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## Fine-tuning of whispering gallery modes in on-chip silica microdisk resonators within a full spectral range

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We investigate an efficient method for fine-tuning whispering gallery mode resonances in disk-type silica microresonators to reach an arbitrary frequency within the free spectral range of the system. This method is based on a post-production hydrofluoric acid etching process to precisely resize the radius of such microresonators. We show the effectiveness of this approach by tuning their resonance frequency within 10 GHz of specific hydrogen cyanide reference lines (P16, P18). This technique allows for simple and exact matching of narrow-linewidth lasers or spectroscopic lines with the high-Q resonances of on-chip silica microresonators. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4789755>]

Resonant optical structures which are able to efficiently store and filter out light are a key requirement in photonics. For applications requiring high transmission in the visible and ultraviolet spectral ranges, silica-based systems are favored due to their high purity and the well-developed silicon technology based processing techniques. In silica, mainly whispering gallery mode (WGM) microresonators in the form of toroids<sup>1</sup> or simple disks and wedges<sup>2</sup> are used. With these types of microresonators, ultimate Q factors in the range of  $10^9$  have recently been shown.<sup>3</sup>

The resonance frequency of WGM microresonators is described by

$$2 \cdot \pi \cdot r_{\text{eff}} \cdot n_{\text{eff}} = m \cdot \lambda. \quad (1)$$

Here,  $r_{\text{eff}}$ ,  $n_{\text{eff}}$  are the effective radius and effective refractive index of the resonator,  $m$  is an integer number corresponding to the number of field maxima around the circumference, and  $\lambda$  is the wavelength of the circulating light. The free spectral range (FSR) can be calculated from Eq. (1) by

$$FSR = \frac{\lambda^2}{2 \cdot \pi \cdot r_{\text{eff}} \cdot n_{\text{eff}}}. \quad (2)$$

One of the main problems for optical microresonator applications is an insufficient tuning range combined with FSRs in the order of nanometers. For coupling a narrow-band light source, e.g., a specific laser or spectral emission line, into the resonator the radius of the system has to be carefully defined. For example, attempts to couple the narrow zero-phonon line of color centers in diamond to WGMs have been reported by several groups,<sup>4,5</sup> but a required reliable tuning method could not be realized so far.

Directly matched manufacturing is not possible in the case of high-Q cavities. Assuming an operational wavelength of 1550 nm and a targeted maximum resonance deviation

of  $\pm 0.1$  nm ( $\pm 11$  GHz), it follows from Eq. (1) that in silica the minimum fabrication precision of disks has to be in the order of  $\pm 0.006\%$ . This high level of accuracy is impossible to achieve with standard up-to-date lithographic methods. The lithographic structuring and the subsequent etching processes both produce random deviations which dominate the achievable final precision. Furthermore, the actual resonance position of a microresonator also depends on other parameters like environmental temperature or humidity. For practical applications, additional active tuning methods have to be used in any case to stabilize a resonance even for under specific conditions perfectly matched resonators.

Several fine-tuning methods were developed to lock high-Q WGM resonances to lasers.<sup>6</sup> The most common technique for tuning silica WGM microresonators is changing the dimension of the resonator through a combination of thermo-optic and thermo-mechanic effects.<sup>7,8</sup> At room temperature, this method is limited to a tuning range of  $\pm 50$  GHz.<sup>9</sup> In a cryostat also controlled nitrogen gas adsorption on the surface of a resonator can be used to change the effective radius of a WGM.<sup>10</sup>

In this paper, we investigate a complementary tuning approach which allows for a permanent coarse frequency tuning with a maximum accuracy of  $\pm 5$  GHz. This is done by applying an additional etch step<sup>11</sup> after an initial measurement of the resonance frequency. It expands the tuning range over the complete FSR. Subsequent reversible thermal fine-tuning is then used for matching a specific frequency line with high precision. We perform quantitative measurements in order to determine the resulting etch rate and the influence on the cavity Q factor. The effectiveness of our combined approach is illustrated by tuning two microdisk resonators with arbitrary resonance frequencies to match specific telecommunication C-band hydrogen cyanide (CHN) reference lines. In contrast to tunable microspheres,<sup>11</sup> silica disk-type microresonators are fabricated by a scalable lithographic process. This allows for design of highly integrated photonic systems with large arrays of resonators even including

