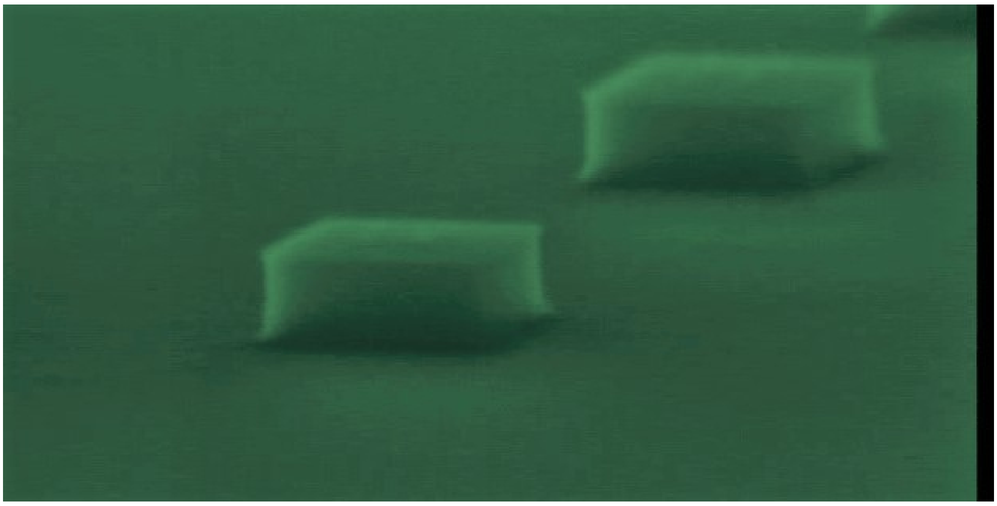
Final Report		
Ente Consiglio Nazionale delle Ricerche Istituto di Fotonica e Nanotecnologie	Tipo di documento Rapporto Tecnico	Classificazione Uso interno CNR
Riferimento	Numero documento 2	Pagine 13
Data 25/11/2016	Revisione Enrico Prati	

Final Report

Short Term Mobility 2016

Silicon single photon emitters based on Er implantation



Autore:	Coautori:	
Dr. Enrico Prati (CNR)	Prof. Takashi Tanii (Waseda Univ.) Maasa Yano (Waseda University) Ayman Abdelghafar (Waseda Univ.)	

Indice delle revisioni			
Revisione numero	Revisionato da	Motivazione	Data revisione

Indice degli acronimi	
SEM	Scanning Electron Microscope
RTA	Rapid Thermal Annealing

Definizioni	

Documenti di Riferimento

Questo Rapporto Finale fa riferimento ad articolo scientifici / rapporti tecnici pubblicati dagli Autori:

[1] E. Prati *et al.*, *Anderson–Mott transition in arrays of a few dopant atoms in a silicon transistor*, Nature Nanotechnology (2012)
[2] E. Prati, Report of the Short Term Mobility 2015 - *Process and pre-characterization of erbium doped silicon transistors* (2015)

SCOPO DEL DOCUMENTO

Questo documento costituisce il Rapporto Finale dell'attività svolta dal Dr. Enrico Prati dal 3 Novembre 2016 al 24 Novembre 2016 presso la Waseda University Tokyo nel contesto dello Short Term Mobility 2016 del CNR. Esso riporta i processi per la creazione di dispositivi di silicio drogati con erbio, un tipo di atomo rilevante per l'integrazione della fotonica nel silicio per le sue proprietà ottiche a 1540 nm, e con ossigeno.

