



Optically active Si-rich Si_3N_4 μ -cavities for sensoristic applications

Federico Ferrarese Lupi

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Optically active Si-rich Si_3N_4 μ -cavities for sensoristic applications

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Contents

1	Introduction	1
1.1	Structure of the work	4
1.1.1	Si-nc photoluminescence guided inside optical structures	4
1.1.2	Design and Simulation of the optical structures	4
1.1.3	Fabrication of process	5
1.1.4	Experimental Setup and Characterization Techniques	5
1.1.5	Optical Losses and PL characterization	5
1.1.6	Circular resonant structures	6
2	Si-ns photoluminescence guided inside optical structures	7
2.1	Principles of emission of Si-ns in Si_3N_4 matrix	8
2.2	Light propagation in a circular resonator	11
2.2.1	Free Spectral Range	16
2.2.2	Finesse	16
2.2.3	Quality Factor	17
3	Design and Simulation of the optical structures	19
3.1	Simulation Softwares	19
3.1.1	Beam Propagation Method	20
3.1.2	FDTD method	23
3.1.3	Harminv	25
3.2	Modal structure of the μ -resonators	27
3.2.1	Geometrical variation	28

3.2.2	45° cut edge Vs Vertical edge	34
3.2.3	Refractive index variation	35
3.3	Coupled Structure	36
3.3.1	Z-Gap	37
3.3.2	X-Gap	40
4	Fabrication Process	43
4.1	Photolithographic mask	43
4.2	Deposition process and parameters	46
4.3	Analysis of the surface	51
4.3.1	Atomic Force Microscopy	51
4.3.2	Scanning Electron Microscopy	53
4.4	XPS	56
4.5	EFTEM analysis	58
5	Experimental Setup and Characterization Techniques	59
5.1	Optical Losses Setup	60
5.1.1	Cut-back Technique	61
5.1.2	Scattered Light Collection Technique	62
5.2	μ -PL measurement setup	64
5.2.1	Shifting Excitation Spot technique	66
6	Optical Losses and PL characterization	69
6.1	Passive WG optimization	70
6.1.1	Annealing treatment and geometrical variation	70
6.1.2	Etching variation	78
6.2	Active material characterization	81
6.2.1	Visible Region	83
6.2.2	Shifting Excitation Spot technique	86
6.2.3	Infrared Region	88
7	Circular resonant structures	91

7.1	Isolated resonators	91
7.1.1	Preliminary considerations	92
7.1.2	Contributions to the total Q	95
7.1.3	Annealing temperature variation	99
7.1.4	Power dependence	100
7.1.5	Ring resonators	104
7.2	Coupled structure	105
7.2.1	Sensitivity of the device	108
7.2.2	Behaviour in the IR region	112
8	Conclusions and future developments	113
8.1	Conclusions	113
8.2	Future developments	115
A	Light Propagation in 2-Dimensional Waveguide	117
A.1	Critical angle condition	118
A.2	Self-consistency condition	119
B	Simulation of the WGM spectrum	123
B.1	FDTD simulation of the coupled structure	123
B.2	Bash script for the FOR LOOP iteration	126
	Bibliography	129
	Acknowledgements	139
	Curriculum Vitae	141

List of Figures

1.1	<i>Left panel - Scheme of the cross section of the coupled structure, in which the variables Z-Gap and X-Gap are graphically defined. Right panel - 3D sketch representing the overall sensing device.</i>	3
2.1	<i>Scheme of the indirect band gap structure of bulk Si. The possible transitions for an electron-hole pair (phonon-assisted radiative recombination, Auger recombination, and free-carrier absorption) are also represented.</i>	8
2.2	<i>Representation of the first kind Bessel function (first and second order) $J_0(k_0, n_{eff}, \rho)$, $J_1(k_0, n_{eff}, \rho)$ and $J_1(k_0, n_{eff}, \rho)$ (left panel) and second kind Bessel function $N_0(k_0, n_{eff}, \rho)$, $N_1(k_0, n_{eff}, \rho)$ and $N_3(k_0, n_{eff}, \rho)$ such functions oscillates like a sine or cosine function but with a decaying amplitude.</i>	14
2.3	<i>Bouncing wave inside a circular resonator, suffering a phase shift $\phi \neq q2\pi$ each round trip.</i>	15
3.1	<i>Structure of a BPM simulation, in which the cross section of the structure under analysis is defined in the plane x-y, while the propagation of the EM wave is in the z direction. . .</i>	21
3.2	<i>Position of various field components on the Yee cell. The E-component are in the middle of the edges while the H-components lie in the center of the faces</i>	24
3.3	<i>Decomposition of a given $f(t)$ signal in a finite number of exponentially decaying sinusoids. (Picture taken from http://ab-initio.mit.edu/wiki/index.php/Harminv)</i>	26

3.4	<i>Example of top view (plane $z=0$) of a 3D simulation of a μ-disk. In figure (a) we can see the excitation source expanding at the beginning of the simulation, while in figure (b) the modal structure is already developed. In the bottom panel (c) we can see the cross section of a 3D simulation of the same disk obtained exploiting the cylindrical symmetry of the system.</i>	27
3.5	<i>Scheme of the cross section of the simulated structure. All the refractive index of each material used in the simulation are also indicated.</i>	29
3.6	<i>Variation of Q factor (left graphic) and the FSR (right graphic) as function of the radius R of the disk. The blue dashed line in the left graphic represent the established minimum value of Q. Such limit for the FSR depends on the practical application of the device. .</i>	30
3.7	<i>Behaviour of the Q in a $3\ \mu\text{m}$ radius disk as function of the thickness: only for disks with h superior than $0.3\ \mu\text{m}$ the minimum condition given by the blue dashed line, is respected. In the inset we can appreciate the broadening of the resonances as the h goes down.</i>	31
3.8	<i>(a)-(g): TR polarized simulated spectra obtained for a μ-disk of radius $4\ \mu\text{m}$ and variable thickness (from $0.1\ \mu\text{m}$ to $0.4\ \mu\text{m}$). The value of m parameter and the number of the modal family is also indicated (e.g. the notation $52^{(1)}$ represent a resonance peak with $m = 52$ and modal family = 1). (h) Graphic of the FSR as function of the wavelength for four different values of h.</i>	32
3.9	<i>Distribution of the EM field of some modes supported by the cavity. (a) and (b) represent the fundamental mode. Figure (h) represent a high losses mode obtained for an h value of $0.1\ \mu\text{m}$</i>	32
3.10	<i>Comparison between the Q factor of TR and TM polarizations of a $4\ \mu\text{m}$ radius and $0.3\ \mu\text{m}$ thickness disk.</i>	33
3.11	<i>Variation of the WGM spectrum (panels (a)-(g)) and FSR (panel (h))for the TM polarization inside a disk of $R=4\ \mu\text{m}$ and $n = 2$ as function of the variation of the height. .</i>	33
3.12	<i>Comparison between the field distribution of the first order modal family inside a disk presenting a vertical edge (a) and 45° edge (b).</i>	34
3.13	<i>Left graphic: Q factor variation as a function of the edge shape of the disk, showing a relevant difference at small wavelength. In the inset the FSR is also shown. Right graphic: TR polarized WGM spectral shape for both μdisks.</i>	34

3.14	<i>Left panel: variation of the WGM position of a 7.5 μm radius disk due to a change of refractive index value. Right panel: linear slope of the maximum of one resonance reported in the Left panel, resulting in a Sensitivity of 60.82 nm/RIU</i>	36
3.15	<i>Scheme of the cross section of the coupled structure. The Z-Gap and X-Gap are also graphically defined.</i>	36
3.16	<i>Left panel: Simulated behaviour of Q in a coupled structure as a function of the Z-Gap distance. Right panel: comparison between the Q and the intensity (measured inside the WG) of a resonance for different Z-Gap values.</i>	38
3.17	<i>Left graphic: simulated WGM spectra of a 3 μm radius μ-disks, for different vertical gap values. The signal is provided by the detector placed on the waveguide. Right graphic: simulated WGM spectra measured inside the μ-resonator. The inset graphic shows a zoom of one particular resonance, putting in evidence the variation of Q factor as a function of the gap distance.</i>	39
3.18	<i>Simulated spectra obtained by varying the lateral X-Gap from an external position outside the disk, to an internal position.</i>	41
4.1	<i>Layout of the two level of photolithographic mask superimposed. The red parts correspond to the passive layer, while the green part represent the active material.</i>	44
4.2	<i>Zoom of one series of disk fields shown in figure 4.1 and detail taken with an optical microscope of the 2.5 μm and 5 μm field.</i>	45
4.3	<i>Particular of the Zone 2, containing active disks and rings coupled with passive waveguides.</i>	45
4.4	<i>Left panel: Profile of the double implantation compared with the energy distribution of the fundamental WGM of a micro-cavity. It is easy to notice that the overlap is not perfect due to the technical limitations of the implanter. Right panel: Percentage of Si excess for different implantation doses samples.</i>	47
4.5	<i>3D image of the morphological status of a 7.5 μm radius disk (a) and analysis of the roughness of the top surface of the disk (b).</i>	52
4.6	<i>AFM image of the defects affecting the fabrication of the μ-rings.</i>	52
4.7	<i>Top image (a) and 3D reconstruction (b) of the AFM analysis on a WG of sample 1 of run 5107-UB. No evident imperfections can be seen.</i>	53

4.8	<i>SEM images showing the real dimensions of two circular resonators: panel (a) and (b) demonstrate a discrepancy of about $0.3 - 0.5 \mu\text{m}$ between the real length of the radius and that previewed by the mask.</i>	54
4.9	<i>SEM image (a) and particular zoomed area (b) showing the 45° angle of the perimetrical walls.</i>	54
4.10	<i>Image of a WG field (left panel) and particular of the WG side walls (right panel) . . .</i>	55
4.11	<i>SEM images of some μ-disks lying on the edge of the sample: on the left picture the cavities are broken by the cut of the sample, so is not possible the WGM detection. On the contrary, in the right image the disks are results to be intact.</i>	55
4.12	<i>Spectral shape of the Si_{2p} and N_{1s} contributions for sample 1 of Run 5107_UB</i>	56
4.13	<i>Si_{2p} (Left panel) and N_{1s} (Right panel) atomic concentration profiles of the studied samples.</i>	57
4.14	<i>Si_{2p} binding energies for the different samples at the same depth.</i>	57
4.15	<i>(a) Cross section EFTEM image showing the SRSO/SRSN bilayer and Si-ncs into the SiO_2 layer. (b) Scheme of the light emitting device structure. Picture taken from Y. Berencén et al. Optics Letters, Vol. 36, No. 14, July 15, 2011</i>	58
5.1	<i>Scheme of the WG losses measurements setup. The dotted red line represent the two different parts in witch the collected light is devided by means of a Beam Splitter (BS) and sent to a detector (D) and to a IR camera (C).</i>	60
5.2	<i>Left panel: representation of $I_{\text{out}}/I_{\text{in}}$ values as a function of sample length. Right panel: $IL(\text{dB})$ representation of $I_{\text{out}}/I_{\text{in}}$ values.</i>	62
5.3	<i>Left: representative picture of the top view of a waveguide with a coupled laser. Right: example of graphic obtained after the treatment of the images obtained by Scattered Light Collection Technique.</i>	63
5.4	<i>Graphical representation of the μ-PL setup used for the characterization of the circular resonators.</i>	64
5.5	<i>Picture of a single (left panel) and multiple (right panel) μ-disk excitation obtained by means a de-focalization the pumping spot. The yellow dye photographies were taken using a 500 nm long-pass filter in order to eliminate the noise due to laser scattering. .</i>	65

5.6	<i>Top view of the μ-PL setup (left picture) and particular of the sample holder and the pumping and recollection objectives (right picture).</i>	65
5.7	<i>Schematic view of the Shifting Excitation Spot technique setup. The sample is excited by a micrometric spot in different position on the same line perpendicular to the sample edge.</i>	67
6.1	<i>Comparison between (a) Propagation and Coupling losses in sample without annealing process (sample 2) and (b) Propagation losses for the not annealed sample and samples treated at 1000°C, 1100°C and 1150°C (sample 4, 5 and 6).</i>	72
6.2	<i>Upper view of a 10 μm WG belonging to sample 5, coupled with a 633 nm laser beam.</i>	73
6.3	<i>Comparison between (a) propagation losses at 1480 nm (b) and 1520 nm in samples that have suffered a different annealing process (samples 4 to 6) and a not annealed sample (blue stars representing sample 2).</i>	75
6.4	<i>Top panels: simulated structure of a 2 μm width WG and thickness of 150 nm, with a covering cladding. (Run 5094_UB) and energy distribution of the fundamental mode at $\lambda = 1480$nm. Bottom panels: structure of a 2 μm width and thickness 300 nm WG (Run 5107_UB) and energy distribution of the fundamental mode</i>	76
6.5	<i>Comparison between losses value for WG structures described in Figure 6.4.</i>	77
6.6	<i>Scheme of the Strip (a) and Rib (b) Si_3N_4 WG and simulation of the spacial distribution of the TE polarized EM field at 780 nm.</i>	79
6.7	<i>Propagation and coupling losses at 780 nm and Laser power of 4.5 mW for samples with different etching: Deep (1 and 2) and Shallow (sample 3) belonging to run 5094_UB).</i>	80
6.8	<i>PL spectra (left panel) and summary of its intensities (right panel) as a function of the Si excess.</i>	82
6.9	<i>Integrated PL emission as a function of the photon flux for two samples of different Si excess: 6.3% (black squares) and 11.9% (red circles).</i>	82
6.10	<i>Optical loss at 780 nm due to the propagation and coupling inside active material's WGs having a Si excess of 6.3%</i>	84
6.11	<i>Propagation Losses calculated by means of the Scattered Light Collection Technique on sample 5 (a) and 6 (b) having high percentage of ionic implantation.</i>	84

6.12	<i>Left panel: optical loss values at 780 nm for three different WG of 5 μm width belonging to samples 1, 2 and 3 of run 5372_UB. Right panel: comparison between PL intensity and optical losses as a function of the Si excess %.</i>	85
6.13	<i>Emitted PL spectra for nine values of position of the pumping spot on the WG.</i>	87
6.14	<i>(a) Spectral dependence of the optical losses of an active strip WG 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 WG for three different positions of the pumping spot: the red shift of the PL spectra is clear when moving the pumping spot inside the sample.</i>	87
6.15	<i>Representation of propagation losses for 11.9, 7.4 and 6.3 % Si excess WGs.</i>	88
7.1	<i>WGM spectra of the same sample obtained by means of a standard PL setup (a) and with a more precise μ-PL setup(b).</i>	93
7.2	<i>Comparison between low resolution spectrum (left panel) and high resolution spectrum (right panel) of a same resonance peak of a $R = 7.5 \mu\text{m}$ disk. The 600 l/mm grating has a maximum resolution of 0.11 nm, leading to a maximum Q factor of 5.63×10^3 while the 1200 l/mm owns a resolution of 0.06 nm corresponding of a Q value of 1.05×10^4 at 760 nm.</i>	94
7.3	<i>PL spectra of TM (black curve) and TR (red curve) polarized WGM in a 2.5 μm radius disk. Looking carefully the two spectra we can see how the two polarizations are not completely separated: it is possible to see part of the first order TR (TM) mode superimposed to the TM (TR) polarized spectrum.</i>	94
7.4	<i>Panel (a): comparison between simulated and experimental FSR. Panel (b): experimental and measured group index. Panel (c): WGM spectrum of μ-disks of different radius.</i>	95
7.5	<i>(a): Behaviour of Q (black spheres) measured at 760 nm as function of the Si excess for $R = 7.5 \mu\text{m}$. The propagation losses at 780 nm are also reported (red triangles). (b): TR polarised μ-PL spectrum corresponding to disks with $R = 7.5 \mu\text{m}$ of samples 1 (grey) and 4 (black). (c): Zoom of the resonance present at 757 nm for sample 4, together with the corresponding Lorentzian fit.</i>	97
7.6	<i>Incidence of the Q contributions in a 7.5 μm (left panel) radius disks of Sample 3 run 5372_UB. The SEM analysis reveals a good circular shape, with small variations.</i>	98

7.7	<i>Incidence of the Q contributions in a 3 μm (left panel) radius disks of Sample 3 run 5372_UB and SEM image of the same disk (right panel).</i>	98
7.8	<i>Behaviour of the Q as function of the wavelength for the TE (blue circles) and TM (green triangles) polarizations in a 3 μm radius disk. Such values are overlapped with the unpolarized WGM specturm of the same cavity.</i>	99
7.9	<i>Variation of Q (graphic on the left) and of the FSR (graphic on the right) in function of the Si dose and annealing treatment - with the same symbol are headed samples with same Si excess dose but different annealing temperature. Both graphics were made using the 600 l/mm grating.</i>	100
7.10	<i>Variation of the WGM position and FWHM as function of the power. Panel (a) shows a red shift associated with a deformation of the resonator edge due to a thermal effect, while panel (c) represent the blue shift due to deformation of the resonator. In both panel (b) and (d) we can notice a very small variation of the Q under different pumping powers.</i>	102
7.11	<i>(a) Spectral power density of a TR polarized μ-PL emission spectrum for a μ-disk of sample 4 ($R=4.5 \mu\text{m}$). (b) SEM image of the corresponding μ-disk.</i>	103
7.12	<i>Top view of a 10 μm radius ring under 488 nm (top picture) and 378 nm (bottom picture) laser pumping. The first picture has been taken putting a 550 nm filter in front of the camera. On the right panel are represented the spectra of three rings of same radius but different width of the WG forming the circular structure: (a) 1.5 μm, (b) 2 μm and (c) 3 μm.</i>	105
7.13	<i>(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.</i>	107
7.14	<i>Comparison between TR and TM polarized spectra of a 7.5 μm disk coupled with a 1 μm wide WG, placed at a Z-Gap of 355 nm and X-Gap of $-1 \mu\text{m}$.</i>	107
7.15	<i>Spectral displacement of a resonance measured by pouring a drop of liquids with different refractive index on a 7.5 μm radius disk for five analytes with different n value.</i>	109

7.16	<i>Linear behavior of the $\Delta\lambda$ as a function of Δn for all the analytes prepared. The slope of the linear fit obtained by fitting all the data is 36.52 nm/RIU (Black line), while for small refractive index changes the sensitivity value is of 51.79 nm/RIU (Blue line). The Red line represent the experimental sensitivity obtained for the same n variation.</i>	111
7.17	<i>Simulated (black line) and experimental (red line) spectra of a 7.5 μm disk bottom coupled with a 1 μm wide WG.</i>	112
8.1	<i>Representation of some possibles improvements on the μ-disk-based sensor, as the microfluidic channel (in light blue over the disk) and the demultiplexing circuitry: in the left panel a bottom cupeling scheme is represented, while in the right panel the μ-rings are horizontally coupled with the bus WG.</i>	115
A.1	<i>Some of the most common optical waveguide configurations: (a) slab WG, (b) strip WG and (c) fiber WG</i>	118
A.2	<i>Scheme of the self-consistency condition for a plane wave traveling in the z direction with an incident angle $\hat{\theta}$.</i>	119
A.3	<i>Number of modes of a bi dimensional WG. (Picture taken from [60])</i>	121

List of Tables

4.1	<i>Passive samples produced in order to study the effect of the annealing treatment on the propagation losses.</i>	49
4.2	<i>Characteristics of the samples belonging to Run 5107_UB.</i>	49
4.3	<i>Deposition parameters of samples 1-6 belonging to Run 5372_UB.</i>	50
4.4	<i>Description of the samples of Run 5506_UB, containing the final coupled structures.</i>	50
6.1	<i>Main parameters of the samples that have suffered a further annealing treatment at different temperature.</i>	71
6.2	<i>Summary of the losses values obtained at 633 nm and 780 nm, for 10 μm passive WG.</i>	74
6.3	<i>Table of the optical losses obtained in the IR region for the passive WGs.</i>	77
6.4	<i>Resume table of the etching type.</i>	78
6.5	<i>Summary of the results of the propagation losses measurements obtained by varying the etching type.</i>	80
6.6	<i>Resume of the active material samples implantation (Run 5107_UB).</i>	81
6.7	<i>CBT results obtained at 780 nm. TSC measurements have been also performed on that samples in which CBT has produced no results.</i>	85
6.8	<i>Summary of the losses values for the active WGs in the infrared region.</i>	89
7.1	<i>Main parameters used in the preparation of the solutions.</i>	108
7.2	<i>Molar fractions of the dissolutions of Methanol and Ethanol. In the third column is reported the resulting refractive index.</i>	109

Resumen en Castellano

En esta tesis, realizada en el departament d' Electrònica de la Universitat de Barcelona se ha presentado un estudio detallado de las propiedades ópticas y sensoras de estructuras resonantes tipo μ -disco fabricados íntegramente en nitruro de silicio (Si_3N_4) enriquecido con silicio (*SRSN*). El estudio se ha llevado a cabo bien en estructuras aisladas o en una configuración acoplada con una guía de onda passiva situada debajo de la cavidad.

La totalidad de la estructura, ya sea la cavidad ó el sistema acoplado, han sido previamente simuladas con el fin de estudiar el comportamiento de los modos resonantes (whispering gallery modes) soportados cuando se cambian las condiciones de contorno geómetricas (por ejemplo el radio, el espesor, la forma del borde del disco aislado...) y del material (índice de refracción y exceso de silicio). Los resultado obtenidos han permitido la realización de estructuras resonantes con factores de calidad superiores a $Q = 10^4$. El objetivo de las simulaciones efectuadas con softwares comerciales ha sido el de maximizar la intensidad transmitida de los modos soportados (WGM) al final de la guía de onda. Este hito ha sido cumplido gracias a la optimización de los parámetros geómetricos relativos (el *X-Gap* y el *Z-Gap*). Una vez optimizada la estructura, se procedió a fabricar las muestras, que fueron realizadas en el *Instituto de Microelectrónica de Barcelona* (IMB-CNM-CSIC), bajo la supervisión de los doctores Josep Montserrat y Carlos Domínguez. El proceso tuvo lugar en una cadena CMOS estándar. La deposición y la implantación de los nitruros se llevó a cabo mediante la técnica LPCVD (Low Pressure Chemical Vapor Deposition) , mientras que la capa de SiO_2 que forma la separación entre la cavidad resonante y la guía de onda fue fabricada por PECVD (Plasma-Enhanced Chemical Vapor Deposition). Finalmente los elementos ópticos han sido definidos mediante dos niveles de

máscara fotolitográfica. Una vez producidas las muestras, se procedió a la realización de un análisis de superficie (SEM, AFM) para evaluar la rugosidad efectiva de las estructuras y las eventuales imperfecciones geométricas, ya que son factores influyentes en el factor de calidad. Además, un estudio EFTEM (Energy Filtered Transmission Electron Microscopy) realizado en muestras similares de nitruro de silicio fabricadas en nuestro grupo revelaron la ausencia de nanocristales de silicio (Si-nc) en la capa activa, evidenciando sólo la presencia de estructuras amorfas (Si-ns).

Por otro lado, utilizando la técnica del Cut-back, se encontraron pérdidas en las guías de onda pasivas inferiores a 1 dB/cm en las dos regiones espectrales analizadas (el visible y el infrarojo cercano). También se analizaron distintas guías de onda, variando su anchura y espesor. El mismo estudio fue llevado a cabo en guías de onda activas con distintas dosis de silicio implantado, obteniendo valores de pérdidas alrededor de 7 dB/cm en el visible (a 780 nm). Además, la utilización de la *Shifting excitation spot technique* (SES) permitió analizar en profundidad las pérdidas por propagación en el interior del material activo y extraer su dependencia en un rango continuo de longitudes de onda pertenecientes al espectro de emisión del fotoluminiscencia (aproximadamente entre 550 nm y 900 nm).

Como resultado de la optimización del material activo en términos de intensidad de fotoluminiscencia y pérdidas ópticas, se consiguieron realizar cavidades resonantes de alta emisión luminosa y buenos factores de calidad. En un nuevo montaje experimental de μ -PL desarrollado íntegramente para el estudio de estos dispositivos, se obtuvieron valores máximos de $Q = 1.4 \times 10^4$ en un rango espectral ancho en el visible. Cabe destacar que los factores de calidad encontrados son los mejores nunca reportados en la literatura para cavidades circulares activas basadas en silicio y sólo están limitados por la resolución de nuestro montaje experimental. La potencia emitida en cada resonancia ha sido medida y cuantificada, resultando en un valor de nW. Este valor permite la utilización de detectores de silicio integrados, ya que estos son capaces de medir potencias de este calibre, permitiendo la fabricación de un sistema integrado que comprenda las cavidades resonantes y los detectores de forma conjunta.

Finalmente, una vez caracterizadas las estructuras aisladas, se procedió al estudio de las estructuras acopladas a la guías de onda. Debido a un problema en la planaridad de la capa de separación situada entre el microdisco y la WG, se obtuvieron factores de calidad inferiores a los de las estructuras aisladas (del orden de 1.48×10^3). No obstante, ha sido posible el desarrollo de una prueba de concepto en la que se plantea la posibilidad de utilizar estas estructuras acopladas como sensores ópticos. Estos sensores integrados monitorean los cambios de índice de refracción efectivo del modo óptico resonante inducidos por variaciones del índice de refracción del material del cual estan rodeados (analito). Con el fin de evaluar la sensibilidad del dispositivo, se han llevado a cabo medidas de μ -PL cambiando el entorno del microdisco y monitorizando el desplazamiento espectral que sufre una determinada resonancia. En particular, se varió el índice de refracción del analito en función de la fracción molar de metanol y etanol en una solución conjunta de metanol y etanol.

El resultado de estas medidas muestra un desplazamiento de 1.37 nm como consecuencia de un índice de refracción de $\Delta n = 0.0038 \text{ RIU}$ (refractive index unit). Desde la pendiente del fit lineal, realizada teniendo en cuenta todos los puntos experimentales, podemos extraer una sensibilidad máxima de 51.79 nm/RIU y un cambio mínimo de índice de refracción de $1.15 \times 10^{-3} \text{ RIU}$.

Teniendo en cuenta estos resultados, creemos que los microdiscos de SRSN tienen un gran potencial como “*building blocks*” de una plataforma fotónica de sensado donde la demultiplexación y detección pueden ser integradas en el mismo chip.

Así, siendo posible la unión de la fotónica y la electrónica en un mismo chip funcional, preveemos que esta nueva tecnología puede ser uno de los puntos clave para el desarrollo de sistemas sensores de bajo coste, alta sensibilidad y una huella electrónica extramadamente pequeña, permitiendo el análisis de baja cantidad de analitos.

Chapter 1

Introduction

One of the most intensely focused area of research and technology is without doubt the Silicon-based photonics. [1] The objective of this applied science is to generate and control the flow of the light at micro-nanometric scale in compounds based on silicon, in order to integrate optics into electronic circuit chips. Indeed photons can be used to carry out functions that traditionally fell within the domain of electronics, such as telecommunications and information processing. [2]-[4]

The most important appeal is given by the fact that Silicon photonic devices can be made using current standard and low-cost semiconductor manufacturing techniques, and also because Si is already used as substrate in integrated circuits fabrication. Further advantages deriving by the use of the Si are represented by the existence of a companion insulator oxide, the silica, having lower refractive index respect to the Si ($n_{Si}=3.5$, $n_{SiO_2}=1.45$) and by its 1.12 eV band gap, ideal for room temperature operation.

Among the numerous challenges in Si-photonics, one still unsolved problem concerns the realization of inexpensive integrated light sources on a Si circuit using *Complementary Metal-Oxide Semiconductor* (CMOS) compatible approaches.[5]-[7] Indeed, the most important drawbacks preventing the fulfillment of this objective is the lack of an efficient light emission from silicon, mainly because of the indirect band gap in its energy band structure. [8] The only way to generate photon emission in bulk silicon is in fact due to an electron-hole phonon-assisted recombination: this kind of low-probability process competes with fast non-radiative recombination paths, such as Auger and free carrier

absorption, quenching in fact the light emission process. [5],[9]

Perhaps the most interesting solution in order to increase the radiative emission probability, is to take advantage of the quantum confinement effects appearing when reducing the Si to nanometric dimension. Therefore, nanostructured Silicon - Silicon nanoclusters (*Si-ncl*) - embedded in a dielectric matrix, has been extensively investigated so far, and results to be one of the most promising material to turn Si in an effective light emitter.[10] The main idea that we will develop in this work is to avail of the described research as a starting point, and apply such know-how in the sensoristic field. The technological field described so far, is indeed giving new opportunities that are growing in parallel to the research of the Si-based integrated source of light: in the last decade in fact, a large variety of Si-based integrated photonic elements found application in the sensoristic field. [11]-[15]

Different approaches of direct detection, not always based on Si materials, have already been reported such as Mach-Zehnder interferometers, [16]-[17] surface plasmon resonator (SPR) [18] and optical waveguide based sensors.[19] Most of them can guarantee very high performances in terms minimum measurable change of refractive index of the analyte (also known as detection limit (DL)) from 10^{-5} to 10^{-8} refractive index unit (RIU). The main drawbacks regarding these mentioned methods are that they require a relatively large interaction length with the analyte, or a bulky light coupling system, decreasing the compactness of the device. On the contrary, sensing photonic structures based on integrated optical resonators such as rings/disks or 2-D photonic crystals [20]-[21] allows robust and compact on-chip integration suitable for high volume production and field use. In particular, circular μ -resonators cavities present reasonably high DL (up to 10^{-4} RIU for Si-based devices [22]-[23] and 10^{-7} RIU for polymeric μ -resonators [24]) and sensitivities (S) of 10^2 nm/RIU [23], 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: CMOS compatibility, high refractive index ($n \geq 2$), efficient photoluminescence (PL) emission in the visible range, fast recombination rates [25] and good characteristics for achieving efficient electrical injection due to the relatively low $Si - Si_3N_4$ band offsets [26]. 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.

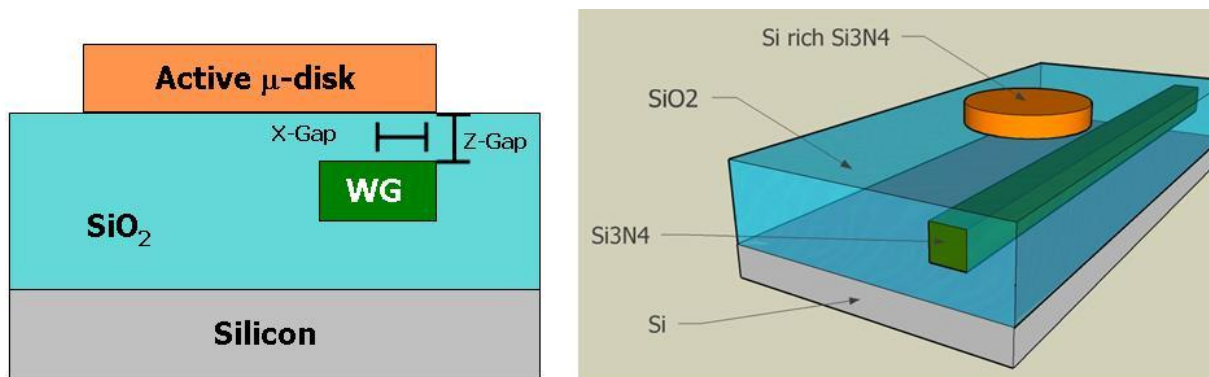


Figure 1.1: *Left panel - Scheme of the cross section of the coupled structure, in which the variables Z-Gap and X-Gap are graphically defined. Right panel - 3D sketch representing the overall sensing device.*

In this thesis, we propose and characterize a basic sensing photonic structure consisting on a μ -disk cavity made of a SRSN material coupled to a passive low losses stoichiometric Si_3N_4 waveguide (WG) placed underneath.

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.

1.1 Structure of the work

Hereafter we will introduce the contents of the chapters composing this thesis. Although many of the processes that will be presented (simulation of the optical structures, production of the samples, characterization etc..) were conducted in parallel, we have decided to describe them separately, in order to provide to the reader a better comprehension of the work.

1.1.1 Si-nc photoluminescence guided inside optical structures

The first chapter is dedicated to the discussion on the physical mechanisms governing the PL emission inside the active dielectric matrix. We will hence compare the characteristics two different dielectric matrices - SiO_2 and Si_3N_4 - describing which advantages they can bring from an optical and electrical point of view. On the base of those assumptions we will justify the choice of the SRSN as active material for the active resonators.

In the second part of the chapter we will introduce the fundamentals of how the light generated with the PL process can be guided inside a circular μ -resonator.

1.1.2 Design and Simulation of the optical structures

In the second chapter the methods and the parameters used during the simulation process of the optical structures will be described: at the first stage the simulation involves the geometric design of the passive WG, representing the connection between the μ -cavity and an eventual detector. Further simulations have been performed in order to study the modal structure of the μ -resonators (i.e. the position of the resonance peaks and electric field spatial distribution of the different modes inside the cavity), which were modified by varying the structural parameters of the optical elements. The aim of this study is to obtain mono-modal behaviours with quite high free optical ranges and quality factors, which were estimated by using a three-dimensional FDTD package.

Finally we will present the optimization of the coupling between the passive waveguide and the active μ -cavities, obtained by modifying the geometrical parameters describing the optical elements - resonator and WG - and its relative position.

1.1.3 Fabrication of process

The third chapter is focused on the description of all the technological process implied in the fabrication of both passive and active optical structures. All the samples were produced using CMOS compatible techniques. The geometrical parameters of the deposition (thickness of the active-passive layers and geometry of the optical structures) are the results of the simulation work presented in the previous chapter.

Furthermore we will show all the morphological analysis, performed in order to estimate the effective quality of the produced samples.

1.1.4 Experimental Setup and Characterization Techniques

The description of the experimental setup in which we have performed all the optical measurements is the subject of the fourth chapter.

In this part of the thesis we will also and explain all the different techniques used in order to measure the losses of the samples and to visualize the resonance PL spectra of the cavities.

1.1.5 Optical Losses and PL characterization

The estimation of the optical losses of the passive WG is the theme that developed in the first part of Chapter 6. Taking advantage of the techniques described in chapter 5, a large variety of passive devices on the the produced samples have been analysed (in both visible and infrared spectral region) and compared in order to find the optimal parameters (geometrical shape and dimensions, etching type, annealing temperature and wavelength dependence) allowing us to fabricate low losses structures.

In the second part of this chapter we will go through the characterization of the active material that we will use inside the μ -cavities. The application of the *Shifting Excitation Spot* technique (SES) to a 2-dimensional WG structure will bring to the achievement of the optical losses in a continuous spectrum in the VIS region.

1.1.6 Circular resonant structures

In this chapter we will present an experimental characterization of the isolated resonator structures, mainly performed by means of visible μ -PL. The PL spectrum emitted by the active material is modulated by the Whispering Gallery Mode (WGM) spectrum, which is characteristic of the cavity. The main objective of this part is therefore to select the best active material in function of the quality factor of the cavity and intensity of emitted PL.

In the last part of this section we show the operational principle of the coupled structures, based on the detection of the induced changes in the effective refractive index of the cavity supported modes.

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.

Chapter 2

Si-ns photoluminescence guided inside optical structures

In the first part of the present chapter we will describe the light emission properties of Si nanostructures (Si-ns), whose important role played in photonics has been already mentioned in the introduction of this thesis. In particular we will stress that the Si-ns emission is several orders of magnitude more efficient than bulk Si.[27] Moreover our attention will be focused on the effect due to the different dielectric matrix in which the Si-ns are grown (that in our case are Silicon Oxide (SiO_2) or Silicon Nitride (Si_3N_4)) and we will motivate the choice of Si_3N_4 matrix in the realization of our devices.

Before starting to describe the design and characterization process of the optical structure, that will be developed in the next chapters, it is important to give some notions about how the light generated inside the active material behaves inside a circular resonator (i.e. μ -disks and μ -rings), forming an optical mode.

Some of the described concepts will also be extended in Section 2.2 to the circular resonator, where useful parameters (e.g. quality factor, free spectral range, the finesse, ecc..) will be introduced.

2.1 Principles of emission of Si-ns in Si_3N_4 matrix

As seen in Chapter 1, even if considered the most important material for the realization of integrated circuits due to its cheap CMOS compatible fabrication technology, the bulk Si presents bad light emitting properties. The main limitation to the achievement of an efficient light emission process in Si is represented by its indirect electronic band gap. In Si in fact, the conduction band minimum and the valence band maximum occur at two different values of crystal momentum (Figure 2.1). This situation makes necessary the emission or absorption of momentum-conserving phonons in order to achieve a radiative recombination of the exciton.

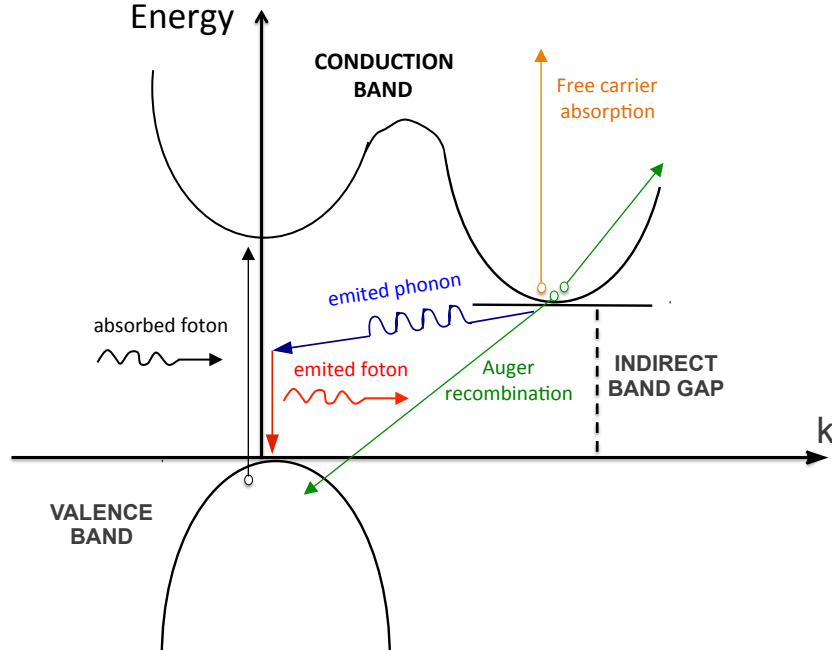


Figure 2.1: Scheme of the indirect band gap structure of bulk Si. The possible transitions for an electron-hole pair (phonon-assisted radiative recombination, Auger recombination, and free-carrier absorption) are also represented.

In bulk Si, competitive non-radiative recombination rates are much higher than the radiative ones and most of the excited electron-hole pairs ($e-h$) recombine non-radiatively. This yields very low internal quantum efficiency ($\eta_{int} \sim 10^{-6}$) - defined as the fraction of

$e-h$ pairs that recombine radiatively - for bulk silicon luminescence.

In addition, non-radiative processes such as Auger - in which the $e-h$ pair give its recombination energy to another electron leading to an intra-band transition - strongly contribute to the quenching of the bulk material light emission. Another non radiative mechanism competing with the radiative one is the recombination of the electrons with the matrix defects: indeed the large mobility of electrons in Si ($1.4 \times 10^3 \frac{cm^2}{V.s}$ reported in ref [28]) results in a non negligible recombination effect contributing to the lowering of the photon emission in bulk undoped Si.

Because of these reasons, while the development of passive elements such as waveguides, splitters and active devices such as modulators has already found success, an efficient electrically pumped CMOS compatible optical source has not been demonstrated so far. In order to improve Si light emission performances is thus fundamental to increase the radiative recombination rate of the formed exciton while decreasing the electrons mobility. One possible solution is given by the possibility to reduce the Silicon to nanometric dimensions (e.g. porous silicon [29], Si nanoparticles [30], Si nanocrystals embedded in Si-oxide [31]-[32]), where quantum confinement effects start to play a fundamental role in the description of light emission mechanism.

The most common method to obtain nanostructured Si, described in a large number of paper, is the nucleation of Si inside a SiO_2 matrix (*SRSO*), achieved by a thermal annealing of Si-rich oxide [33]-[38], Si/SiO_2 or SiO/SiO_2 superlattices [39]. Several other methods are described in literature in order to obtain nanostructured Si and among them it is worth to mention the electrochemical etching [40]-[41], laser-assisted processing [42]-[43], solution synthesis [44].

While visible PL of Si nano-structures have already demonstrated, the physical mechanism of such emission is still not completely understood. In order to find an agreement between experimental results and theoretical prediction, near the mere *quantum confinement mechanism* a number of theoretical models were developed: some of them describe the PL as result of recombination mechanisms due to interface states [45],[46], luminescent defect states [47] and oxygen vacancy states at the $Si=SiO_2$ interface [48]. Despite this large number of possible process, two mechanisms of radiative recombination in Si

2.1. PRINCIPLES OF EMISSION OF SI-NS IN Si_3N_4 MATRIX

nano crystal systems are recognized to be dominant in PL emission: after the creation of the exciton inside the nanocrystal, the radiative recombination can occur either inside the nanocrystal or outside at a localized state on the Si-ns interface. In the latter context is clear that the matrix plays a crucial role in tailoring the electronic structure of the Si-ns, influencing as consequence the PL emission properties.

One drawback preventing to realize stable and efficient electroluminescent photonic devices based on *SRSO* are the relatively high band gap of SiO_2 with respect to the *Si*. The other one is given by the low refractive index difference between the SiO_2 (around $n_{SiO_2} = 1.46$ at 750 nm) and the air ($n_{air} = 1$): due to the low n contrast is indeed difficult to fabricate high Q active SRSO circular μ -cavities directly on the SiO_2 cladding, and the realization of more complex structure as free standing μ -disks is required.[49]-[50]

In order to overcome those issues alternative CMOS compatibles host materials have been investigated, and one of the most valuable candidate has resulted to be the Si_3N_4 .

Beyond the efficient room temperature visible PL, demonstrated for Si-nc embedded in Si nitride matrices by many groups [51]-[54], *SRSN* provides in fact several appealing properties for fabricating compact and efficient emitting devices. Inducing the nucleation of Si clusters inside Si_3N_4 we therefore obtain a compound having both good optical and electrical properties, represented by a higher refractive index ($n_{SRSN} > 2$ at 750 nm) and efficient electrical injection due to the lower electron/hole injection barrier at the *Si-Si₃N₄* interfaces [26] with respect to the *Si-SRSO* one. The conduction mechanism in *SRSN* matrix is indeed governed by shallow-trap-assisted conduction, that is more effective respect to the tunnelling mechanism typical of the *SRSO*. [55]-[56] This mechanism results in the possibility of fabrication of low-voltage electroluminescent devices.[57] Furthermore the high refractive index value implies a greater index contrast with the surrounding material, giving more freedom in the design of optical elements like μ -disks: it is in fact no longer necessary to make use of the aforesaid free standing resonator cavities in order to decrease the losses, as in the *SRSO* case, but they can be created directly on a SiO_2 substrate.

