Publishable Summary for 20IND12 Elena
Electrical nanoscale metrology in industry

Overview

Consumer electronics, innovative quantum technologies, and Internet of Things applications all rely on semiconductors, where reliable characterisation of electrical properties at the nanoscale is essential for European innovation and competitiveness. The measurement of these properties allows the evaluation of critical parameters used to define the performance of electronic materials and components. Currently, Conductive Atomic Force Microscopes and Scanning Microwave Microscopes enable nanoscale electrical characterisation, but they are costly, complicated and, in many cases where they are used, unreliable as measurements are not traceable. This project aims to make such measurements traceable for the first time, with stated uncertainties, and affordable by developing and testing cost effective instrumentation and the first “out of lab” reference standards from DC to GHz. Robust calibration methods and good practice guides using simplified uncertainty budgets will underpin this effort.

Need

Micro- and nano-electronics are considered by the European Commission (EC) as a Key Enabling Technology (KET) with high potential for innovation throughout the economy, currently accounting for 10 % of EU gross domestic product (GDP), and fostering highly skilled employment. A competitive advantage in the semiconductor industry is gained through the exploitation of new materials and processes, translating into improved component performance. This requires a metrological infrastructure allowing a reliable nanoscale characterisation of new materials and devices, particularly in terms of their electrical parameters and properties. The development of electrical nanoscale metrology has also been clearly identified in the Nano-electronics Standardisation Roadmap by the International Electrotechnical Committee (IEC-TC113).

The main metrological problem is the traceability and reliability of the electrical properties measurements at the nanoscale using conductive atomic force microscopes (C-AFM) and scanning microwave microscopes (SMM). As scanning probe microscopy (SPM) methods, C-AFM and SMM are particularly attractive for use in the characterisation of electrical properties at the nanoscale because they allow non-destructive analysis of electrical components. However, the majority of measurements are currently carried out with different instruments, using different reference standards. Thus, they can neither be compared to each other nor used for modelling. This problem is compounded by the lack of established measurement protocols and the lack of easy-to-use reference standards. Furthermore, end-users need best practice guides, worked examples, and access to easy-to-use 3D models to assess the influence of environmental conditions, the influence of the particular instruments used in the measurement, and the type of standards used. Considering these influencing factors, a simplified uncertainty budget can be established for the measurement’s setup, which is required in many quality standards. Additionally, to make measurements economically viable, end-users require a cost-effective instrumentation, particularly for high-frequency measurements.

To address these problems, the project will develop and characterise such an instrumentation and easy-to-use reference standards, establish protocols and good practice guides, and develop easily accessible 3D models and simplified uncertainty budgets. The objective 1 develops and validates devices and procedures to meet the need for traceable measurements with low uncertainties. The objective 2 aims at achieving a high reliability and reproducibility of the measurements by developing techniques to calibrate the C-AFM and SMM instruments, and evaluating uncertainty contributions due to influencing factors. The assessment of the influencing factors is further developed in objective 3 through multi-physics modelling. The objective 4 formalises robust and accurate measurement techniques, based on the C-AFM and SMM to meet the needs of industrial end-users.

Objectives

The overall objective of the project is to establish a European metrological infrastructure and cost-effective technologies for C-AFM and SMM, enabling the industrial actors to conduct traceable measurements of electrical properties on materials and devices at the nanoscale.

The specific objectives are:

1. To develop and validate devices (DC to GHz reference standards with target uncertainties ≤10 %, probes, and measurement microwave electronics), and procedures (broadband impedance matching) for GHz near-field SMM. These developments should be suitable for DC-current measurements, high frequency (from 100 MHz to 50 GHz), material characterisation, and impedance measurements involving C-AFM and SMM techniques.
2. To use the devices and procedures from objective 1 to develop calibration methods for C-AFM and SMM techniques. This should include the quantification of uncertainty contributions due to influencing factors such as those that arise from the standards, the tip-sample interactions, and the measurement instrument in the laboratory environment.
3. To develop reliable 3D multi-physics modelling based on analytical or numerical approaches, to evaluate the effect of the water meniscus (*i.e.*, at the tip-sample interface) on the electrical measurement. This should also include an investigation of the effects of the tip's real shape and composition, and of the tip-sample electromagnetic interactions, on the electrical measurement. The modelling data will be compared to experimental results from objective 2.
4. To establish simplified uncertainty budgets for the C-AFM and SMM techniques using the results from objectives 2 and 3. In addition, to develop calibration methods for the key electrical measurands, including DC-current from fA to µA, DC-resistance from 100 Ω to 100 TΩ and HF-admittance from 100 nS to 100 mS, for the use in industrial applications. To develop 'out-of-the-lab' electrical standards, such as calibration kits, based on micro-size capacitors for the industrial calibration of C-AFM or SMM.
5. To facilitate the uptake of the technology and the measurement infrastructure developed in the project by the measurement supply chain in the micro- and nano-electronics sector (European industry, electrical SPM producers), standardisation organisations (IEC), and end users (NMIs and DIs, and academic and industrial R&D labs).

