



## GOOD PRACTICE GUIDE No 2 EMPIR PROJECT 19ENG05 NANOWIRES

# Methodology for NW thermal measurements

Nolwenn Fleurence<sup>1</sup>, Sarah Douri<sup>1</sup>, Petr Klapetek<sup>2</sup>, Teodor Gotszalk<sup>3</sup>, Eleonora Cara<sup>4</sup>, Luca Boarino<sup>4</sup>

1 Laboratoire national de métrologie et d'essais, 29, avenue Roger Hennequin, 78197 Trappes Cedex France

2 Czech Metrology Institute, Okružní 31, 638 00 Brno, Czech Republic

3 Wroclaw University of Technology, ul.Janiszewskiego 11/17, 50-372 Wroclaw, Poland

4 Istituto Nazionale di Ricerca Metrologica, Advanced Materials Metrology and Life Sciences Division and QR Laboratories, Strada delle Cacce 91, 10135 Torino Italia

### TOC

<b>List of Abbreviations</b> .....	<b>2</b>
<b>Introduction</b> .....	<b>3</b>
<b>SThM traceability</b> .....	<b>3</b>
Preliminary recommendations for SThM measurements:.....	3
SThM device:.....	3
Measurement definition:.....	3
Calibration:.....	5
Uncertainty assessment:.....	7
<b>Sample preparation</b> .....	<b>9</b>
<b>Standalone nanowires measurements using standard SThM tools</b> .....	<b>10</b>
<b>Advanced SThM instrumentation for thermoelectric measurements</b> .....	<b>13</b>
<b>Nanopillars measurements</b> .....	<b>13</b>
<b>References</b> .....	<b>15</b>



## LIST OF ABBREVIATIONS

<b>3D</b>	Three Dimensional
<b>AFM</b>	Atomic Force Microscopy
<b>FDM</b>	Finite Difference Method
<b>GPG</b>	Good Practice Guide
<b>GUM</b>	Guide to the expression of Uncertainty in Measurement
<b>MCM</b>	Monte Carlo Method
<b>MEMS</b>	Micro ElectroMechanical Systems
<b>PDF</b>	Probability Distribution Function
<b>ROI</b>	Region Of Interest
<b>SEM</b>	Scanning Electron Microscope
<b>SiNWs</b>	Silicon Nanowires
<b>SThM</b>	Scanning Thermal Microscopy
<b>VIM</b>	International Vocabulary in Metrology



## INTRODUCTION

This Good Practice Guide (GPG) describes the methodology for determining thermal properties of individual nanowires and thin films related to them using Scanning Thermal Microscopy (S<sub>Th</sub>M) technique in its standard, commercially available configuration and with improved circuitry using transformer bridges. The document focuses on traceability aspects of S<sub>Th</sub>M, sample preparation, measurements at different scanning regimes and data evaluation.

In the framework of Nanowires project also the dual probe S<sub>Th</sub>M technique was developed, which is discussed in Good Practice Guide No 1, together with MEMS platform measurements description and is not part of this document.

## S<sub>Th</sub>M TRACEABILITY

### Preliminary recommendations for S<sub>Th</sub>M measurements:

**Probe manipulation:** Use of an electrostatic wrist strap during probe manipulation is strongly recommended, as S<sub>Th</sub>M thermo-resistive probes are very sensitive to electrostatic discharges.

**Electrical current value:** Value of the electrical current must be maintained below a critical value (generally specified by the supplier) in order to avoid damages or premature wear of the probe.

### Environmental conditions:

- Thermal steady-state conditions are required during measurements: care about the influence of the laser spot that can heat the probe, which leads to drift in the electrical resistance of the thermo-sensitive element of the S<sub>Th</sub>M tip. This can also induce bimetallic effects due to this photothermal effect.
- An optical table must be used to isolate the AFM from vibrations (ground and acoustic vibrations can excite the vibratory modes of the cantilever and disrupt both AFM and thermal measurements).
- Measurements can be performed either in air or in vacuum condition. In air condition both temperature and relative hygrometry have to be controlled to remain stable during the experiment.

### S<sub>Th</sub>M device:

Scanning Thermal Microscopy is a method from the family of Scanning Probe Microscopes (SPM), based on use of a small heated resistive probe scanned in contact to a sample surface. The heat flux between the probe and sample can be used to determine the local thermal conductivity of the sample. Therefore, a key component of a S<sub>Th</sub>M is a Wheatstone bridge that can be used to control the electrical current flowing through the probe and to measure probe resistance. The rest of instrumentation is same as for all the other SPM methods, typically being based on laser based pickup of probe holder (cantilever) deflection which is related to probe-sample force and piezoelectric transducers as actuators.

### Measurement definition:

For thermal conductivity measurement, the S<sub>Th</sub>M is used in its active mode: the probe acts both as a local heater and as a sensor such as illustrated in Figure 1a. To heat the probe, a sufficiently high electrical current  $I$  (A) (several mA or

even several tens of mA depending on the type of the using SThM probe) is used to heat thermoresistive probes by Joule effect while for thermoresistive probes, a laser spot focused on the tip is used to heat it. Currently, the most commonly used probes are thermoresistive probes. In steady state, the hot probe exchanges a constant heat flow corresponding to a thermal conductance  $G_{tot}$  with its surroundings.

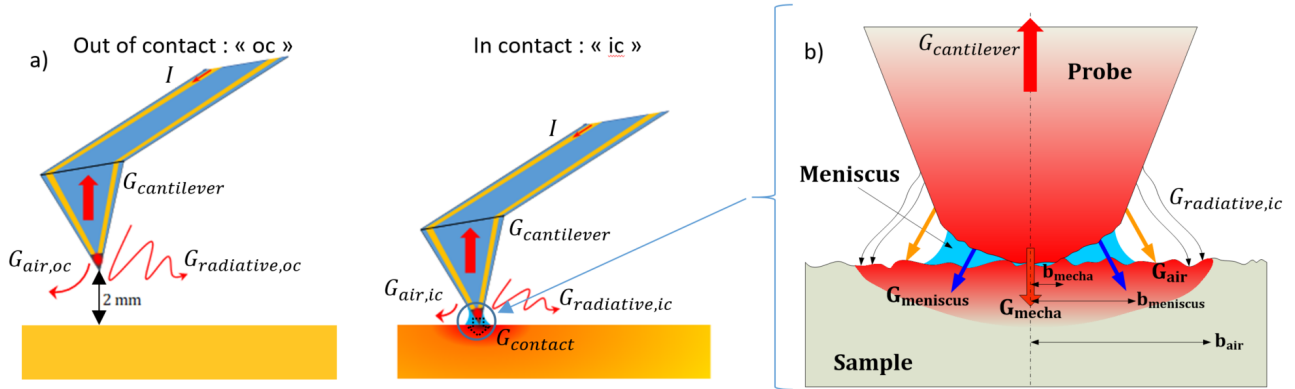


Figure 1: a) Heat transfers from out of contact to in contact configuration in air, b) Zoom on the tip apex and sample surface contact with simplified heat transfer mechanisms in air at the contact between the tip apex and the surface of the sample.

