

Fabrication of high-aspect ratio Si_3N_4 nanobeam resonators

Robin Groth¹ and Mohammad J. Bereyhi¹

¹*Institute of Physics, Swiss Federal Institute of Technology Lausanne (EPFL)*

We describe the fabrication of high-stress Si_3N_4 nanobeam resonators with high aspect ratios exceeding lengths of 3.5mm. The lowest order out-of-plane modes of these nanobeams have quality factors of $Q \geq 10^6$ with fundamental mode frequencies lying in the range of 80 – 500kHz. The beams are fabricated from high-stress, 20 – 50nm-thick films of Si_3N_4 deposited via LPCVD on standard silicon wafers. The beams are patterned via electron beam lithography and deep reactive ion etching. The underlying silicon is etched in KOH and the released structures are dried in a critical point dryer.

Keywords: Optomechanics; silicon nitride; nanobeam resonators; electron beam lithography (EBL); deep reactive ion etching (DRIE); KOH; critical point dryer (CPD)

In the following we give a detailed description of the fabrication process of the nanobeam resonators shown in fig. 1. This process flow has been used to fabricate strain-engineered nanobeams [1], clamp-tapered nanobeams [2] and fractal-like beam resonators [3]. For a theoretical treatment of dissipation dilution in strained nanomechanical resonators, readers are invited to consult [4]. A general overview of the process flow can be found in fig. 2. Each of the steps will be described in the main part of the document. The appendix contains a “gallery of failures” with common issues we have encountered.

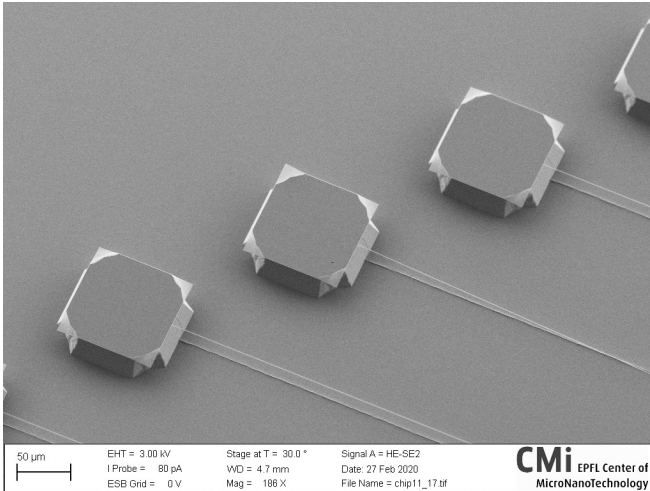


FIG. 1. **Perspective view of three nanobeam resonators suspended above the silicon substrate by clamping their endpoints to rectangular pads.** The central beam collapsed during the release. The beams shown here are 20nm thick, $1\mu\text{m}$ wide and around 1mm long. The thin lines visible on the silicon substrate running in parallel to the beams are a consequence of the anisotropic KOH etch.

ALIGNMENT MARKERS

We start with prime, double-side polished, $\langle 100 \rangle$ silicon wafers[5] with a diameter of 100mm. The process

flow described here should work for a wide range of substrate thickness (we used $525\mu\text{m}$ thick wafers). The alignment markers are transferred to the substrate in the following way. First, the wafers are cleaned in an oxygen plasma[6] for two minutes and at high power (600W, O_2 flow = 400sccm, 0.8mbar). Then we use a standard coating sequence of an automatic coater[7] to cover the wafer with a $1.5\mu\text{m}$ thick layer of ECI 3007 photoresist. The coating sequence contains HMDS priming of the substrate and edge-bead removal. After coating the photoresist is exposed using a mask aligner[8] with a dose of $150\text{mJ}/\text{cm}^2$. After exposure the photoresist is developed in an automatic developer[9]. This is followed by a spin-rinse-dry (SRD) process which removes any unexposed photoresist and developer from the wafer to avoid contamination of the dry etcher. The pattern is transferred to the substrate via continuous deep reactive ion etching [10]. The etch depth should be $\geq 3\mu\text{m}$ to facilitate identification of the alignment markers by the electron beam tool. Too shallow markers carry the risk of not being found by the tool. After the dry etch the resist is removed via a 10-minute, high power oxygen plasma followed by a 10-minute bath in Shipley Remover 1165. The wafers are cleaned in two separate DI-water baths for both rough and fine rinsing. Then the wafers are dried using the dry-only programme of a spin rinse dryer.

