Microfabrication of large-area circular high-stress silicon nitride membranes for optomechanical applications

Serra, Enrico; Bawaj, M.; Borrielli, A.; Di Giuseppe, G.; Forte, Salvatore; Kralj, N.; Malossi, N.; Marconi, L.; Marin, F.; Marino, F.

DOI
10.1063/1.4953805

Publication date
2016

Document Version
Publisher’s PDF, also known as Version of record

Published in
AIP Advances

Citation (APA)

Important note
To cite this publication, please use the final published version (if applicable). Please check the document version above.

Copyright
Other than for strictly personal use, it is not permitted to download, forward or distribute the text or part of it, without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license such as Creative Commons.

Takedown policy
Please contact us and provide details if you believe this document breaches copyrights. We will remove access to the work immediately and investigate your claim.

This work is downloaded from Delft University of Technology. For technical reasons the number of authors shown on this cover page is limited to a maximum of 10.
Microfabrication of large-area circular high-stress silicon nitride membranes for optomechanical applications

E. Serra,1,2 M. Bawaj,3,4 A. Borrielli,1,5 G. Di Giuseppe,3,4 S. Forte,2,6 N. Kralj,3 N. Malossi,3,4 L. Marconi,7,8 F. Marin,7,8,9 F. Marino,8,10 B. Morana,2 R. Natali,3,4 G. Pandraud,2 A. Pontin,7,8 G. A. Prodi,1,6 M. Rossi,3 P. M. Sarro,2 D. Vitali,3,4 and M. Bonaldi1,5,a

1Istituto Nazionale di Fisica Nucleare, TIFPA, 38123 Povo (TN), Italy
2Delft University of Technology, Else Kooi Laboratory, 2628 Delft, The Netherlands
3Physics Division, School of Science and Technology, Università di Camerino, 62032 Camerino (MC), Italy
4INFN, Sezione di Perugia, 06123, Perugia, Italy
5Institute of Materials for Electronics and Magnetism, Nanoscience-Trento-FBK Division, 38123 Povo (TN), Italy
6Dipartimento di Fisica, Università di Trento, 38123 Povo (TN), Italy
7Dipartimento di Fisica e Astronomia, Università di Firenze, Via Sansone 1, 50019 Sesto Fiorentino (FI), Italy
8INFN, Sezione di Firenze, Via Sansone 1, 50019 Sesto Fiorentino (FI), Italy
9LENS, Via Carrara 1, 50019 Sesto Fiorentino (FI), Italy
10CNR-INO, L.go Enrico Fermi 6, 50125 Firenze, Italy

(Received 16 December 2015; accepted 30 May 2016; published online 7 June 2016)

In view of the integration of membrane resonators with more complex MEMS structures, we developed a general fabrication procedure for circular shape SiNx membranes using Deep Reactive Ion Etching (DRIE). Large area and high-stress SiNx membranes were fabricated and used as optomechanical resonators in a Michelson interferometer, where Q values up to $1.3 \times 10^6$ were measured at cryogenic temperatures, and in a Fabry-Pérot cavity, where an optical finesse up to 50000 has been observed. © 2016 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/). [http://dx.doi.org/10.1063/1.4953805]

The optomechanical coupling between a laser beam and a microdevice via radiation pressure is of great interest in quantum-optics and fundamental research.1 In fact, the latest micro- and nano-mechanical resonators, when used in a high-finesse Fabry-Pérot optical cavity, offer great potential for precision sensing2,3 and for manipulation of the quantum state of light.4,5

In many cases the resonators consist of a free-standing high-stress silicon nitride (SiNx) membrane supported by a silicon (Si) frame, where Q-frequency product above $10^{13}$ Hz can be obtained thanks to the large tensile stress (of the order of GPa).6 These setups usually exploit dispersive coupling of the dielectric membrane placed in an optical cavity,7 but there are a number of ongoing efforts to extend the capabilities of SiNx membrane resonators, for instance by coating them with a metal for use in hybrid optical-microwave setups8 or by enhancing optomechanical coupling by patterning of photonic crystal structures.9 We also mention recent studies aiming to understand and possibly overcome the current limits in their mechanical performance.10

Large area free-standing SiNx membranes were originally proposed as TEM windows. Generally, these are fabricated on a Si support by low pressure chemical vapour deposition (LPCVD) and then released by wet etching the Si substrate. For this last step potassium hydroxide (KOH) solutions are typically employed. However, this etching is highly selective along silicon crystal planes and allows precise control of dimensions only if the desired structure can be bounded by <111> planes,11 as in rectangular membranes. On the other hand the integration of the resonator in
complex microsystems requires a greater flexibility in terms of layout. We note that SiNx membrane of arbitrary shape have been obtained by HF etching of a sacrificial SiO$_2$ layer,\textsuperscript{12} but these devices are suspended over the silicon wafer and cannot be used in the membrane-in-the-middle cavity configuration, that is used for optomechanical experiments with semi-transparent membranes.

