

# Report on the activities in Electricity and Magnetism within the LNE between 2021 and 2023

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In this report, we provide a brief outline of the main research and development activities in the field of Electricity and Magnetism since March 2021 at the Laboratoire national de métrologie et d'essais (LNE).

## **1** Fundamental mass metrology

## Kibble balance

LNE continues the developments on the Kibble balance to realise the kilogram with an aimed uncertainty of a few parts in  $10^8$  in vacuum.

After a number of various optimizations, dynamic phases were performed in vacuum. These dynamic phases "2021" show a significant improvement of the noise compared to 2017 for the determination of the field profile (the rejection factor has indeed been improved by a factor of 3). This result is the outcome of several improvements of the experimental process, notably: hollow retroreflectors for interferometers, pillar structure for mechanical structure of the set-up, commercial nanosecond trig generator, new amplified photodetectors with higher gain and lower noise.

An automated system for the control of the optical frequency of the Nd:YAG laser (intended to measure the velocities) has been realized. It completes the optical device and the PLL, allowing locating the lines of the hyperfine structure of the di-iode used for the realization of the meter. A system of control identifies the line of interest and locks the frequency of the laser on this target. In case of loss of the servo due to an external event, the return to the frequency line is also automatic.

The force model determined by the double pendulum model was used again and carefully checked. A comparison was made with other possible force estimates: by calculating the forces from the displacement of other segments of the suspension, but also by the position of the coil with respect to the magnetic circuit. These 3 groups of force estimation are based on simple physical models, and are driven by several numerical parameters (like the length and masses of the different suspension segments for example). Their comparison allows to refine the value of these numerical parameters (known theoretically) and especially to remove most of the possible errors of instrumentation, since each model uses different information.

In case of a non-horizontal beam used as force comparator, horizontal parasitic forces exerted on the coil can cause of bias. A method of measuring the position of the beam has been developed. It has the advantage of being carried out with the habitual instrumentation of the balance, using in particular a commercial interferometer (measuring the vertical position of the end of the beam) and the Gaussian sensors (measuring the position of the coil with respect to the horizontal plane). Under the sole assumption of the verticality of the Gaussian sensor beams, it is possible to determine the inclination of the beam axis of rotation with respect to the horizontal and the inclination of the beam longitudinal axis with respect to the horizontal plane.

Various vacuum measurements were performed from July 2021 to September 2022. Especially, mass measurements were carried out for the comparison CCM.M-K8.2021: the mass standard was a 500 g iridium, identified as DB1. Its mass was determined with a relative type-uncertainty of  $u_r = 1.1 \cdot 10^{-7}$ . This

uncertainty should be considered as the outcome of the first dry run under vacuum of the LNE Kibble balance.

### **Electrostatic balance**

LNE has begun the conception and machining of an electrostatic force balance, aiming at realizing the mass unit at the milligram level in the International System of Units (SI).

The most important parts are designed, machined and assembled: a mass balance mechanism, two circular capacitance actuators (for balancing the force to be measured and for changing the vertical position). The balance mechanism is a fully monolithic parallelogram with 40  $\mu$ m-thick flexure hinges. The two actuators are fixed on the same side of balance mechanism, with their own monolithic adjustment system. Special attention has been paid to the overall symmetry of the system and to cost efficiency.

By using a commercial interferometer, a 8.5 digits voltmeter and  $\pm 1000$  V-source driven by a custom digital PID, the study of the vertical stiffness was done, in particular its dependence (from 8 N/m to lower than 0.01 N/m) relative to the position of the mass centre of the balance mechanism. A very rough weighing of a 2 mg-mass was carried out.

The next steps will be to lower the stiffness by a non-gravitational method, to study the influence parameters for mass measurement standard deviation and to measure the capacitance gradient.

Contact: Matthieu Thomas (matthieu.thomas@lne.fr)

## 2 Capacitance metrology

## Thompson-Lampard calculable capacitance standard

The new calculable electrical capacitance standard will enable realizing the farad in the SI with a combined uncertainty (k = 1) in the order of one part in 10<sup>8</sup>. To achieve this result, various original mechanical systems to position and control macroscopic elements at the nanometer scale have been developed. A measurement system allowing the in-situ control of this positioning is integrated into the standard. The electrodes are held in position in a hexapod structure, which is dissociated from the standard's carrier structure to ensure that no force is applied to change the electrodes' positions. An additional electrode, so-called "movable guard", is introduced into the cavity formed by the electrodes to ensure the operation of the standard. The lateral position of this guard in the cavity must be controlled to within 100 nm along its entire length of travel, which is achieved by means of two piezoelectric actuators. An additional capacitive measurement device between the moving guard and the five electrodes ensures that it is correctly positioned in the cavity. It has been demonstrated that the system described here guarantees the correct positioning of the guard within 80 nm in the cavity, regardless of the cavity's altitude.

