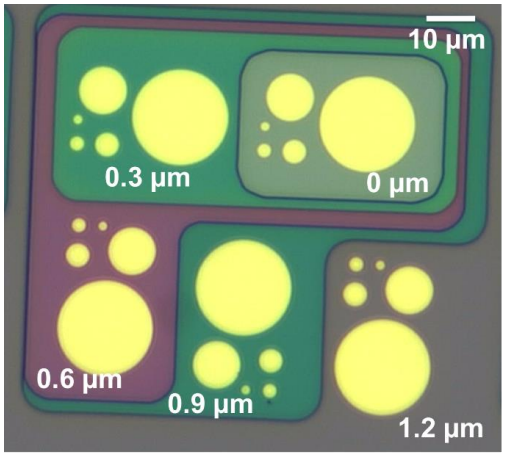
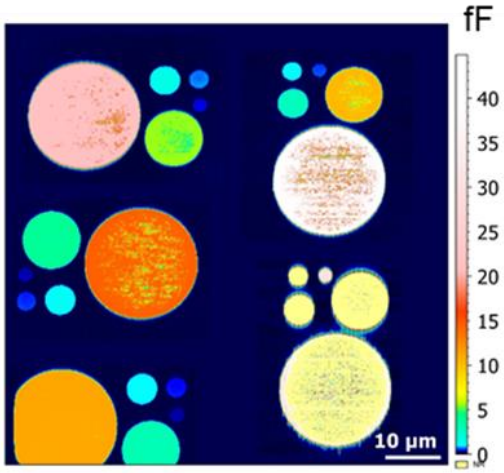
ELENA
Electrical nanoscale metrology in industry

Good practice guide for calibrated admittance measurements using scanning microwave microscopy





Abstract

The measurement of electrical properties at the nanoscale allows evaluating the performance of nanomaterials developed for consumer electronics, innovative quantum technologies, IoT applications and life science research. 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, METAS and LNE with support from JKU, PTB and ULILLE, jointly propose a good practice guide (GPG) on the calibration of SMM for measurement at the nanoscale, covering HF admittance from 100 nS to 100 mS, for use in industrial applications for measurement at the nanoscale. This GPG relies on robust calibration methods, reference samples and simplified uncertainty budgets.

Content

1. Introduction

The scanning microwave microscopy (SMM) is a powerful nanoscale technique to measure the admittance of dielectric thin films or nanocapacitors. SMM thus allows the measurement of the nanoscale capacitance and conductance giving access to the dielectric constant, loss angle tangent and dopant concentration of materials.

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 SMM while drawing up simplified uncertainty budgets, all of these for use in industrial applications.

This good practice guide (GPG) summarizes the use of SMM as a relevant technique for carrying out nanoscale admittance (capacitance and conductance) measurements with a demonstrated traceability to the international system of units (SI). This GPG was established by METAS and LNE, with the support of JKU, PTB and ULILLE.

The calibration of SMM for admittance measurements relies on the use of a calibration kit such as developed by MC2 Technologies [1], [2] or METAS [3][4]. The MC2 technologies calibration kit is composed of MOS capacitors ranging from 0.3 fF to 9.8 fF while the standards proposed by METAS shows a coplanar capacitor structure on a trilayer membrane (SiN/SiO_2/SiN) with capacitance ranging from 1.3 fF to 10.1 fF.

In the following sections, we give a general description of the experimental set-up (SMM principle, environmental conditions, reference samples) detail the calibration methods developed for nanoscale admittance measurements using SMM, describe the software used to calculate uncertainty and present calibration results on SMM systems with simplified uncertainty budgets.

2. Experimental section

2.1. SMM measurement principle

SMM consists of a scanning probe microscope (SPM) interfaced with a vector network analyzer (VNA). The SPM includes an atomic force microscope (AFM) (in both NMIs) or a scanning tunneling microscope. Basically, the conductive tip of the SMM is connected to the microwave source/meter of VNA (see Fig.1) via a impedance matching network.