Indice

1. Introduction

The fabrication of the devices for ErO implantation was carried at the Tanii's Lab at Waseda University, Tokyo. The objective was to fabricate silicon devices for single photon emission, namely silicon pillars to create optical microcavities by implanting Er and O, so photoluminescence may be achieved at 1520-1550 nm at room temperature. In order to take advantage of the stay, the determination of the size of the pillars, planned to be carried during the first week in the proposal, was partially anticipated before the start of the stay in Waseda. This decision allowed to save time during the stay in Japan and to employ it to develop the process in the cleanroom more effectively. According to photoluminescence measurement carried before the stay on bulk silicon substrates, we opted to implant 10^{14} cm^{-2} atoms of Er and O was implanted with a density of 10^{14} cm^{-2}. Notice that such decision follows our previous results that ratio of 1:6 is not needed to increase photoluminescence and 1:1 is sufficient, with benefits in terms of creation of limited number of defects. This ratio has been independently confirmed by a very recent publication on Scientific Report about the super-enhanced photoluminescence in the proximity of the ratio 1:1 [Lourenco et al. Scientific Reports, 2016]. Additional reference transistor devices have been prepared for complementary information extraction and to control effectiveness of implantation recipe.

2. Preparation of the 220 nm SOI substrates

Two different SOI materials have been employed in order to fabricate the resonant cavities. Here the silicon 100 SOI substrate (220 nm of silicon on 3000 nm of silicon oxide) is considered. The 220 nm SOI 6" wafer has been cut after protecting by PMMA by using a DAD321 automatic dicing saw. Wafer was first covered by layer PMMA, so the following process has been employed for removal:

1. Tetrahydrofuran (THF) 10 minutes
2. Acetone 10 minutes
3. Pure water 2 times
4. Repetition of steps 1-3 again.
5. Mixing of H2O2 and H2SO4 for 10 minutes
6. Pure water cleaning
7. BHF for 1 minute
8. H2O H2O2 and ammonium cleaning

The preparation of the diced substrates of 1 cm x 1 cm for electron beam lithography has followed this process:

1. Sample dried by nitrogen
2. Sample heated at 115 C for 1 minute
3. Spinning of the resist at 4000 rpm for 70 seconds (HMDS)
4. Same spin for SAL601
5. Baking 1 minute at 115 C

3. Electron beam lithography design and performing

In order to fabricate the cavities, the CAD design tool of the electron beam lithography machine has been employed. In order to exploit sub wavelength effects including Purcell effect, a range of different cavities has been designed. Two sub-half wavelengths sizes have been designed at 80 mm and 160 nm respectively, a resonant half wavalenght at 220 nm and a similar off resonance 250 nm have been designed, as well as double size at 440 nm and 500 nm respectively. The resonance set is completed by a 880 nm size, while off resonance are 750 nm, 1 um, 1.25, 1.5 2, 2.5 and 3 um respectively. Several combinations are assessed by a 14x14 matrix.

The CAD has been loaded in the Elionix ELS 7500 W EBL. In order to test different exposition doses, four different values have been employed of 40, 80, 160 and 320 uC/cm^2 respectively. The field size was 150 um by 60000 dots. By using a laser displacement meter Iwatsu ST-3708E, before performing the EBL, the thickness of the substrate has been measured of 379.35 um. The vacuum is pumped to 3.0 10^{-5} Pa, next the EBL process is started

After running the EBL, the operations have been completed by baking for 1 minute at 105 C and by using developer for 7m30s.

4. Silicon etching for pillar formation on the 220 nm SOI

At this stage the only resist is in correspondence of the section of the pillars to be etched, so the silicon is removed where resist has been already removed. The etching has been calculate to be around 230-240 nm per minute.

We performed ICP-RIE by SAMCO RIE 101 IPH by employing SF6. The evacuation of the gas line lasts of about 40 minutes. Next the SF6 has been delivered to the machine. The opening of the valve lasts just few seconds. Next the SF6 valve is closed again and the vacuum is pumped again along the pipes. The process is repeated in order to clean the equipment the first time, and also for the second time.

The same process has been repeated for the third time for the same reason. Next, a dummy wafer was loaded and cleaned by O2 gas which has been carried for 10 minutes. Next, after O_2 cleaning, another dummy wafer is loaded as holder of the 1 cm x 1 cm sample, and the SF6 is carried for 1 m and 30 s. Notice that, during etching, the SF6 removes part of the resist.

