Collaborative project*

Project acronym: SNM

Project full title: "**Single Nanometer Manufacturing for beyond CMOS devices**"

Grant agreement no: 318804

Deliverable: D8.7 ("Silicon nanowire based mechanical resonators as mass sensors")

Name of the coordinating person: Prof. Dr. Ivo W. Rangelow, Email: ivo.rangelow@tu-ilmenau.de

List of participants:

Participant no.	Participant organisation name	Part. short name	Activity Type	Country
7	Spanish National Research Council	**CSIC**	RES	Spain

<table>
<tr><td colspan="6">SNM
Work Package 8
Deliverable: D8.7“Silicon nanowire based mechanical resonator as mass sensors”</td></tr>
<tr><td>Lead beneficiary number</td><td>7</td><td>Nature</td><td>R</td><td>Dissemination level</td><td>PU</td></tr>
<tr><td>Estimated Person-months</td><td colspan="5">8</td></tr>
<tr><td>Person-months by partner for the Deliverable</td><td colspan="5"><table><tr><td>CSIC</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr><tr><td>6.00</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr></table></td></tr>
<tr><td>Estimated Delivery Date</td><td colspan="2"></td><td colspan="2">Delivery Date</td><td>06/02/2017</td></tr>
<tr><td>Author</td><td colspan="5">• Jordi Llobet and Francesc Perez-Murano (CSIC Barcelona).</td></tr>
<tr><td>Reviewed by:</td><td colspan="5">• WP8 leader: Zahid Durrani
• WPG3 Leader: Zahid Durrani
• Coordinator: Ivo W. Rangelow</td></tr>
<tr><td rowspan="4">Criteria and Achieved Results</td><td colspan="3">Criteria</td><td colspan="2">Achieved result</td></tr>
<tr><td colspan="3">Fabrication of sub-10 nm suspended silicon nanowires</td><td colspan="2">Suspended silicon nanowires with critical dimension of 6 nm fabricated by o-SPL and FIB lithography.</td></tr>
<tr><td colspan="3">Large array of suspended silicon nanowires</td><td colspan="2">Array of 3480 silicon nanowire fabricated. Metrology analysis performed.</td></tr>
<tr><td colspan="3">Analysis of the performance of suspended silicon nanowires as mass sensors</td><td colspan="2">Mass sensor functionality of suspended silicon nanowire proved. Achieved mass sensitivity: 20 zg/Hz</td></tr>
</table>

Description of the Deliverable

- **Introduction**

This deliverable describes the results on the fabrication and functional performance of suspended silicon nanowires achieved during the realization of task T-8-6 *"Sub-5 nm silicon nanowire field effect transistors and mechanical resonators"*.

The fabrication has been based on FIB lithography, as a continuation of the activity from task T-8-5 "*Preliminary fabrication of devices based on silicon nanowires*". As it has been shown in D8.3, in the reports of that task and in several journal publications, this fabrication method produced suspended silicon nanowires of very small dimensions and exceptional electromechanical properties. In task T-8-6, we focused on reducing the dimensions of the nanowires and proved their manufacturability by producing an array of more than 3000 suspended silicon nanowires. The smallest devices were fabricated by a novel combination of FIB lithography and o-SPL.

With respect to the functional characterization, small quantities of mass were deposited on top of silicon nanowires by electron beam induced deposition. The change of resonance frequency of the nanowires was monitored by electrical read-out, taking advantage of the piezoresistive detection method developed within this project. The resulting value of the mass sensitivity (20 zg/Hz) indicates the appropriateness of the use of these silicon nanowires as mass sensors.

In the following, we depict the main results achieved in the fabrication and functional characterization of the suspended silicon nanowires.

- **Large array of suspended silicon nanowires.**

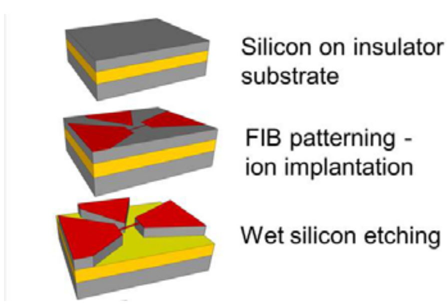

Figure 1. Process for the fabrication of the large array of silicon nanowires.