#### Non-linearity of AH 2700A Capacitance Bridges with an uncertainty level of 1 part in 10<sup>7</sup>

The stability and nonlinearity of a commercial AH 2700A capacitance bridge were studied beyond its specified capabilities using the Thompson-Lampard Calculable Capacitor (TLCC) at LNE. The TLCC allows for continuous variation of measured capacitance between 0.4 pF and 1.2 pF with a resolution of 2 parts in  $10^7$  and stability better than 1 part in  $10^9$  over 2 days. The study confirmed a saw-tooth evolution of bridge non-linearity previously reported in the literature and observed when the bridge was used without internal calibration. Additionally, the dependence of capacitance nonlinearity on various factors such as frequency and capacitance value were analyzed. This work enables automatic calibration of the commercial bridge with an uncertainty level of 1 part in  $10^7$  and allows for quick evaluation of TLCC's non-linearity and monitoring of any changes over time through *in-situ* measurements.

Contact: Olivier Thévenot (olivier.thevenot@lne.fr); Almazbek Imanaliev (Almazbek.Imanaliev@lne.fr)

## **3** QHE metrology

In the perspective of simplifying the operation of the quantum Hall resistance standard (QHRS), namely at lower magnetic field ( $B \le 5$  T), higher temperature ( $T \ge 4.2$  K) and higher measurement current ( $I \ge 100 \mu$ A), for the wider dissemination of the electrical units in the SI, LNE is working on improving the reliability of the technology of quantum Hall devices made from graphene grown by chemical vapor deposition on silicon carbide (G/SiC). This work follows on from the results obtained at LNE in 2015 on the demonstration of the QHRS in a graphene device under relaxed conditions with state-of-the-art accuracy (1.10<sup>-9</sup>) (Nature Nanotechnology 10, 965 (2015)) and concerns the same technology. The present work includes notably the control of the charge carrier density, with a current focus on molecular doping. This work is carried out in collaboration with C2N at CNRS/Université Paris-Saclay and with CRHEA at CNRS.

Towards this goal of simplified operation of the QHRS, LNE has also installed a cryomagnetic system, without liquid helium consumption, based on a pulse-tube cryo-cooler and a closed cycle helium 4 variable temperature insert. A continuous operation of the system has been demonstrated for typically 2-3 months, with a stability of the base temperature at the millikelvin, after upgrading the system with a liquid nitrogen trap to purify the helium gas circulating in the closed-cycle system. LNE has customized the top-loading probe that equips this system and allows fast exchange of samples - notably with homemade wiring - in order to perform precision QHR measurements. LNE has validated this setup with high-precision and high-accuracy measurements of a well-characterized LEP514 GaAs based QHRS, reaching comparable performances to LNE' former wet cryostat.

Moreover, this top-loading probe integrates coaxial wiring all the way down to the TO-8 sample holders in order to perform QHR measurements in the AC regime - in the kHz frequency range - with a target relative uncertainty below  $0.01 \ \mu\Omega/\Omega$  for traceability of impedance units to the SI, complementary to the LNE's Thompson-Lampard calculable capacitance standard.

Contacts: François Couëdo (<u>francois.couedo@lne.fr</u>); Mathieu Taupin (mathieu.taupin@lne.fr)

## 4 Quantum ampere

LNE has developed a practical quantum current standard realizing the new ampere definition based on the elementary charge (J. Brun-Picard et al, PRX 6, 041051 (2016)). It is founded on the application of Ohm's law to the quantum voltage and resistance standards that are combined using a cryogenic current comparator. It was demonstrated that the quantum current standard could generate 10<sup>-8</sup>-accurate currents in the range from 1  $\mu$ A up to 10 mA. Our goal is to equip a new laboratory dedicated to the traceability of current measurements to the SI with the POCG. After having improved the design of the POCG to achieve lower noise and better reliability (more efficient damping circuit, improved grounding scheme), we have developed new elements for the optimized version of the PQCG: a new external current source adapted to the generation of currents from 1 nA up to 100 mA, a new cryogenic current comparator (CCC) made of 20 windings including several winding triplets of same number of turns in order to implement the triple connection of the QHR to the PJVS (to achieve negligible effect of the connection resistances), a new cryogenic system based on a pulse-tube and equipped with a 7 l liquid helium tank to cool down two hermetic PJVS probes. This latter system is characterized by a 600 mW available cooling power and a temperature stability better than 1 mK at 4 K owing to a fine pressure regulation of the helium. It also allows short heating pulses to remove trapped magnetic fluxes in the Josephson arrays. All these improvements aim at realizing the ampere with a reduced measurement uncertainty using the PQCG in a more practical and economical way.

