



Fabrication of phononic crystals on free-standing silicon membranes



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ABSTRACT

Free-standing Si films have been and remain an excellent example to study experimentally the effect of the reduction of the characteristic size on the phonon dispersion relation. A step further in geometrical complexity and, therefore, in increasing the control and manipulation of phonons is achieved by introducing periodicity in the medium to form phononic crystals.

Here we report on the development of the fabrication process of large-area, solid–air and solid–solid two-dimensional phononic crystals, directly on free-standing, single crystalline silicon membranes.

The patterning of the membranes involved electron-beam lithography and reactive ion etching for holes or metal evaporation and lift-off for pillars.

The fabrication was possible due to the external strain induced on the membrane in order to reduce the buckling, which is typically found in large area free-standing structures. As a result, we obtained 250 nm thick structured membranes with patterned areas up to $100 \times 100 \mu\text{m}$, feature size between 100 and 300 nm and periodicity between 300 and 500 nm. The changes in dispersion relations of hypersonic acoustic phonons due to nanopatterning in free-standing silicon membranes were measured by Brillouin light scattering and the results were compared with numerical calculations by finite elements method. Information on phonon dispersion relation combined with a reliable fabrication process for large-scale structures opens a way for phonon engineering in more complex devices.

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1. Introduction

Phononic crystals (PnCs) are in general periodic structures made of materials with different elastic properties, with lattice spacing comparable to the acoustic wavelength [1,2]. In analogy to photonic crystals, where propagation of certain electromagnetic waves is prohibited by creating a photonic band gap, a phononic band gap can appear in properly designed PnCs.

Engineering of the phonon dispersion relation provides the means to impact on related properties of materials in a controlled manner. In this context, silicon nanomembranes, which are component parts in integrated photonic, plasmonic, mechanical and magnonic devices, appear as an excellent example to study the effect of the reduction of the characteristic size on the phonon dispersion relation and its influence on phonon propagation or material properties such as thermal transport [3–5]. The control and manipulation of phonons can be achieved by introducing periodicity in the elastic properties of the nanomembrane to form phononic crystals [6–8]. Alternatively, placing a periodic arrangement of an array of cylindrical resonators on the membrane can result in the appearance of hybrid slow phonons [9].

The fabrication of free-standing silicon PnCs-patterned membranes is typically based on processing of the silicon-on-insulator (SOI) wafer with a thin top silicon device layer. The device layer is patterned by means of lithography and reactive ion etching (RIE) and the buried oxide layer (BOX) is removed to obtain a suspended structure [3, 10–12]. However, this method has many disadvantages. First of all, the suspended area has to be relatively small to prevent from bending or collapsing of the structure after suspension. Second of all, etching of the BOX requires harsh chemicals, such as hydrofluoric acid, and critical point drying.

Alternatively, focused ion beam (FIB) milling can be used to pattern holes, as it is often done in case of photonic crystals [13]. This approach allows fabrication of large scale PnCs with well controlled diameters, but is usually time consuming. Moreover, the profile of the holes is not perfectly straight: the top diameter is larger than the bottom one [14,15].

In this article we present a method to fabricate phononic crystals with dimensions of $100 \times 100 \mu\text{m}$, based on free-standing silicon membranes, which avoids the abovementioned problems. With the presented process the suspended areas can be larger than in case of fabrication with SOI wafers and the integration of Al fabricated structures like, for example, electrodes, plasmonic or phononic structures is easier. This fabrication process was already successfully realized using SiN membranes for holey phononic crystals [16].

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First of all, we show that a thin layer of poly(methyl methacrylate) (PMMA) on top of the membrane induces enough strain to reduce the buckling, typical for thin, stress-free membranes. The exact profile of the membrane with and without the PMMA was obtained using optical profilometry. Then we present a fabrication process for obtaining large area PnCs, both holes and metallic pillars. Finally, we compare the Brillouin spectra of the plain and holey membrane and resolve the phonon modes using finite element method calculations (FEM).

2. Fabrication methods

We have adapted the electron beam lithography (EBL) and RIE pattern transfer process in order to fabricate square-lattice, holey PnCs over large areas of a free-standing membrane. We have also performed EBL and, metal evaporation in order to achieve square-lattice of aluminium nanopillars on the membrane. In our process we used commercially available, single crystal silicon (100) membranes (Fig. 1 inset), with a window size of $3.2 \times 3.2 \text{ mm}^2$ and 250 nm thick (Norcada Inc.).

These ultra-thin silicon membranes were slightly wrinkled due to the low stress and thickness of the membrane film. The buckling is typical for large free-standing membranes, unless external strain is applied [17,18]. In our approach we used a layer of PMMA, approximately 300 nm thick, on the sample, which induced enough strain in order to flatten the membrane.

The membrane profile was measured using optical profilometry on both pristine and PMMA-covered membranes. The pristine membrane shows clear buckling, but after being covered with a thin polymer layer it becomes flat, as shown in Fig. 1. In both of the figures we show a part of the bulk membrane frame as a reference.