LPCVD

After definition of alignment markers, the wafers are prepared for low pressure chemical vapour deposition (LPCVD) of high-stress silicon nitride (step on in fig. 2). The substrate preparation contains a standard RCA cleaning. The LPCVD for our wafers was done in the CMI’s furnaces and by the Hahn-Schickard-Gesellschaft. Typical values for the deposition stress are in the range of 1 – 1.1GPa.

Measuring deposition stress. For quality control of the nitride deposition we add a single-side polished test wafer to the batch. Before LPCVD the bow of the

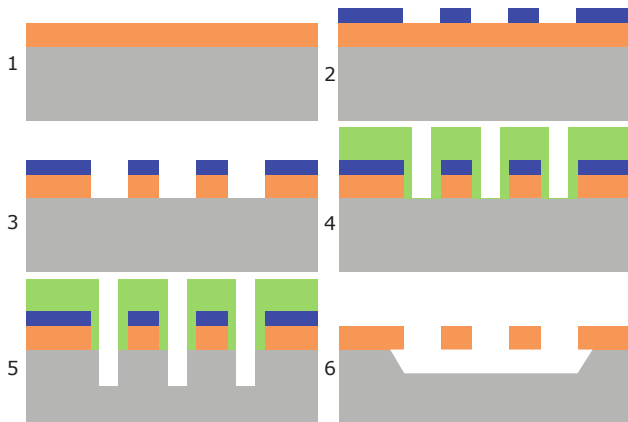


FIG. 2. **Overview of the process flow.** Grey: silicon, orange: silicon nitride, blue: first FOx layer, green: second FOx layer. See text for details.

test wafer is measured using a thin film stress measurement tool[11]. For more accurate measurements the bow can be measured for several wafer orientations. After LPCVD the silicon nitride is removed from the backside using a dry etcher. Then, the bow is re-measured using the thin film stress measurement tool. Using the tool's software and knowing both the thickness of the nitride layer as well as the wafer properties, it is possible to infer the deposition stress of the nitride layer from the change in wafer bow.

Measuring film thickness The thickness of the nitride film is measured with a spectroscopic reflectometer[12].

FIRST EXPOSURE AND PATTERN TRANSFER

The wafer is cleaned and dehydrated through a two minute, high power oxygen plasma (with the same parameters described before). An alternative to oxygen plasma is a dehydration bake for ≥ 5 min at $\approx 120^\circ\text{C}$ followed by at least a minute of cool-down before proceeding with the coating. Note that we cannot recommend this alternative since insufficient dehydration of the substrate can reduce adhesion of the e-beam resist. Without a prior oxygen plasma we have observed lift-off of the e-beam resist during development (see fig. 9 in the appendix).

The e-beam resist used for both exposures is DOW FOx-16 Flowable Oxide. For the first exposure the FOx resist is spin-coated at 6000rpm giving a resist thickness of around 550nm. The wafer is then exposed with a dose of $1100\mu\text{C}/\text{cm}^2$. We write the first exposure with two different spot sizes using the bulk-and-sleeve method to reduce the overall writing time. Regarding the design of the mask, note that the pads should be aligned along the slow-etching planes of silicon for wet etch in KOH (i.e. with edges of the pads aligned vertically and hori-

zontally when viewed from the primary flat of the wafer). A different orientation will lead to a faster undercut of the pads. This can lead to large overhangs of the nitride film which can significantly impact the mechanical properties of the nanobeams and cause a collapse of the beams. Moreover, the pad size should be significantly larger than the maximum beam width (in our case the sides of the pads measure $100\mu\text{m}$) to avoid undercut of the pads in the clamping region of the nanobeams. After exposure the wafer is developed in TMAH25% for 2 minutes followed by a thorough rinse. The wafer is then dried using a nitrogen spray gun.

After inspection of the resist with a microscope, the pattern is transferred to the silicon nitride using a ICP plasma etcher with fluorine chemistry[13]. The etch time required to remove 20-50nm of silicon nitride lies in the range of 20-50 seconds. We use an end-point-detection system based on optical spectroscopy to abort the etch process once the nitride is fully removed. Due to a non-uniform etch rate, we typically overetch slightly to ensure that the nitride is removed everywhere on the exposed areas of the wafer.