5. Silicon etching for pillar formation on the 300 nm SOI

In order to determine the most effective crystal orientation and in order to have highest pillars, a second SOI substrate of 300 nm thickness has been processed with 110 orientation. The same design has been adopted in order to compare the two crystal orientations. All the steps described in the Sections 2-4 have been applied to the 300 nm SOI.

About the specific details of this substrate, when loaded in the Elionix ELS-7500 we used again the laser displacement meter Iwatsu ST-3708E and we determined that thickness was 373.57 um for this substrate. The etching rate was calculated of 2 minutes.

Because after imaging we discovered that the etching rate was insufficient, the fabrication process has been repeated again from scratch on a brand new SOI material. The process is identical , except that RIE was carried for 2:40 minutes, giving the desired result of 300 nm pillars.

6. Imaging of the pillars

220 nm Si 100 pillars (Batch A1)

Imaging has been performed by using a scanning electron microscope Hitachi S-4200 SE. The sample is loaded in the prechamber and air is extracted before inserting in the chamber. Current of the beam is set to 500 pA. Before resist removal, the SEM image reveals that both 40 uC and 80 uC expositions formed the pillars with the exception of 80 nm features at the smallest rectangles. The 40 uC resist , from the top view, shows smaller size than nomjnal. For instance the 220 nm x 220 nm is measured below 200 nm per side. The 220nm x 440 nm pillar is smaller but it is achieved by 250nm x 500 nm.

On the contrary, 80 uC exposition shows that 220 nm x 220 nm corresponds to the nominal size. The birds eye view shows that from the vertical aspect the pillar has some lateral tilt and total etching agrees with around 210-220 nm.

After removal of the resist, the images are taken again (see **Figure 6.1.1-2**)