When the tip is out of contact (“oc”), there are three different heat transfers, characterised by a thermal conductance, involve during the measurement such as conduction through the cantilever  $G_{cantilever}$ , thermal radiation  $G_{radiative,oc}$  and conduction through the gas surrounding the probe  $G_{air,oc}$ . When the tip comes in contact (“ic”) with the sample, a new channel  $G_{contact}$  for the heat transfer appears which allows the hot probe to release a quantity of heat to the sample that depends on the thermal conductance of the contact and that of the thermal conductivity  $k$  of the sample. Depending on the measurement conditions,  $G_{contact}$  can include different types of heat transfers as conduction through the water meniscus formed at the tip-sample contact  $G_{meniscus}$ , through the air close to the contact  $G_{air}$  and heat conduction through the solid-solid contacts between the tip and the sample  $G_{mecha}$  as illustrated in Figure 1b. As the probe cools between out of contact and in contact configuration, its temperature changes which results in variation of its electrical resistance  $\Delta R$ . This variation is measured and linked to the thermal conductivity  $k$  of the studied sample following a measurement model  $h$  involving all thermal conductances  $G_i$  ( $i = 1, \dots, n$ ). The thermal conductivity  $k$  is identified by inverse technique from the measurement model described by the general form [1]:

$$h(k, \Delta R, G_1, \dots, G_n) = 0 \quad [1]$$

It means that measuring thermal conductivity using the SThM technique is an indirect measurement method (6) and that it is challenging to ensure the traceability of measurement performed using this technique. The VIM defines the metrological traceability as the “property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty” (1). Since there are more than one input quantity in the measurement model [1], each of the input quantity values  $\Delta R$  and  $G_i$  should itself be metrologically traceable. One way to ensure traceability is to build a calibration curve based on a set of bulk samples that are calibrated using normalised measurement procedures (2), (3), (4), (5). SThM probe is characterised using this set, calibration curve is constructed and measurement on the unknown sample is performed under the same conditions as measurement on the bulk samples as illustrated in Figure 2. This represents the most straightforward method for obtaining traceable results, namely in contrast to calculating all the thermal conductances  $G_i$  involved during measurements from the geometrical and electrical parameters of the system. Nevertheless, the

calibration curve based methods has its own disadvantages, namely related to bulk materials roughness, impact of probing volume and long term drifts in the electronics.

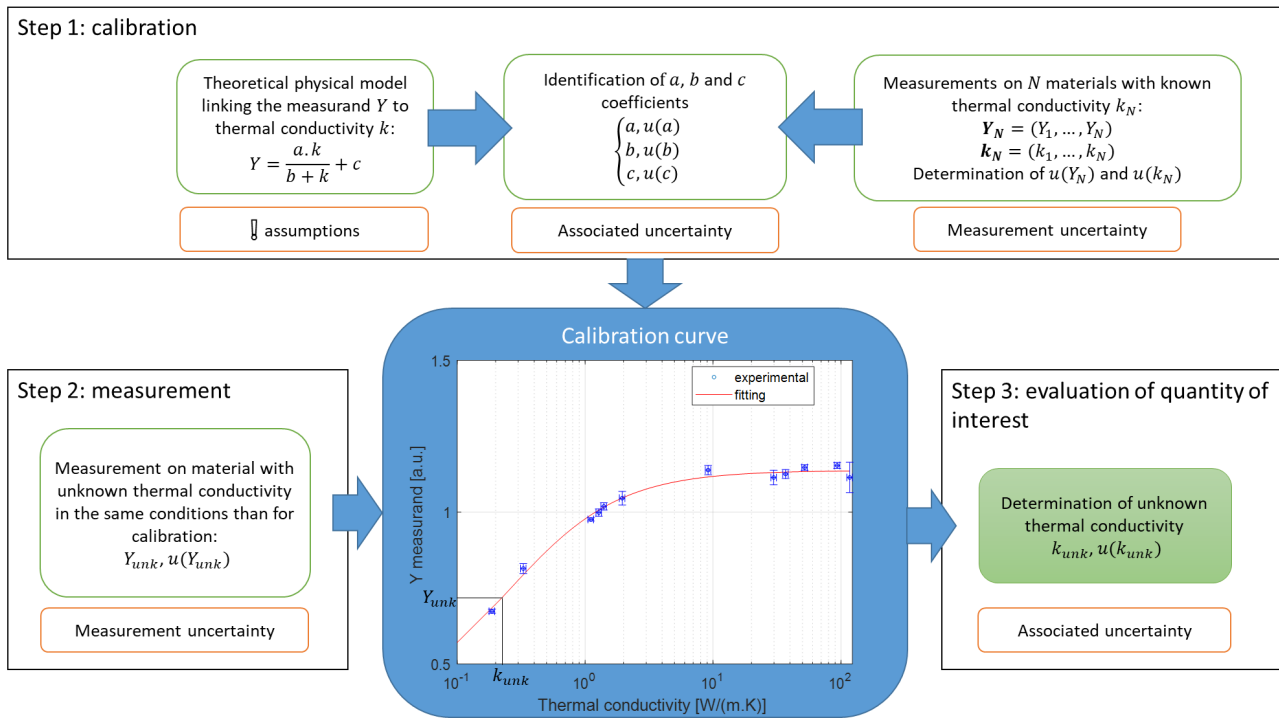


Figure 2: Methodology for traceable thermal conductivity measurement with SThM technique using a calibration curve built with a set of bulk materials with known thermal conductivity.

## Calibration:

### DEFINITION OF THE CALIBRATION SAMPLES:

To ensure the traceability, thermal conductivity value of the calibration samples have to be measured using traceable techniques. The samples have to be homogeneous, isotropic and with roughness as low as possible (ideally with  $R_a < 1 \text{ nm}$ ). They should be select to cover a wide range of thermal conductivity, at least in the range of  $0.1$  to  $10 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ . Examples of calibration materials could be found in (6) such as polymers (poly(methyl methacrylate PMMA), poly-oxymethylene in copolymer POM-C), ceramics (glass, silicon dioxide, alumina) and metals (zinc).

### DEFINITION OF THE MODEL:

In order to link the intermediate measurand  $\Delta R$  to the thermal conductivity  $k$  of the studied sample, it is necessary to establish a physical model describing heat transfers between the probe and the surface sample. As illustrated in Figure 1b, the contact thermal conductance  $G_{contact}$  can be represented, in a simplified way, as:

$$G_{contact} = \frac{G_{mecha} \cdot G_{env}}{G_{mecha} + G_{env}} \quad [2]$$

with  $G_{env}$  the global thermal conductance due to conduction through the meniscus and the air, as well as the radiative contribution and  $G_{mecha}$  the conduction through the solid-solid contacts between the apex of the tip and the sample. As the calibration samples are bulk, isotropic and with sufficient thickness to be assumed semi-infinite,  $G_{mecha}$  can be expressed as:

$$G_{mecha} = 4 \cdot \beta \cdot b_{eff} \cdot k \quad [3]$$

with  $\beta$  a specific coefficient corresponding to the influence of the shape of the thermal contact and  $b_{eff}$  the radius of the thermal contact. As a result, the variation of the total conductance  $\Delta G_{tot}$  between “oc” and “ic” configuration can be described as:

$$\Delta G_{tot} = G_{air,oc} - G_{air,ic} + \frac{G_{mecha} \cdot G_{env}}{G_{mecha} + G_{env}} = \frac{G_{env} \cdot k}{\frac{G_{env}}{4 \cdot \beta \cdot b_{eff}} + k} + G_{air,oc} - G_{air,ic} \quad [4]$$

This function, relative to a  $\frac{A \cdot k}{B+k} + C$  model can be used to determine thermal conductivity, but, as discussed previously, this requires that all the terms ( $G_i$ ) to be expressed by complete physical models, which is complex and involves many influencing parameters. Another solution is to globally identify the coefficients of the model using a calibration curve linking the thermo-electrical response of the probe to the thermal conductivity of the sample. This calibration curve is based on the measurements of a set of bulk calibration materials with well-known thermal conductivities, and the fit these experimental points with the model deduced from phenomenological studies of the measurements to deduce the coefficients of the model as illustrated in Figure 3.