SECOND EXPOSURE AND DRIE

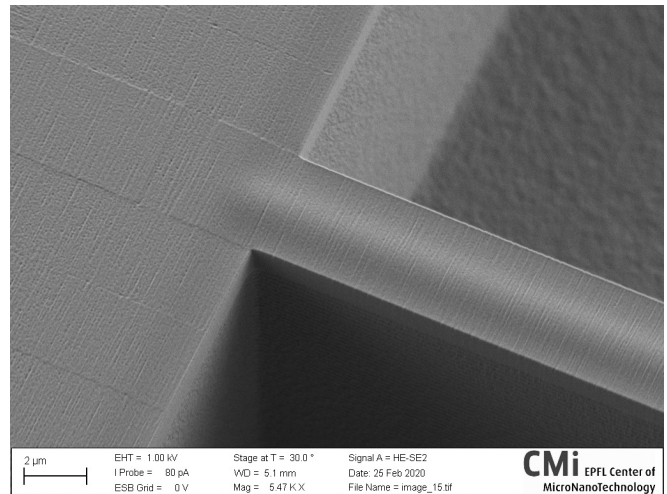


FIG. 3. **SEM image taken after DRIE and oxygen plasma.** The view shows the FOx layer in the clamping region of the nanobeam. The first FOx layer manifests itself in the contour of the second layer.

The second exposure closely follows the procedure for the first exposure described above (oxygen plasma, coating, exposure, development). The only difference lies in the resist thickness and the exposure dose. This time we spin-coat the FOx resist at 2000rpm to obtain a thick resist layer of around 800nm. The thick resist will provide additional protection of the silicon nitride film during deep reactive ion etching DRIE. To ensure sufficient

exposure the dose is increased to $1200\mu\text{C}/\text{cm}^2$. We perform DRIE[14] to create a $15\mu\text{m}$ deep recess in the silicon. Note that the etch depth should be at least twice as deep as the widest part of the beam. This will ensure a fast etch of the silicon during the release. We found that choosing a more gentle Bosch process, i.e. with shorter SF_6 -pulses and longer C_4F_8 -pulses, leads to a higher yield. The dry etch is followed by a 7-minute oxygen plasma at high power to remove contamination from the substrate. The main source of contamination are polymers formed by C_4F_8 -molecules during the Bosch process. See fig. 10 in the appendix for an image of this type of residue. The cleanliness of the substrate as well as the etch depth is controlled in a scanning-electron microscope (SEM, see fig. 3).

DICING

Before dicing we coat the frontside of the wafer with a thick photoresist to protect the structures from damage and contamination. We chose a resist with high viscosity[15] and use a manual coater to ensure that all structures are covered sufficiently. Before coating we do a dehydration bake for at least 5 minutes at 115°C . After coating we do a softbake, also at least for 5 minutes and at 115°C . After coating the wafer is diced[16] into chips measuring $5\times 12\text{mm}$. After dicing the photoresist is removed by immersing the wafer in Shipley Remover 1165 for 10 minutes. Subsequently any organic residues are removed in a 10-minute piranha bath. This step should be followed by thorough rinsing to reduce contamination of the chips. The last step before we can release the structures is the removal of the two FOX layers. For this we dip the chips into BHF (buffered hydrofluoric acid) for one minute. Again, in order to reduce contamination, we rinse the chips for at least 5-10 minutes in DI-water. Now the devices are ready for release.

RELEASE

The structures are released in three steps: wet etch in KOH , neutralisation in HCl and drying with critical point dryer (CPD). The release is the most delicate step of the process since the released beams are very fragile and small turbulences in the liquid suffice to cause collapse of the beams. This is why we are using a customised teflon holder for the entire release.

