

Publication IV

Nikolai Chekurov, Kestutis Grigoras, Antti Peltonen, Sami Franssila, and Ilkka Tittonen. 2009. Localized gallium doping and cryogenic deep reactive ion etching in fabrication of silicon nanostructures. In: Daryush Ila, Paul K. Chu, Naoki Kishimoto, Jörg K. N. Lindner, and John E. E. Baglin (editors). Proceedings of the 2009 MRS Spring Meeting & Exhibit: Symposium DD – Ion Beams and Nano-Engineering. San Francisco, CA, USA. 13-17 April 2009. Warrendale, PA, USA. Materials Research Society. Materials Research Society Symposium Proceedings, volume 1181, 1181-DD07-01, 6 pages. ISBN 978-1-60511-154-4.

© 2009 Materials Research Society (MRS)

Reprinted by permission of Cambridge University Press.

Localized Gallium Doping and Cryogenic Deep Reactive Ion etching in Fabrication of Silicon Nanostructures

Nikolai Chekurov^{1,2}, Kestutis Grigoras^{1,2}, Antti Peltonen^{1,3}, Sami Franssila^{1,2}
and Ilkka Tittonen^{1,2}

¹ Department of Micro and Nanosciences, Helsinki University of Technology,
PO Box 3500, FIN-02015 TKK, Finland

² Center for New Materials, Helsinki University of Technology, PO Box 3500, FI-02015 TKK,
Finland

³ TKK Micronova, Helsinki University of Technology, PO Box 3500, FI-02015 TKK, Finland

ABSTRACT

We present a novel fabrication method to create controlled 3-dimensional silicon nanostructures with the lateral dimensions that are less than 50 nm as a result of a rapid clean room compatible process. We also demonstrate periodic and nonperiodic lattices of nanopillars in predetermined positions with the minimum pitch of 100 nm. One of the uses of this process is to fabricate suspended silicon nanowhiskers.

INTRODUCTION

Instead of using focused ion beam (FIB) in milling the target by bombarding it with gallium ions, the doping of silicon is known to lead to selective masking of the surface in etching [1]. Especially the wet etch process [2, 3] is known to be sensitive for gallium ion doping. More recently, the dry processes have been studied in association with gallium implantation especially reactive ion etching with various chemical compositions and its derivatives such as deep reactive ion etching techniques (DRIE).

The main drawback of the reported methods is a poor selectivity between treated and untreated areas of the sample (dry etching) or crystallographic anisotropy restrictions (wet etching). In dry etching, the selectivity values in the range of 1-2.5 [4] have been demonstrated. In this work we describe a combination of local gallium implantation and cryogenic deep reactive ion etching which enables selectivity of least 2000:1 thus allowing fabrication of deep structures. By using the adjustable etching process one can achieve controlled underetching of the structures creating horizontally suspended nanowhiskers.

EXPERIMENT

The fabrication process consists of only two main steps (figure 1). First selected area of the sample is treated with Ga⁺ ion beam (FEI Helios Nanolab 600) and then DRIE (Oxford Instruments Plasmalab System 100) is used to machine the features by removing the untreated silicon. In analyzing the gallium dose needed to protect silicon from etching in the cryogenic DRIE we found out that moderate amount of 10¹⁶ ions/cm² is enough to produce structures of several μm in height. The dose is several orders of magnitude lower than the one needed for traditional direct FIB milling or FIB – assisted etching (10¹⁶ ions/cm² instead of

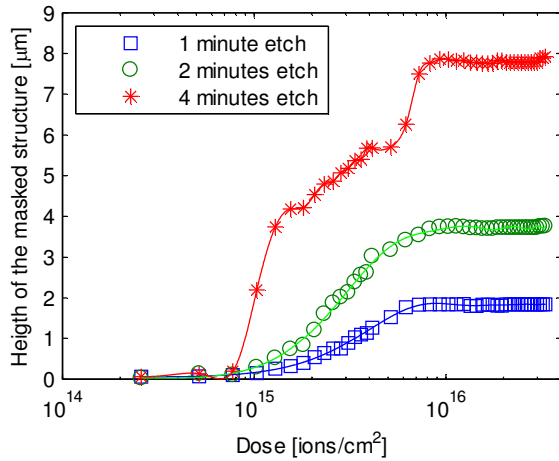


Figure 2. Influence of the amount of the gallium dose as a masking layer, the full height structures with doping dose $> 10^{16}$ ions/cm² indicate that the mask did not fail during etching. The structures with the gallium ion concentration $< 10^{16}$ ions/cm² experience a mask failure which is more severe for longer etching times.