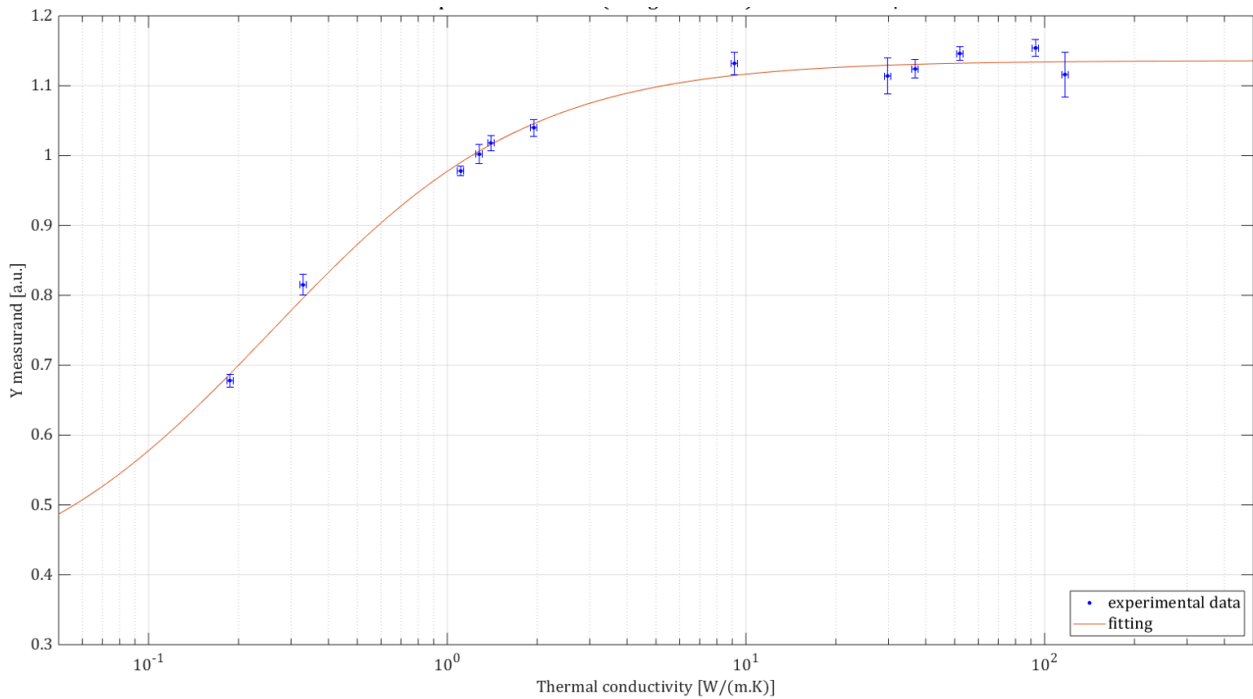


Figure 3: Example of a calibration curve obtained for a thermoresistive probe (KNT probe provided by Kelvin Nanotechnology)

It is important to note that the Figure 3 shows a reduced range of sensitivity to the thermal conductivity for KNT probes, limited to thermal conductivity lower than  $10 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ .

#### DEFINITION OF THE INTERMEDIATE MEASURAND AND RECOMMENDATIONS:

In the studied measurement model, the thermal conductivity  $k$  is the measurand (quantity intended to be measured) as defined in the International Vocabulary in Metrology (1), and the variation of the thermo-electrical response of the probe is an intermediate measurand  $Y$ .

The thermal conductance  $G_{tot}$  can be expressed as a function of the thermo-electrical response of the probe:

$$G_{tot} = \frac{P}{(T-T_a)} = \frac{RI^2}{(T-T_a)}, \quad [5]$$

and the probe electrical resistance  $R(T)$  depends on its temperature  $T$  and follows a quiet linear relationship with its temperature for low variation of temperature:

$$R(t) = R(T_a) \cdot [1 + \alpha \cdot (T - T_a)], \quad [6]$$

with  $R(T_a)$  the electrical resistances of the resistive sensor at the reference temperature  $T_a$  and  $\alpha$  the temperature coefficient of the resistive sensor. When the SThM works with constant current intensity through the probe, the variation  $\Delta R$  with the thermal conductivity can be assumed similar to the evolution of the thermal conductance  $\Delta G$  and be expressed by the following calibration law:

$$\Delta R = \frac{A_R \cdot k}{B_R + k} + C_k. \quad [7]$$

Unfortunately, experiments highlight drifts of the value of  $R(T_a)$  depending on the ageing of the probe. In addition, long term drifts in the electronics and residual temperature of the environment drifts have been observed. In order to limit the influence of these drifts on the measurements, it is recommended to work using a reference sample measured before each measurement on a new sample. This reference sample must be selected so as to have a thermal conductivity within the sensitivity range of the technique ( $k < 10 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ ) and a roughness as low as possible. The intermediate measurand  $Y$  is thus defined as the ratio between  $\Delta R_{sample}$  results on a studied sample and  $\Delta R_{ref}$  results on the reference sample:

$$Y = \frac{\Delta R_{sample}}{\Delta R_{ref}}. \quad [8]$$

As  $\Delta R_{ref}$  does not depend on the thermal conductivity  $k$  of the studied sample, the  $k$ -dependence of  $\Delta R$  expressed in equation [7] remained unchanged when divided by a constant value. The intermediate measurand can thus be expressed as:

$$Y = \frac{\Delta R_{sample}}{\Delta R_{ref}} = \frac{a \cdot k}{b+k} + c. \quad [9]$$

### Uncertainty assessment:

As mentioned in the VIM (1), a calibration consists in a two-steps procedure. In the first place, the relationship between the thermal conductivity value  $k_N$  of the  $N$  calibration samples (and their associated uncertainty  $u(k_N)$ ) and the indications (the intermediate measurand  $Y_N$  measured for each calibration sample) of the SThM (and the associated uncertainty  $u(Y_N)$ ) has to be estimated. In a second place, a measurement result is obtained, from an intermediate measurand  $Y_{unk}$  and its associated uncertainty  $u(Y_{unk})$ , consisting of the estimation of the thermal conductivity  $k_{unk}$  of an unknown sample and its associated uncertainty  $u(k_{unk})$ .

UNCERTAINTY ASSOCIATED WITH THERMAL CONDUCTIVITY VALUES OF THE CALIBRATION SAMPLES:

Uncertainty associated with the traceable measurement of the thermal conductivity of the reference samples will be calculated in accordance with normalised measurement procedures selected for the measurement and the Guide to the Expression of Uncertainty in Measurement (GUM) (7). As an example, the thermal conductivity determined using an indirect and traceable method is described in (8). Using measurements of the thermal diffusivity by the laser flash method, of the specific heat by differential scanning calorimetry, and the density by the Archimedean method, the expanded relative uncertainty associated with the measurement of thermal conductivity by this indirect method has been estimated at 5%.

UNCERTAINTY ASSOCIATED WITH THE INTERMEDIATE MEASURAND VALUES:

Uncertainty associated to the intermediate measurand  $Y$  could be estimated using a classical law of propagation of uncertainty according to the GUM (7). Nevertheless care should be taken to take into account the numerous correlations between the different quantities involved during the measurement process. Another way to estimate the associated uncertainty with the intermediate measurand  $Y$  is, first, to analyse the measurement process used to measure  $Y$ , in a second time, to determine the assigned Probability Distribution Function (PDF) to each input quantity involved in the measurement and then, to propagate the distributions using a Monte Carlo method according to the principles of the Supplement 1 to the GUM (9) as illustrated in the Figure 4. The typical relative expanded uncertainty associated with the intermediate measurand  $Y$  has been estimated to be 2% at most in the framework of the project (6).