For the etch we prepare a beaker with KOH 40% and control that the density measures $1.380\text{g}/\text{cm}^3$. This check is necessary since the etch rate of silicon in KOH depends on the KOH concentration. The smaller the concentration (i.e. density) the greater the etch rate. We use a hotplate to heat up the KOH to 63°C because a higher temperature reduces the required etch time. Much higher

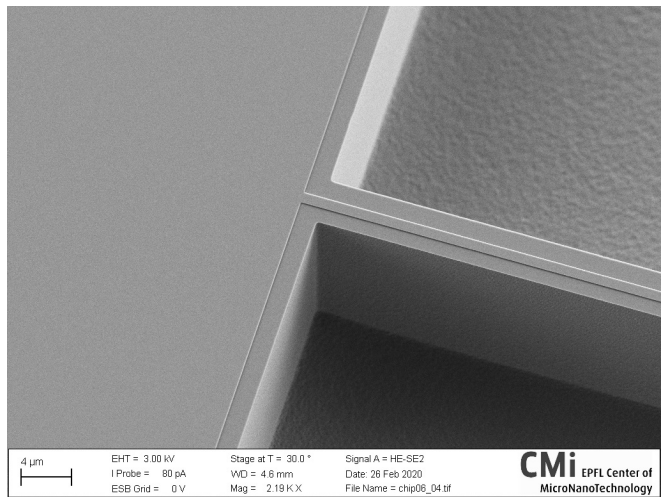


FIG. 4. SEM image taken after BHF and just before release. The view shows the clamping region of the nanobeam similar to fig. 3. The thin silicon nitride film can be clearly distinguished from the underlying silicon.

temperatures are not recommended, since this will lead to bubble formation and the collapse of bubbles can damage the released nanobeams. The required etch time depends primarily on the largest width of the beams and lies typically in the range of 15-30 minutes. During the release we use a magnetic stirrer to maintain a uniform distribution of KOH in the beaker for a constant etch rate. The stirrer should rotate at a low angular velocity to avoid turbulences in the liquid (again to prevent collapse of the beams). When releasing a new design for the first time, we perform a test run of a single chip to calibrate the etch time to ensure that the beams are completely released and the pads are only slightly undercut from the corners. After the etch time elapsed, we rinse the chips in DI-water for at least 10 minutes. This has to be done carefully to avoid damaging the beams due to turbulences in the liquid. Sufficient dilution of KOH is verified by using pH strip. After rinsing we move the chips into a 1:1 $\text{HCl}:\text{H}_2\text{O}$ mixture for at least two hours and at 45°C . Again the liquid is stirred gently. After the neutralisation is complete, we repeat the rinsing procedure described before until the pH-value of the liquid is no longer in the acidic regime (acids can damage the CPD machine). Then we place the chips in ethanol for about 15 minutes under gentle agitation with a magnetic stirrer. Finally, the devices are dried in the CPD. For high-Q resonators it is not recommended to image the released structures with an SEM. This is because small amounts of carbon which are deposited during imaging are believed to increase mechanical losses of nanobeam resonators.

APPENDIX

The appendix contains a “gallery of failure” of common issues we have encountered during the fabrication process. The microscope and SEM images presented here should help with troubleshooting.

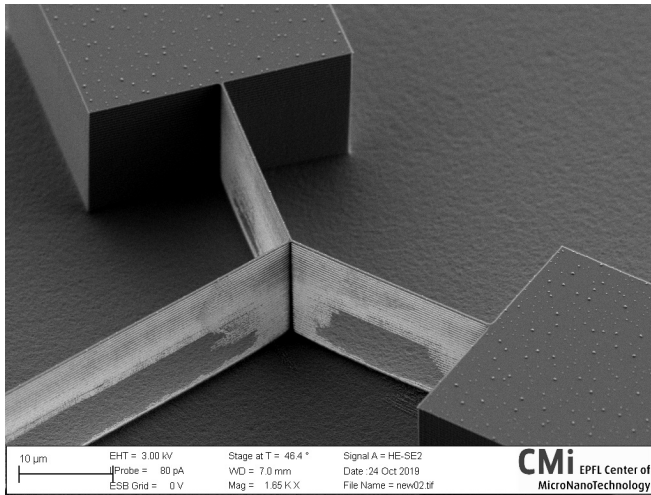


FIG. 5. **Motivation for two electron beam exposures.** The SEM image shows a fractal-like resonator [3] with a beam width of 500nm fabricated with a single e-beam exposure. The high-aspect ratio sidewalls were damaged during DRIE, leaving holes in the walls which can cause collapse of the beams. For a single electron beam exposure, the width of beams needs to be $\geq 1\mu\text{m}$. The process flow presented in this article allows fabrication of thinner beam segments needed for strain-engineered nanobeams [1], clamp-tapered nanobeams [2] and fractal-like beam resonators [3].

