

# Athermalization of a self-assembled rolled-up TiO<sub>2</sub> microtube ring resonator through incorporation of a positive thermo-optic coefficient material in planar bilayers

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### Abstract

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8 For the first time, this paper theoretically and experimentally investigates the thermal stability of optical filters based on 9 self-assembled TiO<sub>2</sub> rolled-up microtube ring resonators (RUMRs) by incorporating positive thermo-optic coefficient (TOC) materials (e.g.,  $SiO_2$  and/or  $Si_3N_4$ ). The influence of the TOC, refractive index, and thickness of the positive TOC materials AQ1 on the filtering performance of the  $TiO_2$  RUMR is theoretically studied. The results illustrate that an increase in temperature AQ2 12 leads to a blueshift in the resonant wavelength of the RUMR-based optical filter, which changes at a rate of -33.3 pm/K owing to the negative TOC of TiO<sub>2</sub> ( $-4.9 \pm 0.5 \times 10^{-5}$ /K). By increasing the thickness of SiO<sub>2</sub> or Si<sub>3</sub>N<sub>4</sub> as a positive TOC AQ3 14 material together with TiO<sub>2</sub>, the temperature-induced resonant shifts of TiO<sub>2</sub>/SiO<sub>2</sub> and/or TiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> RUMRs are theoreti-15 cally obtained. The TIRS varies between -40 pm/K (-22 pm/K) and about 30 pm/K (22 pm/K) for TiO<sub>2</sub>/SiO<sub>2</sub> (TiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub>) 16 RUMRs. It is shown that thermal stability occurs when the thickness of the SiO<sub>2</sub> (Si<sub>3</sub>N<sub>4</sub>) layer is ~16 nm (12 nm). At the end 17 of this study, as a proof of concept, an experiment is demonstrated by fabricating an RUMR based on TiO<sub>2</sub>/SiO<sub>2</sub> on the flat silicon wafer. The experimental results show that the athermalization of the system is experimentally achieved by selecting AQ4 the apricated thickness ratio of TiO<sub>2</sub>/SiO<sub>2</sub>. This novel approach for athermalization of the resonators opens up interesting 20 perspectives on the implementation of vertical and multi-routing coupling between photonic and optoelectronic layers and 21 more specifically in a three-integration fashion.

# <sup>22</sup> 1 Introduction

23 Silicon photonics has been recognized as a mature and 24 appealing platform for photonic integration, owing to its AQ5 remarkable properties. This outstanding photonic platform 26 can provide the opportunity to fulfill several functionalities 27 on a single chip by following complementary metal oxide 28 semiconductor (CMOS) fabrication technology [1, 2]. The 29 high refractive index of silicon and the accessibility of high-30 quality oxides create possibilities for increasing the number

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 A2 Microresonators (Síle Nic Chormaic, Misha Sumetsky, Lan Yang).

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of optical devices by fabricating a low-loss and ultracompact optical waveguide on a single chip [3-9]. Despite this great success in the field of silicon photonics, however, quantum photonics and quantum emitters are mainly operated in the visible wavelengths [10]. Hence, silicon photonic device suffers from non-transparency for visible wavelengths; hence, its application can be dramatically limited in this growing research field [10]. Apart from the materials perspective, silicon-based optical devices are highly wavelength dependent with temperature variations, due to their relatively high thermo-optical coefficient (TOC) (~ $1.84 \times 10^{-4}$ /°C) [5–8]. This can be another important concern in integrated optics; by increasing the temperature in the device, an unwanted wavelength shift is caused in the system, which is not satisfactory for many important applications. For example, in the experiential demonstration of silicon photonics using multiwavelength parallelism, optical filtering, switching and/or the implementation of dense 3D optoelectronic systems, this thermal heating may be one of the biggest obstacles in this system. Moreover, due to the fact that an electrically (optically) pumped semiconductor laser within the chip can

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generate higher temperatures, an unwanted shift may occur
in the laser beam, or some fluctuation and/or instability of
the laser beam may occur. To end this unwanted issue, athermal operation of silicon photonic devices is crucial [11].

Hence, to stabilize the chip temperature to a constant 56 level, the athermal photonic devices (Si or SiO<sub>2</sub> based) 57 have been demonstrated extensively already [5-13]. One 58 straightforward technique to fabricate athermal photonic 59 devices (based Si or  $SiO_2$ ) is to coat them with a polymer 60 as the upper cladding layer and take advantage of the poly-61 mer's negative thermo-optic (TO) coefficient to neutralize 62 the waveguide core's positive TOC coefficient. However, 63 in the case of Si waveguides, because of the high thermo-64 optic coefficient of Si, the biggest challenge here is cou-65 pling almost half the light from the silicon core into the 66 polymer cladding to satisfy the athermal condition. To solve 67 this difficulty, in contrast to the other photonic materials 68 (e.g., Si, InP, GaAs, SiO2), owing to its relatively strong 69 negative TOC of approximately ~  $-1 \times 10^{-4}$ /°C and/or 70 even ~  $-2 \times 10^{-4/\circ}$ C around 1550 nm (depending on the 71 phase of  $TiO_2$ ), it has recently presented itself as a valuable 72 73 photonic material to stabilize chip temperature [13]. As a result, many groups have recently investigated the athermal 74 operation of silicon photonic devices by over-cladding them, 75 depositing TiO<sub>2</sub> on top of them. The CMOS-compatible fab-76 rication has led to the hybrid system of TiO<sub>2</sub>-Si devices 77 (e.g., optical waveguides, ring resonators), which illustrates 78 nearly complete cancelation of thermo-optical dependence 79 in these hybrid devices. It is worth mentioning that a group 80 at MIT has experimentally reported a blueshift (i.e., a shift 81 82 to a shorter wavelength) in the resonant modes of microring resonators through an increase in temperature due to the 83 relatively strong negative TOC of TiO<sub>2</sub> [12]. 84