While the tip scans over the sample surface, it irradiates the microwave signal, highly localized at the apex, over a local region of the sample, allowing simultaneous topographic and electrical characterization of the sample under study.

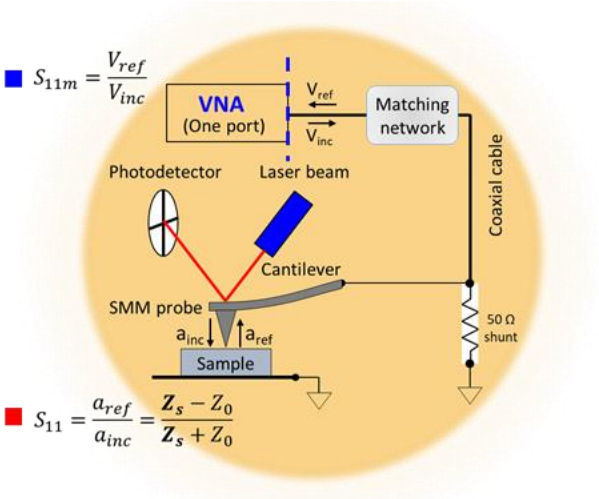


Figure 1. Schematic diagram of the AFM-based SMM setup, showing the matching network between the VNA and the sample under study

Depending on the mismatch between the characteristic impedance (Z_0) and the local impedance of the tip−sample system (Z_s), one part of the incident microwave signal is reflected back travelling from the tip-sample contact point to the VNA and the other part is transmitted throughout the sample.

The ratio between the reflected and incident signals at the tip-sample interface is the so-called S_{11} scattering parameter. Nevertheless, the VNA measure the scattering parameter S_{11m} at the reference plane (vertical blue dashed line in Figure 1), which is affected by the "matching network". Following the one-port VNA calibration procedure [5], S_{11m} measurements recorded by VNA are converted into complex impedance values.

2.2. Environmental conditions

2.2.1. National Metrology Institute environments

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

- Ensure stable lab climate (temperature, humidity) to enable stable operation of the SMM 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. Glovebox at LNE, where the AFM part is located inside.

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 admittance measurements using SMM can still be performed. If the following conditions are satisfied:

$$\mathrm{RH} = (40 \pm 10)\ \%\ \text{and}\ T = (23 \pm 3)\ ^\circ\mathrm{C},$$

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

2.3.1. Vertical MOS capacitor

A first version of calibration kit fabricated by MC2 Technologies is used to calibrate the SMM. As shown in Fig.3, each 45×45 mm^2 substrate has 144 identical 140×140 µm^2 patterns. Each patterns has 4 identical sets of 48 microcapacitors. Each capacitor consists of a circular gold electrode (gold pad height = 281 nm) deposited on silicon dioxide with different thicknesses and a Si (100) substrate strongly doped with boron atoms (p-type) (Fig.4).

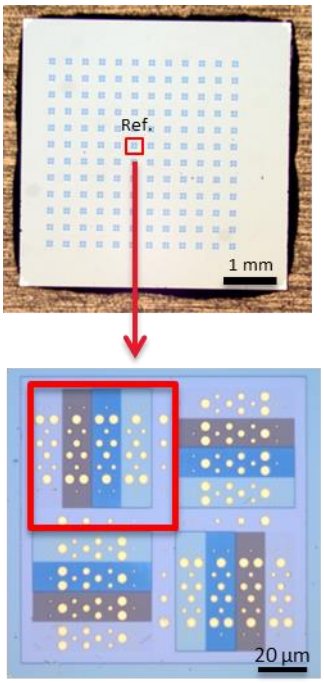


Figure 3. Top: a top view optical image of one of the used calibration sample. In this image, the pad , where the device set were used for the calibration, is labelled and marked. Below: a zoom in optical image of the same pad and the chosen set is framed in red square.