Similarly to what happens for the conduction mechanism, also the origin of PL emission in *Si* rich Si_3N_4 is quite different from that of *SRSO*: authors have demonstrated that

in *SRSN* layers the PL emission comes out from the defect centres, the Si nano-clusters or even the interface states. This is principally due to the different growth kinetics that the Si-nc follow in amorphous nitride host matrix, that is very different than in *SRSO*, and characterized by the formation after the annealing process of smaller Si clusters (around 3 nm of diameter) at a faster rate and lower temperature [59]. This also results in a large number of vibrational configuration allowed in *SRSN* by *Si-N* or *Si-H* interaction (Si-N stretching, *Si-N* breathing, *Si-H* rocking, Si-H stretching [2]) respect to the *Si=O* double bonds of *SRSO*. For all those reasons it is clear that the amorphous or crystalline nature of Si in *SRSN* strongly depends on the growth parameters (e.g. thermal annealing, Si excess etc..) and method of fabrication. All those parameters will be listed more in detail in Chapter 4, where we will describe the fabrication method used in the preparation of our samples.

2.2 Light propagation in a circular resonator

Once we have listed the advantages coming out from the use of the nitride respect to an oxide matrix and the mechanisms governing the PL emission in *SRSN*, it is useful to understand what is its behaviour when generated inside an circular resonator cavity. In particular we focus our attention on the circular resonators, such as disks or rings, with radius of few μm . (Further notions about how the light is confined in a WG can be found in the Appendix A)

First of all it is worth to describe the domain in which we are working and, in the case of an isotropic medium, such description is given by the following set of Maxwell equations:

$$\nabla \times \vec{E}(r, t) + \frac{\partial \vec{B}(r, t)}{\partial t} = 0 \quad \text{MAXWELL-FARADAY EQUATION} \quad (2.1)$$

$$\nabla \times \vec{H}(r, t) - \frac{\partial \vec{D}(r, t)}{\partial t} = j \quad \text{AMPÈRE'S CIRCUITAL LAW} \quad (2.2)$$

$$\nabla \cdot \vec{D}(r, t) = \rho \quad \text{GAUSS'S LAW} \quad (2.3)$$

$$\nabla \cdot \vec{B}(r, t) = 0 \quad \text{GAUSS'S LAW FOR MAGNETISM} \quad (2.4)$$

where $\vec{H}(r, t) = \frac{1}{\mu_0} \vec{B}(r, t)$ (for non-magnetic materials) and $\vec{D}(r, t) = \epsilon(r) \vec{E}(r, t)$. In the case in which no free charge are present and there is not current flowing ($j = 0$), the temporal dependence of the solutions is given by an oscillating function ($e^{-i\omega t}$), so that the previously set of equations becomes:

$$\nabla \times \vec{E}(r) - i\omega \vec{B}(r) = 0 \quad (2.5)$$

$$\nabla \times \vec{H}(r) + i\omega \vec{D}(r) = 0 \quad (2.6)$$

$$\nabla \cdot \vec{D}(r) = 0 \quad (2.7)$$

$$\nabla \cdot \vec{B}(r) = 0 \quad (2.8)$$

At this point, if we apply the curl operator on the Equation 2.5, and substituting in it the Equation 2.6, we obtain the master equation describing the temporal and spacial evolution of the electric field:

$$\nabla \times \nabla \times \vec{E}(r) - k_0^2 n^2(r) \vec{E}(r) = 0 \quad (2.9)$$

where we have substituted $k_0 = \omega/c_0$, $c_0 = 1/\sqrt{\mu_0\epsilon_0}$ and $n^2(r) = \epsilon(r)/\epsilon_0$. Similarly we can obtain the master equation of the magnetic field B by taking the curl of Equation 2.6 and substituting in it the Equation 2.5.

In the case of a circular μ -resonator the master equation can be completed introducing the azimuthal symmetry of the refractive index, given by $\vec{n}^2(r) = \vec{n}^2(\rho, z)$. Taking in account the mathematical expression for the double curl $\nabla \times \nabla \times \vec{E} = \nabla \nabla \cdot \vec{E} - \nabla^2 \vec{E}$, and substituting the relation $\nabla \cdot \vec{E} = -\frac{1}{n^2(\rho, z)} \vec{E} \cdot \nabla n^2(\rho, z)$ [61] we obtain:

$$\nabla^2 \vec{E} + \nabla \left(\frac{1}{n^2(\rho, z)} \vec{E}(r) \cdot \nabla n^2(\rho, z) \right) + k_0^2 n^2(r) \vec{E}(r) = 0 \quad (2.10)$$

2.2. LIGHT PROPAGATION IN A CIRCULAR RESONATOR

The same equation can be derived for the magnetic field. It is possible to demonstrate that those two equations, assuming a piecewise homogeneous medium, can be brought back to the Helmholtz equation:

$$\nabla^2 \vec{U}(r) + k_0^2 n^2(r) \vec{U}(r) = 0 \quad \text{HELMHOLTZ EQUATION} \quad (2.11)$$

where $U(\vec{r})$ can be either the electric or magnetic field. Furthermore, by substituting the expression of the Laplacian operator in cylindrical coordinates (always allowed by the symmetry of the system under examination), we then obtain:

$$\left[\frac{\partial^2}{\partial \rho^2} + \frac{1}{\rho} \frac{\partial}{\partial \rho} + \frac{1}{\rho^2} \frac{\partial^2}{\partial \phi^2} + \frac{\partial^2}{\partial z^2} + k_0^2 n^2(\rho, z) \right] \vec{U}(r) = 0 \quad (2.12)$$

The problem can be further simplified considering that a real circular resonator is not a cylinder of infinite height, so we can assume that the radiation inside the cavity travels on a plane rather than in a three dimensional structure.

We can now separate each component of Equation 2.12 with $U(r) = P(\rho) \cdot \Phi(\phi) \cdot Z(z)$, obtaining the following equations, in which n_{eff} represents the effective index:

$$\left(\frac{d^2}{dz^2} + k_0^2 (n^2(z) - n_{eff}^2) \right) \vec{Z}(z) = 0 \quad (2.13)$$

$$\left(\frac{d^2}{d\phi^2} + m^2 \right) \vec{\Phi}(\phi) = 0 \quad (2.14)$$

$$\frac{d^2 \vec{P}(\rho)}{d\rho^2} + \frac{1}{\rho} \frac{d\vec{P}(\rho)}{d\rho} + \left(k_0^2 n^2(\rho) - \frac{l^2}{\rho^2} \right) \vec{P}(\rho) = 0 \quad (2.15)$$

The possibles solutions to the former Equations are the following:

- Equation 2.13 can be solved following the standard slab mode calculation already reported in Appendix A, keeping in mind that the U_z function is continuous at the interface for the TE modes, and is discontinuous for the TM ones.

2.2. LIGHT PROPAGATION IN A CIRCULAR RESONATOR

- The solutions of Equation 2.14 are functions similar to $\Phi_m = \Phi_n^0 e^{im\phi}$, where m is a positive integer ($m = 1, 2, 3, \dots$)
- The radial part of the system - represented by the Equation 2.15 - can not be solved analytically, but can be approximated with a *Bessel* function of first kind and order l , depending of the zone in which we are calculating the field dispersion: inside the resonator ($r < \rho$) the solution is given by a first order like $P(\rho) \approx J_l(k_0, n_{eff}, \rho)$ (represented in the left panel of Figure 2.2), while outside the resonator ($r > \rho$) the solution is *Hankel* function, that is a linear combination of first and second kind Bessel function (the second kind function are also called *Neumann* functions $N_p(k_0, n_{eff}, \rho)$), and it can be written as $P(\rho) \approx J_l(k_0, n_{eff}, \rho) + iN_p(k_0, n_{eff}, \rho)$.

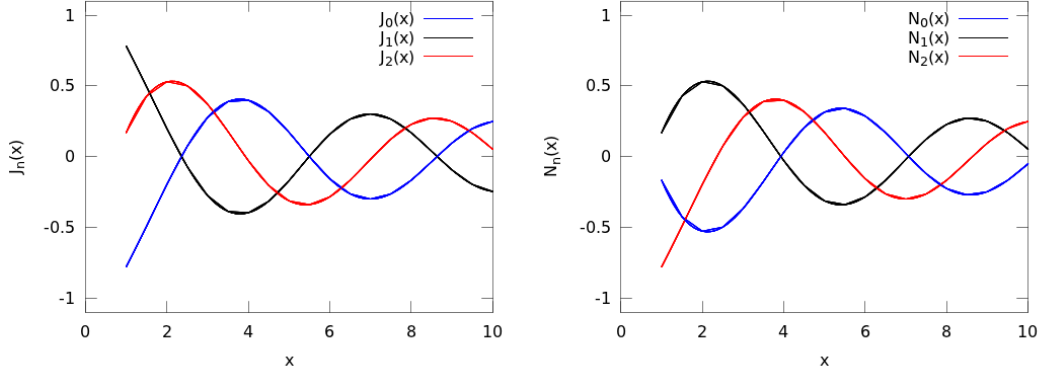


Figure 2.2: Representation of the first kind Bessel function (first and second order) $J_0(k_0, n_{eff}, \rho)$, $J_1(k_0, n_{eff}, \rho)$ and $J_2(k_0, n_{eff}, \rho)$ (left panel) and second kind Bessel function $N_0(k_0, n_{eff}, \rho)$, $N_1(k_0, n_{eff}, \rho)$ and $N_2(k_0, n_{eff}, \rho)$ such functions oscillates like a sine or cosine function but with a decaying amplitude.

Once defined the equations describing the field distribution inside the resonator we can find which are the modes (the eigenvalues of those equations) supported by the cavity and its corresponding frequencies. A mode (or *Whispering Gallery Mode* - WGM) is therefore characterized by two numbers (m and l) for which the equations 2.13, 2.14 and 2.15 admit a non zero solution.

The parameter m in Equation 2.14 is the order of the WGM, representing the number

2.2. LIGHT PROPAGATION IN A CIRCULAR RESONATOR

of nodes along ϕ , and is given by imposing the self-consistency condition deriving from the particular system that we are analysing. As reported in Appendix A, for the linear WG the EM field has to reproduce itself after a double reflection process: similarly, the EM field inside a circular resonator has to reproduce itself after a complete round trip (see figure 2.3 in which a the bouncing of a phasor Φ inside the resonator is represented). Considering that in a circular cavity the total path of a round trip is given by $2\pi R$ - being R the radius - the solutions of the Equation 2.14 are thus restricted to the values of k_0 satisfying the condition:

$$k_0 = \frac{m}{n \cdot R} \rightarrow m = 1, 2, 3, \dots \quad (2.16)$$

from which is immediate to find the corresponding discrete values of frequency ν supported by the cavity:

$$\nu = m \frac{c}{2\pi R} \rightarrow m = 1, 2, 3, \dots \quad (2.17)$$

where $c = \frac{c_0}{n}$, being c_0 the light speed in the free space, and $k_0 = \frac{2\pi\nu}{c_0}$.

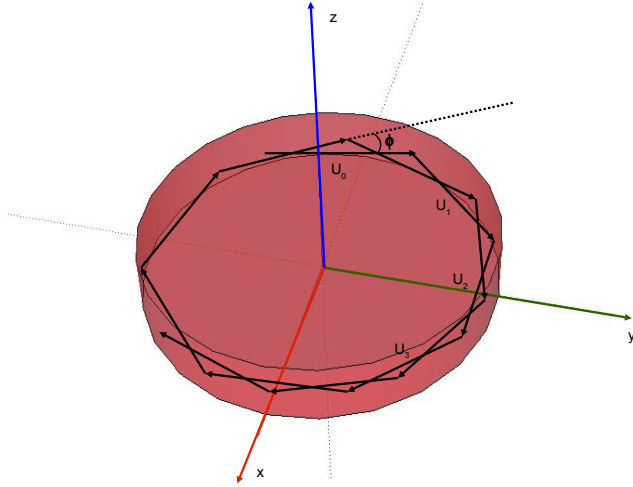


Figure 2.3: *Bouncing wave inside a circular resonator, suffering a phase shift $\phi \neq q2\pi$ each round trip.*

In the following we will introduce some of the main parameters that we will use in the analysis of the μ -cavities, that are the Free Spectral Range, the Quality Factor and the Finesse of the resonator.

2.2.1 Free Spectral Range

The Free Spectral Range (FSR) is defined as the spacing in wavelength or in frequency existing between two consecutive resonances, defined as m and $m+1$. Such distance, inside a circular resonator of radius R , can be written as the difference between the following equations:

$$m\lambda_m = n_{eff}2\pi R \quad (2.18)$$

$$(m+1)\lambda_{m+1} = \left(n_{eff} - \frac{\partial n_{eff}}{\partial \lambda}(\lambda_m - \lambda_{m+1}) \right) 2\pi R \quad (2.19)$$

where n_{eff} represent the effective refractive index and λ_m is the wavelength value of the maximum of the m^{th} resonance. Therefore the aforesaid difference can be written as :

$$FSR = \delta\lambda_{FSR} = \lambda_m - \lambda_{m+1} = \frac{\bar{\lambda}^2}{n_g 2\pi R} \quad (2.20)$$

in which the group index is defined as $n_g = n_{eff} - \frac{\partial n_{eff}}{\partial \lambda}\lambda$ and the average wavelength is given by $\bar{\lambda}^2 \approx \lambda_m \cdot \lambda_{m+1}$.

As we can see in Equation 2.20 the FSR is inversely proportional to radius of the cavity, and this is a very important property that we have to take in account during the design of the resonators.

Furthermore, the FSR can be also used as an indirect measure of the group velocity, defined as $v_g = \frac{\partial \omega}{\partial k_0}$, with k_0 propagation constant of the wave-packet of angular frequency centred at ω . Such velocity is also related to the group index, being v_g the velocity of the wave-packet inside the medium, so that $v_g = \frac{c_0}{n_g}$. If we substitute this definition in the Equation 2.20 we find:

$$v_g = \frac{c_0 \cdot FSR \cdot 2\pi R}{\bar{\lambda}^2} \quad (2.21)$$

2.2.2 Finesse

We can now define a positive parameter giving information on the losses inside the cavity: the Finesse ($\mathcal{F}$) of the resonator. This particular variable can be described as the ratio

between the spacing of the modes supported by the cavity ν_f (*FSR*) and the *FWHM* $\delta\nu$ of the resonances.

$$\mathcal{F} = \frac{\nu_f}{\delta\nu} \quad (2.22)$$

Considering the magnitude ratio ξ of two phasors after a complete round trip inside the resonator (round-trip amplitude attenuation), induced by the internal reflections and absorption of the medium, the $\mathcal{F}$ can be also defined with the following formula:

$$\mathcal{F} = \frac{\pi\xi^{1/2}}{1 - \xi} \quad (2.23)$$

In a perfectly lossless resonator, the $\mathcal{F}$ goes to infinite, as the attenuation term ξ is equal to zero. Furthermore the intensity of the radiation inside the resonator is linked to $\mathcal{F}$ by the relation:

$$I = \frac{I_{max}}{1 + (2\mathcal{F}/\pi)^2 \sin^2(\phi/2)} \quad (2.24)$$

If $\mathcal{F}$ is very high, the intensity, that is a periodic function of ϕ with period 2π , presents a sharp peak centred in all the $\phi = q2\pi$ values.

2.2.3 Quality Factor

At this point it is important to define another parameter that we will use a lot in the characterization of the optical μ -resonators: the Quality Factor (Q). The Q factor represent the ration between the energy stored inside the cavity ($U_c(t)$) and the energy lost at every cycle.

$$Q = \frac{2\pi(\text{stored energy})}{\text{energy loss per cycle}} = 2\pi \frac{U_c(t)}{-T \frac{dU_c(t)}{dt}} = -\frac{\omega U_c(t)}{\frac{dU_c(t)}{dt}} \quad (2.25)$$

where T is the period of the oscillation and ω is its angular frequency. The solution of the last equation are functions like $U_c(t) = U_c(0)e^{-\frac{\omega t}{Q}}$. On the other hand, $U_c(t)$ can also be related to standard loss coefficients (α) via the group refractive index (n_g) of the propagating mode, so that $U_c(t) = U_c(0)e^{-\frac{\alpha \cdot c_0 \cdot t}{n_g}}$. By comparing both expressions we can deduce a relation between Q and α in the form of:

$$\alpha = \frac{\omega \cdot n_g}{Q \cdot c_0} = \frac{2\pi \cdot n_g}{Q \cdot \lambda} \quad (2.26)$$

2.2. LIGHT PROPAGATION IN A CIRCULAR RESONATOR

The Q factor has a great importance since it can supply information about the photon lifetime τ_p inside the cavity. As consequence of the presence of an attenuation in the plane wave amplitude, the photon lifetime inside of the resonator is a finite value τ_p . The inverse of this value is called Full Width at Half Maximum (*FWHM*) and it represents the spectral broadening of the resonance peak.

$$\delta\nu = \frac{1}{2\pi\tau_p} \quad (2.27)$$

By using the Equation 2.27 the Q factor can be thus expressed as:

$$Q = \frac{\nu_0}{\delta\nu} = 2\pi\nu_0\tau_p \quad (2.28)$$

Finally, substituting in Equation 2.22 in 2.28 we can found the correlation between Q and $\mathcal{F}$:

$$Q = \frac{\nu_0}{\nu_F} \mathcal{F} \quad (2.29)$$

As reported in Equation 2.25 the total Q of a cavity is inversely proportional to the energy lost in one cycle. Such energy lost is given by the sum of all losses contribution (e.g. material losses due to bulk absorption and propagation, surface scattering, surface absorption, etc..). It is therefore possible to decompose the inverse of Q as the sum of the inverse Q associate to all those components:

$$\frac{1}{Q_{tot}} = \frac{1}{Q_{rad}} + \frac{1}{Q_{mat}} + \frac{1}{Q_{scat}} + \dots \quad (2.30)$$

A more complete description and evaluation of all the source of losses will be given in Chapter 6.

Chapter 3

Design and Simulation of the optical structures

In the last decade many commercial and free optical simulation software packages have been developed in order to study the propagation of the light inside dielectric material. Most of them are based on two principal computational techniques: the *Beam Propagation Method* (BPM) [62] and the *Finite Difference in Time Domain method* (FDTD) [63], both conceived in the '70s. The principles of these two numerical algorithm will be described in the following sections together with the application that have been found in our work. The first part of the simulation step concerns the study of the isolated structures: the geometrical variables have been varied in order to optimize the involved parameters (i.e. Quality factor, FSR, optical losses, transmitted PL intensity).

In the last part of the chapter is described the simulation of the final structure composing the sensor device, constituted by the circular resonator vertically coupled to the WG.

3.1 Simulation Softwares

Here after we will describe the different tools involved in the simulation process and its functioning. Depending on the optical element under analysis we will perform simulations using a different algorithm. In particular the WG dimensions have been optimized

using a BPM tool (the *BeamPROPTM* produced by the *Rsoft Design Group*) [64], while the resonators have been simulated by means of a FDTD software package (the *MEEP*, developed in the Massachusetts Institute of Technology) [65].

3.1.1 Beam Propagation Method

The Beam Propagation Method (BPM) is a computational technique applied in order to investigate the dynamics of an Electromagnetic (EM) field propagating through arbitrary inhomogeneous medium. It is typically used in order to solve the intensity and modes in WG structures. With this method, the mode profile of an unusual WG such as *y-couplers* can be determined with ease. The BPM exploit a Finite Difference (FD) method in order to solve the Helmholtz equation 2.11 under parabolic approximation. Such approximation implies the paraxiality condition on the main direction of propagation, that is also probably the most important limitation to the use of this technique. Indeed the EM field under analysis can be calculated primarily along one axis making the BPM deficient in modelling complicated structures.

Similarly to what happens applying a Fast Fourier Transform (FFT) to a periodic signal, the BPM is based on an algorithm able to decomposes a mode into a superposition of plane waves, each travelling in a different direction. The basic idea here is to split a complicated problem into a simpler problem for which solutions are obvious.

All these individual plane waves, which are sinusoids of different frequencies, are propagated through a finite distance defined by the calculation area, through the optical structure until the point where the field needs to be determined has arrived.

Thanks to the linearity of the wave equation, the original signal can be then re-obtained by adding all those sinusoidal waves in order to get back the spatial mode.

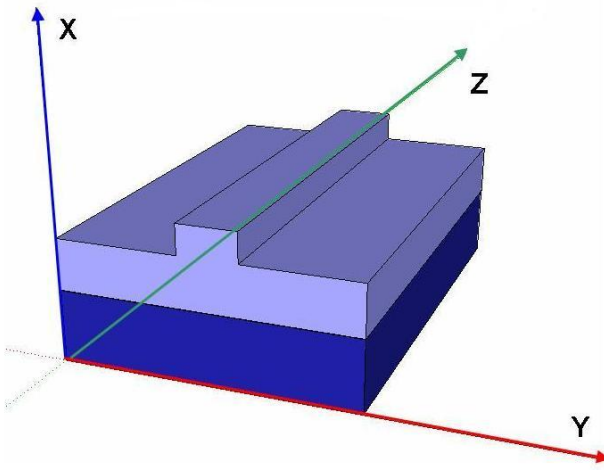


Figure 3.1: *Structure of a BPM simulation, in which the cross section of the structure under analysis is defined in the plane x - y , while the propagation of the EM wave is in the z direction.*

In this section the basic approach is illustrated by formulating the problem under the restrictions of a scalar field (neglecting the polarization effects) and paraxiality, which means that the propagation is restricted to a narrow range of angles. It is important to notice that some commercial software give the possibility to overcome those limitations by using a wide-angle and bi-directional extensions to BPM, coming out from a more accurate approximation of Helmholtz equations, that will not be discussed here.

The Helmholtz equation comes out from the time-independent form of the general wave equation:

$$\left(\nabla^2 - \frac{1}{c^2} \frac{\partial^2}{\partial t^2} \right) a(r, t) = 0 \quad (3.1)$$

where $a(r, t) = U(r)T(t)$ and $T(t) = e^{i\omega t}$.

$$(\nabla^2 + k^2)U(r) = 0 \quad (3.2)$$

assuming $k = \frac{\omega}{c}$. From Equation 3.2, using the scalar field assumption, we can write the wave equation as:

$$\frac{\partial^2 U}{\partial x^2} + \frac{\partial^2 U}{\partial y^2} + \frac{\partial^2 U}{\partial z^2} + k(x, y, z)^2 U = 0 \quad (3.3)$$

In eq 3.3 the electric field has been introduced in the scalar form as $E(x, y, z, t) = U(x, y, z)e^{-i\omega t}$ and the notation $k(x, y, z) = k_0 n(x, y, z)$ has been introduced for the spa-

tially dependent wavenumber, with $k_0 = 2\pi/\lambda$ being the wavenumber in free space. In BPM the whole geometry of the system is so defined by the refractive index distribution $n(x, y, z)$.

It is important to remark that in typical WG problems the most rapid variation in the field U is the phase variation due to propagation along the guiding axis. Therefore if we assume that the guiding axis is predominantly along the z direction, it is beneficial to factor such rapid variation out of the problem by introducing a slowly varying field u by means of the following assumption:

$$U(x, y, z) = u(x, y, z)e^{i\bar{k}z} \quad (3.4)$$

In this case $\bar{k}$ (*reference wavenumber*) is a constant number that represents the average phase variation of the field U . Such reference wavenumber is normally expressed in terms of a reference refractive index, $\bar{n}$, by the equation $\bar{k} = k_0\bar{n}$. At this point, if we introduce the 3.4 into the Helmholtz equation 3.3 we obtain the expression for the slowly varying field:

$$\frac{\partial^2 u}{\partial z^2} + 2i\bar{k}\frac{\partial u}{\partial z} + \frac{\partial^2 u}{\partial x^2} + \frac{\partial^2 u}{\partial y^2} + (k^2 - \bar{k}^2)u = 0 \quad (3.5)$$

The so obtained equation is completely equivalent to the exact Helmholtz equation, aside for its expression in terms of u . Assuming that the variation of u with z is sufficiently slow, the first term of eq 3.5 can be neglected respect to the second; such assumption is also called *slowly varying envelope approximation* and in this context it is also referred to as the paraxial or parabolic approximation. Taking in account that the Equation 3.5 can be reduced to:

$$\frac{\partial u}{\partial z} = \frac{i}{2\bar{k}} \left(\frac{\partial^2 u}{\partial x^2} + \frac{\partial^2 u}{\partial y^2} + (k^2 - \bar{k}^2)u \right) \quad (3.6)$$

Equation 3.6 is the basic three dimensional (3D) BPM equation under the restriction of scalar field and paraxiality.

One of the limitations of BPM is the possibility to introduce in the simulation only a

monochromatic wave, and not a broad excitation source.

The BPM method has been used in order to evaluate the optical losses of the rib WGs. (see Chapter 6)

3.1.2 FDTD method

The *Finite Difference in Time Domain* (FDTD) method is a powerful computational electromagnetic modeling technique permitting to solve time dependent Maxwell equations in a discretized spacial domain. The calculation window is therefore divided into a discrete grid and then the EM fields are evolved in time using discrete time steps. By increasing the grid elements and reducing the time steps, the FDTD solution of the simulation becomes a closer approximation for the true continuous equations, so it is possible to simulate the behaviour of the EM field inside an optical structure.

One of the main advantages of FDTD method (with respect for example to BPM) is that it is possible to introduce a multiple wavelength excitation source inside the structure and to simulate the temporal evolution of the EM field inside the region of interest. Since is a technique based on the time domain, with a single FDTD simulation and by means of *Discrete Fourier Transform* (DFT) we can obtain the frequency response (that we can than translate in wavelength) of the optical system under analysis. Indeed, by applying a temporal pulse, we obtain a frequency spectrum centred in the frequency of interest.

The field distribution in an isotropic medium are given by Maxwell equations (*MKS* units) previously defined in Section 2.2 by the Equations 2.1, 2.2, 2.3 and 2.4 , where j , μ and ϵ are assumed to be given function of space and time. If we consider a region of space which contains no flowing currents or isolated charge in ($J = 0$) and in which μ and ϵ are constant, the only source of problem is the incident wave on the system. With those assumptions the eq 2.1 and 2.2 are equivalent to the following system of scalar equation (Maxwell's curl equations in Cartesian coordinates):

$$\begin{cases} \frac{\partial H_x}{\partial t} = \frac{1}{\mu} \left(\frac{\partial E_y}{\partial z} - \frac{\partial E_z}{\partial y} \right) \\ \frac{\partial H_y}{\partial t} = \frac{1}{\mu} \left(\frac{\partial E_z}{\partial x} - \frac{\partial E_x}{\partial z} \right) \\ \frac{\partial H_z}{\partial t} = \frac{1}{\mu} \left(\frac{\partial E_x}{\partial y} - \frac{\partial E_y}{\partial x} \right) \end{cases} \quad (3.7)$$

$$\begin{cases} \frac{\partial E_x}{\partial t} = \frac{1}{\epsilon} \left(\frac{\partial H_z}{\partial y} - \frac{\partial H_y}{\partial z} \right) \\ \frac{\partial E_y}{\partial t} = \frac{1}{\epsilon} \left(\frac{\partial H_x}{\partial z} - \frac{\partial H_z}{\partial x} \right) \\ \frac{\partial E_z}{\partial t} = \frac{1}{\epsilon} \left(\frac{\partial H_y}{\partial x} - \frac{\partial H_x}{\partial y} \right) \end{cases} \quad (3.8)$$

These Maxwell's equations describe a situation in which the temporal change in the E field is dependent upon the spatial variation of the H field, and vice versa. As stated before the FDTD method works firstly discretizing Maxwell's equations in finite differences in time and space domain and then solving them numerically.

The most common method to solve numerically the eq. 3.7 and 3.8 is based on Yee's mesh: as shown in figure 3.2, the E and H field components are calculated at points on a grid with grid points spaced Δx , Δy , and Δz apart. Afterwards the E and the H field components are then interlaced in all three spatial dimensions.

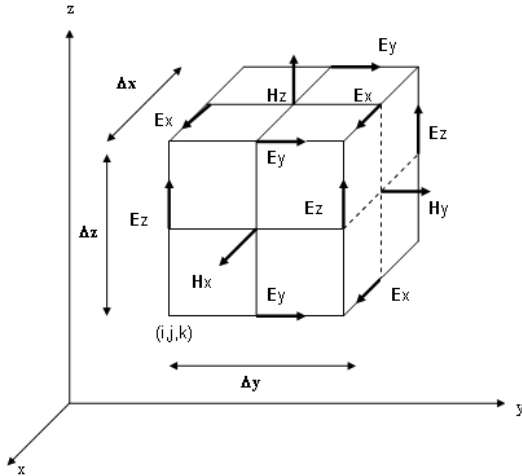


Figure 3.2: *Position of various field components on the Yee cell. The E -component are in the middle of the edges while the H -components lie in the center of the faces*

Furthermore, time is broken up into discrete steps of Δt . The E field components are

then computed at times $t = n\Delta t$ and the H fields at times $t = (n + 1/2) \Delta t$, where n is an integer representing the compute step. For example, the E field at a time $t = n\Delta t$ is equal to the E field at $t = (n - 1) \Delta t$ plus an additional term computed from the spatial variation, or curl, of the H field at time t .

This method results in six finite difference equations describing the electric and magnetic waves, that can be used to compute the field at a given mesh point, denoted by integers i, j, k .

$$H_{x(i,j,k)}^{n+1/2} = H_{x(i,j,k)}^{n-1/2} + \frac{\Delta t}{\mu \Delta z} (E_{y(i,j,k)}^n - E_{y(i,j,k-1)}^n) - \frac{\Delta t}{\mu \Delta y} (E_{z(i,j,k)}^n - E_{z(i,j-1,k)}^n) \quad (3.9)$$

$$E_{x(i,j,k)}^{n+1} = E_{x(i,j,k)}^n + \frac{\Delta t}{\epsilon \Delta y} (H_{z(i,j+1,k)}^{n+1/2} - H_{z(i,j,k)}^{n+1/2}) - \frac{\Delta t}{\epsilon \Delta z} (H_{z(i,j,k+1)}^{n+1/2} - H_{y(i,j,k)}^{n+1/2}) \quad (3.10)$$

These equations are then iteratively solved, alternating between computing the E and H fields at subsequent $\frac{\Delta t}{2}$ intervals.

3.1.3 Harminv

One of the most important problems that we have to solve in our simulation process is to find the resonant modes (frequencies and decay rates) of circular resonators. To this goal we have exploited Harminv [66], a free program associated with *MEEP* useful in order to solve the problem of harmonic inversion: without going too much into detail, such problem consists in the determination of the frequencies, decay constants, amplitudes, and phases of a signal that consists of a sum a finite number of exponentially decaying sinusoids in a given bandwidth. The fundamental assumption is to have a finite length system discretized in time: if these conditions are satisfied Harminv analyzes the signal $f(t)$ and determines all the value aforesaid variables (the frequencies ω_n , the decay constant α_n , the Q - that is a dimensionless quantity- absolute value of the amplitudes $|a_n|$ and the phase shift of the sinusoids ϕ_n).

$$f(t) = \sum_k A_k \sin(\omega_k \cdot t + \phi_k) e^{-\alpha_k \cdot t} \quad (3.11)$$

The Q factor is here defined as the number of periods for the energy in the mode - represented by the squared amplitude - to decay by $e^{-2\pi}$. Considering that the decay constant is the inverse of the previously defined photon lifetime τ_p , the value of Q can be determined using the Equation 2.28 as:

$$Q = 2\pi \frac{\nu_0}{\alpha_k} \quad (3.12)$$

The main advantage with respect to the packages using algorithms based on the Fast Fourier Transform (FFT), that represents any data as a sum of sinusoidal components, is that Harminv provides much better accuracy: indeed it assumes a specific form for the signal and is not thus limited by the uncertainty principle as in the FFT case.

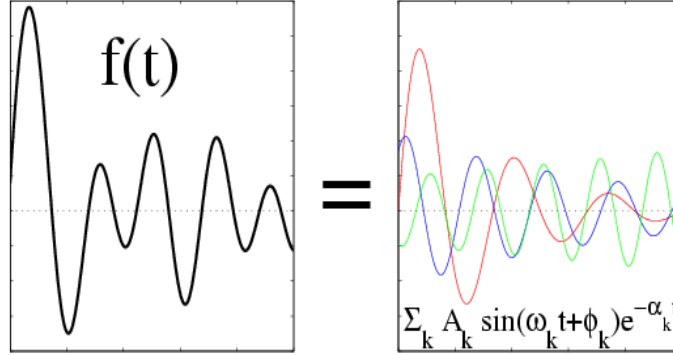


Figure 3.3: *Decomposition of a given $f(t)$ signal in a finite number of exponentially decaying sinusoids.*
(Picture taken from <http://ab-initio.mit.edu/wiki/index.php/Harminv>)

Harminv is also often more robust than directly least-squares method normally used in the data fitting process, which can have problematic convergence, since it re-expresses the problem in terms of simply finding the eigenvalues of a small matrix. Furthermore Harminv uses a low-storage “*filter diagonalization method*” (FDM) [67]-[68] for finding the sinusoids near a given frequency interval.

3.2 Modal structure of the μ -resonators

In the previous section we have described the different methods used to simulate the behaviour of the EM field in waveguide-based photonic systems. In this section we address the problem of solving the modal structure of the μ -disks, simulate the emission spectrum modulated by the cavity and to find the distribution of the EM field inside the resonator. Later on in section 3.3 we will describe the design of the final coupled system, composed by the active optimized μ -disk bottom coupled with a passive waveguide. An example of simulation performed using the MEEP is contained in Appendix B, together with the bash script used in order to perform multiple simulations by iteratively varying the involved parameters.

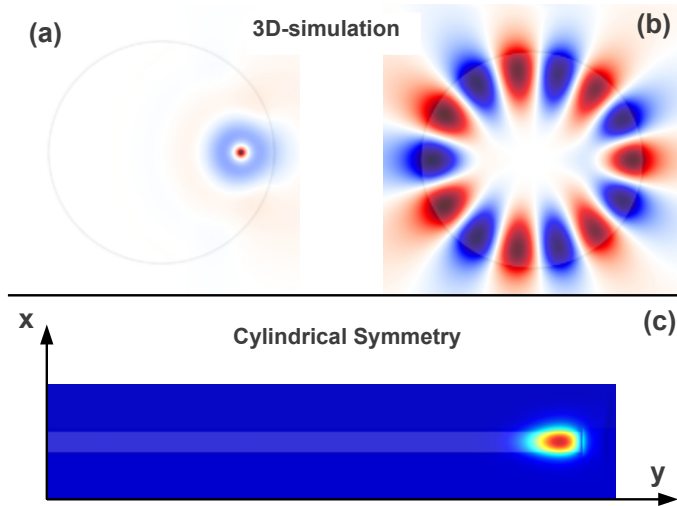


Figure 3.4: *Example of top view (plane $z=0$) of a 3D simulation of a μ -disk. In figure (a) we can see the excitation source expanding at the beginning of the simulation, while in figure (b) the modal structure is already developed. In the bottom panel (c) we can see the cross section of a 3D simulation of the same disk obtained exploiting the cylindrical symmetry of the system.*

As explained in section 3.1.2 the calculation window in which the photonic structure is defined has to be discretized in both the spacial and temporal domains. The correct choice of the spacial and temporal resolutions will depend on the precision of the results that we want to obtain.

We have both realized 2D and fully 3D simulations of the system under study in a polyhedral cell. Simulation exploiting the rotational symmetry of a rectangular cell have also been done: such method is useful in order to reduce the total calculation time and CPU usage in all the systems having cylindrical symmetry, and in particular it is really useful

when calculating the field distribution inside complex structure needing an high spatial resolution (like slot WG).

In those simulations we have treated the μ -resonator has a passive system containing a broad excitation source, placed in one side of the cavity (panel (a) in figure 3.4). In our case the experimental bulk emission spectrum of the material (previously obtained by standard PL measurements) has been simulated as the pulse of temporal duration corresponding to the spectral width of approximately 150 nm centred in its maximum wavelength. A detector exploiting the Harminv tool have been put inside the disk in order to obtain the informations about the modes (real and imaginary part of the frequency, Q factor and intensity). All those informations permit us to reconstruct the shape of the resonance spectrum, obtained as the sum of all the single resonance spectra in the wavelength axis. The resonances can not be represented as a single Dirac delta function but only as a Lorentzian curve, which dispersion is described by:

$$f(\lambda, \lambda_0, Q, I) = I \left[\frac{FWHM}{(\lambda - \lambda_0)^2 + FWHM^2} \right] \quad (3.13)$$

The parameters involved in the representation of the Lorentzian curve (FWHM, intensity and centre of the maximum) were taken from the results of the Harminv calculation.

The polarization of the excitation source has been chosen, depending on the particular simulation problem, between TE (parallel to the substrate surface) or TM (perpendicular to the substrate surface).

3.2.1 Geometrical variation

The shape of the WGM spectrum, the Q factor of the resonances and the number of the modal families supported by the cavity, depend strictly on two main geometrical parameters: the height and the radius of the cavity. In order to study such dependence we have realized simulations of disks and rings with radius between 2.5 and 10 μm and height values between 0.1 and 0.4 μm . The refractive index vales are that indicated in Figure 3.5 (in the simulation performed in this section the WG is not present). In the following we will explain why those particular values for R and h where chosen and what are their limitations.

3.2. MODAL STRUCTURE OF THE μ -RESONATORS

Looking at the sensoristic application of the device, it is very important to design the active cavities with low loss geometry. One of the most important parameters in order to estimate the potentiality of the sensor is in fact the *Detection Limit* (DL), defined as the minimum refractive index change that provides a shift equal to the FWHM (a more detailed definition will be provided in Section 7.2.1): it is therefore clear that to an increase of the Q corresponds a rise of the DL. We have indeed calculated that losses lower than 65 dB/cm at 780 nm are needed in order to obtain a Q factor of at least 10^4 , that is the necessary condition permitting to reach a DL value comparable with that of the literature ($DL = 10^3$).

The left graphic of Figure 3.6, in which the Q factor is represented in function of the wavelength and the radius for a constant value of $h = 0.3 \text{ }\mu\text{m}$: the more we increase R , the higher Q values. For R greater than $3 \text{ }\mu\text{m}$ the minimum condition is accomplished in almost all the excitation spectrum.

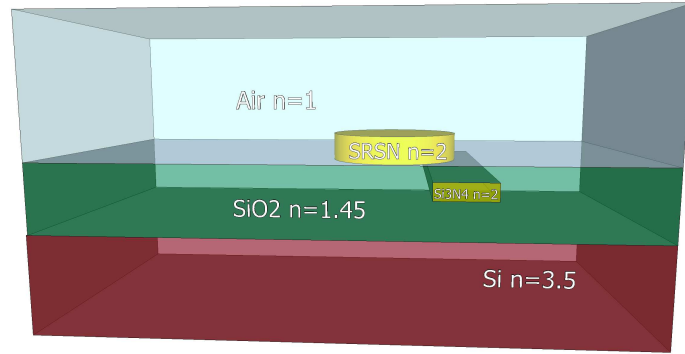


Figure 3.5: *Scheme of the cross section of the simulated structure. All the refractive index of each material used in the simulation are also indicated.*

Afterwards, taking in to account this simulation, during the design process of the photolithographic mask (described in Section 4.1), only disks with $R > 3 \text{ }\mu\text{m}$ will be considered. Another fact that we can appreciate in Figure 3.6 is the lowering of Q as the wavelength rise: this is principally due to the geometrical de-confinement of the mode due to the small height of the disk. In view of the application of the μ -cavities in the sensoristic field it is also worth to examine the behaviour of the *Free Spectral Range* (FSR, defined as the

3.2. MODAL STRUCTURE OF THE μ -RESONATORS

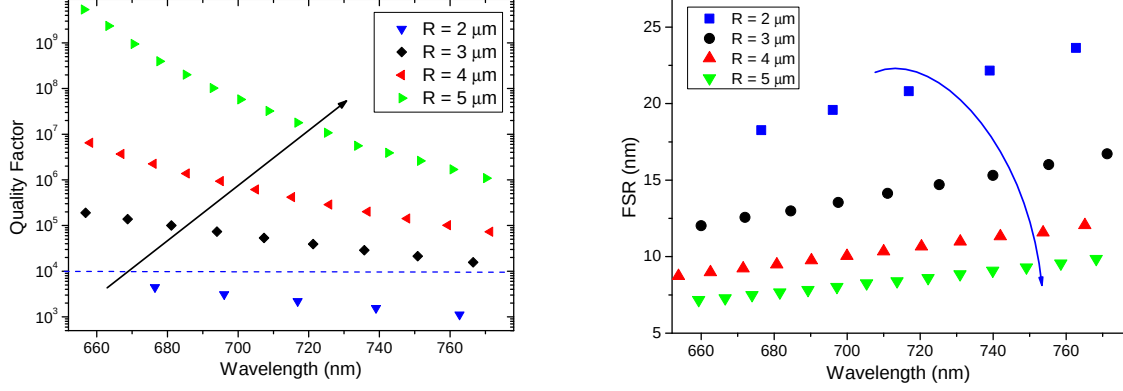


Figure 3.6: Variation of Q factor (left graphic) and the FSR (right graphic) as function of the radius R of the disk. The blue dashed line in the left graphic represent the established minimum value of Q . Such limit for the FSR depends on the practical application of the device.

spectral distance between two different resonances of a same modal family) as a function of R . It is clear that, if the sensor will work by measuring the changes of the position of the resonances due to a variation of n , a spectrum with high FSR is suitable, in order to separate two resonance having consecutive m . From the Equation 2.21 we can extract the inverse relation between FSR and R . In this case, in contrast with what stated for the Q factor, it is not possible to establish a limit value of FSR because it depends on the precise practical application of the cavity. For this reason, even though that high R values bring to high Q , it is not convenient to work with cavities with $R > 10 \mu\text{m}$, as it will generate a number undesired high order modal families.

The next step will be hence to study the dependence of the Q as a function of the height of the cavity. We keep the radius fixed to a value of $4 \mu\text{m}$ in order to have a Q factor superior than the previously given minimum limit of 10^4 obtained with $h = 0.3 \mu\text{m}$ and we variate h around that value, from 0.1 to $0.4 \mu\text{m}$. The results of those simulation are illustrated in figure 3.7, in which it is clear that only for values superior than $0.25 \mu\text{m}$ is possible to accomplish the Q_{limit} condition.

3.2. MODAL STRUCTURE OF THE μ -RESONATORS

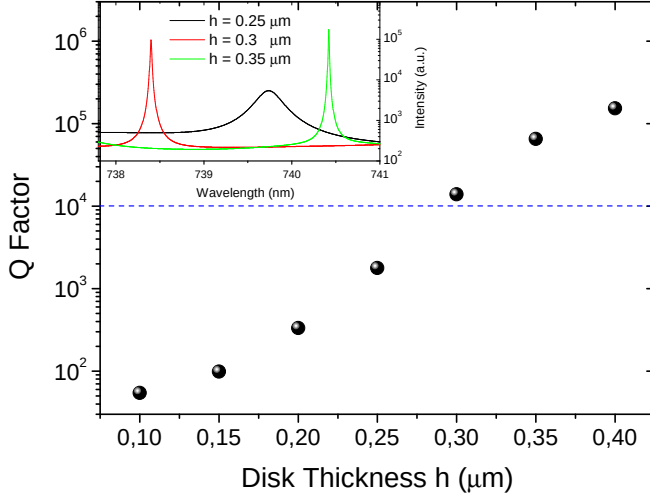


Figure 3.7: *Behaviour of the Q in a $3 \mu\text{m}$ radius disk as function of the thickness: only for disks with h superior than $0.3 \mu\text{m}$ the minimum condition given by the blue dashed line, is respected. In the inset we can appreciate the broadening of the resonances as the h goes down.*

At this stage of the discussion about the simulation process, it is worth to analyse the behaviour of the system when subjected to incident waves having different polarization. The simulation package give also the possibility to change such parameter both directly in the source or in the detector.

