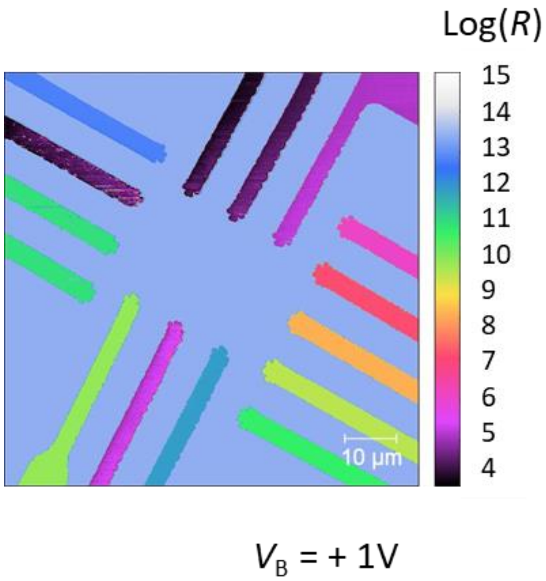
Good practice guide for calibrated resistance and current measurements using conductive probe atomic force microscopy



Abstract

The measurement of electrical properties at the nanoscale allows evaluating the performance of nanomaterials developed for consumer electronics, innovative quantum technologies, and IoT applications. Local DC resistances and high frequency (HF) impedances are among the most prominent properties to measure for nowadays advanced devices. Currently, Conductive probe Atomic Force Microscopy (C-AFM) and Scanning Microwave Microscopy (SMM) are two main techniques used for the characterization of these properties. Although powerful, these two techniques suffer from major drawbacks: costly, complicated implementation, and lack of traceability. Measurements are thus unreliable. The 20IND12 EMPIR project ELENA aims at pioneering the traceability of such measurements, with stated uncertainties and increasing the affordability of these methods.

Within this project, LNE, CNRS and DFM with the support of BAM, PTB, TUBITAK, jointly propose a good practice guide (GPG) on the calibration of C-AFM for measurement at the nanoscale covering DC current from 1 fA to µA, and DC resistance from 1 kΩ to 1 TΩ, for use in industrial applications. This GPG relies on robust calibration method, reference samples and simplified uncertainty budgets. It applies on two operating modes: spectroscopic mode based on I-V curves and image mode, both with AFM tip in permanent contact to the scanned surface of the reference sample.

Content

1. Introduction

As technological progress continues to push back the limits of device manufacturing to incredibly small scales, a commensurate development of measurement tools and methods is inevitable. Conductive probe atomic force microscopy (C-AFM) is a scanning probe microscopy technique widely used to measure currents and resistances at the nanoscale. Its popularity has recently increased due to the versatility of its applications in mainstream technologies such as memory, photovoltaics and semiconductors.

Despite the considerable contributions of C-AFM to understanding nanomaterials and nanodevices' properties, measurements have remained prone to numerous artefacts-inducing factors and restricted to qualitative comparisons.

The three-years ELENA project: Electrical nanoscale metrology in industry (start date: 1st Sept 2021) [1] aims at establishing a European metrological infrastructure and cost-effective technologies for both techniques C-AFM and the Scanning Microwave Microscopy (SMM) providing means for industries to conduct traceable nanoscale measurements of materials and devices electrical properties. One part of the project focused on the development of reference samples and calibration methods for C-AFM while drawing up simplified uncertainty budgets, all of these for use in industrial applications.

This good practice guide (GPG) summarizes the use of C-AFM as a relevant technique for carrying out nanoscale resistance and current measurements with a demonstrated traceability to the international system of units (SI). The GPG was established by LNE, CNRS and DFM with the support of BAM, PTB and TUBITAK.

The calibration of C-AFM for resistance and current measurements relies on reference samples and procedures such as jointly developed by LNE and CNRS [2, 3]. The samples designed by LNE are composed of Surface Mount Device (SMD) resistors or Junction Field Effect Transistor (JFET) SMD diode. The C-AFM is calibrated over 8 or 9 decades by scanning (or contacting at a fixed position) the AFM tip successively on each electrodes of the reference sample. Because of difficulties met to measure resistance in the order of 100 Ω (even with metallic tip) and present lack of reference resistance values larger than 1 TΩ, the GPG presented below only concern the resistance range 1 kΩ to 1 TΩ and current range 1 fA to 1 μA. Moreover, this GPG was drawn-up for C-AFM operating either in spectroscopic (I-V curve) mode or in image mode, both with AFM tip in permanent contact with the sample surface.

In the following sections, we give a general description of the experimental set-up (C-AFM principle, environmental conditions, reference samples ...) detail the calibration methods developed for nanoscale resistance and current measurements based on C-AFM, and present calibration results on C-AFM systems with simplified uncertainty budgets. At the end of this document, a first annex describes the properties of SMD resistors and JFET diodes used for the reference sample while a second annex details the various possible artefacts in C-AFM measurements.