As a result, athermal microresonators have become 85 attractive structures because of their insensitivity to tem-86 perature, strong confinement of light within a small volume 87 and high Q factor for high-speed data transmission, as well 88 as power reduction in optical communication systems and 89 more importantly for thermal stability [3–8, 14–17]. Dif-90 ferent types of athermal resonators such as microspheres, 91 microdisks, microtoroids and microrings have been studied 92 and fabricated already [18, 19]. One intriguing optical reso-93 94 nator [18] is based on a self-assembled rolled-up microtube (RUM) resonator. RUM resonators are easily integrated into 95 coupled arrays, conveniently defined in materials design by 96 97 thin film deposition techniques, and efficiently deployed with high sensitivity due to their hollow cores and ultrathin 98 walls [20-28]. RUMs can be used for a broad range of pho-99 tonic applications, including optofluidic components, opti-100 cal signal processing and metamaterial devices [20, 22, 24, 101 25, 29, 30]. Like other photonic devices, the optical signals 102 generated by RUMs are extremely vulnerable to changes in 103 temperature, owing to their ultrathinness [24, 25]. 104

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In this paper, we propose for the first time the use of 105 athermal rolled-up microtube (RUM) ring resonators to 106 overcome temperature sensitivity. We suggest hybrid 107  $TiO_2$ -SiO<sub>2</sub> and/or  $TiO_2$ -Si<sub>3</sub>N<sub>4</sub> RUMs as athermal resonators 108 for optical filtering, which can greatly improve the thermal 109 stability of TiO<sub>2</sub>-based RUMs by incorporating SiO<sub>2</sub> and/ 110 or  $Si_3N_4$ , which possess a positive TOC and therefore the 111 ability to compensate the commonly observed blueshift of 112 TiO<sub>2</sub> RUM resonators. 113

To reach this goal, the general scheme of an athermal 114 RUM resonator for optical filtering is outlined in Sect. 2. The 115 theory of effective thermo-optic coefficients is presented in 116 Sect. 3. In Sect. 4, the temperature sensitivity of TiO<sub>2</sub> RUMs 117 for different temperatures and wall thicknesses is theoreti-118 cally studied. Furthermore, the shift in optical resonances 119 as a function of SiO<sub>2</sub> thicknesses and Si<sub>3</sub>N<sub>4</sub> thicknesses 120 with a fixed thickness of TiO<sub>2</sub> is investigated. In Sect. 4, the 121 optical filtering performance of photonic RUMs, including 122 wavelength shift and extinction ratio, is analyzed by a three-123 dimensional finite difference time domain (FDTD) method. 124 Finally, as a proof of concept, an experiment is demonstrated 125 by fabricating RUMs based on TiO<sub>2</sub>-SiO<sub>2</sub> on the flat silicon 126 wafer as substrate. To study the (in)sensitivity of the optical 127 resonance modes to the temperature, the fabricated devices 128 are excited under different input powers and the experi-129 mental results show that the non-shift of the optical modes 130 is achieved, which may be a result of having an athermal 131 hybrid RUM based on the appreciated thickness of these 132 two materials. This novel idea is an important milestone in 133 the implementation of vertical and multi-routing coupling 134 between photonic layers in three optoelectronic devices, 135 where the stability of the optical signals is really essential. 136

# 2 Scheme of the on-chip hybrid (athermal) rolled-up microtube ring resonator

A 3D layout of an optical filter based on an athermal hybrid 139 RUM resonator is proposed in Fig. 1a, which is monolithically 140 integrated with a nanophotonic waveguide. This suggested 141 device is composed of a hybrid RUM, which is coupled to a 142 straight single-mode silicon waveguide located on the substrate 143 surface. Similar to our previous works [25, 31], the proposed 144 device can be easily fabricated on a silicon wafer coated with 145 SiO<sub>2</sub>. By following the parameters from our previous works, 146 in this theoretical work, on-chip silicon waveguides are set to 147 a 220-nm-thick silicon layer on 4-µm-thick SiO<sub>2</sub> to avoid any 148 coupling light to the substrate. To experimentally study the 149 temperature dependence of our novel hybrid resonator, it is 150 feasible to implement the electrodes next to the waveguides 151 and integrate them with the RUM (see outlook for more details 152 and/or [28]). In this scheme, the electrode can be designed on 153 top of the 2D nanomembrane, and when the nanomembrane is 154

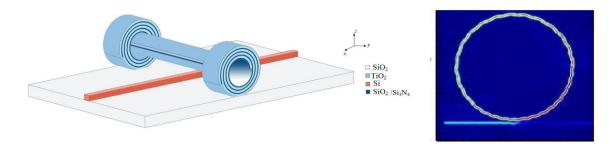


Fig. 1 Schematic of the proposed athermal optical filter based on an RUM, composed of a TiO<sub>2</sub>/SiO<sub>2</sub> (TiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub>) bilayer, positioned on top of a straight silicon waveguide on an SiO<sub>2</sub> substrate

rolled up, the electrodes are monolithically integrated within 155 the tube windings [29, 32]. However, in our theoretical inves-156 tigation, we simply change the temperature in the software 157 used, which leads to changes in the refractive index that are 158 then automatically considered in our calculation. We simply 159 used the FDTD solution of LUMERICAL software. In this 160 software, there is a model for each material that the TOC can introduce. Moreover, there is the possibility of adjusting the operating temperature to the desired value for this quantity.