In this paper we describe a general fabrication procedure for opto-mechanical membranes based on Deep Reactive Ion Etching (DRIE) through-wafer etching. DRIE, that was already used to produce SiN membranes for filtration and separation,\textsuperscript{13} allows the fabrication of membranes of any shape, because the design is not constrained by the crystal planes as in the case of KOH etching. At the same time it enables the fabrication of complex hybrid systems around the membrane. This is required to ease the integration of the SiNx membrane with on-chip mechanical isolation, useful for improving the overall reproducibility of the resonator by reducing the mechanical coupling with the sample holder. We remark that this strategy has recently allowed the production of optomechanical microresonators where quality factors higher than 10$^6$ can reproducibly be obtained for specifically designed normal modes.\textsuperscript{14,15} So far SiNx resonators suspended from phononic band-gap isolation structures have been built by combining DRIE with a wet etch release of the SiNx layer, but the process could not attain an optimal control of the membrane cleanliness.\textsuperscript{16,17}

Membranes were fabricated following the steps shown in Figure 1(a)-1(j). As substrate we employed a double-side-polished Si wafer with a thickness of 500 $\mu$m and a RMS surface roughness lower than 1 nm. Prior fabrication the wafers were cleaned by immersing them in 99\% HNO$_3$ and in 65\% HNO$_3$ at 110 $^\circ$C. A 200-nm-thick SiO$_2$ layer was then deposited by LPCVD using tetraethylorthosilicate (TEOS) as precursor [step(a)]. The LPCVD TEOS has a low compressive stress and works as etching stop layer during the DRIE etch. The fabrication followed with the LPCVD deposition of a 100-nm-thick SiNx layer [step (b)]. The deposition recipe was optimized for a residual tensile stress of about 1 GPa,\textsuperscript{18} measured by wafer curvature (TENCOR Flexus FLX-2908). After the back-side etching [step (c)], a low-stress 1-$\mu$m-thick layer of pure Al was sputtered on the front side [step (d)]. This layer served to protect the SiNx during the DRIE etch. A 6-$\mu$m-thick oxide layer was deposited on the backside by means of plasma enhanced chemical vapor deposition (PECVD) [step (e)]. Circular (square) holes with a diameter (side) up to 1.5 mm were patterned into this layer [step (f-g)]. The Si was then locally removed [step(h)] by means of DRIE (two pulse BOSCH process in Omega i2L Raper). The etching rates were about 1.38 $\mu$m/cycle for Si and 5 nm/cycle for the PECVD oxide. Finally, the SiNx membrane was released by first stripping the Al layer [step(i)] using a PES solution and then by etching the TEOS oxide [step (j)] by means of an HF-based solution. We point out that metal and oxide layers are only protective and do not concur in determining the shape. The etching of the silicon frame, that defines the membrane’s shape, is fully realized by DRIE.

We focus the analysis of the mechanical resonance frequencies on the circular membranes, due to their original shape with respect to the more common square membranes. The theoretical resonance frequencies in a circular membrane are given by the expression $f_{mn} = f_0 \alpha_{mn}$ where $\alpha_{mn}$ is the $n$-th root of the Bessel polynomial of order $m$, and $f_0 = \frac{1}{2\pi} \sqrt{\frac{T}{\rho R}}$ ($T$ is the stress, $\rho$ the density, $R$ the radius of the membrane). The modal shapes of the first 0$n$ and 1$n$ modes are shown in Figure 2.