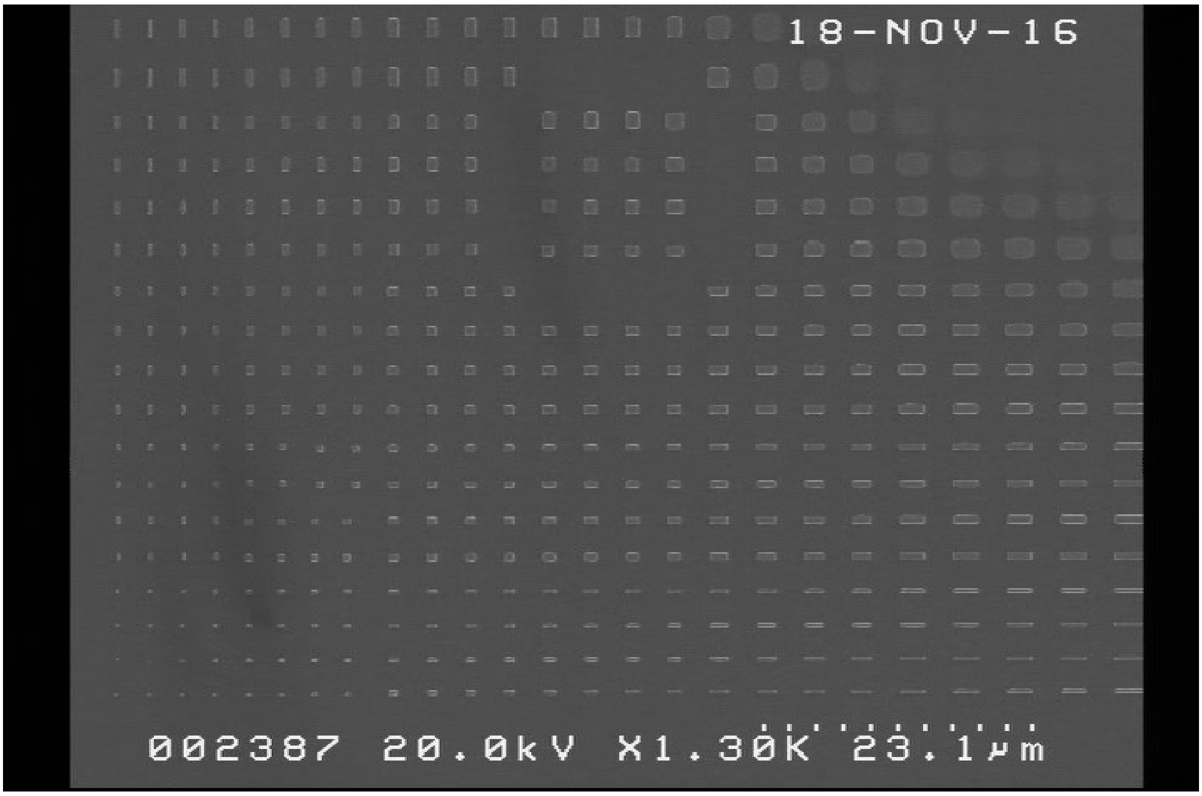


Figure 6.1.1. SEM image of the 220 SOI 100 silicon pillars field for exposition of 160 uC after removal of resist.

.

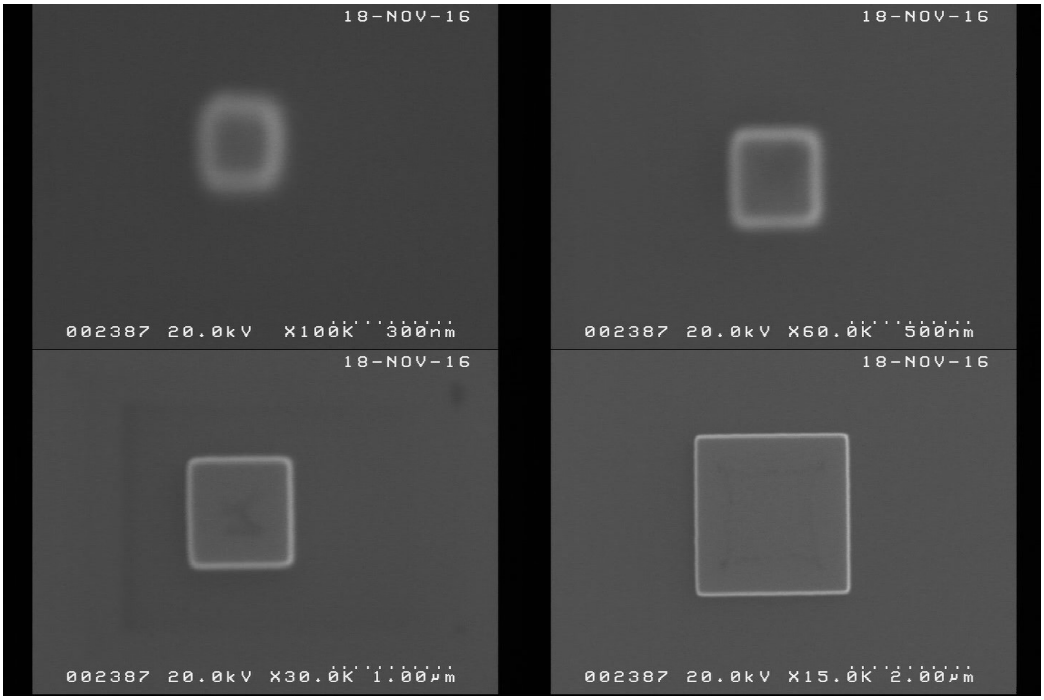


Figure 6.1.2 . SEM image of the 220 SOI 100 silicon pillars at 80 uC exposition after removal of resist: Left-Up: 220 nm x 220 nm. Right-Up: 440 nm x 440 nm, Left-Down: 880 nm x 880 nm. Roght-Down: 2.5 um x 2.5 um.