The shape of the simulated spectra for values of h between 0.1 and $0.4 \mu\text{m}$ are reported for both TE (that in the case of a circular resonator will be defined as TR) and TM polarizations in graphics 3.8 and 3.11. The numbers reported near each peak represent the value of the variable m of that peak, while the apex indicate the modal family (the number 1 is the fundamental).

The small peaks that we can see on the right of resonances 49^3 and 48^3 in graphic 3.8 (*g*) are due to a small coupling of the perpendicular polarization (in this case the TM), weakly revealed by the detector. Comparing the graphic 3.8 and 3.9 we can establish the order of the modal family of each resonance peak. We can easily note in figure 3.9 (*f*) that for h values bigger than $0.3 \mu\text{m}$ a quasi second order vertical mode appears. The design of the system will be realized in order to deal only with monomodal μ -cavities in the vertical direction of TE (so the notation of such modes will be $(x, 0)$).

3.2. MODAL STRUCTURE OF THE μ -RESONATORS

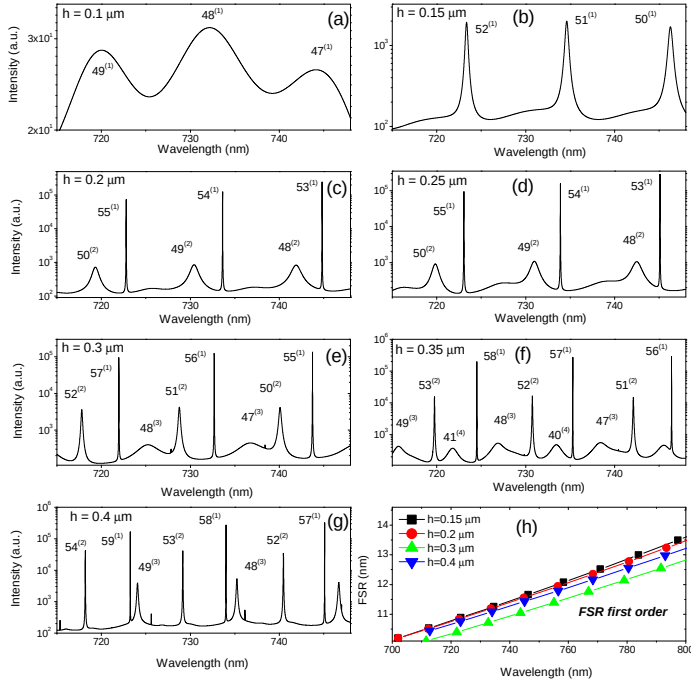


Figure 3.8: (a)-(g): TR polarized simulated spectra obtained for a μ -disk of radius $4 \mu\text{m}$ and variable thickness (from $0.1 \mu\text{m}$ to $0.4 \mu\text{m}$). The value of m parameter and the number of the modal family is also indicated (e.g. the notation $52^{(1)}$ represent a resonance peak with $m = 52$ and modal family = 1). (h) Graphic of the FSR as function of the wavelength for four different values of h .

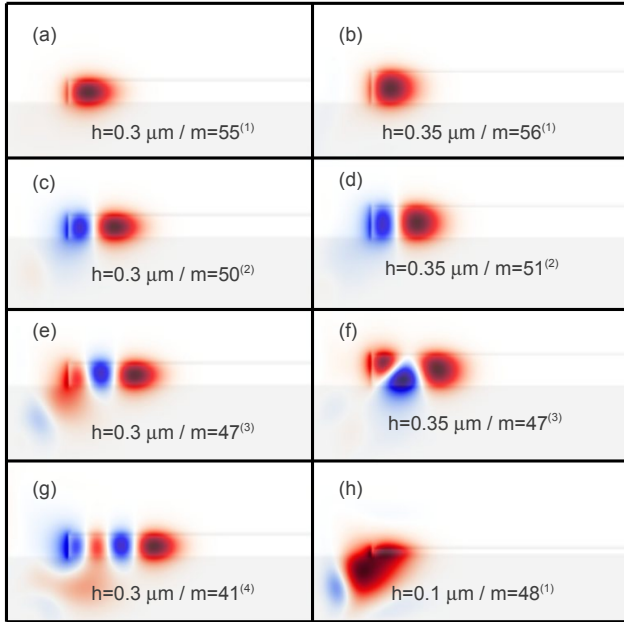


Figure 3.9: Distribution of the EM field of some modes supported by the cavity. (a) and (b) represent the fundamental mode. Figure (h) represent a high losses mode obtained for an h value of $0.1 \mu\text{m}$

From the comparison of the graphics in Figure 3.10, in which the the Q factor of a $4 \mu\text{m}$ resonator for both TR and TM polarizations are shown in function of different thickness

3.2. MODAL STRUCTURE OF THE μ -RESONATORS

values: for all the reported h values the TR polarization present higher Q factor respect to the TM ones. Nevertheless, the previously defined Q limit condition is accomplished also for the TM, so in principle for our sensoristic application both polarizations can be exploited.

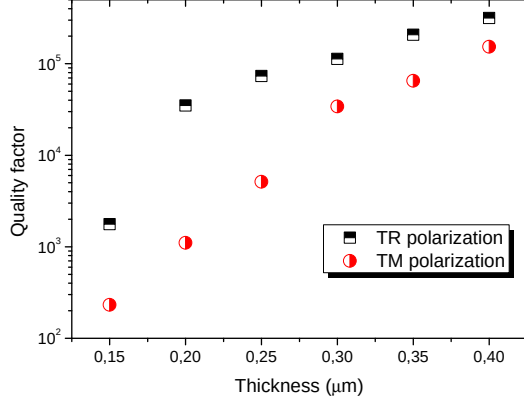


Figure 3.10: Comparison between the Q factor of TR and TM polarizations of a $4\ \mu\text{m}$ radius and $0.3\ \mu\text{m}$ thickness disk.

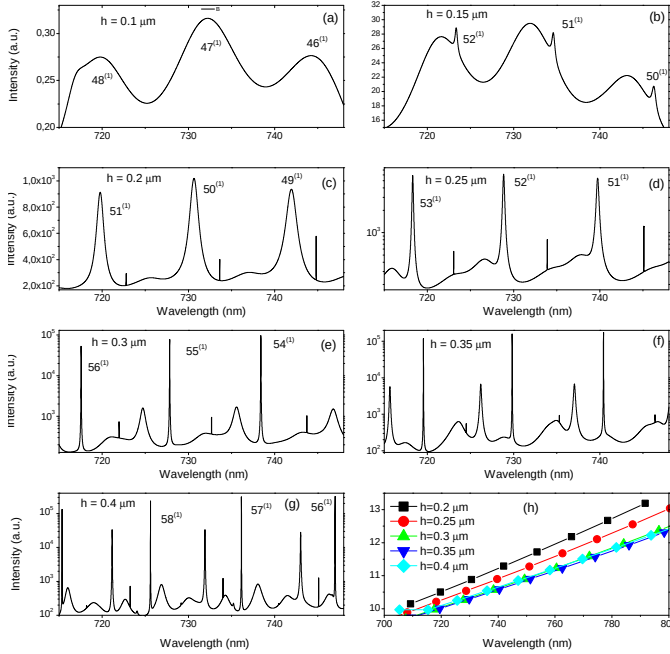


Figure 3.11: Variation of the WGM spectrum (panels (a)-(g)) and FSR (panel (h)) for the TM polarization inside a disk of $R=4\ \mu\text{m}$ and $n=2$ as function of the variation of the height.

In synthesis in this section we have chosen the geometrical parameters that will be used in the design of the optical structures: $R = 3$ to $10\ \mu\text{m}$ and $h = 0.25$ to $0.35\ \mu\text{m}$.

3.2.2 45° cut edge Vs Vertical edge

A further parameter regarding the cavity geometry that has been evaluated is the angle of its edge. During the fabrication process there is indeed the possibility to vary the etching process to create cavities with 90° vertical or 45° edges. The advantage of the last mentioned configuration is that the mode is developed more inside in the resonator, decreasing the losses due to scattering with the superficial imperfection (see Figure 3.12). Unfortunately the utilized tool does not include the possibility of simulating a superficial roughness, so the consideration regarding the etching type can be made only from a qualitative point of view, by checking if there is some remarkable variation between the two kind of etching due to a geometrical shape of the edge, lowering the Q factor under the Q_{limit} condition.

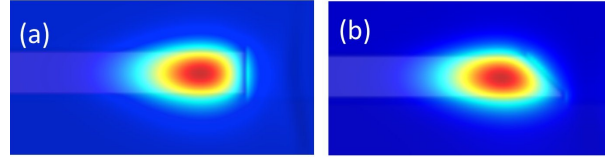


Figure 3.12: Comparison between the field distribution of the first order modal family inside a disk presenting a vertical edge (a) and 45° edge (b).

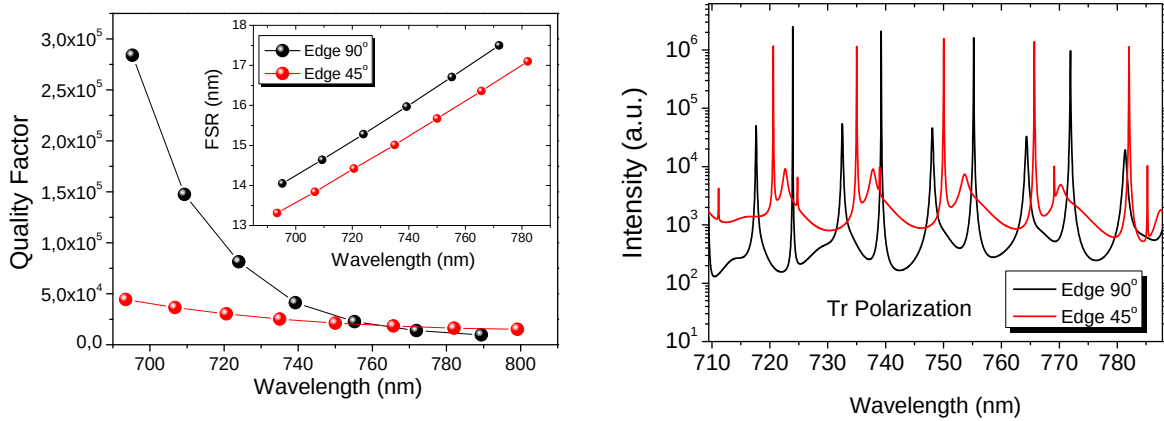


Figure 3.13: Left graphic: Q factor variation as a function of the edge shape of the disk, showing a relevant difference at small wavelength. In the inset the FSR is also shown. Right graphic: TR polarized WGM spectral shape for both μ disks.

Figure 3.13 (a) shows the results of the simulation obtained for a $4\ \mu\text{m}$ radius disk: the optical resonators with 90° angle edge (vertical etching) have superior Q factor for small wavelengths values respect to the 45° angle disks. Nevertheless, as we will see afterwards, in this spectral region the Q is governed by the material loss: for this reason and since the simulated Q is always above the Q_{limit} , it might be interesting to produce both kind of structures to estimate the eventual losses due to the superficial scattering.

3.2.3 Refractive index variation

All the previous simulations have been performed using disks standing on a SiO_2 substrate and surrounded by air. In this section we will study the changes in WGM resonance spectrum if we vary the refractive index of the surrounding material (n_{surr}). In particular, the physical quantity that we want to observe is the Sensitivity of the disk, given by the formula:

$$S = \frac{\Delta\lambda}{\Delta n_{surr}} \quad \text{SENSITIVITY} \quad (3.14)$$

in which S is defined as the wavelength displacement of a resonance ($\Delta\lambda$) as a result of a change of one Refractive Index Unit (RIU) Δn_{surr} .

Simulations have been performed by varying the third decimal number of the n_{surr} around a $7.5\ \mu\text{m}$ radius cavity. More precisely we want to simulate the n that we will use in the practical proof of concept (reported in Chapter 7). The red shift showed in panel *a* of Figure 3.14 is a great result in terms of Sensitivity of the device, comparable with that reported in the literature ($10^2\ \text{nm/RIU}$) for similar structures measuring the refractive index variation. [23]

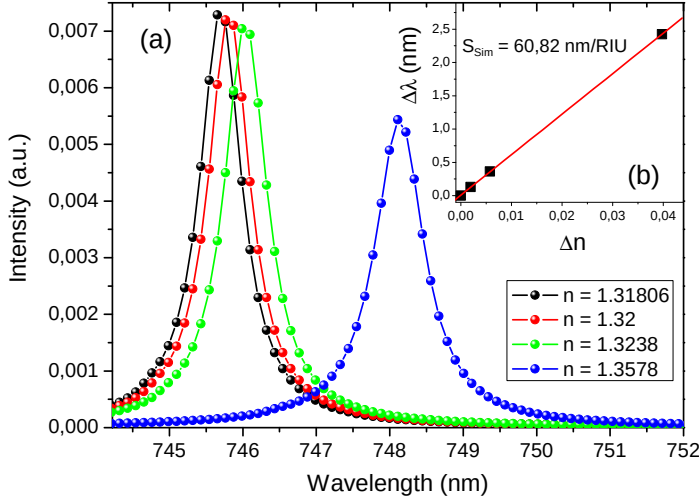


Figure 3.14: *Left panel: variation of the WGM position of a $7.5 \mu\text{m}$ radius disk due to a change of refractive index value. Right panel: linear slope of the maximum of one resonance reported in the Left panel, resulting in a Sensitivity of 60.82 nm/RIU*

3.3 Coupled Structure

The last step of the simulation process is to join all the parameters optimized in the previous sections for the isolated structures, and implement a simulation of the coupled structure. As stated before the system is given by a μ -disk or μ -ring vertically coupled with a WG . The WG is therefore created in the oxide layer below the disk as shown in figure 3.15. In this case the presence of the WG under the disk breaks the cylindrical symmetry, so the simulation will be performed in a fully 3D calculation window.



Figure 3.15: *Scheme of the cross section of the coupled structure. The Z-Gap and X-Gap are also graphically defined.*

The two principal parameters involved in this simulation, apart from that previously described for the isolated structures, are the distance between the bottom surface of the

resonator and the top surface of the *WG* (defined as *Z-Gap*), and the distance between the center of the *WG* and the edge of the resonator (*X-Gap*). By changing those two parameters it is possible to tune the coupling of the radiation generated inside the cavity into the *WG*.

As the presence of the *WG* induce a further source of losses on the cavity, the *Q* factor can be also modulated by varying the *Z-Gap*.

Our objective is therefore to find a compromise among *Q* and transmitted intensity. Due to the fact that the light is directly generated inside the resonator, the critical coupling condition is not mandatory, as it is for the passive μ -resonators, externally coupled with a broad band light source or a tunable laser.

As we will see in next sections a good set of coupling parameters (*X-gap*, *Z-gap*) will bring to acceptable *Q* factor (over the 10^4 limit) and optimized intensity at the end of the bus *WG*.

3.3.1 Z-Gap

As previously remarked, the *Q* factor of the μ -cavity has a direct and strong dependence on the gap thickness. The intuitive explanation to the observed behaviour is that, as the presence of a waveguide under the μ -disk implies an additional source of losses due to evanescent coupling, there is a consequent reduction of the *WGM* *Q* factor according with equation 2.30.

3D FDTD simulation of the coupled structure have been hence executed with the aim of studying such dependence and finding the best gap separation value between the top surface of the waveguide and the bottom of the μ -disk.

In the following we will show simulated TM polarized spectra of a $3\ \mu m$ radius and $0.3\ \mu m$ height μ -disk, keeping the *X-gap* constant to the zero position in order to deal only with the fundamental *WGM* family and have clearer comprehension of the behaviour of the system. The two principal parameters that have been taken into account for optimizing the coupling conditions are the *Q* factor of the optical system (μ -resonator + *WG*) and the intensity of the resonances measured at the end of the bus *WG*. For this reason, in the simulation, two virtual intensity detectors have been placed in different positions of

3.3. COUPLED STRUCTURE

the system: one inside the disk on the opposite side respect to the WG position, used to evaluate the intensity of the resonances inside the μ -resonator and the other at the end of the WG .

This last detector helps us to obtain the value of the WGM intensity after the coupling. For completeness, we have to remark that the simulations take in account only the radiative losses of the structures and not the losses due to the material itself.

The results of such simulations demonstrate that the smaller is the gap between the waveguide and the disk, the lower is the Q of the coupled structure. This result is well illustrated in the left panel of Figure 3.16, where the spectral behaviour of the Q factor, measured inside the disk for different vertical gaps (Z -Gap) is shown.

According to what stated before, we can appreciate the decrease of Q value with the decreasing of the Z -Gap, which is inversely related to an increase of the overall cavity losses originated by the evanescent coupling with the waveguide.

The more the WG is far from the cavity, the more the resonator behaves like an isolated cavity: in our case this condition is accomplished with $Z - Gap = 0.4 \mu m$. It is afterwards clear that gap values below $0.2 \mu m$ would not be of practical use, because the low Q - lower than 10^4 - will penalize the sensing DL.

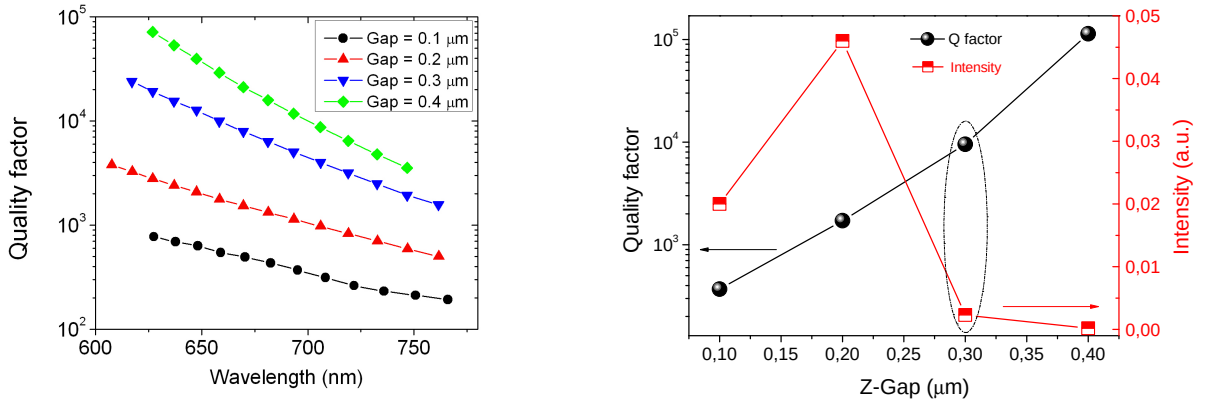


Figure 3.16: *Left panel: Simulated behaviour of Q in a coupled structure as a function of the Z -Gap distance. Right panel: comparison between the Q and the intensity (measured inside the WG) of a resonance for different Z -Gap values.*

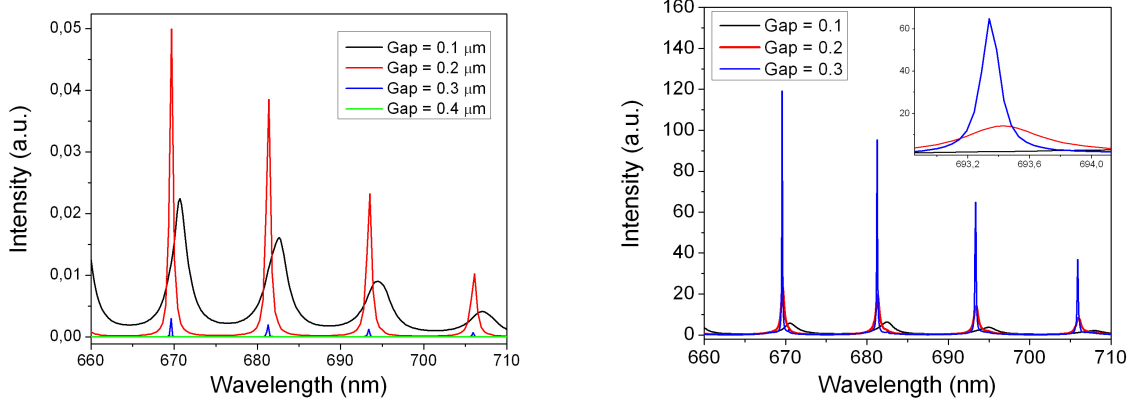


Figure 3.17: *Left graphic: simulated WGM spectra of a 3 μm radius μ-disks, for different vertical gap values. The signal is provided by the detector placed on the waveguide. Right graphic: simulated WGM spectra measured inside the μ-resonator. The inset graphic shows a zoom of one particular resonance, putting in evidence the variation of Q factor as a function of the gap distance.*

On the other hand, the growth of the Q obtained moving away the WG from the resonator is accompanied by a drastic reduction of the transmitted intensity, measured in a detector placed inside the WG. In the right panel of Figure 3.16 we see in fact that for a Q value exceeding the previously defined limit of 10^4 the intensity decrease of more than an order of magnitude (from 0.45 a.u. for a Z-Gap of 2.5 μm to 0.02 a.u. for a Z-Gap=0.3 μm). From the cross comparison between the intensity of the spectra obtained by the two detectors and the overall quality factors (Figure 3.17 *left* and *right*), it is possible to understand that, while the WGM intensity within the disk increases with the vertical gap distance, the highest intensity values at the end of the WG are found at distances between 0.2 and 0.3 μm. A gap value greater than 0.3 μm will correspond to an under-coupling regime (overall Q above half of the Q of the isolated disk) while below 0.2 μm the regime is that of over-coupling and the presence of the waveguide starts to visibly modify even the real part of the effective refractive index, since the WGM spectral position shifts.

In summary, the intersection of the information obtained by the simulation suggested an optimum coupling distance between 0.2 and 0.3 μm. Therefore, as we will see in the chapter 4, samples with different gaps within this range were realized (from 205 to 310 nm) by varying the oxide thickness between WG and μ-resonator.

3.3.2 X-Gap

The last parameter involved in the design of the coupled structure is the *X-Gap*. In the simulations, we have defined it as the relative displacement between the centre of a $1\ \mu m$ WG and the most external point of the circumference of the μ -disk, keeping the *Z-Gap* constant to $0.25\ \mu m$, as established in the previous section. The *X-Gap* origin value corresponds to the situation in which the WG centre is aligned with the vertical projection of the disk lateral edge. The minus (plus) sign corresponds to a situation in which the centre of the WG is moved towards (outwards) the μ -disk centre.

Figure 3.18 shows that, by changing the X-gap, it is possible to tune the intensity of the WGM detected at the end of the bus WG.

The first important result that we can extract from this simulation is that the higher intensity of the resonances is obtained for a value of $X - Gap = 0\ \mu m$, corresponding to the position in which the WG lies exactly under the volume of the WGM. For positive values of *X-Gap* higher than $0.2\ \mu m$ no EM field is detected at the end of the bus WG.

The comprehension of this behaviour plays a fundamental role, as we will see in chapter 4, during the horizontal alignment of the photolithographic masks: such process is in fact subjected to an alignment mismatch of about $1\ \mu m$.

Another interesting result concerns the appearance of the second order WGM family for X-Gap values below $-0.1\ \mu m$. Figure 3.18 shows how it is clear that negative *X-Gap* values contribute to the lowering of the first order WGM family.

This result is very interesting in view of the application in sensoristic field, thought that the problem of working with high Q factor μ -cavity (corresponding to high R values) was the apparition of undesired WGM families of superior order: during the sensor measurement such modal families might superimpose to the first order family (normally used as reference in the detection), making difficult the estimation of the n_{eff} index variations. This problem can be overcome using WG narrower than the EM profile of the WGM and playing with the *X-Gap* parameter, selecting in fact one particular modal family and filtering the others.

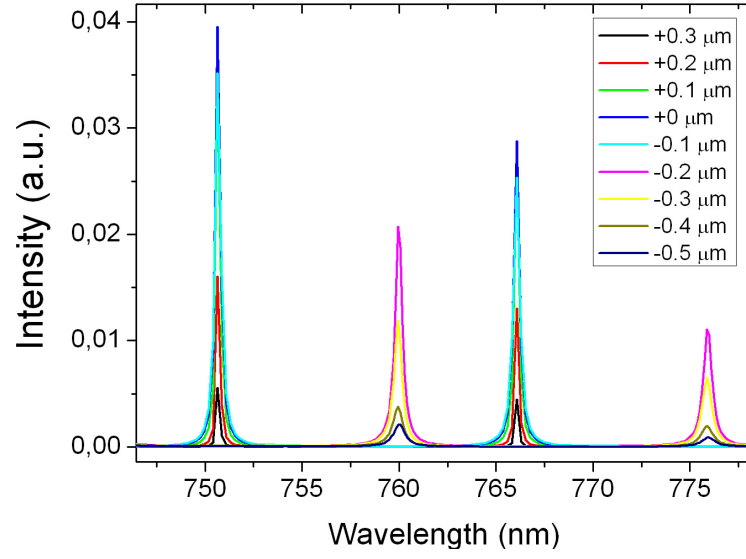


Figure 3.18: *Simulated spectra obtained by varying the lateral X-Gap from an external position outside the disk, to an internal position.*

Chapter 4

Fabrication Process

The main objective that we have tried to achieve in this thesis is to realize a micrometric device behaving as a sensor using a fully compatible CMOS technology. For this reason, all the samples have been produced using standard silicon microfabrication technology. In this chapter we will illustrate the fabrication process of the micro structures: the geometrical parameters were previously simulated, as reported in Chapter 3, and their realization requires the design of a adequate photolithographic mask. All the samples were produced in the *Institut de microelectrònica de Barcelona* (IMB-CNM-CSIC) of Bellaterra (Barcelona) under the supervision of Dr. J. Monserrat and Dr. C. Dominguez. Moreover we will describe in detail the parameters used in the epitaxial growth of the active material performed by means of *Low-Pressure Chemical Vapor Deposition* (LPCVD) technique and the implantation process of the Si.

A detailed characterization of the produced samples have finally been performed by means of AFM, XPS and EFTEM in order to assess the quality of the samples.

4.1 Photolithographic mask

Keeping in mind the values of the optimized parameters arising from the simulations we have designed two levels of photolithographic mask. By combining the two levels and the implantation process it is possible to define isolated active and passive optical elements

or vertically coupled structures (waveguides, rings, disks).

The pattern of the mask is composed by four separated zones, schematically represented in Figure 4.1, designed in order to ease the cut of the sample and consequently the measurement. Since the pattern is repeated only one time on the wafer, and there is not a small dice reported many times, it is very important to have the possibility to separate one zones without damaging the others.

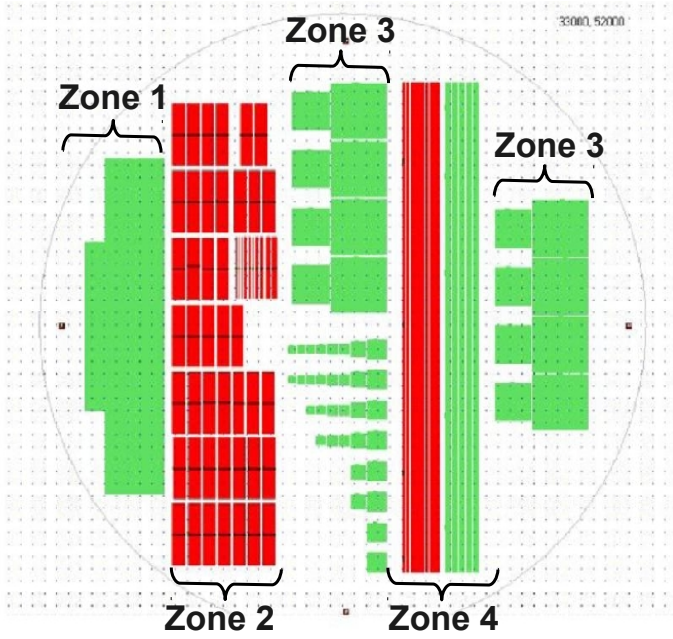


Figure 4.1: *Layout of the two level of photolithographic mask superimposed. The red parts correspond to the passive layer, while the green part represent the active material.*

The first zone (labeled as *Zone 1* in Figure 4.1) contains only the bulk material, with the facets size in the order of tenth of mm , without any optical structure. In this lateral part of the sample it is possible to study the properties of the material (e.g. photoluminescence intensity and the optical loss in planar waveguide using the SES technique), that will be described in the following chapters.

Adjacent to the bulk material, the *Zone 2* showed in Figure 4.1, we can find many combination of active resonators with variable radius and passive waveguide with different size, coupled each other with different values of relative positioning. In this zone it is possible to execute passive measurements, coupling an external light source from the WG to the disk, or active measurements directly pumping the resonators and collecting the light out-coming from the WG. The coupled WG have a length of 1 cm .

The central and right zones (*Zone 3*) of the wafer are composed by squared fields of μ -disks with variable radius from $2.5\mu\text{m}$ to $10\mu\text{m}$ and μ -rings with radius between $2.5\mu\text{m}$ and $30\mu\text{m}$. These zones were conceived to study the properties of one or more isolated μ -cavities by focusing or defocusing the pumping spot on the sample.

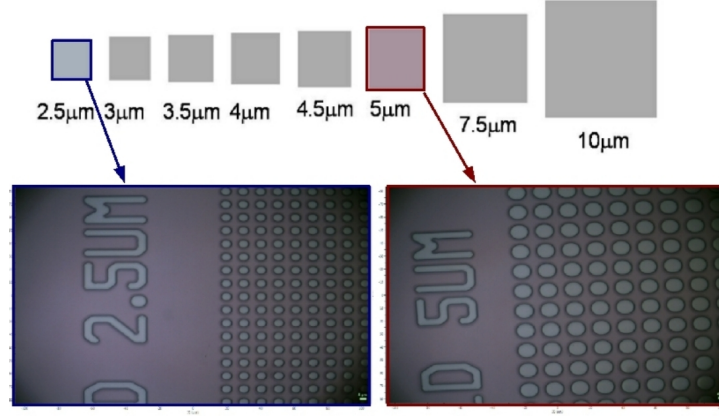


Figure 4.2: Zoom of one series of disk fields shown in figure 4.1 and detail taken with an optical microscope of the $2.5\mu\text{m}$ and $5\mu\text{m}$ field.

Finally the *Zone 3* is composed by groups of active and passive WG with different size (from $1\mu\text{m}$ to $6\mu\text{m}$): this size is very important to evaluate the optical loss of the material. The pattern defined by the photolithographic process consist in 10 groups of waveguides, and each group contain 8 waveguides of different widths ($10, 6, 5, 4, 3, 2, 1.5, 1\mu\text{m}$).

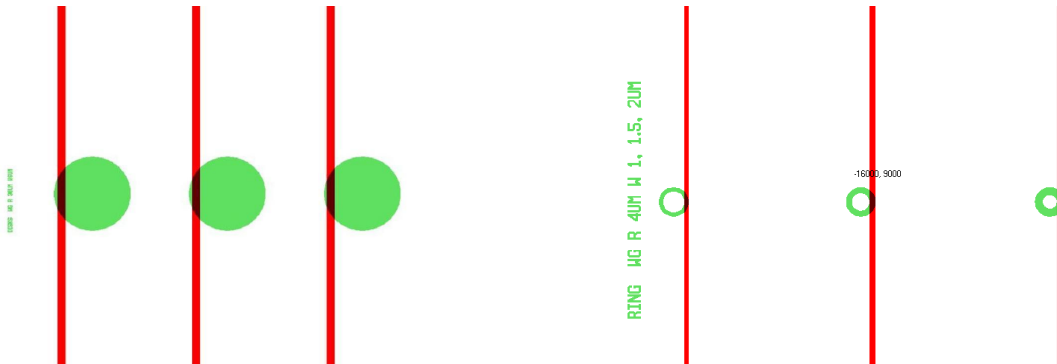


Figure 4.3: Particular of the *Zone 2*, containing active disks and rings coupled with passive waveguides.

4.2 Deposition process and parameters

The samples under analysis have been produced using standard complementary metal-oxide-semiconductor (CMOS) compatible processes. The wafer on which the structures were defined are a PPA type (p-type Si doped with Boron), previously cleaned with a $H_2SO_4 + H_2O_2$ treatment (*Piranha Etch*).

Initially, 2 μm of SiO_2 was thermally grown (1100°C, wet ambient) on top of crystalline Si wafers, becoming the optical cladding of the active structures. After another $H_2SO_4 + H_2O_2$ treatment layer of stoichiometric Si_3N_4 (of variable thickness between 250-350 nm) was subsequently deposited by means of low-pressure chemical vapour deposition technique (LPCVD) at 800°C. In the case of the active material the previous layer afterwards suffered a Si ion implantation followed by an annealing in N_2 atmosphere at variable temperature between 900°C and 1100°C.

As stated in Chapter 3 the thickness of the active material allows obtaining monomodal behaviours for the TR polarization (parallel to the disk surface). Moreover the selected values for the active layer thickness should be low enough to prevent stress induced issues. The implantation consisted of a 150 keV energy process (*I1*) followed by a second one at 90 keV with a lower dose (*I2*). A typical implantation profile is shown in the left panel of Figure 4.4 . The ion energies and doses of the double implantation - previously simulated by means of *TRIM* software package [69]-[70] - were chosen to achieve a flat Si excess profile and to optimize the overlap with the vertical distribution of the fundamental optical mode. The ideal situation would be in fact that of having the implantation profile maximum as close as possible to the maximum of the vertical energy distribution for the fundamental mode, in order to couple as much light as possible to the propagating mode. Nevertheless, the limited available energy for the implanter prevents achieving this condition and the Si-excess distribution is centred slightly over the gravity centre of the fundamental mode at 750 nm. We have produced a set of samples where the implantation doses were varied, covering a range of Si excesses from 11.8% (sample 1 of run 5107) to 2.4% (sample 5 of run 5506). (Right panel of Figure 4.4)

Finally, the photonic structures have been defined by means of standard photolithographic techniques and Reactive Ion Etching (RIE).

4.2. DEPOSITION PROCESS AND PARAMETERS

In the case of the coupled structures, a passive stoichiometric Si_3N_4 rib WG (150 nm thick) has been also defined by standard photolithographic processes within the SiO_2 cladding, with different values of X-Gap, as reported in Section 3.3.1.

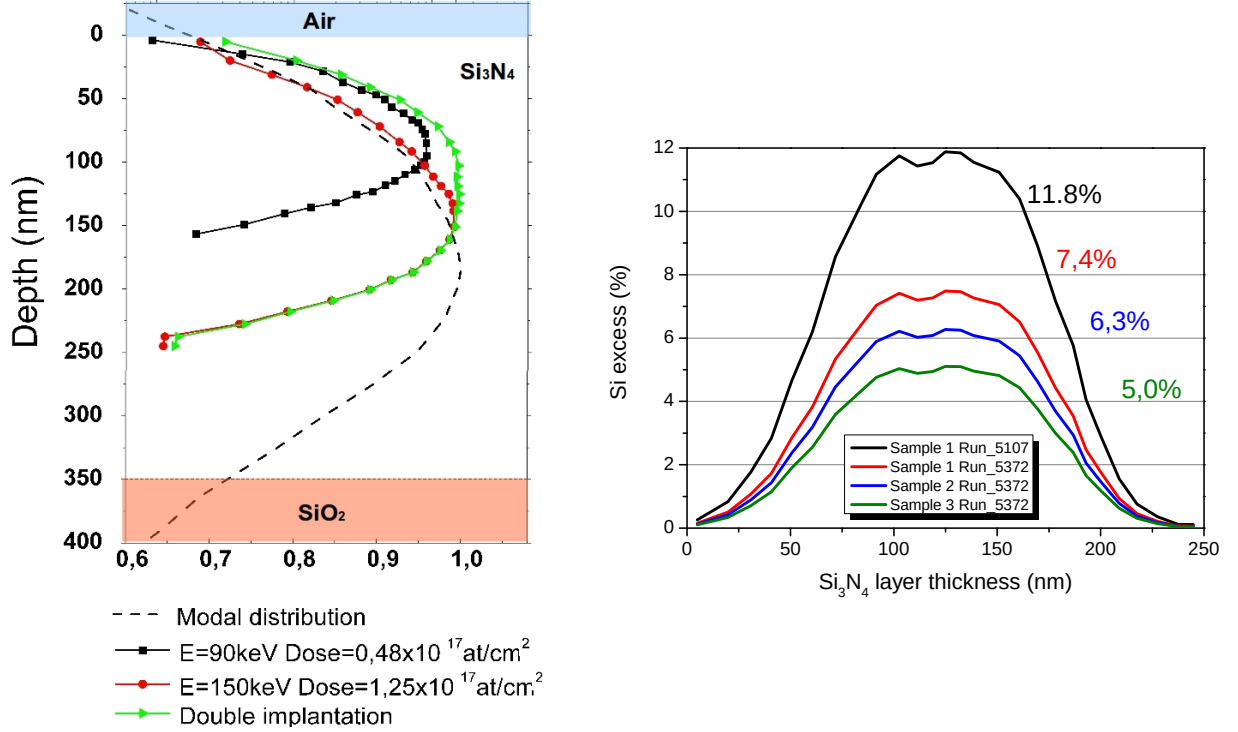


Figure 4.4: *Left panel: Profile of the double implantation compared with the energy distribution of the fundamental WGM of a micro-cavity. It is easy to notice that the overlap is not perfect due to the technical limitations of the implanter. Right panel: Percentage of Si excess for different implantation doses samples.*

In order to perform the alignment process a *contact/proximity* type mask aligner is used. Each level of mask contains some alignment patterns placed on the left and on the right side of the mask. In our case the alignment pattern is represented on the first mask by a squared marker and by a cross-shaped marker on the second mask. The alignment is obtained by overlapping the two markers: when the four sides of the square matches with the sides of the cross the alignment is completed. In such process, the precision of the alignment depends mostly on the ability of the technician, and can be as high as 1 μm .

4.2. DEPOSITION PROCESS AND PARAMETERS

In Tables 4.1, 4.2, 4.3, 4.4 we summarize the active material parameters of the samples produced during my PhD. Each run of samples have been produced in order to optimize a particular set of parameter, so we can associate to them a precise function:

- Run 5094_UB: characterization of the passive material in terms of optical losses due to geometrical variation, annealing process and etching type. The superficial analysis was also performed on these samples. (Table 4.1)
- Run 5107_UB: study of the variation of the optical properties of the structures under different implantation dose. Some single implantation sample were also produced. Furthermore two samples (the 7 and 8) without implantation, but with high thickness were produced in order to evaluate possible stress effect or deformation of the structure (Table 4.2)
- Run 5372_UB: optimization of some parameters (baking temperature, dose and energy of the implantation) with the goal of finding a compromise between material losses and PL intensity. (Table 4.3)
- Run 5506_UB: study the vertical coupling of the optical structures. The passive Si_3N_4 layer has a thickness of 153 nm while the active *Si-rich-Si₃N₄* has thickness of 304 nm (Table 4.4)

4.2. DEPOSITION PROCESS AND PARAMETERS

Table 4.1: *Passive samples produced in order to study the effect of the annealing treatment on the propagation losses.*

Sample 5094_UB	Si_3N_4 Thickness (nm)	Protection SiO_2 layer (nm)	Si_3N_4 Etching	Annealing treatment for 4h	Annealing Before / After lithography (B/A)
1	150.3	200.5	Total	Not annealed	/
2	150.3	200.5	Total	Not annealed	/
3	150.3	200.5	75 nm	Not annealed	/
4	150.3	180	Total	1050°C	A
5	150.3	180	Total	1100°C	A
6	150.3	180	Total	1150°C	A

Table 4.2: *Characteristics of the samples belonging to Run 5107_UB.*

Sample 5107_UB	Si_3N_4 Thickness (nm)	Implantation energy (keV)	Implantation dose ($\times 10^{17} \frac{at}{cm^2}$)	Annealing treatment for 4h	Annealing Before / After lithography (B/A)
1	300	150/90	1.25/0.48	1100°C	B
2	350	150/90	1.25/0.48	1100°C	B
3	250	150/90	1.25/0.48	1100°C	B
4	300	150/90	0.62/0.24	1100°C	B
5	300	150	1.35	1100°C	A
6	300	90	0.8	1100°C	A
7	500			1100°C	A
8	500			1100°C	B

4.2. DEPOSITION PROCESS AND PARAMETERS

Table 4.3: *Deposition parameters of samples 1-6 belonging to Run 5372_UB.*

Sample 5372_UB	Si_3N_4 Thickness (nm)	Implantation energy (keV)	Implantation dose ($\times 10^{17} \frac{at}{cm^2}$)	Annealing treatment for 4h	Annealing Before / After lithography (B/A)
1	300	150/90	0.75/0.29	1100°C	B
2	300	150/90	0.62/0.24	1100°C	B
3	300	150/90	0.50/0.19	1100°C	B
4	300	150/90	0.75/0.29	950°C	B
5	300	150/90	0.62/0.24	950°C	B
6	300	150/90	0.50/0.19	950°C	B

Table 4.4: *Description of the samples of Run 5506_UB, containing the final coupled structures.*

Sample 5506_UB	Si_3N_4 Thickness (nm)	SiO_2 Thickness (nm)	Implantation energy (keV)	Implantation dose ($\times 10^{17} \frac{at}{cm^2}$)	Annealing treatment for 4h
1	304	205	150/90	0.5/0.19	950°C
2	304	250	150/90	0.5/0.19	950°C
3	304	310	150/90	0.5/0.19	950°C
4	304	355	150/90	0.5/0.19	950°C
5	304	310	150/90	0.25/0.1	950°C
6	304	355	150/90	0.25/0.1	950°C
7	304	205	150/90	0.5/0.19	950°C
8	304	310	150/90	0.25/0.1	950°C

4.3 Analysis of the surface

After the growth process a large series of analysis on the surface of the samples have been performed, in order to consider the actual quality of the produced optical structures. It is thus fundamental to certify the perfect geometrical shape in order to avoid any geometrical imperfection, otherwise affecting the optical properties and consequently the good functioning of the devices.

4.3.1 Atomic Force Microscopy

To ensure that no morphological factor will cause optical losses changing we performed some Atomic Force Microscopy analysis on the profile of eight active waveguides belonging to the first implanted run (the *5107-UB*). In particular we have characterised *sample 1*. The surface analysis with AFM has revealed a very good top surface without inhomogeneities or irregularities. The roughness results to be on average lower than 1 *nm*. Taking in account this result we can exclude the processing quality as the main source of the losses.

In figure 4.5 we show an AFM image of one of the micro-disk structures designed, where it is evident the high quality of the disk surface and shape.

We believe that this measurement, as we will discuss later, allows us to assume that surface scattering/surface absorption losses are not the limiting factor determining the quality factors values of our cavities.

In figure 4.6 we show that, for the case of the studied ring resonator arrays the situation is significantly different, since the top surfaces present a valley-like distortion of several tens of nanometers. This will affect significantly both the consistency of the solution of the propagating mode as well as increase the losses due to the interaction of the mode with this distorted and rough surface: this effect, maybe due to diffraction phenomena during the photolithographic process, bring as consequence a dramatic reduction by orders of magnitude of the Q factors with respect to the expected ones for ideal simulated structures.

