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Silicon nanowires: fabrication and quantitative dimensional characterisation by AFM

EMPIR 19ENG05 project NanoWires

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ABSTRACT

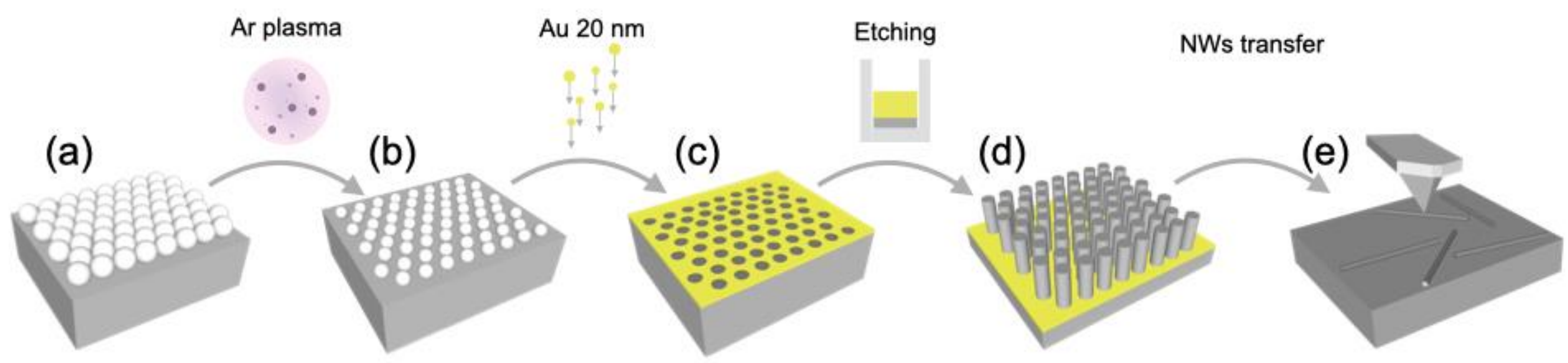
Silicon nanowires (NWs) are fabricated by means of nanosphere lithography and metal-assisted chemical etching (MACE) to obtain high aspect ratio nanostructures.

This study reports an interlaboratory comparison on the measurements of dimensional parameters of nanowires by AFMs among some European National Metrology Institutes, since robust methods to measure nanowires is lacking.

The measurands investigated are NW diameter (measured as top-height) and sidewall roughness (Ra, Rq, Rz, Rsk, Rku parameters), extracted from the top profile along the nanowire length. In fact, both are key parameters to understand if the fabrication process was carried out in a correct way. Moreover, the knowledge of these parameters is essential to achieve the expected functional characteristic of energy harvesting systems.

In this work the reproducibility due to different instruments of exactly the same set of nanowires are studied. Measurements show a good agreement, with a combined standard uncertainty of the diameter less than 3%, and well within 5% for Ra and Rq values. Concerning the roughness, no standard or guide exists for assessing the uncertainty associated with it, so we propose and investigate a new methodology based on Monte-Carlo approach.

FABRICATION



(a) Deposition of polystyrene nanospheres NSs (diameter 200 nm) on the silicon wafer by spin coating at different rotational speed.
(b) Reduction of NSs diameter to 100 nm by argon plasma.
(c) Deposition of 20 nm of Au and lift-off of the NSs to obtain the pattern of circular voids on the gold layer.
(d) **Metal-Assisted Chemical Etching** to obtain the porous silicon nanowires with very high aspect ratio (height > 15 µm), not achievable by dry etching methods.
(e) Transfer of the nanowires NWs on a solid silicon substrate with markers, where they lay horizontally and their sidewalls can be probed by atomic force microscopy.

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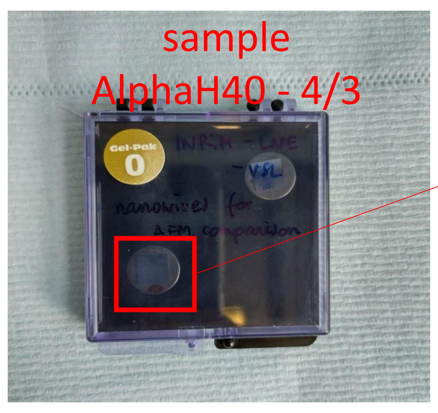
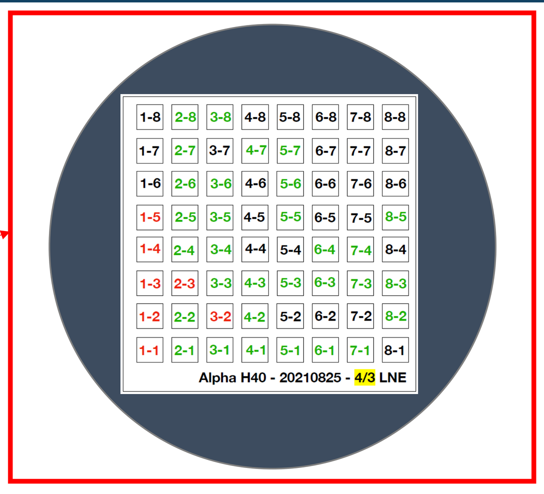
AFM CHARACTERISATION