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 - [2] M. J. Beryhi, A. Beccari, S. A. Fedorov, A. H. Ghadimi, R. Schilling, D. J. Wilson, N. J. Engelsen, and T. J. Kippenberg, *Nano Lett.* **19**, 2329 (2019).
 - [3] S. A. Fedorov, A. Beccari, N. J. Engelsen, and T. J. Kippenberg, *Phys. Rev. Lett.* **124**, 025502 (2020).
 - [4] S. A. Fedorov, N. J. Engelsen, A. H. Ghadimi, M. J. Beryhi, R. Schilling, D. J. Wilson, and T. J. Kippenberg, [arXiv:1807.07086](https://arxiv.org/abs/1807.07086) [cond-mat, physics:physics] (2018), arXiv: 1807.07086.
 - [5] We use L14295 wafers from Siegert wafer (diameter: $100\pm 0.3\text{mm}$, grade: prime, growth: FZ, type: P, dopant: B, orientation: $\langle 100 \rangle \pm 0.5^\circ$, resistivity: $3000\Omega\text{cm}$, thickness: $525 \pm 25\mu\text{m}$, finish: double side polished, TTV $< 5\mu\text{m}$, bow $< 30\mu\text{m}$, warp $< 30\mu\text{m}$, particles at $0.3\mu\text{m} < 10$).

- [6] Machine used: GIGAbatch Series Microwave Plasma Systems from PVA TePla.
- [7] Machine used: ACS200 Gen 3.
- [8] Machine used: MA6 Gen3, i-line (355-365nm).

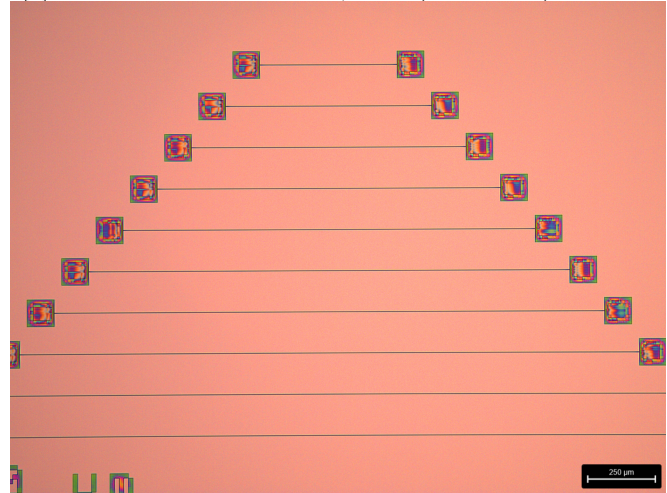


FIG. 6. **Microscope image showing FOx resist exposed with poor proximity effect correction (PEC).** The pads appear in different colours due to variations in the FOx thickness caused by variation in the local dose. Correct PEC settings will ensure that the local dose is constant over the whole design leading to a constant FOx thickness and a uniform colour when viewed under a microscope (see fig. 7).

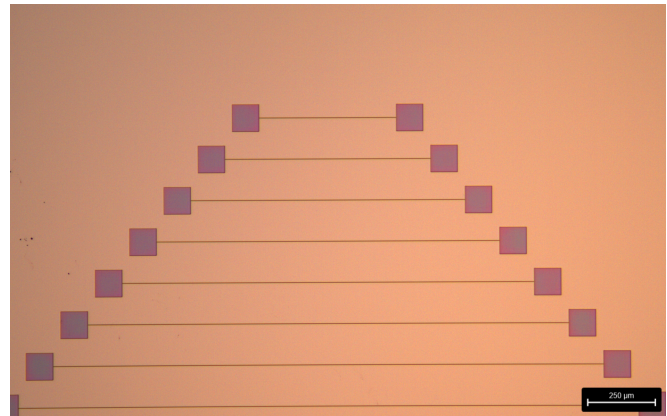


FIG. 7. **Microscope image showing FOx resist exposed with optimised PEC settings.**

- [9] Machine used: ACS200 Gen 3.
- [10] Machine used: Alcatel AMS 200 SE, dry etcher, fluorine chemistry.
- [11] Machine used: Toho Technology FLX 2320-S.
- [12] Machine used: FilMetrics F20-UV.
- [13] Machine used: SPTS APS Dielectric Etcher, dry etcher, fluorine chemistry.
- [14] Machine used: Alcatel AMS 200 SE, dry etcher, fluorine chemistry.
- [15] We selected the photoresist AZ9260 and a resist thickness of $15\mu\text{m}$.
- [16] Machine used: Disco DAD321, automatic dicing saw.