2. Experimental section

2.1. C-AFM measurement principle

In C-AFM, a conductive probe with a sharp nano-sized tip acts as a top electrode brought into contact with the surface of a sample while applying a potential difference (V_{in}) relative to a back electrode (Figure 1). The small currents flowing through the system are measured using a current amplifier, typically ranging from 100 fA to 10 μA for most commercially available microscopes. The amplifier can be a linear amplifier based on resistors (R) or logarithmic amplifier using a diode (D) depending on kind of C-AFM and measurement mode: current or resistance.

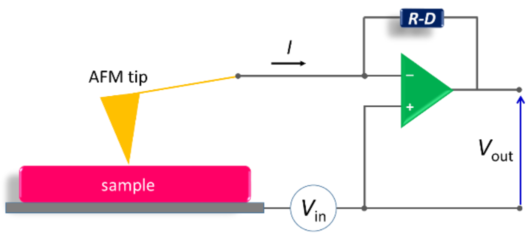


Figure 1. Schematic diagram of the C-AFM measurement circuit composed of resistors, programmable voltage source (V_{in}) and trans-impedance amplifier.

By sweeping the potential difference while the tip is fixed in contact with the sample, current versus voltage (I-V) curves are acquired. I-V curves are essentially used to extract resistance values or to characterize the electric behaviour of components and devices. Alternatively, current variation maps are acquired at a given applied voltage (V_{in}) by scanning the AFM tip in contact mode across a defined sample surface area.

It's important to note that the tip of the AFM is in direct physical contact with the sample which may cause damage both to the sample and the tip. This aspect shall be carefully evaluated. If damage cannot be ruled out, it is recommended (i) to measure the topography of the sample separately before the resistance or current measurements (ii) to measure the resistance of the tip before and after performing the resistance measurements on the sample, particularly in case of resistances $R \leq 100$ kΩ.

2.2. Environmental conditions

2.2.1. National Metrology Institute environments

The following environmental conditions support stable operation of the C-AFM with minimum mechanical, thermal, electrical drift and ensures very accurate measurements:

- Ensure stable lab climate (temperature, humidity) to enable stable operation of the C-AFM with minimum mechanical, thermal, and electrical drift;
- In the best case the C-AFM microscope shall be installed in a glove box under a dry nitrogen atmosphere (RH < 1 %) and the complete set-up (glove box, measurement circuit, etc) located in an electromagnetically shielded environment (Faraday cage) under stabilized room temperature (air conditioning system) at 20 °C or 23 °C with a temperature regulation of ± 0.3 °C or better;
- Avoid opening windows, direct sunlight on the experiment, and other thermal sources influencing temperature and humidity nearby the measurement setup.

Figure 2. The AFM device at LNE is located inside a glove box for maximum environmental control and stability.

Log humidity, temperature, type of atmosphere, and pressure in the laboratory or the glove box to allow tracing back implausible C-AFM measurements and check possible influences of one of these parameters.

2.2.2. "Out of the Lab" environment

In less restricted environmental conditions, generally met in Industry or Academia, accurate traceable measurements of resistance and current using C-AFM can still be performed. If the following conditions are satisfied:

$$HR = (40 \pm 10)\% \text{ and } T = (23 \pm 3)\ °C,$$

then the user will easily draw up a simplified uncertainty budget as explained later in this GPG. No temperature or relative humidity correction will need to be applied by the user on the measured values if these conditions are respected.

2.3. Calibration sample

For calibration of C-AFM for resistance and current measurements, a reference sample with SMD resistors of known resistance and enabling to cover a wide resistance range is required (Annexe1). This multiple resistance wide range reference sample will also allow one to cover a wide current range but in discrete (per decade) current values as function of the (per decade) resistance values and the applied bias voltage. Alternatively, another reference sample fitted with a JFET SMD diode characterized by a known I-V curve will enable to cover continuously a wide current range by providing any current values depending on the bias voltage (Annexe1). Such reference samples have been recently developed by LNE with two designs.

The first kind of samples (Type 1A) described in [2] covers the resistance range 100 Ω to 100 GΩ while the second type (Type 4A) enables to cover the resistance range 1 kΩ up to 1 TΩ and a current range from 10 fA to 1 µA (Figure 3).

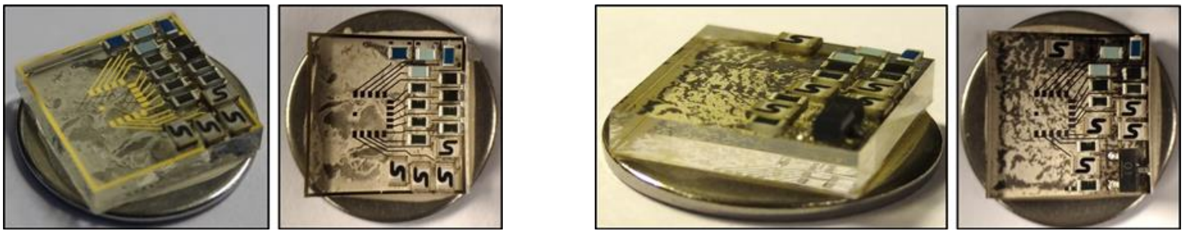


Figure 3. LNE reference samples: Type 1A (left) and Type 4A (right)

It is worth noticing that the resistance and current values of the LNE reference standards are determined using calibrated equipment composed of a probe station connected to a programmable voltage source and a high-precision ammeter. For this goal, the rectangular gold pads are used as terminals to calibrate the corresponding resistance and current values relative to the back electrodes, which include the resistance of the SMD resistors and the metallic (Au or Pt) connection lines.