Contacts: Sophie Djordjevic (sophie.djordjevic@lne.fr); Wilfrid Poirier (wilfrid.Poirier@lne.fr)

## 5 Electrical nanometrology

This activity is focused on the development of metrological instrumentation (reference calibration samples, probes, and electronics) and calibration methods for measuring electrical quantities at the nanoscale using electrical scanning probe microscope (eSPM). Reducing the total standard uncertainty down to a few percent in relative value under optimal conditions forms one of the main challenges in eSPM. In addition to improving the accuracy of electrical quantities' measured in eSPM, this enables the determination of the materials' inherent electrical properties (e.g., permittivity, doping concentration, mobility) at the nanoscale.

We published a study on the nanoscale metrological quantification of the dielectric constants of two high-  $\kappa$  materials, lead zirconate titanate (PZT) and lead magnesium niobate-lead titanate (PMN-PT), in the GHz range. This work was based on the previously reported results on the measurement of capacitances ranging from 0.2 fF to 10 fF using a scanning microwave microscope (SMM) with a combined relative uncertainty of 3 % (k = 1), We highlighted the strong weight of dimensional measurements on the combined relative uncertainties in the capacitance calibration procedure. We introduced a novel approach for correcting the lateral dimension measurements of micro-capacitive structures using the microwave electrical signatures, especially for rough surfaces of high- $\kappa$  materials. Moreover, we derived a new analytical expression for the capacitance calculations, taking into account the contribution of fringing electric fields. Consequently, we succeeded in determining the dielectric constant of PZT = 445 and PMN-PT = 641 at ~3.6 GHz, with combined relative uncertainties of 3.5% and 6.9%, respectively. This work provided a general description of the metrological path for a quantified measurement of high dielectric constants with well-controlled low uncertainty levels.

More recently, we demonstrated a controlled uncertainty at a level of a few percent for the calibration of the SMM measurement using a modified Short Open Load (mSOL) method in differential mode. This result was demonstrated using various types of representative conductive AFM probes. Furthermore, we developed a new approach in SMM to measure quantifiable variations of tan $\delta$  and to map its local variations. We foresee a particularly strong potential of the proposed method in elucidating the functional efficiency of local nanostructures for high-speed switching and heat dissipation in operating devices.

In addition, we developed multilayer-based GaAs samples as reference systems for the calibration of dopant concentrations  $(5 \cdot 10^{16} \text{ cm}^{-3} - 2 \cdot 10^{19} \text{ cm}^{-3})$  adapted for SMM measurements on individual GaAs nanowires. Secondary-ion mass spectrometry (SIMS) was used to characterize the same set of multilayer samples. The uncertainties on SIMS measurements have been largely investigated in the literature providing concentration values within an overall 10 % uncertainty and concentration ratios with a typical uncertainty of 1 %. To investigate the uncertainty on our SMM measurements, we compared the relative variations between the dopant concentrations of the different layers measured by SMM and SIMS, and found a good agreement within  $\pm 10$  %. A same level of agreement was obtained for doping concentration ratios for our developed GaAs multilayers samples, we propose their use as reference samples for further calibrated measurements of dopant concentrations on axial and radial pn junction nanowires. This will constitute a first step toward the calibration of dopant concentrations on vertical semiconducting nanowires with a 10 % uncertainty. Further investigations are in progress and first results on doping concentrations measured by SMM on the top surface of vertically aligned nanowires.

Part of this work has been developed in the frame of the projects Nanowires (19ENG05) and Elena (20IND12), which received funding from the EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme. In particular, the project Elena aims at pioneering the traceability of such measurements, with stated uncertainties. The project also aims at increasing the affordability of these methods by developing and testing cost effective

instrumentation and reference standards spanning the range from DC to GHz. Robust calibration methods and good practice guides using simplified uncertainty budgets will underpin this effort.

Contacts: Khaled Kaja (<u>khaled.kaja@lne.fr</u>); Jose Moran (<u>jose.moran@lne.fr</u>); François Piquemal (francois.piquemal@lne.fr)

## 6 Power and Energy

LNE is an active member of the European Metrology Network Smart Electricity Grids since its creation, May 2019. Important contribution in the last year might be outlined on the elaboration of the Strategic Research Agenda and its roadmaps (Power quality, Instruments Transformers and Sensors, respectively DC grids) and of the network NewsLetters published in November 2021, respectively September 2022.