(1)

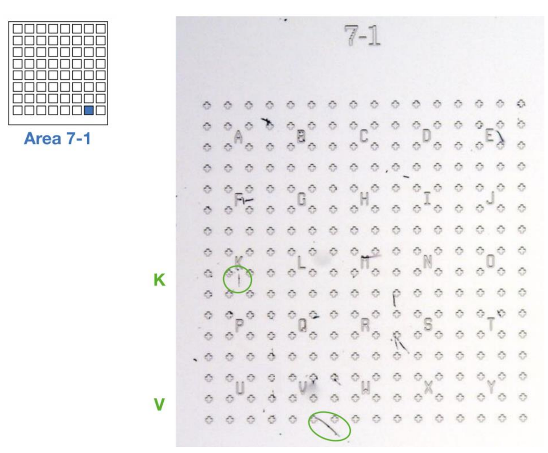


The silicon chip with NWs is installed with carbon tape onto a metallic disk

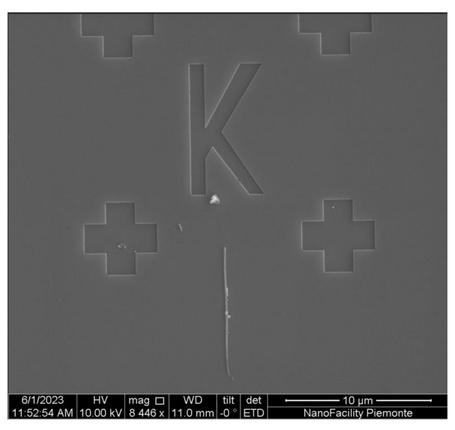


Scheme of the silicon chip with different areas marked with numbers, letters and crosses. The green areas has isolated NWs, while the red areas present NWs agglomerates.