A large and dense array of suspended silicon nanowires using the process depicted in figure 1 was successfully fabricated. The result is shown in figure 2. It consists on an array of more than 3000 suspended silicon nanowires.

As it is seen in figure 1, the process is simple and flexible, which facilitates its manufacturability. The aim of fabricating a large array is to proof that the process is reliable and reproducible. Because of this, a metrological analysis has been performed by VSL. No defects are present in the arrays (missing, broken or not suspended silicon nanowires). As a result of the analysis, we determined which are the limitations of the method in terms of dispersion of the dimensions.

.

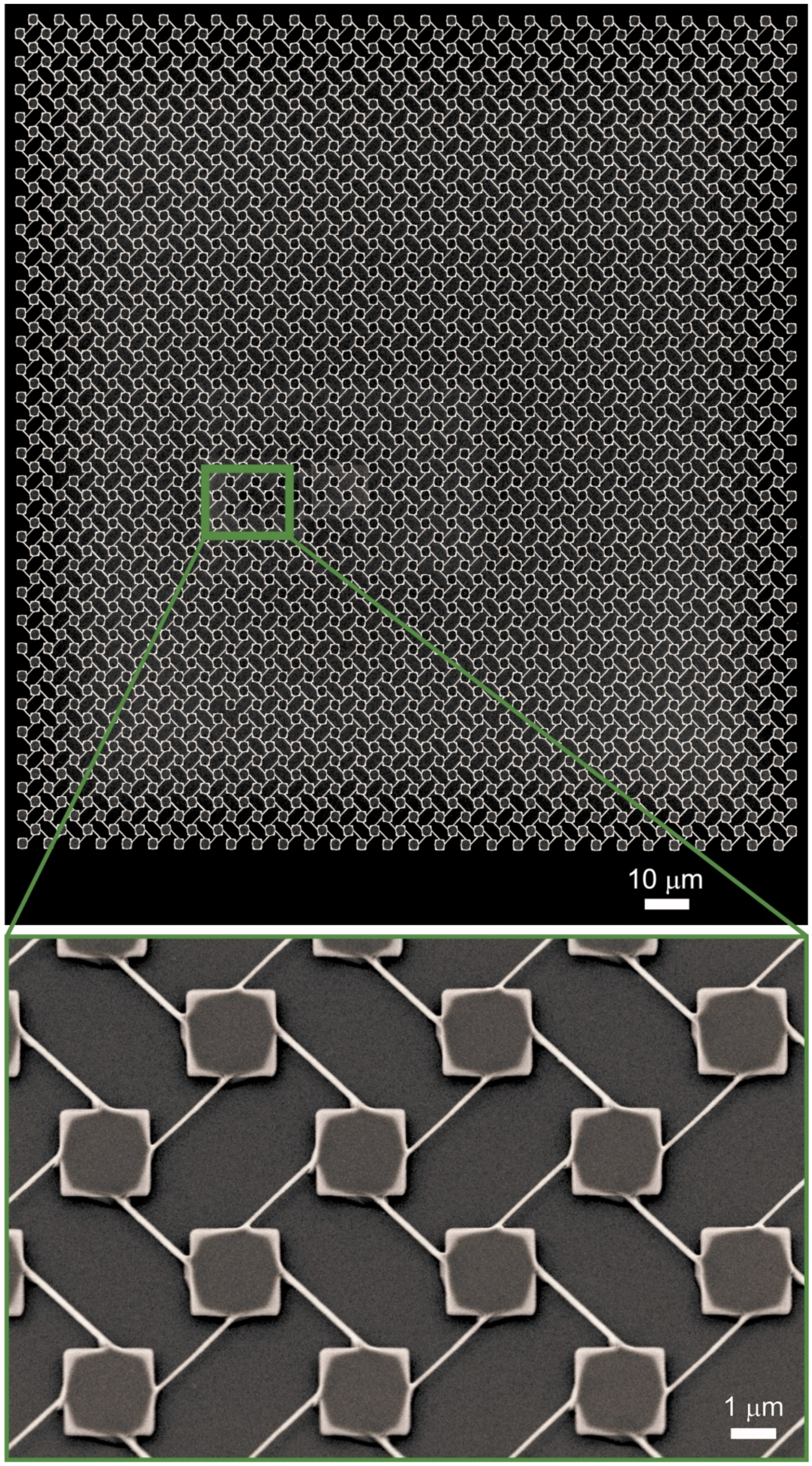

Figure 2. Large array of suspended silicon nanowires fabricated by FIB lithography. The array consists on 3480 suspended nanowires. The top SEM image shows the whole array and the bottom SEM image a zoom in the area marked in green,