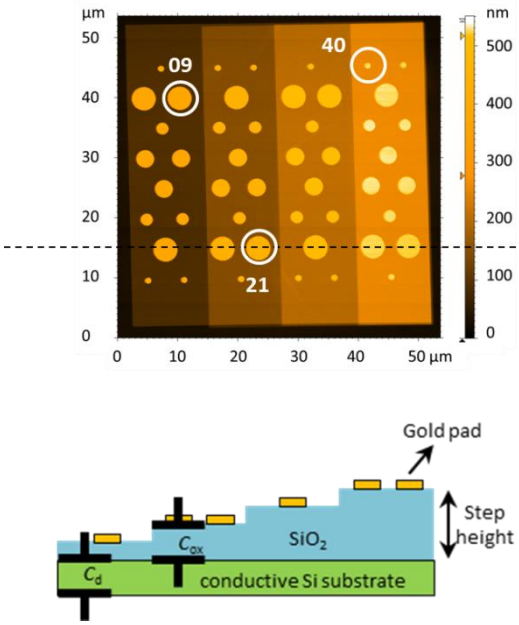


Figure 4. Top: AFM image of an area where all 48 microcapacitors stay. Below: cross section view taken along the dashed line shown in the top panel. The scale is not proportional.

The doping concentration N_a of the substrate was extracted from resistivity measurements (N_a = 8.2 × 10^{18} atoms/cm^3), this quite high value makes the parasitic depletion capacitance C_d negligible compared to its dielectric capacitance C_{ox}. Because of the variation of SiO_2 thickness (from 50 nm to 220 nm with about 50 nm steps) and the lateral size of the gold electrode (with diameters varying from 1 µm to 4 µm), capacitance values of fabricated microcapacitors vary from 300 aF to 10 fF. More details about the fabrication process of calibration sample are described elsewhere [1].

It must be noted here that the knowledge of the capacitance values results from calculation using the measured values of the dimensional parameters of the capacitors (thickness of the dielectric layer and the area of the top electrode). These parameters can be measured with certain uncertainty by AFM or scanning electron microscope (SEM) techniques. The capacitance calculations also depend on the

relative permittivity ε_r value of the silicon dioxide, usually set to 3.9 as nominal value [8][9]. These calculations, relying on an analytical approach or using a finite element modeling, consider the effects of fringing fields.

The uncertainty associated with this calibration structure was reported in [10]. These are composed of the uncertainties on dimensional measurement of the MOS capacitors (top electrode areas and dielectric layer thickness), those on the dielectric constant of the SiO_2 layer and the uncertainties on the depletion capacitance. For clarity purpose, a typical uncertainty budget acquired on the MC2 calibration kit is presented in Tab. 1.

Table 1. Uncertainty budget for the calculation of the capacitance of the first version of MC2 calibration kit

Uncertainty budget (%)	Type	Top electrode radius = 2 µm SiO_2 layer thickness = 50 nm	Top electrode radius = 0.5 µm SiO_2 layer thickness = 200 nm
Area measurement	A, B	0.9	2.4
Thickness measurement	A, B	1.3	0.7
Permittivity ε_r (SiO_2)	B	1.0	1.0
Depletion capacitance	B	2.0	0.8
Capacitance values (fF)		9.472 ± 0.263	0.272 ± 0.009

Using this uncertainty budget, a second version of the capacitance calibration kit was proposed and manufactured (Fig. 5).

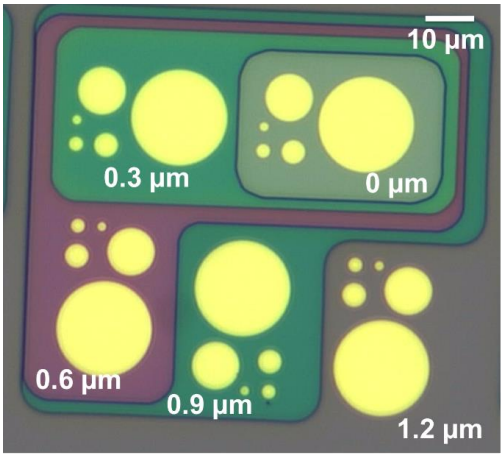


Figure 5. A top view optical image of one of the second version of the calibration structure.