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integrated waveguide couplers. Here, we complement the large-scale fabrication process with an efficient and precise fine-tuning step. We would like to point out that direct monitoring of resonances on-chip automatically accounts for possible shifts due to interactions with the substrate and other integrated elements.

For analyzing the mode structure of the microresonators, the light from a tunable external cavity diode laser (New Focus Velocity Series) with a wavelength between 1520 nm and 1570 nm was coupled into the resonator via the evanescent field of a tapered optical fiber. The laser combines a small linewidth (300 kHz) with a large mode-hop free scanning range (up to 30 GHz). The fiber tapers had diameters of approx. 3  $\mu\text{m}$  and allowed for stable coupling under contact and non-contact conditions. Disk-type silica microresonators with diameters between 40 and 60  $\mu\text{m}$  were used. The silica layer had a thickness of 2  $\mu\text{m}$  and was thermally oxidized on a silicon substrate. The microdisks were 10  $\mu\text{m}$  underetched at the rim such that the separation from the substrate was nearly 20  $\mu\text{m}$ . A scanning electron micrograph of a typical microdisk resonator with 25  $\mu\text{m}$  radius is shown in Fig. 1(a).

By shifting the frequency of the laser over the distinct WGMs, light was coupled into the resonator and the modes were observed as Lorentzian shaped dips in the transmitted laser power. For measuring the actual resonance position of a WGM, the laser was shifted to the middle of that resonance (i.e., to the middle of the Lorentzian dip) and a wavemeter (Burleigh WA-1500) was used for an exact frequency determination.

At a wavelength around 1550 nm, the FSR of our microresonators is 10.6 nm. At this wavelength, the resonators show single mode behavior and only two orthogonal polarization modes are observable within one FSR. This highly simplifies the need for re-identification of the modes after subsequent etching steps. The resonances of several resonators scatter widely within one FSR. This is shown in Fig. 1(b) for resonators with the same nominal diameter all on one chip.

During such a measurement series, the highest Q factor we observed in our resonators was  $10^6$  with a linewidth of around 200 MHz. The measured Q factors are comparable with the results of other groups achieved in directly etched chip-based microresonators with similar size.<sup>3</sup> Higher Q factors are only possible within silica resonator designs with a surface tension induced thermal reflow polishing, e.g., in microtoroids or microspheres. However, these designs are

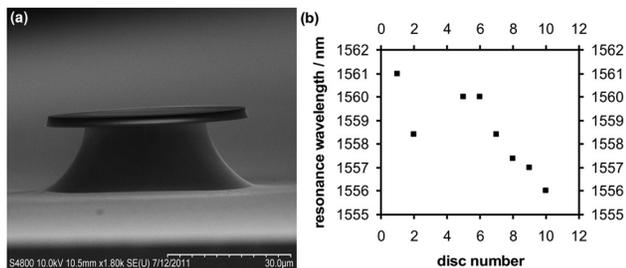


FIG. 1. (a) Scanning electron micrograph of a silica microdisk with 25  $\mu\text{m}$  radius. (b) Measured resonance frequencies of several silica disk-type microresonators all produced in the same etching process. The scattering of these values is well within one FSR of the system.

not compatible with standard clean room process lines and require additional laser treatment. Another option to achieve higher Q factors is to reduce the radiation losses using resonator designs with much larger effective radii.<sup>3</sup> Here, a precise manufacturing process is even more challenging and reliable tuning methods still have to be found.