The PMMA 950k (Allresist) was spun at 4000 rpm for one minute, followed by one hour bake at 100 °C in an oven. We designed PnCs with hole diameters between 100 and 250 nm with a constant period of 300 nm. The EBL (Raith 150-TWO) was performed at 20 kV acceleration voltage, with a different dose for each hole diameter ($320\text{--}360 \mu\text{C}/\text{cm}^2$) in order to reduce the proximity effect. The writefield area was of $100 \times 100 \mu\text{m}$ and this was the maximum size of the phononic structures. After development in 1:3 methyl isobutyl ketone:isopropanol (MIBK:IPA) the samples were post-baked for one minute at 80 °C on a hot plate. This step additionally hardened the PMMA mask before the RIE.

The pattern was transferred to silicon using the Bosch process (Alcatel AMS-110DE). The source power was set to 500 W and the flow of SF_6 and C_4F_8 gases was of 150 sccm and 100 sccm, respectively. The etching time varied between 30 and 60 s, depending on the hole diameter. Finally, after pattern transfer the samples were placed in a plasma system (PVA Tepla), and cleaned in 50 sccm O_2 at 400 W for one minute.

For the preparation of PnCs made of metal nanopillars the same spin-coating and EBL process as described previously were used, adjusting the exposure dose between 360 and $380 \mu\text{C}/\text{cm}^2$. The EBL was followed by metal evaporation of 5 nm Ti and 70 nm Al (ATC

Orion, Telemark). In this case we fabricated structures with the pitch of 400 and 500 nm and pillar diameter of 300 nm. Lift-off was done in acetone bath at 50 °C.

3. Experimental technique

Measurements of the dispersion relations of phonons propagating both in pristine membranes and phononic crystal membranes were performed by means of Brillouin light scattering (BLS). Brillouin light scattering spectroscopy allows the investigation of thermally activated acoustic phonons in the GHz range of frequencies (0.2–250 GHz). BLS is a well-established technique commonly used in non-destructive testing of elastic properties and structural phase transitions in bulk materials [19–21]. BLS has been also found as an excellent tool to characterize phonon propagation in the nm-scale systems, such as ultra-thin free standing membranes [22] and phononic crystals [9]. BLS experiments provide information on the relative change in the frequency (Stokes and anti-Stokes components) of laser light undergoing inelastic coherent scattering by acoustic phonons. For opaque or semi-transparent materials, the main contribution to the scattered light comes from the surface ripple mechanism. Brillouin spectroscopy measurements were performed on a six-pass tandem type Fabry–Perot interferometer (JRS Scientific Instruments) in the p–p (incident and scattered light polarization parallel to the plane of incidence) backscattering geometry. The light source was an argon gas laser generating light at $\lambda = 514.5 \text{ nm}$. For the backscattering geometry the angle of the laser beam incidence onto a given surface studied is equal to the scattering angle and denoted by θ . The magnitude of the scattering wave vector \mathbf{q} is given by [19–21]:

$$q = \frac{4\pi \sin\theta}{\lambda}. \quad (1)$$

For periodic structures such as phononic crystals, the scattering wave vector \mathbf{q} is defined by momentum conservation: $\mathbf{q} = \mathbf{k} + \mathbf{G}$, (2) where \mathbf{k} denotes the wave vector of an acoustic phonon and \mathbf{G} is a reciprocal lattice vector.

To get a correct dispersion relation for the acoustic waves propagating in 2D phononic crystal membranes we use the FEM approach. The FEM calculations were performed using frequency domain module (COMSOL Metaphysics) and the silicon material properties are gathered in Table 1.

4. Results and discussion

Using the above-described procedure we successfully fabricated free-standing holey PnCs with thickness of 250 nm and with a variety of filling factors (relation between hole area and unit cell area), ranging from 10% to 50%, as shown in Fig. 2. The largest filling factor corresponds to the hole diameter of 240 nm, which means that the walls between the holes are 60 nm thick. There is still room for improvement and achieving even larger filling factor, as the limit for standard nanofabrication is around 20 nm.

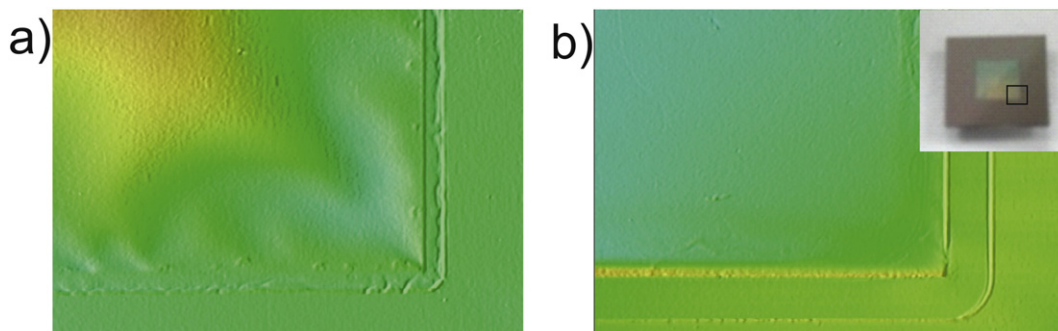


Fig. 1. Optical profilometry images of a) pristine and b) PMMA-covered membranes. Inset: photo of a Norcada chip indicating the studied area.

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