We have measured the modal frequencies from a thermal spectrum acquired using a Michelson interferometer (Fig. 3), with the sample kept in a vacuum chamber. In Fig. 4(a) we show the experimental resonance frequencies divided by the respective $\alpha_{mn}$, for the first $m = 0$ and $m = 1$ order modes. We expect a constant value, equal to $f_0$. It appears that the lower modes slightly (but systematically) deviate from the predicted behavior, with a maximum spread of just 4\%, probably due to boundary effects, likely influenced by the clamping. We can extrapolate an asymptotic experimental value of $f_0 \approx 114$ kHz, to be compared with $f_0 = 118.6$ kHz that is calculated using the nominal parameters $R = 0.75$ mm, $T = 1$ GPa and $\rho = 3200$ kg/m$^3$. The agreement is very good. We also remark that measurements taken at different times (entailing few degrees variations of the room temperature) yield fluctuations of the experimental $f_0$ by $2 - 3\%$.

An interesting property of the circular membrane is that the effective modal mass depends on the modal index, as opposed to the square membranes. Namely, the effective mass of the 0$n$ modes for a centered, $\delta$–like readout is $M_{0n} = M (J_1(\alpha_{mn}))^2$, where $M$ is the physical mass of the membrane and $J_m$ is the Bessel polynomial (the first values of $J_0,J_1,J_2,J_3 = 0.269,0.116,0.074,0.054$, respectively).
FIG. 1. (a)-(j) Fabrication steps. (k) Section view of the finished device. (l) Optical microscope image of a circular membrane of diameter 1.5 mm.

0.043). The mass is lower at higher index because the modal displacement is more concentrated in the center, while it remains homogeneous for a square membrane. For comparison, in the case of a square membrane the effective mass of the odd modes is $M/4$. A reduced effective mass, increasing the susceptibility, is a useful property in opto-mechanics experiments. In the realistic case of a
centered, Gaussian readout with $1/e^2$ width $w$, the effective mass becomes:

$$M_{0n} = M \left( \frac{J_1(\alpha_{0n})}{\frac{1}{w} \int J_0(\alpha_{0n} \tau / R) \exp \left( -\frac{2\tau^2}{w^2} \right) d\tau} \right)^2. \quad (1)$$

In Fig. 4(b) we report the experimental values of $M_{0n}$ for the first modes, derived from the areas $A_{0n}$ of the thermal peaks in the displacement spectrum using $A_{0n} = \frac{k_b T_K}{k_B(2\pi f_0)^2}$ ($k_B$ is the Boltzmann constant and $T_K$ the temperature). They are compared with the theoretical values calculated for a realistic $w = 0.15$ mm (showing a good agreement) and with a pointlike readout.

We have verified by Finite Element (FE) analysis that the expected quality factor is similar to that of a square membrane with the same modal frequency of the first mode. In fact, for each mode, a FE routine can evaluate the energy stored in different parts of the systems and can estimate the overall quality factor from the loss angle assigned to each part.\textsuperscript{19} To validate our procedure we have verified (Figure 5(a)) that the quality factors of the modes of the square membrane evaluated by FE are in good agreement with the values expected from the theory. For a square membrane of side 1.52 mm, thickness 100 nm and internal stress 1 GPa, the quality factor envelope can be evaluated as:\textsuperscript{20}

$$Q_{\text{square}} \approx \frac{1.1 \times 10^7}{1 + 1.35 \times 10^{-13} f^2} \quad (2)$$

where $f$ is the frequency of the modes and the intrinsic loss has been set as $1/Q = 2 \times 10^{-3}$, the room temperature value.\textsuperscript{10} The case of a circular membrane is then solved with the same FE routine, obtaining quite similar values (Fig. 5(a)). Note that the loss $(1/Q)$ grows with the frequency because higher order modes have an increasing number of nodal lines.

The simulation can take into account also the loss from the substrate supporting the membrane, that has not been obtained in closed form yet. In fact the borders of the membrane bends during the oscillation and the first layer of the substrate is subjected to a mechanical strain. This contribution is

FIG. 2. Modal shape of the first $0n$ and $1n$ modes of a circular membrane. The color scale, from light gray to red, is proportional to the absolute displacement from the equilibrium position.

