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Enabling electromechanical transduction in silicon nanowire mechanical resonators fabricated by FIB ion implantation

J. Llobet [a], M. Sansa [a], M. Gerbolés [a], N. Mestres [b], J. Arbiol [b,c], X. Borrisé [d], F. Pérez-Murano [a]

[a] Institut de Microelectrònica de Barcelona (IMB-CNM CSIC), Bellaterra 08193, Catalonia, Spain

[b] Institut de Ciència de Materials de Barcelona (ICMAB-CSIC), Campus UAB. 08193- Bellaterra, Catalonia, Spain

[c] Institució Catalana de Recerca i Estudis Avançats (ICREA), 08010 Barcelona, Catalonia, Spain

[d] Institut Català de Nanociència i Nanotecnologia (CIN2), Campus UAB. 08193- Bellaterra, Catalonia, Spain

e-mail: jordi.llobet@csic.es, Francesc.Perez@csic.es

We present the fabrication of silicon nanowire (SiNW) mechanical resonators by a process based on focused ion beam local gallium implantation, selective silicon etching and diffusive boron doping. We obtain SiNWs with good electrical conductivity and lateral dimensions as small as 25 nm. During the fabrication process, gallium implantation induces the amorphization of silicon that, together with the incorporation of gallium into the irradiated volume, makes the electrical resistivity to increase to values higher than 3 Ω•m, resulting in an unacceptable too high resistance for electrical transduction of the SiNW mechanical oscillation. We show that the conductivity of the SiNWs can be restored by performing a high-temperature doping process, which allows to recover the crystalline structure of the silicon and to achieve a controlled resistivity of the structures. Raman spectroscopy and TEM microscopy are used to characterize the recovery of crystallinity, while electrical measurements show a resistivity of 10^{-4} *Ω•m. This resistivity is adequate to obtain an optimal electromechanical transduction mechanism that enables to characterize the high frequency mechanical response of suspended silicon nanowires by electrical methods.*

1. Introduction

Nanometer scale structures and devices offer multiple advantages that arise from their small dimensions. For example, they can be more energetically efficient, they empower the integration of multiple devices in a single monolithical substrate and they can provide improved or new properties to systems compared to their micrometer-sized equivalent. Mechanical resonators are very promising building blocks for the realization of miniaturized systems like mass sensors or integrated oscillators for telecommunications. [1,2] Size reduction of suspended structures towards the nanoscale allows to build up high resonance mechanical resonators, which present unprecedented sensitivities for mass detection.[3–5] The overall performance of a nanomechanical resonator is not only dictated by the dimensions and resonance frequency, but also by the rest of its mechanical and material properties (quality factor, elastic constant, Young modulus, density, built-in stress), morphology (including surface smoothness and clamping) and by the possibility to detect its oscillation in an efficient and sensitive way. In this sense, when developing nanomechanical resonators, the interplay between fabrication, materials properties and system integration issues must be considered to obtain an operative, efficient device with optimal performance.

Despite the progress being made in the area of nanomechanical devices and systems, a robust, simple and scalable method of fabrication is still not available. Methods based on electron beam lithography (EBL) are usually employed [6], but they rely on a complex sequence of fabrication processes (resist deposition, alignment, e-beam exposure, lift-off and high resolution reactive ion etching) which are difficult to scale-up or present critical features to ensure their reproducibility at the nanoscale. Higher fabrication throughput can be achieved by means of using high resolution optical lithography [7,8] but then the complexity of the process increases either by the use of expensive lithography tools or by the need of additional oxidation/etching processes to further reduce the dimensions of the silicon nanowire. One alternative approach consists on the use of nanometer scale objects to build-up the resonator. Nanomechanical resonators with outstanding performance have been realized by using carbon nanotubes [3,9] and silicon nanowires grown by the vapor liquid solid mechanism [10,11].