2.4. Sample preparation method

Ideally, the sample under study (SUT) should be located close to the reference sample and both samples are glued on insulating holder (*i.e.*, not electrically connected to ground). This holder is fitted with a back electrode electrically connected to a bias voltage source. If the system does not allow the 2 samples to be placed next to each other, the SUT should be positioned at the place of the reference sample once the C-AFM calibration is complete. The SUT will be then removed once the SUT measurements are performed and replaced by the reference sample for calibration checking. If the SUT is of small size, *i.e.*, an area smaller than around 30 mm^2, then it can be positioned and glued on the free area available on the reference sample proposed in [2,3].

3. Measurement Procedure

3.1. Calibration of C-AFM

3.1.1. Calibration of C-AFM in terms of resistance

The calibration protocol presented here is applied to C-AFM operating as well in spectroscopic (I-V) mode as in image mode and consists in 5 steps (Table 1). The first two steps are common two both modes. The first step aims to eliminate any tip contamination by repeatedly scanning over a fixed line (typically a few tens of nanometres) on the sample surface (i.e., by disabling the slow scan axis). The effective contamination removal was associated with a stable measurement of a minimal resistance value [2,3].

As second step, the AFM tip is positioned at a fixed location in contact with the electrode surface with a constant applied force, typically 900 nN.

The third step corresponds to the measurement itself. For spectroscopic mode, I–V curves are extracted by sweeping the applied voltage between, typically −1 V and +1 V while for image mode, a surface of 60 µm × 60 µm is scanned with 512 × 512 pixels covering all the resistive electrodes by applying a constant bias voltage, typically V_{in} = +1 V. Then, the scan is repeated with the reversed polarity of the bias voltage, i.e. V_{in} = -1 V. The use of the two polarities (*i.e.* ± 1V), is particularly recommended when the C-AFM instrument is equipped with a highly doped diamond coated tip.

In the fourth step, the measured resistance values R_{meas} are determined, either from I-V slope by applying a regression model on the data in the spectroscopic mode, or in the image mode, by extracting a region of each electrode from the resistance map, then by performing a histogram and fitting data to Gaussian distribution. The type A uncertainty corresponding to found resistance values is given by the standard deviation of the data fits.

The last step, common to both modes, consists in comparing R_{meas} values against the calibrated resistance values R_{cal} and determining the correction factor to apply to C-AFM reading system. Optionally, the process can be repeated to check the agreement with R_{cal} values.

Table 1. C-AFM calibration procedure for resistance measurements

Step	Image mode	Spectroscopic mode
1	Eliminating tip contamination (continuous fixed-line scanning)	
2	Positioning tip	
3	Scanning a surface (60 µm x 60 µm), at two voltage polarities	Collecting I-V curves for each electrode
4	From resistance map, extracting the region of each electrode, calculating resistance from histogram	Finding resistance values from I-V slope
5	Determining the correction factor from deviation $\Delta R = (R_{meas} - R_{cal})$	

3.1.2. Calibration of C-AFM in terms of current

There are two approaches to calibrate C-AFM for current measurements as well in spectroscopic mode as image mode.

- **SMD resistors**

The first way involves the use of a multi-resistance reference sample (for example the Type 1A or Type 4A sample provided by LNE) and as above by scanning the AFM tip successively on each resistive electrodes. Therefore, the current range will be covered over 9 or 10 decades but in discrete values (for example, applying V_B = 1 V on resistive electrodes which have resistances of 1 TΩ, 1 GΩ, and 1 MΩ, the delivered current values will be: 1 pA, 1 nA, and 1 µA).

The calibration procedure described above (Table 1) remains unchanged for C-AFM operating in image mode, except for the fourth step dealing with DC current map and the last step which compares measured current values, I_{meas}, against calibrated current values I_{cal} (Table 2).

For the spectroscopic mode, only the third step and fourth step are changed. For each electrode, the current measurement consists in periodically reversing the polarity of the applied voltage V_{in}, for example V_{in}^{-} = -1V, then V_{in}^{+} = +1 V giving rise to two current values I^- and I^+ then repeating this cycle several times, typically 10 times. In the fourth step, the mean current value is extracted by averaging the n data $\langle I_j \rangle = (I_j^+ - I_j^-)/2$ with j varying from 1 to n and calculating the corresponding standard deviation.