FIG. 3. Typical noise power spectral density (PSD) of the interferometer output; the modal frequencies can be clearly seen above background noise.
negligible when the SiNx layer is directly grown on silicon, but it may become relevant in our case, as the membrane is grown over a SiO$_2$ layer. At room temperature the loss angle of SiO$_2$ is as low as $1/Q = 5 \times 10^{-3}$ and we have not found an additional contribution. At liquid helium temperature the expected loss for SiNx reduces to $1/Q = 2 \times 10^{-5}$, while for SiO$_2$ it grows to $1/Q = 7 \times 10^{-4}$. In this case the substrate becomes the main source of loss and limits the maximum quality factor to $5 \times 10^6$, as shown in Figure 5(b). We note here that the loss is set by the contribution of the border and the frequency dependence can no longer be seen in the simulation data. We point out that these results are necessarily approximate, as the intrinsic loss angle value can be strongly dependent on the deposition procedure or on the thickness of the adhesion layer.

The mechanical quality factor $Q$ of the modes of different membranes has been measured both at room and at cryogenic temperatures, by driving the different resonances with a piezoelectric glued on the sample mount, and observing the ring-down with a Michelson interferometer. At room temperature, the $Q$ values are very scattered, ranging from few thousands up to $2 \times 10^7$. This feature is very common in SiNx membranes, and is due to the coupling with the frame and, through it, with the sample holder. At cryogenic temperatures, the values of $Q$ are still scattered, but globally higher. With a 1.5 mm diameter membrane on a 5 mm side, square frame clamped between two copper plates, we could measure a maximum $Q$ of $0.65 \times 10^6$, at 8 K. With a 1 mm side, square membrane close to the edge of a $5 \times 20$ mm$^2$ frame, glued on the opposite edge to a copper block, we could measure $Q$ values up to $1.3 \times 10^6$ at 13 K. As we aim to develop a general-purpose device, with
features independent from the details of the experimental apparatus, we did not optimize the efforts to improve the best measured quality factor by trying different mounting systems. We also note that, even if the meaningful clamping losses do not permit a better assessment, the FEM calculations indicate that the loss from the SiO₂ adhesion layer limits the expected $Q$ for membranes of our size to about $5 \times 10^{6}$ (Figure 5(b)). In this context, we estimate that a $Q$ higher than $10^{6}$ it is enough to motivate the future development of on-chip isolation system for these membranes.

We characterized the optical properties of a circular membrane of nominal radius $R = 0.6$ mm, with thickness $L_d = 97.27 \pm 0.01$ nm and index of refraction at $\lambda = 1064$ nm $n_R = 2.0210 \pm 0.0005$, measured during production [step (b)] by variable angle spectroscopic ellipsometry (VASE). These values have been confirmed by polarization-resolved transmission measurements again at $\lambda = 1064$ nm, performed on the final, diced samples. These latter measurements provide an intensity reflectivity at normal incidence $|r_d|^2 = 0.355 \pm 0.002$, perfectly consistent with the expression for the amplitude reflection and transmission coefficients of a dielectric membrane of thickness $L_d$ and refractive index $n$,

$$
    r_d = \frac{(n^2 - 1) \sin \beta}{2in \cos \beta + (n^2 + 1) \sin \beta},
$$

(3)

$$
    t_d = \frac{2n}{2in \cos \beta + (n^2 + 1) \sin \beta},
$$

(4)

where $\beta = nkL_d = 2\pi nL_d/\lambda$. When $n = n_R + in_1$, with the small but nonzero imaginary part $n_1$ providing a measure of optical absorption. In order to accurately evaluate it, we have placed the membrane between two Al cylinders, and we have inserted it in a $L = 9.03$ cm long cavity at room temperature formed by two spherical mirrors of radius of curvature 7.5 cm. The high-frequency component of the Pound-Drever-Hall (PDH) signal used to lock the cavity was acquired for the preliminary analysis of the mechanical modes at a vacuum chamber pressure of $1.7 \times 10^{-2}$ mbar. The fundamental eigenfrequency is found to be $f_01 \sim 348.5$ kHz, consistent with the theoretical expectation if we assume $R = 0.615$ mm. The detected optical cavity modes as a function of the membrane position are also consistent with the theoretical model obtained with the cavity and membrane parameters. Finally, the cavity finesse, $T_T$, as a function of the membrane position has been measured (see Fig. 6) by the ringdown technique. The laser beam, deflected by an acousto-optic-modulator (AOM) and sent into the cavity, is switched off in 50 ns after the transmitted light has reached a threshold level. The leakage of transmitted light is monitored to estimate the cavity decay-time, which is fitted with a single exponential form whose time constant, $\tau$, is related to $T_T$ via $T_T = \pi c \tau / L$. Repeating the finesse measurement for different positions of the membrane as reported in Fig. 6, allows to estimate $n_1$ and the roughness of the membrane. For fitting the finesse data we consider the transfer function of a cavity with a membrane in the middle. The cavity consists of two semi-cavities; denoting with $z$ the position of the membrane with respect to the cavity center, and assuming for the reflection and transmission coefficients of the mirrors $r_1 = r_2 = \sqrt{R}$ and $t_1 = t_2 = \sqrt{T_T}$, we derive the following expression for the intensity transmission:

$$
    T_c = \frac{|T_t d|^2}{1 + 2r_d \sqrt{R} \cos(2kz)e^{ikL} + r_d^2 e^{2ikL}t_d^2}. 
$$