(2) Sample inspection with **optical profilometer**

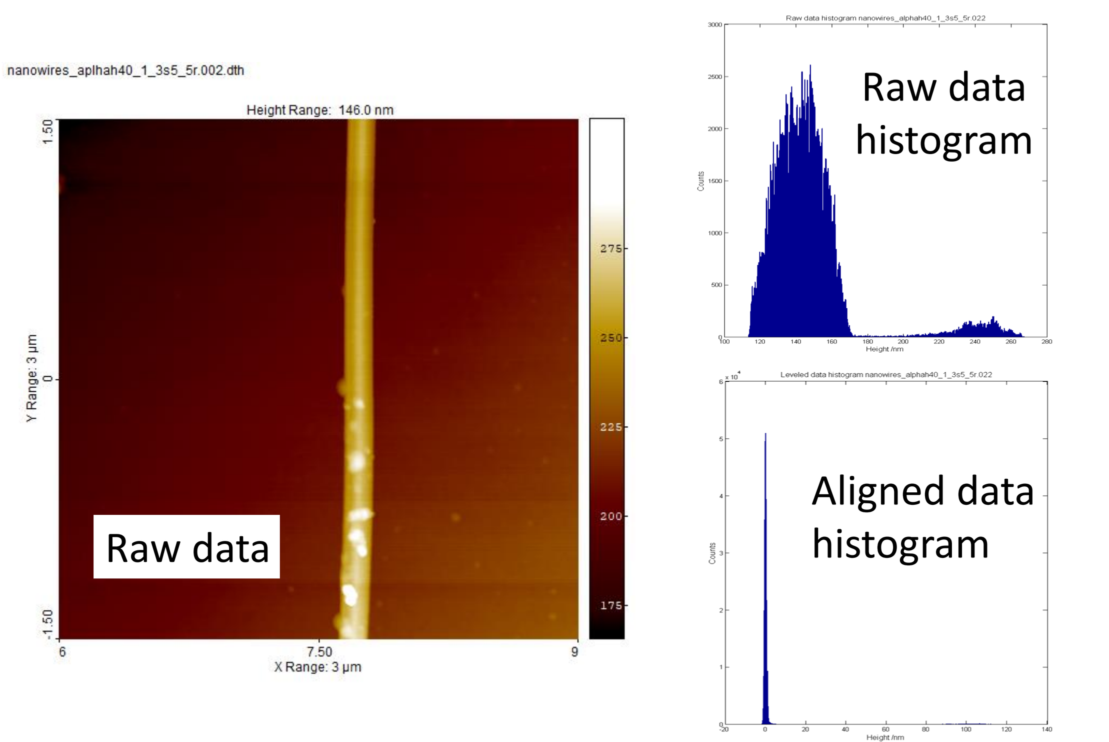


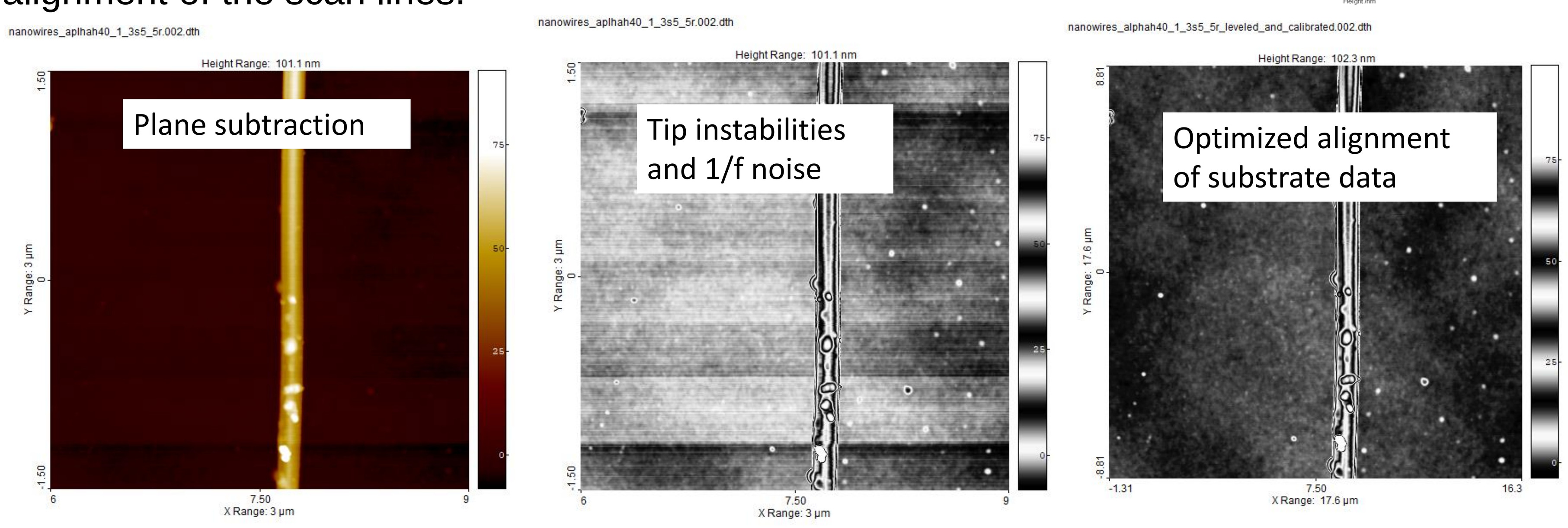
(3) Inspection of the selected areas with **SEM**



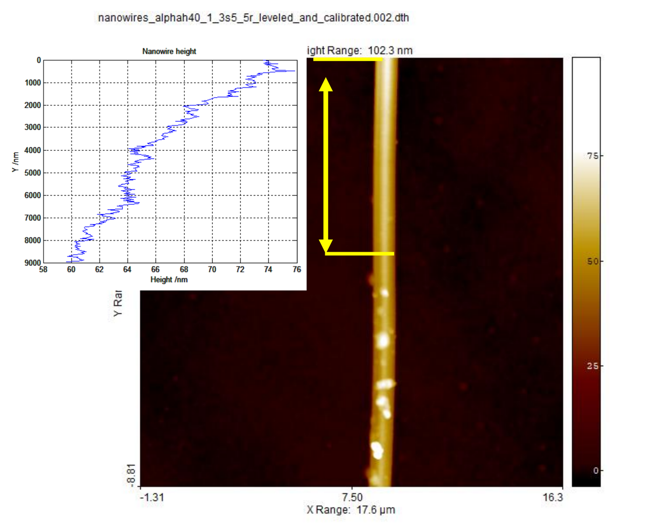
(4) Measurements of the selected NW by means of **AFM**.