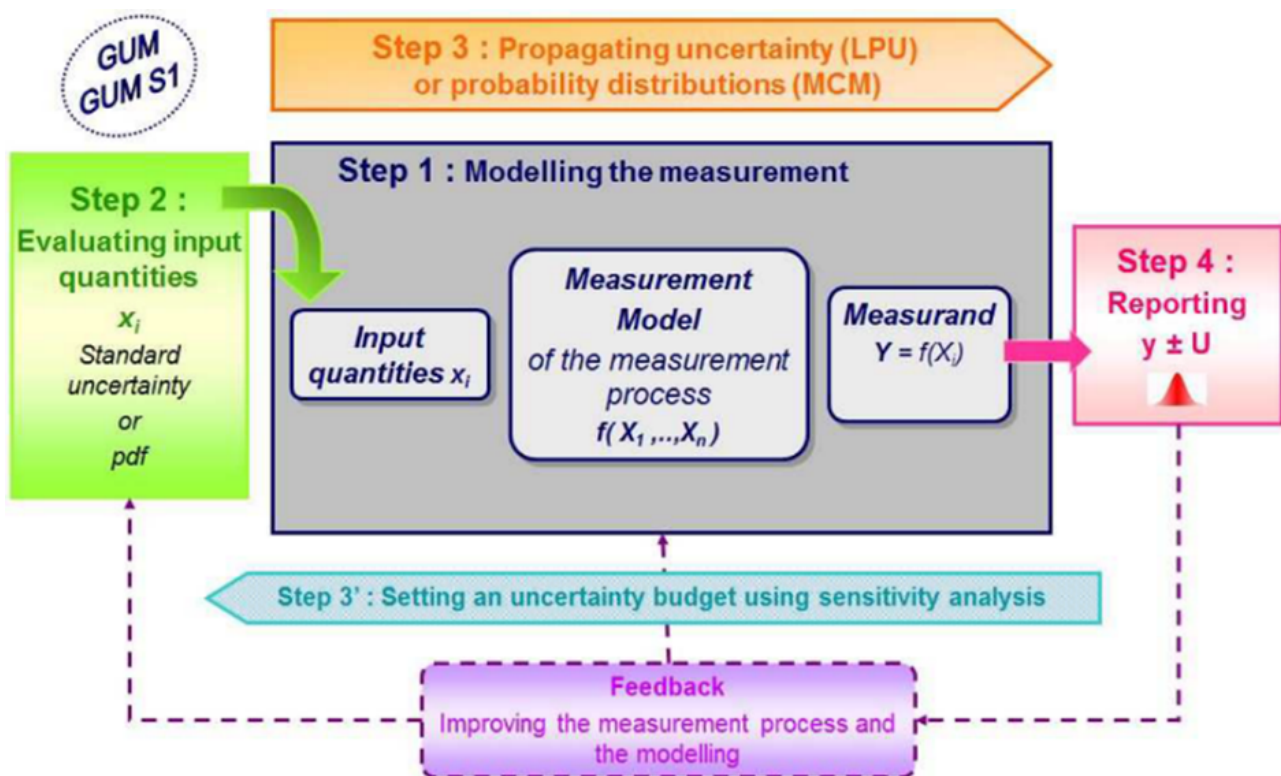


Figure 4: Main steps of an evaluation of measurement uncertainty according to the GUM or GUM-S1 : Modelling the measurement, Evaluating input quantities, Propagating, Reporting and Setting an uncertainty budget

UNCERTAINTY ASSOCIATED WITH THE IDENTIFIED THERMAL CONDUCTIVITY FOR AN UNKNOWN MATERIAL:

The identification of the thermal conductivity  $k_{unk}$  of an unknown material from the calibration curve is performed using the evaluation of the coefficients  $a$ ,  $b$  and  $c$  of the equation [9] and an inversion procedure. Parameters  $a$ ,  $b$  and  $c$  could be estimated using an Ordinary Least Squares method. However, this method provides an uncertainty associated with these coefficients that does not take into account the uncertainty associated with both the thermal



conductivity of the calibration sample and the intermediate measurand. Two methods can be used to identify the  $a$ ,  $b$  and  $c$  parameters taking uncertainties into account: Monte Carlo Method (MCM) or Bayesian statistical inversion (9).

- ❖ Using MCM, we can perform a Monte Carlo simulation to generate plausible calibration data based on the measurement uncertainty obtained for the intermediate measurand  $Y_N$  and for the thermal conductivity values  $k_N$ . This simulation is based on the algorithm described in Figure 5. At each Monte Carlo run:
  - the calibration data are sampled from Gaussian distributions with parameters  $k_N$ ,  $u(k_N)$ ,  $Y_N$  and  $u(Y_N)$ ,
  - the calibration model is estimated based on the simulated calibration data,
  - the intermediate measurand on an unknown sample is injected,
  - the calibration model is inverted in order to obtain an estimate of the corresponding thermal conductivity.

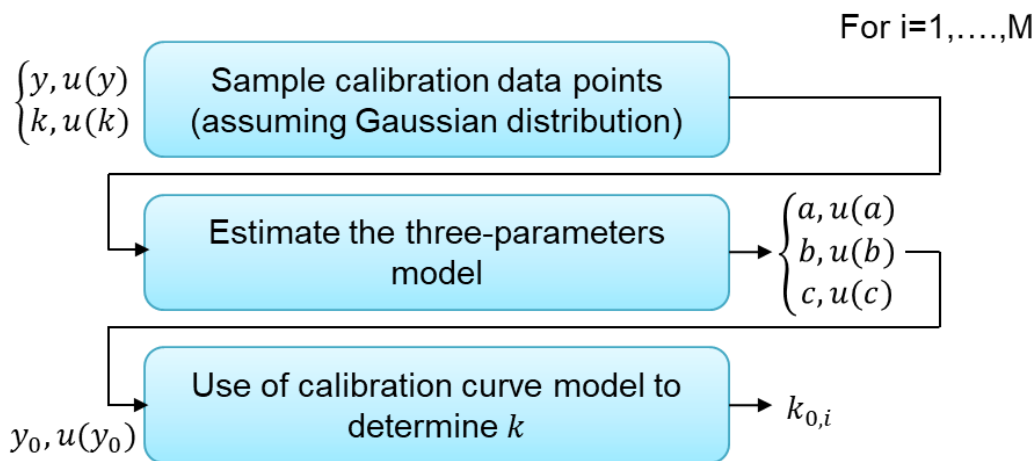


Figure 5: Steps of the Monte Carlo algorithm

- ❖ The advantage of using Bayesian statistical inversion is the joint estimation of the calibration parameters and the thermal conductivities from fresh SThM measurements in addition to the calibration data. Details of the procedure can be found in (6). Under specific conditions, i.e. ensuring strictly steady-state environmental conditions, minimising the roughness of studied materials, ensuring that measurement condition are the same between calibration and measurement (diffusive heat transfer regime), it is possible to perform traceable measurement of thermal conductivity with SThM technique and to reach an uncertainty value of less than 10 % for thermal conductivity lower than  $10 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ .

**SAMPLE PREPARATION**

Two types of individual nanowires measurements were tested in the project. Porous silicon nanowires (p-Si) arrays, shown in Figure 6 (left), with diameter of 100 nm and length around 20  $\mu\text{m}$  were fabricated on n+ doped silicon surface by polystyrene nanosphere self-assembly (nanospheres lithography - NSL) and metal-assisted chemical etching (MACE) (see GPG1 for process flow and details) (10). After that, the nanowires were transferred to a solid Si substrate

with a SiO<sub>2</sub> layer of 90 nm (in some cases 300 or 500 nm of SiO<sub>2</sub> can be chosen) having known thermal properties. The transfer was done by picking the nanowires with a piece of lab cloth by means of electrostatic attraction and by realising them randomly on the substrate. The flat surface covered with nanowires was then inspected by SEM to find nanowires with good characteristics (e.g. isolated, entire, absence of other nanowires fragments). To make sure the chosen single nanowires can be found to perform the SThM measurement, local platinum deposition was performed to fabricate markers as “find me” structures. Pt markers were deposited by focused ion beam-induced Pt gas injection deposition inside the vacuum chamber of the electron microscope. As shown in Figure 6 (right), the contamination is reduced to a minimum by avoiding focusing on the nanowires with the ion beam, but using an e-beam for approximate alignment to the region of interest (ROI).

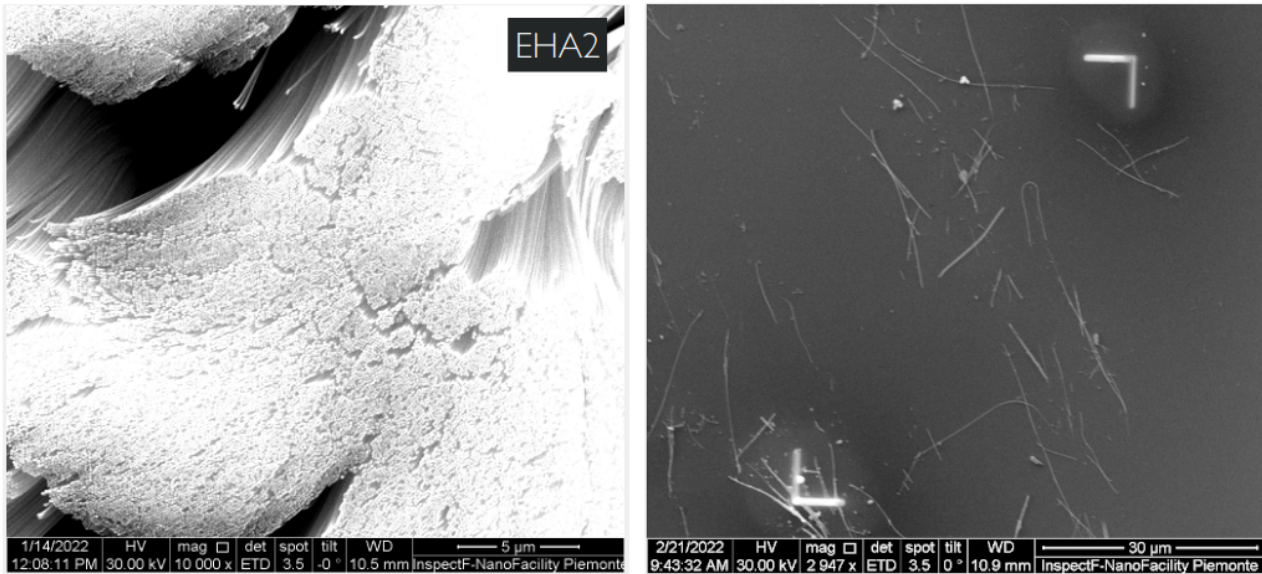


Figure 6: The SEM image on the left reports the array of porous silicon nanowires fabricated by NSL and MACE while the SEM image on the right shows the single nanowires spread randomly on the flat 90 nm SiO<sub>2</sub> on Si substrate and at right, in proximity of the two “L” L-shaped Pt markers surrounding the region of interest (ROI). The contamination halo is visible but, and it’s not extending to the dispersed nanowires in the ROI.

Second, nanowires can be embedded inside a matrix with very low thermal conductivity and measured as grown. The benefit of this approach is that we do not mix the radial and axial thermal conductivity, as in case of nanowires placed on the surface.

**STANDALONE NANOWIRES MEASUREMENTS USING STANDARD SThM TOOLS**

Standalone nanowires, spread on sample surface, can be measured using SThM only if the used scanning regime prevents their movement across the surface. For standard imaging, a tapping mode can be used, which is however incompatible with SThM operation, which needs a good thermal contact between probe and sample. A potential solution is to use Force Volume regime or some off-resonance tapping regime, all performing individual force-distance curve measurements at each sample point. In this way the lateral movement of the probe is done only when the probe is not in contact with the surface. A side benefit of using this type of measurement is a better defined contact force and contact time. Tools for loading and processing force volume data in Gwyddion open source software were developed recently, which simplifies the data processing. A summary of the steps used for traceable measurement of the standalone nanowires is as follows:

- use SThM settings with high probe temperature to increase the sensitivity to thermal conductivity changes

- perform probe calibration using a set of samples with known thermal conductivities, use the Force Volume regime with higher contact forces (e.g. >100 nN). Hold the maximum force value for at least 10 ms and sample data points during it. Use the force distance curve sampling rate allowing the probe to thermalize, the reaction time depends on the probe and can be determined by inspecting the approach and retracting part of the thermal signal, these should overlap.
- find the area of interest on the NW sample, perform measurement on the nanowire with enough large spatial resolution (at least 10 pixels across the nanowire), use Force Volume with the same settings as during the calibration.
- evaluate the data to construct the calibration curve and to construct a map of the SThM output on the NW sample, use the SThM bridge values at the maximum force, which usually also corresponds to an extrema in the SThM bridge signal.
- when evaluating the effective thermal conductivity of the nanowire, use an area that is not affected by topography artefacts - flat parts, top of the nanowire, etc.

As an example of standalone NW measurement, thermal properties of individual nanowires were measured using Scanning Thermal Microscopy (SThM) on Dimension Icon Scanning Probe Microscope (Bruker) with VITA SThM electronics (Anasys Instruments) and VITA-DM-GLA probes. SThM probes were calibrated using a set of bulk material samples measured by French national metrology institute LNE in the framework of FP7 project Quantiheat using laser flash method. Moreover, to relate the SThM measurements to the numerical model, we used silicon dioxide steps on silicon developed by Glasgow University (11), with known effective thermal conductivities, taken from Ref. 1. Measurements were performed in Force Volume mode to prevent movement of nanowires over the surface, observed in contact mode. Moreover, this provided better control over contact force, which was set to 90 nN. In all measurements, including calibration, the SThM signal was referenced to a value far from the sample to prevent impact of electronic drifts. An example of the topography and thermal signal image on a single nanowire is shown in Fig. 7.