Progress beyond the state of the art

Despite the key importance of traceable nanoelectrical measurements of materials and devices in nowadays technologies, no appropriate metrology infrastructure has been established so far neither in the academic literature nor in the industrial R&D sector.

DC-current and resistance standards with target uncertainties in the order of 10 % and dedicated calibration set-up will be developed for C-AFM. This will involve the testing of instruments able to provide results over wide ranges, from µA down to fA, and from 100 Ω up to 100 TΩ.

In the GHz-range, only one capacitance calibration sample has been commercially available and very commonly used for the calibration of SMM measurements. In a close collaboration with the sample's manufacturer, the project will propose a novel design for an improved capacitance calibration sample with new features enabling the reduction of the uncertainties down to a few percent.

To facilitate the use of C-AFM and SMM techniques within the industry, the project will address the development of industry-compatible measurement instrumentation, and "out-of-the-lab" standards that are not only operational in a research laboratory. In the case of SMM, currently challenged with costly setups, the project will develop two instruments based on six-port and software defined radio (SDR) techniques and a new class of AFM probes.

So far, the literature is still lacking available three-dimensional multi-physics models for simulating the nanoelectrical measurements in C-AFM and SMM at the nanoscale. In this direction, the project will build a first body of numerical simulation works enabling the modelling of the water meniscus at the interface between the AFM tip apex and the sample surface. These simulations will be extended to cover the effects of the tip's shape and/or radius of curvature.

The aim of this project is to quantify and establish a complete uncertainty budget for the measurements of key electrical measurands, the DC-current, the DC-resistance, and the HF-admittance using C-AFM and SMM with the help of new devices, procedures and numerical or analytical models. This will be achieved by means of environment-controlled measurements on various reference samples as well as studies about tip-sample interaction to firstly formulate a complete uncertainty budget which can then be separated into contributions with different level of impact and modified into a simplified version for industrial use. In the work on case studies, a different, easier and more practical approach will be developed. The end-user oriented way to provide the uncertainty budget will mainly be based on reference samples as well as a software product which will be capable of determining the uncertainty from calibration measurements.

Results

Instrumentation and reference standards (Objectives 1 and 4)

A comprehensive investigation was carried out on a third design of the tunnelling junction based reference samples and highlighted four main issues which make definitively these samples not appropriate to calibrate C-AFM as an "out of the lab" current standard. As an alternative approach, a new architecture has been designed for the standard samples combining the current and resistance reference on the same chip. The principle of this design relies on incorporating available diodes with the resistance structures to facilitate their practical use and reinforce their stability during test measurements. The samples were fabricated and validation tests are in progress.

Resistance-array standards covering the resistance range 100 Ω – 100 GΩ have been designed, fabricated and fully characterised. It has been demonstrated that such reference sample enables the calibration of C-AFM with a combined relative uncertainty lower than 2.5% over an extended range from 10 kΩ to 100 GΩ. This work has involved a home-made wide-range current measuring device (WCMD) designed and fabricated by one of the partners. The results have been reported in a paper published by a peer-reviewed journal in November 2023. New resistance-array reference samples have been designed and fabricated with the aim at calibrating C-AFM set-up in the extreme resistance values, i.e. much higher than 1 TΩ. Measurements are in progress.

New calibration kits based on micrometer size Metal Oxide Semiconductor (MOS) capacitors have been codesigned and fabricated by a manufacturer. These standards dedicated for the calibration of SMM in terms of capacitance covering the capacitance range from 100 aF up to 40 fF. Successful SMM measurements were recently performed on these calibration kits and validate the fabrication of these devices. Two other standards for SMM measurements have been designed. One design, containing gold structures on a SiO_2 membrane; which is suitable for coaxial tips with 20 µm or larger outer diameter is ready for use. The second design with ITO layers of different thickness for the calibration of thin coaxial tips is now in its third production run.