It is composed of 400 identical patterns composed of 20 MOS capacitors ranging from 0.30 fF to 39.38 fF. It exhibits larger top electrodes and thicker SiO_2 layers to improve the uncertainty associated with dimensional measurement. The doping density of the substrate was also increased to reduce the uncertainty contribution of the depletion capacitance. The doping density amounts to $(2.00 \pm 0.04) \cdot 10^{20}$ atoms/cm^3. The uncertainty budget associated with the second version of the calibration kit is shown in Tab. 2.

Table 2. Uncertainty budget for the calculation of the capacitance of the second version of calibration kit.

Uncertainty budget (%)	Type	Top electrode radius = 10 µm SiO_2 layer thickness = 300 nm	Top electrode radius = 1 µm SiO_2 layer thickness = 1200 nm
Area measurement	A, B	0.16	1.53
Thickness measurement	A, B	0.60	0.44
Permittivity ε_r (SiO_2)	B	1	1
Depletion capacitance	B	0.06	0.04
Capacitance values (fF)		39.38 ± 0.47	0.30 ± 0.01

2.4. Sample preparation method

To reduce the impact of parasitic capacitances during SMM measurements, the sample to be measured should be located close to the capacitance calibration kit and the conductive back electrode of all sample should be connected to ground. In SMM systems where the vertical position of the SMM head is not maintained fixed, it is necessary to adjust the height of samples at a same vertical level, within ± 20 µm of difference, to guaranty the same reference plane (vertical blue dashed line, Fig. 1).

If the sample under study consists of a bare dielectric film, it is necessary the deposition of small gold electrodes on the sample surface. Thus, we respect the same vertical MOS capacitor configuration during SMM measurements.

A good electric contact must be established between the SMM tip and the top electrode of the MOS capacitor to enable a correct reading of the S_{11} parameter. It is typically done by applying a relatively strong contact force (≈ 2 µN). Several scan may be required to fully clean the area. It can be seen in Figure 6 where the same pattern of the second version of the MC2 calibration sample is scanned over multiple times with a strong applied force.

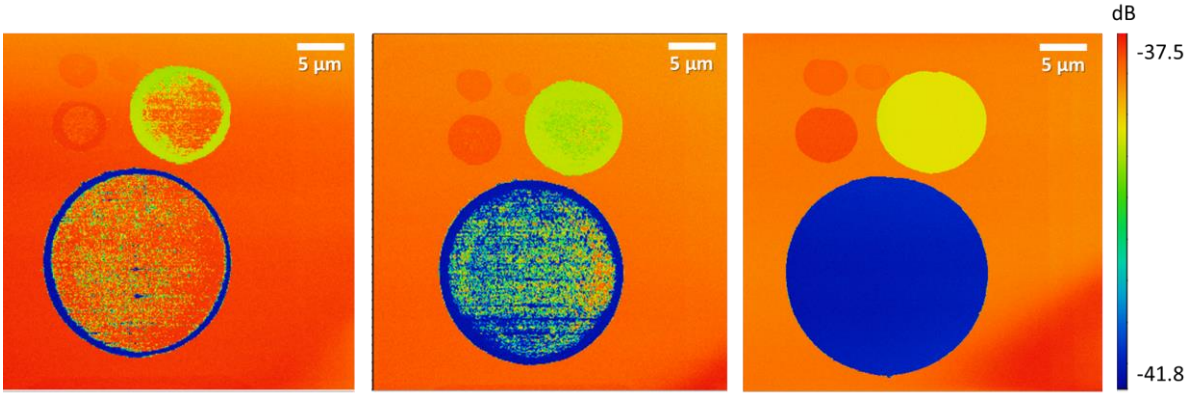


Figure 6. S_{11} amplitude contrast over the second version of the MC2 calibration sample acquired with an important tip-sample interface force (5 µN). left: first image; center: 5th image; right: 8th image

