a and 5.22b, after aligning and fixing the fiber optics, absorption sensors were submerged in a small volume of the solution to be measured. With this principle of operation, a crossed effect may occur: an unavoidable fiber optics bending is produced at the edges of the recipient. It causes the radiation of the higher modes in the fiber optics and a change in the light power. In order to assure that the power variation measured at the device output is only due to particle detection and not to fiber optics bending, the device has to be carefully placed on the sensing volume, trying to keep the fiber optics as straight as possible.

A second generation of this sensor was developed in order to overcome the double-facet polishing and the effect of the fiber optics bent. To do so, input and output waveguides where placed on the same side of the chip (fig. 5.22c). Light injected from the fiber optics was coupled to the input waveguide, which was bent 180º before injecting light into the sensing region. Finally, power was collected by a wider

waveguide and a multimode output fiber optics. With this configuration, fiber optics could remain straight during the measurement.

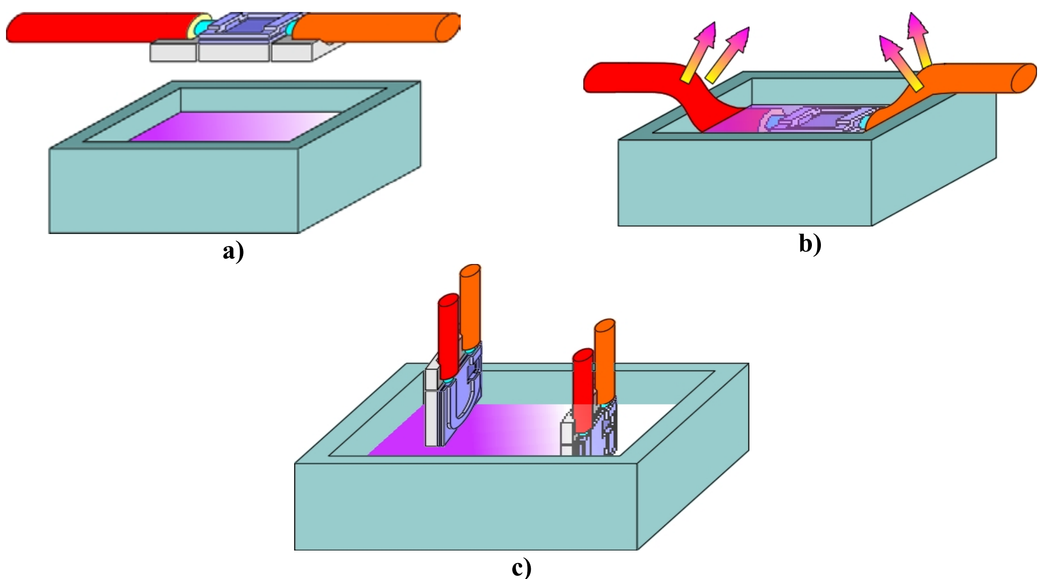


Fig. 5.22: Crossed effects on the 1st generation absorption sensor due to fiber optics bending. a) input and output waveguides where placed on both sides of the chip. b) When it was dipped in the solution, a fiber optic bent was produced, that caused fiber optics higher order modes to be radiative and polarization changes, resulting in a power decrease at the output due to fiber optics bent. c) 2nd generation optochemical sensors, where power was injected and collected by the same side of the chip, overcoming the bending effects.

In order to obtain the maximum available power at the sensing region, losses in the device must be minimized. According to **[6]**, three main factors could be identified: attenuation (or intrinsic losses) both at the waveguide and at the bend region, radiation losses at the bending and losses produced at the straight-bend and bend-bend transitions. The sensor has been designed not to have this latter type of transitions, since it has been observed that losses at these points are 4 times higher as compared to straight-bend transition **[6]**. Thus, the optimum device would have a straight input waveguide, which is 180º bent. After this curvature, the waveguide is straight again (then, two straight-bend transition should be taken into account) and, after the sensing region, light is collected by a straight wider output waveguide.