A method was developed to automatically determine the width and length of each nanowire in the array. For this, a large set of SEM images at low magnification were taken. The analysis was done for the horizontal and vertical wires separately. For both types more than 1,500 wires were analysed. The procedure based on edge detection. The threshold for the edge detection was set to 75 % of the average intensity in the central part of the structure. The length was calculated from the central profile of the nanowire with the same threshold. Afterwards, the width was calculated for each individual image line along 50 % of the established length for each wire. The values of length and width are calculated as the average, median and mode for each set of widths. In the next figure, we report the analysis for the horizontal and vertical nanowires (the image was taken so that the silicon nanowires appear ether vertical or horizontal, in order to facilitate the analysis).

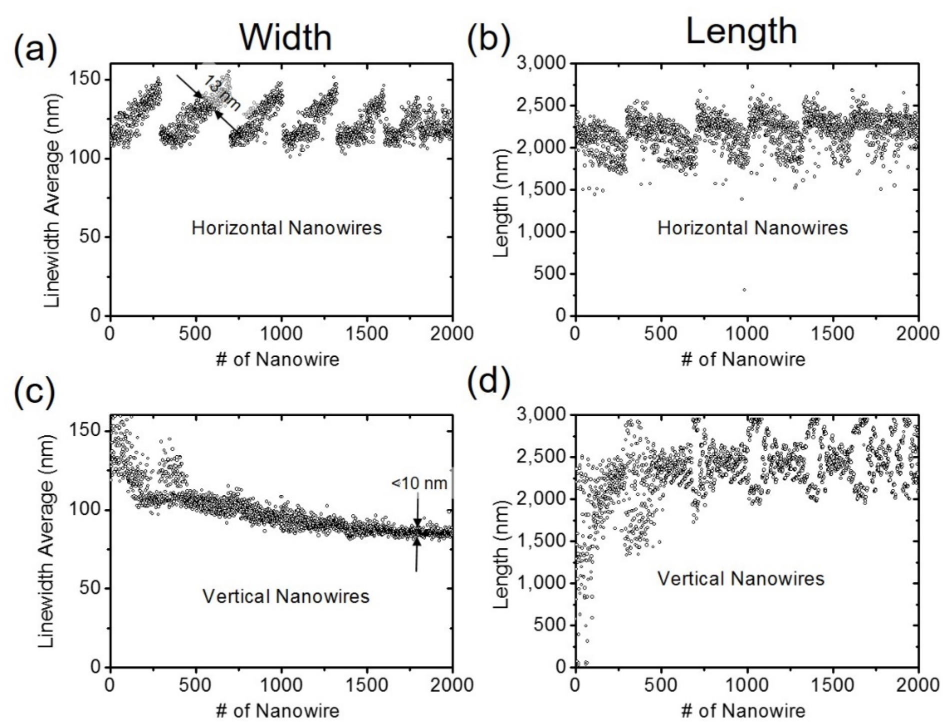

Figure 3. Analysis of the dimensions of the suspended silicon nanowires in the array of figure 2. The analysis has been performed from SEM images in which the suspended silicon nanowires were either vertical or horizontal. Both orientations have been analysed separately,

From the data depicted in figure 3, three different sources of dispersion can be observed. The data is ordered according to the temporal sequence in which the SEM image were taken.

a) The variation in length and width of the vertical nanowires is attributed to an effect of the SEM imaging (drift) due to unbalanced thermal variations. The periodic variations for the length and width of the horizontal nanowires are also attributed to some artefact of the SEM images.

b) The large dispersion in the length for some vertical nanowires is attributed to an error in the design file. They correspond to the nanowires located at the edges of the array.

c) Finally, there is an intrinsic dispersion of the data (see for example the values of the average width for the vertical nanowire after structure number 500) which is attributed to the limitation of the fabrication process itself.

Effects a) and b) can be ruled out in the analysis of the intrinsic dispersion in the dimensions due to the fabrication process, since they can be solved by acquiring proper SEM images and correcting the array design. In consequence, we can infer that the dispersion in width attributed to the fabrication process is below 10 nm.