3. Measurement Procedure

3.1. Imaging conditions

Imaging conditions - SMM images are best acquired in contact mode, where the tip remains in contact with measured surface during the whole measurement process and ensures a homogenous electrical contact. Recorded signals consists of topography and electrical data ($S_{11,m}$-Magnitude and $S_{11,m}$-Phase).

Choice of the VNA operating frequency - Before landing the SMM probe on the sample surface, a full frequency sweep of the VNA signal is performed over its operation frequency range with a tip hovering above the sample surface but not in contact yet. The microwave frequency with the lowest $S_{11,m}$ - Magnitude signal is chosen for resonance based impedance matching systems.

Image processing – During SMM scanning, artifacts commonly seen in AFM imaging like tilt and scan line artifacts are present. Those can be corrected by the following algorithm on

- *First order polynomial background removal (surface features masked)*
- *Mean background substraction*
- *Polynomial align row algorithm applied to the SiO_2 surface (Top electrodes masked)*

In addition to this "classical" AFM data treatment, if an unshielded SMM probe is used, it is required to deal with the parasitic capacitance between the AFM cone and sample and the AFM cantilever and sample. Pratically, it is done by mean background substraction and zero leveling of each SiO_2 terraces.

3.2. Calibration of SMM

The calibration of SMM requires at least three capacitors with known capacitance values. A poor and arbitrary selection of these three capacitors without established criteria can lead to obtain erroneous results on capacitance measurements after SMM calibration. The capacitors of the reference device are selected according to the following criteria:

- they present a clean surface confirmed by AFM,
- they ensure a good and homogeneous electrical contact between the SMM tip and their top electrode,
- they cover the full desired calibration range,
- their capacitances satisfy approximately the inequality $|C_i - C_j| \geq \Delta C_{max}/2$,

where i and j (≠ i) ranging from 1 to 48 and ΔC_{max} is the difference between the highest and lowest capacitance values $\Delta C_{max} = C_{max} - C_{min}$. Depending on the capacitance value of the device under test (DUT), it is important to choose a triplet of capacitors, the range of which covers the expected value of the DUT. For example, for the DUT with a capacitance around 0.8 fF, a triplet with capacitances around 0.3 fF, 1 fF and 10 fF would be more suitable than the one with capacitances around 1 fF, 5 fF and 10 fF.

The calibration of the SMM consists in converting the raw measured reflection coefficient $S_{11,m}$ into the complex impedance of the sample under study Z_s using the modified Short Open Load (SOL) calibration method proposed by Hoffmann *et al.* [5]. The quantities $S_{11,m}$ and Z_s are related by two equations:

$$S_{11} = \frac{Z_s - Z_r}{Z_s + Z_r} \quad (1)$$

$$S_{11,m} = e_{00} + e_{01}\left(\frac{S_{11}}{1 - e_{11}S_{11}}\right) \quad (2)$$

where S_{11} is the expected reflection coefficient and e_{00}, e_{01}, and e_{11} are three complex parameters (also known as error parameters) to be determined from $S_{11,m}$ measurements on three reference structures with known capacitance values. Z_r is a non-zero reference impedance, which is typically set at 50 Ω. It is important to note that this calibration requires all measurements (for reference and DUT devices) to be performed with the same tip and at the same RF frequency. The whole calibration process needs to be repeated to calibrate the SMM measurement at other frequencies.