164 For example, if the device must be simulated at a temperature of 312 K, the operating temperature is set to 312, whereas 165 the TOC of the material is constant. In the system under study, 166 167 the silicon waveguide with a cross section of  $400 \times 210 \text{ nm}^2$ was chosen to realize single-mode transmission. Furthermore, 168 16- $\mu$ m TiO<sub>2</sub>-SiO<sub>2</sub> (TiO<sub>2</sub>-Si<sub>3</sub>N<sub>4</sub>) RUM in the free-standing part 169 was utilized. The parameters were tailored such that the effec-170 tive index of the RUM ( $n_{\rm eff}$ =2.076 at  $\lambda$ =1.55 µm) matched the 171 effective index of the single-mode silicon waveguide. The opti-172 cal profile of this proposed athermal TiO<sub>2</sub> RUM-based filter is 173 depicted in Fig. 1b, where the temperature is set to room tem-174 perature (300 K) and the resonant wavelength is 1546.84 nm. 175 A 60-nm gap between athermal RUM and silicon waveguide 176 is engineered for a maximum transmission coefficient [26, 27]. 177

#### 3 Theory of effective thermo-optic 178 coefficient (TOC) 179

It is known that thermal heating induces a temperature-180 dependent refractive index change in the constituent materials. 181 For the RUM resonator, changing the temperature leads to a 182 change in the index of the resonator material and consequently 183 leads to a shift in the resonant wavelength  $\lambda_0$  toward a shorter 184 (blueshift) or longer (redshift) wavelength. The shift with 185 respect to temperature, which is called a temperature-induced 186 resonant shift (TIRS), is given by [9]: 187

$$\frac{\mathrm{d}\lambda}{\mathrm{d}T} = \frac{\partial n_{\mathrm{eff}}}{\partial T} \frac{\lambda_0}{n_o},\tag{1}$$

where  $\lambda_0$  is the resonant wavelength,  $n_{\rm eff}$  is the effective 189 index, T is the temperature, and  $n_{o}$  is the group index. In 190 Eq. 1, we assume that the optical mode is tightly confined 191 within the tube wall. Therefore,  $n_{\rm eff}$  corresponds to the effec-192 tive refractive index of RUM by considering power confine-193 ment within individual regions such as the tube wall, hol-194 low core, and surrounding region. Owing to the asymmetric AQ6 5 geometry of RUM (see Fig. 1a), the analytically calculated 196 values need to be considered for three parts of the RUM (i.e., 197 the two leg parts and a middle part). By taking advantage 198 of the coupling area between RUM and optical waveguide, 199 only the middle part of RUM needs to be considered, which 200 was already analytically calculated in our previously pub-201 lished works [9, 12]. It should be noted that the terms in 202 Eq. 1 are wavelength dependent. To decrease or eliminate 203 the temperature dependence of the RUM, it is suggested to 204 use materials with the opposite thermo-optic coefficient in 205 planar grown bilayers, which can be a promising scheme to 206 achieve athermalization. In this regard, the effective index 207 of the whole RUM structure will change in Eq. 1. There-208 fore, the effective thermo-optic coefficient of the RUM wall 209 before rolling up is 210

$$n_{\rm eff} = \Gamma_{\rm o} n_{\rm o} + \Gamma_{\rm a} n_{\rm a}, \tag{2}$$

where  $\Gamma_0$  and  $\Gamma_a$  are modal confinement factors for original and athermal layers, and  $n_0$  and  $n_a$  are refractive indices 213 of these regions, respectively. Consequently, the effective 214 thermo-optic coefficient becomes 215

$$\frac{\partial n_{\rm eff}}{\partial T} = \Gamma_{\rm o} \frac{\partial n_{\rm o}}{\partial T} = \Gamma_{\rm a} \frac{\partial n_{\rm a}}{\partial T}.$$
(3)

In this work, the modal confinement and positive TOC of the 217 athermal layer (SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>) are engineered to balance the 218 modal confinement and negative thermo-optic coefficients 219 of the RUM wall (TiO<sub>2</sub>). 220

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# 3.1 Filtering performance, thermal stability,and athermalization

223 To filter the specific wavelength using conventional (and/or athermal) RUM, a winding number and asymmetric param-224 eter should be engineered. For optical filtering, standard 225 wavelength in telecom band (O-band to C-band), asymmet-226 ric parameter, and wall thickness of TiO<sub>2</sub> RUM are con-227 sidered  $5\pi/4$  and 345 nm, respectively. Furthermore, it has 228 already been found that the TOCs of TiO<sub>2</sub>, SiO<sub>2</sub>, and Si<sub>3</sub>N<sub>4</sub> 229 are  $-4.5 \times 10^{-5}$ /K [12],  $+2 \times 10^{-5}$ /K [11], and  $+4 \times 10^{-5}$ /K 230 [9], respectively. All simulations are based on the 66th azi-231 muthal mode and the first radial mode. 232

