Fabrication of patterned silicon nitride nanomembranes at the LKB

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Thin silicon nitride nanomembranes are attracting growing attention following a novel fabrication method which consists in patterning them with a phononic crystal. In engineering the vibrational mode profile, the dominant mechanisms of loss, radiation loss and intrinsic material loss, are simultaneously addressed and mitigated. The fabrication method employed by the optomechanics group at the Laboratoire Kastler Brossel is presented here; in particular, we only employ basic lithography techniques and wet etching processes.

Keywords: lithography, soft clamping, phononic crystal, silicon nitride, wet etch, KOH

FABRICATION PROCESS

In this report, we present the process used by the optomechanics group at the Laboratoire Kastler Brossel for fabricating 'soft-clamped' silicon nitride (SiN) membranes [1]. The (100) Si wafers were bought from Si-Mat [2], with stoichiometric SiN deposited on both sides of the Si by low-pressure chemical vapor deposition. Two kinds of wafers are currently used in our work, and the method described below has been tested on both kinds: the first is a 500 µm thick Si wafer, with intrinsic resistivity ($\rho > 10\,000\ \Omega$.cm), and layers of 100 nm of SiN; the other is a 300 µm tick Si wafer, with a resistivity of $\rho \approx 1 - 30\ \Omega$.cm, and a SiN thickness of 35 nm.

As with most fabrication methods reported by other groups for SiN membranes, patterned or otherwise, the fabrication process is based on the wet etching of the Si frame in a warm KOH bath. KOH possesses a high selectivity of SiN to Si, and SiN can thus be used as a hard mask to locally etch the Si at a high rate. This property can be used for fabricating various shapes of anchored membranes or strings of SiN [1,3-5].

Releasing an unpatterned SiN membrane by itself is a relatively straightforward process, with high yield and high margin of error. Conversely, it has repeatedly been observed that the release of a membrane patterned in such a way than thin structures exist within it requires more meticulous handling of the samples [3,6]. Moreover, the addition of air holes in the SiN membrane (see Figure 1) adds another channel for the KOH to etch the Si, which must absolutely be blocked for the release to be successful. Several approaches involve the use of watertight PEEK holders, protecting the membrane side [1,3].

Here, we present an alternative etching process which differs slightly from the usual approach to release these PnC structures; the membrane side is protected, rather than by an external holder, by a thick layer of photoresist lying on the surface on the chip for the duration of the KOH etch.

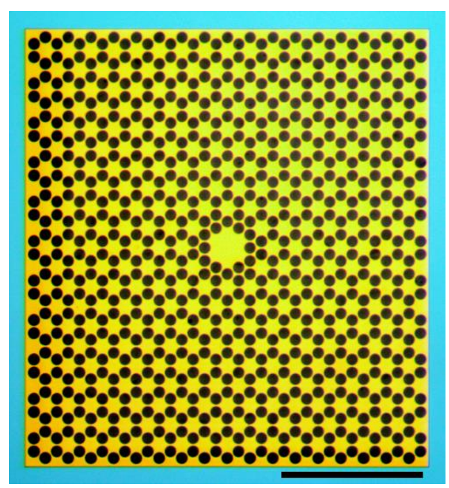

Figure 1- Optical micrograph of a patterned SiN membrane.
In yellow, the freely standing SiN; in blue, the SiN above Si; in black, air holes. The black scale bar represents 1 mm.

The bought wafers are initially diced into 1cm × 1 cm chips, before being bathed in an ultrasound bath of hot acetone, at 50°C. After each acetone bath, a chip is systematically rinsed in isopropanol before drying with N_2. The procedure is then as follows, illustrated in figure 2:

- Step 1: a single chip is rinsed as above.
- Step 2: before any further procedure, the side of the chip on which the membrane will be fabricated (the *frontside)* is covered with a protective coating of photoresist AZ 5214 E. The frontside is chosen based on best surface cleanliness. The resist is then spun onto the other side of the chip (the *backside)*, and a window is patterned by h-line laser lithography (on the Microtech Laserwriter LW405C). The developer used is AZ 726 MIF.
- Step3: the SiN window on the backside is etched by reactive ion etching (RIE) involving a mixture of CHF3 and SF6 gases.
- Step 4: the sample is cleaned in a hot ultrasound acetone bath, followed by a dip in IPA.
- Step 5: resist is spun onto the frontside, which is patterned with the phononic crystal structure by laser lithography.
- Step 6: the SiN on the frontside is etched by RIE
- Step 7: another acetone/IPA cleaning cycle is undergone.
- Step 8: for the duration of the KOH etch, the frontside is protected with a thick photoresist unaffected by KOH: ProTEK PSB [7].
- Step 9: the Si is etched in a KOH bath, with a concentration of approximately 30%. The etch consist of two phases: first, the KOH is heated to 85°C, into which the sample is dipped, held by a Teflon holder keeping it vertical during the duration of the etch (see Figure 3). To ensure homogeneity throughout the liquid, the KOH is stirred with a magnetic stirrer at 200 rpm. When an estimated less than 50 µm of Si remain, the temperature is decreased to 75°C to reduce the amount of H_2 bubbles generated, and the stirring is interrupted.
- Step 10: after the KOH etch is fully finished and the SiN membranes are fully released (which can be judged by eye), the samples are transferred to a in a piranha solution (with a H_2O_2:H_2SO_4 ratio of 1:4) in order to remove the ProTEK. The process is finalized by a dip in deionized (DI) water, and IPA.
- (Optional) Step 11: in case the piranha etch was not successful in fully etching the Protek, a second, stronger piranha etch is undergone, with a H_2O_2:H_2SO_4 ratio of 1:1.

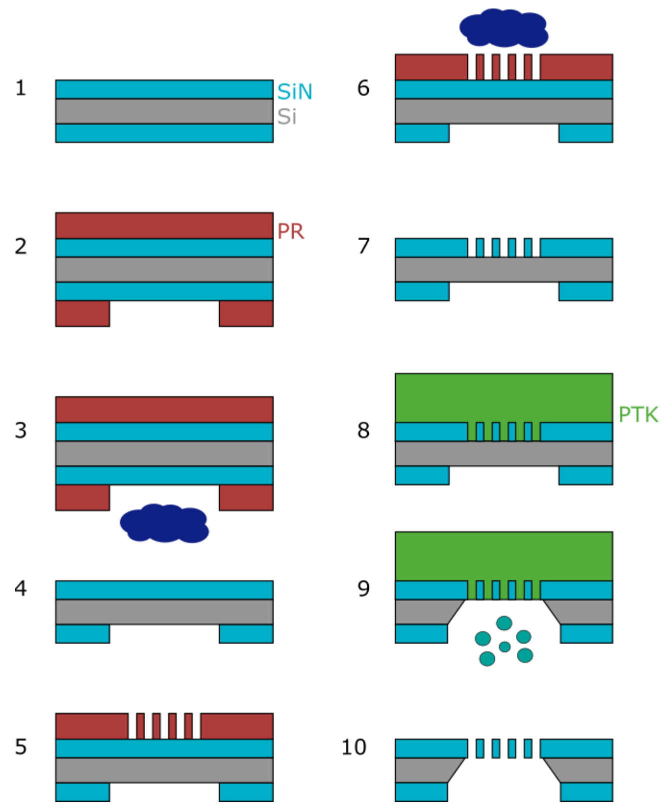

Figure 2- Illustrated fabrication process.
Blue: SiN; gray: Si; red: photoresist (PR); green: ProTEK PSB (PTK)

The precision in the width and height of the membrane window was of the order 1 µm, with an accuracy of the order of 1 µm, and good reproducibility from one bath to another. The centering of the PnC pattern was also determined, by measuring the distance of an outermost hole to the membrane border and comparing that to its design value. We found a precision in that distance of 2 µm, with an accuracy of 2 µm.

ADITIONNAL REMARKS

- Step 9: the empty Teflon holder is heated in the KOH until the temperature of 85°C is reached, to prevent a temperature dip when the sample is inserted. When 85°C are reached, the holder is removed from the solution, the chip is loaded, and the holder is finally returned to the solution.
  The verticality of the Teflon holder is employed to minimize viscous drag normal to the membrane surface during manipulations in the KOH; it serves as a way to ensure that the H_2 bubbles generated by the KOH etch do not affect with excessive force onto the membranes as well. Once the chip is loaded onto the holder, it is never removed until the process is fully terminated.
- Step 10: After the membranes are released, the sample is treated as delicately as possible. Any dip in a solvent or DI water simply consists of gently lowering down the holder into the solution, and letting is clean by diffusion for approximately 10 minutes.

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