For the optical characterization of the microresonators, the experimental setup was thermally stabilized by a closed loop controlled thermoelectric Peltier element. Using this setup, it was possible to tune the temperature of the microchip between 15  $^{\circ}\text{C}$  and 40  $^{\circ}\text{C}$ . This allowed for a thermal tuning of our resonators over a range of nearly 40 GHz. The corresponding calibration curve for the tuning at temperatures around 25  $^{\circ}\text{C}$  is shown in Fig. 2. The resulting shift rate of 1.15 GHz/K agrees well with the theoretical estimation based on Gregor *et al.*<sup>12</sup> For the material parameters of pure fused silica ( $n_{1550\text{nm}} = 1.444$ ,  $\alpha = 0.55 \times 10^{-6} \text{ K}^{-1}$ ,  $\beta = 11.5 \times 10^{-6} \text{ K}^{-1}$ ),<sup>13,14</sup> a theoretical shift rate of 1.65 GHz/K was estimated.

In order to shift the spectral resonance position of a microresonator as close to a targeted wavelength that it was within reach by thermal tuning, we implemented a technique from standard silicon semiconductor processing. After standard reactive ion etching steps, it is possible to chemically polish the resulting surfaces by shortly dipping the whole sample into a wet chemical etching solution. Any sharp edges or residual surface roughness from ion bombardment are rounded and etched faster compared to smoother structures on the chip. Therefore, this technique seems also suitable for successively removing material from the surface in a lab post-production process.

For fine-tuning of the silica/silicon material system, we prepared a buffered hydrofluoric (HF) acid solution ( $\text{NH}_4$  (conc.)/HF(conc.)/ $\text{H}_2\text{O}$ , 3:1:3) which has high silica etch selectivity against silicon. The etching rate was then regulated by diluting this solution with water resulting in additional 1:10 and 1:100 etching solutions. The removal rates of these solutions were then tested on unstructured silicon wafers with thermally grown silica on top by a stepwise etching. The measurement of the surface removal rate was done by white light interferometry in a wavelength range between 400 and 900 nm. With this technique, removal rates for thermally grown silica were measured dilution dependent as 20 nm/s, 2 nm/s, and 0.15 nm/s. It was observed that the

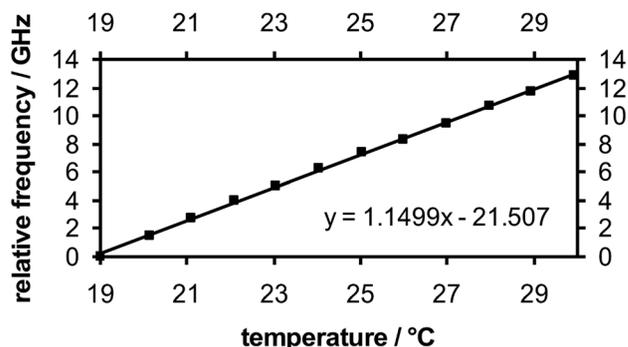


FIG. 2. Temperature tuning curve for the closed-loop controlled thermoelectric Peltier element of our setup. After each temperature change, the system had 5 min for complete stabilization before measurement.

removal rate in the original solution was too high for a specific removal of just a few nm of silica. In the 1:100 solution, the removal rate was too low for a substantial resonance shift; this makes the results hard to reproduce. Also with this solution, the removal rate showed a quite inhomogeneous distribution over the wafer surface. Only the dilution of 1:10 resulted in a highly reproducible isotropic material removal of 2 nm/s. Since we were looking for a fast and reliable process, only one dilution was chosen as tradeoff between ease and reliability allowing a fast single-step tuning process. The removal rate in the selected dilution is not only slow enough for small resonance shifts but also suitable for larger FSR-wide tuning steps. If ultimate tunability with highest precision is needed a higher dilution of the etching solution can be used with less reliability.