When the device serves as an optical filter, a broadband 233 light (including  $\lambda_1 \dots \lambda_i \dots \lambda_n$  wavelengths) from the input port 234 coupled to the RUM, then the specific wavelength (i.e.,  $\lambda_i$ )— 235 which is called resonance mode-separates from the input 236 signal according to the resonance condition and also the 237 238 properties of the RUM along the axial direction, and finally sends the rest of the input wavelengths  $(\lambda_1 \dots \lambda_{i-1}, \lambda_{i+1} \dots \lambda_n)$  to 239 the through port. Figure 2a shows the influence of tempera-240 241 ture on the transmission spectrum of an optical filter based on 16 µm TiO<sub>2</sub> RUM with three winding numbers (for more 242 information about our simulation technique, see [26-28]). AQ7As illustrated in Fig. 2a, the wavelength 1546.84 nm is fil-244 tered at 300 K (room temperature), and by increasing the 245 temperature to 312 K, the filtered wavelength experiences 246 blueshift (i.e., shift to the shorter wavelength) because of 247 the negative TOC of TiO<sub>2</sub>. Hence, the effect of tempera-248 ture on the resonant wavelength for an RUM based on TiO<sub>2</sub> 249 with a diameter of 16 µm at the different winding numbers 250 is presented in Fig. 2b. This figure indicates that when the 251

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temperature increases, the resonance modes are clearly shifted to the shorter wavelength.

As can be easily calculated, the resonance peaks as a func-254 tion of temperature yields a rate of  $d\lambda_0/dT = -33.3$  pm/K for 255 the different winding numbers, which corresponds to a dn/ 256  $dT = 5.8 \times 10^{-5} \text{ k}^{-1}$ . As mentioned above, this blueshift in 257 the resonant wavelength results from the negative TOC of 258 TiO<sub>2</sub>. Moreover, a variation in the resonant wavelengths as 259 a function of winding numbers is shown in Fig. 2b. This 260 phenomenon leads to the much larger effective refractive 261 index, which will result in higher mode confinement. Since 262 the resonant wavelength varies directly in relation to the 263 effective refractive index, it can be observed that the reso-264 nant wavelength increases as the wall thickness increases. In 265 Fig. 3, resonant modes characteristic of the athermal  $TiO_2$ 266 microtube are represented by overlaying an SiO<sub>2</sub> layer on 267 TiO<sub>2</sub> bilayer membrane at different temperatures (which vary 268 between 304 and 312 K). As a result, by increasing the thick-269 ness of the SiO<sub>2</sub> layer a redshift in the resonant wavelengths 270 occurs due to the positive TOC of SiO<sub>2</sub> ( $+2 \times 10^{-5}$ /K), as 271 well as increasing the whole thickness of the tube wall. 272

It is also worth mentioning that the effective refractive 273 index in TiO<sub>2</sub>/SiO<sub>2</sub> RUM is not the same as pure TiO<sub>2</sub> 274 RUM, because of the dissimilar refractive indices of pure 275  $TiO_2$  ( $n_{TiO_2} = 2.45$ ) and  $SiO_2$  ( $n_{SiO_2} = 1.44$ ). As a result, 276 it can be explained why the resonant wavelengths are dif-277 ferent in these two types of RUMs (pure TiO<sub>2</sub> RUM and 278 hybrid TiO<sub>2</sub> RUM) such that the TiO<sub>2</sub>-SiO<sub>2</sub> RUM with a 279 thicker wall presents a shorter resonant wavelength shift 280 than RUM composed only of TiO<sub>2</sub> with 345-nm thickness 281 (because the effective refractive index of hybrid RUM is 282 much smaller than that of pure TiO<sub>2</sub> RUM). Therefore, 283

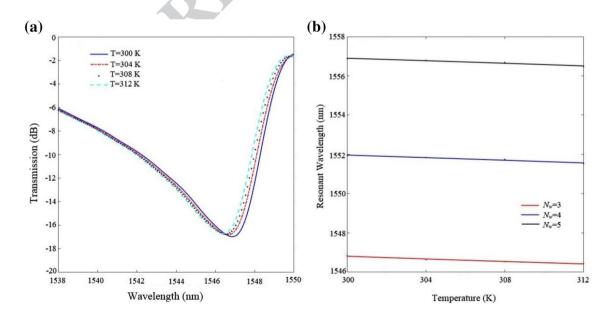


Fig. 2 a Transmission spectrum of an 8-µm-radius TiO<sub>2</sub> with wall thickness of 345 nm at different temperatures

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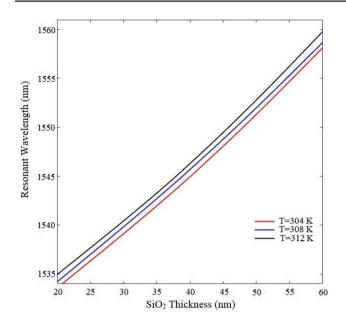


Fig. 3 Variations of the resonant wavelength of TiO<sub>2</sub>-SiO<sub>2</sub> microtube versus the thickness of SiO<sub>2</sub> layer with a constant TiO<sub>2</sub> thickness of 115 nm