(5)

In the ideal case $R = 1$ and $n$ is real, so that $r_d^2 + r_d = \exp \{2i \text{arg}(r_d)\} = \exp \{2i \phi_r\}$, the eigenfrequencies of the cavity modes are given by the zeros of the denominator of the cavity transmission $\cos(kL + \phi_r) + |r_d| \cos(2kz)|^2 = 0$, which gives the results in Ref. 24

$$
    2kL = \nu \frac{2\pi}{\Delta V_{FSR}} = -2\phi_r + 2\cos^{-1}[|r_d| \cos(2kz)].
$$

(6)

When $R < 1$ and in the presence of absorption the resonance frequencies of the cavity modes are determined by the maxima of the transmission $T_c$ given by Eq. (5), and the finesse can be obtained from the width of the transmission peaks. Roughness can be introduced following Refs. 25–27, according to which any optical element with a given roughness is responsible for a modification of the
wavefront and therefore leads to a scattering loss from the incident optical mode into all the other cavity modes. This scattering into other modes induced by an effective optical roughness $\sigma_{\text{opt}}$ can be modeled by including a factor $\sqrt{\exp[-(2k\sigma_{\text{opt}})^2]}$ multiplying $r_d$ in eq. (3). Fig. 6 shows the best fits for the set of data (red symbols) taken for the 1.2 mm–diameter circular membranes. We have taken $n_1$ and $\sigma_{\text{opt}}$ as fitting parameters, while we have kept fixed all the other parameters ($L_d = 97$ nm, $n_R = 2.021$, $L = 9.03$ cm, $\lambda = 1064$ nm, and $F_0 = 53500 \pm 100$ corresponding to $\mathcal{R} = 0.9999413$). While the $\lambda/2$ periodicity is caused by the absorption which is maximum at the antinodes and minimum at the nodes of the cavity field, the membrane roughness causes scattering which instead does not significantly depend upon the membrane axial position (as shown in Fig. 6). The fit gives an imaginary part of the refractive index $\tilde{n}_1 = (1.97 \pm 0.08) \times 10^{-6}$ and $\tilde{\sigma}_{\text{opt}} = (287 \pm 4)$ pm. As a comparison, the results for a 1 mm side, 50 nm thickness membrane by Norcada$^{28}$ are reported with fitting parameters $n_1 = (1.00 \pm 0.01) \times 10^{-5}$ and $\sigma_{\text{opt}} = (280 \pm 10)$ pm. The circular membranes we fabricated present a similar effective optical roughness with respect to a commercial square one but a lower absorption. As regards standard AFM measurement of roughness, we measured for our membranes $\sigma_{\text{rms}} = 0.7 \pm 0.1$ nm over scan areas of $1 \mu m \times 1 \mu m$.

In conclusion, we developed and validated a general fabrication procedure for free-standing large area high-stress SiNx membranes of any shape with a good dimensional precision by using DRIE etching. Possible improvements of the process could be obtained by the use of silicon substrates with a lower initial roughness. This work is a crucial step toward the integration of SiN$_x$ membranes in hybrid systems and on-chip mechanical isolation.

We mention that some interesting development have been recently reported$^{29,30}$ published after the submission of this work. Here small membranes (about $100 \times 100 \mu m^2$) are supported by trampolines that act as flexure elements, and demonstrate the effectiveness of a careful engineering in obtaining high quality factors. These resonators also have a very small mass, obtained at the expense of a reduced heat management capability, and therefore target different experimental regimes compared to large membranes.

This work has been supported by MIUR (PRIN 2010-2011) and by INFN (HUMOR project). A.B. acknowledges support from the MIUR under the “FIRB-Futuro in ricerca 2013” funding program, project code RBFR13QUVI.