(5) Pre-processing of the image. Since a simple plane correction is not sufficient to correct tip instabilities and 1/f noise, the image is aligned by isolating the data points representing the substrate and then correcting each scan line for tilt and offset of the substrate. This results in a very sharp histogram for the substrate data indicating good alignment of the scan lines.

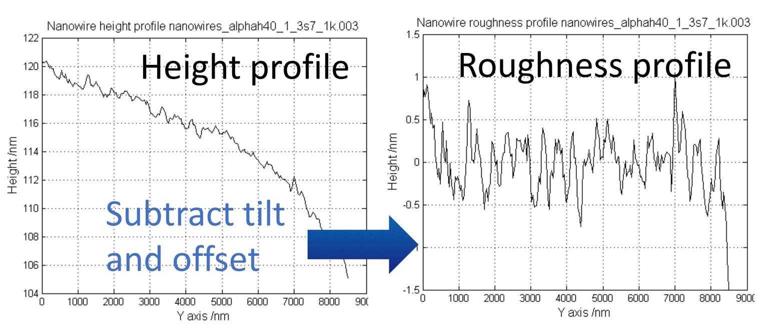




(6) Extraction of the **cross-section height** in zones free from contaminations, in order to calculate the diameter of the wire.



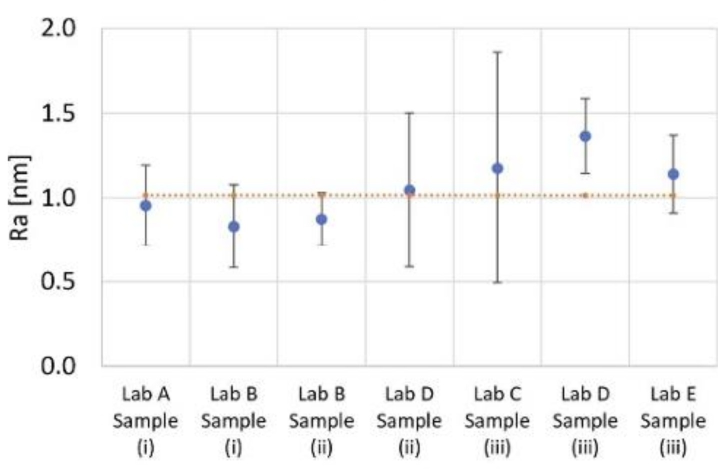
(7) Extraction of the **roughness parameters** from the profile at the top of the NW.

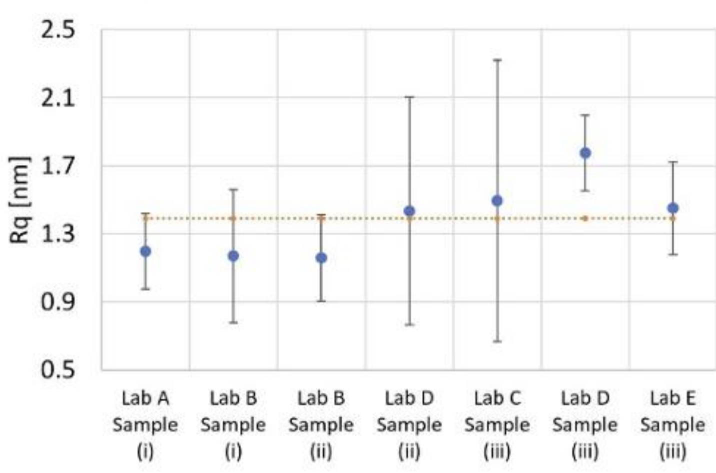


INTERLABORATORY COMPARISON

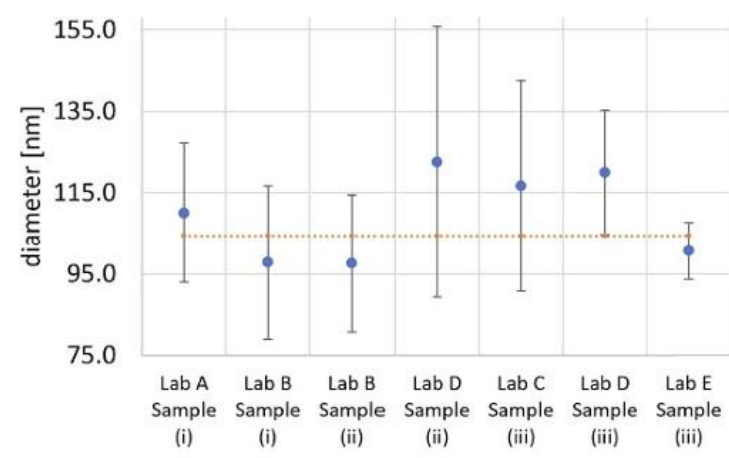
The NWs fabricated by Metal-Assisted Chemical Etching are deposited onto three different chips. These samples are circulated between 5 different laboratories.