Different tip designs for SMM have been intensively discussed. The fixing mechanism of the probe onto the instruments' probe holders has been defined and production of tips has started.

Four types of matching circuits have been characterised with respect to their gain and added noise. The proposed measurement protocol has been executed by all participating partners, and the results have been reported. Currently a publication on this is in peer review.

An SDR and a six-port are envisaged as electronics for the SMM. The SDR has been successfully tested when measuring known impedances. The six port electronics is still in the development phase.

Calibration methods for C-AFM and SMM and quantification of uncertainty contributions (Objective 2)

A first draft of procedure to calibrate C-AFM in terms of resistance has been established and successfully tested. The adaptation of this method applied to non-conventional mode (peak force SSRM) is investigated.

The development of robust SMM calibration techniques for traceable measurements of HF admittance is in progress. SMM measurements have been performed to compare the two existing calibration techniques: the modified Short-Open-Load (SOL) approach and the tip retraction technique, with a focus to use low spring constant AFM tip.

For the calibration of both techniques, the uncertainty contributions to be quantified budget are subdivided in four categories.

The first category concerns the uncertainty contributions from the reference standards. For the newly developed resistance standards, the major uncertainty components originated from the sample temperature, and voltage effects and do not exceed 0.1%. Dimensional parameters of the new capacitance calibration kit have been measured by AFM and Scanning electron microscopy (SEM) leading to improved uncertainty components in contrast with the former design. A new approach using LCR meter has been implemented to determine the dielectric constant of the SiO_2 layers of the calibration kit.

The second category is related to environmental conditions. C-AFM measurements on array resistance standards have shown no effect from relative humidity within an uncertainty of a few percent. Influence of the tip on C-AFM measurements is currently being investigated using different kinds of tip. It is worth noticing that unexpected photovoltaic effect was observed. In the case of SMM techniques, measurements have shown noticeable influence of humidity only for capacitances smaller than 100 aF. The results are in line with preliminary measurements on nano-sized capacitors which highlighted the impact of the water meniscus on SMM measurements. Moreover, the relation between temperature, humidity and instrument drift has been investigated. SEM images have been analysed to determine the geometry of several commercial probes commonly used in SMM. The deviations from nominal values will be used to evaluate the impact on impedance characterisation based on 3D EM simulations. Finally a data set from the results regarding impact of water on the C-AFM and SMM measurements is being prepared.

The error sources related to non-environmental operating parameters provide the third category of uncertainty contributions. Repetitive measurements have been performed on resistance reference sample with C-AFM operating at different conditions (scan speed and scan direction, measurement times, tip, bias voltage) and in different modes (permanent contact, intermittent contact, imaging or spectroscopic). The data treatment is on-going. Similarly, SMM measurements have been performed on a capacitance calibration kit using five different probes. Depending on the tip used, the dispersion of results can vary from 3% to 9%.

Finally, the last category of uncertainty contributions concerns the measurement instrument itself. The uncertainty budget has been established for the calibration at microscale of the resistance and current standards leading to a combined uncertainty limited to 0.1%. The uncertainty budgets are being prepared for the calibration of the C-AFM set-ups. In the case of SMM set-up, the estimate of the uncertainty contributions from measurement microwave electronics is in progress. A method to determine the linearity error has been defined and simultaneous measurements of scattering parameters S_{11} by Six-port reflectometer and vector network analyser (VNA) are on-going. The comparison of different methods for calibration of the spring constant of the SMM cantilever is also in progress.

Reliable 3D multi-physics modelling (Objective 3)

The modelling of the meniscus was carried out after a selection among three different analytical expressions applicable in a finite element (FE) simulation (COMSOL® software). The chosen model showed a reduced number of parameters and enabled the variation in the width and height of the water meniscus, and the thickness of the water layer covering the surface of the sample. This model was used to perform a self-calibration of the tip-sample model. The simulations of the water layer’s influence, with and without the meniscus has been conducted in the case of SMM measurement, and is on its way for C-AFM measurement. A particular focus is made on the water layer’s influence when the calibration samples are used. A numerical twin of the SMM experiment, constructed using a self-calibration procedure, allows to assess the role of water meniscus in spite of the unknown electrical properties of water at such scale.

The meniscus was also modelled using Bezier curves and the simulations were conducted with the SFEPy package. A realistic shape of the water meniscus can be obtained using software that takes into account the surface energies, the real shape of the tip, and its hydrophylic nature. This model can be used in FE software in order to model the influence of the water meniscus in a more realistic manner.