As an example, in figure 4 it is shown the distribution of width for the Vertical nanowires (figure 3.a) where only the last 500 nanowires have been considered, in order to rule out the drift effect mentioned above. It confirms the statement that a width dispersion below 10 nm is achieved.

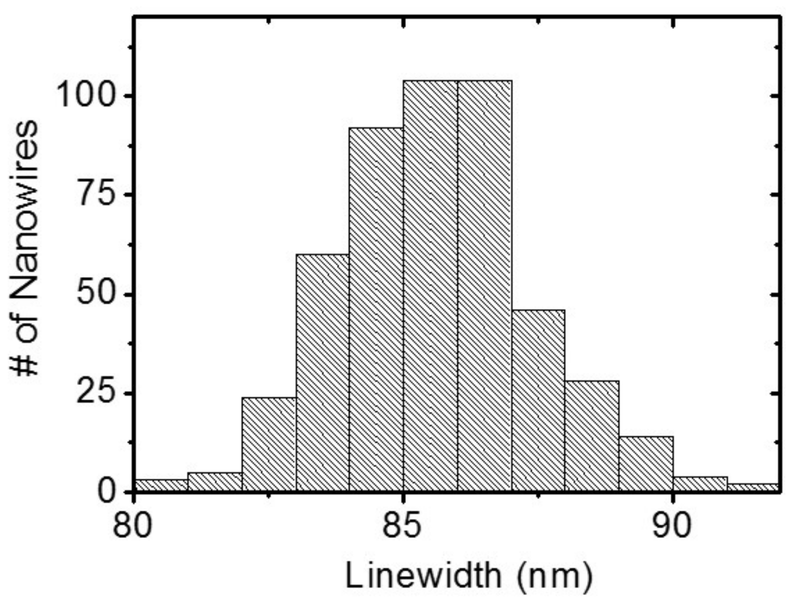

Figure 4. Analysis of the dimensions of the linewidth of suspended silicon nanowires, corresponding to the last 500 suspended nanowires in figure 3.a

Next table summarizes the main results of the analysis:

	Vertical	**Horizontal**
Number of NWs	1,740	1,740
Density of NWs	> 1 SiNW / mm^2	
Average length	2300 nm	2400 nm
Length dispersion	100 nm	100 nm
Average width	86 nm	130 nm
Width dispersion	<10 nm	15 nm

Table 1. Data extracted from the analysis of the dimensions of the array of suspended silicon nanowires shown in figure 2

- **Sub-10 nm suspended silicon nanowires**

  To fabricate the smallest silicon nanowires, a process based on the combination of o-SPL and FIB implantation was developed. It is a novel approach with the advantage of simplifying the overall process and the potential for high density integration. In a first step the contacting pads and the clamping sites are defined by FIB implantation onto the top silicon layer. Then, o-SPL defines the nanowires. RIE is used to etch silicon everywhere except in the areas patterned by FIB and o-SPL. In the final step the buried SiO_2 is etched by SiO-etch in order to release the fabricated silicon nanowires. It is important to note that this process is completely resistless. Next figure shows the overall process, which is a joint development between CSIC-Madrid and CSIC-Barcelona.