300 nm Si 110 pillars (Batch A2)

The 300 nm Si 110 pillars are inserted into Hitachi S-4200SE. The pillars are observed by bird eye view. The 880nm and the 440nm square pillars look well formed and they fulfill the nominal size at 80 uC. The largest pillars appear not well formed because of residual material between pillars. By moving to the 40 uC exposition, it appears that all the devices are well formed, both 220 nm square and 2500 nm square. The view is moved to top view. The images look similar as those of 220 nm pillars on 100 SOI. (see **Figure 6.2.1-2**)

The removal of the resist has been performed by O_2 plasma asher for 10 minutes

The 300 nm SOI pillars have been observed again by S-4200 Hitachi SEM to check the complete removal of top resist and the quality of the etching. This substrate is based on 110 silicon which reveals different etching rate. As we performed 2 m, and the height of pillars is of about 250nm, we determine the new sample etching time to be added of 24 seconds, which we round to 30 s.

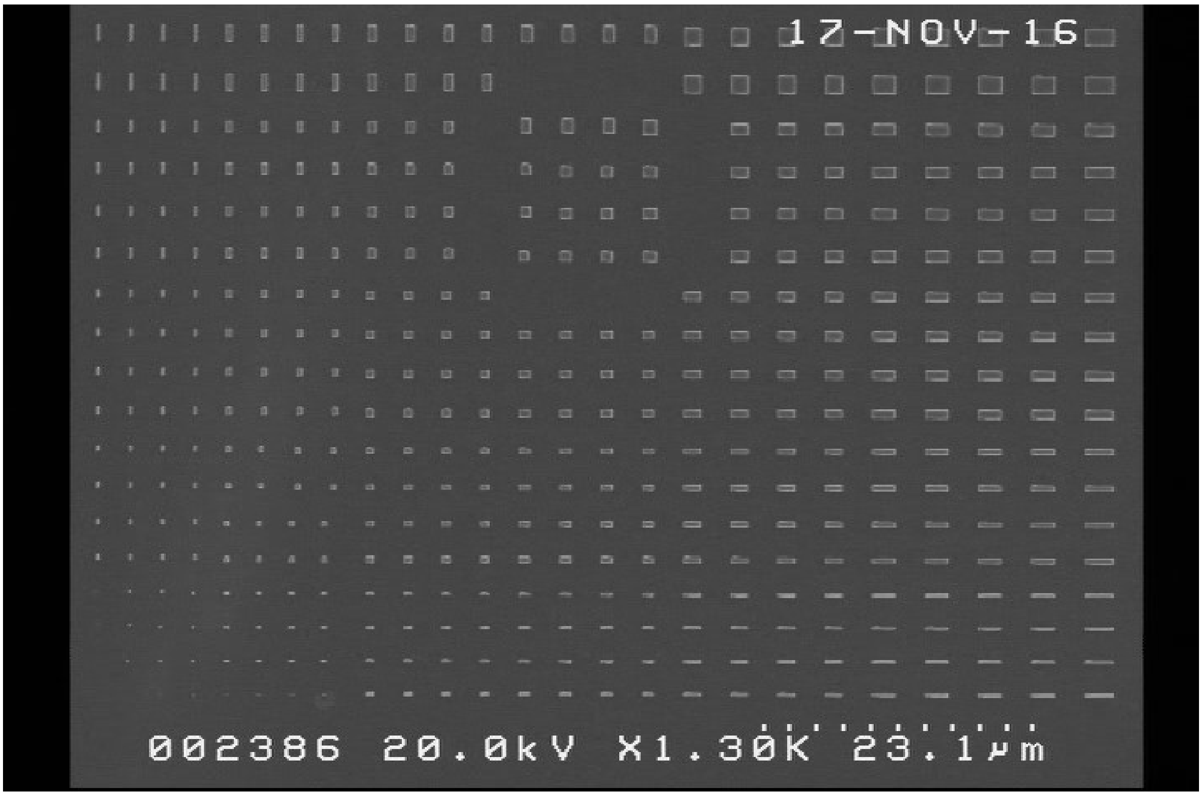


Figure 6.2.1 . SEM image of the 300 SOI 110 silicon pillar field at 40 uC exposition.