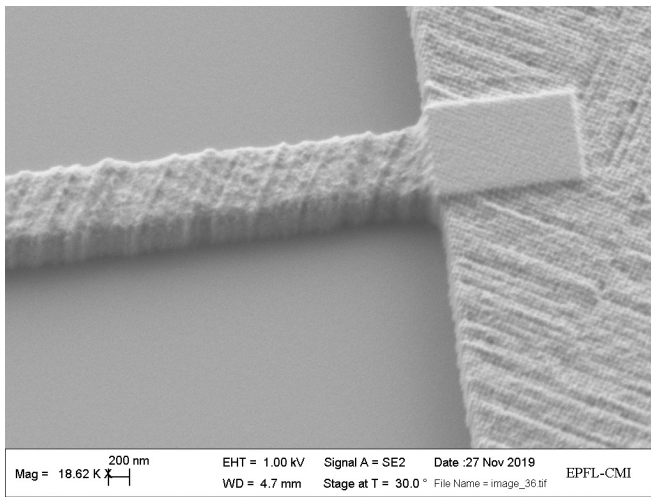


FIG. 8. **SEM image of the first FOx layer.** Here the dose was set too low and the FOx was underexposed. In the small overlap region at the clamp, the combined dose of both electron beams was sufficient to ensure sufficient exposure of the FOx resist.

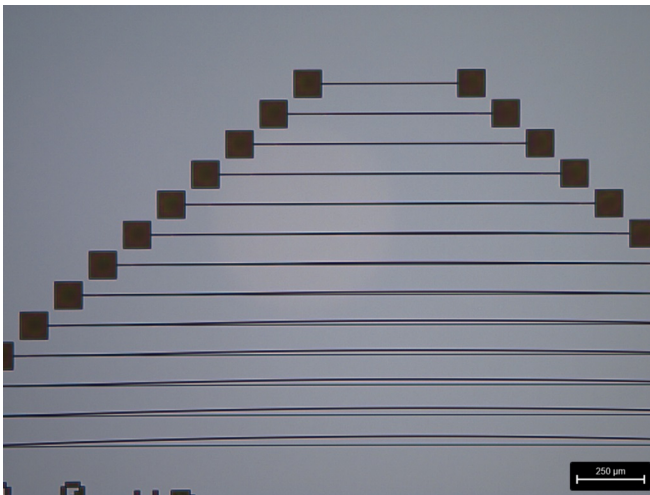


FIG. 9. **Microscope image taken after the second exposure showing lift-off of the second FOx layer.** Straight lines correspond to the first FOx layer and bend lines to the second FOx layer. The second layer slipped off from the first layer during development in TMAH. On the pads the adhesion between the two FOx layers was sufficient to keep the second FOx layer in place.

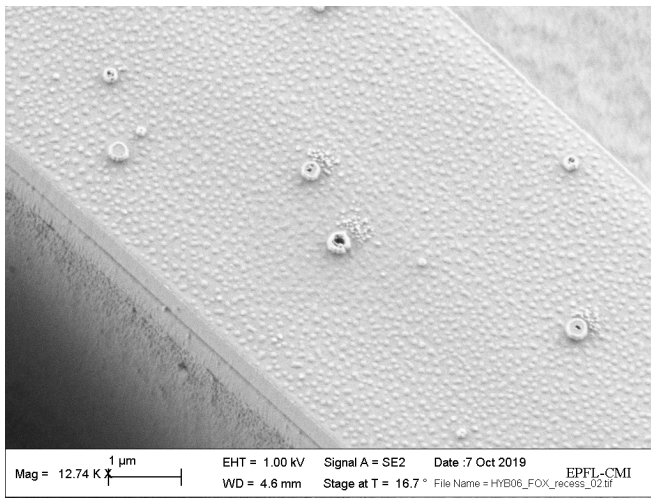


FIG. 10. SEM image of a nanobeam taken after DRIE with insufficient plasma cleaning. The residual polymers formed by the passivation layer during the Bosch process are clearly visible on top of the FOX layer.