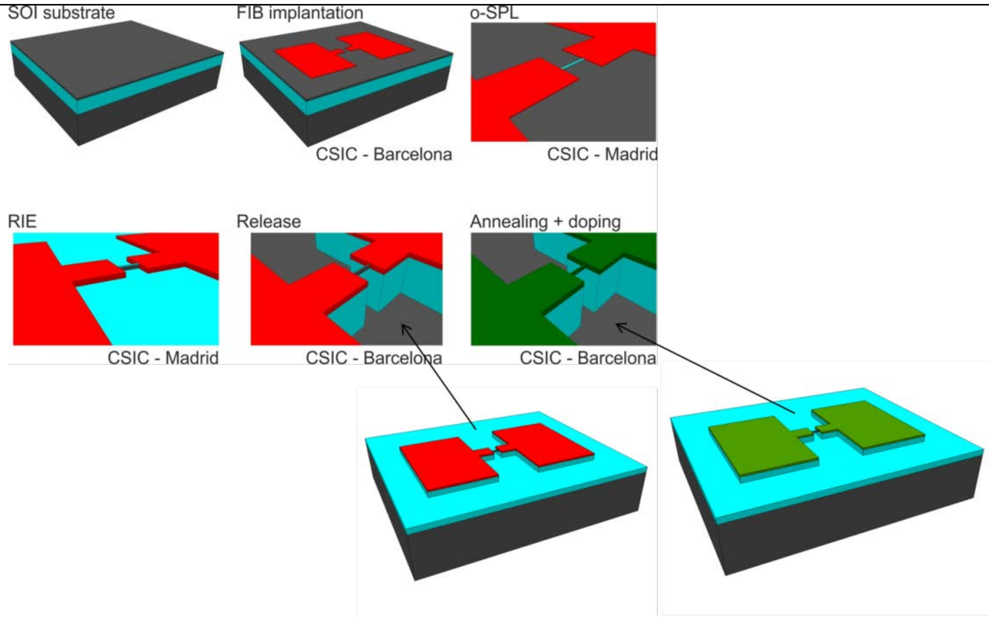

Figure 5. Process definition for the fabrication of suspended silicon nanowires by the combination of FIB implantation and o-SPL. It involves processes to take place at CSIC-Madrid and CSIC-Barcelona

During year 2, a lot of effort has been developed to achieve the compatibility between FIB implantation and o-SPL. The most critical step is that the reactive ion etching process to transfer the pattern into silicon has to be adequate simultaneously for the areas defined by o-SPL and for the areas defined by FIB implantation. Next figure shows that sub-10 nm resolution is achievable. Electrical characterization of these devices is now being addressed.

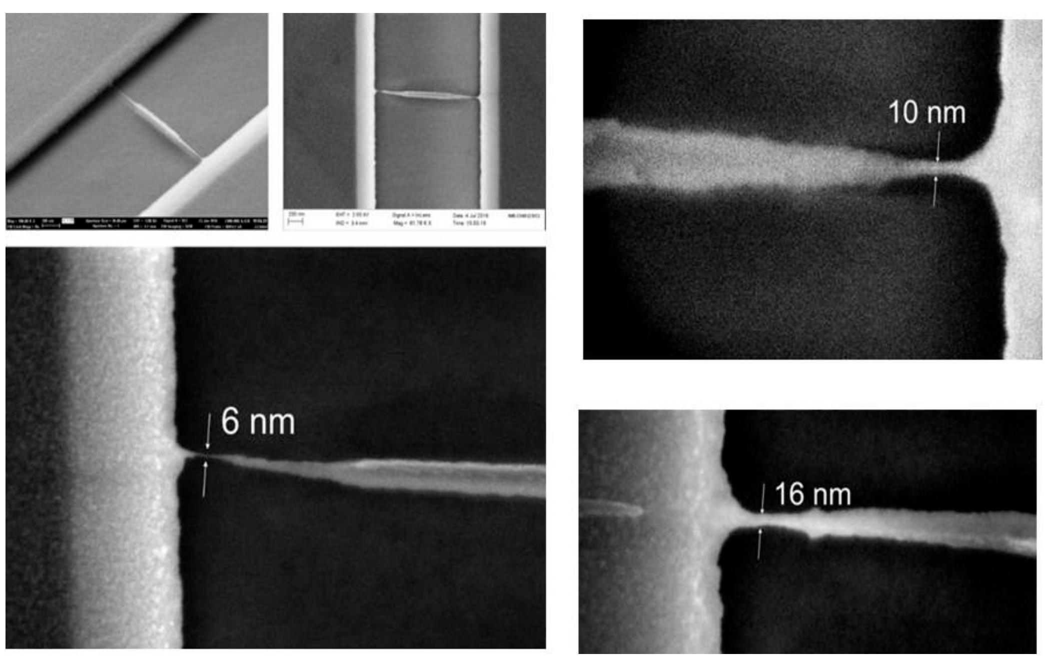

Figure 6. Examples of silicon nanowires fabricated by the combination of o-SPL and FIB, showing that sub-10 nm resolution is achievable.

The fact that the suspended nanowire section appears smaller at the edges is related

with the definition of the nanowires during the o-SPL process. We have observed that the oxidation rate depends on the gallium exposure dose: in the border of the clamps, the exposure decreases gradually, causing an increase of the oxidation rate.
FIB lithography alone is also capable of fabricating sub 10 nm Si Nws, as shown in figure 7 but the rate of success for such critical dimension is quite low, reason why integration with o-SPL has been preferred.