According with this result, in the following our attention will be focused mainly on the

μ -disks.

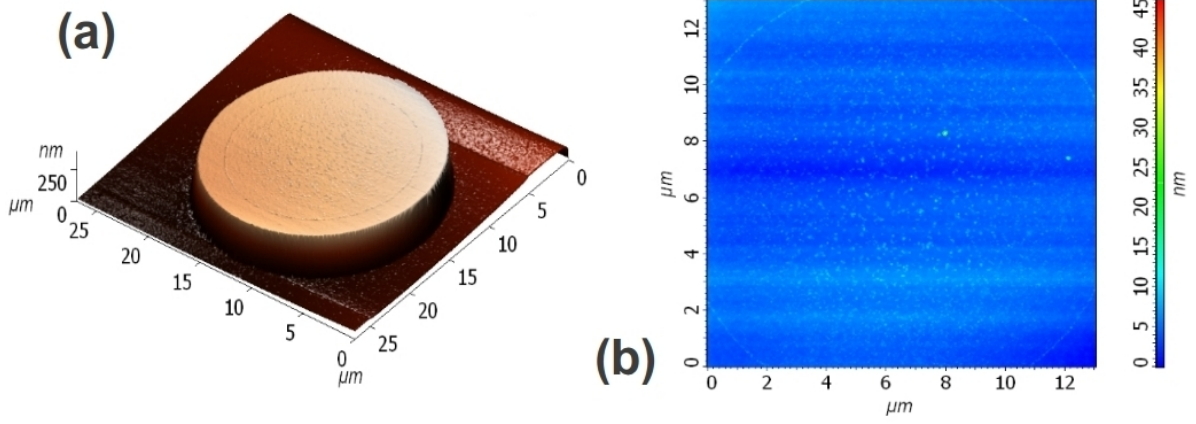


Figure 4.5: 3D image of the morphological status of a $7.5 \mu\text{m}$ radius disk (a) and analysis of the roughness of the top surface of the disk (b).

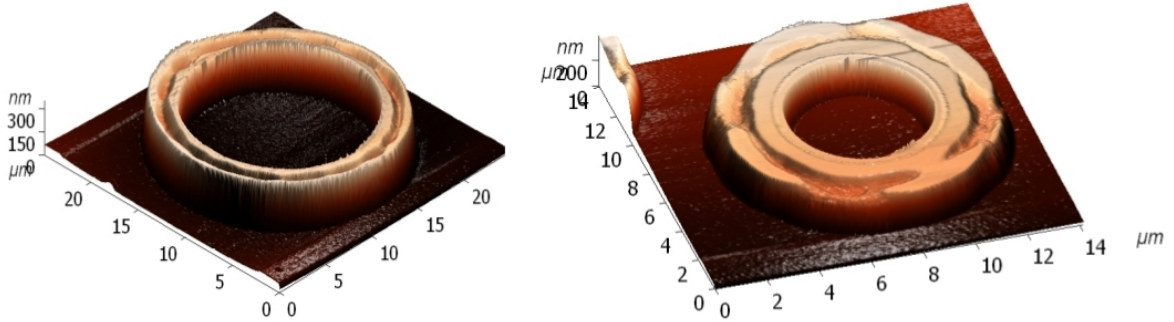


Figure 4.6: AFM image of the defects affecting the fabrication of the μ -rings.

Such problem does not affect the realization of the WG structures that, as we can see in Figure 4.7 do not present any sign of breaking or imperfection.

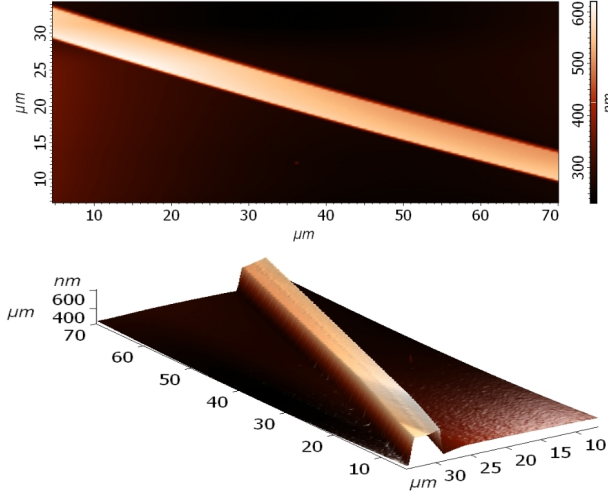


Figure 4.7: *Top image (a) and 3D reconstruction (b) of the AFM analysis on a WG of sample 1 of run 5107-UB. No evident imperfections can be seen.*

4.3.2 Scanning Electron Microscopy

The SEM analysis of the μ -cavities have revealed some issues, that we have to take in account during the optical characterization of the samples.

The first one concerns the slight discrepancy, represented in Figure 4.8, between the dimensions designed on the photo-mask and those of the fabricated structures: the proximity method used for illuminating the photo-resist through the mask entails diffraction effects enlarging the features of the devices, since it is not placed in contact with the resist. This will increase the FSR of the supported modes at the wavelengths of interest. Another evident effect can be noticed in Figure 4.8 (a): some circular structures present a polygonal shape, which is more evident for smaller cavities. Polygonal features are already present on the photo-mask, which is associated to a resolution limitation on the mask fabrication step. We think that this issue may increase the overall losses of the system, probably becoming the limiting source of loss (overcoming material losses) for the case of small cavities.

Furthermore the lateral walls seems not to be perfectly vertical (Figure 4.9 (b) and 4.10). In fact, the wall angle of both resonators and WGs is close to 45° . This is associated to the nature of the plasma (SF_6) used in the reactive ion dry etching procedure for etching the Si_3N_4 . This procedure is very selective with respect to SiO_2 and leaves a smooth

lateral surface.

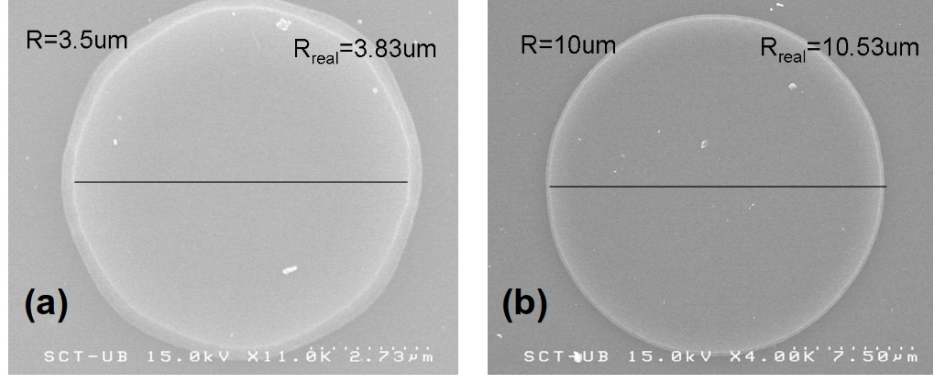


Figure 4.8: *SEM images showing the real dimensions of two circular resonators: panel (a) and (b) demonstrate a discrepancy of about $0.3 - 0.5 \mu\text{m}$ between the real length of the radius and that previewed by the mask.*

On the contrary it provides a quite low anisotropy. It was expected that (see Section 3.2.2), in the case of the disks, the supported modes would be squeezed towards the inner part of the structure, reducing the *FSR* and probably the surface scattering losses contribution .

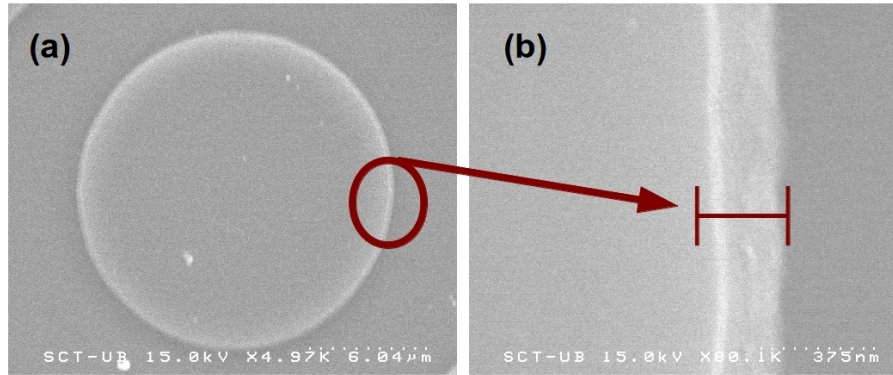


Figure 4.9: *SEM image (a) and particular zoomed area (b) showing the 45° angle of the perimetrical walls.*

The SEM images provide also information about the edge characteristics after the samples

are cleaved. This is quite important, as we will discuss in Section 5.2, for the characterization made in the μ -PL setup, because the easiest structures to measure are the ones close to the edge, whose emitted light arrives to the collecting objective without screening from other structures.

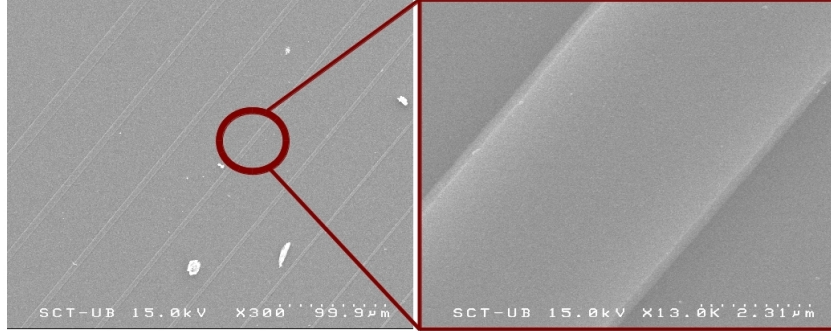


Figure 4.10: *Image of a WG field (left panel) and particular of the WG side walls (right panel)*

In that setup, we have experimentally observed that some of the cavities that we specifically pump are providing a PL spectrum without the WGM features, while other clearly show a PL modulated by the WGM.

Figure 4.11 clarifies this issue, revealing that the sample cut can break the structures, thus breaking their optical cavity nature. The ideal situation would be that where the cut passes between two cavity columns, as in the right image.

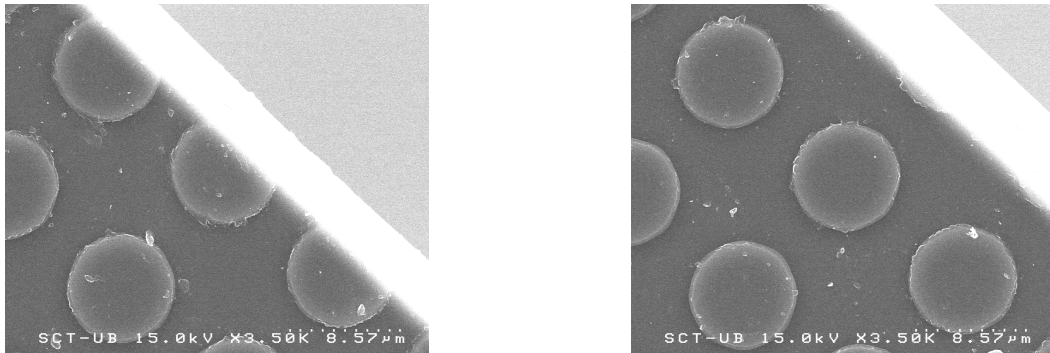


Figure 4.11: *SEM images of some μ -disks lying on the edge of the sample: on the left picture the cavities are broken by the cut of the sample, so is not possible the WGM detection. On the contrary, in the right image the disks are results to be intact.*

4.4 XPS

X-ray Photoelectron Spectroscopy (XPS) measurements have been performed on the active material of a set of wafers with *Si* rich Si_3N_4 . The initial intention justifying this study was to have an idea of the implantation profile of samples with different implantation doses, though this has been only possible in part.

The analysed samples were the *sample 1* of Run 5107_UB and *samples 1, 2 and 3* of Run 5372_UB. The characteristics of those samples were listed in Tables 4.2 and 4.3.

We have focused the analysis on the presence of *Si* and *N* on the samples (although *Ar*, *O* and *C* were also measured). On figures 4.12 we show the typical binding energy spectrum of those species, presented for the particular case of *sample 1* of run 5107_UB. It is interesting how the contribution associated to Si is changing the spectral shape on the top and bottom of the sample, because on these parts Si is present in the SiO_2 and has a lower binding energy.

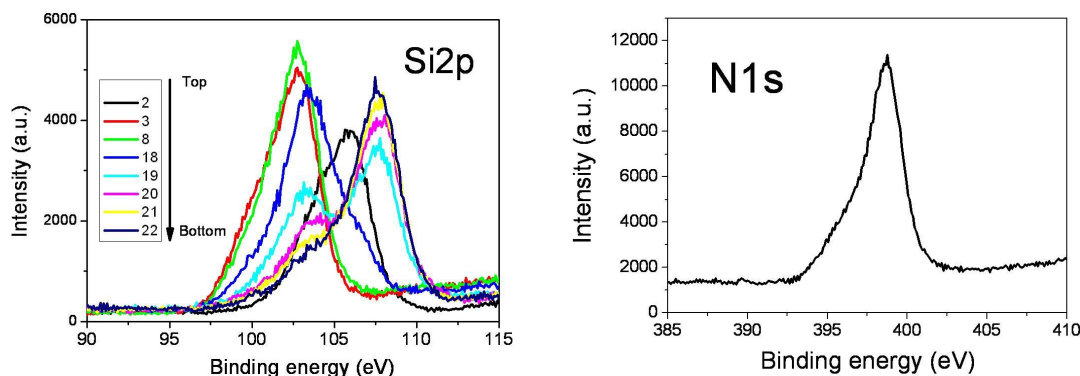


Figure 4.12: *Spectral shape of the Si_{2p} and N_{1s} contributions for sample 1 of Run 5107_UB*

We have analysed the atomic concentration profile for the different measured samples, but it is not possible to extract much significant information from this. It is only possible to say that run 5107_UB was slightly thicker than the 5372_UB, but the Si concentration seems similar for all the samples.

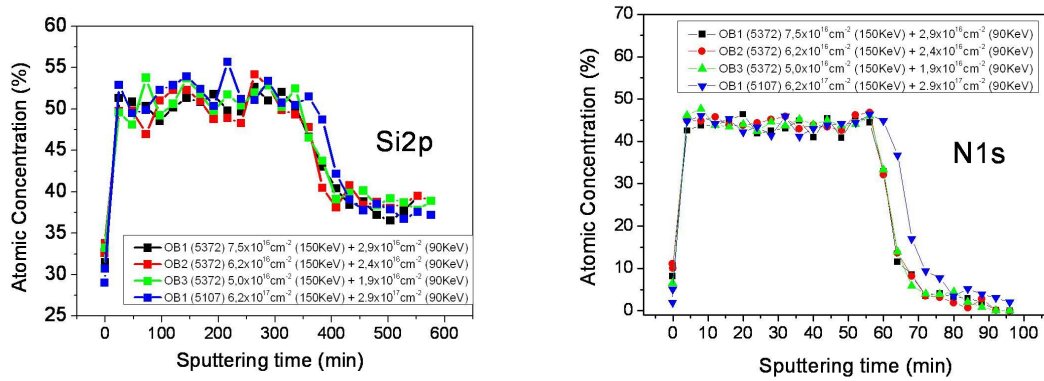


Figure 4.13: Si_{2p} (Left panel) and N_{1s} (Right panel) atomic concentration profiles of the studied samples.

There is however a difference in the spectral shapes of the Si_{2p} contribution at the central part. In fact it seems that the sample with higher implantation dose is shifted towards higher binding energies, which is probably an indication of a high number of $Si-Si$ bonds associated to the high presence of Si in this sample.

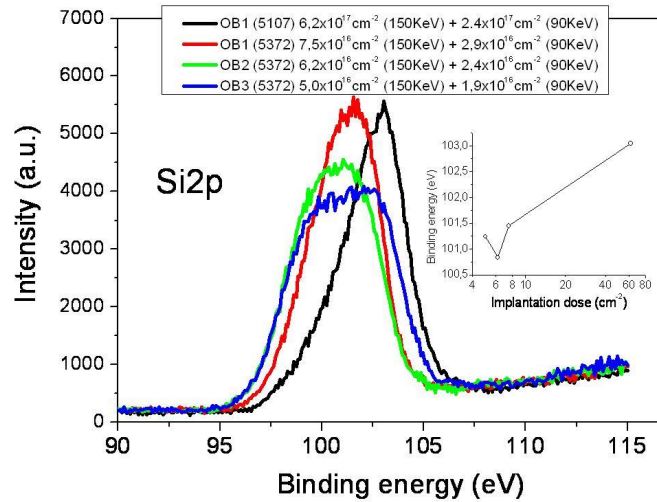


Figure 4.14: Si_{2p} binding energies for the different samples at the same depth.

4.5 EFTEM analysis

As discussed in Section 2.1 it is very important to know how the Si excess behaves inside the Si_3N_4 , in order to understand the real origin of the photoluminescence.

With this goal Energy-Filtered Transmission Electron Microscopy (EFTEM) analysis on samples with a more complex cross-section produced in our group has been performed. The structure under study is a Silicon Nitride MNOSLED composed by a 3 nm thick tunnel oxide SiO_2 surmounted by a 26 nm thick Si_3N_4 layer. The deposition parameters used for the fabrication of the Si_3N_4 layer are the same used in our samples. Both layers were successively implanted with Si ions. Afterwards, the wafers were annealed in an N_2 atmosphere at $1000^\circ C$ for 1 hour in order to induce phase separation. (for further details see [55])

The results have revealed the absence of Si crystalline nanostructures inside the active layer Si_3N_4 , even in the case of the highest Si excess.

Indeed, as we can see in Figure 4.15 (a), in the Silicon Nitride layer at the border of the polycrystalline silicon (pc-Si) no Si -ncs were observed, in accordance others works , while in the SiO_2 layer small Si -nc ($3.8 \pm 0.5 \mu m$) were revealed.

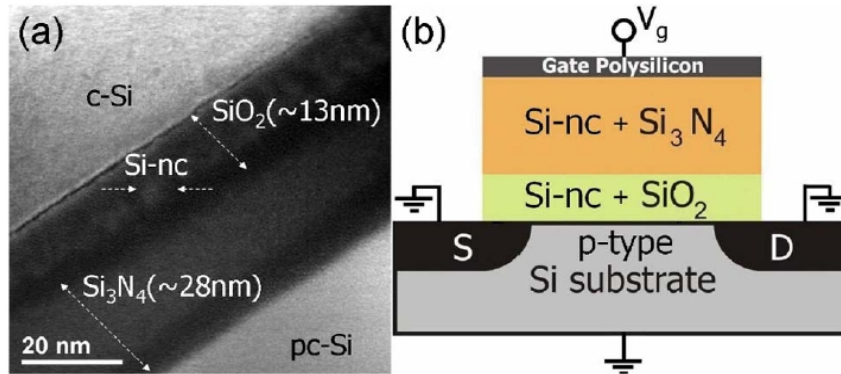


Figure 4.15: (a) Cross section EFTEM image showing the SRSO/SRSN bilayer and Si-ncs into the SiO_2 layer. (b) Scheme of the light emitting device structure. Picture taken from Y. Berencén et al. *Optics Letters*, Vol. 36, No. 14, July 15, 2011

Chapter 5

Experimental Setup and Characterization Techniques

In the previous chapters we have described the materials used and the fabrication of the active and passive optical components under analysis. Their optical properties have been characterized by means of different measurement techniques, which have been performed in two different setups. The first one has been designed in order to estimate the optical losses in waveguide structures (WG). As we will see more in detail in Chapter 6 the characterization process of the WG plays an essential role in the optimization of the characteristic parameters of the material (variation of the implantation dose and implantation profile) and the geometry of the optical elements (as the roughness or the deformations of the lateral walls of the structure). In this setup it is possible to operate using two different standard techniques covering a wide region of spectrum in the Visible (*VIS*) and the Near-Infrared (*NIR*).

On the other hand a high spectral resolution micro-photoluminescence (μ PL) set-up has been conceived in order to estimate the fundamental parameters of the active micro-resonators. Within the same setup is also possible to perform losses measurements of simple structures (planar or strip WG) in a continuous spectrum of wavelength between 450 and 900 *nm*.

5.1 Optical Losses Setup

In this section we will describe the experimental set-up through which we have measured the optical losses of the strip WG.

The input line is composed by a tapered optical fiber mounted on a nano-positioning stage through is possible to couple the Laser with the WG. The laser sources available in the laboratory are an *He-Ne* laser (633 nm) and a Fabry-Perot Benchtop Laser Source at 780 nm for the *VIS*, and a *Tunics-BT* laser, tunable between 1460 and 1540 nm, for the *NIR*.

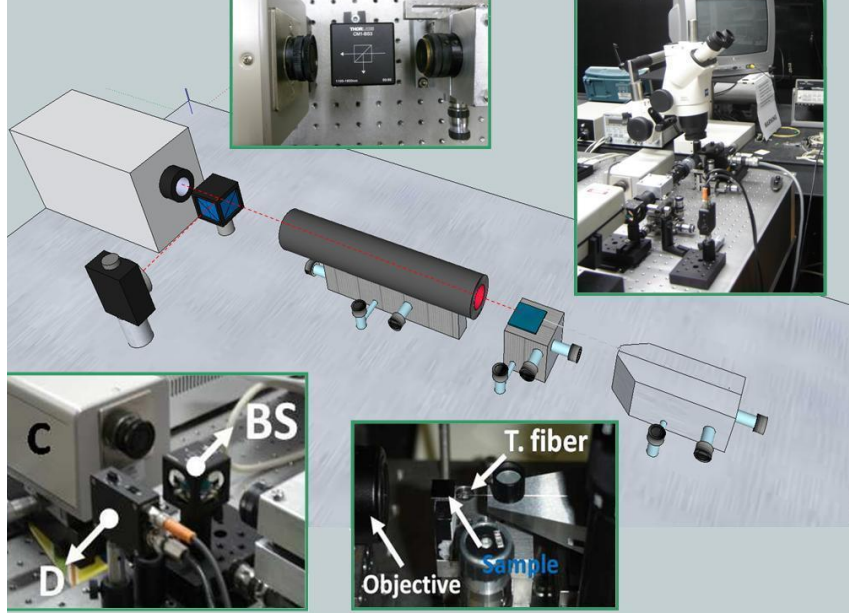


Figure 5.1: Scheme of the WG losses measurements setup. The dotted red line represent the two different parts in witch the collected light is divided by means of a Beam Splitter (BS) and sent to a detector (D) and to a IR camera (C).

The rough approach between optical fiber and the sample is controlled using a stereo microscope (*Zeiss Stemi 2000-C*). The out coming radiation is then collected by a system of lenses situated after the sample. A beam splitter placed in front of the lenses allows the division of the beam in two elements: one part is directed to a *VIS-NIR CCD* camera and the other one to a photodiode detector. Through the camera is possible to visualize

the shape of the optical mode, to evaluate the value of the modal order (in the case of multimodal WG) and maximize the fiber-waveguide coupling.

Two different detectors have been used to measure the intensity of the transmitted light in the range between 400 and 1800 nm: the first detector is the *Thorlabs PDA100A*, working in the *VIS* zone (400 – 1100nm), and the other one is the *PDA50B* and has been used in the *NIR* (800 – 1800nm).

The collection line is also equipped with a pinhole used to select only the radiation coming out through the *WG*.

5.1.1 Cut-back Technique

Propagation loss measurements at 780 and 1520nm have been performed in the described setup by means of the *cut-back technique* (CBT): this technique consists in comparing the values of the transmitted light through the WG of samples of different length. If a signal intensity I_{in} comes out from the input fiber and is coupled into a 2D-waveguide the light is attenuated as a function of the length L following an exponential law, which is the *Beer-Lambert law*. The light at the output (I_{out}) of a waveguide of length L is thus:

$$I_{out} = \frac{I_{in}}{C} e^{-\alpha'_{prop} L} \quad \text{BEER-LAMBERT LAW} \quad (5.1)$$

where α'_{prop} is the propagation loss coefficient in cm^{-1} (if the L is in cm) and C is the coupling factor, that is due to a not optimum coupling between the input fiber and the waveguide.

The total insertion loss (IL) of such a waveguide is thus:

$$IL(dB) = 10 \cdot \log\left(\frac{I_{out}}{I_{in}}\right) = -\alpha'_{prop} \cdot L \cdot 10 \cdot \log(e) - 10 \cdot \log(C) = -\alpha_{prop} \cdot L - \alpha_{coupling} \quad (5.2)$$

The output signal has been measured for at least three pieces of sample with different length each (5.1.1 left). It is possible to extract the propagation loss coefficient α_{prop} expressed in dB/cm and the corresponding coupling loss coefficient $\alpha_{coupling}$ in dB respectively from the slope and offset of a linear fit in logarithmic scale of the data.

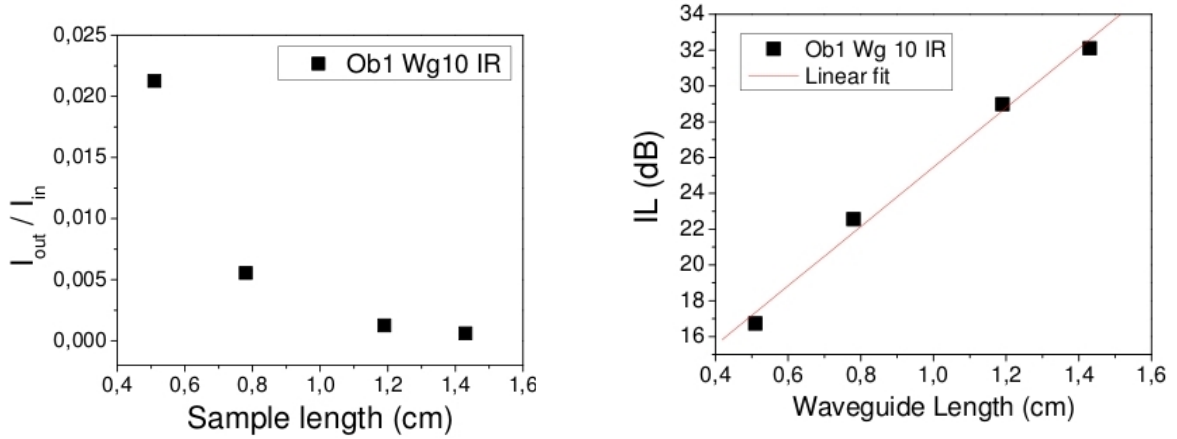


Figure 5.2: Left panel: representation of I_{out}/I_{in} values as a function of sample length. Right panel: $IL(dB)$ representation of I_{out}/I_{in} values.

The $\alpha_{coupling}$ represent the amount of light lost in the input coupling process: this kind of losses depends mostly on type of the experimental setup (e.g. normal or tapered fiber coupler) and on the coupling method used (e.g. grating coupler or cut edge coupling), and do not affect the α_{prop} value. As shown in figure 5.1, in our setup we insert the light inside the WG on the sample by means of a tapered fiber, placed perpendicularly with one edge of the sample. In this case the $\alpha_{coupling}$ depends dramatically on the quality of the cut of the sample's edge. For this reason there is the possibility to find samples having small α_{prop} coefficient but high $\alpha_{coupling}$.

For the completeness we have to remark that, due to the minimum resolution of the detector and the power of the pumping laser used in our setup, we can not measure losses values lower than 1 dB/cm using the *CBT*.

5.1.2 Scattered Light Collection Technique

One limitation of the Cut-back technique is the difficulty to cut samples of short length. If a sample posses very high losses values, it might be impossible to detect light at the end of the *WG*. In this case it is useful to perform *Scattered Light Collection Technique (SLCT)* measurements. This method consists in collecting the light scattered from the top of the *WG* with a *VIS* or *IR* camera, and analyse with a software the intensity decay along the

sample. In case of small losses value the fundamental requirement for the applicability of the *SLCT* is that the WG under analysis has to be long enough in order to appreciate the decreasing of the scattered light along the total WG length.

Thanks to this method we are able to estimate very high losses values. However it is more complicated and less reliable than the cut-back, because some assumptions have to be taken in account that not always are accomplished. First of all we have to assume that the responsivity of the detection system is linear with the variation of the received light. This fact needs to be verified in the data-sheet of our instrument and eventually a correction has to be applied.

The second assumption is that the scattering inside the WG is linear with the light intensity all along the sample.

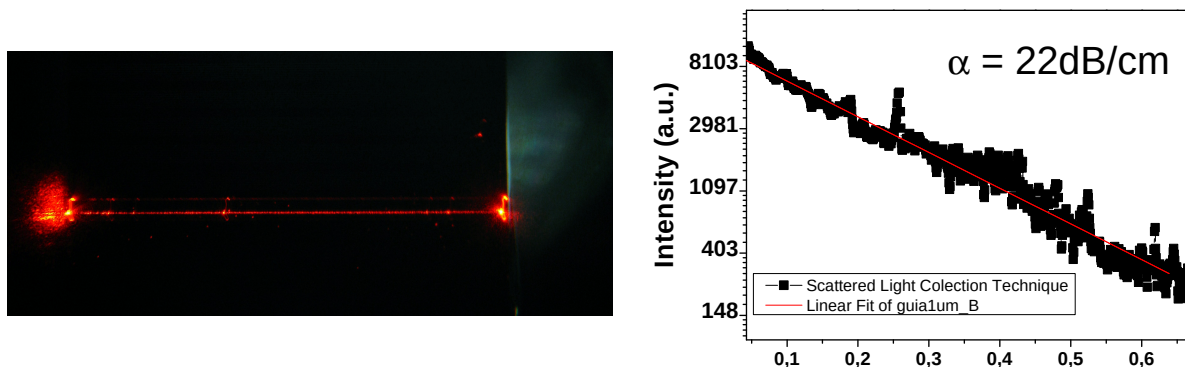


Figure 5.3: *Left: representative picture of the top view of a waveguide with a coupled laser. Right: example of graphic obtained after the treatment of the images obtained by Scattered Light Collection Technique.*

Finally, as we can see in the left picture of Figure 5.1.2, only the central part of the sample can be taken in to account in the losses measurement, because of the great scattering at the in-out edges.

Due to the poor intensity provided by most of the lasers available in our laboratory, such measurements can be performed only at 633 nm, that can provide enough intensity. Since the He-Ne laser output is not directly injected in a tapered fiber we have used a fiber coupler optimized for this wavelength (*Thorlabs PAF-X-11-C*).

During the measurement it is possible to notice spikes due to over-scattering of the light

with the imperfection of the structure: such spikes contribute to rise the noise of the measurement, but do not have physical importance in the calculation of the losses coefficient. This issue can be solved using a digital filter implemented in the analysis software, that can automatically find and eliminate them.

5.2 μ -PL measurement setup

Room temperature μPL measurements on a single resonator have been performed using two different pumping lines (see Fig 5.4 for a scheme of the setup): one is represented by an Argon laser (especially the lines 476 nm and 488 nm) and a solid state laser emitting at 370 nm. These two lasers work both in CW, but it is also possible to modulate the pumping light with a chopper or an Acousto-Optic modulator (Brimrose FQM-80-2) in order to modulate the beam with a maximum resolution of 200 ns.

A long working distance (20.0 mm) objective (Mitutoyo Plan Apo 20x) was used to focus the laser beam on the samples, having the possibility of a single resonator excitation since the well focalized spot can have a diameter of a few μm (Figure 5.5 left).

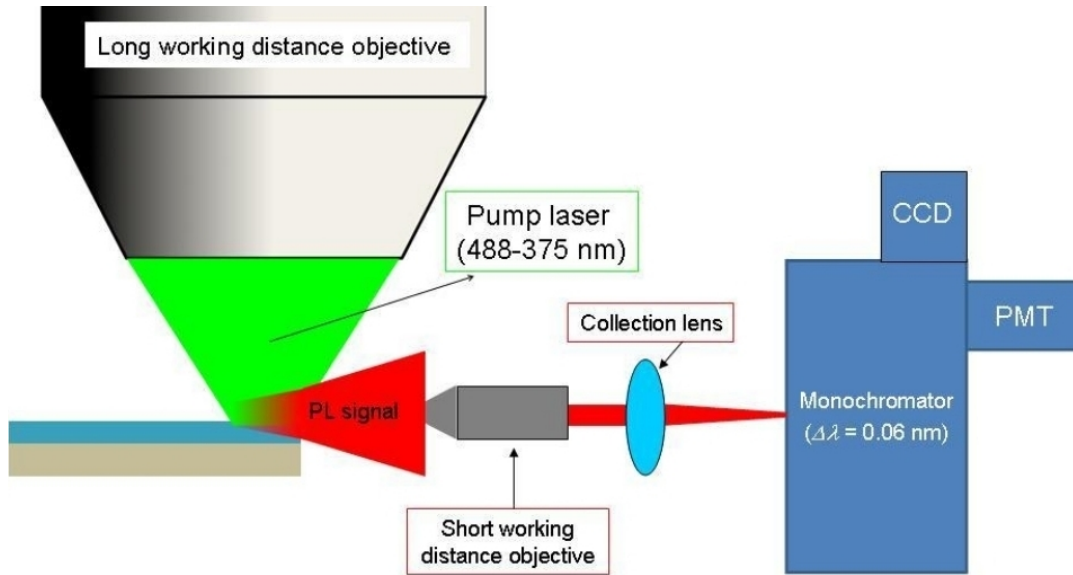


Figure 5.4: Graphical representation of the μ -PL setup used for the characterization of the circular resonators.

5.2. μ -PL MEASUREMENT SETUP

The objective is in fact mounted on a micro-positioner, allowing the de-focalization of the spot: this option is useful in order to generate a spot of different size, in order to adapt the pumping to the whole set of samples with different radius, or to excite more than one resonator. (Figure 5.5 *right*).

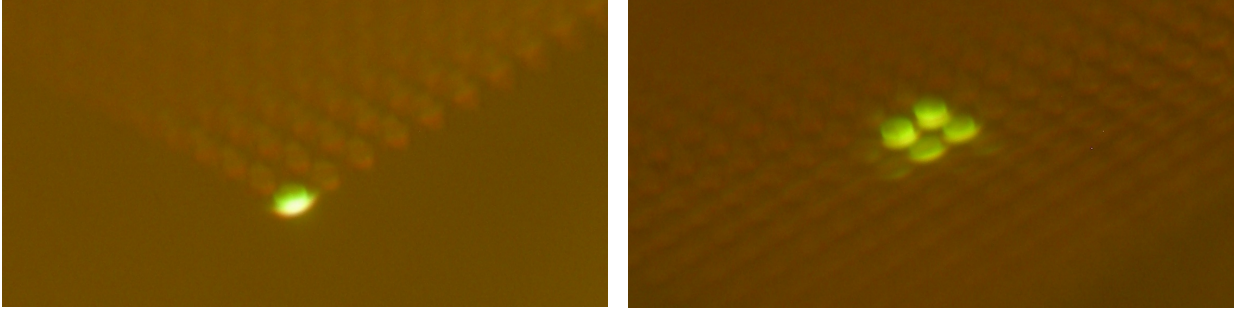


Figure 5.5: *Picture of a single (left panel) and multiple (right panel) μ -disk excitation obtained by means a de-focalization the pumping spot. The yellow dye photographs were taken using a 500 nm long-pass filter in order to eliminate the noise due to laser scattering.*

As the spot is smaller than the resonators top area it is thus possible to study the variation of the resonances shape and intensity ass a function of the variation of the spot position on the resonator. The microdisks/microrings close to the cleaved sample edge were excited vertically, while the WGM emission was monitored in the plane of disks, using collection short working distance objective. ($NA = 0.4$).

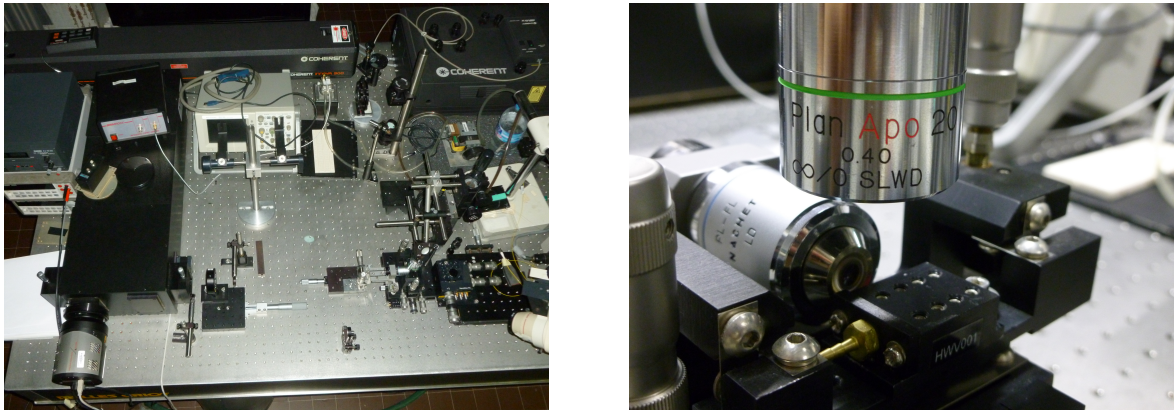


Figure 5.6: *Top view of the μ -PL setup (left picture) and particular of the sample holder and the pumping and recollection objectives (right picture).*

Following in the collection line after the objective, a micrometric pinhole was placed in order to block the out-of-plane PL emission, reducing the noise of the measurement. A linear polariser was also set in the collection line to select the *TR* or *TM* polarized emission. The collected signal was afterwards focused on a high-resolution monochromator (*SpectraPro 2750*, focal length 750 mm, f/9.8) with a three gratings turret: two working in the VIS (a 600 g/mm with spectral resolution of 0.16 nm and a high precision 1200 g/mm grating with resolution of 0.06 nm) and one for the IR (600 g/mm). One exit of the monochromator is coupled to a visible *CCD* camera (*Acton Pixis 100*), while in the other has been placed a PMT.

The principal issue to solve in order to perform a measurement that could be representative regards the quality of the cut, which has revealed of great importance, since it has to be done through the internal part of a resonator array, and in such a way that the resonator remains intact (see Figure 4.11). We have experienced that we can have a good detection of WGM from the three rows closer to the edge, since the on-plane PL emission of resonators placed on further rows becomes screened by closer non-pumped resonators. Indeed one of the hardest task of this measurement is to pump a resonator which is on the first row and not broken by the cut.

Nevertheless the use of the *CCD* camera permits to simplify the alignment operation respect using a PMT: it is in fact possible to evaluate in real time the alignment of the system by monitoring the presence of resonances in a large wavelength window (~ 30 to 50 nm depending on the used grating) even in a low light emitting samples.

Finally, the total on-plane emission can be quantified by measuring the signal collected by the objective (which only collects a finite solid angle) with a calibrated photodetector, and then integrating to the 2π angle in which the on-plane μ -disk emission is distributed.

5.2.1 Shifting Excitation Spot technique

A further application of the described μ PL setup is given by the possibility of performing Shifting Excitation Spot technique measurements (SES). The *SES* consists of measuring a series of PL spectra by varying the position of the excitation spot respect to the edge of the sample (where the *PL* emission is recollected). This method present some similarity

to the previously described *CBT*, but with a fundamental difference: in the *CBT* the light source was an external laser coupled with the WG structure, while in the case of *SES* technique the light is generated by the material and directly coupled in the WG. For this reason the *SES* technique can only be applied to WG (planar or strip) containing a light emitting material.

The light coming out from the facet of the sample has been recollectd in horizontal configuration: the pumping spot position has been varied by moving the sample without changing the relative position of the system objective-sample holder. Inside the material the emitted light undergoes to an attenuation that follows an exponential law function of the distance between the pumping spot and the sample facet. All the spectra collected spectra can be compared each others to find a loss curve along the PL emission spectrum (in the range between 550 and 900 *nm*).

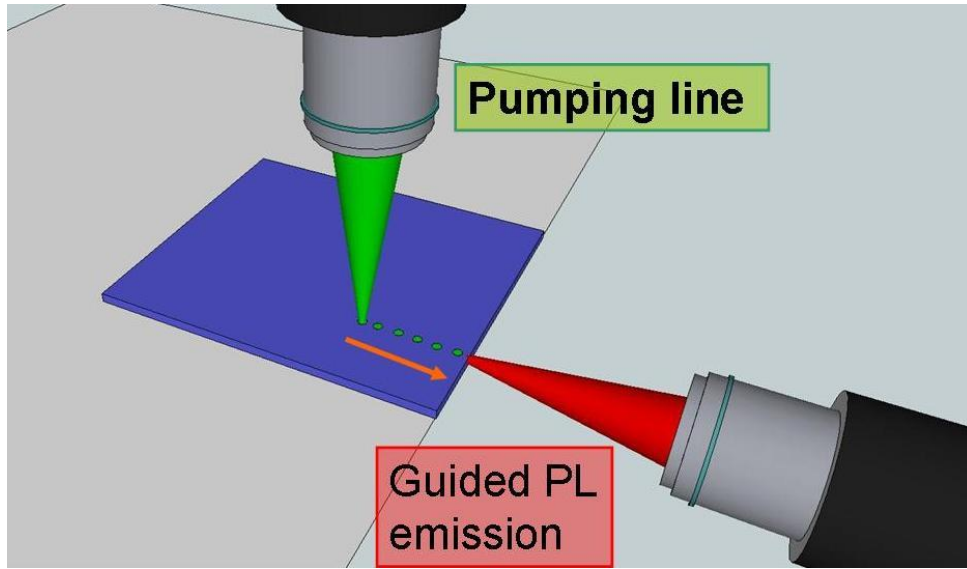


Figure 5.7: *Schematic view of the Shifting Excitation Spot technique setup. The sample is excited by a micrometric spot in different position on the same line perpendicular to the sample edge.*

Nevertheless it is important to remark that, at the best of our knowledge, such technique has only been applied to planar WG [71] in which only a finite percentage of optical radiation, depending on the spot-sample edge distance, can be collected by the objective.

This causes an overestimation of the losses and a $1/r$ correction has to be also applied since the emission is isotropic in the planar direction (r being the distance to the sample edge). Furthermore on the planar WG case, much care must be taken to keep the objective collection efficiency constant while the excitation source moves within the sample and the edge emitting region (with acceptable k vectors) becomes wider.

In this work, on the contrary, the *SES* technique will be applied to strip WG: we believe that this new approach provides a significant upgrade since there is no correction to make on the measured data, being the generated light coupled in the WG collected at the output always in the same condition, independently to the pumping position.

Chapter 6

Optical Losses and PL characterization

The optimization process of the active and passive material, that will be at the base of the final device, is mostly achieved by estimating the optical losses of the material itself. In this chapter we will show the results concerning the optical losses measurements performed in the VIS and IR spectral region on both passive stoichiometric Si_3N_4 and active SRSN straight WG.

In the first section our attention will be focused on the optimization of passive WGs: in order to obtain low losses structures we have taken in to account the following geometrical and material parameters:

- i) Geometrical parameters of the WG (width and height).
- ii) Annealing time and temperature.
- iii) Etching type (shallow or deep etching).