Table 1Shift in resonantwavelength of $TiO_2$ microtubeby utilizing different thicknessesof the $SiO_2$ layer between 40	SiO <sub>2</sub> thickness (nm)	Resonant wavelength (nm)
and 45 nm at 304 K	41	1545.74
	42	1545.54
	43	1546.05
	44	1546.84
	45	1547.74
<b>Table 2</b> Shift in resonant wavelength of $TiO_2$ microtube by utilizing different thicknesses of the $SiO_2$ layer between 40	SiO <sub>2</sub> thickness (nm)	Resonant wavelength (nm)
and 45 nm at 308 K	41	1545.52
	42	1546.04
	43	1546.84
×	44	1547.44
	45	1548.2

the range of thickness of SiO<sub>2</sub> layers for athermalization 284 must be increased from 40 to 45 nm, as shown in Fig. 3. 285 Further simulation results are presented in Tables 1, 2, 286 and 3 (see "Appendix"). It is indicated that to generate an 287 athermalization behavior in our hybrid RUM, the appreci-288 ated thickness of SiO<sub>2</sub> needs to be 44 nm at 304 K, 43 nm 289 at 308 K, and 41 nm at 312 K. 290

<b>Table 3</b> Shift in resonantwavelength of $TiO_2$ microtubeby utilizing different thicknessesof the $SiO_2$ layer between 40	SiO <sub>2</sub> thickness (nm)	Resonant wavelength (nm)
and 45 nm at 312 K	41	1546.85
	42	1547.54
	43	1548.34
	44	1549.04
	45	1549.95

A trend in the temperature-induced resonant shift 291 (TIRS) in the athermal TiO<sub>2</sub>/SiO<sub>2</sub> RUM versus the thick-292 ness of the SiO<sub>2</sub> layer is plotted in Fig. 4a, wherein the 293 total thickness of bilayers remains constant at 115 nm. As 294 can be seen in this figure, by increasing the thickness of 295  $SiO_2$  from 5 up to 30 nm, the resonant wavelength shifted 296 toward the longer wavelengths. As the results from TIRS AQ8 show, it can be increased from a range of -40 pm/K to 298 about 30 pm/K. This figure proposes that by choosing the 299 thickness of SiO<sub>2</sub> around 16 nm, TIRS compensation can 300 be well achieved. 301

Furthermore, athermalization of TiO<sub>2</sub> RUM utilizing 302  $Si_3N_4$ , whose refractive index  $(n_{Si_2N_4} = 1.99)$  is much 303 closer to the refractive index of TiO<sub>2</sub> (at the telecom wave-304 length), is shown in Fig. 4b. In this figure, the total thick-305 ness of bilayers is still 115 nm. Figure 4b shows the TIRS 306 under the influence of changing the thickness of  $Si_3N_4$  for 307 the hybrid TiO<sub>2</sub>–Si<sub>3</sub>N<sub>4</sub> RUM. 308

As expected, TIRS compensation occurs with a smaller 309 thickness of  $Si_3N_4$  ( $t_{Si_3N_4} = 12 \text{ nm}$ ) as compared with  $SiO_2$ , 310 owing to the closer refractive index of Si<sub>3</sub>N<sub>4</sub> with respect 311 to  $TiO_2$ . Additionally, more light confinement between 312 these layers is elegantly expected. By comparing the 313 athermalizing behavior of these two hybrid RUMs (i.e., 314 TiO<sub>2</sub>-SiO<sub>2</sub> and TiO<sub>2</sub>-Si<sub>3</sub>N<sub>4</sub> RUMs), it is found that the 315 smoother variations of TIRS with respect to the thick-316 ness of the athermal layer can be achieved by selecting 317 the material which has a closer refractive index to the 318 original planar bilayers of an RUM. As a conclusion and 319 prior to the experimental part, the TOC of both materi-320 als has already been known (i.e., TiO<sub>2</sub> and SiO<sub>2</sub>), and by 321 following  $\frac{dn_{\text{eff}}}{dT}$  (and/or  $\frac{d\lambda_0}{dT}$ ) and wavelength shift ( $\Delta\lambda$ ), it is 322 feasible to estimate the value for the temperature. Based AQ9 3 on the calculations, when the input power was  $85\mu$ W, the 324 temperature within the RUM will be estimated to be 25 °C 325 and at 1.7 mW changed to around 65 °C. 326

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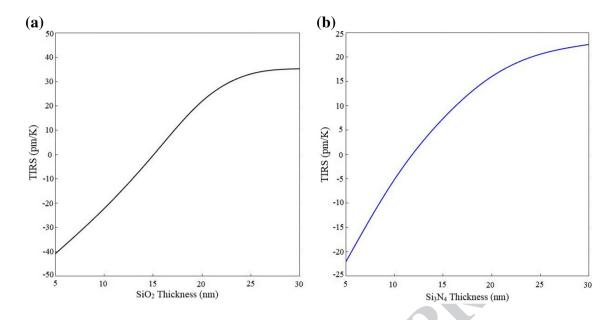


Fig. 4 a Temperature-induced resonant shift (TIRS) of  $TiO_2$ -SiO<sub>2</sub> RUM as a function of the thickness of SiO<sub>2</sub> layer, while the total thickness of the RUM remains constant at 115 nm

### 327 4 Experimental part

# 4.1 Fabrication of the hybrid (athermal) RUMs based on TiO<sub>2</sub>-SiO<sub>2</sub>

A theoretical study of athermal resonators-based on-chip hybrid RUMs was conducted, followed by an investigation into how the thickness variation of  $SiO_2$  and/or  $Si_3N_4$ within the  $TiO_2$  RUMs' walls can influence the performance of these proposed devices. In this section, as a proof of concept, the fabrication of the isolated (i.e., not integrated with photonic waveguides) athermal TiO<sub>2</sub> 336 RUMs in combination with SiO<sub>2</sub> is presented. To study the 337 temperature-(in)sensitive resonance modes, the fabricated 338 devices are excited using a continuous wavelength (CW) 339 laser ( $\lambda = 442$  nm) under different exciton powers. 340