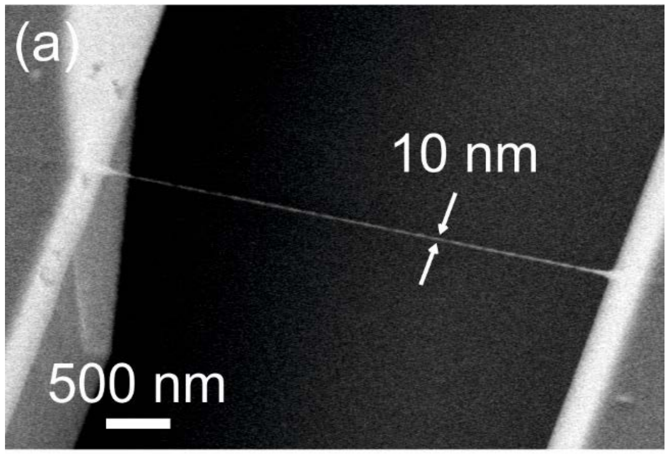

Figure 7. Suspended SiNw fabricated by FIB lithography, the measured with is approximately 10 nm

- **Evaluation of mass sensitivity of suspended silicon nanowires as nano-mechanical resonators**

For the determination of the mass sensitivity of the suspended silicon nanowires fabricated by FIB lithography, the fabricated devices consist on mechanically coupled silicon nanowires. The reason is that, as presented in previous years, the electromechanical (piezoresistive) transduction is highly dependent on the stress and deformation of the structure. By using a system of coupled nanomechanical resonators, the sensitivity is enhanced and some parasitic effects can be ruled out.

Small amounts of mass have been deposited by focused electron beam induced deposition (FEBID). This allows to deposit the mass at specific locations of the nanomechanical resonator. Due to instrumental limitations related with the system used for FEBID, the minimum amount of mass to be deposited is limited.

Figure 7 shows an example of FEBID mass deposition on a nanomechanical resonator based on three silicon nanobeams. The gas precursor for the FEBID process is trimethyl (methylcyclopentadienyl) platinum. It is known that the density of the resulting material (basically platinum with carbon) is close to 12500 Kg/m^3. From the SEM images, we estimate a volume of $8{\cdot}10^{-21}$ m^3. In consequence, the mass corresponding to a single deposit is approximately of 100 fg. Although this value of mass is only an estimation obtained from geometrical considerations, it is enough for a first determination of the mass sensitivity.

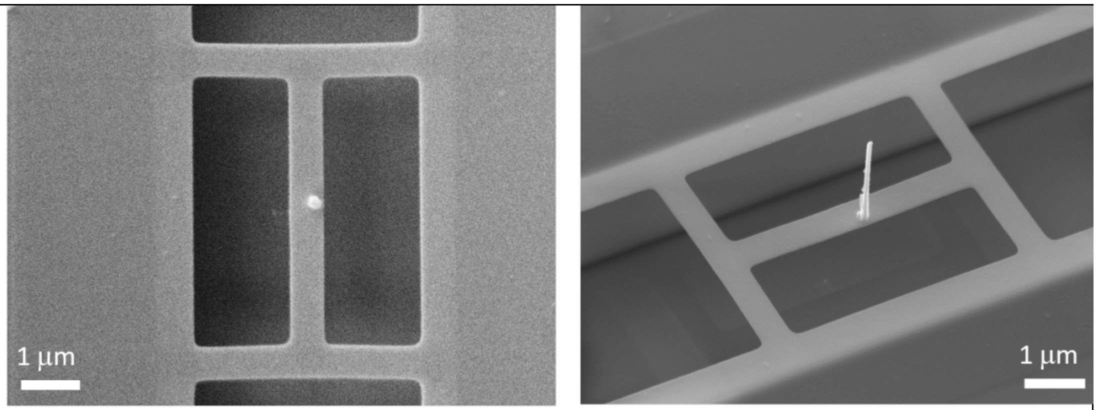

Figure 8. Example of mass deposition by FEBID on top of a nanomechanical resonator based on coupled silicon nanobeams fabricated by FIB implantation

We analyse the mass sensitivity of the devices. The coupled devices present several resonant modes, which resonance frequency changes upon the deposition of mass. The identification of the modes requires a careful investigation by finite element modelling. Next figure shows two examples of experiments with multiple mass deposition.