Although the PL emission range of the active material is located inside the VIS region, the estimation of the optical losses in the IR will be important in view of the possibility, already investigated by other authors [72]-[75], of implanting the WG with rare earth (e.g. Er^{3+} ions) emitting in the IR.

The important informations concerning the material and geometrical optimization obtained through the described study will be then compared with the results of the simulation and successively used in the design of the resonators and the coupled structure.

The main topic of the second part of the chapter will be the characterization of the active material. The obtained results in terms of PL intensity and optical losses, will assume a great importance during the selection process of the best active material: a fundamental parameter such as the Q factor is in fact directly dependent from the losses induced by the presence of Si excess inside the Si_3N_4 matrix (contained in the α coefficient in Equation 2.26).

Besides the cut back technique, mostly used in this chapter, the active material samples will be characterized by means of the SES technique inside the WGs. We have already state in Section 5.2.1 that this technique helps us to obtain an estimation of the losses of the active material in a continuous range of wavelength determined by the PL emission spectrum of the material.

To provide a better understanding of the characterization process, small tables summarizing the parameter involved at each stage of the optimization, will be added in all the following sections.

6.1 Passive WG optimization

6.1.1 Annealing treatment and geometrical variation

The first aspect that we will analyse in this section is the incidence of the annealing treatment on the optical loss of the passive material. It is worth to remember that a passive WG placed below the active resonator in the final coupled device will have to suffer the annealing performed in order to achieve the phase separation in the active material layer: we have therefore to make sure that the quality of the passive WG is not subjected to degradation after this process, comparing the obtained results with the losses values of samples having the same characteristics but that have not suffered an annealing process (*sample 2*). Taking into account those considerations we decided to fabricate passive samples in which annealing temperature has been varied between 1000

and 1150°C (*samples 4, 5 and 6* of run 5094_UB), since this is the range of temperatures that were scheduled for the thermal annealing, performed in an inert environment of N_2 (see Table 6.1).

The measurements on all the described samples were performed in both the VIS (633 and 780 nm) and IR (1480 and 1520 nm) spectral regions and for a set of WG with different width (between 1 μm and 10 μm wide) to estimate at the same time the influence of the annealing and the WG width on the optical losses.

Sample	Si_3N_4 Thickness (nm)	Protection SiO_2 layer(nm)	Annealing treatment for 4h	Annealing Before / After lithography (B/A)
2 5094_UB	150.3	200.5	Not annealed	/
4 5094_UB	150.3	180	1000°C	A
5 5094_UB	150.3	180	1100°C	A
6 5094_UB	150.3	180	1150°C	A
7 5107_UB	500	/	1100°C	A
8 5107_UB	500	/	1100°C	B

Table 6.1: *Main parameters of the samples that have suffered a further annealing treatment at different temperature.*

In addition we will compare the described wafers with samples pertaining to a following run (*samples 7 and 8* of run 5107_UB) composed by passive WG having a thicker core (about 500 nm): this further study can be useful in order to understand if there is a thickness limitation for the growth of a Si_3N_4 layer, due to stress issues. At the same time, on these structures, we can check if to a rising of the WG heigh correspond a diminution of the optical loss, especially in the IR region.

Visible region

Using the IR CCD camera it was also possible to visualize on a screen the shape of the mode transmitted by the WG: this has permitted us to make a first analysis of the

structure of modes supported by Si_3N_4 WGs. If in the smallest structures (1, 1.5, 2, 3 μm) the intensity of transmitted light clearly belongs to a first order mode, we can not say the same thing for the biggest ones. The multi-modal behaviour can be easily observed by coupling the laser with a 10 μm width WG: making small movements with the micro-positioning stage we can observe how the relative position between WG and fibre influences on the excitation of particular higher order modes.

The first relevant result that we can observe on *samples 4, 5 and 6* is that there is not clear dependence of the propagation losses on the WG width: as we can see in Figure 6.1 (a) and (b), there is maybe a slight lowering of the optical loss as consequence of the increase of the WG lateral dimension, but it is masked under a error bar. Such huge error is due to the fact that the measured losses lies under the minimum resolution of the experimental result.

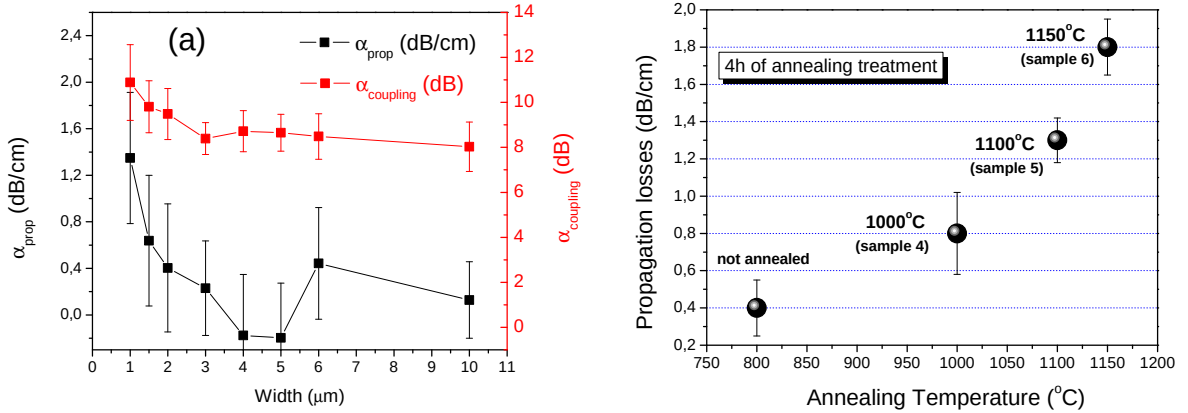


Figure 6.1: Comparison between (a) Propagation and Coupling losses in sample without annealing process (sample 2) and (b) Propagation losses for the not annealed sample and samples treated at 1000 $^{\circ}C$, 1100 $^{\circ}C$ and 1150 $^{\circ}C$ (sample 4, 5 and 6).

For the same reason, the strange behaviour of the WGs of 4 and 5 μm width pertaining to *sample 2* - in which the loss value decrease under the zero - is not related with some sort of optical gain, but is simply the error due to an experimental limitation.

For these reason, from now on, we will assume the main source of the measured propagation losses as an intrinsic characteristic of the material itself, at least in the VIS region.

Afterwards we have compared the results obtained on samples with (*sample 2*) and without an extra annealing process (*sample 4, 5 and 6*). It is immediate to see that their values are quite similar, even if the sample 2 has slightly lower losses: both Figure 6.1 (*a*) and (*b*) show propagation losses values about 2 dB/cm , while maintaining coupling losses around 10 dB (depending on the quality of the sample's cut). This suggests us that the increasing of the propagation losses at 780 nm subsequent to an annealing process is limited to a maximum of 1 dB/cm .

The results obtained so far are a quite good starting point, also if compared with the results reported in literature obtained for similar nitride WG structures. [76]-[78]

This is an excellent result towards the overall goal of this work, since the passive WG can be considered already as optimized. In fact the annealing temperatures used to obtain phase separation and the consequent formation of Si-nc in the Si_3N_4 matrix, are not affecting the losses of the passive WG in the visible.

Another interesting result concerns the losses variation between two different wavelengths of the visible spectrum. Using Scattered Light Collection Technique described in Section 5.1.2, we have estimated the propagation losses at 633 nm (Figure 6.2).

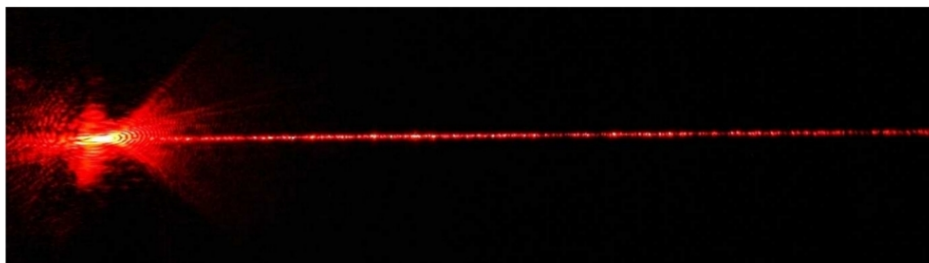


Figure 6.2: *Upper view of a $10\text{ }\mu\text{m}$ WG belonging to sample 5, coupled with a 633 nm laser beam.*

Though being very low, the values at 633 nm are slightly higher than the ones measured at 780 nm and they do not seem to follow the same increase as function of the annealing temperature obtained at 780 nm .

In Table 6.2 and 6.3 we can find the summary of the optical losses results that we have obtained for the widest WGs ($10\text{ }\mu\text{m}$) in the samples under study for both visible analysed wavelengths.

OB	VIS (633 nm)	VIS (780 nm)	
	Propagation (dB/cm)	Propagation (dB/cm)	Coupling (dB)
2 (5094_UB)	/	0.4	8.5
4 (5094_UB)	2	0.8	9.2
5 (5094_UB)	5	1.3	13.3
6 (5094_UB)	3.5	1.8	11.2

Table 6.2: Summary of the losses values obtained at 633 nm and 780 nm, for 10 μm passive WG.

According to these results we can suppose that transmission in the VIS region is not significantly affected by the annealing processes or the WG width. That being so, prospective losses problems in active WG (starting with the run 5107_UB) will be related principally to the Si excess.

Infrared Region

The first result obtained in the IR region is an evident rise of the optical loss of 6-7 dB/cm with respect to that obtained in the VIS. At 1480 nm propagation losses result to be around 8 $\frac{dB}{cm}$ - with a small increase with the decrease of WG width - for all the annealed samples. (Figure 6.3 (a)) It is also important to notice that the values concerning the WGs with width of 1 μm were not reported because of the small signal transmitted, comparable with the noise level of the detector.

In this spectral region, the propagation losses of the annealed samples seem to be subjected to a remarkable lowering of approximately 9 dB/cm with respect to the not annealed WGs. Indeed, the high temperature annealing treatments may help to improve these losses values: it is indeed likely that the presence of *N-H* bonds, *Si-H* bonds or even *O-H* bonds within the upper cladding cause absorption in the analysed region. Especially, the former kinds of bonds act as an absorption centre and their low energy tail lead to undesirable absorption loss in the IR region.[79]-[80] On the other hand, the optical loss of the annealed Si_3N_4 still continue to remain high if compared with the VIS region: this behaviour suggests that the mayor cause of the losses has to be identified in another factor, independent from the annealing temperature.

Furthermore, similarly to the VIS case, the analysed samples seems to be insensitive to changes between 1000 and 1150°C of the annealing temperatures.

Concerning the coupling losses, we have to remark that they are only related to the characteristic of the experimental setup and to the width of the WGs: as we can see in table 6.3 their value is always around 7 dB. The characterization at 1520 nm confirms the results extracted at 1480 nm.

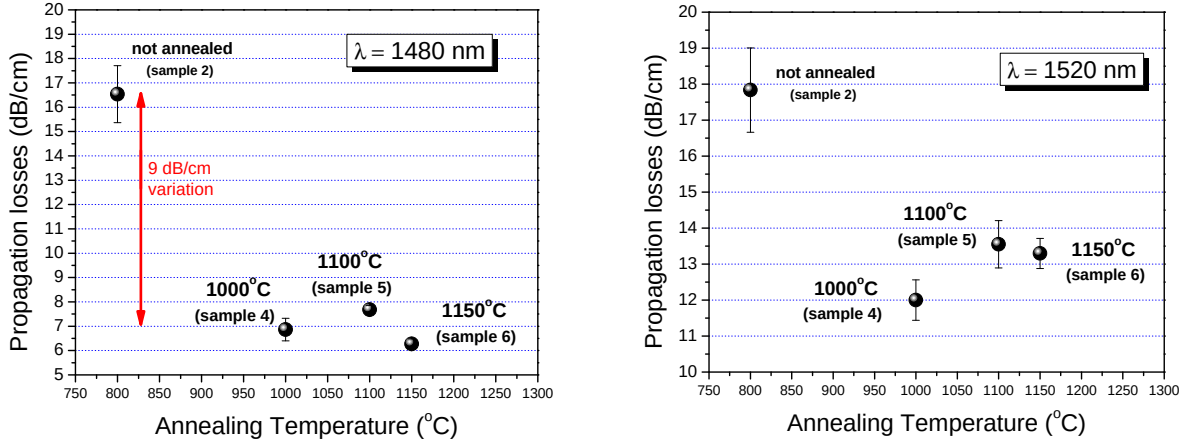


Figure 6.3: Comparison between (a) propagation losses at 1480 nm (b) and 1520 nm in samples that have suffered a different annealing process (samples 4 to 6) and a not annealed sample (blue stars representing sample 2).

We can also notice that, differently than to found for VIS region 6.1 (a) and (a), in the majority of the samples we were not able to analyse the narrowest WG (1 μm) (Figure 6.3). The main reason is due to the smaller beam size at the focus of the tapered fibre with respect to the IR case.

Having established that there is no remarkable variation of the losses between samples with annealing temperature ranging between 1000°C and 1150°C, the last variable operating in our system is the the thickness of the WG. Sure enough, for the case of *samples 7* and *8* of run 5107_UB, the losses measurements in IR region revealed very low losses values ranging between 0.8 and 1.7 $\frac{\text{dB}}{\text{cm}}$. This result, comparable with the results published by others groups in the IR region [81] allows us to conclude two facts:

- i) The losses in the IR region reported in samples of run 5094_UB are mainly related to

the small thickness of the layer and not to material losses, since for a much thicker sample, losses are very low.

- ii) Stress is not an issue and layers as thick as 500 *nm* can be deposited without the appearance of significant losses.

In order to have more insight on the conclusion i), we have done simulations (using the Beam Propagation Method) of the energy distribution of the fundamental mode for a WG geometry equal to the measured structures.

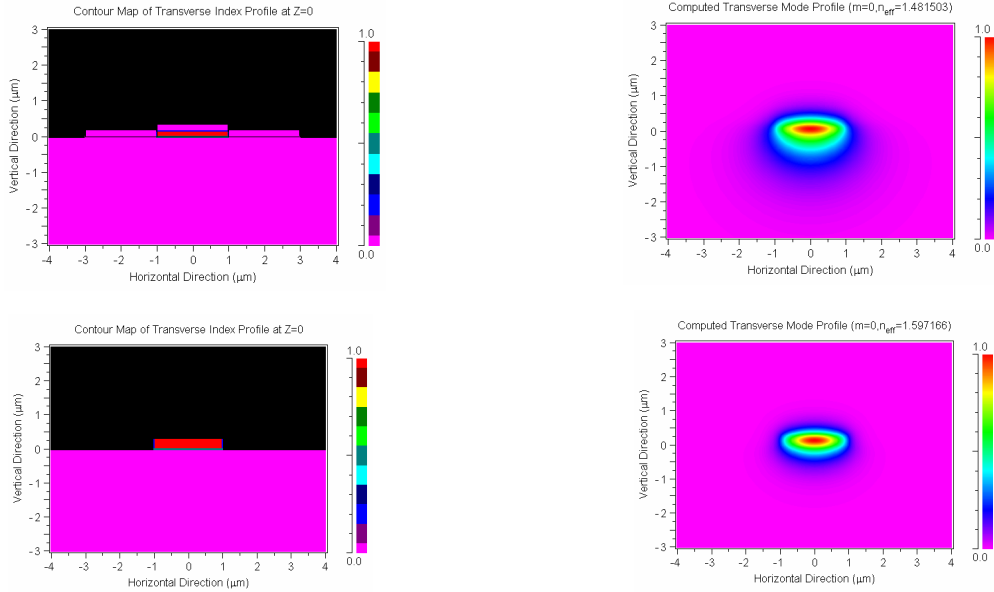


Figure 6.4: *Top panels: simulated structure of a 2 μm width WG and thickness of 150 nm, with a covering cladding. (Run 5094_UB) and energy distribution of the fundamental mode at $\lambda = 1480\text{nm}$. Bottom panels: structure of a 2 μm width and thickness 300 nm WG (Run 5107_UB) and energy distribution of the fundamental mode*

It is worth to remind that the thickness of these samples was chosen to optimize the transport of radiation in the VIS region, and not in the IR. Indeed, if we compare the simulated modal structure of a 150 *nm* thickness WG (top panels in Figure 6.4) and that of a 300 *nm* WG (bottom panels in Figure 6.4) we can notice that the second one results to be more confined inside the WG.

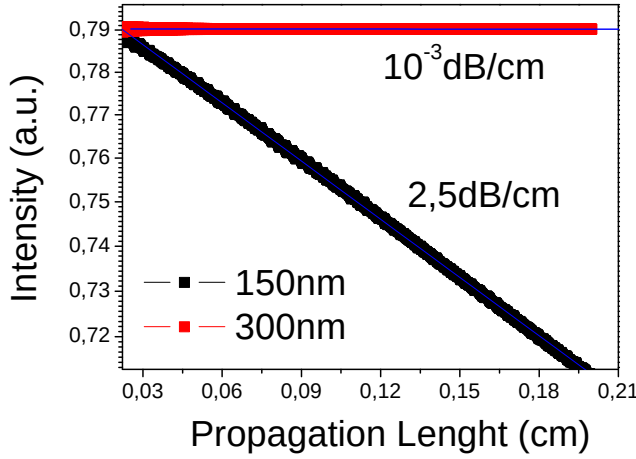


Figure 6.5: Comparison between losses value for WG structures described in Figure 6.4.

Table 6.3: Table of the optical losses obtained in the IR region for the passive WGs.

Sample	IR (1480 nm)		IR (1520nm)	
	Propagation (dB/cm)	Coupling (dB)	Propagation (dB/cm)	Coupling (dB)
2 (5094_UB)	16.5	10.2	17.8	9.8
4 (5094_UB)	6.8	11.1	12.1	12.2
5 (5094_UB)	7.6	14.7	13.5	14.3
6 (5094_UB)	6.27	15.1	13.2	16.1
7 (5107_UB)	0.8	15.1	/	/
8 (5107_UB)	1.7	13.6	/	/

Simulations also confirm that even a 300 nm thickness WG (red points in Figure 6.5) the losses values are three orders of magnitude lower than a 150 nm of thickness WG (black points in Figure 6.5). Taking in account that the experimental values differ, the qualitative observation of higher losses for a 150 nm thick sample is explained.

In conclusion, the analysed passive Si_3N_4 WGs would be suitable for IR if the thickness is high enough (300 nm is indeed a good choice). Through these results we can deduce that passive Si_3N_4 WG with a adequate geometrical structure are a good choice for both

IR and Vis region, and is not necessary to resort to SOI structures.

In the following a table summarizing the results in the IR region is shown.

6.1.2 Etching variation

Between the whole set of process involved in the realization of a low loss WG structure, the etching may play an important role, because it defines the effective geometry of the optical structure. In order of establish which is the dependence of the optical loss from the geometrical shape of the passive WG, measurements were performed on *samples 1, 2* and *3* of the run 5094_UB. The characteristics of these samples are summarized in the following table.

Sample 5094_UB	Etching Type	Thickness (nm)
1	Total	150.3
2	Total	150.3
3	75 nm	150.3

Table 6.4: *Resume table of the etching type.*

As we can see in Table 6.4, the substantial difference between those samples lies on the etching type: in the *samples 1* and *2* the photolithographic process removed completely the Si_3N_4 material surrounding the WGs (forming *strip* WG structures), while for *sample 3* only 75 nm were etched with the consequent formation of *rib* WGs.

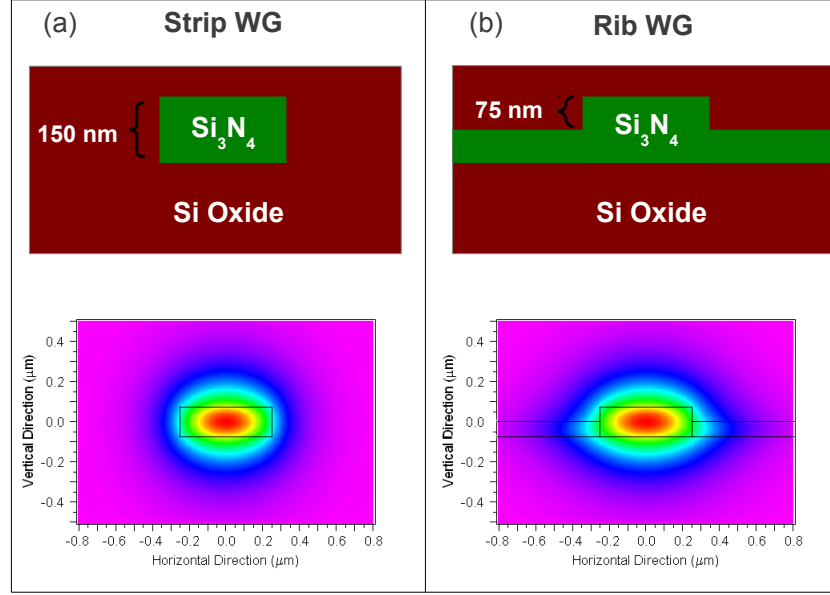


Figure 6.6: Scheme of the Strip (a) and Rib (b) Si_3N_4 WG and simulation of the spacial distribution of the TE polarized EM field at 780 nm.

In the strip WGs the mode is highly confined by the side-walls (see the panel (a) on Figure 6.6): however in presence of significant roughness, those side-walls can begin a not negligible source losses. Taking in account that, rib structures have been also realized, where the presence of the lateral walls are limited to the top of the WG.

In the IR region, the measurements performed on *samples 1* and *3* the propagation revealed that the loss coefficient remains roughly constant around $16\text{--}18 \frac{\text{dB}}{\text{cm}}$, due to the thickness limitation described in the previous section. It is thus impossible to establish if there is a real improvement using one particular etching type.

As expected, the results for the visible region are sensibly better, due to the higher confinement of the light at smaller wavelength, and the loss coefficients extracted are close to the minimal sensitivity of the experiment. For this reason, also in this case, it is hard to establish and compare the real value of propagation losses, though we are pretty sure they can be lower than $1 \frac{\text{dB}}{\text{cm}}$ for all the studied widths. Only a slight increasing of the optical loss can be found in the shallow etching WG, if compared with the deep etching

ones, but we can not extract any real conclusion from this measurements because such variation remains inside the error bar. (see figure 6.1.2).

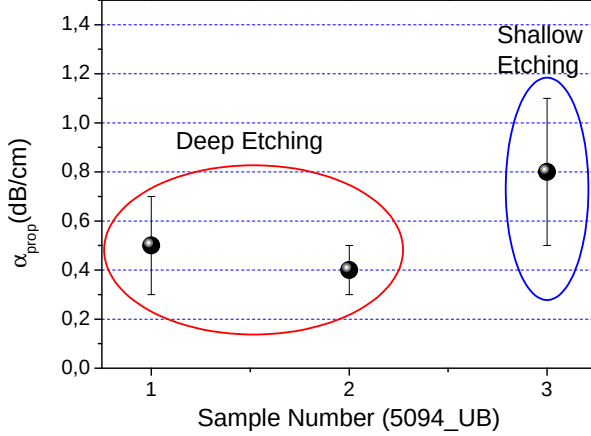


Figure 6.7: *Propagation and coupling losses at 780 nm and Laser power of 4.5 mW for samples with different etching: Deep (1 and 2) and Shallow (sample 3) belonging to run 5094_UB).*

As partial confirmation of this behaviour we can state that during the alignment operation it is easy to see how light diffuses onto this under-etched layer, probably causing the rising of the propagation losses.

In the Table 6.5 are summarized the results obtained for a representative set of WG belonging to *samples 1, 2 and 3* in both the VIS and IR.

Sample (5094_UB)	Etching Type Deep (D) or Shallow (S)	VIS Losses at 780 nm (dB/cm)	IR Losses at 1520 nm (dB/cm)
1	D	0.5	16
2	D	0.5	16
3	S	0.8	18

Table 6.5: *Summary of the results of the propagation losses measurements obtained by varying the etching type.*

In conclusion from the results obtained we can notice that there is no such appreciable difference between the Strip or Rib WG and both structures can grant low losses, below $1 \frac{\text{dB}}{\text{cm}}$ in the VIS region.

6.2 Active material characterization

Having found the best characteristics useful for the good functioning of the bus WG, the main goal became the optimization of the active material, in order to find the best compromise between optical losses and intensity of the PL emission.

In this section we will describe the optimization process of the active material, performed on WGs pertaining to the runs 5107_UB and 5372_UB. In particular we will focus our attention over the samples that have suffered a multiple implantation with silicon at different doses. Such study assumes a fundamental importance looking at the realisation of the active μ -cavities, since the Si excess rules both the intensity of the emitted PL and the Q factor of the resonator.

Other samples obtained with a single ion implantation will be also taken in to account, although in this case the characterization has not brought to remarkable results.

Table 6.6: *Resume of the active material samples implantation (Run 5107_UB).*

Sample	Si_3N_4 Thickness (nm)	Implantation energy (keV)	Implantation dose ($\times 10^{17} \frac{at}{cm^2}$)	Si Excess %
1 5107_UB	300	150/90	1.25/0.48	11.9
2 5107_UB	350	150/90	1.25/0.48	11.9
3 5107_UB	250	150/90	1.25/0.48	11.9
4 5107_UB	300	150/90	0.62/0.24	6.3
5 5107_UB	300	150	1.35	13.0
6 5107_UB	300	90	0.8	14.2
1 5372_UB	300	150/90	0.75/0.29	7.4
2 5372_UB	300	150/90	0.62/0.24	6.3
3 5372_UB	300	150/90	0.50/0.19	5.1
1 5506_UB	300	150/90	0.25/0.1	2.5

First of all PL intensity measurements were performed to establish the best material in

terms of light emitted intensity: we have characterised and compared the PL emission properties of a piece of bulk active material on all the samples. The results show an increase of the intensity with the rise of the concentration for a same pumping flux. (see Figure 6.8)

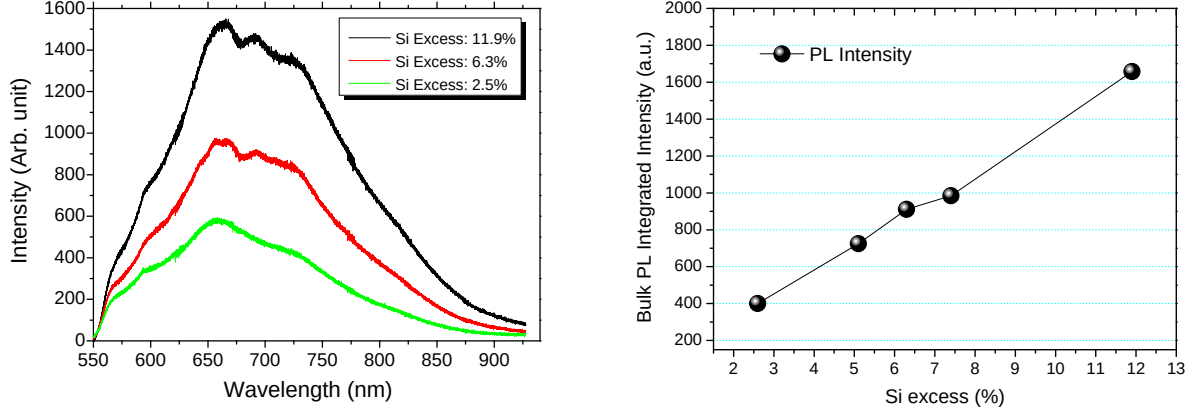


Figure 6.8: *PL spectra (left panel) and summary of its intensities (right panel) as a function of the Si excess.*

It is also interesting to note that the behaviour with the pumping flux Φ (represented in Figure 6.9) is linear, which means that, at least for this flux range, there are no flux dependent non-radiative recombination mechanisms (typical of Auger-like processes) competing with the spontaneous emission process.

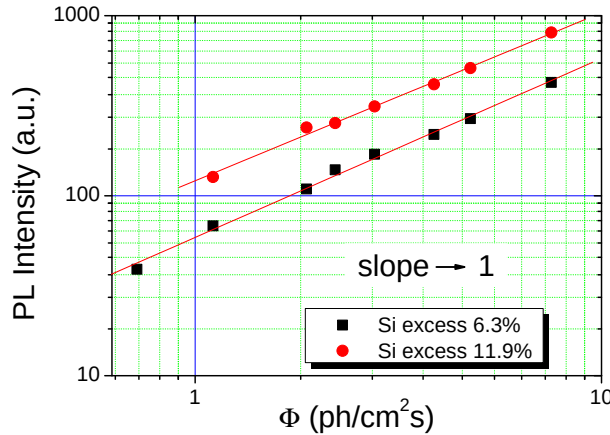


Figure 6.9: *Integrated PL emission as a function of the photon flux for two samples of different Si excess: 6.3% (black squares) and 11.9% (red circles).*

At this point it is very important to establish the exact behaviour of the losses as a

function Si excess.

With this goal we have taken advantage of the same experimental setup that we have used for the optimization of the passive material. Apart from the previously illustrated standard techniques (the *cut-back* and *top-scattering techniques*), the active material provides the possibility of performing the SES technique.

For the completeness we have to point out that also samples with different thickness have been produced (Samples 1 2 and 3 of run 5107_UB), but this study has not provided any significant result: the losses values reported were in fact below the minimum sensitivity of the experimental setup, so it was impossible to discern any potential different behaviour as a function of this geometrical parameter.

Finally it is worth to remind that during the design process we have identified a minimum thickness that a small radius μ -disk must have in order to reach values of Q factor higher than 10^4 (Figure 3.7). Therefore, the thickness of the core of the WG studied in this section is approximately the double respect to the WGs described in the previous section, that were intended as an optical bus channel. As a consequence, according to Figure 6.5, the radiative losses have to be very low, also in the IR region.

6.2.1 Visible Region

In the VIS region we have taken into account samples 1 and 4 of 5107_UB and *samples 1, 2 and 3* pertaining to run 5372_UB. In this case, as expected, the increase of losses with the increase of Si excess is remarkable. Samples containing a high implantation dose could not be even analysed (*sample 1, 5 and 6*) because the intensity of transmitted light is lower than the minimum measurable by our detector. *Sample 4* was the only sample that could be measured in the VIS, actually presenting quite low losses of about 6 dB/cm (Figure 6.10).

In order to overcome the limits arising from cutback technique and thus quantify the propagation losses of the high implantation dose samples we performed the measurements using the Scattered Light Collection Technique at 633 nm. Both Figures 6.11 (a) and (b) show that the values are between 100-200 dB/cm and we believe that those would be of the same order at 780 nm.

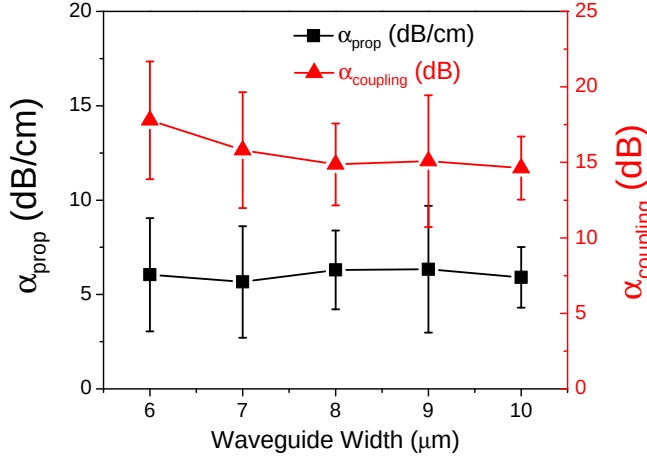


Figure 6.10: Optical loss at 780 nm due to the propagation and coupling inside active material's WGs having a Si excess of 6.3%

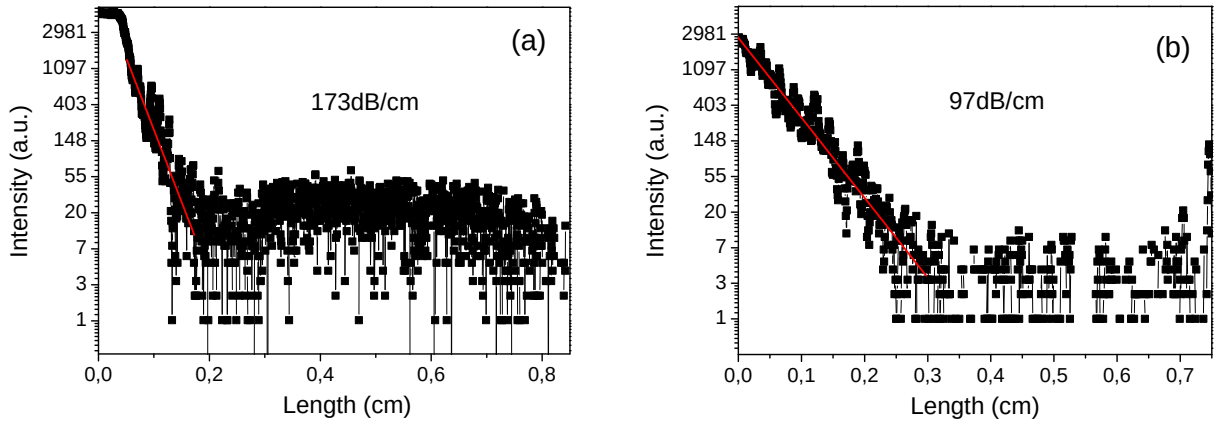


Figure 6.11: Propagation Losses calculated by means of the Scattered Light Collection Technique on sample 5 (a) and 6 (b) having high percentage of ionic implantation.

Looking at the latter results it is clear that the material obtained with high implantation doses or a single implantation are not suitable for building any active photonic device working in the visible region because of the unacceptable propagation losses. On the other hand, *sample 4* seems to be a good candidate as constituent material of the resonators, since it is the only sample having optical loss leading to a potential Q factor of 10^4 , according with Equation 2.26. We decide hence to produce a further run of samples with a value of Si excess dose around 6.3%.

Loss measurements in a $5\ \mu\text{m}$ wide WG have been done using cut back technique (reported

6.2. ACTIVE MATERIAL CHARACTERIZATION

on Figure 6.12) confirming a significant reduction of the loss value for the low Si excess samples.

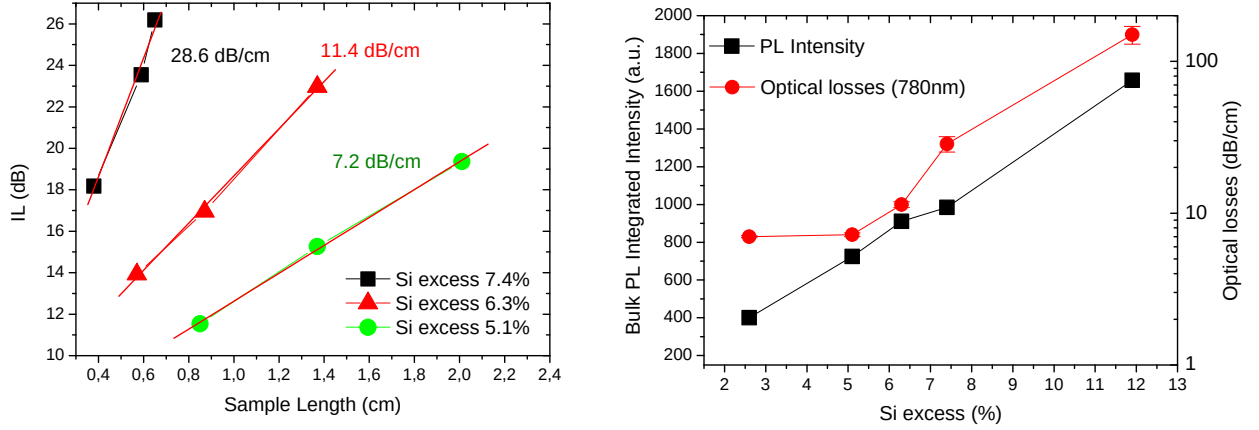


Figure 6.12: *Left panel: optical loss values at 780 nm for three different WG of 5 μ m width belonging to samples 1, 2 and 3 of run 5372_UB. Right panel: comparison between PL intensity and optical losses as a function of the Si excess %.*

On the following table we summarize the results obtained for the active WG.

Table 6.7: *CBT results obtained at 780 nm. TSC measurements have been also performed on that samples in which CBT has produced no results.*

Sample	Si Excess dose %	VIS (633 nm) Propagation (dB/cm)	VIS (780 nm) Propagation (dB/cm)
1 (5107_UB)	11.9	/	Above setup limit
4 (5107_UB)	6.3	15	9.3
5 (5107_UB)	13.0	173	Above setup limit
6 (5107_UB)	14.2	97	Above setup limit
1 (5372_UB)	7.4	/	28.6
2 (5372_UB)	6.3	/	11.4
3 (5372_UB)	5.1	50 (with SES)	7.2

6.2.2 Shifting Excitation Spot technique

Thanks to *SES* technique we were able go further on the analysis of the propagation losses present in the material and to extract their dependence in a continuous range of wavelength inside the PL emission band (approximately between 550 and 900 *nm*).

It is worth to remind that the application of this technique is possible only in WG composed by an active material. Previous works report the use of such technique only in planar bi-dimensional WG, needing some correction in order to eliminate the errors in the recollection of the out-coming light intensity, depending on the excitation spot position (see chapter 10 of [5]). In order to solve this problem, taking in account the conclusions formulated in the previous sections (the losses do not depend on the WG width, at least for wide WGs) we decide to apply the *SES* technique inside a 10 μm wide, using an excitation spot having diameter approximately of the same size of the WG.

Figure 6.13 shows the PL spectra measured for many different distances to the edge, resulting in a clear variation of their shape and intensity. The values reported in the legend are the distances between the excitation spot and the edge of the sample. On Figure 6.14 are shown the results of this study in terms of optical losses, reported in *dB/cm*, for the case of a strip WG with 2.5% Si excess (*sample 3* of run 5372_UB).

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. On the inset of Figure 6.14 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 of Figure 6.14, where we identify Rayleigh scattering from Si nano-clusters 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 λ^{-4} behaviour and the contribution of direct absorption losses starts to play a non negligible role. This explain the higher losses obtained with the *CBT* at 633 nm respect to that extracted at 780 nm.

The deviation from the Rayleigh scattering behaviour reported in Figure 6.14 can be as-

sociated to interband excitation of carriers to the valence band and is not expected to vary significantly with pump flux.

It is worth noting though that the quantification of this contribution is not possible to be done with the SES technique since only one point of the sample is pumped and the propagating waveguide region remains in a unexcited state, not susceptible to carrier absorption.

Nevertheless, as we will see hereafter, we have checked that the measured quality factors of the μ -disks noticing that they are not varying (at least on a quantifiable way) with pump flux on the full spectral range covered by the PL emission.

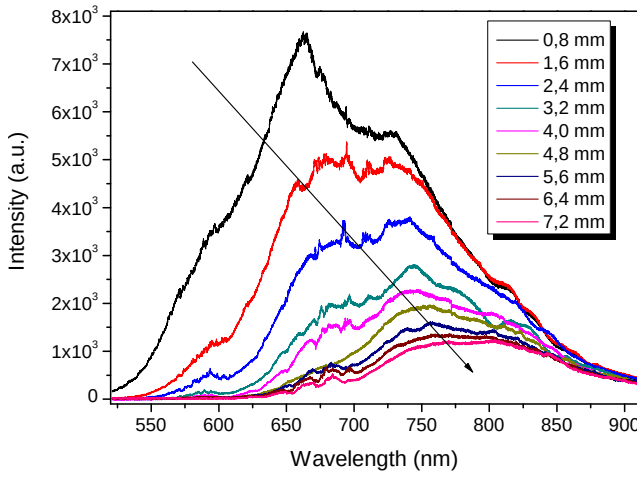


Figure 6.13: *Emitted PL spectra for nine values of position of the pumping spot on the WG.*

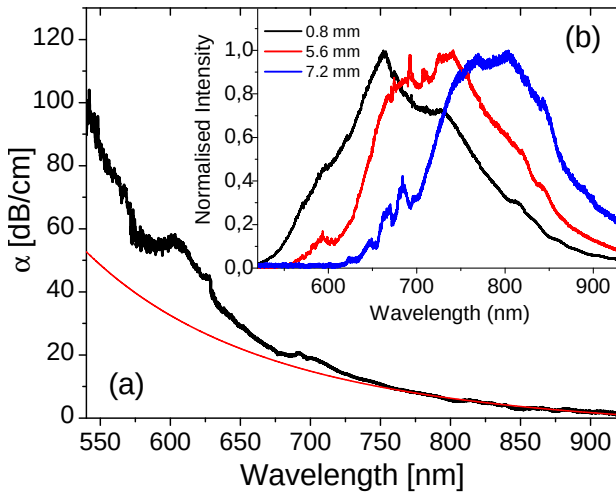


Figure 6.14: (a) *Spectral dependence of the optical losses of an active strip WG with 2.5% Si excess, as extracted from the SES measurements. The fit using a Rayleigh type scattering dependence is also shown.* (b) *Guided spectrum collected at the output of the WG for three different positions of the pumping spot: the red shift of the PL spectra is clear when moving the pumping spot inside the sample.*

6.2.3 Infrared Region

The optical losses analysis on the active WG in the IR region have been performed at 1480 nm following the described standard process. As we can see in Figure 6.15, the propagation losses for *sample 4* are very low, similarly to what measured on *samples 7* and *8* of the run 5107_UB (samples without implantation and with high thickness), since we are dealing with low Si-excess in the active region and a nice geometrical configuration for IR propagation (see simulation of Figure 6.4). In fact, the losses slightly increases with the Si excess, as we see by comparing the left figure with the right one that corresponds to *sample 6*.

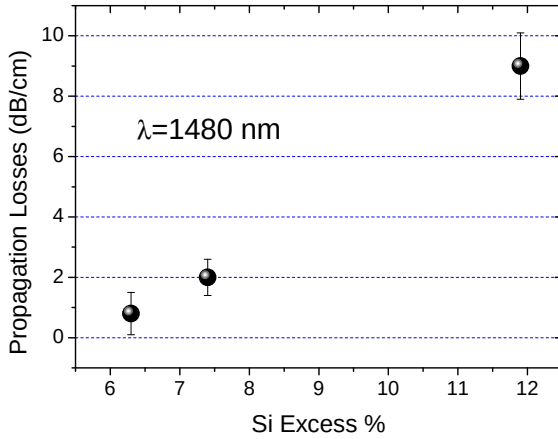


Figure 6.15: Representation of propagation losses for 11.9, 7.4 and 6.3 % Si excess WGs.

The previous statement is further confirmed by the results shown in Figure 6.15, obtained for *sample 1*, which demonstrate that the increase of losses is strongly dependent to the increase of the ion implantation in the WG. The propagation losses value decrease from 9 dB/cm to 1 – 2 dB/cm from *sample 1* to *sample 4*, while the implantation dose was decreased by a factor of two. Taking into account all these results, we believe that non implanted Si_3N_4 WG with about 300 nm core thickness will show negligible losses in the IR.

Table 6.8: *Summary of the losses values for the active WGs in the infrared region.*

Si Excess %	IR (1480 nm)	
	Propagation (dB/cm)	Coupling (dB)
11.9	9	14
7.4	2	12
6.3	1.5	17

Chapter 7

Circular resonant structures

In the following chapter we will explain how all the design, fabrication and characterization process described so far, will finally merge into the realization of the integrated sensor. Such device will monitor the changes of the effective refractive index of the resonant optical modes induced by variations of the refractive index of the surrounding material.