The devices are fabricated using an elegant well-established roll-up method which has been discussed extensively in the literature [20–25, 29–31, 33–36]. A schematic of the fabrication procedure is shown in Fig. 5. Standard photolithography was used to fabricate an array of U-shaped [25, 31, 33, 35] photoresist (AR-P 3510, Allresist GmbH) patterns on an Si substrate [25, 35]. The photoresist pattern 347

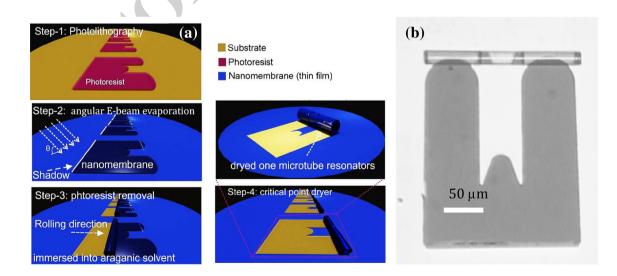


Fig. 5 a Schematic of the fabrication procedure of microtubes from a photoresist sacrificial layer

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348 (with a thickness of around 2  $\mu$ m) defines the final shape of 349 the RUM and serves as a sacrificial layer.

Then, a 115-nm-thick layer of differentially strained 350 TiO<sub>2</sub>-SiO<sub>2</sub> is deposited on the surface using tilted elec-351 tron beam evaporation [20, 25, 31, 33]. It is worth men-352 tioning that in this fabrication process, TiO<sub>2</sub> was deposited 353 from pure titanium pellets (Kurt J. Lesker Company) in an 354 oxygen background (the pressure inside the chamber was 355  $2.6 \times 10^{-4}$  mbar), with strain being generated by deposit-356 ing parts of the layer at different rates. To fabricate the first 357 sample (called "device no. 1" in this paper), the SiO<sub>2</sub> layer 358 was deposited with a low (about  $\approx 0.3$  Å/s) deposition rate 359 and a thickness of 15 nm, and then the TiO<sub>2</sub> layer (with a 360 thickness of 100 nm) as second layer was deposited with a 361 high (about  $\approx 3.4$  Å/s) deposition rate. For fabricating the 362 second sample (called "device no. 2" in this paper), the SiO<sub>2</sub> 363 layer was deposited with a low (about  $\approx 0.4$  Å/s) deposition 364 rate and a thickness of 25 nm, and then the TiO<sub>2</sub> layer (with 365 a thickness of 90 nm) as a second layer was deposited with 366 a high (about  $\approx 3.8$  Å/s) deposition rate. The photoresist was 367 then dissolved with dimethyl sulfoxide (DMSO; VWR Inter-368 national S.A.S.), leading to relaxation of the nanomembrane, 369 which is rolled up into RUMs with an average diameter of 370 10 µm, and then dried in a critical point dryer to avoid col-371 lapse due to surface tension. It is worth mentioning that after 372 rolling, the tube sits on the vertical wall formed by the mate-373 rial deposited on the side of the sacrificial layer. This fea-374 ture is important to avoid light losses to the substrate when 375 the microtube is employed as an optical microcavity. As an 376 example, Figs. 5b and 6b show the top of the viewed optical 377 images of the RUMs (with different lobs) fabricated in this 378 manner. In both figures, the outline of the U-shaped pattern 379 is clearly visible. In the inset of Fig. 6b, details of the RUMs 380 are shown which highlight the high quality of the fabrication 381 process. The membrane is rolled tightly-so that the wind-382 ings are in contact with each other-and completely. The 383 bridge-like middle segment is elevated above the substrate 384 to prevent optical leakage, and the axial mode-inducing 385

lobe-like patterns on the middle segment and both ends are clearly defined.

### 4.2 Optical characterization and results

In this work, the optical characterization (i.e., excitation and 389 detection of optical resonant modes) from the fabricated 390 athermal microtubes (with different thicknesses of  $SiO_2$ ) is 391 performed using a commercial confocal laser µ-PL spectros-392 copy at room temperature, supplied by Renishaw inVia, as 393 schematically depicted in Fig. 6a. It should be remarked that 394 a  $\mu$ -PL experiment is a free-space approach for excitation of 395 the resonance modes within the RUMs. It has been reported 396 in [20, 31, 37] that the origin of PL emission in these fab-397 ricated devices is likely silicon nanocrystals embedded in 398 SiO<sub>2</sub> for light, and also trace amounts of residual photoresist 390 on the sample and/or unspecified defect centers similar to 400 those found in silica. 401

However, it is proof that the titanium dioxide material of 402 the tube wall has very poor PL properties [31]. However, this 403 drawback of using TiO<sub>2</sub> microtubes can be easily addressed 404 by using SiO<sub>2</sub> as another layer of the tube's material. A  $\mu$ -PLAQ11 <sub>15</sub> setup was performed using an He-Cd laser with an excita-406 tion line of 442 nm. The pump beam is routed and focused 407 through a  $50 \times$  microscopic objective lens (with a spot size 408 of  $\approx 1 \,\mu\text{m}^2$ ) onto the fabricated RUMs, which is mounted 409 to a precision XYZ stage. The luminescence signals from 410 the examined tube are collected by the same objective and 411 diffracted in an optical spectrometer with 1200 lines/mm. 412 An electrically cooled charge-coupled device (CCD) camera 413 was used for spectral analysis. 414