Research activities in 2021 and 2022 are in relation with power quality of AC and DC electric grids. The increasing number of electrical goods connected to the electrical grid is leading to an increase in interferences and disturbances affecting the power line communications and smooth function of the grid. The European project JRP 18NRM05 SupraEMI (2019 - 2022) targeted the measurements of emissions in the frequency range from 2 kHz to 150 kHz (supraharmonics). The project aimed to answer the question:

## How to compare the emissions measurements carried out in the laboratory with the emissions measured on site?

As partner of this project, the main role of LNE was to provide the reference measurements of conducted emissions of mass-market household appliances. Measurements were performed in laboratory test environment according to the family of standards EN 55016 (CISPR 16). To study the characteristics of device emissions under different conditions of the low voltage electrical network, measurements were performed also on-site. The comparison between laboratory and on-site measurements outlined that the CISPR 16 method and devices are quite sensitive, and the inherent noise of the network can make difficult the detection of emission of equipment with broad spectrum. On the contrary, high-level and narrow-band emissions are well detected. However, when turning to the grid measurements, common effort and research are still needed before defining standards to determine the emissions in a comparable, repeatable and traceable way.

New measurement and technological challenges arrive from DC field. The European project JRP 20NRM03 DC Grids ("Standardisation of measurements for DC electricity grids") aims to develop traceable measurement and characterization of PQ parameters to support standardization in further development and future use of DC grids. Active partner in the JRP consortium, LNE is working close to other NMIs to the development of new reference DCPQ setups on DC specific PQ parameters such as ripple and in-rush.

Contact: Daniela Istrate (daniela.istrate@lne.fr)

## 7 High Voltage/High Current metrology

## Development of a standard measuring system for traceable high voltage pulses up to 500 kV in the nanosecond and sub-nanosecond range

A calibration system has been developed for the characterization of high voltage pulses of amplitudes up to 500 kV with rise times as low as few hundreds of picoseconds. The developed measuring system is composed by two major components, a voltage divider and a termination load. Other components have been also developed, high-voltage connectors to facilitate the insertion of the measuring system to other industrial systems and transition cones which are necessary for the calibration. The voltage divider is the central component of this measuring system as it reduces the high voltage amplitudes to adequate levels

which could be measured through a calibrated oscilloscope. The voltage divider designed through analytical and numerical calculations. Its performances in terms of measuring accurately and precisely nanosecond and sub nanosecond pulses depend also on the characteristics of the transmission line termination load since the reflections from an inadequately matched line termination load could lead to the misunderstanding of the measured waveform at the output of the divider. A 50  $\Omega$  high accurate line termination load has been developed and characterized. It has high insulation properties for voltage amplitudes up to 500 kV and a maximum reflection coefficient of -27 dB as a function of frequencies up to 2 GHz.

The characterization of the complete measurement system was carried out in two steps. Firstly, the measurement system was characterized at low levels of input power by two different methods, namely, VNA characterization and calibration through a SI traceable high frequency attenuation measurement method. The results obtained from these two methods were compared to CST and spice modelling results and all of these results were found to be in good agreement with each other. These characterizations demonstrated that the developed measurement system has a division ratio of around 85 dB, a linear phase response and a flat bandwidth from 300 kHz to at least 2 GHz. Calibrated high accurate attenuators could be added at the output of the divider to increase the division ratio up to 110 dB without degrading the metrological performance of the measuring system. In the second characterization step, this system was tested at high power levels through nanosecond pulse generators. Different high voltage pulses of voltage peaks as high as 300 kV unipolar and 420 kV bipolar were successfully achieved with rise times ranging from 400 ps to 10 ns. The measurement uncertainties for these high voltage measurements were calculated to be 3.4 % for the voltage peaks and 87 picoseconds for the temporal parameters.

Additional investigations are underway to study the possibility to extend the range of frequency up to at least 3 GHz in order to measure pulses with rise time of 100 ps. Indeed, during the characterization phase, the calibrations did not pushed so far for frequencies above 2 GHz since the first principal goal of the system was achieved.

Contacts: Mohamed Agazar (mohamed.agazar@lne.fr); Mohammad Saif Khan (mohammad-saif.khan@lne.fr)

## 8 AC resistance and current measurements up to 1 MHz

#### Traceability of AC resistances up to 1 MHz

Since 2019, LNE launched an internal project on the calibration of AC resistances in low-frequency range from 10 m $\Omega$  to 2 M $\Omega$ . The project has two scientific and technical objectives: develop an automated measurement method for calibrating AC resistances with a maximum uncertainty at 20 kHz of 50  $\mu\Omega/\Omega$  and 50  $\mu$ rad on AC-DC difference and impedance phase respectively. The second objective is to extend the frequency bandwidth up to 1 MHz (currently limited to 20 kHz). The complementary method is based on the use of a vector network analyser (VNA) with targeted uncertainties of at most 5 m $\Omega/\Omega$  and 5 mrad respectively on AC-DC difference and impedance phase up to 1 MHz.