3.3. Simplified uncertainty budgets

The combined standard uncertainties result from the main uncertainty contributions that are summarized in Tab.3 for the first version of MC2 reference sample. As expected, the largest contribution comes from the SMM calibration uncertainty. Details are given below. No change of the error parameters (e_{00}, e_{01}, and e_{11}) has been detected from the measurements carried out on the reference pattern. The repeatability levels observed on the measurements of $C_{ref\text{-}09}$, $C_{ref\text{-}21}$, and $C_{ref\text{-}40}$ have been found between 0.2% and 1.1%. The uncertainty contribution from parasitic capacitances becomes non negligible for the smallest capacitance, reaching 0.7%.

Table 3. Main uncertainty contributions for 3 values measured on MC2 calibration kit – first version (A61 sample): C_{05} (8.79 fF), C_{23} (1.36 fF), C_{32} (0.33 fF).

Uncertainty budget for C_i (%)	Type	C_{09}	C_{21}	C_{40}
Histogram	A	0.1	0.6	2.1
Repeatability	A	0.5	1.1	0.2
SMM calibration	B	2.1	2.9	2.5
Parasitic capacitances	B	0.02	0.18	0.72
Water meniscus	B	0.2	0.2	0.2
Combined uncertainty u_{Cm}		2.2	3.2	3.3

The SMM calibration uncertainty is estimated from the uncertainties of capacitance calculation of the selected capacitors $C_{ref\text{-}09}$, $C_{ref\text{-}21}$, and $C_{ref\text{-}40}$ were taken from Tab.2.

Similarly, the uncertainty budget associated with capacitance measurement by SMM calibrated on the second version of the calibration sample is shown in Tab. 4.

Table 4. Main uncertainty contributions for 2 values measured on MC2 calibration kit – second version (B03 sample): C_{01} (39.37 fF), C_{16} (11.11 fF).

Uncertainty budget for C_i (%)	Type	C_{01}	C_{16}
Histogram	A	0.17	0.13
Repeatability	A	TBD	TBD
SMM calibration	B	0.68	064
Parasitic capacitances	B	0.01	0.03
Water meniscus	B	0.2	0.2
Combined uncertainty u_{Cm}		0.73	0.7

A non negligible improvement can be observed when compared to the calibration performed with the first version of the calibration sample.

3.3.1. SMM calibration for capacitance measurements

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

- **Environmental condition**

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

This condition allows the user to do not apply correction on the measured values from temperature and relative humidity effects.

- **SMM tip**

As mentioned before, if the SMM measurement is performed with an unshielded tip, a subtraction method should be used to deal with the parasitic capacitances. The performance of this treatment is improved when an AFM tip with a large form factor as the parasitic capacitances are less prone to change over location in this configuration.

3.3.2. Software for SMM calibration

The software developed by METAS dedicated to SMM Calibration can be used. The measurement model is based on the One-Port calibration algorithm well known from VNA metrology. The user will be guided through the main steps from S_{11m} raw data to corrected S_{11} data. As directed by the mSOL technique [5], the user selects at least three standard regions/patterns using masks. It is then necessary to assign the references standards to these masks. To compute the error terms, two sources of uncertainties can appear here. The one coming from the definition of the standards, the other from the measurement system itself. The mathematical propagation of the uncertainties is provided by the library UncLib [11], also developed at METAS. Once the S_{11} measurements are corrected, it is then

possible to display various information, such as the equivalent impedance, resistance, and capacitance, permittivity or dopant density. Results are displayed in the form of map scans of the corrected values and associated uncertainties.

4. Calibration results

The calibrated SMM was used to perform electric properties measurement on various samples.

4.1. Calibration of SMM for capacitance measurements in NMI

Two versions of the calibration structures were measured by the SMM. An SMM image was acquired over the first version (A64) of the calibration sample to perform the calibration, then on the second version (B03) which serves as test sample, and finally back on the A64 sample to check the instrument calibration. In figure 7, the average relative deviation between the computed and measured capacitance of the test sample using the calibrated SMM.