Another alternative is to fabricate nanomechanical resonators by direct milling using a focused ion beam (FIB) [12]. In this case, high energy gallium ions are used to locally remove material from local selected areas. The main advantage resides in the fact that no resist is needed, preventing difficulties that appear when the surface to be structured is not flat or it contains pre-patterned features of significant topography. However, the use of FIB processing for fabrication is limited due to its low throughput. In addition it can potentially induce material damage [13–15].

A strategy to overcome this limitation is to expose the silicon surface at lower doses in order to modify the silicon substrate via ion implantation of gallium. The implanted silicon acts as a mask for a subsequent etching, which can be either wet or dry etching. The process has been used for many years [16,17]. Several groups have shown that the process is useful for the fabrication of nanostructures in silicon [18–26], silicon dioxide [27], diamond [28], titanium [29], aluminum oxide [30] among others materials. The performance of the implanted area to be used as a mask for transfer pattern has been demonstrated for wet etching [17,19,21,23] and for dry etching [18,22]. The approach of using the implanted layer as a mask for transferring features to the underlying bulk silicon has also been used for the realization of micromechanical devices [20].

On the other hand, it has also been demonstrated the use of the gallium implantated layer as the structural device material [17,19,21,23,31]. However, the electrical conductivity of the FIB implanted area is poor, due to the damage caused by the exposure to the ion beam, preventing their use as functional nano-electromechancial devices with electrical read-out. Up to now, very few attempts have been reported to improve electrical properties of the gallium implanted layers [24].

In this article, we present the fabrication and characterization of nano-electro-mechanical resonators by using the ultra-thin gallium implanted layer as a structural and functional conductive material. The conductivity of the SiNWs is optimized by performing an annealing process in a boron environment after the FIB ion implantation and wet chemical etching. The overall fabrication process is a very convenient way for the prototyping of nanomechanical resonators with electrical read-out because it combines a simple and fast fabrication method together with good electromechanical properties. We demonstrate its applicability by the characterization, by purely electrical methods, of the frequency response of mechanical resonators based on double clamped silicon nano-beams, indicating that they present an efficient electromechanical transduction mechanism.

2. Fabrication process and material characterization

The fabrication of suspended silicon nanowires made of the ultra-thin FIB gallium implanted layer is simple and reliable, and it does not require a critical surface preparation, since it proceeds correctly even in the presence of silicon native oxide. To perform the pattern design and exposure, we have used dedicated control software [32] that allows to define complex structures and to implement blind navigation, avoiding electron and ion irradiation in undesirable areas. For the structures that we show in this article, FIB gallium implantation is typically performed at 10 pA, with areal fluences of $1 \cdot 10^{16}$ at/cm^2 and an acceleration voltage of 30 kV. Currents from 1 pA to 2 nA and fluences from $3 \cdot 10^{15}$ to $3 \cdot 10^{16}$ at/cm^2 are suitable. Can you say something here on the typical implantation depth and on the expected thickness of the released structures? TRIM simulation ???

For this acceleration voltage the implantation is expected to occur at the surface silicon layer and up to a depth of 40 nm ??…..

Once all the structures are defined via FIB implantation, the etching of the underlying Si is performed. We use the wet silicon etchant TMAH (tetramethilammonium hydroxide) at 25% of concentration and at a temperature of 80ºC, resulting in a silicon etching rate of 0.4 μm/s for the (100) plane and 0.9 μm/s for the (110) plane. We take advantage of the anisotropic etching rate of TMAH to obtain released or non-released nanowires according to their specific orientation. Fig. 1 shows two examples of arrays of silicon nanowires fabricated by this method on a (100) silicon surface. The nanowires of the structures shown in Fig. 1.a are defined following the <110> direction, resulting in non-released nanowires because the (111) plane presents a very low etching rate, preventing the occurrence of under-etch. In the case of figure 1.b, the nanowires are defined following the <100> direction, and in this way, they become released after the TMAH etching because the perpendicular plane is etched as well. We observe in the structures of Fig.1 that the clamping pads become also under-etch. By defining the sides of the clamping pads in the proper direction, the under-etch area can be minimized or maximized, which has important implications for the final mechanical behavior of the structures. We have used a modelling software to determine the proper orientation of the designs in order to obtain the desired shape. In the supplemental information we show results of the modelling of the etching process and its comparison with the fabricated structures.