In 2023, a new calibration bench (Wheatstone bridge) for AC resistances up to 20 kHz was developed. The Wheatstone bridge is based on a dual AC voltage source and PXI modules. The dual AC voltage source supplies the Wheatstone bridge and the PXI modules that are used as a voltage injection system. A 10 MHz synchronization solution with an external signal has been used for the two instruments. The calibration bench has been validated for the resistors defined in two terminal-pair (2TP), this bridge has been used to calibrate the standards resistors defined in 2TP of LNE. High impedance stages are developed to adapt the bench for the resistors defined in four-terminal pairs (4TP), the adapted bench has also been used to calibrate standard resistors defined in 4TP.

The second method to measure AC resistors between 20 kHz and 1 MHz and based on the use of a VNA has been validated. This method is conditioned by the possibility to model the resistance frequency behaviour by polynomial regressions (up to a few MHz). Most standard resistors at LNE are designed with UHF connectors and the traceability of the VNA is ensured only with BNC or Type-N connectors. Two analytically calculable RF adapters for Type-N to UHF connectors were designed, the mechanical assembly and characterization of these adapters were validated.

## Design and modelling of a shunt for current measurements at 10 A and up to 1 MHz

The LNE has investigated the possibility to extend the calibration capabilities of high current sensors up to 10 A and 1 MHz and thus to improve the traceability of AC current measurements.

Firstly, LNE has developed a completely calculable (electromagnetic and thermal responses) current shunt standard for 50 mA based on theoretical basis and innovated design: at 1 MHz the phase shift and transposition deviation are - 0.01 mrad and 15  $\mu\Omega/\Omega$  respectively. In 2023, a calculable current shunt of 0.08  $\Omega$  for a current of 10 A was developed and validated. An optimization study of the electrical resistivity and temperature coefficient of the resistive disks as a function of the thickness of the deposited layer was performed. The current shunt of 10 A was used to ensure uncertainties less than 0.2 % on the high current sensor calibration up to 10 A and 1 MHz.

Secondly, LNE has developed a traceable calibration method to measure shunts up to 10 MHz. The measurement method, based on the use of a vector network analyser (VNA), allows the AC-DC difference and impedance phase of a shunt to be measured simultaneously with relative uncertainties less than  $1 \cdot 10^{-3}$  at 1 MHz.

Contact: Mohamed Ouameur (mohamed.ouameur@lne.fr)

## 9 RF and MW metrology

## Specific absorption rate (SAR) and vector electric field measurement & EMPIR project Vector SAR

This activity is complementary to the JRP Vector SAR (completed by mid-2020) which aimed to establish the traceability of SAR measurement when using vector E-field probes. The focus of the project is to characterize tissue equivalent liquids and to develop the measurement systems and methods for vector E-field probe calibration in several telecommunication frequency bands between 400 MHz and 6 GHz.

The complex permittivity is characterized using a VNA to measure the *S* parameters of coaxial cells filled with reference liquids or tissue equivalent liquids. A permittivity extraction procedure using the Line-Line method, derived from the "Through-Reflect-Line" (TRL) calibration technique, is established. There is a very good agreement between the calculation and measurement results of the relative permittivity and conductivity of the ethanol reference liquid with less than 3 % difference between the measured and the calculated relative permittivity. For the tissue equivalent liquids formulated on the polysorbate-based recipes proposed by NPL, the measured permittivity and conductivity are consistent with the target values as the difference between them is less than 5 %. The uncertainty evaluation for dielectric measurements of tissue equivalent liquids using an analytical approach and specific Matlab library Metas.Unclib is performed. The sources of uncertainty such as measurement repeatability, cable flexure, VNA imperfection (noise floor, trace noise, linearity, directivity ...) have been taken into account in the uncertainty budget of permittivity characterization. In fact, the uncertainty of complex permittivity measurements of liquids will contribute to the establishment of traceability in the E-field measurement.

The vector E-field probe calibration systems were developed to operate in several telecommunication frequency bands between 400 MHz and 6 GHz, using reference waveguides for the corresponding frequency bands. Matching windows for the different waveguides have been developed and the tightness and the good power transfer into the liquid (reflection coefficient less than -10 dB at the frequencies of

interest) have been verified. An automatic positioning system with mechanical supports for holding probes and waveguides were also developed allowing the E-field probe under calibration to be positioned in the different reference waveguides. A physical and analytical model of the calibration system were devised to establish the traceability of E-field and SAR measurements when using electro-optic vector E-field probes, by determining the vector correction factor of the probe (ratio between E-field measured by the probe and electro-optic converter voltage output). This is done by measuring the *S* parameters of the section between the reference waveguide input and the probe tip and second, of the whole measurement chain from the reference waveguide input to the electro-optical converter output. The numerical approach based on the Monte Carlo method were used for estimating the uncertainty of the vector E-field probe calibration. The estimation of uncertainty sources related to the measurement repeatability, probe positioning, and thickness of waveguide sections by measurements are in progress.