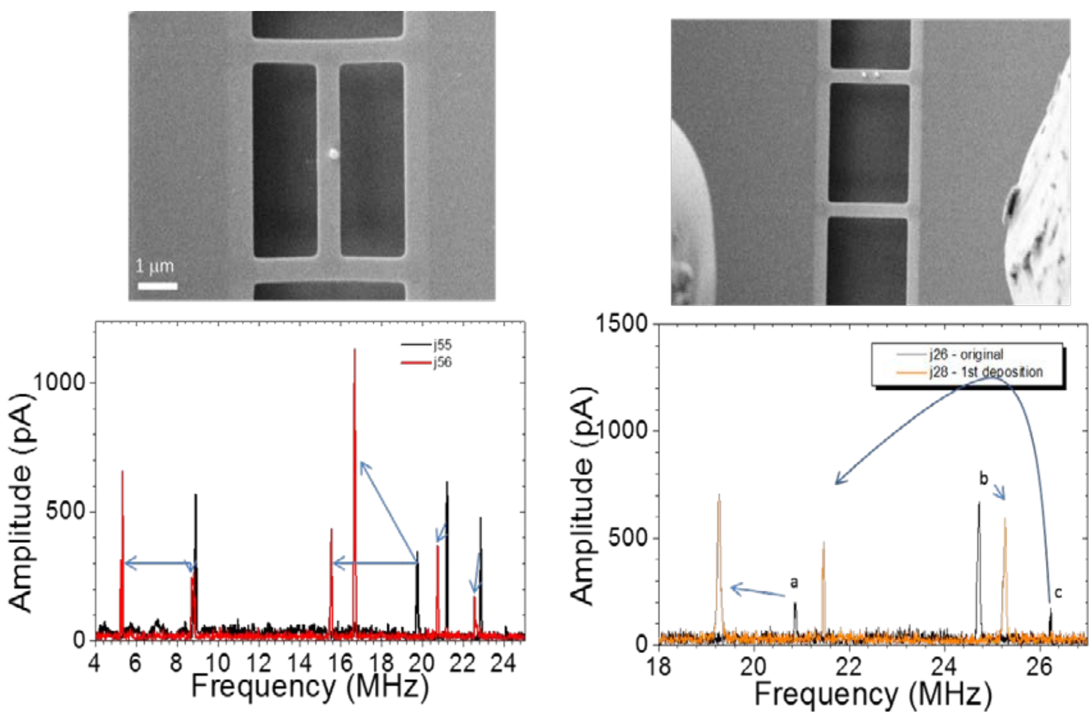

Figure 9. Example of mass deposition by FEBID on top of two nanomechanical resonators based on coupled silicon nanobeams fabricated by FIB implantation.

If we consider the change of resonance frequency for the device of the right column in figure 7, we estimate a change of frequency of 5 MHz. It implies a mass sensitivity of these devices of around 0.05 Hz/zg (best result achieved for piezoresistive silicon NEMS is 0.7

	Hz/zg, Mo Li et al, Nat. Nanotech. 2, 114 (2007)). This value should be achievable by a further reduction of the dimensions of the silicon nanowires esp. due to the combination of o-SPL and FIB.
Explanation of Differences between Estimation and Realisation	The fabrication of suspended silicon nanowires to be used as nanomechanical resonators for mass sensing has been mainly performed by FIB lithography. The reason is that this method has resulted to be extremely powerful in terms of flexibility and simplicity, and the resulting nanowire present exceptional nanoelectromechanical properties that facilitate their electrical read-out. Combination of FIB and o-SPL has been used to show that sub-10 nm suspended silicon nanowires can be fabricated. It has been proven that the resulting silicon nanowires can be used as high sensitivity mass sensors. The value of the mass sensitivity is close to the best reported values for piezoresistive NEMS on silicon, and it can be improved by further reduction of the dimensions of the silicon nanowires.
Metrology comments	A metrological study has been carried out to estimate the dispersion in the dimensions of the suspended silicon nanowires. A large array of suspended silicon nanowires has been fabricated for this purpose. The results are reported in this deliverable.