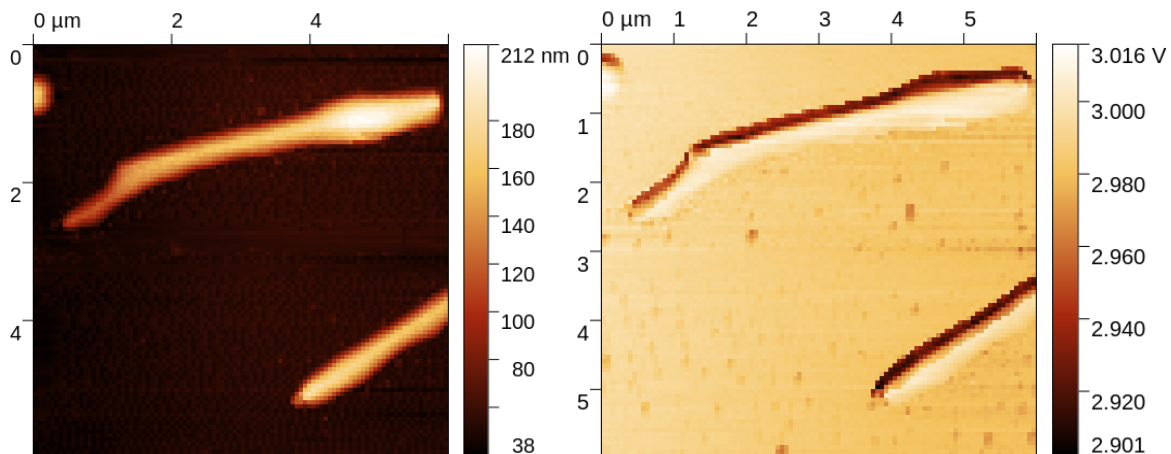


Fig. 7: SThM measurement on a NW sample, left: topography, right: thermal signal

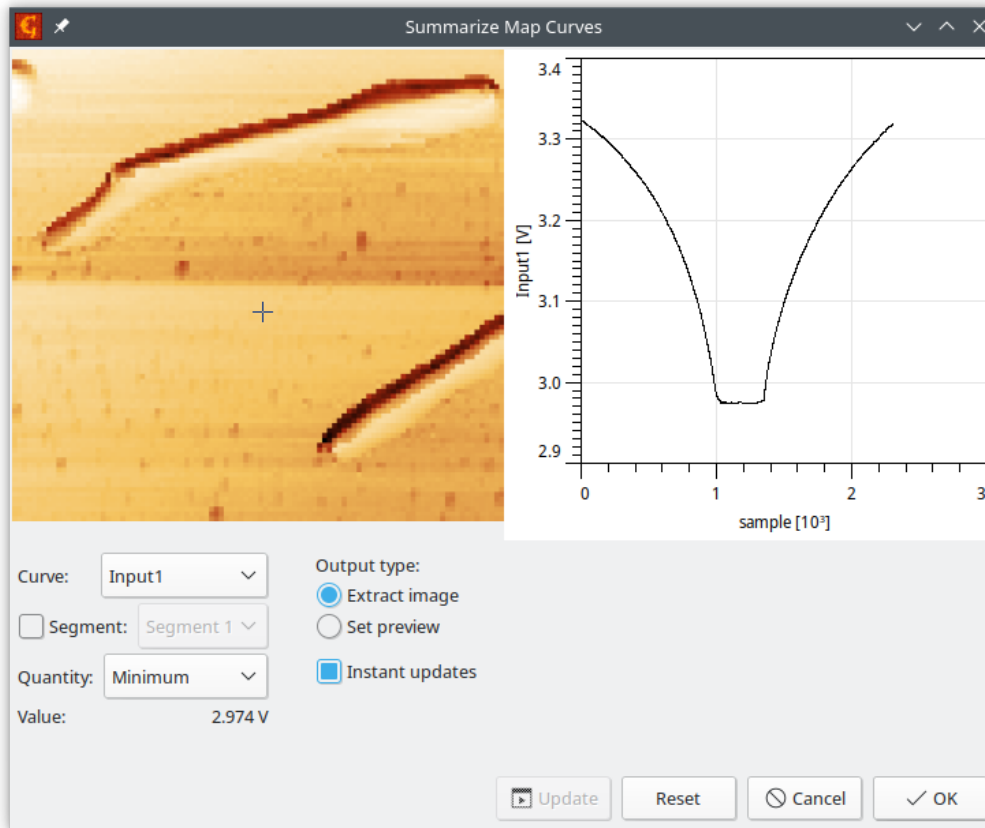


Fig 8: force volume data in Gwyddion open source software showing the SThM channel

Measured data were processed using Gwyddion open source software (12). An example of force volume data as opened in Gwyddion open source software is shown in Fig. 8. Data processing consisted of obtaining a probe calibration curve from bulk and thin film samples. This calibration curve was used to determine an effective thermal conductivity of the nanowire, as illustrated in Fig. 9. For this, a profile across the SThM signal of the nanowire was taken. The profile is highly affected by topography artefacts, therefore the peak value, corresponding to the top of the nanowire and minimum probe-sample contact radius was used, similarly to the numerical model discussed below. The substrate around nanowire, characterised earlier right after the SThM probe calibration was taken as a reference, to prevent impact of long term drifts of VITA electronics. The measured effective thermal conductivity of the nanowire at its top was  $(2.6 \pm 0.5)$  W/m/K. Uncertainties were determined from calibration curve fitting and data variance, however no full uncertainty budget was established yet.

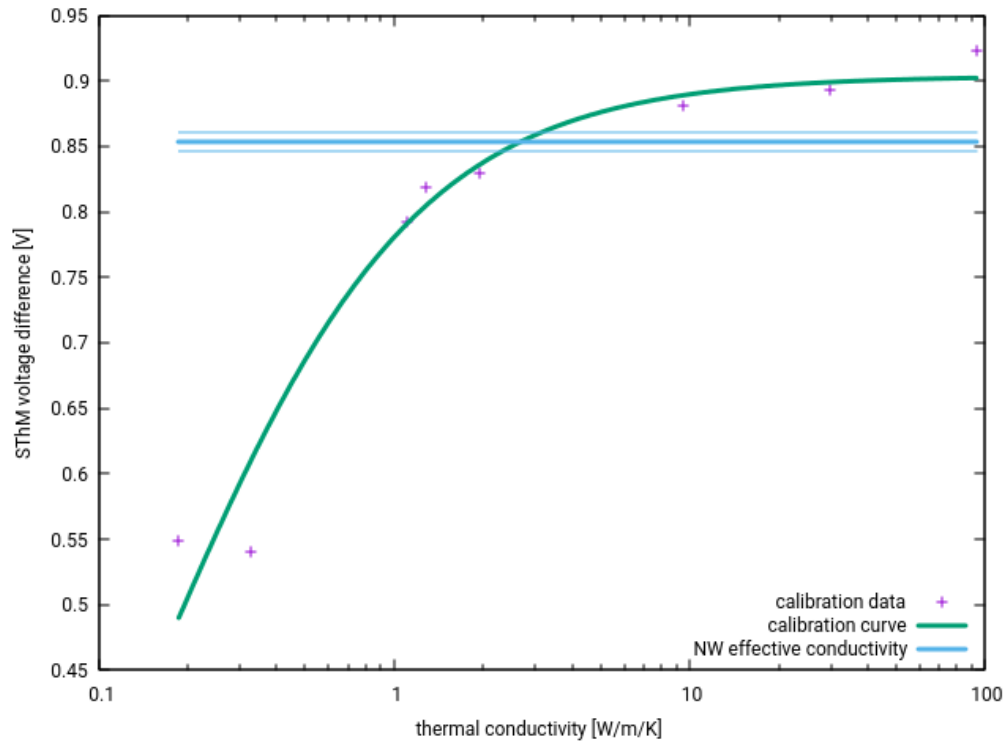


Fig. 9: SThM calibration curve showing the intersection with measured SThM signal on top of the nanowire. Thin blue lines show the error margins coming from the experimental data.