The E-field probe calibration system has been finalized in the WR187 waveguide frequency band (3.95 GHz to 5.85 GHz). In frequency bands between 400 MHz and 4 GHz, the realization of calibration systems using corresponding waveguides will be proposed as part of the maintenance of the references.

Contact: Thi Dao Pham Bui (thi-dao.bui@lne.fr), Djamel Allal (<u>djamel.allal@lne.fr</u>), Jean-Marie Lerat (Jean-Marie.Lerat@lne.fr)

#### Nontraditional impedance S parameter measurement: balanced impedance, extreme impedance

The aim of this project is to implement *S* parameter measurements traceability for nontraditional impedance domains, focusing on balanced impedance and extreme impedance.

Work on the second part, devoted to extreme impedances, and more generally to traceability of nanoscale S parameters measurements was on hold but it was able to start again in September last year with the recruitment of a PhD candidate in collaboration with IEMN-University of Lille.

These first months have allowed us to review the state of the art on the measurement of S parameters at the nanometer scale and to determine the resulting problematic, linked to the measurement of extreme impedances, both low and high, and the question of traceability that results from this. Work then began on the study of calibration techniques to guarantee this traceability, taking into account the dimensional scale constraint, and possibly the impedance domain to be treated. For the moment, the one-port standard oriented LRRM calibration technique, is considered, rather than transmission line based calibration techniques, such as TRL, which would lead to excessively large aspect ratios between the width and length of the patterns to be produced. Work has also begun on taper structures that will allow the transition from micrometric patterns compatible with the spacing of the measuring tips to nanometric patterns with at least a 1:100 ratio on the coplanar sections that will be produced. An on-wafer probe station recently developed at IEMN and allowing for nanometric translation control accuracy on X, Y and Z and micro-degree accuracy on rotation will be used to validate this work.

Contact: Djamel Allal (djamel.allal@lne.fr)

#### EMPIR project FutureCom (RF Measurements for Future Communications Applications)

The objective of this European project is to develop the metrology of RF components, circuits and subsystems operating under real operating conditions, where LNE is working on the evaluation of on-chip signal and power integrity, the development of a technique for characterizing power integrity by programming an FPGA circuit, and the evaluation of electromagnetic interference on a typical FPGA board. LNE is also working on the traceability of industrial-grade connectors, specifically the 4.3-10 connector.

Different structures were defined and they were designed based on thin-film microstrip line (TFMSL) technology, and simulated using 3D EM software. The structures are typically coupled lines with

variations such as trace width, trace spacing and the presence of aggressors of varying size and distance, in order to produce effects on the common and differential modes and on the conversion between modes. The chips are being manufactured at CNRS-LAAS to perform comparative measurements of signal integrity, power integrity and electromagnetic interference at the chip level among several project partners.

Concerning the evaluation at the level of FPGA PCBs, an experiment was set up to implement a VNA by programming an FPGA in order to evaluate the power integrity at the level of the power distribution network (PDN), by measuring their S parameters and therefore their impedance, and to analyse the effect of the decoupling capacitors mounted in parallel to the PDN traces. Thus, S-parameter measurements were carried out on several FPGA boards, where the FPGA applies currents and reads the corresponding voltages, then translates the ratios into impedance and S-parameters as a function of frequency. The second step was to set up a similar measurement, using a VNA and a comparative analysis is underway.

The work on traceability of 4.3-10 connectors consisted in the development of traceable adapters from Type-N to 4.3-10 with respective diameters of 7 mm and 10 mm. These adapters based on an air line structure do not contain any dielectric and are modelled using 3D EM simulation, based on dimensional characterisation of the different parts. Two adapters were made with a 4.3-10 male and female interface. The simulations are performed both on the adapters separately and also when they are interconnected (cascaded). De-embedding procedures are then used to characterise commercial adapters that allow any component with 4.3-10 connectors to be traceably measured.

Contact: Djamel Allal (djamel.allal@lne.fr)

#### EMPIR project TEMMT (Traceable measurements at millimetre-wave and terahertz frequencies)

This European project, starting in May 2019, aims to establish the SI traceability of three electrical quantities (S-parameters, high-frequency power and the complex permittivity of dielectric materials) at millimetre and terahertz frequencies. This traceability is important for many emerging applications, exploiting communications and electronics technologies - *e.g.* fifth generation (5G) mobile networks, Internet of Things (IoT), connected and autonomous vehicles (CAV), space-based radiometers for earth remote sensing, security images, *etc*.

The project has four main scientific and technical objectives that aim to ensure traceability: S-parameters of connected components (objective 1), S-parameters of components on wafer (objective 2), power measurement up to 750 GHz (objective 3), and measurements of material properties (relative permittivity) (objective 4).