At this point, it becomes necessary to analyze the effect that geometrical factors have on the losses, concretely, the radius and the width of the bend waveguide. Clearly, attenuation in bend waveguides would increase as the radius of curvature

increases, since the optical path is enlarged. However, as the radius increases, radiation losses are lower. Thus, a compromise between attenuation and radiation losses must be reached. Losses in 180º waveguides (without straight-bend transitions) with widths of 20μm and 50μm are presented in fig. 5.23a. As can be seen, losses in the wider waveguide are significantly lower as a function of the radius. From these results, it could be concluded that the wider waveguide should be the one with the bending, that is, that the sensing region should be placed near to the device input. However, when straight-bend transitions are studied (fig. 5.23b), it is observed that losses in the 50μm waveguides are much higher. This can be associated to the fact that modes in a bend waveguide tend to displace towards the external part of the arc. Thus, the wider the waveguide, the larger the displacement. Thence, when bend-straight transition occurs, position and shape of the modes are so distorted that larger losses are produced.

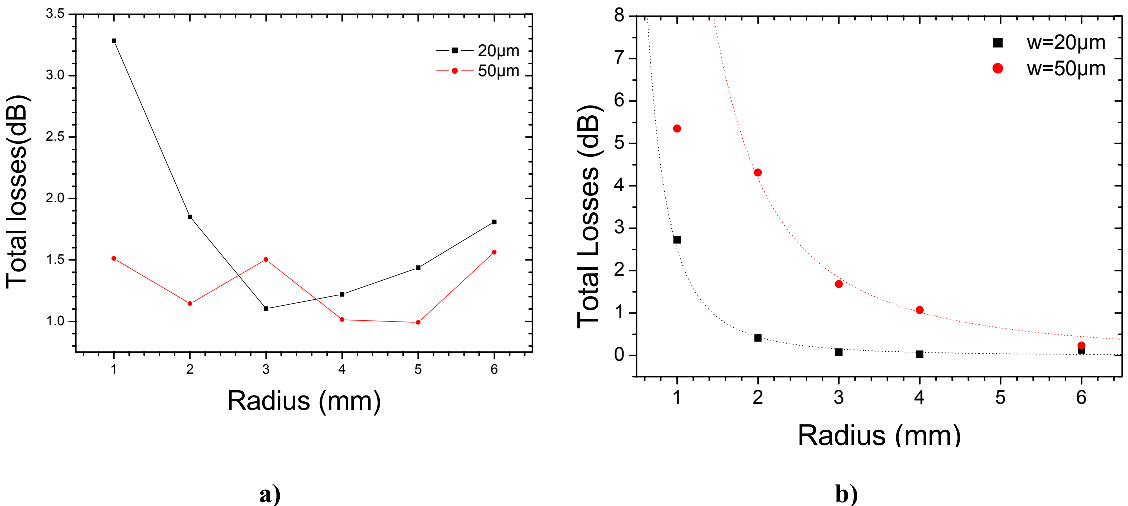
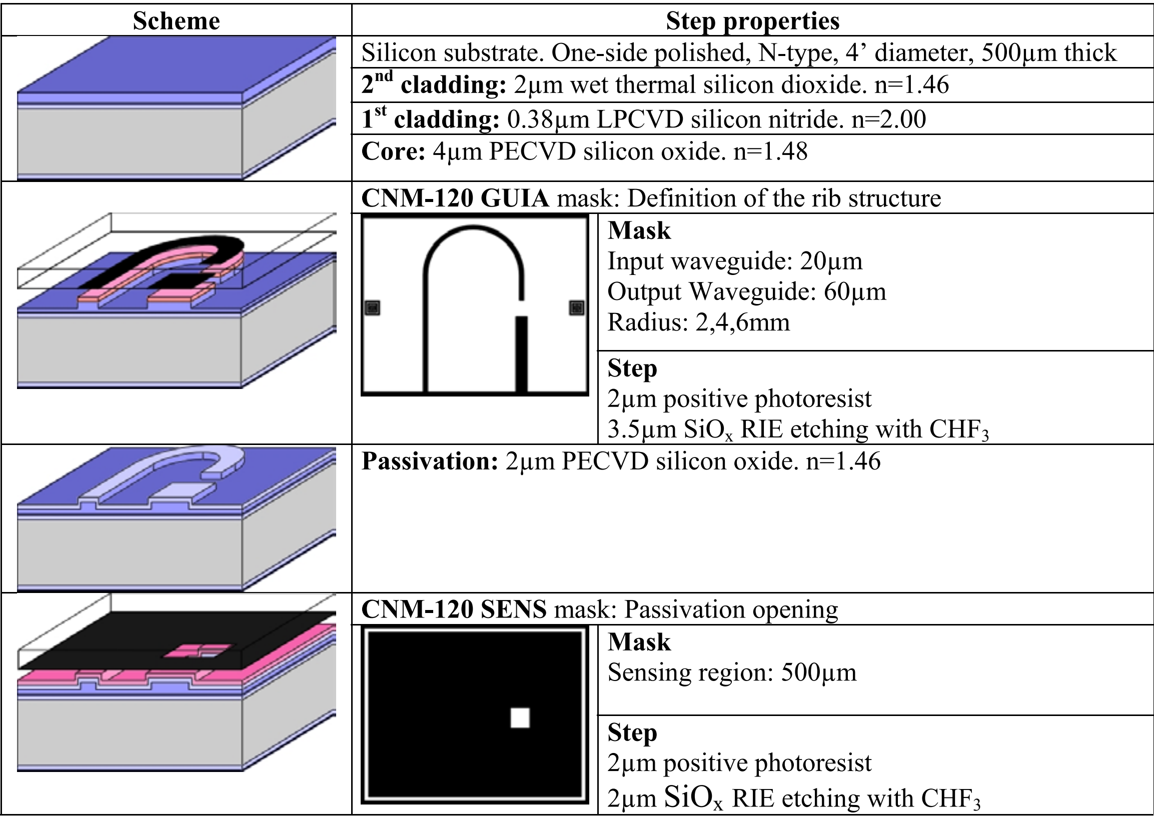