As explained in the introduction of this thesis, the fulfilment of this objective pass through the characterization of the isolated resonators, looking mainly at the optimisation of the Q factor of the WGMs and the intensity of the single resonance, that is hence the subject of the first part of this chapter.

Afterwards we will present an optical characterization of the coupled photonic system (optimized active μ -disk and passive waveguide) and an evaluation of its sensoristic performance. At this stage we will demonstrate that the proposed structure emits few nW in a single resonance and show competitive sensitivities and a detection limit, comparable with that in the literature for similar structures.

7.1 Isolated resonators

Looking at the results of Section 6.2 concerning on the realization of a good active material, it is clear the need of a material with low content of Si excess, in order to lessen the optical loss of the material itself. Taking in account this premise, we have analysed the

behaviour of the active material when used as base component of a resonator.

The optimization process of the active material composing the circular resonators involves the standard parameters that we have defined and simulated in the previous Chapters (i.e. the Q factor, the FSR and the PL intensity). Our attention will thus concentrated on variables pertaining to the active material, and governing the aforesaid parameters: for this reason in the present chapter we will take in to account samples belonging to run 5372_UB, having different annealing temperature and Si excess - the first three *samples* (1, 2 and 3) were annealed at $1100^{\circ}C$ while the *samples* 4, 5 and 6 at $950^{\circ}C$ (see Table 4.3). With this set of wafers we will try to obtain informations about the best implantation dose and annealing temperature: in practice we will try to reproduce the same study of Section 6.2.1, that it could not be performed using the losses measurement inside the WGs, due to the insufficient resolution limit of our setup. On the contrary, in this case we are confident to understand if there is some variation due to the annealing temperature by evaluating the trend of Q factor.

7.1.1 Preliminary considerations

Before starting the mere discussion regarding the characterization, it can be useful to show how the use of a μ -PL setup improves a conventional PL setup. With this purpose it is interesting to look at two PL spectra represented in Figure 7.1, obtained looking to the resonance modulated PL emission on the same set of μ -disk: the graphic on panel (a) was taken with a simple PL setup composed by a milimetric pumping spot and a Photomultiplier in the VIS range positioned behind a 350 mm focal length monochromator. The second spectrum was instead taken using a more complex μ -PL setup, described in section 5.2. The difference between the two spectra are evident even at a first sight: in Figure 7.1 (a) the quality factor is lower due to the smallest resolution of the monochromator and to the fact that we can not be sure that we are looking only at the PL produced inside one single disk. In this case the obtained spectrum is probably the result of the convolution between resonances of different order, with the corresponding maximum very near in wavelength one to the other. The consequence is a lowering of the reported Q associated to a broadening of the $FWHM$.

In any case, in both spectra it is clear the resonance intensity and Q lowering for wavelengths smaller than 675 nm. This fact is certainly associated to the growth of the optical loss of the material in that range, which was already demonstrated by the SES measurements performed inside the active WGs structures.

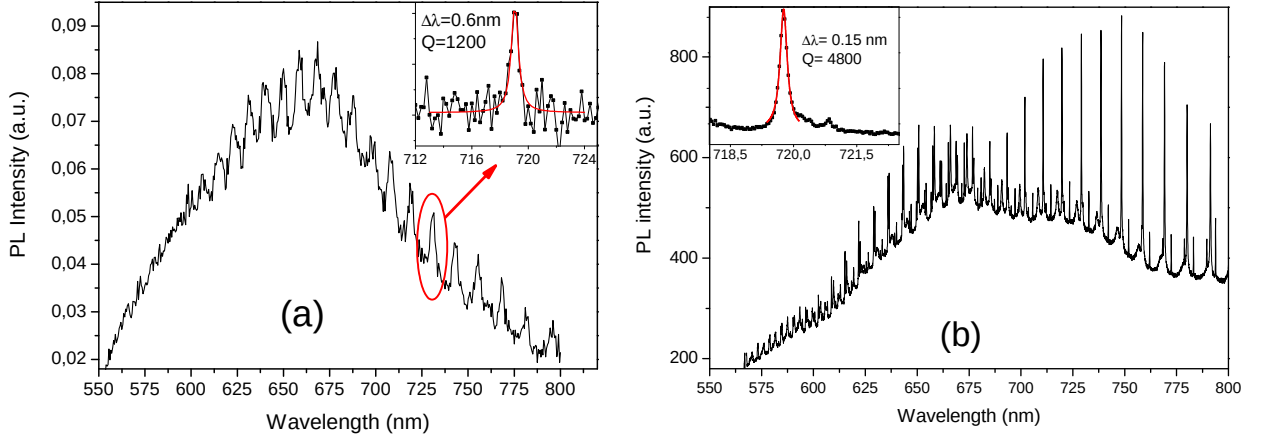


Figure 7.1: WGM spectra of the same sample obtained by means of a standard PL setup (a) and with a more precise μ -PL setup (b).

Depending on the particular experiment we want to perform, with the μ -PL setup gives us the possibility to change between the 600 l/mm and the 1200 l/mm diffraction grating. The first one can provide only a general trend of the Q value, due to its lower resolution and not its exact maximum but can guarantee with high amount of intensity to the CCD. On the other hand the 1200 l/mm grating results to be fundamental in order to perform high resolution measures in some region of interest of the emission spectrum, to the detriment of the light intensity. In Figure 7.2 it is possible to appreciate the resolution difference due to the grating type.

A further advantage coming out from Figure 7.1 (b) is that it is possible to distinguish up to three different families of radial modes, while in panel (a) this operation is impossible. As we can see in Figure 7.3, using a polariser we are also able to isolate the families with vertical polarisation (TM) and horizontal (TR): according to the simulation results we can confirm that the first modal family belonging to the TR polarization is the one that reach the higher value of intensity inside the cavity.

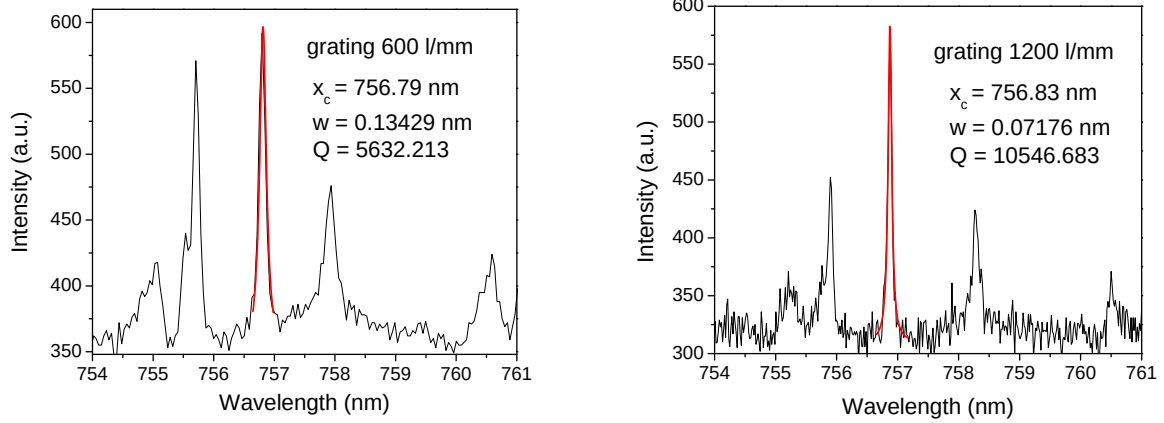


Figure 7.2: Comparison between low resolution spectrum (left panel) and high resolution spectrum (right panel) of a same resonance peak of a $R = 7.5 \mu\text{m}$ disk. The 600 l/mm grating has a maximum resolution of 0.11 nm, leading to a maximum Q factor of 5.63×10^3 while the 1200 l/mm owns a resolution of 0.06 nm corresponding of a Q value of 1.05×10^4 at 760 nm.

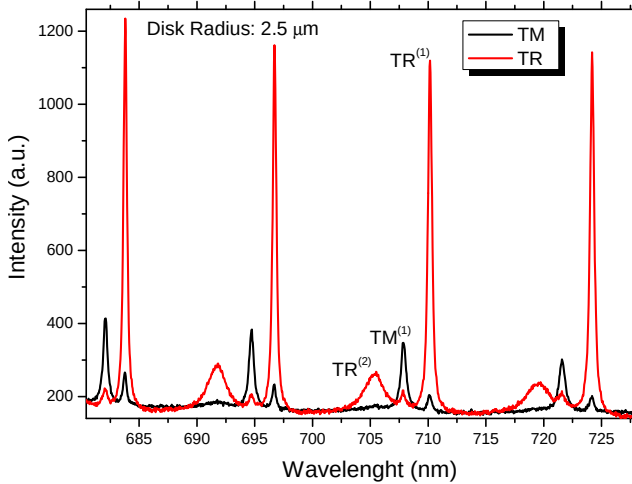


Figure 7.3: PL spectra of TM (black curve) and TR (red curve) polarized WGM in a $2.5 \mu\text{m}$ radius disk. Looking carefully the two spectra we can see how the two polarizations are not completely separated: it is possible to see part of the first order TR (TM) mode superimposed to the TM (TR) polarized spectrum.

Finally, in order to complete this preliminary characterization of the disks, we can compare the effect that a change of the radius of the μ -disk has on the resonances peaks. In this case the devices that we have analysed are 3, 4 and 5 μm radius resonators with vertical etching. According to the results obtained by the simulation we can observe a

diminution of the FSR between consecutive resonances as a function of the increase of disk radius. As we can see in the panel (a) of Figure 7.4 the experimental results (squared points) fit well with the simulated data (continuous lines), and also the experimental group index (panel (b)), calculated through the Equation 2.20, has a good matching with the simulation (dotted lines), with a average variation lower than 0.1.

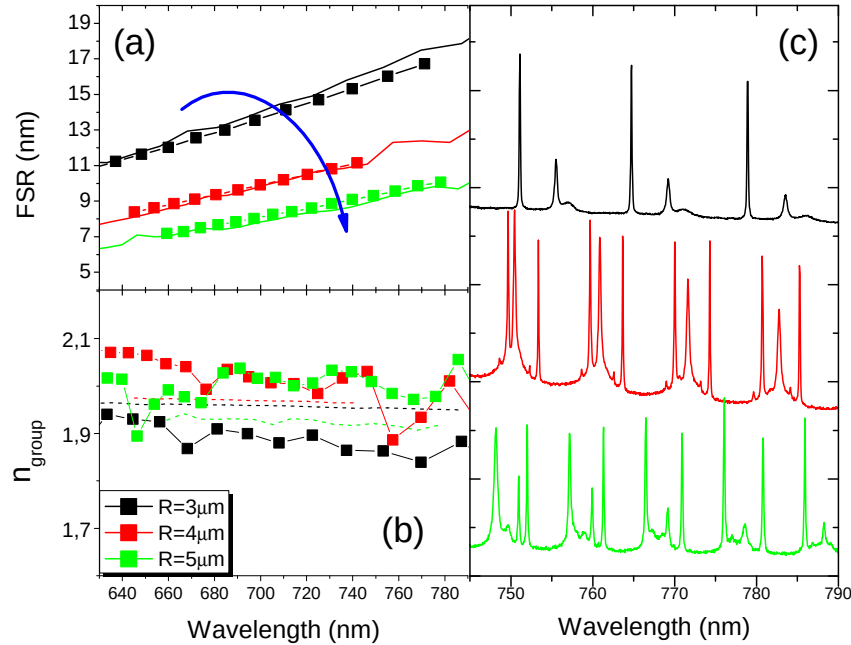


Figure 7.4: Panel (a): comparison between simulated and experimental FSR . Panel (b): experimental and measured group index. Panel (c): WGM spectrum of μ -disks of different radius.

7.1.2 Contributions to the total Q

Starting from the results that we have discussed in Section 6.2 concerning only the PL intensity, we should conclude that the best material will be that of *sample 1* of run 5372_UB. However, as we will see ahead in this chapter, the high losses value of the samples with high content of Si in excess reduces sensibly the Q factor.

We have already mentioned that the overall Q of a circular μ -resonator (Q_{tot}) can be

expressed as the sum of four main independent contributions enclosed in the equation:

$$Q_{tot}^{-1} = Q_{mat}^{-1} + Q_{rad}^{-1} + Q_{ssc}^{-1} + Q_{sa}^{-1} + Q_{shape}^{-1} + Q_{setup}^{-1} \quad (7.1)$$

where the elements of the sum represent respectively:

- Q_{mat} : is the component related to the active material composition and is governed by the absorption and volume scattering losses. We can extract its value from the CBT and SES measurements showed in the previous chapter.
- Q_{rad} : include the theoretical radiation losses of the cavity, and is mostly dependent on the geometrical parameters - i.e. height of the resonator and its radius. The estimation of this contribution comes out from the simulation of a resonator of perfectly circular shape.
- Q_{shape} : represent the maximum achievable Q due to the irregularities of the circular resonator's shape - especially for small radius disks - due to fabrication issues revealed through SEM analysis. In principle Q_{rad} and Q_{shape} could be contained in single contribution, but in this case we want to emphasize the differences between the simulated and real geometry of the cavity.
- Q_{ssc} : takes in account the surface scattering (AFM analysis have revealed small superficial roughness, so this terms must be negligible).
- Q_{sa} : is the surface absorption term.
- Q_{setup} : define the upper limit of measurable Q related to the particular experimental setup.

If we consider, in order to discard geometrical limitation, only well shaped μ -resonators with optimized radius and thickness, several reasons indicate towards the material losses as the limiting factor for the measured Q values, the main one being its clear dependence with the Si excess present in the matrix.

We can get to this conclusion by noticing the direct relationship existing with the inverse of the active material optical losses, graphically represented in Figure 7.5: in panel (a) it

is clear that only for values of main implantation dose inferior to $5 \times 10^{16} \frac{\text{at}}{\text{cm}^2}$ the resonator reach a Q greater than 10^4 at 780 nm, that is the established Q_{limit} value that we want to achieve. In particular, for a $7.5 \mu\text{m}$ radius disk pertaining to *sample 3* we obtain a Q of 1.4×10^4 , that is to the best of our knowledge, the highest Q reported in literature for active planar circular Si based resonators.

The dependence of Q on the Si implantation also results in a huge difference existing between spectra of low and high implantation dose, underlined in panel (b) of the same figure.

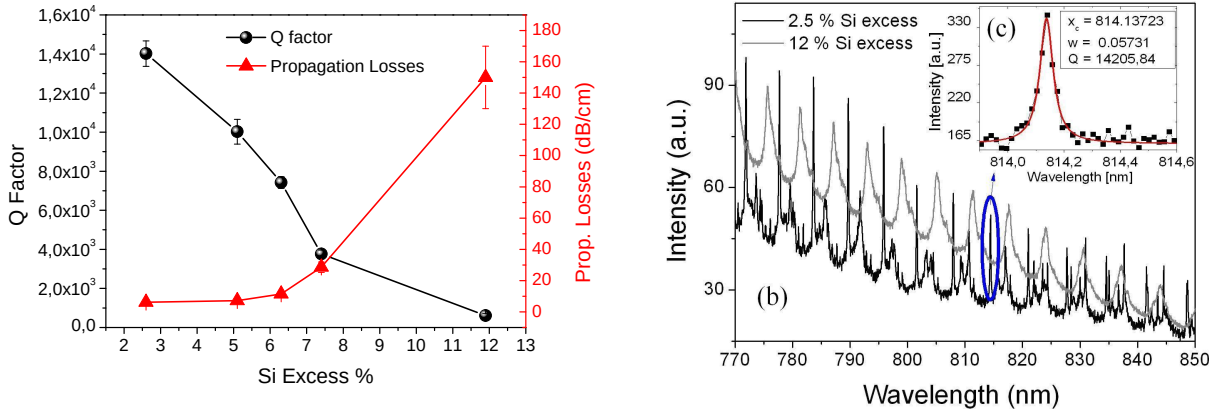


Figure 7.5: (a): Behaviour of Q (black spheres) measured at 760 nm as function of the Si excess for $R = 7.5 \mu\text{m}$. The propagation losses at 780 nm are also reported (red triangles). (b): TR polarised μ -PL spectrum corresponding to disks with $R = 7.5 \mu\text{m}$ of samples 1 (grey) and 4 (black). (c): Zoom of the resonance present at 757 nm for sample 4, together with the corresponding Lorentzian fit.

The measurements reported so far and shown in Figure 7.5 have been performed on a $7.5 \mu\text{m}$ radius disk in order to not to be limited by the Q_{shape} component: indeed, taking in account that all disk have been designed to have $Q_{\text{rad}} > 10^4$, the Q_{shape} seems to play an important role especially for small radius cavities.

For giving support of this hypothesis we can take the results of the measurement of the Q in function of the wavelength for two disks of different radius belonging to the sample with lowest implantation (*sample 3*), reported in Figure 7.6 and 7.7.

In the case of $7.5 \mu\text{m}$ radius cavity there are no limiting factor except for the maximum resolution of the grating inside the monochromator, so the measured Q follows the Q_{setup}

7.1. ISOLATED RESONATORS

slope (red line panel in left panel). On the contrary, in the case of a $3\ \mu\text{m}$ radius disk with irregular shape (right panel) the Q_{measured} lies in on a certain distance (for low λ values even one order of magnitude) below the Q_{setup} , although not being limited from Q_{rad} and Q_{mat} , confirming our hypothesis.

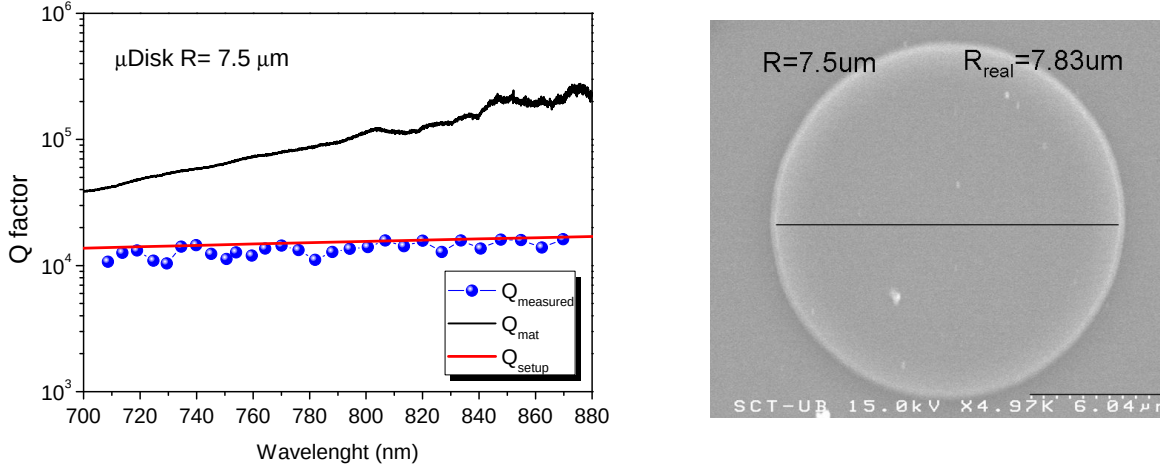


Figure 7.6: Incidence of the Q contributions in a $7.5\ \mu\text{m}$ (left panel) radius disks of Sample 3 run 5372.UB. The SEM analysis reveals a good circular shape, with small variations.

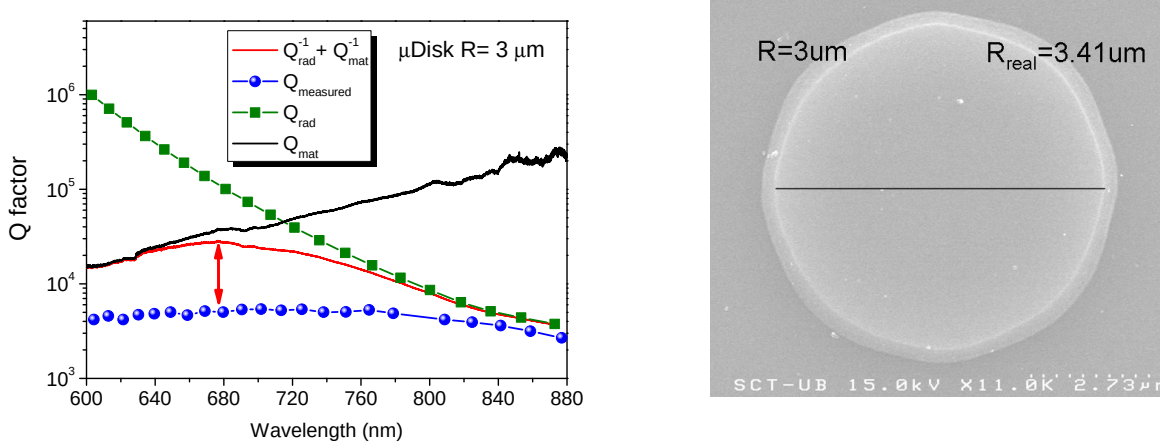


Figure 7.7: Incidence of the Q contributions in a $3\ \mu\text{m}$ (left panel) radius disks of Sample 3 run 5372.UB and SEM image of the same disk (right panel).

All the previous hypothesis were based on the assumption that, for μ -cavities having regular shape, the surface-related contributions to Q should be independent on the Si excess for the same μ -disk radius, and their contribution to the total Q value is low. Indeed the low top-surface roughness measured by AFM measurements performed on various samples, allows us to disregard the contribution of the Q_{ssc} and Q_{sa} in this spectral range.

It is also interesting to underline that measurements performed using a polarizer in the recollection line have revealed that the first WGM of a same family order show higher Q in the TR than in TM polarization (see Figure 7.8). This is an expected result, because of the mayor deconfinement of TM polarization and, we will see in the Section 7.2, this properties will a play an important role inside the coupled structure.

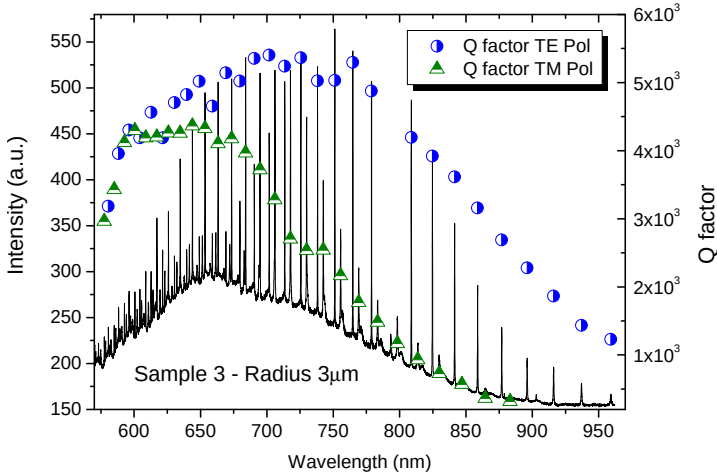


Figure 7.8: *Behaviour of the Q as function of the wavelength for the TE (blue circles) and TM (green triangles) polarizations in a $3\ \mu\text{m}$ radius disk. Such values are overlapped with the unpolarized WGM spectrum of the same cavity.*

7.1.3 Annealing temperature variation

When looking for better comprehension of this behaviour is useful to analyse the left graphic of Figure 7.9. We can divide the six samples in two groups of different temperature (*samples 1-3* / $T = 1100^\circ\text{C}$ and *samples 4-6* / $T = 950^\circ\text{C}$) inside which the dose of Si in excess has been varied from the higher to the lower. Figure 7.9 also shows that in all cases the process of ionic implantation of lower dose (Dose: $5.0 \times 10^{16}\ \text{cm}^{-2}$ / Energy 150 KeV - Dose: $1.9 \times 10^{16}\ \text{cm}^{-2}$ / Energy 90 KeV) results in higher Q values.

For what concerns to the temperature variation it is more difficult to draw clear conclusions even if the higher temperature seems to give the best results.

Taking in account all these results we can conclude that the best samples in terms of Q of the resonances are the wafer samples 3 and 6. It is important to state that this graphic has been realized using the 600 l/mm grating, so we can only extrapolate an approximate dependence of Q from the material, and not its real maximum value.

In the right panel of Figure 7.9 the evolution of FSR is represented in function of wavelength in the case of a disk of radius $R = 3.5 \mu m$. The FSR dependence from the different materials deposited is not sufficiently clear to derive any conclusion: indeed the variations of FSR as a function of the different materials remains inside the error due to the maximum resolution of the grating employed (0.12 nm for 600 l/mm grating). However, the variation is very low, so we can conclude that approximately the refractive index of the material in the range of implantation explored remains constant.

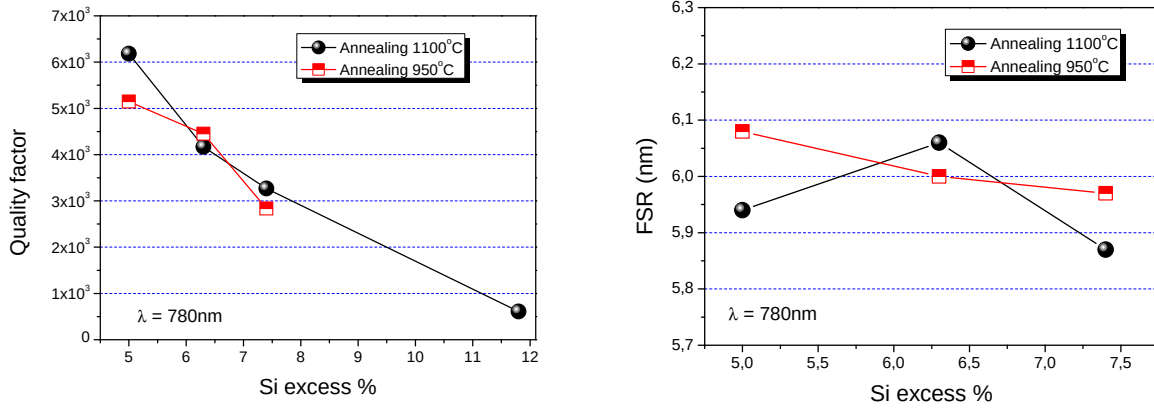


Figure 7.9: Variation of Q (graphic on the left) and of the FSR (graphic on the right) in function of the Si dose and annealing treatment - with the same symbol are headed samples with same Si excess dose but different annealing temperature. Both graphics were made using the 600 l/mm grating.

7.1.4 Power dependence

In order to check if there is a pump flux associated induced losses, which is typical of carrier absorption mechanisms and are not present in the absence of an external excitation

mechanism, we have done experiments at different pump fluxes. Indeed, it has been reported in literature that, in the case of substoichiometric SiO_2 μ -disk systems, carrier absorption losses prevail at high pumping fluxes, which is mainly a consequence of the relatively long lifetime (tens of μs) of the excited carriers (in situations where Auger or stimulated emission processes are not dominant mechanisms). [83] With this goal we have performed high resolution μ -PL measurements on two μ -disk of $R = 4.5 \mu\text{m}$ and $R = 10 \mu\text{m}$ belonging to a sample with 5.3% Si excess, varying the pump flux from 15 to 300 mW for a spot as big as the disk surface. For this reason the power density associated to the two cavity are different, and decrease as the cavity become bigger.

In Figure 7.10 we show the results for a restricted spectral region for different pump densities. First of all we can notice that in these samples only a very small broadening of the WGM resonances can be detected. To quantify the effective broadening of the peaks we have compared the Q factor as a function of the flux density (red spheres in Figure 7.10 panel (b) and (d)), finding that the diminution of Q at high pump values is not remarkable. In order to identify the cause of this behaviour, we have experimentally established an upper limit to the decay lifetime of the PL emission of 200 ns (the temporal resolution of our experimental setup), almost two orders of magnitude faster than in substoichiometric SiO_2 . Authors in [25] report a maximum recombination lifetime of 50 ns. Therefore, the total recombination probability of the carriers generated in this material is so high that carrier absorption effects are not generating measurable losses, so that Q values do not decrease.

Looking with further attention to the panels (a) and (c) it is also possible to note two different competing processes: in the case of small radius disk represented on panel (a) the maximum of the resonance peak is red shifted when increasing the power, while for a larger disk (c) such maximum is subjected to a blue shift.

The red shift can be associated to a dilatation of the cavity due to a thermal effect, and is more evident as the emitted PL intensity saturates (Figure 7.10 (b)).[84] On the other hand, for a bigger cavity (and smaller power density), the former effect is not dominant, and we obtain a blue shift of the resonances due to a lowering of the refractive index inside the disk (Figure 7.10 (c)). Contrarily to the previous case, the applied power density is not sufficient in order to saturate the increasing of the PL. In any case, as already said,

none of the two presented effects contribute to the lowering of the Q factor, that remains approximately constant over a wide range of pumping powers. Other authors reported a similar behaviour also in SRSO free standing cavities, confirm that excited carrier effects and thermal heating are competing effects. [85]

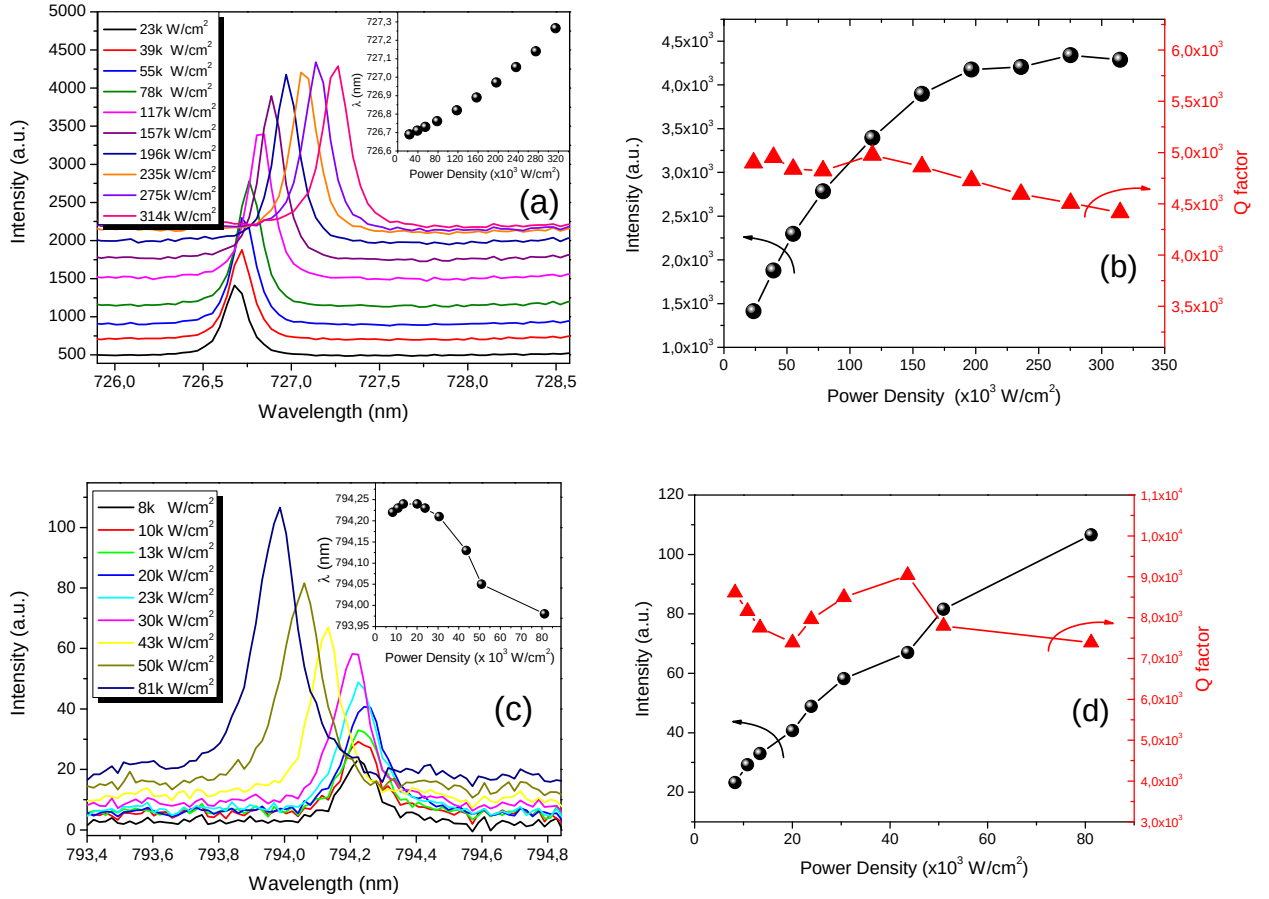


Figure 7.10: Variation of the WGM position and FWHM as function of the power. Panel (a) shows a red shift associated with a deformation of the resonator edge due to a thermal effect, while panel (c) represent the blue shift due to deformation of the resonator. In both panel (b) and (d) we can notice a very small variation of the Q under different pumping powers.

An immediate implication of all these described effects is that it is possible to have high emitted power with high Q , in contrast to what occurs in Si nanoclusters embedded in SiO_2 . [49]

Furthermore we have measured that the power contained by a single resonance in the range between 700 and 770. The main panel of Figure 7.11 reports the quantified μ -PL spectrum (TR polarized) obtained under a 476 nm pumping from a single μ -disk with $R = 4.5 \mu\text{m}$, being the active material of sample 4 (5.3% of Si excess). Whispering gallery mode resonances are clearly observable over an offset PL signal that has the same spectral shape of the PL emission obtained from the bulk material. The latter signal is not coupled to supported modes of the disk. The area of the curve provides a total power emitted on plane of $0.6 \mu\text{W}$ ($0.12 \mu\text{W}$ contained within the resonances), associated with a minimum power efficiency of $\sim 3 \times 10^{-6}$. We have measured an order of magnitude higher efficiency ($\sim 3 \times 10^{-5}$) by decreasing the pumping wavelength to 378 nm, which is a result of the increasing of the excitation cross section of the emitting species. Those efficiency values can be much further improved by optimizing the overlap of the pumping spot shape and the emitting region within the disk that is actually coupled to the supported modes. In fact, the calculated modal volumes for the fundamental modes (few cubed micrometers) are much smaller than the pumped volume (several tens of cubed micrometers).

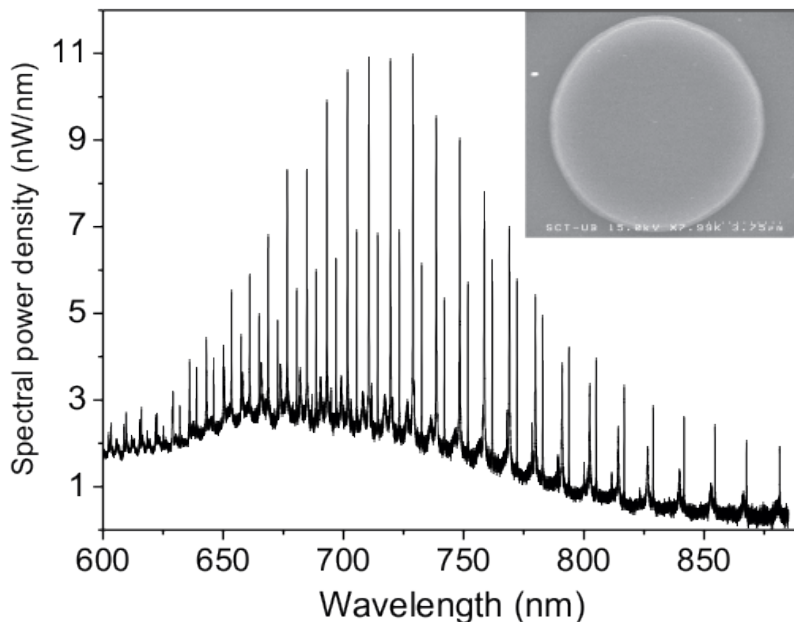


Figure 7.11: (a) Spectral power density of a TR polarized μ -PL emission spectrum for a μ -disk of sample 4 ($R=4.5 \mu\text{m}$). (b) SEM image of the corresponding μ -disk.

The measured power values are well above the minimum sensitivity of the state-of-the-art visible silicon-based photodetectors, which would in principle allow demultiplexing and detecting the emitted signal within the same chip.

7.1.5 Ring resonators

Using the same procedure than that described in the case of the μ -disks, some measurements have been performed on μ -ring resonators with the same radius but different waveguide width. It is important to remind that in this case the word waveguide is referred to the curved WG forming the structure of the ring. The width of the ring's waveguide is the parameter governing the generation of a determined modal family of order superior than one.

As we expected, the more we increase the width the less modal families we obtain: in Figure 7.12 this behaviour is shown for a 10 μm ring with waveguides of 1.5 μm , 2 μm and 3 μm (*(a)*, *(b)* and *(c)* respectively). These properties can be very useful in the realization of the sensor because one of the requested characteristics that the device must have is a clear spectrum with less resonances as possible, in order to facilitate the detection and demultiplexing, while maintaining Q factors above 10^4 .

Unfortunately due to some issue emerged during the fabrication and photolithographic process it was not possible to produce good coupled structures, in order to perform a proof of concept also on the rings. Anyway the μ -disk resonators remain our first choice for the realization of the sensor because of its suitability for the electrical pumping.

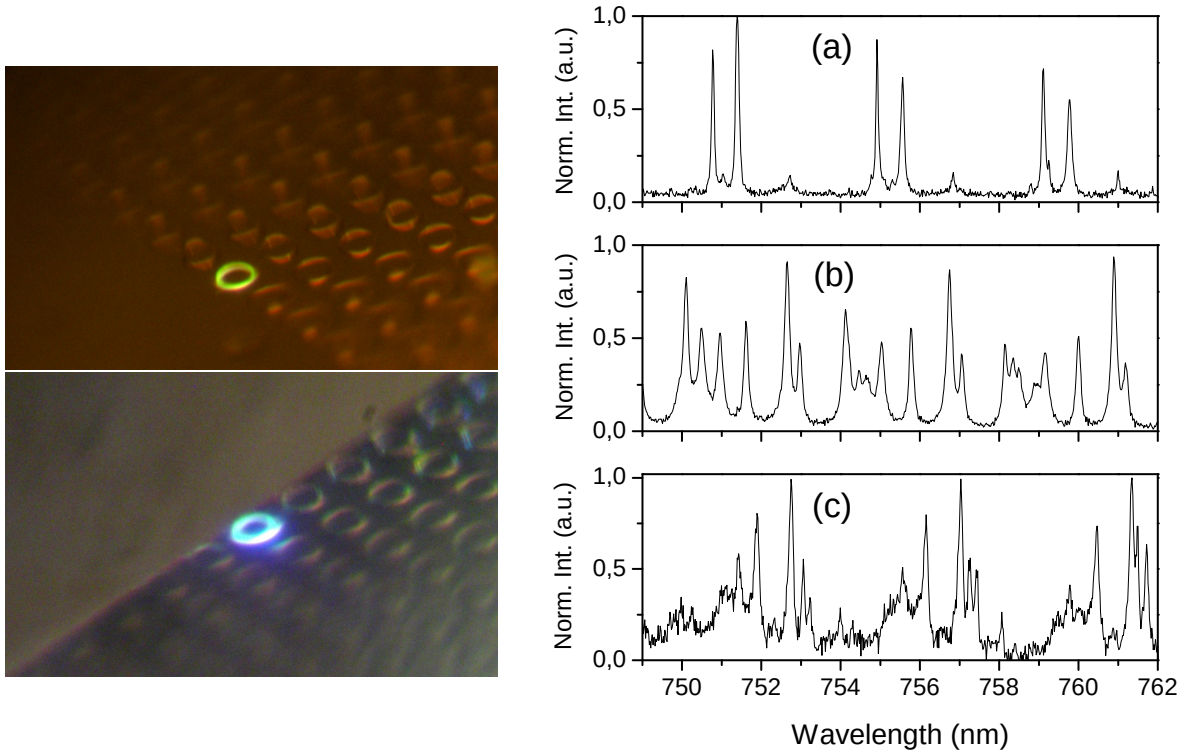


Figure 7.12: Top view of a $10\ \mu\text{m}$ radius ring under $488\ \text{nm}$ (top picture) and $378\ \text{nm}$ (bottom picture) laser pumping. The first picture has been taken putting a $550\ \text{nm}$ filter in front of the camera. On the right panel are represented the spectra of three rings of same radius but different width of the WG forming the circular structure: (a) $1.5\ \mu\text{m}$, (b) $2\ \mu\text{m}$ and (c) $3\ \mu\text{m}$.

7.2 Coupled structure

The last step of this work concerns characterisation of the coupling between the two optimized optical element (WG and μ -disk). In the discussion regarding the realization of the mask in Section 4.1 we stated an upper limit as high as $1\ \mu\text{m}$ for the alignment mismatch between the two structures (depending on the skills of the operator). With that possible mismatch in mind, we designed on the photo-masks a set of 20 coupled structures for a given combination of disk radius and waveguide width, each one with a different ideal lateral displacement ($X\text{-Gap}$). Despite that in this work had the possibility to evaluate the influence on the system only of the $Z\text{-Gap}$.

From the experimental point of view, there are in fact two issues preventing us to perform a detailed characterization of these structures on the basis of its relative *X-Gap*: the first one is that, as said before, the WG-resonator relative position in the *X* axis cannot be determined accurately. The second one is that the mechanical polishing is not optimized, so the profile of the device present a deformation in correspondence to the coupling area, which has been only partially attenuated by a polishing process done on the top of the separation *SiO₂*.

As a consequence of this deformation, the supported *WGM* are displaced more inside the cavity and the maximum *Q* factors that we have found within the set of 20 structures are about $Q \approx 1.4 \times 10^3$, an order of magnitude lower than those obtained on the isolated disks. This result was obtained in devices where the *X-Gap* shows the maximum value outside the resonator, represented in the simulation with a negative sign, and with the smallest WG width, in order to limit the overall variation with respect to the designed structure. We are quite confident that the quality of the technical processing of the coupled structures could be greatly improved, leading to a rise of the experimental *Q* of the device.

However, we are of the opinion that the *Q* value and the intensity of the signal extracted from the coupled WG are already competitive for a proof of concept sensing measurement. As reported in Section 3.3.1 the amount of intensity coming out from the bus WG and the *Q* are strictly connected to the *Z-Gap* value. For this reason samples with different *SiO₂* thickness within the optimum range (from 205 to 310 *nm*) and passive waveguide widths of 1 μm were realized to verify the simulated predictions.

The main results of this study, reported in Figure 7.13 (b), confirms the expected qualitative behaviour as function of the increasing of the *Z-Gap*: to the lowering of the the TM-polarized *PL* intensity measured at the output of the waveguide corresponds a rise of the *Q* factors of the resonances.

Considering that the device with higher *Z-Gap* has the best result in terms of *Q* and that the out coming intensity can be easily measured by the CCD, we have decided to perform the sensoristic proof of concept principally on such device.