To determine the resolution of the process, we created arrays of nanopillars with masks of different shapes. For this experiment the smallest available ion current (1.5 pA) and the shortest dwell time (100 ns) available were utilized. The treatment was repeated for 40 cycles resulting in $4 \cdot 10^{16}$ ions/cm². Figure 3 shows such pillars with square masking as well as parts of electronic masks and resulting structures from the same viewing angle and in scale. The measured widening of the structures was from 17 nm to 25 nm. A minimum linewidth was measured by creating a line pattern with varying thickness and spaces between the lines. As a result, 43 nm wide trenches and 45 nm wide lines were obtained.

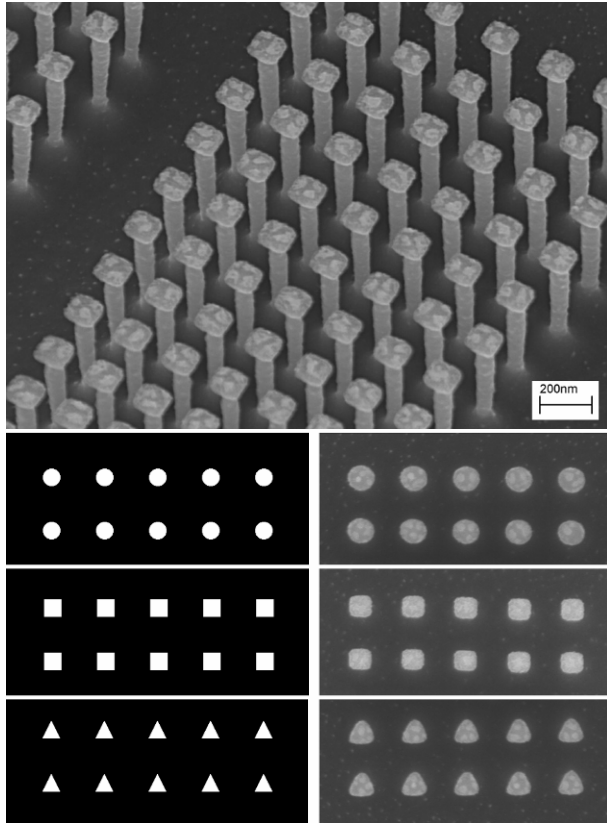


Figure 3. Nanopillar resolution test. Arrays of nanopillars (top) were fabricated, pictured directly from above (right) and compared to the starting mask (left). The widening of the structures measured to be in the range of 17-25 nm.

3D suspended structures can generally be fabricated with the same resolution as the non-suspended ones. Additionally, by using a relatively high etching temperature (-80 °C instead of -120°C), shrinkage of gallium doped layer occurs, making it possible to create nanobridges down to 20 nm wide (Figure 4).

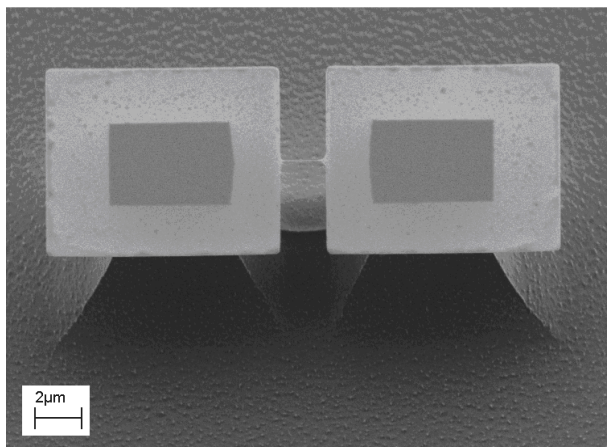


Figure 4. Smallest nanobridge obtained by etching at elevated (-80°C) temperature. The length of the bridge is 2 μm, width < 20 nm and thickness < 30 nm.