Table 2. C-AFM calibration procedure for current measurements with SMD resistors

Step	Image mode	Spectroscopic mode
1	Eliminating tip contamination (continuous fixed-line scanning)	
2	Positioning tip	
3	Scanning a surface (60 µm x 60 µm), at two voltage polarities	Collecting I-V curves $<I_j>$ by switching successively voltage polarities along *n* cycles.
4	From DC current map, extracting the region of each electrode, calculating current from histogram	Finding mean current values from averaging the *n* data $<I_j>$.
5	Determining the correction factor from deviation $\Delta I = (I_{meas} - I_{cal})$	

- **SMD JFET diode**

The second way to calibrate the C-AFM in terms of current is to use a JFET SMD diode (available on the Type 4A reference sample). In spectroscopic mode, the AFM tip being in contact to the surface of the electrode electrically connected to the SMD diode, sweeping the voltage from 0.1 V up to 0.7 V enables to calibrate the C-AFM over continuous current values ranging from 10 fA up to 100 µA. In image mode, repeating a scan of a defined area of the electrode at different voltages permits to provide a calibration of C-AFM for any current value of interest in the same range (10 fA – 100 µA). It is worth mentioning here that possible voltage shift can occur with the use of highly doped diamond coated tip due to photoconductive effect and create significant error, much larger than 10% in relative value. It is then highly recommended either to determine the voltage shift by carrying out I-V curve on electrode connected to a SMD resistor, or to use metallic tip (PtSi, CrPt …).

The Table 3 gives the scheme of the calibration procedure, with first two steps unchanged. In the third step, for the spectroscopic mode, the bias voltage is swept from 0.1 V up to 0.7 V by constant voltage increments, which provide the current values desired by the user. Care has to be taken on the voltage sweeping speed, which must be compatible with the bandwidth of the current amplifier used. Repeating at least three times the I-V curves is recommended to estimate the type A uncertainty associated to the calibrated I-V curve (step 4). For the image mode, the third step consists in repeating the scanning the AFM tip on a defined area of the electrode at different voltages. In the 4th step, the current value is calculated by performing an histogram and fitting data to Gaussian distribution. Then, the correction factor is determined from deviation $\Delta I = (I_{meas} - I_{cal})$ at each voltage values (5th step).

Table 3. C-AFM calibration procedure for current measurements with SMD diode

Step	Image mode	Spectroscopic mode
1	Eliminating tip contamination (continuous fixed-line scanning)	
2	Positioning tip	
3	Scanning the electrode surface on a defined area at different voltages	Collecting I-V curves.
4	From each DC current map, calculating current from histogram	Determining the mean I-V curve.
5	Determining the correction factor from $\Delta I = (I_{meas} - I_{cal})$ at a given voltage	

3.2. Simplified uncertainty budgets

3.2.1. C-AFM Calibration for resistance measurements

The robust and simplified uncertainty budget drawn-up below requires to fulfil three conditions for use in an industrial environment:

- **Environmental condition**

  HR = (40 ± 10)% and T = (23 ± 3) °C.

This condition allows the user to do avoid applying a correction on the measured values from temperature and relative humidity effects.

- **AFM tip**

The use of highly-doped diamond coated tips is recommended for covering almost all resistance ranges, from 100 kΩ up to 1 TΩ. This choice relies on the robustness and the widely use of such tips in contrast with metallic tips, which are more fragile. The drawback of this is an increased uncertainty for 100 kΩ (by a factor of 10).

However, for the smallest resistance values, *i.e.* ranging from 1 kΩ to 10 kΩ, the user can consider the uncertainty value mentioned in the corresponding case only if a metallic tip is used. This is not valid for diamond tip (too resistive).

- **Spectroscopic mode versus image mode**

The use of I-V spectroscopic mode to calibrate the C-AFM is preferred, which is found more precise than the image mode and allows to cover the complete resistance range (100 Ω to 1 TΩ). The Type A uncertainty value is then calculated from the linear regression applied to the I-V straight line (between – 1V and + 1V).

The calibration of C-AFM in image mode remains possible and precise enough on the more limited resistance range, *i.e.* from 100 kΩ and 1 TΩ. The Type A uncertainty will be then given by the standard deviation associated to the Gaussian fit of the recorded histogram.

The simplified uncertainty budgets are given in Table 4 for the I-V mode and in Table 5 for the image mode. All uncertainties reported in this GPG are given at one standard deviation (k = 1) corresponding to a 68% confidence level in the case of a normal distribution [4].

Table 4. I-V mode

Uncertainty budget	1 kΩ -10 kΩ	100 kΩ	1 MΩ	(10-100) MΩ	1 GΩ	10 GΩ	100 GΩ	1 TΩ
Type A	< $3·10^{-3}$	$3.2·10^{-3}$	$1·10^{-4}$	< $3·10^{-4}$	$5·10^{-4}$	$1·10^{-4}$	$2·10^{-4}$	$1.2·10^{-3}$
Calibration	$6.9·10^{-4}$	$6.9·10^{-4}$	$6.9·10^{-4}$	$6.9·10^{-4}$	$1.5·10^{-3}$	$1.5·10^{-3}$	$1.9·10^{-3}$	$2.3·10^{-3}$
Environment	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1·10^{-2}$
Tip resistance	$1.0·10^{-2}$	$1.0·10^{-2}$	$1.0·10^{-3}$	≤ $1·10^{-4}$	< $1·10^{-4}$	< $1·10^{-4}$	< $1·10^{-4}$	< $1·10^{-4}$
Total uncertainty	**$1.1·10^{-2}$ (*)**	**$1.1·10^{-2}$**	**$2.3·10^{-3}$**	**$2.0·10^{-3}$**	**$2.5·10^{-3}$**	**$2.4·10^{-3}$**	**$2.7·10^{-3}$**	**$1.0·10^{-2}$**