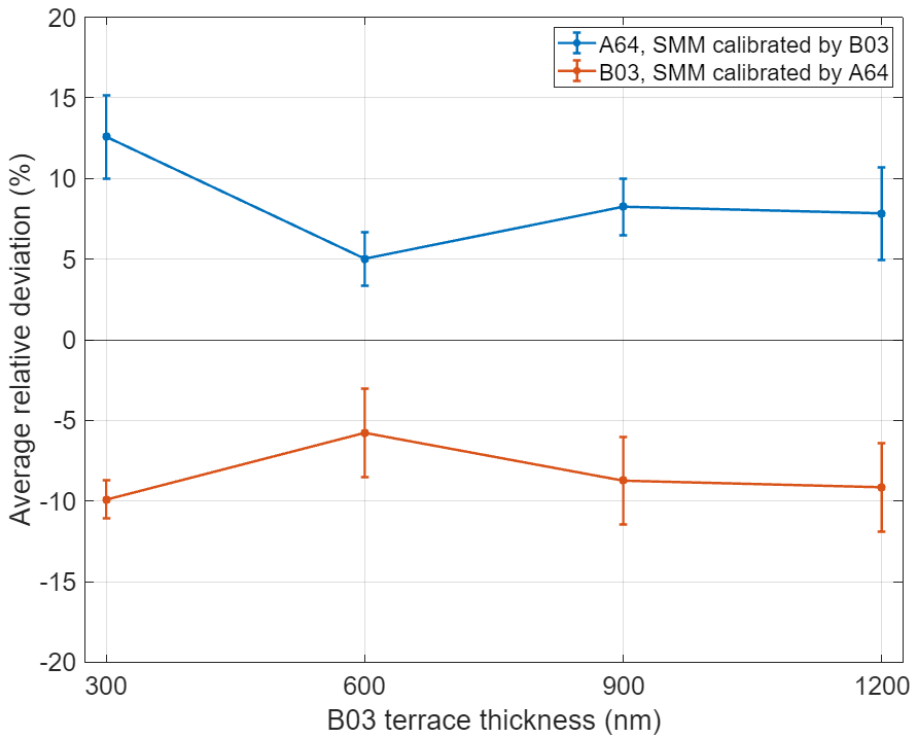


Figure 7. Average relative deviation between the computed and measured capacitance of the test sample using the calibrated SMM.

A systemic constant error of 8% is observed between the two structures. It is believed that a difference in the dielectric constant of the SiO_2 layer in both structures. Moreover, parasitic capacitance may still be unaccounted for in the first calibration sample. Additional measurements are planned to better understand the origin of this deviation.

4.2. Calibrated SMM measurements of dielectric constant on high-κ materials

The SMM calibrated on the first version of MC2 calibration kit (A64) was used to perform capacitance measurement over two high-κ samples, namely PZT and PMN-PT. Those two samples were provided to LNE by Electrosciences Ltd. Gold pad of various diameters were deposited on top of the high-κ layer to serve as top electrode of a capacitor in which the dielectric material is the high-κ layer. The capacitance value associated with each gold pad on the test samples were measured by the calibrated sample. The dimension of the top electrode and thickness of the high-κ layer was measured with a controlled uncertainty using SEM (for thickness measurement) and AFM (for top electrode area) images. Numerical simulations were conducted to estimate the dielectric constant of the high-κ layer with an uncertainty of 3.5% for the PZT sample and of 10.6% for the PMN-PT sample due to difficulty in the area determination caused by a rough surface. The evolution of the dielectric constant of the PZT layer as a function of the excitation frequency was evaluated between 1.5 to 5.5 GHz. A slight decrease with a slope of $\frac{\Delta\varepsilon_r}{\Delta f} = (18.4 \pm 1.6)\,\mathrm{GHz}$ was found which is in agreement with results reported elsewhere [12]. Additional details can be found in [13].

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