After exciting the athermal RUM (device no. 1 or device 415 no. 2) using the incident laser beam, the silicon nanocrystals embedded in the SiO<sub>2</sub> within the tube wall emit light 417 which can propagate and circulate in the ring-like microtube nanomembrane wall. When the wavelength of the emitted light satisfies the constructive interference condition  $\pi D = m\lambda/n$  (where *D* is the tube diameter, m is the integer 421

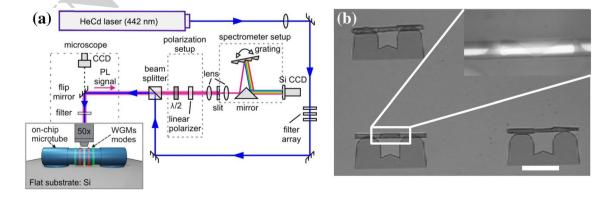


Fig. 6 a Schematic illustration of the micro-PL setup to excite and detect optical resonances

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mode number,  $\lambda$  is the resonant wavelength, and n is the refractive index of the tube wall), optical resonance modes are formed as the peaks on top of the PL background in a fixed spectral range from 550 to 850 nm. To show the results more clearly, only small parts of this large spectral range are shown in Figs. 7b and 8b as different colors under different excitation powers, respectively.

The set of the peaks shown have FSRs = 10 nm and 8 nm, respectively, which is the difference between two adjacent fundamental peaks that is in good agreement with the calculated value (by the simple equation  $FSR = \frac{\lambda_{\text{res}}^2}{n\pi D}$ , where n is the refractive index, D is the diameter of RUM, and  $\lambda_{\text{res}}$  is the wavelength of the resonant mode). Apart from the observation of resonance modes from the athermal RUMs (device no. 1), as shown in Fig. 8b, the device (with a thickness of 15 nm of SiO<sub>2</sub> within the tube's wall) was excited with different powers using a  $\mu$ -PL setup. Figure 7b displays the results of a less than 2 nm shift in the optical modes at different powers (blueshift or shot to the shorter 440 wavelength). 441

Because of the negative TOC of the TiO<sub>2</sub>, this blueshift 442 can occur. Based on our theoretical work, we find that it is 443 necessary to increase the thickness of SiO<sub>2</sub> to overcome this 444 blueshift. By following the apricated thickness of the TiO<sub>2</sub> 445 and SiO<sub>2</sub> (i.e., SiO<sub>2</sub> layer with a thickness of 25 nm and 446 then the TiO<sub>2</sub> layer with a thickness of 90 nm, respectively), 447 now the fabricated device acts as the athermal microtube. 448 Optical characterization of the fabricated device no. 2 shows 449 non-shift of resonance modes, while the exaction power of 450 the laser increases (or in other words, while the tempera-451 ture of the tube increases). These non-shifted modes can be 452 explained by the fact that the negative TOC of TiO<sub>2</sub> causes 453 a shift to the shorter wavelengths (a blueshift) around 2 nm, 454 while the positive TOC of SiO<sub>2</sub> with the apricated thick-455 ness (i.e.,  $t_{SiO2} = 25$  nm) leads to an equal shift ( $\Delta \lambda = 2$  nm) 456 but toward the longer wavelengths (a redshift). Thus, in the 457

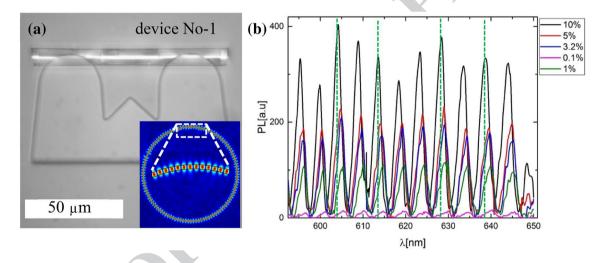


Fig. 7 a Top view optical microscopy image of a hybrid RUM (with a thickness of 15 nm of SiO<sub>2</sub> and 100 nm of TiO<sub>2</sub> layer)

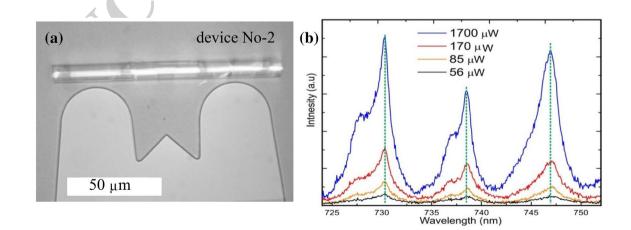


Fig. 8 a Top view optical microscopy image of an athermal RUM (with a thickness of 25 nm of SiO<sub>2</sub> and 90 nm of TiO<sub>2</sub> layer)

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experiment, it is proven that the unwanted shift can easily 458 disappear when the apricated thickness ratio between two 459 materials is selected, as shown in Fig. 9b. Additional opti-460 cal measurements are performed for other athermal devices, 461 exhibiting similar results to those presented in Fig. 9b. 462 This validates the feasibility of athermal mass production 463 of RUMs. In our work, we used  $TiO_2$ -SiO<sub>2</sub> as an example 464 material, but the fabrication process can be easily adapted 465 to RUMs made out of other materials such as  $TiO_2$ -Si, 466 TiO<sub>2</sub>-SiOX, TiO<sub>2</sub>-SiN<sub>x</sub>, or TiO<sub>2</sub>-Y2O<sub>3</sub>. 467

#### 4.3 Future outlook 468

Fig. 10 a Top view optical

from Ref [25]

microscopy image of a TiO<sub>2</sub> RUM fully integrated with nano-waveguides on a single chip. Taken with permission

In this paper, monolithic integration of athermal RUMs on photonic waveguides is proposed and theoretically investi-470 gated. Apart from this idea, it would be of great interest to experimentally demonstrate and fabricate this device, which would be a crucial ingredient for advanced optoelectronic applications such as 3D stacked chip integration.