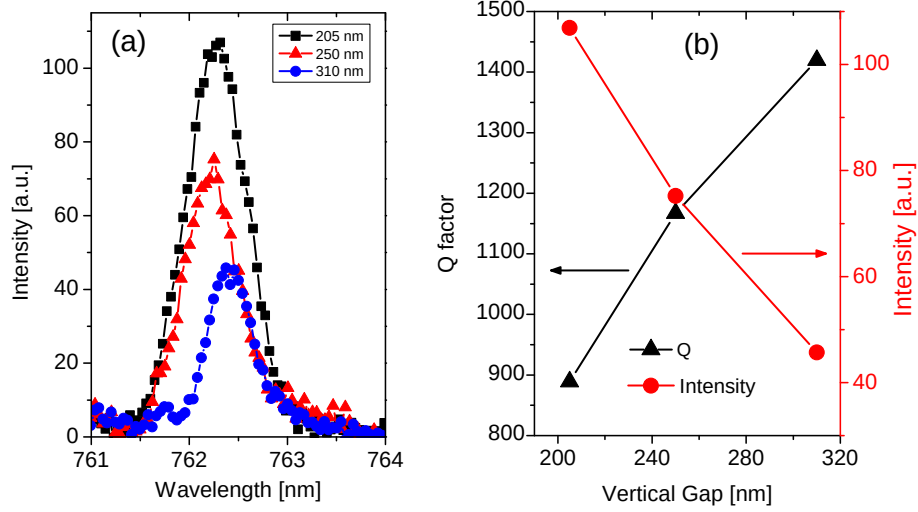


Figure 7.13: (a) TM -polarized μ -PL intensity of a resonance at about 762 nm for different gap distances between the bus waveguide and the μ -disk. (b) Quality factor (black triangles) and μ -PL resonance intensity (red circles) as a function of the vertical gap distance.

In this case only the TM polarization is coupled to the WG : this is probably because the Z -gap may be too high to extract a significant TR polarized signal (TM is less confined on the disk than TR , which improves the evanescent coupling efficiency). As a demonstration of this fact, in Figure 7.14 we report the TR and TM polarized spectra of a $7.5\ \mu\text{m}$ radius disk surrounded by air and coupled with a $1\ \mu\text{m}$ WG , in which it is clear that the TR spectrum do not show appreciable resonances.

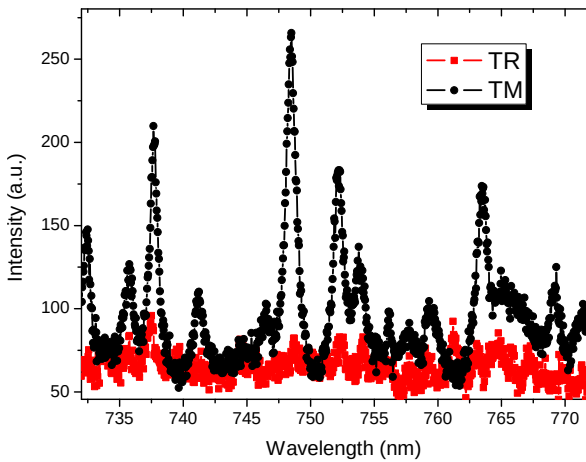


Figure 7.14: Comparison between TR and TM polarized spectra of a $7.5\ \mu\text{m}$ disk coupled with a $1\ \mu\text{m}$ wide WG , placed at a Z -Gap of 355 nm and X -Gap of $-1\ \mu\text{m}$.

7.2.1 Sensitivity of the device

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. In particular we have varied the refractive index of the analyte as a function of the molar fractions of Methanol and Ethanol (x_{Met} and x_{Et} respectively) in a Methanol-Ethanol solution.

The molar refraction R_M of a given substance can be expressed by the Lorentz-Lorenz equation:

$$R_M = \left(\frac{n^2 - 1}{n^2 + 2} \right) \cdot \frac{M}{\rho} \quad \text{MOLAR FRACTION} \quad (7.2)$$

where n is the refractive index of the solution at a certain wavelength, M is the molecular mass and ρ is the density. Those parameters take the following values for Methanol and Ethanol around $\lambda = 750 \text{ nm}$:

	Methanol	Ethanol
R_M	7.93648116	12.8158663
n	1.31604	1.3578
M	32.04	46.07
ρ	0.7918	0.789

Table 7.1: *Main parameters used in the preparation of the solutions.*

When dissolving the two substances, the molar refraction of the resulting solution is additive and becomes:

$$R_{M(Et, Met)} = x_{Met} R_{M(Met)} + x_{Et} R_{M(Et)} \quad (7.3)$$

According to the previous definitions, we have produced the following set of ten solutions with different molar fractions of the two substances:

7.2. COUPLED STRUCTURE

x_{Met}	x_{Et}	n
0.9658254	0.03417465	1.31806
0.9578315	0.0481685	1.31887
0.932111	0.67889	1.32
0.9030093	0.0969907	1.32159
0.8629	0.1371	1.3238
0.8033	0.1967	1.32691
0.715	0.285	1.33126
0.579917	0.420083	1.33735
0.36565	0.63435	1.34587
0	1	1.354587

Table 7.2: Molar fractions of the dissolutions of Methanol and Ethanol. In the third column is reported the resulting refractive index.

This characterization has been performed on a coupled structure with $R = 7.5 \mu m$ and $Z\text{-gap} = 310 \text{ nm}$, which gave the best result in terms of Q .

The results of this measurement are showed in Figure 7.15, in which we can appreciate an overall resonance displacement of $\Delta\lambda = 1.37 \text{ nm}$ as a consequence of a change in refractive index of $\Delta n = 0.038 \text{ RIU}$.

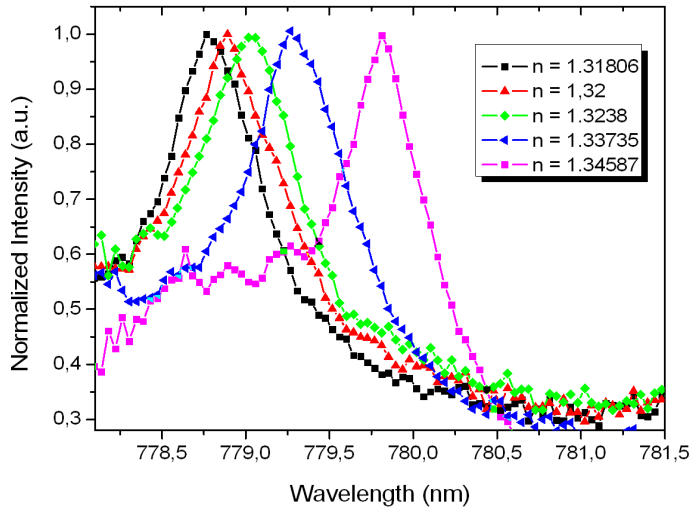


Figure 7.15: Spectral displacement of a resonance measured by pouring a drop of liquids with different refractive index on a $7.5 \mu m$ radius disk for five analytes with different n value.

From the slope of the linear fit, realized taking in account all the points of the experimental results (black line in Figure 7.16), we can extract the maximum sensitivity of our

device, defined as the resonance wavelength shift for RIU , so that:

$$S = \Delta\lambda/\Delta n = 36.52nm/RIU \quad \text{SENSITIVITY OF THE DEVICE} \quad (7.4)$$

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. [11] [23]

It is worth to note that for analytes with higher Ethanol concentration (with molar fraction superior than $x_{Et} = 0.1967$ in Figure 7.16) the experimental data deviate from the theoretical prediction. This is probably a consequence of the fact that the characterisation associated to each analyte is done in sequence after the previous analyte evaporation and without cleaning the device between two consecutive measurements. We have proceeded in this way in order to preserve the alignment conditions. The Methanol used to make the solutions has a purity percentage of 99.9%, while the Ethanol presents a value of 99.8%, thus presenting a percentage of other volatile substances (e.g. Water, Acetone, Acetaldehyde or Isopropyl) and non volatile elements as metals. We believe that, due to the higher impurity percentage in high ethanol concentrations solutions, a deposition of a non volatile residual over the sample occurs, creating a thin layer over and aside the disk, slightly reducing the sensitivity of the system. The six points fit give in fact a value of sensitivity of $S = 51.79 \text{ nm}/RIU$ (Blue line in Figure 7.16) that is much closer to the simulated behaviour. (Black line in Figure 7.16)

It is also important to establish a definition for the sensor DL , in terms of minimum measurable refractive index variation (Δn_{min}).

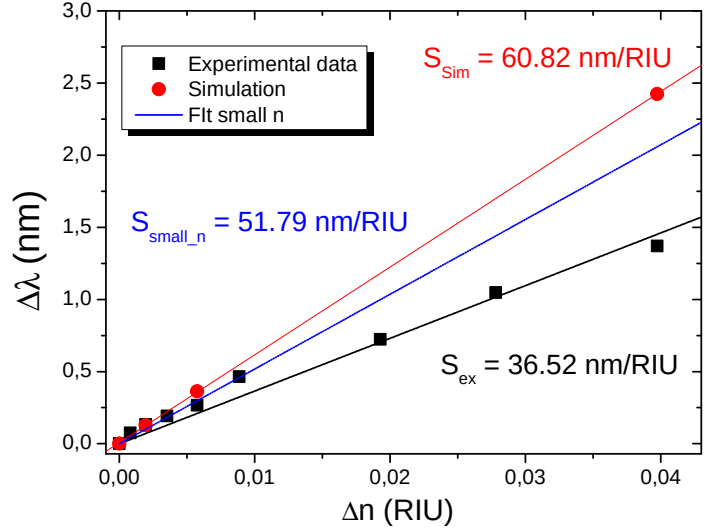
In [86], this limit is defined as the subjective ability to distinguish a displacement of a single resonance.

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 (Figure 7.16). 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 $\delta\lambda = 0.06 \text{ nm}$

shift, so that the detection limit of our measurement is:

$$DL = \Delta n_{min} = \frac{\delta\lambda}{S} = 1.15 \times 10^{-3} RIU \quad \text{DETECTION LIMIT} \quad (7.5)$$

Figure 7.16: *Linear behavior of the $\Delta\lambda$ as a function of Δn for all the analytes prepared. The slope of the linear fit obtained by fitting all the data is 36.52 nm/RIU (Black line), while for small refractive index changes the sensitivity value is of 51.79 nm/RIU (Blue line). The Red line represent the experimental sensitivity obtained for the same n variation.*



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 also useful to 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. $DL_{FWHM} = 1.7 \times 10^{-2}$. The described results are slightly lower than that reported the state of the art CMOS compatibles devices [23], but obtained with the previously described advantages given by the active $SRSN$ material.

It is worth to note that the reported S value, and consequently the DL, 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 μ -resonator/waveguide vertically coupled system on a silicon chip demonstrating Q factors above 2×10^4 on the IR region.

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.

7.2.2 Behaviour in the IR region

The last result that we present in order to complete the study performed on the coupled structure, concerns the measurements that we have made in a transmission configuration, using a tunable laser in the IR region ($1480 - 1560 \text{ nm}$). The results of such study have been than compared with that obtained by the full $3D$ simulation, and are summarized in Figure 7.17.

First of all the low Q is due to the not optimized thickness of the disk and WG for the IR region, according to the results of the simulation.

The second result is that the position of the peaks measured experimentally is quite coincident with that of the simulation. This is an interesting results when thinking to a possible implantation of the samples with rare earth material (e.g. Erbium ions) emitting in the IR region.

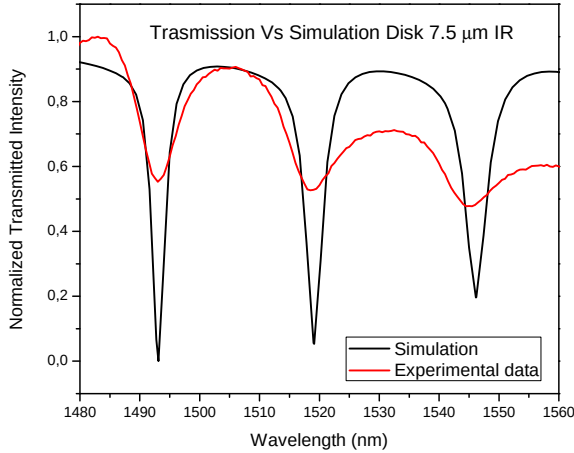


Figure 7.17: *Simulated (black line) and experimental (red line) spectra of a $7.5 \mu\text{m}$ disk bottom coupled with a $1 \mu\text{m}$ wide WG.*

Chapter 8

Conclusions and future developments

8.1 Conclusions

In this thesis we have presented a thorough study on the optical and sensoristic properties of an active SRSN μ -disks-based sensor, in which the light source is directly integrated in the device. The characterization has been performed in both isolated optical resonator and when coupled to a passive Si_3N_4 waveguide placed underneath. The whole structure has been initially simulated in order to study the behaviour of the supported WGM when subject to a geometrical variation (i.e.: radius, thickness, shape of the edge of the isolated μ -disk): the obtained results granted the realization of μ -resonators with Q factor exceeding 10^4 , that is the necessary condition in order to reach a DL of the same order of that of the literature - $DL = 10^3$.

The coupled structure has been then simulated with the main goal of maximizing the WGM intensity transmitted at the end of the WG. This task has been fulfilled through a careful optimization of the geometrical parameters (i.e.: X -Gap and Z -Gap). Simulations performed on the coupled structure revealed also a Sensitivity of the sensor of 60.82 nm/RIU for a 7.5 μm radius cavity.

The fabrication process of the sample has been carried out using standard CMOS compatible process. The deposition and the implantation of the Si_3N_4 has been performed by means of LPCVD technique, while for the SiO_2 deposition the PECVD.

On the produced samples we have performed a superficial analysis (SEM, AFM) revealing the presence of some geometrical imperfections for μ -cavities with radius inferior than $3\ \mu m$, and the presence of a small superficial roughness (average value lower than $1\ nm$). Furthermore, the EFTEM analysis revealed the absence of Si crystalline nanostructure inside the active layer.

Using the Cut-back technique, low losses under $1\ dB/cm$ have been found in both VIS and IR spectral range in the passive WG of different width. On the other hand, applying the SES technique on an Si-rich WG structure we have been able to extract the losses value of active material in a wide and continuous range of wavelength, defined inside the PL spectrum.

As a result of a careful optimization of the active SRSN 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 for cavities with radius exceeding $7.5\ \mu m$. 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. Furthermore the estimation of the total on plane emitted power brought to values up to few nW on a single resonance. This is a very important result because such value of power and the VIS PL emission of the SRSN allow demultiplexing and detecting the emitted signal within the same chip by means of Si-based detectors.

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.

Through a proof of concept, we have demonstrated that these structures are very sensible to the surrounding material and are able to detect refractive index changes with sensitivities of $51.79\ nm/RIU$ and minimum measured refractive index change of $1.15 \times 10^{-3}\ RIU$.

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 demultiplexing and detection can be integrated on the same chip.

8.2 Future developements

Since it is possible to merge photonics and electronics in the same chip, we foresee this new technology as the key to low cost advanced sensor systems showing high sensitivity and extremely small footprint, allowing very small quantities of analyte to be measured. The successive step towards the realization of the integrated sensor is represented by the introduction of a the microfluidic channel on the top of the μ -disk, as represented in Figure 8.1. Microfluidic channels can be readily integrated with mature polymer technology, promising features for lab-on-a-chip applications. The transparency of the polymeric material constituting the channel still allow the optical pumping (performed using a laser or a UV LED) while giving the possibility of a continuous flowing of the analyse on the cavity. This characteristic is fundamental in order to avoid the problems that we have noticed due to the accumulation analyte residual, and will permit the estimation of the variation of the refractive index in a continuous range of time.

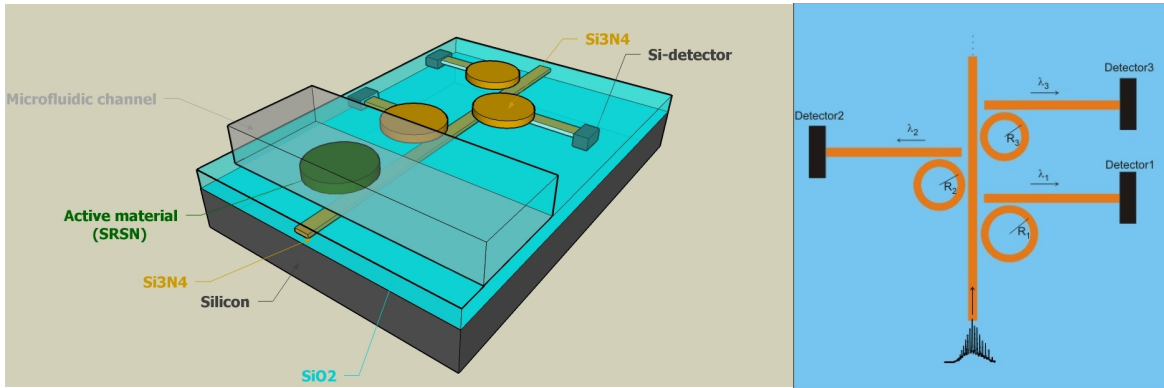


Figure 8.1: Representation of some possible improvements on the μ -disk-based sensor, as the microfluidic channel (in light blue over the disk) and the demultiplexing circuitry: in the left panel a bottom coupling scheme is represented, while in the right panel the μ -rings are horizontally coupled with the bus WG.

The demultiplexing of the obtained PL signal is another important issue that has to be solved. The vertical coupling scheme of the proposed device does permit the realization of a passive photonic circuitry placed in the oxide layer or in the same plane of the cavity, allowing the extraction of the light generated within the resonator. Many possible

schemes of demultiplexing are compatibles with our structure: the first one foresee the routing of signals of different wavelengths to different photodetectors (Si detectors for visible light, SiGe for IR) by means of passive circular resonators (represented in yellow in figure 8.1). The μ disk-ring could be coupled with the WG in both bottom or lateral coupling configurations. [87].

The demultiplexing of the signal can be also achieved using COMS compatible Photonics crystals super prisms, [88]-[89] or the Arrayed Waveguide Gratings (AWGs).[90]

The possibility of an electrical pumping of the active material is another interesting objective that can be explored in the future. The most important issue that has to be overcome is the excessive thickness of the active material in order to obtain an acceptable (one possible solution is the use multilayered active material as component of the resonator).

Appendix A

Light Propagation in 2-Dimensional Waveguide

An optical WG represents the fundamental elements providing the connection between the various optical devices and components. More specifically, in our case rib WG have been used in order to transport up to the detection system the light generated inside the active resonator.

A WG can be described as a light channel guiding EM waves by total internal reflection. Several kind of optical WGs are currently used in Integrated Optics, generally consisting of a slab, strip, or cylinder (see Figure A.1 (a), (b) and (c) respectively) made of high refractive index dielectric material (n_H), surrounded by a low refractive index (n_L) dielectric material. For simplicity in this section we will take in account a symmetric planar dielectric WG in which the incident rays form an angle $\hat{\theta}$ with the z axis in the y - z plane. In this chapter we will associate to each optical ray flowing inside the WG a transverse electromagnetic (TEM) plane wave. As consequence, the total *EM* field flowing inside the WG can be written as the sum all of these plane waves.

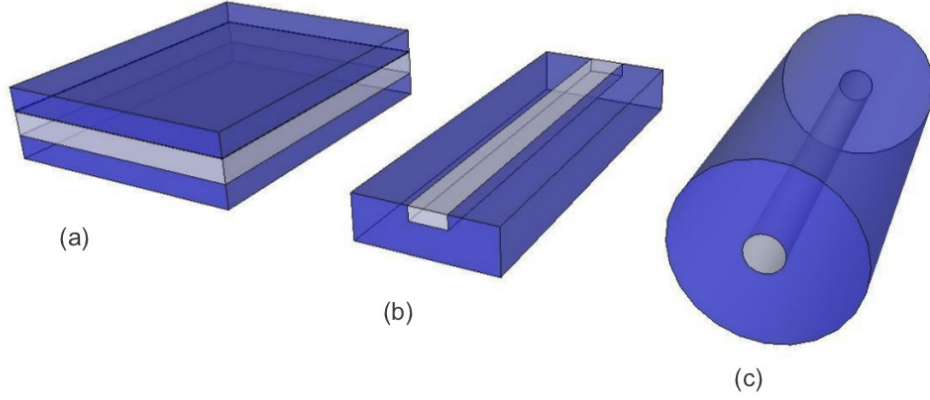


Figure A.1: *Some of the most common optical waveguide configurations: (a) slab WG, (b) strip WG and (c) fiber WG*

A.1 Critical angle condition

At this stage, let's consider a monochromatic *TEM* plane wave, having wavelength $\lambda = \frac{\lambda_0}{n_H}$ (λ_0 defines the wavelength of the plane wave in the free space), in a (x, y, z) Cartesian coordinate system and traveling in the previously defined symmetrical WG structure along the z direction (see Figure A.2). Its phase velocity is defined as $c = \frac{c_0}{n_H}$ (being c_0 the speed of the light traveling in the free space) and the wavenumber as $k = n_H k_0$ (with wave-vector components $k_x = 0, k_y = n_H k_0 \sin \hat{\theta}$ and $k_z = n_H k_0 \cos \hat{\theta}$).

The critical angle ($\hat{\theta}_c$), for which the condition of internal reflection is accomplished, can be defined starting from the Snell's law:

$$n_H \sin \hat{\theta}_H = n_L \sin \hat{\theta}_L \quad \text{SNELL'S LAW} \quad (\text{A.1})$$

and finding the angle $\hat{\theta}_H = \hat{\theta}_c$ for which $\hat{\theta}_L$ is equal to $\pi/2$. the equation:

$$\hat{\theta}_c = \arcsin \left(\frac{n_L}{n_H} \right) \quad \text{CRITICAL ANGLE} \quad (\text{A.2})$$

As shown in Figure A.2, if $\hat{\theta}_H$ is smaller than the complement of the critical angle $\bar{\theta}_c$ the light is then transported through the n_H medium by total internal reflection at the

boundaries without radiating into the surrounding medium.

$$\bar{\theta}_c = \frac{\pi}{2} - \arcsin\left(\frac{n_L}{n_H}\right) = \arccos\left(\frac{n_L}{n_H}\right) \quad \text{TOTAL REFLECTION} \quad (\text{A.3})$$

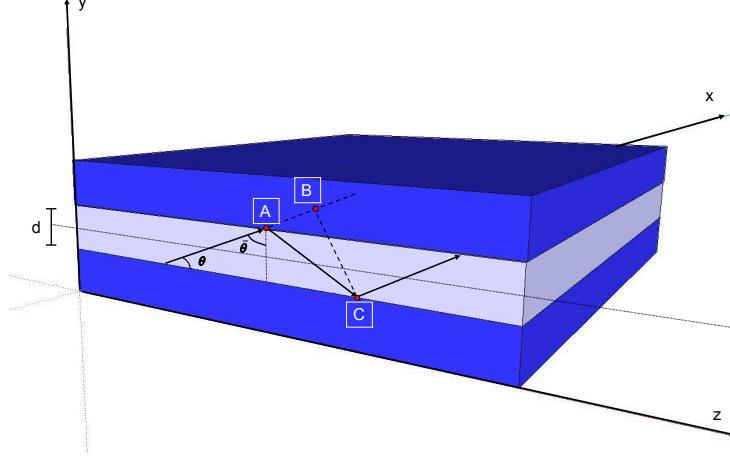


Figure A.2: Scheme of the self-consistency condition for a plane wave traveling in the z direction with an incident angle $\hat{\theta}$.

A.2 Self-consistency condition

We now impose a self-consistency condition requiring that a wave reproduces itself after a double reflection process. All the EM fields satisfying this condition are *eigenmodes* (or more simply *modes*) of the waveguide. Therefore the modes are fields that maintain the same transverse distribution and polarization at all distances along the z axis.

For values of θ satisfying the self-consistency condition, the resulting plane wave after two reflection is shifted respect to the original wave by a value given by the equation A.4 (see Figure A.2).

$$\overline{AC} - \overline{AB} = 2d\sin\hat{\theta} \quad (\text{A.4})$$

Furthermore a phase term ϕ_r must be taken into account, that is introduced by each internal reflection at the dielectric boundary. As result of self-consistency, the phase shift between the two waves must be zero or a multiple of 2π , so that:

$$2d\frac{2\pi}{\lambda}\sin\hat{\theta} - 2\phi_r = 2\pi m \quad \text{SELF-CONSISTENCY} \quad (\text{A.5})$$

with m positive integer. The same equation can also be written as $2k_y d - 2\phi_r = 2\pi m$ introducing in the Equation A.5 the wavevector.

The reflection phase shift ϕ_r is a function of the angle $\hat{\theta}$ and depends also on the polarization of the incident wave. The *EM* field is named as TE polarized if the Electric field lies in the x direction, while we define a TM polarized field when the Magnetic field is in the x direction. In the case of TE polarization the total internal reflection phase shift is given by the equation:

$$\tan\frac{\phi_r}{2} = \sqrt{\left(\frac{\sin^2\bar{\theta}_c}{\sin^2\hat{\theta}} - 1\right)} \quad (\text{A.6})$$

in which ϕ_r varies between π and 0 while $\hat{\theta}$ varies from 0 to $\bar{\theta}_c$. Using Equation A.5 and A.6 we can obtain:

$$\tan\left(\pi\frac{d}{\lambda}\sin\hat{\theta} - m\frac{\pi}{2}\right) = \sqrt{\left(\frac{\sin^2\bar{\theta}_c}{\sin^2\hat{\theta}} - 1\right)} \quad (\text{A.7})$$

This latter equation represent the *self-consistency condition for TE modes*, and is a transcendental equation in one variable ($\sin\theta$). By solving Equation A.7 we can find the reflection angle $\hat{\theta}_m$ of a determinate mode m . Indeed all the $\hat{\theta}_m$ angles lie between 0 and $\bar{\theta}_c$ and they correspond to wave-vectors with components $(0, n_1 k_0 \sin\hat{\theta}_m, n_1 k_0 \cos\hat{\theta}_m)$. The z components of this wave-vector represent the propagation constant β_m .

Using the same reasoning it is possible to find the reflection angles θ_m and propagation constants β_m for the TM modes.

Moreover the number of the modes supported (M) by the WG is proportional to its numerical aperture ($NA = \sqrt{n_H^2 - n_L^2}$), that is the angle of acceptance of rays coming from

air into the WG. So for TE polarization M is represented by:

$$M = 2 \frac{d}{\lambda} \cdot NA \quad \text{SUPPORTED MODES} \quad (\text{A.8})$$

where d is the height of the slab with higher refractive index.

The description that we have given so far deals with the propagation inside a planar WG, while in this thesis we will see that the light flux is normally guided in rectangular WG: respect to the previous case, in which the light is confined only in the y direction, we have to extend the self-consistency conditions of Equation A.5 introducing the confinement in the x direction. In the case of a squared WG, in which the facets are equal to d the self consistency conditions are given by:

$$\begin{cases} 2k_x d = 2\pi \cdot m_x, & m_x = 1, 2, \dots \\ 2k_y d = 2\pi \cdot m_y, & m_y = 1, 2, \dots \end{cases} \quad (\text{A.9})$$

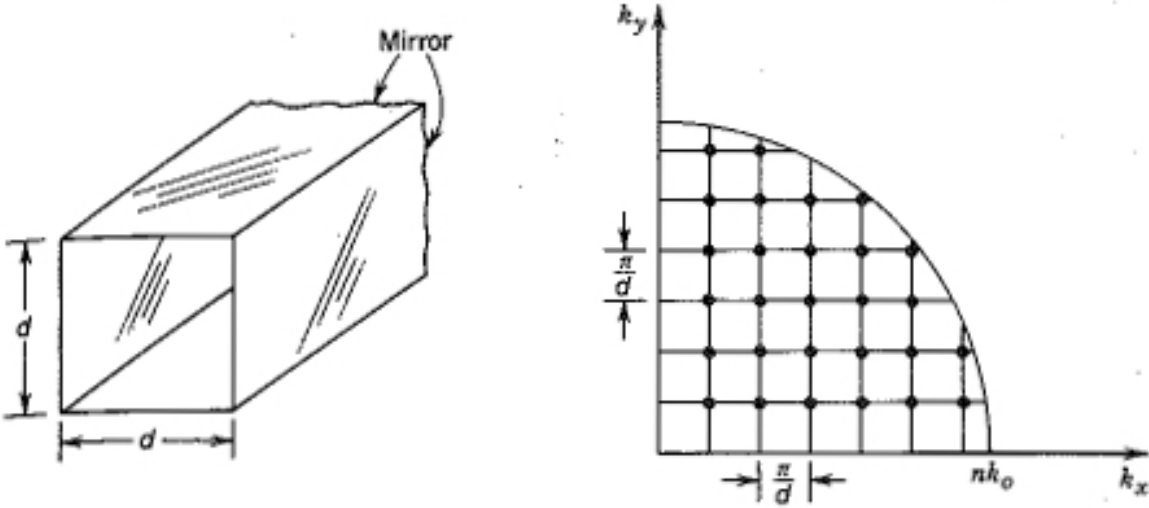


Figure A.3: *Number of modes of a bi dimensional WG. (Picture taken from [60])*

The number of modes M can be determined by counting the number of nodes inside a

quarter circle of radius nk_0 in the k_x - k_y diagram showed in Figure A.3. If this number is large, it may be approximated by the ratio of the area $\pi(nk_0)^2/4$ of a unit cell $(\pi/d)^2$ resulting in the equation:

$$M \approx \frac{\pi}{4} \cdot \left(\frac{2d}{\lambda} \right)^2 \quad (\text{A.10})$$

Since there are two polarizations for each mode, the total number of modes is given by $2M$. According to what stated before for the unidimensional WG, if the material is a dielectric we have to take in account the NA so that :

$$M \approx \frac{\pi}{4} \cdot \left(\frac{2d}{\lambda} \right)^2 \cdot NA^2 \quad (\text{A.11})$$

Appendix B

Simulation of the WGM spectrum

B.1 FDTD simulation of the coupled structure

Hereafter we will show an example of 3D FDTD simulation coupled structure, implemented by means of the MEEP simulation tool.

In this case a $3\ \mu m$ radius disk is coupled with a $1\ \mu m$ wide WG at a Z-Gap distance of $0.25\ \mu m$.

Definition of the material parameters

(define-param n 2.0)	<i>Refractive index of disk</i>
(define-param enne 1.31806)	<i>Refractive index of analyte</i>
(define-param s 1.45)	<i>Refractive index of substrate</i>
(define-param h 0.3)	<i>Height of disk</i>
(define-param r 3)	<i>Radius of the disk</i>
(define-param b 0.2)	<i>Height of waveguide</i>
(define-param a 1)	<i>Width of waveguide</i>
(define-param l 8)	<i>Waveguide length</i>
(define-param d 0.25)	<i>Z-Gap</i>
(define-param e 5)	<i>Width of substrate</i>

<code>(define-param g 0)</code>	<i>X-Gap</i>
<code>(define-param pad 0.5)</code>	<i>Padding between waveguide and edge of PML</i>
<code>(define-param dpml 0.5)</code>	<i>Thickness of PML</i>

Definition of the geometrical parameters

<code>(define sx (+ (* 2 r) 1 pad dpml))</code>	<i>Cell size x</i>
<code>(define sy (+ 1 2 pad dpml))</code>	<i>Cell size y</i>
<code>(define sz (+ (* 2 h) pad dpml))</code>	<i>Cell size z</i>
<code>(define cd (/ h 2))</code>	<i>Center of the disk in z</i>
<code>(define ct (* -1 (+ (/ b 2) d)))</code>	<i>Center of the waveguide in z</i>
<code>(define lt r)</code>	<i>Center of the waveguide in x</i>

<code>(set! geometry-lattice (make lattice (size sx sy sz)))</code>	<i>Geometry of the system</i>
--	-------------------------------

Definition of the optical structures

<code>(set! geometry (list</code>	
<code>(make block (center 0 0 0) (size infinity infinity infinity)</code>	
<code>(material (make dielectric (index enne))))</code>	
<code>(make block (center 0 0 (* (/ e 2) -1)) (size infinity infinity e)</code>	
<code>(material (make dielectric (index s))))</code>	<i>Substrate</i>
<code>(make cylinder (center 0 0 cd) (height h)</code>	
<code>(radius r) (material (make dielectric (index n))))</code>	<i>μ-Disk</i>
<code>(make block (center (+ lt (- 0.3 g)) 0 ct) (size a infinity b)</code>	
<code>(material (make dielectric (index n)))))</code>	<i>Waveguide</i>

```
(set! pml-layers (list (make pml (thickness dpml))))
(set-param! resolution 30)
```

Definition of the source

(define-param fcen 1.33)	<i>Frequency centre of the pulse</i>
(define-param df 0.4)	<i>Pulse width (in frequency)</i>
(define-param nfreq 2000)	<i>Number of frequencies at which to compute flux</i>

```
(set! sources (list
  (make source
    (src (make gaussian-src (frequency fcen) (fwidth df)))
    (component Ex) (center (* (- r cd) -1) 0 cd) (size (+ h 0.1) 0 (+ h 0.01) ) )))
```

Placing the detectors inside the structure

```
(define trans
  (add-flux fcen df nfreq
    (make flux-region
      (center lt -3.5 ct) (size (- a 0.01) 0 (- b 0.01)) ) )))
```

Running the simulation

```
(run-sources+ 2500
  (at-beginning output-epsilon)
  (at-time 1 output-efield-x)
  (at-every 100 output-efield-z)
  (at-time 200 output-efield-z))
```

```
(after-sources (harminv Ex (vector3 (+ (* r -1) 0.1) 0 cd) fcen df))
(at-end output-efield-z))
```

Display all the detected signals

```
(display-fluxes trans) Print out the flux spectrum
```

Graphical representation of the modal structure

Once obtained the position and the intensity of the supported WGM, the task is to represent them using a Lorentzian fit. The centre and the FWHM of each mode are defined using the results of the Harminv tool. Using a basic bash scripting we than overlap all the resonances.

B.2 Bash script for the FOR LOOP iteration

This simple bash script permits to run multiples MEEP simulations changing the parameters previously defined. In this case we want to vary the radius r of the disk between 3 and 10 μm , the refractive index n of the active material between 2 and 2.1 and the modal number m between 55 and 95.

```
for ((t=3; t<=10; t=t+1))
do
  for ((j=200; j<=210; j=j+10))
  do
    for((i=55; i<=95; i=i+1))
    do

      k='echo "scale=2 ; $j/100" —bc -l '
      GUILE_WARN_DEPRECATED = "no" mpirun -np 2 meep-mpi r = $t m = $i
n = $k $NOMCTL.ctl > output
```

B.2. BASH SCRIPT FOR THE FOR LOOP ITERATION

```
grep -E 'command-line ||harminv' output >> modi-r$t-n$k.out
grep -Ev 'command-line ||frequency' modi-r$t-n$k.out >> out-r$t-m$i-n$k.dat
cat out-r$t-m$i-n$k.dat >> out-r$t-n$k.dat
rm -f out-r$t-m$i-n$k.dat
done
sed 's/,/ /g' out-r$t-n$k.dat > temp1
echo "(" > temp2
awk -f /home/one.awk temp1 >> temp2
awk -f /home/two.awk temp2 > temp1
sed 's/.$//' temp1 > temp2
sed 's/.$//' temp2 > temp1
sed 's/.$//' temp1 > temp2
rm -f temp1
rm -f temp2
echo "Building result-h$p-n$k.txt containing data points.."
cp out-r$t-n$k.dat data.txt
perl /home/scriptperl.pl > spettro-r$t-n$k.txt
rm -f data.txt
rm -f output
rm -f result.txt
rm -f out-r$t-n$k.dat
done
done
```


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Curriculum Vitae

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Conference contributions

- 1) Poster presentation: J.M. Ramírez, F. Ferrarese Lupi, Y. Berencén, D. Navarro-Urrios, L. López-Conesa, J.M. Rebled, S. Estradé, F. Peiró, O. Jambois, A. Tenggatini, A. Anopchenko, N. Prtljaga, L. Pavesi, J-M. Fedeli, B. Garrido; “*Experimental*

evidences of energy transfer between Si nanoclusters and Er^{3+} ions under electrical pumping"; Congress: E-MRS Spring Meeting 2012 (Strasbourg, France, May 14-18, 2012)

- 2) Poster presentation: L. López-Conesa, J.M. Rebled, S. Estradé, Y. Berencén, F. Ferrarese Lupi, J.M. Ramírez, Blas Garrido, J.-M. Fedeli, and F. Peiró; "*Site specific (EF)TEM characterization of Er^{3+} implanted silicon nanophotonic and nanoelectronic devices*", Congress: E-MRS spring meeting 2012, (Strasbourg, France, May 2012)
- 3) Oral presentation: F. Ferrarese Lupi, D. Navarro-Urrios, J. Rubio-Garcia, J. Monserrat, C. Dominguez, P. Pellegrino, B. Garrido; "*Visible light emitting Si rich Si_3N_4 μ -disk resonators for sensoristic applications*", Congress: Silicon Photonics and Photonic Integrated Circuits conference in SPIE Photonics Europe, (Brussels, Belgium, April 2012)
- 4) Poster presentation: J. M. Ramírez, F. Ferrarese Lupi, Y. Berencén, D. Navarro-Urrios, O. Jambois, L. López-Conesa, J.M. Rebled, S. Estradé, F. Peiró, A. Tengattini, N. Prtljaga, A. Anopchenko, L. Pavesi, J.M. Fedeli, B. Garrido; "*Silicon-based light sources: Er route for the first injected Silicon laser emitting at 1.54 μ m*", Congress: 16th European Conference on Integrated Optics and Technical Exhibition - ECIO 2012, (Sitges, Barcelona, April 2012)
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 - 8) Poster presentation: F. Ferrarese Lupi, D. Navarro-Urrios, J. Monserrat, C. Dominguez, P. Pellegrino, B. Garrido; “*High Q optically active Si rich Si_3N_4 μ -cavities*”, Congress: III Jornada IN2UB, (Barcelona, Spain, 2010)
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 - 10) Poster presentation: D. Navarro-Urrios, A. Pitanti, F. Ferrarese Lupi, O. Jambois, N. Daldosso, L. Pavesi, B. Garrido; “*Si nanoclusters coupled to Er^{3+} ions in a SiO_2 matrix for optical amplification*”, Congress: 1st Conferencia Española de Nanofotonica - CEN2010, (Segovia, Spain, 2010)
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 - 12) Oral presentation: D. Navarro-Urrios, Y. Lebour, F. Ferrarese Lupi, O. Jambois, B. Garrido, A. Pitanti, N. Daldosso, F. Gourbilleau, R. Rizk and L. Pavesi; “*Quantification of the optically active Er^{3+} content and its direct excitation cross section in Er^{3+} doped SiO_2 sensitized by Si nanoclusters*”; Impurity Based Electroluminescent Devices and Materials (IBEDM), September 30th - October 3rd 2009, Tossa de Mar, Barcelona, Spain.
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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 a building block for detection systems with increased complexity, in which demultiplexing and detection could be readily integrated on the same chip.

Index Terms—Active sensor, cavity resonators, microcavity, microdisk, optical losses, optical sensors, photoluminescence (PL), 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} 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 handheld 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: 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 paper, we propose and characterize a basic sensing photonic structure consisting on a μ -disk cavity made of an 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.

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.

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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 paper, 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 nanowatt in a single resonance and show competitive sensitivities (about 36 nm/RIU) and a DL 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 vapor deposition technique. The thickness of this layer has been chosen to ensure monomodal behavior 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 950 $^\circ\text{C}$. The ion energies and doses of the double implantation were chosen to achieve a flat Si excess profile and to optimize 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%. 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. In Fig. 1, we show a atomic force microscopy (AFM) 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 this paper, we will mainly focus on the experimental results of the 7.5 μm radius disks, which are representative of the whole set.

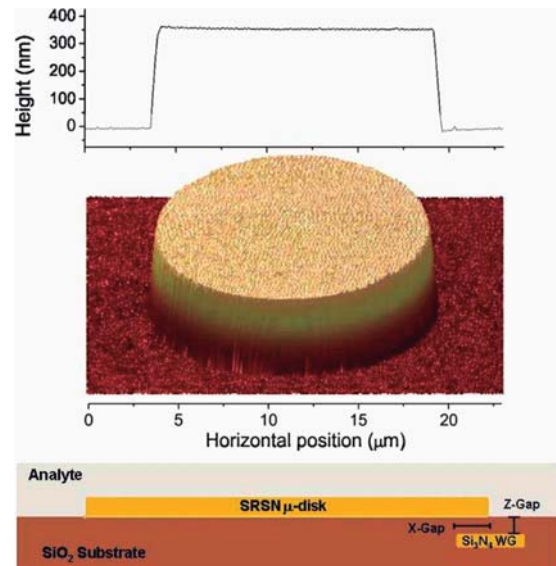


Fig. 1. (Top and central panels) AFM image of an isolated 7.5 μm radius μ -disk. (Bottom panel) Scheme of the cross section of the coupled structure. The Z-gap is defined as the vertical distance between the bottom of the μ -disk and the top of the WG, while the X-gap represents the center of the WG 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 Fig. 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 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 in 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

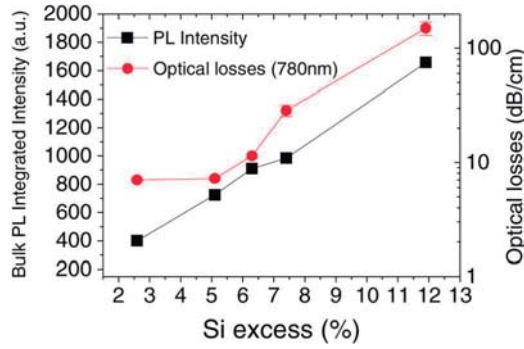


Fig. 2. Integrated PL intensity (black squares) and waveguide optical losses at 780 nm (red dots) as a function of the Si excess present on the active material.

propagation losses along the emission spectrum of the active material using the same μ -PL setup described previously. 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 traveling 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 characterize the active material in terms of the emitted PL intensity of the bulk material and propagation losses on strip waveguides.

The black squared curve in Fig. 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 in Fig. 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 analyze their wavelength dependence. The SES technique was used to extract this information along the spectral range of the emission band. In Fig. 3, we report those results in decibel per centimeter for the case of a strip waveguide with 2.5% Si excess. Increasing the Si excess did not modify the spectral behavior of the losses but only scaled their values similarly to that previously showed for the cut-back experiments.

On the inset of Fig. 3, we report the normalized spectrum resulting from three different positions of the excitation spot. The redshift 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

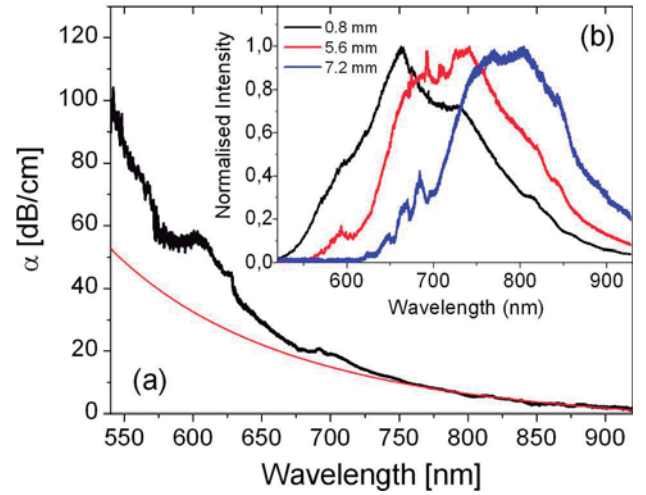


Fig. 3. (a) Spectral dependence of the optical losses of an active strip waveguide with 2.5% Si excess, as extracted from the SES measurements. The fit using a Rayleigh type scattering dependence is also shown. (b) Guided spectrum collected at the output of the waveguide for three different positions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

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 $1/\lambda^4$ behavior and the contribution of direct absorption losses starts to play a nonnegligible role.