(*) C-AFM fitted with metallic tip only

Table 5. Image mode

Uncertainty budget	100 kΩ	1 MΩ	10 MΩ	100 MΩ	1 GΩ	10 GΩ	100 GΩ	1 TΩ
Type A	$3.5·10^{-2}$	$6.1·10^{-3}$	$4.0·10^{-3}$	$5.1·10^{-3}$	$3.9·10^{-3}$	$4.9·10^{-3}$	$7.2·10^{-3}$	$2.9·10^{-2}$
Calibration	$6.9·10^{-4}$	$6.9·10^{-4}$	$6.9·10^{-4}$	$6.9·10^{-4}$	$1.5·10^{-3}$	$1.5·10^{-3}$	$1.9·10^{-3}$	$2.3·10^{-3}$
Environment	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1.9·10^{-3}$	$1·10^{-2}$
Tip resistance	$1.0·10^{-2}$	$1.0·10^{-3}$	$1·10^{-4}$	< $1·10^{-4}$	< $1·10^{-4}$	< $1·10^{-4}$	< $1·10^{-4}$	< $1·10^{-4}$
Total uncertainty	**$3.6·10^{-2}$**	**$6.5·10^{-3}$**	**$4.4·10^{-3}$**	**$5.5·10^{-3}$**	**$4.6·10^{-3}$**	**$5.5·10^{-3}$**	**$7.7·10^{-3}$**	**$3.1·10^{-2}$**

3.2.2. C-AFM Calibration for current measurements

As previously mentioned, the C-AFM can be calibrated in terms of current along two approaches, the first using SMD resistors of the multi resistance reference sample (for example Type 1A or Type 4A LNE sample) and biased by the voltage source, the second one involving the JFET diode from Type 4A reference sample (also biased by the voltage source). Here, the simplified uncertainty budgets are only established for C-AFM operating in spectroscopic mode.

The Table 6 details the simplified uncertainty budget for the first approach. It relies on the total Type B uncertainty related to the calibration of reference sample and the same uncertainties are considered for environment and tip resistance given in Table 4. From experience, the Type A uncertainty is assumed not exceeding $5{\cdot}10^{-4}$ (in relative value) for $I \geq 1$ pA and in the order of $1{\cdot}10^{-3}$ for 1 fA< I < 1 pA.

Table 6

Uncertainty budget	1 fA	10 fA	100 fA	1 pA	10 pA	100 pA	1 nA to 1 µA
Type A uncertainty	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$
Calibration	$2.5{\cdot}10^{-3}$	$2.3{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.5{\cdot}10^{-3}$	$1.5{\cdot}10^{-3}$	$6.9{\cdot}10^{-4}$
Environment	$1{\cdot}10^{-2}$	$1{\cdot}10^{-2}$	$1{\cdot}10^{-2}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$
Tip resistance	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$1{\cdot}10^{-4}$	$1{\cdot}10^{-3}$
Total uncertainty	**$1{\cdot}10^{-2}$**	**$1{\cdot}10^{-2}$**	**$1{\cdot}10^{-2}$**	**$2.7{\cdot}10^{-3}$**	**$2.5{\cdot}10^{-3}$**	**$2.5{\cdot}10^{-3}$**	**$2.3{\cdot}10^{-3}$**

Table 7 gives the simplified budget uncertainty when the JFET diode source is used as the reference current standard. The calibration uncertainties change and estimated at the level of 1.7% as simplified uncertainty. Using the same environmental conditions, particularly for the temperature, *i.e.* T = (23 ± 3) °C, the uncertainties related to environment are significantly increased, varying from 3.2% to 14.6% for current ranging from 10 fA up to 1 µA respectively and dominate all the other uncertainty contributions. To guarantee a total uncertainty much lower than 10%, it is recommended for the user to keep the room temperature at T = (23 ± 1) °C or to correct the measured current if the temperature is outside this range. The table 7 reports the simplified uncertainty budget for T = (23 ± 1) °C.

Table 7

Uncertainty budget	10 fA	100 fA	1 pA	10 pA	100 pA	1 nA	10 nA	100 nA	1 µA
Type A	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$	$5{\cdot}10^{-4}$
Calibration	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$	$1.7{\cdot}10^{-2}$
Environment T = (23 ± 1) °C	$1.1{\cdot}10^{-2}$	$1.6{\cdot}10^{-2}$	$2.1{\cdot}10^{-2}$	$2.5{\cdot}10^{-2}$	$3.0{\cdot}10^{-2}$	$3.5{\cdot}10^{-2}$	$3.9{\cdot}10^{-2}$	$4.4{\cdot}10^{-2}$	$4.9{\cdot}10^{-2}$
Tip resistance	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$1{\cdot}10^{-4}$	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$
Total uncertainty	**$2.0{\cdot}10^{-2}$**	**$2.3{\cdot}10^{-2}$**	**$2.7{\cdot}10^{-2}$**	**$3.0{\cdot}10^{-2}$**	**$3.5{\cdot}10^{-2}$**	**$3.9{\cdot}10^{-2}$**	**$4.3{\cdot}10^{-2}$**	**$4.7{\cdot}10^{-2}$**	**$5.2{\cdot}10^{-2}$**

4. Calibration results

Results are given here for two different measurement conditions. The first one concerns the calibration of C-AFM at DFM, *i.e.* in NMI environment, the C-AFM operating in spectroscopic mode for resistance measurements. The second example deals with results obtained with C-AFM operating in CNRS-GeePs, *i.e.* in "out of the lab" environment, with the C-AFM operating in image mode for current measurements. Other examples can be found from comprehensive collaborative works between CNRS and LNE reported in [2,3].