During the fabrication process, Ga implantation induces the amorphization of silicon in the irradiated volume, that, together with the incorporation of Ga, increases the electrical resistivity of the wires to values higher than 3 Ω·m. In order to overcome this limitation, we have included an additional step consisting on the post-annealing at high temperature in a boron environment, allowing the recovering of the crystalline structure and the improvement of the electrical conductivity of the silicon nanowire.

Firstly, we study the recovery of the crystal structure in the gallium implanted volumes by Raman spectroscopy and TEM. It is desirable that SiNWs will be made of a crystalline material not only because they will present better electrical properties and eventually a good transduction mechanism, but also because it is known that the mechanical properties of crystalline silicon are better than the ones of amorphous silicon. Raman characterization allows quantifying the crystalline damage in low dimensional structures. [33] Raman spectra are obtained on six different silicon chips from the same wafer submitted to different annealing process. The first chip is used as the undamaged crystalline silicon reference. The other five contain 100 x 100 μm^2 areas of FIB implanted gallium at fluences of $1\cdot10^{16}$ at/cm^2 . After the implantation, each chip has been submitted to a thermal annealing at different temperatures (without thermal process, 200ºC, 400ºC, 600ºC and 800ºC) for 40 minutes in a nitrogen atmosphere. Figure 2 clearly shows a very large reduction of the LO-phonon mode intensity at ~521 cm^{-1}, which is associated with a large amount of damage. The crystalline structure of silicon, which is damaged after the FIB ion implantation, is partially recovered after a high temperature annealing concordant with the recovery of the LO-phonon intensity. A quantification of the damage recovery after the thermal treatment can be extracted from the evolution of the LO-phonon Raman intensity $1-I_{imp} / I_{virgin}$, with annealing temperature, and reaches an average value above 80% for the annealing performed at 800 °C.

The crystalline structure integrity of silicon after the ion implantation has also been studied using TEM (Transmission Electron Microscope). Two lamellae coming from the same silicon chip and containing two previous FIB implanted areas have been fabricated. The first lift-out lamella has been extracted before an annealing (Fig. 3.a-c) and the second one after a 600ºC and 30 minutes annealing step (Fig. 3.d). It is observed that the silicon becomes amorphized up to a depth of 39 nm (Fig. 3.a). The atomic concentration of gallium and oxygen is analyzed by electron energy loss spectroscopy (EELS) showing only a residual oxygen presence in the implanted area (Fig 3.b-c marked as 2) because of the thin native oxide intrinsically grown on both lamella faces. After the annealing, the disordered volume of silicon is partially recovered showing the formation of nanometer scale grains of polycrystalline silicon containing crystalline defects and twinned silicon structures, in agreement with the previous Raman spectroscopy measurements. In figure 3.d it is observed an epitaxial crystalline recovery showing the formation of twins from the bulk silicon.

After the Ga implantation, the conductivity of the silicon nanowire is almost entirely suppressed, which precludes the exploitation of the electromechanical properties of the nanostructure for the building up of functional electro-active devices. For nanometer scale mechanical resonators, mechanical to electrical signal transduction at high frequency becomes a challenge, imposing additional requirements to the fabrication process. In particular, control of the conductivity of the nanowire is critical to obtain good electromechanical transduced signals.

While high temperature annealing allows to recover the crystal structure, outer diffusion of dopants from the suspended silicon nanowires provokes that, after the annealing, the conductivity of the nanowires is lower than that of the original bulk doped silicon. In order to overcome this limitation, the annealing is performed in a boron environment, so that the conductivity is improved at the same time that the crystal structure is recovered.