Figure 9 represents an artistic view of an on-chip athermal rolled-up microtube which is monolithically

integrated with optical waveguides, as an example. As 477 shown in Fig. 9a, the 2D nanomembrane can be designed 478 well on top of the electrode, and it is rolled up together 479 with the electrodes to form a tubular shape [37]. In this 480 way, the electrodes are monolithically integrated with tube 481 windings (see Fig. 9b). It is worth stating that in recent 482 years, a step forward in integrating the rolled-up micro-483 tube onto planar waveguides or other photonic as well as 484 integration electrodes within tube winding has been real-485 ized by our group and other research groups (see Fig. 10 486 as an example). 487

To achieve a fully integrated system with a low cost and ultracompactness, and to use it in different areas, more attention should be paid to this proposal for bringing these two possibilities (i.e., integrating waveguides and electrodes into one single tube) within a single athermal RUM, as the next step.

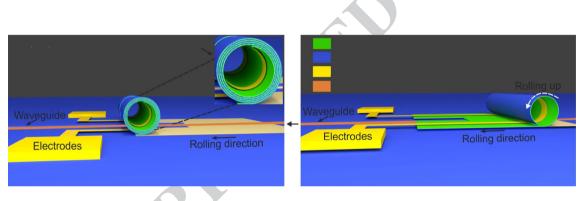
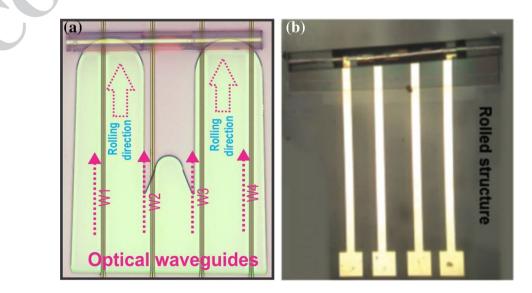


Fig. 9 a and b Schematic of the proposed athermal optical filter based on an RUM, composed of a TiO<sub>2</sub>/SiO<sub>2</sub> (TiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub>) bilayer, positioned on top of a straight silicon waveguide and electrodes on an SiO<sub>2</sub> substrate



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# 494 **5 Conclusion**

One important concern in integrated optics is thermal heat-495 ing, which leads to unwanted wavelength shifts in the inte-496 grated system. This shift is often unsuitable for important 497 applications: for example, optical filtering. To this end, it 498 is necessary to fabricate athermal resonators instead of the 499 conventional resonators in the integrated system. In this 500 study, an athermal TiO<sub>2</sub> RUM with SiO<sub>2</sub> (Si<sub>3</sub>N<sub>4</sub>) layer is 501 proposed and designed for filtering with thermal stabil-502 ity. The transmission spectra and filtering performance of 503 TiO<sub>2</sub> RUM are investigated at different temperatures. The 504 simulation results indicate that the negative TOC of TiO<sub>2</sub> 505 leads to a blueshift in resonant wavelength with different 506 wall thicknesses; consequently, the rate of wavelength shift 507 508 is -33.3 pm/K. To compensate for the shift in wavelength, the behavior of TIRS in an athermal  $TiO_2/SiO_2$  ( $TiO_2/Si_3N_4$ ) 509 RUM versus an SiO<sub>2</sub> (Si<sub>3</sub>N<sub>4</sub>) layer thickness—where SiO<sub>2</sub> 510 and Si<sub>3</sub>N<sub>4</sub> are positive TOC materials—is studied. The 511 results show that increasing SiO<sub>2</sub> (Si<sub>3</sub>N<sub>4</sub>) thickness results 512 in redshift of the resonant wavelengths, and this increment 513 leads to increasing TIRS in the ranges of -40 pm/K and 514 about 30 pm/K, and - 22 pm/K and 22 pm/K, for TiO<sub>2</sub>/SiO<sub>2</sub> 515 and TiO<sub>2</sub>/Si<sub>2</sub>N<sub>4</sub> RUMs, respectively. Also, compensation is 516 achieved at a thickness of ~16 nm and 12 nm for the  $SiO_2$ 517 and Si<sub>3</sub>N<sub>4</sub> layers respectively. 518

Finally, for a proof of concept, an experiment is made on fabricating RUMs based on TiO2/SiOx on the flat silicon wafer as substrate. They were excited with different powers using a  $\mu$ PL setup. The result confirms the athermal idea by displaying the non-shift of the optical modes at different powers.

# 525 Appendix

In this section, athermalization of  $TiO_2/SiO_2$  RuM by modifying SiO<sub>2</sub> thickness when the thickness of  $TiO_2$  layer is 115 nm is investigated in Tables 1, 2, and 3 for operating temperatures 304 K, 308 K, and 312 K, respectively.

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