The surface roughness of oxide wafers before and after etching was analyzed on a KLA Tencor P 16 profilometer. The scan length was 100  $\mu\text{m}$ , the scan speed 2  $\mu\text{m/s}$ , the vertical scan range 13  $\mu\text{m}$ , and the nominal resolution 0.0078  $\text{\AA}$ /point. The scan length of 100  $\mu\text{m}$  was chosen as it represents roughly the perimeter of a small disk. Increasing the scan length and hence the scan time increases the chance to catch background noise in a normally operating clean room. The average roughness  $R_a$  of the silica before etching was  $\sim 1.5$  nm, after etching a roughness of  $\sim 1.4$  nm was measured. The results clearly showed that etching of 600 nm of oxide does not change the surface roughness parameters significantly. The measured average roughness of the etched surface corresponds well with other experimental data for the surface quality of directly etched microresonators<sup>3,11</sup> and proves the comparability of the microdisk Q factors.

To estimate the etching times for the microresonators, we assumed a maximum shift in the range of one FSR. This corresponds to a possible size reduction of 170 nm in radius. With the selected etching solution, the expected etching times are then in the range between a few seconds and 1.5 min. Assuming an isotropic etching on all sides of the disks, then the upper and lower surface will be etched, too. The silica layer thickness will be reduced from 2  $\mu\text{m}$  to 1.7  $\mu\text{m}$  at the maximum etching time. Although this is a reduction of more than 10%, it was verified by numerical simulations with a commercial FDTD mode solver (Photon Design Fimmwave) that no relevant change in effective radius of the WGMs is caused by this effect and also the mode structure mainly stays the same in result of this thinning process.

By applying the etching technique to the resonator samples, we found that the previously determined etching rates on planar wafer structures are not directly transferrable to the microdisk structures. We measured a shift rate of  $+(3.77 \pm 0.03)$  GHz/s which corresponds to a mean silica removal rate of 0.5 nm/s. This result deviates from the rate determined with unstructured silicon wafers by a factor of 4. The reason for this is not fully understood, yet. The side walls of the resonators may still be passivated from the plasma etching process, in this case the etch rate should rise after a substantial material removal from the side wall. For very short etching times (below 10 s), a slightly higher etching rate was observed. This can be explained by the dilution of the etching solution that is consumed in the process. Also,

it is assumed that the height to length aspect ratio and the curved surfaces of the structures have an influence on the etching rate.

In order to demonstrate the reproducibility of our approach, the WGM resonances of two different resonators located on the same chip are shifted within 10 GHz of two telecommunication C-band CHN reference lines. This was achieved by means of a repeated etching process. The six etching steps are shown in Fig. 3(a) for two equivalent microresonators. It can be seen in Fig. 3(a) that the frequency distance between both modes remained stable at around 200 GHz throughout the whole measurement process. Also, the two curves develop absolutely identically during the etching steps. The first 4 steps each had durations of 60 s, the following steps had reduced etching times of 30 s and 6 s. With this procedure, it was possible to achieve a matching of the two resonance lines to the P18 and P16 CHN reference lines with a residual distance of 7.76 GHz and 14.66 GHz, respectively. Fig. 3(b) shows the final frequency matching of the WGMs in one of the microdisk resonators by shifting the residual distance by means of temperature tuning and thus pairing the resonator exactly with the CHN standard reference line (P16). This technique turned out to be highly reproducible as the same behavior could be replicated on other chip samples also within only one applied tuning step.

The resulting Q factors are only slightly influenced by the tuning procedure and remained above  $10^5$  or higher throughout the whole measurement process (see Fig. 4). As