4.1. Calibration of C-AFM for resistance measurements

The C-AFM set-up of DFM has been calibrated by applying the procedure described in part 3.1.1 (Table 1). The LNE reference sample B04-03 (Type 1A) has been used for four resistance values: 100 MΩ, 500 MΩ, 1 GΩ and 10 GΩ.

As shown in Figure 4.1a, the observed relative deviations are found significant in respect with the total combined uncertainty (Table 1). It can be noted as a positive finding that this deviation is constant, mainly due to the amplifier gain used (10^9 V/A) which can be then easily corrected.

The total combined uncertainties on these four values are calculated from the simplified uncertainty budget in Table 8, and considering the Type A uncertainties corresponding to the DFM measurements.

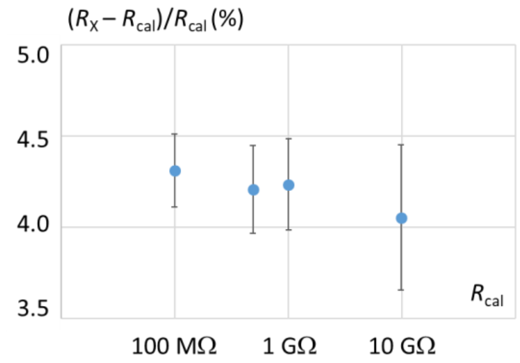


Figure 4.1a Relative deviations between resistance values measured with C-AFM at DFM and calibrated values from reference sample B04-03 (Type 1A). The error bars represent the total combined uncertainty.

Table 8

Uncertainty budget	100 MΩ	1 GΩ	10 GΩ	100 GΩ
Type A	$1.5{\cdot}10^{-4}$	$3{\cdot}10^{-4}$	$4{\cdot}10^{-4}$	$3{\cdot}10^{-3}$
Calibration	$6.9{\cdot}10^{-4}$	$1.5{\cdot}10^{-3}$	$1.5{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$
Environment	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$
Tip resistance	$\leq 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$
Total uncertainty	**$2.0{\cdot}10^{-3}$**	**$2.4{\cdot}10^{-3}$**	**$2.5{\cdot}10^{-3}$**	**$4.0{\cdot}10^{-3}$**

4.2. Calibration of C-AFM for current measurements

The C-AFM set-up at CNRS-GeePs has been calibrated following the calibration procedure given in part 4.1.2. (Table 2). Bias voltages at two polarities + 1 V and – 1 V have been applied to the SMD resistors of the LNE reference sample B01-Au1 (Type 4A) so that the delivered calibrated current values ranges from 1 pA up to 1 µA. For this calibration, the C-AFM was fitted with highly doped diamond coated tip.

The Figure 4.2A shows that the current values measured by the C-AFM agree very well with the reference values provided by the calibration sample, *i.e.*, within ± 1%, except at 1 pA for which a significant deviation of – 4.3% is observed.

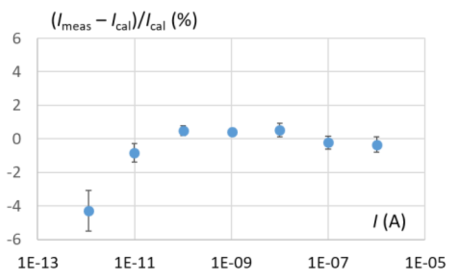


Figure 4.2a Relative deviations between current values measured with C-AFM at CNRS and calibrated values from reference sample B01-Au1 (Type 4A). The error bars represent the total combined uncertainty.

Table 9 below lists the components of the simplified uncertainty budget associated to this calibration. All combined standard uncertainties are between 0.3% and 0.6% except at 1 pA for which the total uncertainty reaches 1.2%.