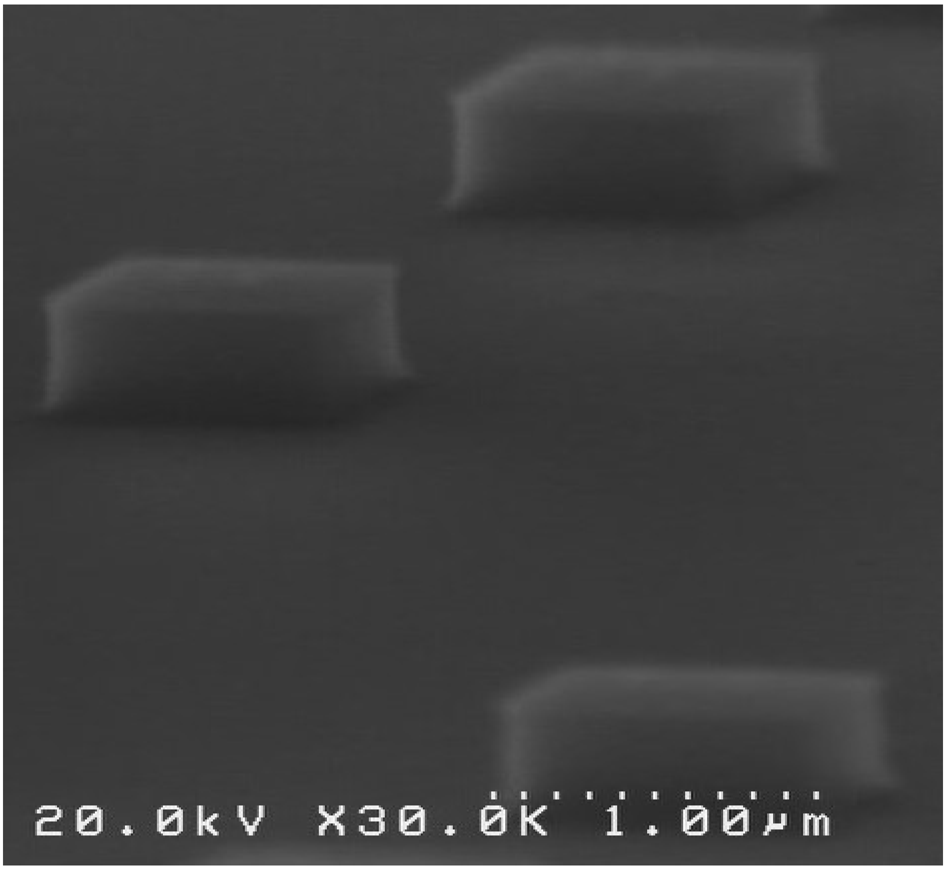


Figure 6.1.2 . SEM image of the 300 nm SOI 110 silicon pillar 880 nm x 880 nm at 80 uC exposition after removal of resist.

7. Reference devices for electroluminescence and photoluminescence

Batch B (Transistors) In order to complement pillars with additional information we prepared reference silicon devices for electrical confirmation of the formation of -140 meV defects. We determined a number of aspect ratios, including 5 um wide and 2 um wide devices, with length of 500 nm and 200 nm respectively. We adopted also a L=500 nm and W=100 nm and two narrow devices of 40 nm and 100 nm respectively, with lenght of 200 nm and 100 nm respectively.

The chip is selected for preliminary characterization by IV characteristics by Keithley 4200 Semiconductor Characterization System. The software provides and automated IV curve by sweeping the gate voltage between -6 and 6 V, at a drain voltage typical of 100 mV.

In addition we characterized a test device not planned for ErO doping (marked as E4).