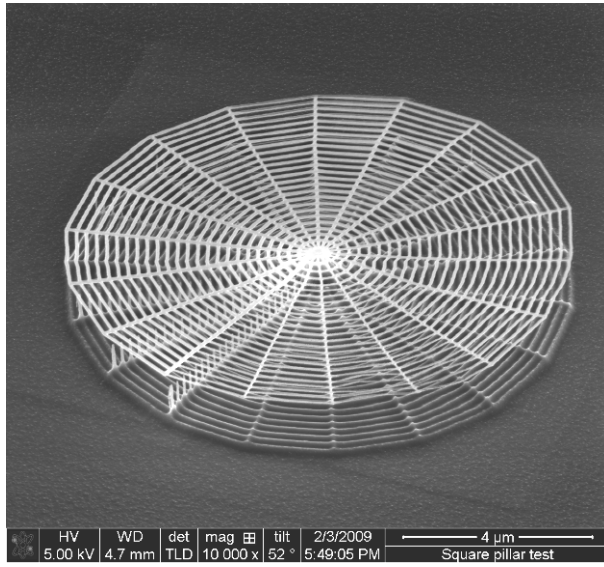


Figure 5. A floating web formation demonstrating toughness and extremely low stress of the resulting suspended nanowhiskers. The structure (10 μm in diameter) consists of crossing nanobridges from 50 nm to 100 nm wide. There is only little stress-related buckling visible.

DISCUSSION

Assuming that the penetration depth of the 30 keV gallium ions in silicon is around 30 nm, the selectivity between treated and untreated silicon is well over 2000 as structures up to 80 μm high were obtained in the mask stress experiments. The lateral widening of the structures was measured to be between 17 nm and 25 nm and the overall process resolution is 10 pairs of lines / μm . An achievable height-to width aspect ratio is measured to be more than 15:1, which means that e.g. pillars 600 nm high and 40 nm in diameter are possible. The repeatability of the process within one run is excellent, as several thousands of identical structures can be produced (Figure 6). The processing time is limited by the speed of the FIB – writing, because the speed of the DRIE – step is more than sufficient; 2 $\mu\text{m}/\text{min}$, and thus typical etch times are well below one minute. The FIB step takes, depending on the resolution and treated area, from microseconds to several minutes. By using different FIB – currents and view fields it is possible to create structures at an area of 10 x 10 μm^2 even with 150 μm times 150 μm bonding pads in under 15 minutes, so several design/fabrication/measurement cycles can be accomplished in one working day. For the freestanding structures, our current achievement is a nanowire with dimensions of 2 μm x 20 nm x 30 nm.

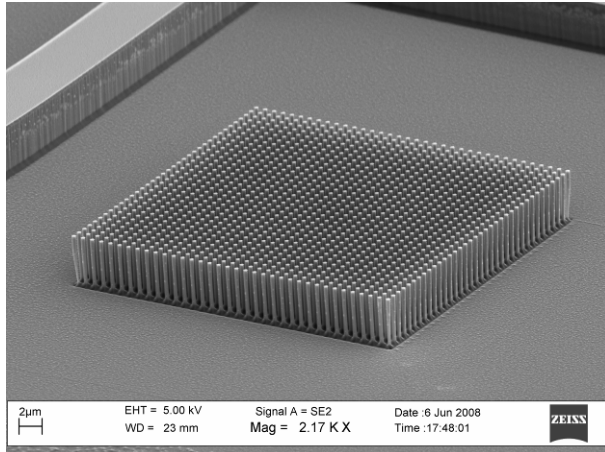


Figure 6. An array of more than 1000 identical nanopyllars 300 nm in diameter and 4 μm high.

CONCLUSIONS

The fabrication method producing silicon structures with a line width under 50 nm and with an aspect ratio of more than 15:1 has been developed. This quick, all-dry process can be utilized for several purposes in nanotechnology research and prototyping phases. Possible research areas, which would greatly benefit from possibility to quickly realize nanometer-sized structures are plasmonics and metamaterial or quantum phenomena research.

ACKNOWLEDGMENTS

Nikolai Chekurov acknowledges Magnus Ehrnrooths foundation for financial support.

REFERENCES

- [1] A. J. Steckl, H. C. Mogul, S. Mogren, *Applied Physics Letters*, **60**, 1883 (1992)
- [2] J. Brugger, G. Beljakovic, M. Despont, N. F. De Rooij, P. Vettiger, *Microelectronic engineering*, **35**, 401 (1997)
- [3] B. Schmidt, S. Oswald, L. Bischoff, *J. of the Electrochemical Society*, **152**, G875 (2005)
- [4] H. X. Qian, W. Zhou, J. Miao, L. E. N. Lim, X. R. Zeng, *Journal of Micromechanics and Microengineering*, **18**, 35003 (2008)