Table 9

Uncertainty budget	1 pA	10 pA	100 pA	1 nA	10 nA	100 nA	1 µA
Type A uncertainty	$1.2{\cdot}10^{-2}$	$5.1{\cdot}10^{-3}$	$1.4{\cdot}10^{-3}$	$1.0{\cdot}10^{-3}$	$3.2{\cdot}10^{-3}$	$3.0{\cdot}10^{-3}$	$4.1{\cdot}10^{-3}$
Calibration	$1.9{\cdot}10^{-3}$	$1.5{\cdot}10^{-3}$	$1.5{\cdot}10^{-3}$	$6.9{\cdot}10^{-4}$	$6.9{\cdot}10^{-4}$	$6.9{\cdot}10^{-4}$	$6.9{\cdot}10^{-4}$
Environment	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$	$1.9{\cdot}10^{-3}$
Tip resistance	$< 1{\cdot}10^{-4}$	$< 1{\cdot}10^{-4}$	$1{\cdot}10^{-4}$	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$	$1{\cdot}10^{-3}$
Total uncertainty	$\mathbf{1.2{\cdot}10^{-2}}$	$\mathbf{5.7{\cdot}10^{-3}}$	$\mathbf{2.9{\cdot}10^{-3}}$	$\mathbf{2.5{\cdot}10^{-3}}$	$\mathbf{3.9{\cdot}10^{-3}}$	$\mathbf{3.8{\cdot}10^{-3}}$	$\mathbf{4.7{\cdot}10^{-3}}$

5. Annex 1 - Reference sample

5.1. Surface Mount Device (SMD) resistors

SMD resistors are used in multi-resistance reference samples (also referred to as array resistance standard). Each resistor in the array can be individually biased by a voltage source to produce a stable current reference over a wide range. This method allows for precise calibration across the entire measurement range from fA to µA. There are several advantages of SMD resistors for calibration of resistance:

Wide Range of Values: SMD resistor arrays can cover a broad range of resistances from 100 Ω to 1 TΩ. Each resistor can be individually biased by a voltage source, providing stable and precise current reference across the entire measurement range of C-AFMs, often decided by the range of their pre-amplifier. A wide range is essential for comprehensive calibration, ensuring that the C-AFM can accurately measure currents at both low and high ends of the spectrum.

Stability and Precision: SMD resistors are known for their high stability and precision. They are manufactured with tight tolerance specifications, ensuring that their resistance values remain consistent over time and under varying environmental conditions. This stability is crucial for reliable calibration.

Compact and Robust Design: The compact nature of SMD resistors allows for the integration of multiple resistors into a single reference sample. This design not only saves space but also enhances the robustness of the calibration setup, making it more convenient and durable for repeated use.

Ease of integration: SMD resistor arrays can be easily integrated into standard electronic circuits. Their compatibility with existing measurement systems simplifies the calibration process, allowing for straightforward connection to the voltage source and measurement equipment.

5.2. Junction Field Effect Transistor (JFET) SMD diode

The JFET diode source is a highly effective method for generating continuous range of current values, particularly useful for lower currents ranging from 10 fA to 100 µA. There are several advantages of JFET diode source for calibration of current:

Wide and Continuous Range: The JFET diode source can produce a continuous range of current values by sweeping the voltage across the diode. This capability is particularly valuable for calibrating instruments that need to measure very low currents, as it covers a range from 10 fA to 100 µA. This wide range ensures comprehensive calibration across the entire measurement spectrum of the C-AFM, given that a suitable preamplifier is supplied.

Stability and Precision: The behaviour of JFET diodes under varying voltages is well-characterized and highly predictable provided that its temperature dependence is taken into account. This makes them ideal for generating table and repeatable current references. The predictable I-V (current-voltage) characteristics allow for precise control over the generated current.

Low Noise and High Sensitivity: Proper JFET diodes are known for their low noise characteristics, which is critical when measuring very small currents. The high sensitivity of these diodes ensures that even minute changes in current can be accurately detected and measured.

Compact and Robust Design: JFET diodes are small and robust, making them suitable for integration into compact reference samples. Their durability ensures consistent performance over long periods, which is essential for reliable calibration.

5.3. Ensuring traceability to the SI

The resistance and current values provided by the SMD resistors and JFET diode source used in the reference sample are initially calibrated. To do this, LNE uses a 4-probe station coupled to a programmable voltage source and a high-precision ammeter, all the set-up being in an electromagnetically shielded environment under stabilized air temperature (typically 23 ± 0.1) °C and relative humidity (typically 40 ± 0.3)%. Two different calibrated ammeters are used depending on the resistance and current ranges: a digital voltage multimeter (DVM) or a very low noise (≈ $fA/Hz^{1/2}$) current amplifier associated with the same DVM. All these instruments are calibrated at LNE using their own primary standards ensuring traceability to the International System of Units (SI).

6. Annex 2 - Imaging and Measurement Artefacts in C-AFM

Although C-AFM is a versatile tool for nanoscale electrical characterization, it is susceptible to various imaging and measurement artefacts. These artefacts can compromise data accuracy and reliability. Understanding their origins and implementing strategies to mitigate them is crucial for obtaining precise measurements. Here are common artefacts in C-AFM and methods to avoid them.

6.1. Tip-Related Artefacts

6.1.1. Tip Contamination and Wear

Artefact Description: Over time, the AFM tip can become contaminated with residues from the sample or environment, or it may wear down, altering its shape and conductive properties. This can lead to inconsistent contact and inaccurate current measurements.

Avoidance Strategies:

- Regular Cleaning: Clean the tip regularly using appropriate solvents or plasma cleaning techniques.
- Tip Replacement: Frequently inspect and replace tips to ensure they remain sharp and conductive.
- Use of Robust Tips: Utilize highly doped diamond or platinum-coated tips, which are more resistant to wear and contamination.