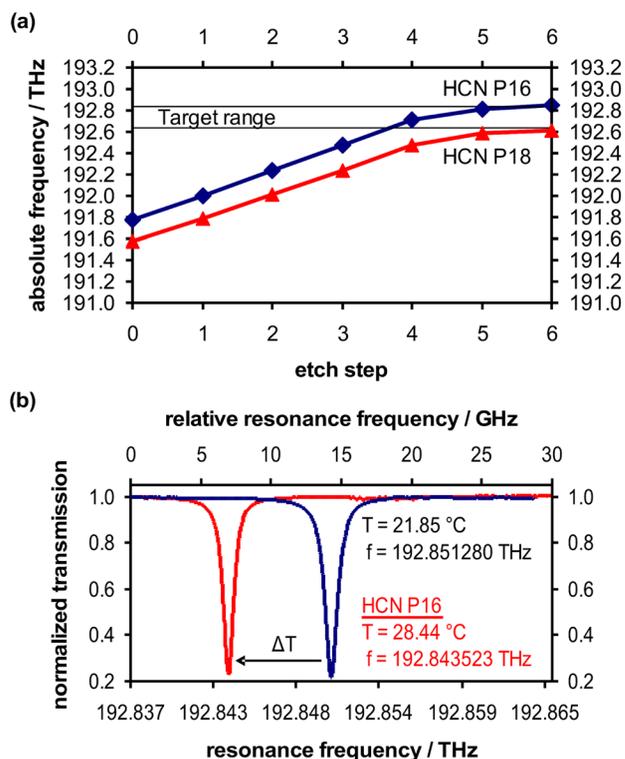


FIG. 3. (a) Simultaneous tuning of two different microresonators located on the same chip. The graphs show the reproducibility of the different etching steps and between consecutive steps with same processing time. The first 4 steps each had a duration of 60 s, the following steps had reduced etching times of 30 s and 6 s. (b) After approaching a specific resonance line (HCN P16), final tuning within a few GHz was done by simple thermal shifting with the Peltier element from our temperature control setup.

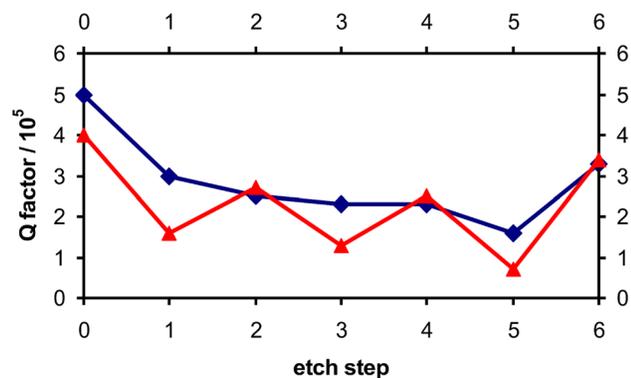


FIG. 4. Measured Q factors for the two microresonators after the consecutive etching steps described in the caption of Fig. 3.

the surface quality of the silica does not degrade during the etch process, it can be assumed that even the higher Q factors observed in directly etched disk or wedge resonators with much larger radii should be preserved. The presented tuning approach is not size-dependent and can also be used on such resonator designs.

In conclusion, the presented tuning method offers an efficient fine-tuning approach for shifting the WGM resonance positions of on-chip silica microresonators. It can complement standard fabrication processes and is thus compatible with mass-production of quasi-planar photonic structures. By dipping the microchips into a diluted buffered HF solution, we were able to shift the resonances during 60 s etching steps at a rate of  $+(3.77 \pm 0.03)$  GHz/s. By reducing the etching time with the selected dilution, a controllable minimal shift of +10 GHz is observed. The high reliability of this process also allows for tuning with comparable precision over a full FSR with a single etching step. The resulting Q factors are barely affected. A precise fine-tuning for canceling residual frequency mismatch can finally be done by using other techniques, e.g., temperature fine-tuning. By local integrated

heaters, this fine-tuning could also be applied to individual resonators. It is also possible to use an additional etching solution with higher dilution to achieve ultimate tunability with much higher precision at the prize of a lower reliability due to the unpredictability of the resulting etch rates. In this case, several repeated etching and measurement steps are required making this process less appealing for the large scale production of chip-based resonator designs. The presented method should also be applicable for other quasi-planar silica microresonators at various sizes, e.g., wedge or toroidal. By changing the etching solution, also resonators in different material systems could be handled.

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