Fig. 5.23: a) Total losses as a function of the radius in a 180º bent waveguide for two different waveguide widths. b) Transition losses as a function of the radius for two waveguide widths.

The second objective was to study the possibility of making a self-alignment system. By far, the most important bottleneck in integrated optics is the light coupling. Then, if waveguides could be easily aligned with fiber optics, this major drawback would be overcome. Moreover, it has to be noted that the sensing is done only in the sensor chip, then, it would be extremely useful to preserve the rest of the setup for further measurements. For that reason, a three-pieced setup was developed. The main idea was defining a setup that allowed changing and replacing sensors fastly and

without alignment. Sensor fabrication process starts as the MZI, with the 1st mask used for defining the core and the 2nd mask for obtaining the sensing region (table 5.9).



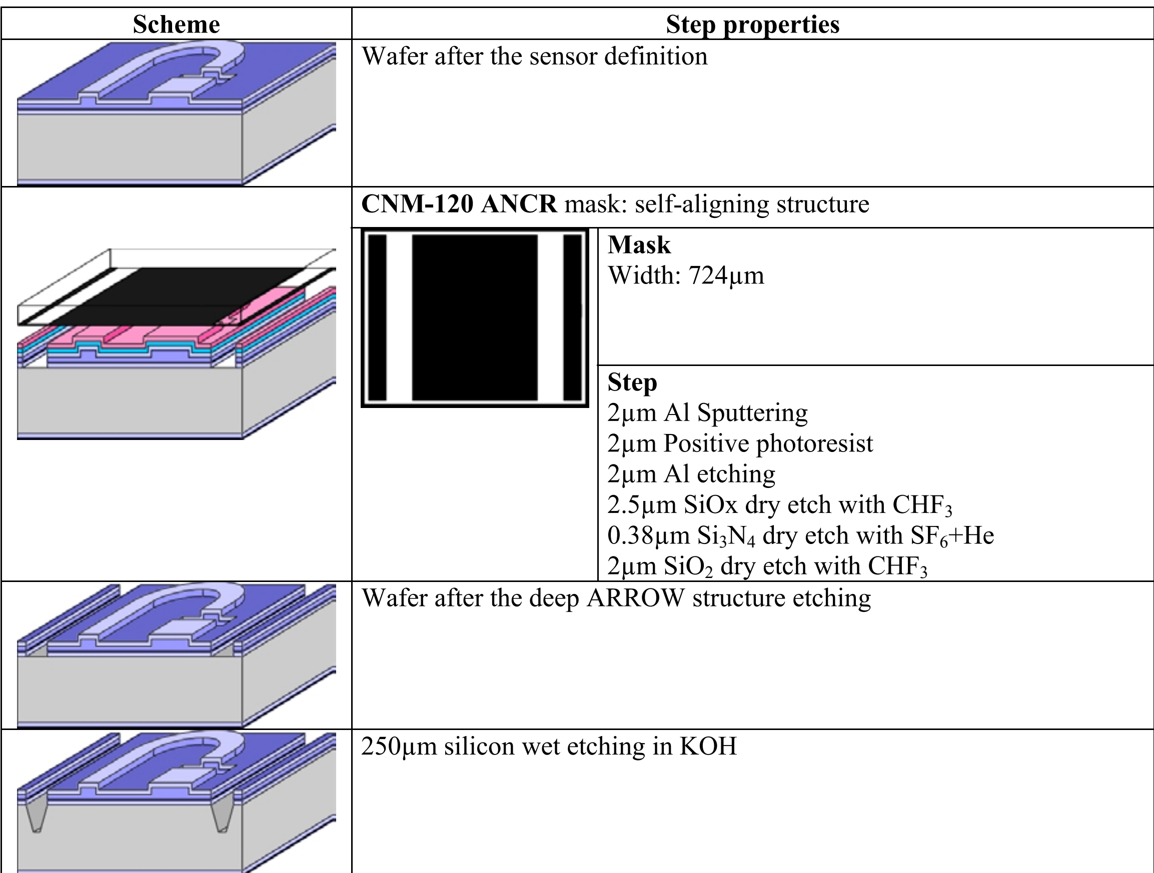
Scheme	Step properties	
	Silicon substrate. One-side polished, N-type, 4' diameter, 500µm thick	
	2nd cladding: 2µm wet thermal silicon dioxide. n=1.46	
	1st cladding: 0.38µm LPCVD silicon nitride. n=2.00	
	Core: 4µm PECVD silicon oxide. n=1.48	
	CNM-120 GUIA mask: Definition of the rib structure	
		Mask Input waveguide: 20µm Output Waveguide: 60µm Radius: 2,4,6mm
		Step 2µm positive photoresist 3.5µm SiO_x RIE etching with CHF_3
	Passivation: 2µm PECVD silicon oxide. n=1.46	
	CNM-120 SENS mask: Passivation opening	
		Mask Sensing region: 500µm
		Step 2µm positive photoresist 2µm SiO_x RIE etching with CHF_3

Table. 5.9: Technological steps used for the waveguide and the sensing region definition.