For the first objective, after setting up the dimensional and electrical (S-parameter) measurement facilities for the 1.35 mm connector, the final step was to participate in an inter-laboratory comparison of S-parameter measurements with the NPL and the PTB. Each laboratory used a separate traceability path. The NPL used direct traceability via characterised air lines, the PTB used direct traceability *via* characterised air lines, the PTB used direct traceability *via* characterised offsets and the LNE used indirect traceability *via* a classical calibration kit and a characterised air line for a *posteriori* verification of the correct definition of the kit standards. The result of the comparison shows in general a better-expected agreement between the two direct traceability paths, and a deviation with the indirect method for the measurement of low values of S-parameters. This demonstrates the need to implement direct traceability in the laboratory.

For the second objective, LNE was to take part in an inter-laboratory comparison of S-parameters using the probes measurement system implemented in 2021 to cover the frequency band between 110 GHz and 170 GHz. Unfortunately, the network analyser broke down and was not repaired in time due to the shortage of electronic components, which prevented us from carrying out our measurements.

For the third objective, after developing and validating the primary power calibration means (microcalorimeter) from 110 GHz to 170 GHz in 2021, LNE took part in an international comparison at

secondary level in the following two frequency bands [110 GHz - 170 GHz] and [500 GHz - 750 GHz]. The participants in the comparison have measured successively a Rohde & Schwarz thermoelectric probe (Germany) and two VDI PM5 and PM5B calorimeters (USA). The LNE values from 110 GHz to 170 GHz and 500 GHz to 750 GHz demonstrate good consistency of results between the thermoelectric probe and the calorimeters.

Contact: François Ziadé (françois.ziade@lne.fr), Djamel Allal (djamel.allal@lne.fr)

#### High frequency power calibration: traceability and extension towards terahertz frequencies

This project, started in 2020, aims to consolidate and extend the traceability of average power measurements to terahertz frequencies (170 GHz). Given the scarcity of bolometric power probes and the difficulty in obtaining thermoelectric power probes compatible with our primary reference (microcalorimeter), this large-scale project should ensure the sustainability of the LNE's CMCs by developing and producing HF power probes based on thermoelectric technology. These new standards should be integrated into new primary (microcalorimeter) and secondary (HF power transfer setup) standards that will be developed and operational at the end of the project. In the framework of this project, LNE is developing power transfer standards as well as primary power standards (microcalorimeters): coaxial transfer standard in 1.85 mm connector and waveguide transfer standards (R620, R900, and R1400).

Regarding the development of a 1.85 mm coaxial power standard for the [DC - 67 GHz] frequency bandwidth, work in 2021 focused on improving the matching of the device without the 1.85 mm connector. Several designs have been implemented: to date, the best results in terms of matching have been obtained for a tractorial shape: the input reflection factor is less than 14 dB up to 40 GHz and then less than -18 dB from 40 GHz to 67 GHz.

During 2022 the research work focused on the development of rectangular waveguide power standards in the three frequency bands [50 GHz - 75 GHz], [75 GHz - 110 GHz] and [110 GHz - 170 GHz]. Several designs were developed and a first one selected for fabrication for the [110 GHz - 170 GHz] bandwidth. An innovative approach was explored by optimising the load value and characteristic impedance of the coplanar CPW line. The electromagnetic and thermal simulation results in the [110 GHz - 170 GHz] band are very promising since the reflection factor is lower than -15 dB and the efficiency higher than 0.8 over the whole frequency band and the DC voltage at the output of the sensor is close to 1 mV for an injected power of 10 mW. These theoretical results are comparable to the performance of a Rohde & Schwarz power sensor measured during the European TEMMT project. The first prototype will be built on a 200  $\mu$ m high-resistivity silicon (SiHR) substrate in 2023.

The year 2022 was also marked by the purchase of two frequency multipliers to cover the [50 GHz - 75 GHz] and [75 GHz - 110 GHz] frequency bands, as well as a new power source in a 1.85 mm connector. The latter enables calibrations to be carried out from DC to 67 GHz and thus meets the new broadband demands of industry.

Contact: François Ziadé (francois.ziade@lne.fr)

## **10** List of publications

D. RICHERT, J. MORAN-MEZA, K. KAJA, B. GAUTIER, A. DELVALLEE, D. ALLAL, F. PIQUEMAL « Traceable nanoscale measurements of high dielectric constant by scanning microwave microscopy » 2021, *Nanomaterials*, 11(11), article n°3104, 19p.

S. DJORDJEVIC, R. BEHR, D. DRUNG, M. GOTZ, W. POIRIER « Improvements of the programmable quantum current generator for better traceability of electrical current measurements » 2021, *Metrologia*, vol 58, n°4, 045005, 16p.