The capacitance produced by the tip apex and the tip cone was modelled analytically using simplified hypotheses. SEM images and GWYDDION software were used to estimate the parameters of the model, in particular the radius of curvature of the tip. The analytical model was compared to COMSOL® calculations, showing a fair agreement. The tip wear during SMM measurements was evaluated and the tip shape was modelled from SEM images. FE simulations have been conducted with the aim of evaluating the influence of the tip shape, especially the apex radius but also its general shape estimated from SEM images, on the evaluation of a known capacitance by SMM. It was found that the curvature radius influences the result, contrary to the working frequency.

A novel structure of microwave probe was proposed, which corresponds to a modification of a commercial solution. The main idea is to support a guided microstrip propagating mode along the probe. For this, a microstrip line and a ground plane were designed according to the ceramic substrate thickness. The probe was simulated, leading to the calculation of the amplitude and phase-shift of the complex reflection coefficient. The electric field in the vicinity of the probe was modelled, leading to the conclusion that the probe is more sensitive to the electrical and dielectric properties of the materials, compared to their magnetic properties. The study of the lateral and in-depth resolution of SMM started, with an initial focus on the electromagnetic coupling between two close devices.

Simplified uncertainty budgets and case studies (objective 4)

The algorithm for calculating the simplified uncertainty budget was developed further. A strong emphasis is put on working with reference samples and using them to calibrate instrument in the industrial environment. One partner has developed a software package to facilitate this task and make it available to the end user. The software is called "SMM Calibration" and is currently being tested within the consortium.

In the frame of a first case study involving a leading European graphene manufacturer, optical data on graphene layers have been collected. Models for ellipsometric analysis of graphene were developed. C-AFM and SMM measurements are also planned on provided graphene-based FET structures. A second case study using ITO layers as conductivity standards was initiated. ITO layers were produced and micro structured by two partners. Ellipsometric measurements and modelling were performed, and models were developed to determine the conductivity from optical data and to correlate the findings with direct electrical measurements. Currently, a third case study involving high-k thin film materials for FET devices provided by an industrial collaborator is in progress. The samples have been received by the project partners and the measurements are being performed. The case study will continue during the last six months of the project.

Impact

To promote the uptake of the project's results and to share insights generated throughout the project, the results were shared broadly with scientific and industrial end-users. Two papers have been published in open-access peer-reviewed international journals, two papers are undergoing peer review revision and two other papers are in preparation. In addition, two articles were published one in a large-audience industrial journal and the second in industrial newsletters. 43 presentations were given at conferences among which 23 are leading international conferences. A one-day workshop for users working on energy nanomaterials was organised, gathering 40 attendees. An international summer school dedicated to nanometrology is also prepared and will be held on June 2024 at Frejus (France) with an expected audience of 60 attendees. The project was presented at standardisation committees and was posted regularly on social media. Two webinars for training in the field of standardisation were broadcasted with remote project partner attendees.

Impact on industrial and other user communities

Equipment manufacturers of C-AFM and SMM will increase sales as their users become confident about the ability of these instruments to quantify electrical properties at the nanoscale with stated uncertainties. This will be enabled by establishing reliable calibration procedures and developing "out-of-the-lab" reference standards. These new standards and measurement probes will be fabricated by means of industrially proven and standardised methods, to facilitate their widespread uptake and commercialisation. High dimensional and parametric reproducibility of the fabricated DC to GHz reference standards will allow their wide dissemination without the need for extensive and costly calibration of individual elements. The IP generated here will be exploited through licensing agreements with foreseen EU instrument manufacturers. Innovation in developing methods for C-AFM and SMM will be accelerated through the use of the new fast and reliable 3D modelling software developed in this project. This new software will be made open-source to enable a wider uptake.

Four manufacturers already showed strong interest to commercialise the reference standards for C-AFM and SMM produced in this project. Two of them have been provided with resistance calibration samples fabricated by one partner. In the case of SMM, already two tight bilateral collaborations have been established between two manufacturers and two partners on the development of impedance calibration kits. Moreover, one industrial stakeholder and one NMI have been provided with such DC resistance standards for testing.

Impact on the metrology and scientific communities

The project will achieve a landmark progress in the broader use of nanoscale electrical measurements by filling the gap of a missing calibration and traceability infrastructure, thus, moving away from poorly reproducible and relatively inaccurate measurements. The project aims at achieving traceable and quantifiable measurements with well-defined calibration procedures. The SI traceability with simplified, yet accurate uncertainty budgets will stimulate international comparisons of electrical quantities at the nanoscale, accelerating the progress and the uptake of instrumentation and methods within the communities. Such a development will provide a roadmap for the future development of C-AFM and SMM.