The plots reported the results of different roughness parameters from this intercomparison study. The values reported are mean values for each laboratory, and the error bar represent the standard uncertainty of the mean. The orange line represent the weighted mean.





Moreover, the plot on the right reports the NW diameter obtained by extracting the mimimum value of the cross-section top height. This because one can observe variations in heigths values, due to the deposition of the wires onto the substrate.



UNCERTAINTY ESTIMATION

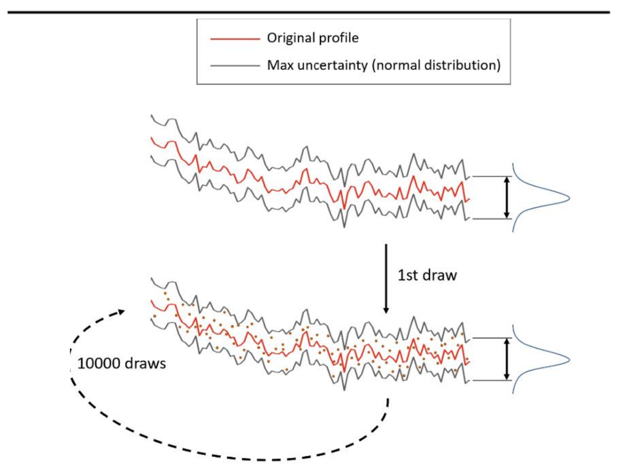
For calculating uncertainty of roughness parameters, we generate 10000 profiles according to the following formula:

$$P_i = P_0 + u_r \cdot P_0 \cdot \mathbf{N_1} + \delta_{noise} \cdot \mathbf{N_2}$$

where P_i is the profile generate at the i-th iteration, P_0 is the original profile, u_r is the relative uncertainty of the Z-calibration coefficient (based on the relative standard uncertainty of the used step height standard), δ_{noise} is the Z-noise of the instrument (noise measurement on a very flat and smooth surface), and $\mathbf{N_1}, \mathbf{N_2} \in \mathbb{R}^{n_p}$ are random vectors of size n_p (number of pixel in the profile) extracted from a normal distribution.

Afterwards, for each iteration we evaluate roughness parameters, and then parse the obtained distributions until 95 % coverage is reached, so these represent the expanded uncertainty for each roughness parameter.

Roughness parameter	Laboratory	Expanded uncertainty $(k = 2)$ U (nm)
Ra	Lab A	0.01
	Lab B	0.09
	Lab C	0.05
	Lab D	0.01
	Lab E	0.03
Rq	Lab A	0.01
	Lab B	0.10
	Lab C	0.07
	Lab D	0.01
	Lab E	0.04



Each participant evaluate the uncertainty of NWs top-height diameter according to $d = C \cdot d_{mean} + \delta_{noise}$, where d is the top-height diameter, d_{mean} is the average of the diameter form experimental data, C is the calibration of the instrument, and δ_{noise} is the instrumental noise.

Laboratory	Expanded uncertainty of the NW diameter $(k = 2)$ U (nm)
Lab A	5.0
Lab B	6.2
Lab C	7.1
Lab D	5.4
Lab E	6.8

CONCLUSIONS

- This study reports an interlaboratory comparison which describes a method for the dimensional analysis of NW. This is important in order to understand the fabrication process, but also to investigate functional behaviour of nanowires in energy harvesting devices.
- A good agreement between roughness and diameter measurements from different laboratories is shown, and a novel approach for uncertainty estimation of roughness parameters is reported.
- Further information can be found here: Luigi Ribotta et al., AFM interlaboratory comparison for nanodimensional metrology on silicon nanowires, 2024, Meas. Sci. Technol., 35, 105014, DOI 10.1088/1361-6501/ad5e9f

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1) INRiM Istituto Nazionale di Ricerca Metrologica 2) PiQuET Piemonte Quantum Enabling Technology 3) QR Lab Micro&NanoLaboratories 4) Laboratoire National de Métrologie et d'Essais LNE 5) VSL National Metrology Institute 6) Politecnico di Torino 7) UGA Université Grenoble Alpes