A previous work [24] showed that the annealing of suspended silicon nanowires by passing an electrical current through them (Joule heating) allows to decrease their resistivity two orders of magnitude. However, they report morphological changes in the nanowire that may reduce its mechanical properties. Moreover, Joule heating can only be performed in individual, double clamped silicon nanowires. In our case, the nanowires are doped by annealing the sample in close proximity to a boron nitride wafer into the furnace. We have used a customized atmospheric furnace to perform the annealing/doping at a temperature of 850ºC-1000ºC. In this way, batch fabrication is possible because multiple suspended silicon nanowires can be anneal/doped at the same time, and it can be applied to single or double clamped silicon nanowires as well as to other types of nanomechanical resonators. To study the influence of this thermal step we study the presence of gallium after the annealing by microanalysis characterization using a SEM equipped with an energy dispersive X-ray (EDX) detector. The characterization shows the evolution of the gallium signal after the ion implantation (Fig. 4a) and after the doping at high temperature (Fig. 4b). Remarkably, gallium is not detected after the annealing process, and the oxygen signal does not increase after this step, discarding the growth of silicon dioxide on the structures. Boron dopant atoms cannot be detected because of their low atomic number.

3. Fabrication and characterization of electrically active suspended silicon nanowires

We present now the overall fabrication method that we have developed for the definition of ultra-thin (< 40 nm) free-standing mechanical structures. It presents the advantages of being simple, fast, and flexible, allowing the implementation of multiple designs with high dimensional control. To increase the fabrication throughput, a mix and match strategy with optical lithography has been implemented: the areas corresponding to the contacting pads and lines for electrical measurements are defined by optical lithography and standard boron ion implantation, while the areas corresponding to the nanomechanical structures, which require higher resolution, are defined by FIB Ga^+ implantation. A similar process was proposed in ref [24].

Chips cut from silicon on insulator (SOI) wafers are employed as starting material The SOI layer has a (110) orientation and an initial resistivity of around 10^{-3} $\Omega\cdot$m (boron doping). A 1µm thick SiO_2 layer is grown on top by dry oxidation. The SiO_2 layer is patterned by optical lithography and wet chemical etching. Using the remaining SiO_2 as a mask, the silicon surface is selectively doped by boron ion implantation at an energy of 12 keV and at a fluence of $1\cdot10^{16}$ at/cm^2. After this implantation, the SiO_2 mask is removed.

The next step consists in a direct ion implantation of gallium to define the nanostructures. An alignment with the previously micro-patterned areas is performed by imaging the surface with the SEM: when using the inlens detector, the boron implanted areas appear brighter than the unmodified surface while the FIB gallium implanted areas appear darker (see supplemental information).

After the gallium implantation, the chip is immersed in the TMAH solution. The etching time used for these structures is 5 minutes, to ensure that the whole thickness of the SOI layer is completely etched away. Then, the chip is transferred to the atmospheric furnace for annealing and doping.

Fig. 5 shows a measurement of the conductivity of several nanomechanical resonators based on suspended silicon nanowires, after the annealing and doping process (TIME and TEMPERATURE conditions). Figures 5.a and 5.c shows devices based on double-clamped suspended silicon nanowire. All the nanowires have a rectangular cross-section with a thickness (height) of 40 nm and width in the range from 80 nm (Fig 5.a) to 250 nm (Fig 5.c). The device in figure 5.a was entirely defined by FIB implantation, while the device in figure 5.c was realized by the mix-and-match ?? with optical lithography process as explained above. In both cases, the devices present an ohmic behavior (see figures 5.b and 5.c) and the value of the nanowire resistivity is around $1\cdot10^{-4}$ $\Omega\cdot$m, which corresponds to a boron impurity concentration of $4.5\cdot10^{18}$ at/cm^3. The conductivity of these devices is optimal to electrically detect the frequency response of the silicon nanowire oscillation as it is shown in the next section.