The uptake of SMM and C-AFM for use in characterising electrical properties in materials and components will increase and application areas will be extended through the publication of good practice guides and the provision of calibration samples and open source software for robust modelling. The improved reproducibility of these methods will speed up the scientific progress in the development and understanding of nano-electronics materials. The development of novel and improved nanoscale characterisation methods will be accelerated due to a better understanding of the tip-sample interaction volume and other influencing factors enabled by the validated new 3D-modelling and the uncertainty analysis developed in this project.

Impact on relevant standards

The development of good practice guides, calibration procedures, and protocols for measuring electrical properties at the nanoscale will impact the international standards directly. This will be facilitated by the direct engagement of project partners with the relevant standards committees (IEC, CEN, ISO). Project partners are already active members of these committees, including IEC TC113, which is the natural entry point for the standardisation in nano-electronics. A subgroup was established acting as the interface between the project and the international Standards Developing Organisations (SDO) to optimise mutual understanding and to deliver guidelines, protocols, and procedures. These will result in a minimum of two standards which will be published within the regular time frame of 36 months after approval of the New Work Item Proposal (NP).

As a follow up to the Preliminary Work Items (PWIs) established by the subgroup, two NPs have been circulated inside IEC TC 113 to the participating and observing IEC National Committees for the voting process, the first on capacitance measurements by SMM, the second on resistance by C-AFM. Both NPs have been voted positively. The scopes of the standards were extended due to achievements in the project from "capacitance" to "impedance" measurement and from "resistance" to "current and resistance" measurement. The plan is to circulate the first Committee drafts (CD) after the next IEC/TC 113 plenary meeting to be held in April, 2024.

It is worth noticing that the consortium takes also active part in the standardisation of ellipsometry as a metrological technique. Within DIN NA 062-01-61, the standard series DIN50989-x is developed which is intended as a sample independent definition and best practice guide for ellipsometry. This standard series is developed into an international standard series ISO23131-x, which are written in ISO/TC 107.

Longer-term economic, social and environmental impacts

Europe hosts the world-leading manufacturers of SPM instrumentation and the development of its new modes. Reliable eSPM metrology will accelerate the progress in nano-electronics, ensuring that Europe maintains its leading position. These techniques are currently used by industrial and academic R&D labs in three Key Enabling Technologies considered by the EC as major drivers of the economic growth in Europe: nanotechnology, micro and nano-electronics, and advanced materials. This project develops the underpinning metrology to quantify the nanoscale electrical properties, which are critical to increase the innovation, the competitiveness, and the resilience of these industries.

As an environmental consideration, the reduction of waste in semiconductor manufacturing is critical. Defects identified early in the complex production process due to well-calibrated inspection techniques save many energy-expensive processes as the product goes to waste. Reliable metrology at the nanoscale will also enable a faster route to market of critical low carbon industrial technologies, including renewable energy sources, more efficient energy electronics, and digital data processors. Both waste reduction and faster market access will have significant impact on the key EC target of Green Manufacturing.

List of publications

1. Kaja, Khaled et al (None) '3D Imaging and Quantitative Subsurface Dielectric Constant Measurement Using Peak Force Kelvin Probe Force Microscopy', *Advanced Materials Interfaces*, 2300503 p. 1-10. Available at https://doi.org/10.1002/admi.202300503
2. Piquemal, François et al (None) 'A multi-resistance wide-range calibration sample for conductive probe atomic force microscopy measurements', *Beilstein Journal of Nanotechnology*, 14 p. 1141-1148. Available at https://doi.org/10.3762/bjnano.14.94

"This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/"

Project start date and duration:	1st September 2021 for 36 months	
Coordinator: *François Piquemal, LNE* Tel: +33 1 30 69 21 73 E-mail: francois.piquemal@lne.fr Project website https://projects.lne.eu/jrp-elena/		
Internal Funded Partners: 1. LNE, France 2. BAM, Germany 3. CMI, Czech Republic 4. DFM, Denmark 5. METAS, Switzerland 6. PTB, Germany 7. TUBITAK, Turkey	External Funded Partners: 8. CEA, France 9. CNRS, France 10. ISC, Germany 11. JKU, Austria 12. ULILLE, France 13. UofG, UK	Unfunded Partners:
Linked Third Parties: 14. INSA Lyon, France (linked to CNRS)		