To relate the effective thermal conductivity to thermal conductivity of the nanowire itself, a simple Finite Difference method (FDM) model was used (13). The model was based on the assumption that the heat transfer is diffusive, which might be correct for the amorphous nanowire, however it might have large errors for both air heat transfer for small probe-sample separations and for crystalline materials. Probe geometry for the model was estimated using blind tip estimation on a rough silicon sample, providing average probe radius of 60 nm, diameter the nanowire was determined from tapping mode Atomic Force Microscopy measurements. The voxel size for the model was 5 nm. The FDM model provides heat power flowing through the probe-sample system, which is not the same signal as SThM voltage difference. SThM voltage difference relies on the electronics settings, environmental factors and probe-sample area geometry and converting it to heat power is not a simple task. For the modelling, we assumed that all the effects, apart from the probe-sample area geometry changes, were constant during the experiment. To relate the numerical model to the experimental data, we simulated the thin film sample developed by Glasgow University, using the same probe and all the settings, similarly as it was done in the experiment. As the effective thermal conductivities of the thin film sample were known, this allowed us to set up a relation between the effective thermal conductivity, measured using SThM, and heat power in the FDM model. To provide a coarse estimate of the uncertainty related to the numerical model we performed simulations with different probe radii, different contact resistances and different substrate conductivities, reflecting our experimental uncertainties, however, it should be noted that this numerical simulation is only a simple estimate and a better numerical model would be necessary to prevent potential model related biases. The estimated thermal conductivity of the nanowire under these assumptions is  $(3.1 \pm 1.2)$  W/m/K.

## NANOPILLARS MEASUREMENTS

In order to perform measurement on nanowires in conditions that respect the requirement of diffusive heat transfer for traceable measurement, we selected nanowires with dimensions higher than the mean free path of the material.

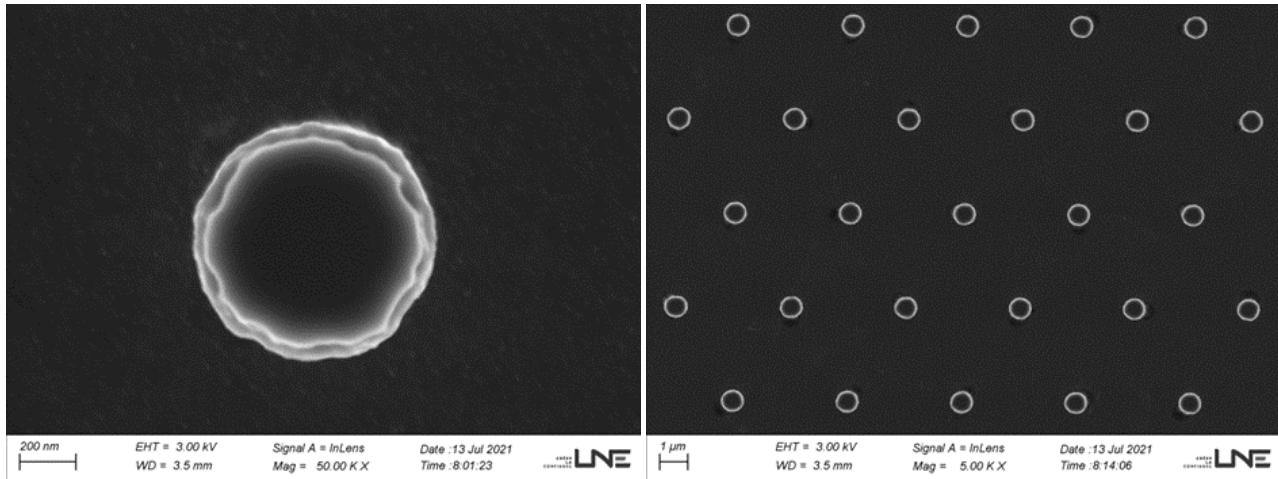


Figure 10: SEM image of Si nanowires F1-D08-P40 produced by TUBS for WP1

The selected silicon nanowires are regular distributed, vertical standing nanowires (illustrated in Figure 10), produced by TUBS in the framework of the project. The dimensional parameters of the nanowires have been characterised in WP1.

S<sub>Th</sub>M measurements have been performed using scan contact mode with a commercial NTEGRA scanning probe microscope from the NT-MDT company. Results on S<sub>Th</sub>M measurements show that, due to high aspect ratio, it is really difficult to maintain a good contact between the probe and the sample in scan contact mode. As observed on Figure 11, the contact is lost when the probe tries to scan the nanowire structure. For this figure, the scan direction is parallel to the cantilever direction with worse results when the scan direction is perpendicular to the cantilever. It could be due to the movement of the SiNW: when the probe is in contact with the SiNW, the applied force could be sufficient to bend the top of the SiNW. The strong deformation observed in the shape of the nanowire is due to the correlation between the shape of the thermal tip and the shape of the nanowire. This measurement artefact regularly observed with the topography mode, is also visible with the thermal mode.



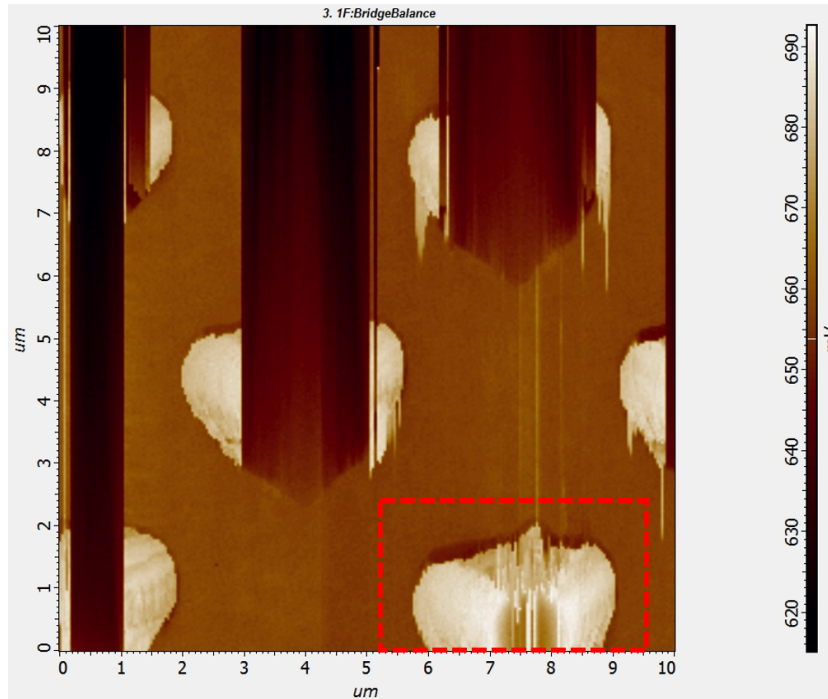


Figure 11: NTEGRA thermal contrast image on the TUBS F1-D08-P40 Si nanowires

Analysis of the only nanowire that is relatively correctly scanned (identified in the red rectangle in figure 11) shows that only the central part of the image is relevant for thermal contrast study. The thermal profile established using MountainsLab® software shows that the diameter of the relevant part of the scan is about 500 nm which is lower than the diameter of the top of the SiNW (around 700 nm). That means that the thermal radius of the thermal exchange surface could be estimated to 200 nm for the KNT probe in air condition.