An example of the usefulness of the fabrication process for the experimentation on nanoelectromechanical structures is shown in figure 5.e, realized by using the combination of optical lithography and ion beam exposure. In this case, the device is made of several double clamped suspended silicon nanowires connected in parallel. The conductivity of the device is monitored in real time while the nanowires are sequentially cut by the FIB operating in the milling mode. Figure 5.f reveals how the ion milling process acts to decrease the conductivity of each individual nanowire.

4. Electrical detection of the frequency response of suspended silicon nanowires

We now study the mechanical behavior of nanoresonators fabricated using the previously described process. The devices consist on double clamped silicon nano-beams with a side-gate nearby for electrostatic actuation. An SEM image of one of the fabricated devise is shown in Fig. 6.a. The length of the nanowire is 3.6 μm and the dimensions of the cross-sectional area are 40 nm (thickness) x 230 nm (width). The flexibility of the process allows to implement multiple designs, incorporating not only suspended nanowires but also suspended membranes, enabling the investigation of nanomechanical phenomena like mode coupling and non-linear behavior.

As the suspended silicon nanowires are now conductive at the end of the fabrication process, the electrical read-out of their mechanical oscillation is enabled. We measure their frequency response by using a frequency modulation (FM) electrical down-mixing method, which has been previously demonstrated to provide outstanding performance for the electromechanical characterization of nanomechanical resonators based on double clamped bottom-up grown silicon nanowires. [34] The measurements are performed inside the vacuum chamber of the FIB system using three micromanipulators with electrical connection for low-resistance and low-noise electrical measurements. [35] This set-up allows for electrical characterization up to frequencies of 150 MHz

Fig. 6.b shows the measurement results for the SiNW mechanical resonator shown in figure 6.a, with a resonance frequency close to 97 MHz. The shape of the resonance peak is the one usually obtained when using the FM down-mixing detection method. The quality factor of the resonance is around 400. The electromechanical response of these structures is comparable to the results obtained in structures fabricated using other methods [34,36]. In addition to the resonance shown in Fig. 6.b, additional resonance modes are observed due to the presence of the membranes, indicating the appearance of mechanical coupling. Moreover, we observe little variability between the frequency response of different resonators with the same design, confirming the high repetitiveness of the fabrication process.

5. Conclusions

A fast and flexible fabrication method based on ion implantation, wet etching and an annealing/doping process has been used to realize double clamped, conductive silicon nanowire mechanical resonators. A mix and match fabrication approach based on the combination of serial (FIB ion implantation) and parallel (photolithography and boron ion implantation) processes permits to increase the fabrication throughput.

From the material point of view, this fabrication process eliminates issues typically associated with ion beam Ga^+ implantation. The high temperature annealing process recovers the crystalline structure of silicon and the high temperature boron doping allows to control the electrical conductivity of SiNWs. In consequence, suspended SiNWs with good mechanical and electrical properties are obtained. The electrical characterization of the frequency response shows a good mechanical response and an efficient electromechanical transduction mechanism.

In comparison with other approaches to fabricate nanomechanical resonators, the described process is much simpler and flexible, and it allows obtaining functional devices in less than one day of clean room work. While the fabrication of nanostructures and devices by the combination of FIB implantation and silicon etching has been demonstrated since more than 20 years, we believe that the fact that the conductivity of the resulting layer can be controlled and that the mechanical properties of the resulting suspended structures are good will push further the use of the method for the prototyping of nanomechanical structures, with the aim of experimenting new geometries and phenomena at the nanoscale. Moreover, we would not discard a possible further increase of the fabrication throughput (for example, by doing the gallium implantation through stencil masks [37]), opening the possibility to apply this methodology to obtain devices and integrated nanosystems at industrial scale.

Acknowledgments:

ANEM, Atometer, SGR, Nanocanales, Infraestructura, explora

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