Once the passivation has been opened, following steps are focused on obtaining the self-aligned structure (table 5.10). This will be based on deep silicon wet etching on both sides of the sensor. This process will allow to obtaining smooth, accurately defined trenches in which the second part of the setup would be easily matched.

In order to define the deep trenches used for self-aligning purposes, a deep etch of the ARROW structure was required. Since this technological process needs large etching times, there are not any photoresist able to stand this process available at CNM. Thence, an aluminium mask has been used. To do so, 2µm aluminium has been sputtered and the self-aligned region is defined. After the elimination of the photoresist, the etching of the structure starts. Once the silicon substrate is reached, a wet etching in

KOH (that also etches the Al mask) is done until a depth of 250µm, ending the technological process for the sensor part.



Scheme	Step properties	
	Wafer after the sensor definition	
	CNM-120 ANCR mask: self-aligning structure	
		Mask Width: 724µm
		Step 2µm Al Sputtering 2µm Positive photoresist 2µm Al etching 2.5µm SiOx dry etch with CHF_3 0.38µm Si_3N_4 dry etch with SF_6+He 2µm SiO_2 dry etch with CHF_3
	Wafer after the deep ARROW structure etching	
	250µm silicon wet etching in KOH	

Table 5.10: Definition of the deep trenches that would allow self aligning with the fiber optics.

The second part of the setup will allow aligning the sensor chip with the reaction chamber. Its technological steps are relatively simple (Table 5.11). However, they must have a high dregree of accuracy in order to fit with the deep silicon trenches defined in the sensor device. Moreover, since the self-aligning structures have been done in the front side, this second piece should have a hole exactly in the sensing region, so as to permit the correct behavior of the device.