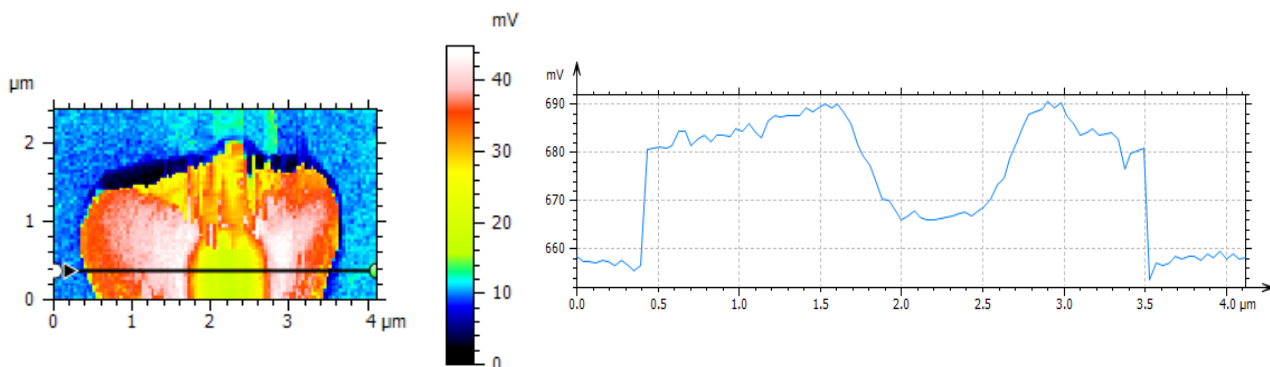


Figure 12: MountainsLab® traitement of the NTEGRA thermal contrast image. Analysis focuses on one nanowire from figure 11.

Another method to perform SThM measurement on nanowires is the Force-Volume (or Pinpoint™) mode, as mentioned in the previous chapters. Using this mode, it is possible to scan the surface of the sample without moving it. After each contact location, the probe withdraws from the surface of the sample to move above another location before landing on this new location. Some examples of thermal contrast images performed using this mode are presented in figures 13 and 14.



Figure 13: SThM measurement on the TUBS F1-D08-P40 Si nanowires, left: topography, right: thermal signal. Measurements were performed using Park NX-Hivac AFM with resistive SThM module and KNT probe.

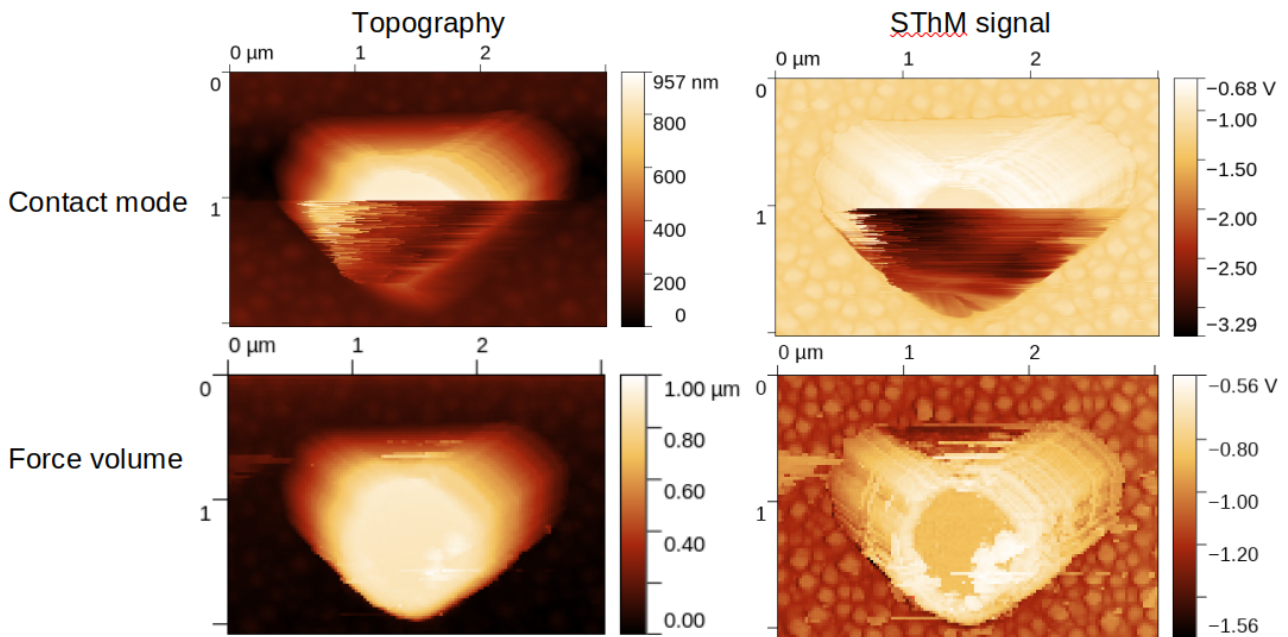


Figure 14: SThM measurement on the TUBS nanowires showing the difference between contact mode and Force-Volume mode.

It should be noted that using these “non permanent” contact modes (force volume mode for Bruker equipment and Pinpoint™ mode for Park Systems equipment) are useful for nanowires measurement. They limit the risk of displacement or movement of the studied structure. If these modes are currently not yet characterised to perform SThM traceable and quantitative measurements, they are promising techniques to perform measurement on nanostructured materials like nanowires.

**REFERENCES**

1. International vocabulary of metrology - Basic and general concepts and associated terms (VIM), 3rd edition. *JCGM 200:2012*. 2012.
2. ISO 22007-1:2017. Determination of thermal conductivity and thermal diffusivity - Part 1: General principles. 2017.
3. ISO 22007-2:2022. Plastics — Determination of thermal conductivity and thermal diffusivity — Part 2: Transient plane heat source (hot disc) method. 2022.
4. ISO 22007-3:2008. Plastics — Determination of thermal conductivity and thermal diffusivity — Part 3: Temperature wave analysis method. 2008.





5. ISO 22007-4:2017. Plastics — Determination of thermal conductivity and thermal diffusivity — Part 4: Laser flash method. 2017.
6. N. Fleurence, S. Demeyer, A. Allard, S. Douri and B. Hay. Quantitative Measurement of Thermal Conductivity by SThM Technique: Measurements, Calibration Protocols and Uncertainty Evaluation. *Nanomaterials*. (2023), 13(17), 2424; <https://doi.org/10.3390/nano13172424>.
7. GUM 1995 with Minor Corrections Evaluation of Measurement Data—Guide to the Expression of Uncertainty in Measurement; JCGM; Guide 100:2008; Joint Committee for Guides in Metrology (JCGM): Sèvres, France 2008
8. Hay, B.; Hameury, J.; Filtz, J.-R.; Haloua, F.; Morice, R. The metrological platform of LNE for measuring thermophysical properties of materials. *High-Temp. -High Press.* 2010, 39, 181–208
9. Guide to the Expression of Uncertainty in Measurement—Part 6: Developing and Using Measurement Models; JCGM GUM-6: 2020; Joint Committee for Guides in Metrology (JCGM): Sèvres, France 2020
10. E. Cara, L. Mandrile, F. Ferrarese Lupi, A. M. Giovannozzi, M. Dialameh, C. Portesi, K. Sparnacci, N. De Leo, A. M. Rossi, L. Boarino, Influence of the long-range ordering of gold-coated Si nanowires on SERS, *Scientific reports*, 8, 1, 11305 (2018)
11. Eloïse, Guen & Chapuis, Pierre-Olivier & Rajkumar, Ravishkrishnan & Dobson, Philipp & Mills, Gordon & Weaver, Jonathan & Gomès, Séverine. (2020). Scanning thermal microscopy on samples of varying effective thermal conductivities and identical flat surfaces. *Journal of Applied Physics*. 128. 10.1063/5.0020276.
12. D. Nečas, P. Klapetek, Gwyddion: an open-source software for SPM data analysis, *Central European Journal of Physics*, 10 (2012) 181-188
13. P. Klapetek, J. Martinek, P. Grolich, M. Valtr, N. J. Kaur, Graphics cards based topography artefacts simulations in Scanning Thermal Microscopy, *International Journal of Heat and Mass Transfer* 108 (2017) 841–850