Batch C (Substrates) As additional reference, we prepared three substrates for photoluminescence not used for pillars. In order to easily identify implanted regions at a later stage during optical characterization, 10 square pads of 100 um x 100 um were prepared by electron beam lithography on each substrate, where implantation is performed.

8. Oxigen implantation process

Oxigen implantation of substrates has been performed first. Pillars (**Batch A1- A2**) have been implanted by oxygen implanter ULVAC UP-250. The dose is fixed to 1.0 10^{14} cm^{-2} implanted entirely over the substrate, for 50 minutes to achieve the objective dose. Mask with apertures has been used only for implanting the Erbium.

Batch B has been implanted by oxygen at a dose of 2.5 10^{13} cm^{-2}over the transistor.
Batch C has been implanted by oxygen at a dose of 2.5 10^{13} cm^{-2}, 1.0 10^{14} cm^{-2}, and 4.0 10^{14} cm^{-2} respectively.

9. Er implantation process

Mounting the Er ion source requires 1 day vacuum pumping. The ion dose was previously characterized and normalized so, according to available flux, the exposition time was calibrated. The samples are loaded into a vacuum chamber and the vacuum achieves 10^{-5} Pa. Temperature of ion source is set to about 600 C. The filament current was set to 0.61 A. The ion source consists of Er with Au and Si. To start ion emission, the ion source is heated by current up to 3.5 in steps of 0.2. The emission of the filament goes around 10-15 uA nominal value. Typical range for the extractor is aroun -4/-5 kV. We set 3.6 A and -5.4 kV to have -13.6 uA emission current. We move to better conditions as -4.96 kV which is sufficient to extract at -10.2 uA. Next the Ion Gun Controller is turned on with ACC=10 kV. Alignment of the top of the ion source is done manually and next CL and OL are controlled in 3-6 kV range The current is finally set to 1.4 nA and fine adjusted by CL and OL coarse plus fine control.

Pillars (**Batch A1- A2**) have been implanted after depositing a resist mask with apertures. The apertures were 150 μm x 150 μm so all the pillars were implanted simultaneously. The Erbium beam was larger than the aperture. Resist was ZEP520A: thickness 350 nm spin curve 4000rpm. After Erbium implantation the resist was removed by oxygen plasma. Cleaning was performed by using acid piranha solution.

Thermal annealing was done at 900 C for 30 minutes and next substrate in HF to remove oxide film (which is of about 1.5 nm).

Batch B (transistors) has been implanted at lower doses compatible with previous tests therefore 3 10^{13} and 1 10^{14} (implanted in this case before the oxigen). Implantation energy was 45 keV. The recipe of 900 annealing on new transistors on SOI has been performed. See STM Report of 2015.

Batch C (substrates) As additional reference, we implanted three substrates at 2.5 10^{13} cm^{-2}, 1 10^{14} and 4 10^{14} respectively, by 15 minutes, 1h and 4 h respectively. The current was set as 600 uA. For Implantation energy was 25 keV. For substrates, we put a carbon tape to see secondary electrons, as we needed some marks.

10. Conclusions

To summarize, the following silicon nanostructures have been assessed:

Main devices:

Batch A1 (pillars) 220 nm SOI 100-silicon:
Er dose : 1 10^{14} cm^{-2}
Oxigen dose: 1 10^{14} cm^{-2}
Batch A2 (pillars) 300 nm SOI 110-silicon:
Er dose: 1 10^{14} cm^{-2}
Oxigen dose: 1 10^{14} cm^{-2}

Reference devices:

Batch B (transistors)
Er dose: 3 10^{13} and 1 10^{14} cm^{-2}
Oxigen dose: 2.5 10^{13} cm^{-2}
Batch C (substrates)
Er dose: 2.5 10^{13}, 1 10^{14} and 4 10^{14} cm^{-2}
Oxigen dose: 2.5 10^{13}, 1.0 10^{14}, and 4.0 10^{14} cm^{-2}

The **implantation of Er in silicon is likely to become a viable source of single photon** and full characterization will be carried in CNR in Milan according to the plan.