6.1.2. Tip-Sample Contact Issues

Artefact Description: Poor contact between the tip and the sample can result in fluctuating or unstable current measurements. This can occur due to improper tip engagement or sample surface roughness.

Avoidance Strategies:

- Optimize Contact Mode Settings: Adjust the force setpoint and feedback parameters to ensure stable tip-sample contact.
- Surface Preparation: Smooth the sample surface through polishing or coating to minimize roughness.
- Use of Conductive Adhesives: Employ conductive adhesives or conductive mounting techniques to enhance contact stability.

6.2. Electrical Artefacts

6.2.1. Leakage currents and parasitic capacitance

Artefact Description: Leakage currents and parasitic capacitance can introduce noise and erroneous readings, particularly in low-current measurements. These artefacts arise from the measurement circuit and surrounding environment.

Avoidance Strategies:

- Shielding and Grounding: Properly shield and ground the AFM system to minimize electrical interference.
- Use of Low-Noise Electronics: Utilize low-noise preamplifiers and high-quality cables to reduce electrical noise.
- Environmental Control: Maintain a stable and low-humidity environment to reduce leakage currents.

6.2.2. Tip Resistance

Artefact Description: The resistance of the AFM tip itself can affect the accuracy of current measurements, especially at higher currents. This resistance can add to the measured resistance, leading to overestimated values.

Avoidance Strategies:

- Tip Calibration: Calibrate the tip resistance separately and subtract it from the measured values.
- High-Conductivity Tips: Use tips with low intrinsic resistance, such as those made from highly doped diamond or metal coatings.

6.3. Sample-Related Artefacts

6.3.1. Surface Contaminants

Artefact Description: Contaminants on the sample surface, such as adsorbed water or organic residues, can alter the electrical properties and affect measurements.

Avoidance Strategies:

- Sample Cleaning: Clean the sample thoroughly before measurements using solvents, UV-ozone treatment, or plasma cleaning.
- Environmental Control: Conduct measurements in a controlled environment, such as a dry nitrogen atmosphere, to prevent recontamination.

6.3.2. Non-Uniform Sample Properties

Artefact Description: Variations in the sample's material properties, such as inhomogeneities or defects, can lead to inconsistent measurements across different regions.

Avoidance Strategies:

- Sample Characterization: Perform thorough initial characterization of the sample to identify and account for inhomogeneities.
- Localized Calibration: Calibrate the AFM system using reference materials that match the sample's properties as closely as possible.

6.4. Measurement-Related Artefacts

6.4.1. Thermal Drift

Artefact Description: Changes in temperature can cause thermal expansion or contraction of the AFM components and the sample, leading to drift in the measurement.

Avoidance Strategies:

- Temperature Control: Maintain a stable temperature in the AFM environment using thermal enclosures or active temperature control systems.
- Wait Time: Allow the system to equilibrate to the ambient temperature before starting measurements.

6.4.2. Hysteresis and Creep

Artefact Description: Mechanical hysteresis and creep in the AFM piezoelectric scanner can lead to distortions in the recorded images and measurements over time.

Avoidance Strategies:

- Scanner Calibration: Regularly calibrate the piezoelectric scanner to correct for hysteresis and creep.
- Slow Scan Rates: Use slower scan rates to minimize the effects of mechanical creep.

6.5. Data Processing Artefacts

6.5.1. Signal Noise and Interference

Artefact Description: Electrical noise and signal interference can introduce artefacts into the measurement data, obscuring true signals.

Avoidance Strategies:

- Signal Filtering: Apply signal filtering techniques to remove high-frequency noise from the data.
- Averaging Multiple Scans: Perform multiple scans and average the results to reduce random noise.

6.5.2. Image Processing Errors

Artefact Description: Processing steps such as image flattening, filtering, and alignment can introduce artefacts if not performed correctly.

Avoidance Strategies:

- Careful Image Processing: Use advanced image processing software and carefully verify each step to avoid introducing artefacts.
- Manual Review: Manually review processed images to identify and correct potential artefacts.

By understanding and addressing these potential artefacts, users can significantly improve the accuracy and reliability of C-AFM measurements.

References

[1] https://projects.lne.eu/jrp-elena/

20IND12 ELENA project, founded by the European Metrology program for Innovation and Research (EMPIR) for the period from Sept 2021 to Aug 2024

[2] F. Piquemal, K. Kaja, P. Chrétien, J. Morán-Meza, F. Houzé, C. Ulysse and A. Harouri, *Beilstein J. Nanotechnol.*, 14, 1141–8, 2023

[3] F. Piquemal, P. Chrétien, J. Morán-Meza, F. Houzé, C. Ulysse and A. Harouri, *Physica Status Solidi A, Jan. 2025.* http://doi.org/10.1002/pssa.202400711

Physica Status Solidi A, Volume 222, Issue 14: Special Issue: ALTECH 2024 – Analytical Techniques for Precise Characterization of Nano Materials, July 2025

[4] Joint Committee for Guides in Metrology - JCGM. Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement. JCGM 100 2008.

https://www.bipm.org/en/publications/guides/gum.html