IV. SINGLE μ -DISKS CHARACTERIZATION

In view of the realization of a low DL system, one of the most important parameters is the quality factor 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

$$Q = 2\pi n_g / \lambda \alpha = \lambda / \text{FWHM}. \quad (1)$$

In Fig. 4, we represent the two main contributions to the total quality factor of the μ -disk. The black curve is associated with the material-related propagation losses as extracted from the experimental data in Fig. 3. The scattered data represent the quality factor associated with the radiative losses of the geometrical structure, as extracted from finite-difference time domain (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 factor as a function of wavelength starts rising, while beyond a critical value it starts to decrease, dominated then by the geometrical

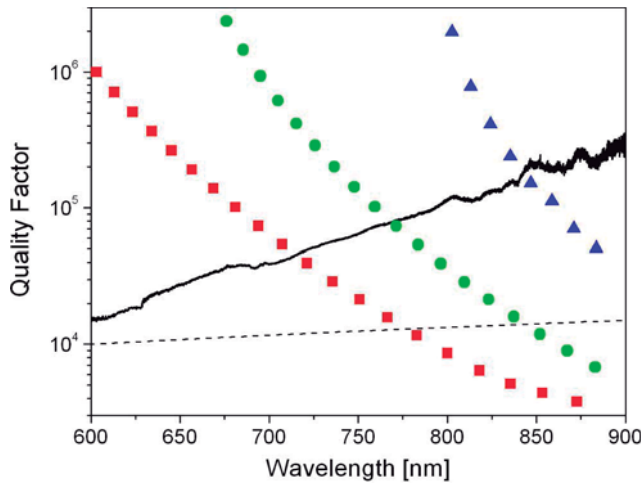


Fig. 4. Black continuous line represents the Q factor related to the material losses reported in Fig. 3. The dotted curves (squares, circles, and triangles) are the radiative Q factor of μ -disks of different dimension (3, 4, and 5 μ m radius) obtained by FDTD simulations. The dashed line represents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

losses. The direct correlation between material losses and experimental Q factor has been reported by us in a recent work [14].

In Fig. 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 factor for the case of 2.5% Si excess.

The experimental μ -PL characterization of the μ -disk of 7.5 μ m radius with 2.5% Si excess is reported as a black curve in Fig. 5. This sample showed the best results in terms of Q factor. We have indeed observed a saturation of the experimental Q factor over the analyzed spectral range up to 1.4×10^4 (inset of Fig. 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 (see Fig. 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 nanowatt 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 factor 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 factor 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

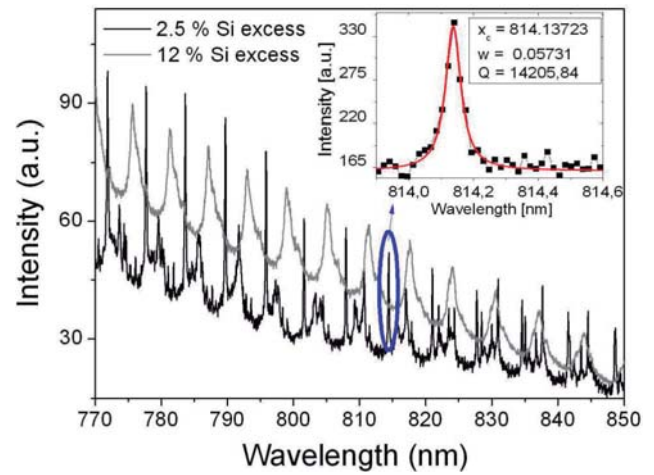


Fig. 5. μ -PL spectrum of a 7.5 μ m radius μ -disk with Si excesses of 12% (gray) and 2.5% (black). On the inset, we show a particular resonance at 814 nm of the 2.5% Si excess μ -disk, where Q factor of 1.42×10^4 is measured.

principle allow demultiplexing and detecting the emitted signal within the same chip.

V. ACTIVE μ -DISKS COUPLED TO PASSIVE Si_3N_4 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 optimized 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 μ m. 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 1 μ m were realized to verify the simulated predictions. The main results of this study are reported in Fig. 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 factor 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 with 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

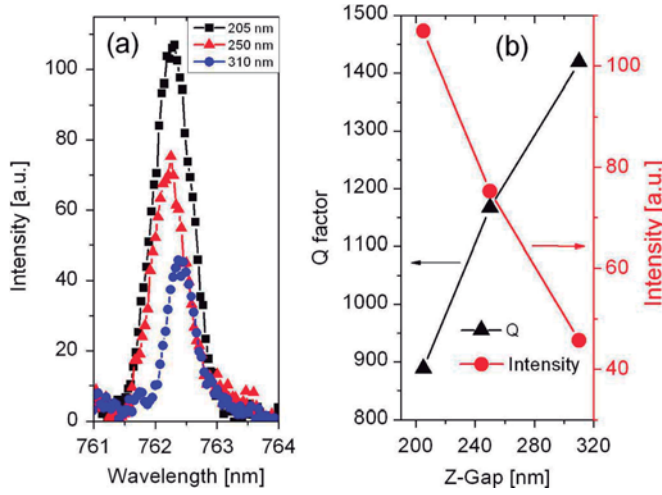


Fig. 6. (a) TM-polarized μ -PL intensity of a resonance at about 762 nm for different gap distances between the bus waveguide and the μ -disk. (b) Quality factor (black triangles) and μ -PL resonance intensity (red circles) as a function of the vertical gap distance.

the following, we will show results corresponding to the sample with the thickest gap.

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 $R = 7.5 \mu\text{m}$ and $Z\text{-gap} = 310 \text{ nm}$, which gave the best result in terms of Q factor. 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 Fig. 7(a), in which we can appreciate an overall resonance displacement of $\Delta\lambda = 1.37 \text{ nm}$ 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 refractive index unit, so that

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

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 $\Delta n_{\min}$. In [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

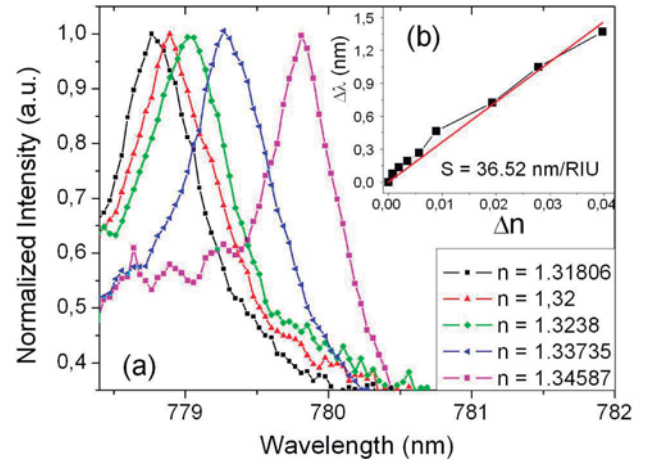


Fig. 7. (a) Spectral displacement of a resonance for five analytes with different n . (b) Linear behavior of the $\Delta\lambda$ as a function of Δn for all the analytes prepared. The slope of the linear fit is 36.52 nm/RIU .

distinguish down to a $\delta\lambda = 0.06 \text{ nm}$ shift, so that the DL of our measurement is

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

However, the previous definition of DL does not allow to reliably compare 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., $\text{DL}_{\text{FWHM}} = 1.7 \times 10^{-2}$. The described results are slightly lower than that reported the state-of-the-art CMOS compatible 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 with respect to μ -disks due to the greater surface in contact with the volume in which the WGM is developed.

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 nanowatt 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 demultiplexing and detection can be integrated on the same chip.

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High Q light-emitting Si-rich Si_3N_4 microdisks

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We report on the optical properties of active silicon (Si)-rich Si_3N_4 microdisk cavities in the visible range. We have studied the correlation between the quality (Q) factor of the cavities and the active material deposition parameters. Microphotoluminescence measurements revealed subangstrom whispering gallery modes resonances and a maximum Q of 10^4 around 760 nm. These values improve significantly the best results reported so far for Si-based light-emitting circular resonators in the visible range. In contrast to what is reported for Si-rich SiO_2 -based microcavities, we demonstrate the absence of a spectral widening at high pump fluxes associated to carrier absorption mechanisms, which allows high emitted power without degrading the Q of the cavity. These results open the route toward the monolithic integration of those structures into more complex circuits including Si photodetectors. © 2011 Optical Society of America

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There is a broad agreement that silicon (Si) will become the future material for nanophotonics. The reason for this is that Si allows optical devices to be made cheaply using standard semiconductor fabrication techniques and integrated with microelectronic chips [1]. Among other applications, Si photonic structures and devices promise high bandwidth, densely integrated information systems with low operation and dissipation powers, as well as low-cost sensors with high sensitivity and specificity. However, one of the main drawbacks that explain why Si photonics is still not dominant with respect to compound semiconductors and Si microelectronics lies in the difficulties associated with making Si a host material for efficient light emission. Among the several alternatives to obtain highly efficient Si-based emitting materials, Si nanoclusters embedded in an SiO_2 matrix has been probably the most investigated, since they can even provide optical gain in the visible region [2]. An interesting strategy has been to combine the light emission properties of those materials and the optical properties of circular microcavities (such as disks or rings) [3–6]. However, the work addressing this topic reveals maximum quality (Q) factors on the emitted light of 10^3 [4–6] obtained at low pump powers, i.e., with low photoluminescence-emitted powers. Indeed, carrier absorption (CA) mechanisms decreased those Q at high pumping fluxes, thus limiting the possible applications of those devices [5,6]. Similarly to Si-rich SiO_2 , Si-rich Si nitrides (SRSN) systems have also revealed efficient light emission in the visible range [7], in addition to a low barrier mismatch between Si and Si_3N_4 that allows efficient electrical excitation [8]. Compact devices deposited over SiO_2 cladding layers can be achieved with SRSN materials thanks to their relative high refractive indices ($n \geq 2$). As an active medium for circular microcavities, SRSN has been only studied for applications in the IR region of the spectrum [9,10], aiming to exploit the sensitization effect when Er^{3+} ions are present in the matrix. In this Letter, we report an experimental characterization of visible light-emitting microdisks (μ -disks) made of SRSN materials, in which Q values of about 10^4 have been measured with high emit-

ting powers, about three times greater than those made of Si-rich SiO_2 .

The samples under analysis have been produced using standard complementary metal–oxide–semiconductor (CMOS) compatible processes. Initially, $2\text{ }\mu\text{m}$ of SiO_2 was thermally grown ($1100\text{ }^\circ\text{C}$, wet ambient) on top of crystalline Si wafers, becoming the optical cladding of the active structures. A 300-nm-thick layer of stoichiometric Si_3N_4 material was subsequently deposited by using the low-pressure chemical vapor deposition technique, which afterward suffered a Si ion implantation followed by an annealing in N_2 atmosphere at $1100\text{ }^\circ\text{C}$. The thickness of the active material allows obtaining monomodal behaviors in the direction perpendicular to the disk surface for the transverse component of the electromagnetic field in the in the direction of the radius (TR) polarization (parallel to the disk surface). The implantation consisted of a 150 keV energy process (I1) followed by a second one at 90 keV with a lower dose (I2), aiming to obtain a flat Si excess profile. In Table 1, we summarize the active material parameters of the samples reported in this manuscript. Energy-filtered transmission electron microscopy measurements on sample 1 did not reveal Si crystalline nanostructures.

The photonic structures have been finally defined by means of standard photolithographic techniques. We have fabricated μ -disks with radii ranging from 3 to $10\text{ }\mu\text{m}$ with average top surface roughness lower than 1 nm, as

Table 1. Sample Parameters: Implantation Doses, Nominal Si Contents, and Associated Si Excess Values^a

Sample	I1/I2 Dose ($\times 10^6\text{ cm}^2$)	Si Content (at. %)	Si Excess (%)
1	12.5/4.8	50	11.9
2	7.5/2.9	47.7	7.4
3	6.2/2.4	46.5	6.3
4	5.0/1.9	45.8	5.1

^aThe Si excess percentage is defined as $(1 - 0.75x)/(1 + x)$, where x is the ratio of the atomic concentrations of nitrogen and Si.

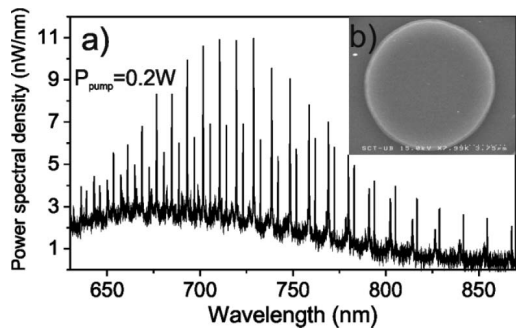


Fig. 1. (a) TR-polarized μ -PL emission spectrum of a μ -disk of sample 4 ($R = 4.5 \mu\text{m}$). (b) SEM image of the corresponding μ -disk.

extracted from atomic force microscopy (AFM) measurements. As an example, a scanning electron microscope (SEM) image of a μ -disk with a radius (R) of $4.5 \mu\text{m}$ is shown in Fig. 1(b). Waveguides with different widths ($1\text{--}10 \mu\text{m}$) have been also produced in order to characterize the optical losses of the active material. Those measurements have been done by using the cutback technique at 633 and 780 nm.

High spectral resolution microphotoluminescence (μ -PL) experiments were performed at room temperature with the goal of characterizing the on-plane spectral emission of a single μ -disk. We have used the 476 nm line of an Argon laser and a solid state laser emitting at 370 nm as excitation sources. A long working distance objective was used to focus the laser beam on the top surface of the disks providing a spot size of few micrometers. Another objective ($\text{NA} = 0.4$) was used to collect the on-plane μ -PL emission, which was afterward focused on a high-resolution monochromator ($\Delta\lambda = 0.06 \text{ nm}$) coupled to a visible CCD camera. A linear polarizer was also placed in the collection line to select the TR or TM polarized emission. Finally, the total on-plane emission has been quantified by measuring the signal collected by the objective (which only collects a finite solid angle) with a calibrated photodetector, and then integrating to the 2π angle in which the on-plane μ -disk emission is distributed. It is worth mentioning that we are currently optimizing a structure in which an Si_3N_4 waveguide is placed below the μ -disks, aiming to extract the emitted power efficiently without damaging the quality of the cavities.

The main panel of Fig. 1 reports the quantified μ -PL spectrum (TR polarized) obtained under a 476 nm pumping from a single μ -disk with $R = 4.5 \mu\text{m}$, being the active material of sample 4. Whispering gallery mode resonances are clearly observable over an offset PL signal that has the same spectral shape of the PL emission obtained from the bulk material. The latter signal is not coupled to supported modes of the disk. The area of the curve provides a total power emitted on plane of $0.6 \mu\text{W}$ ($0.12 \mu\text{W}$ contained within the resonances), associated with a minimum power efficiency of $\sim 3 \times 10^{-6}$. We have measured an order of magnitude higher efficiency ($\sim 3 \times 10^{-5}$) by decreasing the pumping wavelength to 370 nm, which is a result of the increasing of the excitation cross section of the emitting species. Those efficiency values can be much further improved by optimizing the overlap of the pumping spot shape and the emitting region within the disk that is

actually coupled to the supported modes. In fact, the calculated modal volumes for the fundamental modes (few cubed micrometers) are much smaller than the pumped volume (several tens of cubed micrometers).

We have studied the performance of the active materials associated with the different samples in terms of three magnitudes: (i) PL of the bulk material, (ii) optical losses of the waveguides, and (iii) Q of the μ -disk. The latter is inversely proportional to the internal losses (α) within the resonator ($Q = \lambda/\Delta\lambda = 2\pi n_g/\lambda\alpha$, where n_g is the group refractive index of the mode).

Regarding the PL spectra (not reported here), we have observed that the PL intensity roughly scales with the pumping flux and the implantation dose. On the other hand, we have determined that the propagation losses on the waveguides increase: (i) with the Si excess (red circles in Fig. 2(a)) and (ii) roughly by a factor of 2 when moving to 633 nm. The first behavior seems directly related with the inverse of the Q values of the μ -disks, as also shown in Fig. 2(a). In Fig. 2(b), we illustrate this effect for the cases of sample 1 and sample 4 ($R = 7.5 \mu\text{m}$ in both cases). Resonances on sample 1 appear much wider than those of sample 4, in which a multimodal behavior observed is associated with several radial modes of different order. In fact, the maximum Q values are those corresponding to sample 4, which has lower Si excess. In this case, subangstrom resonances are observed on a spectral range of several tens of nanometers around 760 nm, leading to Q values as high as 10^4 (see Fig. 2(c)). Those Q values are, to the best of our knowledge, the best ones reported so far in light-emitting Si-based circular microresonators in the visible range. We address the material losses as the limiting factor for the measured Q values since there is a strong dependence on the Si excess present in the matrix and a clear correlation with the inverse of the optical losses of the waveguides [11]. Furthermore, we observed a decreasing of Q at lower

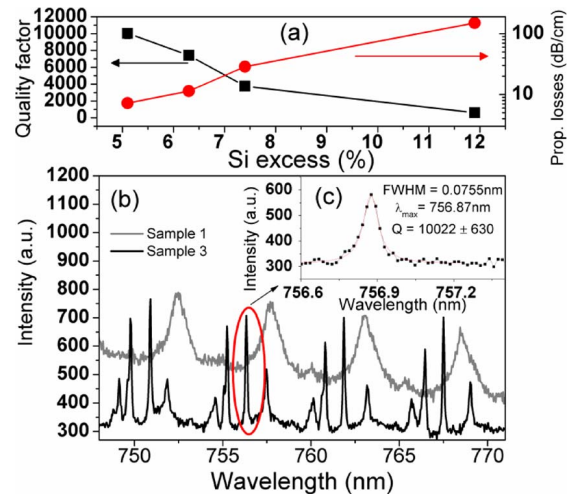


Fig. 2. (Color online) (a) Behavior of Q (black squares) around 760 nm as function of the Si excess for $R = 7.5 \mu\text{m}$. Propagation losses at 780 nm are also reported (red circles). (b) TR-polarized μ -PL spectrum corresponding to disks with $R = 7.5 \mu\text{m}$ of samples 1 (gray) and 4 (black). (c) Resonance present at 756.87 nm for sample 4 together with the corresponding Lorentzian fit.

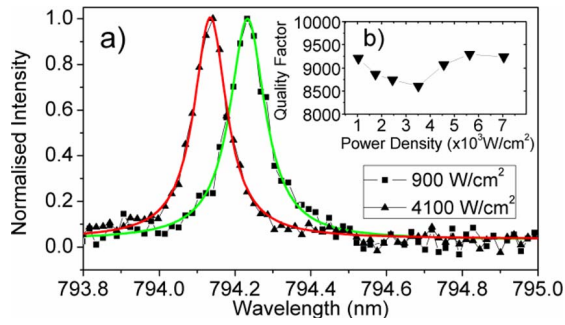


Fig. 3. (Color online) (a) μ -PL spectra of a resonance at 794 nm for a 10 μ m disk in sample 4 at low (squared points) and high (triangular points) power densities. (b) Q as a function of the pump power density.

wavelengths, in agreement with the mentioned increasing of the optical losses.

We have also investigated the effect that the pumping power may induce the optical losses of the material and, therefore, the Q values of the cavities. Indeed, in the case of Si-rich SiO_2 μ -disk systems, CA losses prevail at high pumping fluxes, which is mainly a consequence of the relatively long lifetime (tens of microseconds) of the excited carriers (in situations where Auger or stimulated emission processes are not dominant mechanisms) [12]. On the contrary, in SRSN μ -disks, CA mechanisms do not dominate the total losses of the material. In Fig. 3, we show the results of a μ -disk of sample 4 ($R = 10 \mu\text{m}$): the Q values remain around 9000 over a wide range of pumping powers. We have also experimentally established an upper limit to the decay lifetime of the PL emission of 200 ns (the temporal resolution of our experimental setup), almost 2 orders of magnitude faster than in Si-rich SiO_2 . It is worth noting that the authors in [13] report a maximum recombination lifetime of 50 ns. Therefore, the total recombination probability of the carriers generated in this material is so high that CA effects do not generate measurable losses, so that Q values do not decrease. An immediate implication is that it is possible to have high emitted power with high Q , in contrast to what occurs in Si-rich SiO_2 . In fact, we have measured that the power contained by a single resonance in the range between 700 and 770 nm can be as high as a few nanowatts (e.g., see Fig. 1), while keeping Q factors close to 10^4 . On the other hand, free carrier refraction effects at high powers are not totally absent since they are likely originating a slight decreasing of the material refractive index that produces the subnanometer blueshift reported in Fig. 3.

In conclusion, we have done a thorough study of Si-rich Si_3N_4 μ -disk resonators, where we have quantified on-plane emitted powers up to 0.6 μW (few nanowatts in a single resonance) and measured Q values of 10^4 on a wide spectral range around 760 nm. We have also established a direct relationship among the Si excess, the waveguide optical losses, and the inverse of the Q of

the cavities. In addition, we have demonstrated that an increasing of the pumping flux does not generate a spectral widening of the resonances, which is in contrast to that observed in other reports of Si-rich SiO_2 -based μ cavities. The present result allows high emitted power within the resonances while maintaining Q values close to 10^4 . Indeed, these power values are well above the minimum sensibility of state-of-the-art visible Si-based integrated photodetectors [14], which would allow detecting the emitted signal within the same chip. On the basis of these results, we foresee the use of visible light-emitting SRSN μ -disk resonators as CMOS-compatible integrated light sources in photonic platforms with increased complexity for sensing and telecommunications applications.

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Optically active substoichiometric Si_3N_4 μ -cavities

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We report on the morphological and emission properties of microdisk resonators with an optically active disk material made of luminescent substoichiometric Si_3N_4 matrix. We demonstrate the importance of optimising the material by finding a compromise between the photoluminescence properties and optical losses. A careful design of the cavities for optimising the radiative quality factors has been also performed, enabling us to produce active disks with low intrinsic losses. Subnanometer whispering gallery mode (WGM) resonances have been experimentally detected, showing quality factors as high as 1200

around the wavelength of 719 nm. These values are among the highest previously reported values in Si-nanocluster-based systems and are actually limited by the spectral resolution of our experimental setup. In addition, an increasing of the pumping flux is not generating a spectral widening of the resonances, thus not affecting the quality of the cavities. This is in contrast to what observed in other reports of Si-nc based microcavities, where significant enlargements are observed associated to carrier absorption losses.

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1 Introduction One of the main relevant challenges in optoelectronics concerns the inexpensive integration of efficient light sources on a silicon-based circuit by using complementary metal-oxide semiconductor (CMOS) compatible approaches [1].

An interesting strategy is to combine the light emission properties of Si nanostructures (Si-nc), which is several orders of magnitude more efficient than bulk Si [2], and the optical properties of μ -cavities. In this context, active μ -resonator circular cavities (such as disks or rings) could generate a great impact not only in the short scale telecommunications application area but also in other fields like contamination and biochemical sensing.

Few works in the literature address such active microcavities, where Si-nc are present within a SiO_2 matrix [3–6]. While in Ref. [3] the low quality factors reported were associated to inhomogeneous line broadening due to the slight dispersion of microdisk diameters within the excited area, in Refs. [4–6] quality factors between 1000 and 3000 were measured. However, at least for the two latter works, carrier absorption mechanisms decreased them dramatically with pump flux.

To the best of our knowledge, as an active medium for microdisk resonators, substoichiometric silicon nitrides (SRSN) has been only studied for applications in the IR region of the spectrum, [7,8] aiming to exploit the sensitization effect when Er^{3+} ions are present in the matrix.

However, SRSN systems have also revealed efficient light emission in the visible range [9], which could provide alternative routes towards the fabrication of optically active Si devices that need to be explored. In addition, the relatively low barrier mismatch between Si and Si_3N_4 makes this material suitable for efficient electrical excitation [10]. From the point of view of the passive photonic properties, SRSN materials present refractive indices higher than $n=2$, which allows producing highly performing compact devices deposited over SiO_2 cladding layers.

In this work we address the design and experimental characterisation of a system composed by an SRSN active disk deposited over a SiO_2 cladding layer, aiming to provide a better understanding of the characteristics and potentialities of this active cavity as a CMOS compatible light source.

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We have initially followed two parallel guidelines:

(i) The design and simulation of the μ -disks, which has involved the study of their modal structure (Section 2).

(ii) A characterisation and optimisation of the active material in terms of its photoluminescence (PL) intensities (Section 3.2) and the optical losses measured in waveguides (Section 3.3)

Using optimised recipes for both the active materials and cavity morphologies we have finally fabricated active μ -disks. In Section 3.4, we present an experimental characterization of single μ -disks by means of visible μ -PL measurements.

2 Simulation results The simulated spectral analysis of the μ -disks has been made by using of a three-dimensional Finite Difference Time Domain method (FDTD) package [11]. We have analysed the position of the resonance peaks and electric field spatial distribution of the different whispering gallery modes (WGM) inside the cavity, which are modified by varying the structural parameters of the optical element (radius, disk height and cladding thickness). The aim of this study was to obtain monomodal behaviours in the vertical direction (perpendicular to the disk surface) for the TE polarisation (parallel to the disk surface) with quite high free spectral ranges (FSR) and radiative quality factors (Q_{rad}) higher than 10^4 . The spectral region of interest was around $0.7 \mu\text{m}$, where the experimental PL emission of the active material was present (see Fig. 4).

In Fig. 1 we show the results for Q_{rad} , which decreases with the wavelength as a consequence of a confinement factor lowering of the supported modes. In addition, Q_{rad} increases with the disk radius, just reflecting the fact that the bending losses of the supported modes decrease.

It is also worth noting that we have optimised the disk thickness to be $0.3 \mu\text{m}$, showing a maximum confinement factor while staying monomodal in the vertical direction.

The Q_{rad} results are several orders of magnitude higher than what would be expected for equivalent designs using substoichiometric SiO_2 as an active material. This is a consequence of a much lower refractive index with respect to SRSN and would prevent producing compact substoichiometric SiO_2 cavities over SiO_2 .

Considering the technical limits of the experimental μ -PL setup that will be described in the following, it is difficult to experimentally study a cavity in which high order radial families are showing intense contributions.

Resonators with radii around $4 \mu\text{m}$ are a good compromise between high Q_{rad} values of the 1st radial family modes and low contribution of higher order radial families.

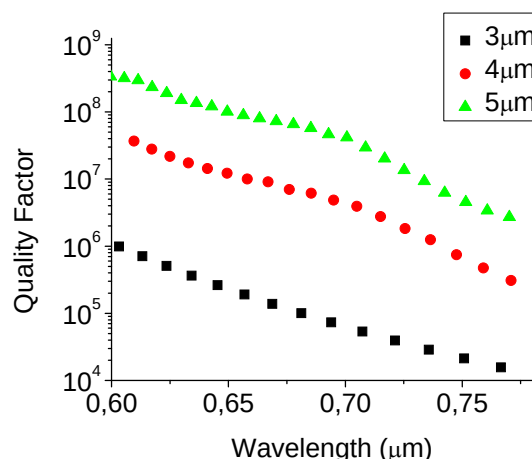


Figure 1 Spectral dependence of the quality factor associated to the 1st order radial family modes. Values corresponding to μ -disks of different radii (3, 4 and $5 \mu\text{m}$) are reported.

As an example, in Fig. 2 we show the PL spectral dependence (TE polarization) of a $3 \mu\text{m}$ disk. Two different radial families are present, the fundamental one being much more intense and narrower than the second order one, which would be hardly observable experimentally. For the shortest wavelengths reported on the graph a third order radial family is also supported weakly. It is worth to note that the modes order has been confirmed by checking their spatial energy distribution.

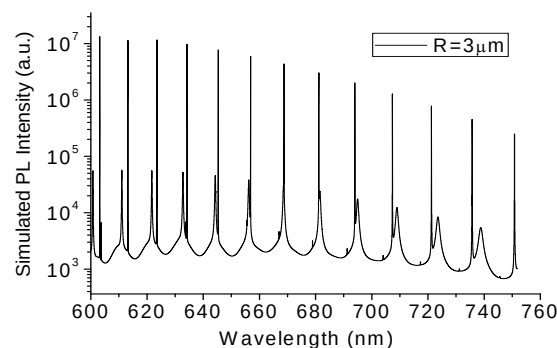


Figure 2 Simulated on plane PL emission for a disk of $R=3 \mu\text{m}$.

3 Sample characterisation

3.1 Sample preparation The samples under analysis have been produced using standard CMOS compatible processes. As a first step, $2 \mu\text{m}$ of SiO_2 was deposited on top of crystalline silicon wafers, acting as an optical cladding for the resonators. A layer of stoichiometric Si_3N_4 material has been subsequently deposited by using the LPCVD deposition technique, which has afterwards suffered a double ion implantation of Si followed by an an-

nealing procedure in N_2 atmosphere at 1100 °C. The double implantation has been realised with the aim of obtaining a flat Si excess profile (see left panel of Fig. 3). We have performed EFTEM measurements on sample 1 and it was not possible to observe the formation Si nanocrystals within the active material. For the sake of clarity, we will only show results on two samples belonging to a wider set, labelled as sample 1 and 4 (see Table 1).

Sample	Si_3N_4 Thickness (nm)	Implantation energy (keV)	Implantation Dose ($\times 10^{17}$ at / cm^2)	Annealing treatment
1	300	150 / 90	1.25 / 0.48	1100 °C for 4h
4	300	150 / 90	0.62 / 0.24	1100 °C for 4h

Table 1 Implantation parameters for samples 1 and 4.

As it is shown in Fig. 3, the limitation in the available implantation energy makes the Si-excess distribution to be centred slightly over the gravity centre of the fundamental mode at 660 nm, which is not the optimised situation in order to couple as much light as possible to the supported modes (whose gravity centre is displaced slightly towards the SiO_2 cladding).

The photonic structures have been defined by means of standard photolithographic techniques. In particular, we have designed disks with radius from 3 μm to 10 μm and waveguides with variable width (1-10 μm) for the characterisation of the optical losses. On the basis of what stated in Section 2, the disks have a thickness of 0.3 μm . The surface analysis with AFM has revealed a very good top surface without inhomogeneities or irregularities (see Fig. 3). The roughness results to be on average lower than 1 nm, so we can neglect this source of optical losses.

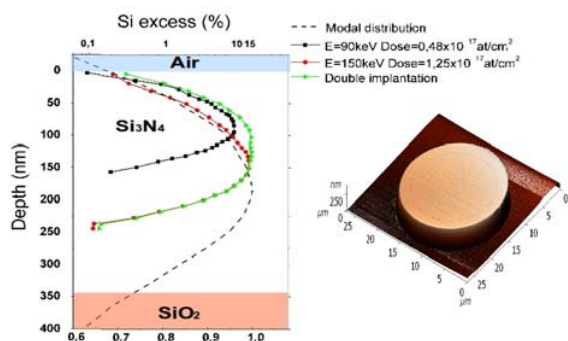


Figure 3 Left. Cross section of the device structure, implantation profile and modal energy distribution. Right. AFM analysis of a disk of radius 10 μm .

3.2 Standard PL characterisation Standard PL measurements were performed using the 488 nm line of an Ar

laser. We have characterised and compared the PL emission properties of a of bulk region of active material on samples 1 and 4. Stoichiometric samples were not showing any PL.

Figure 4 shows the normalised spectra of both samples. A strong interference pattern is observed with a relatively short free spectral range (60-90 nm), which is related to the presence of a 2 μm thick SiO_2 cladding layer in addition to the 0.3 μm of Si_3N_4 material that the light has to travel before it is reflected backwards by the Si substrate.

In any case, it is clear that a red-shift of the PL maximum is observed when the Si implantation dose is increased.

In Fig. 5 we analyse, for those samples, the PL intensity and its evolution as a function of the pumping photon flux (Φ). It is observed that the PL intensity for sample 1 is about 1.8 times that of sample 4, almost scaling with the implantation dose. It is also interesting to note that the behaviour with Φ is linear, which means that, at least for this flux range, there are no flux dependent non-radiative recombination mechanisms (such as Auger-like processes) competing with the spontaneous emission process.

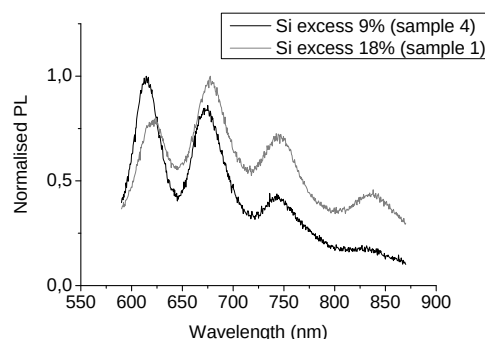


Figure 4 Normalised PL emission for samples 1 (gray) and 4 (black).

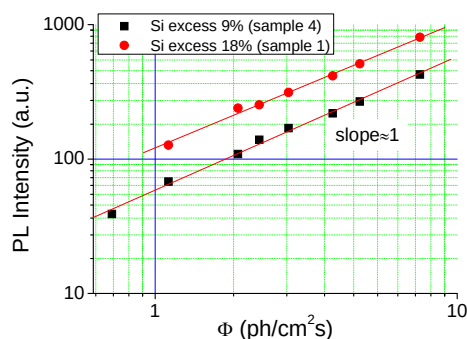


Figure 5 Integrated PL emission as a function of the photon flux for both samples.

3.3 Optical losses in rib-waveguides In this section we will describe the optical losses results in the visible region for both samples. We have used both the Cut-back and the Scattered Light Collection techniques.

The comparison between the results of these two samples shows a dramatic increase in optical losses with the amount of Si excess inside the Si_3N_4 matrix. Sample 1 could not be analysed by the cut-back technique because the intensity of transmitted light was well below the sensitivity limit. As it is shown in Fig. 6 for different waveguide widths, sample 4 presented acceptable propagation losses of about 6 dB/cm and 15 dB of coupling losses. Propagation losses below 1 dB/cm were determined on stoichiometric (not implanted) Si_3N_4 waveguide samples, which allow us to affirm that losses are increasing with Si excess in the matrix.

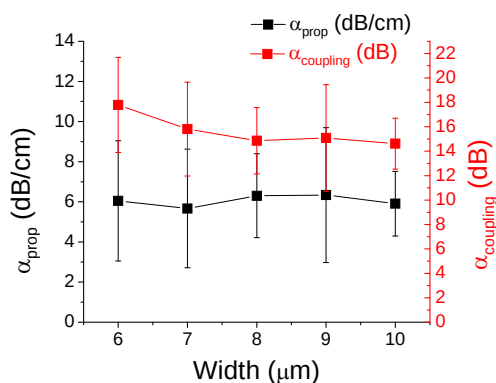


Figure 6 Propagation and coupling losses in VIS region (780 nm) for different waveguide widths for sample 1.

To overcome the limits arising from the cutback technique on sample 1, we performed measurement of the scattered light at 633 nm. By using this technique values close to 200 dB/cm for propagation losses in sample 1 have been detected. In view of application of the analysed material it is important to compare these results with that of Section 3.2: even if sample 1 presents higher PL intensity, it shows propagation losses values that will strongly deteriorate the total quality factor (Q) of an eventual cavity.

It is also worth noting that on sample 4 there is a propagation losses increase of about 10 dB/cm when moving from 780 to 633 nm, and that at 1.55 μm they were as low as 1 dB/cm (the lower detection limit of our setup).

The physical origin of the measured propagation losses is directly related with the amount of Si excess in the matrix. Since losses are decreasing with wavelength, we believe that they are probably a consequence of a combination between Rayleigh scattering from Si nanoclusters and direct absorption losses from the species that are likely generating the PL emission

3.4 μ -PL characterisation Room temperature μ -PL measurements have been performed by pumping with the 488 nm line of an Ar laser. A long working distance objective was used to focus the laser beam on the samples, having the possibility of a single resonator excitation since the spot can reach a diameter as small as 5 μm .

Low resolution measurements (~ 2 nm) have been performed in order to study the FSR and the distribution of resonance peaks. On the other hand, to extract the Q values, high resolution measurements were done (~ 0.6 nm). Even if the PL intensity was higher for sample 1, the WGM of the disks were hardly appreciable. This is an expected result explained on the basis of the high propagation losses reported for this sample in the previous section. On the contrary, sample 4 presented strong WGM resonances, as it can be seen on the low resolution spectrum of Figure 8 ($R = 4 \mu\text{m}$).

Thus, it appears clear that, since the physical geometry and surface quality of the disks are independent on the implantation dose, the observation of WGM on sample 4 and not on sample 1 is related to the significant difference of the optical losses reported for the waveguide devices.

We have compared the obtained μ -PL spectra with the results of the FDTD simulations. At this point it is worth to remember that the FSR is defined by:

$$FSR = \frac{\bar{\lambda}^2}{2\pi R n_g} \quad (1)$$

where R is the radius of the disk, $\bar{\lambda}$ is the average wavelength in vacuum between the two resonance peaks and n_g is the group refractive index.

The agreement between simulations and experiment can be checked by comparing the results obtained for the FSR (extracted by determining the position of each resonant peak) and the extracted n_g (dashed curves of bottom and top panel respectively of Fig. 7).

It is also worth to note that an increase of the disk radius is reflected on a FSR reduction (in agreement with Eq. (1)), that results in a group refractive index insensitive to the disk radius, which is also consistent with performed simulations. The extracted n_g is thus compatible with what expected on the simulations for a Si_3N_4 like material. The uncertainty of the resonance positions translates in non-negligible variations of the extracted n_g , which prevents us to extract conclusions about the effect of the implantation on the material refractive index.

For the case of a 5 μm radius disk there is a slight disagreement between experiments and simulations. This is due to the fact that, in this case, also the second radial family is well supported. Thus, in the experiments, we are observing the superposition of resonances of the first two radial families, effectively resulting in an enlargement and shifting of the peaks and a false decrease of FSR (increase of the n_g) with respect to the real one for the first family.

On the other hand the total quality factor (Q) can be expressed as follows:

$$Q = 2\pi \frac{n_g}{\lambda \alpha} = \frac{\lambda}{\Delta\lambda} \quad (2)$$

where α are the overall optical loss coefficient of the propagating mode.

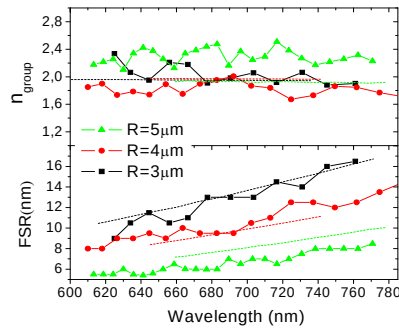


Figure 7 Bottom panel. FSR for the different disks reported together with simulations of the results for a 3 μm disk. Top panel. Group refractive index determined by using Eq. (2). Simulated results are reported as dashed lines.

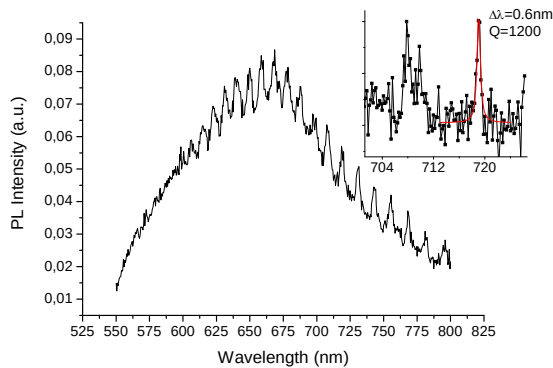


Figure 8 Main Panel. Low resolution μ-PL spectrum of a 4 μm radius microdisk. Inset. High resolution spectrum of a particular part of the spectrum. The red curve shows a Lorentzian fit of one of the resonances.

The inset of Fig. 8 shows a restricted part of the spectrum acquired at the resolution limit of the experimental setup (narrowest slit widths). In this configuration we obtain $\Delta\lambda = 0.6$ nm (equal to the minimum resolution), establishing a lower bound of $Q = 1200$ at $\lambda_0 = 719$ nm.

We have also observed that the spectral width of the resonances broadens as the wavelength decreases until the resonances even disappear. This result is in contrast with the simulated behavior of Q_{rad} , which was decreasing with wavelength (see Fig. 1). We believe that this experimental behaviour is related with an increasing of the material optical losses at lower wavelengths. This is confirmed by our

observation of different optical losses in the waveguide devices for 633 nm and 780 nm, where much higher losses were reported for the former wavelength on sample 4.

Furthermore, we can decompose Q in four different contributions, as follows:

$$Q^{-1} = Q_{rad}^{-1} + Q_{mat}^{-1} + Q_{ssc}^{-1} + Q_{sa}^{-1} \quad (3)$$

where the inverse of Q_{rad} , Q_{mat} , Q_{ssc} and Q_{sa} are related to radiation, material (bulk absorption and propagation), surface scattering and surface absorption losses, respectively.

As it was previously mentioned, AFM measurements performed on sample 4 show a high quality of the surface and shape of the disk, so we can neglect the last two terms in Eq. (3). Q_{rad} is related to the disk geometry and has been calculated by means of FDTD simulations to be greater than $Q_{rad} > 10^5$ for 4 μm disks. On the other hand, Q_{mat} is directly related to the material losses at the resonance wavelength, which we have already determined to be lower than 10 dB/cm, which means a $Q_{mat} \approx 10^5$. However, if we invert Eq. (2), we can extract an experimental value of α larger than 600 dB/cm. Therefore the 0.6 nm width of the observed resonances is effectively limited by the spectral resolution of our system, and not by the quality of the device, which might be as high as $Q \approx 10^5$.

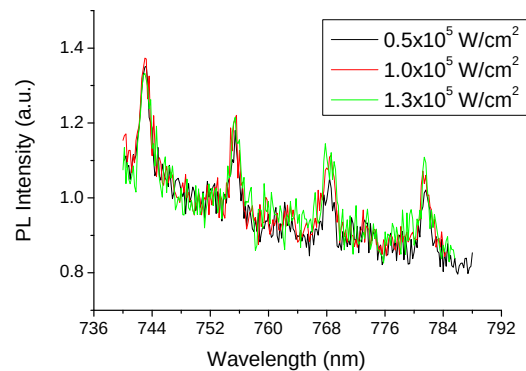


Figure 9 Different highly resolved spectrum obtained at different pump photon fluxes.

Moreover, we have performed experiments for different photon fluxes in order to check if there are flux induced losses. This behaviour is typical of carrier absorption mechanisms, not present in the absence of an external excitation mechanism [12]. In Fig. 9, we report the results obtained for a restricted region of the spectrum for different pump densities, where no broadening is detected.

4 Conclusions In conclusion, we have made a comprehensive study of active substoichiometric Si₃N₄ μ-disk resonators, where, on the one hand we have designed and optimised the photonic structure and on the other hand we

have studied the optical properties of the active material in order to find a compromise between PL intensity and optical losses.

Both studies have been combined in order to fabricate high quality disk resonators from the morphological and optical point of view. In fact we have demonstrated quality factors as high as 1200 at around 720 nm, actually limited by our setup resolution (about 0.6 nm in the best case).

Finally we have demonstrated that increasing the pumping flux does not generate a measurable spectral widening of the resonances, related to carrier absorption phenomena.

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