A. KAZEMIPOUR, J. HOFFMANN, M. WOLLENSACK, M. HUDLICKA. J. RUFENACHT, D. STALDER, D. ALLAL, G. GAUMANN, M. ZEIER « Standard Load Method: A New Calibration Technique for Material Characterization at Terahertz Frequencies » 2021, *IEEE Transactions on Instrumentation and Measurement*; vol 70; 10p.

K. KAJA, D. MARIOLLE, N. CHEVALIER, A. NAJA, M. JOUIAD « Erratum: Sub-10 nm spatial resolution for electrical properties measurements using bimodal excitation in electric force microscopy » 2021, *Review of Scientific Instruments*, Vol 92 ; n°4, Erratum, 049901n, 1p.

N. MEZIERES, B. FUCHS, L. LE COQ, J-M. LERAT, R. CONTRERES, G. LE FUR « On the Antenna Position to Improve the Radiation Pattern Characterization » 2021, *IEEE Transactions on Antennas and Propagation*, Vol 69, n°9, p5335-5344 ; 10p DOI: 10.1109/TAP.2021.3060894.

F. PIQUEMAL, J. MORAN-MEZA, A. DELVALLEE, D. RICHERT, K. KAJA « Progress in Traceable Nanoscale

Capacitance Measurements Using Scanning Microwave Microscopy » 2021, *Nanomaterials*, 11(3), article n°820, 22p.

K. KAJA, D. MARIOLLE, N. CHEVALIER, A. NAJA, M. JOUIAD « Sub-10nm spatial resolution for electrical properties measurements using bimodal excitation in electric force microscopy » 2021, *Review of Scientific Instruments*, Vol 92, n°2, 023703, 13p.

J. DOUMOURO, E. PERROS, A. DODU, N. RAHBANY, D. LEPRAT, W. POIRIER « Quantitative Measurement

of the Thermal Contact Resistance between a Glass Microsphere and a Plate » 2021, *Physical review applied*, Vol 15 , n°1, 014063, 8p DOI:10.1103/PhysRevApplied.15.014063.

N. MEZIERES, B. FUCHS, L. LE COQ, J-M. LERAT, R. CONTRERES, G. LE FUR « Fast Antenna Characterization Improvement by Pattern Rotations » 2021, *IEEE Transactions on Antennas and Propagation*, Vol 69, n°5, mai, 9234653, p2952-2957, 6p, DOI:10.1109/TAP.2020.3031479.

W. POIRIER, D. LEPRAT, F. SCHOPFER « A resistance bridge based on a cryogenic current comparator targeting sub-10-9 measurement uncertainties » 2021, *IEEE Transactions on Instrumentation and Measurement*, vol 70, 9143174, 1000314, 14p DOI 10.1109/TIM.2020.3010111.

D. MOULOUA, A. KOTBI, G. DEOKAR, K. KAJA, M. EL MARSSI, M. A. EL KHAKANI, M. JOUIAD, « Recent progress in the synthesis of MoS2 thin films for sensing, photovoltaic and plasmonic applications : A review » 2021, *Materials*, 14, 3283. https://doi.org/10.3390/ma14123283.

O. ŞEN, M. ÇINAR, A. KRIZ, T. PAVLÍČEK, J.M. LERAT, M. WOJCIECHOWSKI, B. PINTER, M. RODRÍGUEZ and

F. PYTHOUD, « EURAMET supplementary comparison on calibration of RF current monitoring probe » 2021 *Metrologia*, Volume 58, Number 1A, https://iopscience.iop.org/article/10.1088/0026-1394/58/1A/01004.

A. KOTBI, M. IMRAN, K. KAJA, A.RAHAMAN, EL M. RESSAMI, M. LEJEUNE, B. LAKSSIR, M. JOUIAD, « Graphene and g-C3N4-Based Gas Sensors », 2022 *Journal of Nanotechnology*; Vol 2022 ; Article n°9671619 ; 20p ; https://doi.org/10.1155/2022/9671619

M. OUAMEUR, B. ABDOURAHAMANE KASSOUM, F. ZIADÉ, AND D.ALLAL, « Measurements of AC Resistances up to 10 MHz using a VNA and Calculable Adapters», 2022 *IEEE Transactions on Instrumentation and Measurement*, vol 71, DOI : 10.1109/TIM.2022.3193729

M. OUAMEUR, R. VASCONCELLOS, M. AGAZAR, O. THÉVENOT AND E. PATOIS, "New impedance bridge at LNE for the two terminal-pair AC resistor calibration up to 20 kHz", 2022 *Metrologia*, vol 59, n°5, DOI: 10.1088/1681-7575/ac8485

S. MAŠLÁŇ, G. HINDS, M. OUAMEUR AND F. OVERNEY, "Project LiBforSecUse: Quality assessment of electric vehicle Li-ion batteries for second use applications", 2022 *Cal Lab: The International Journal of Metrology*; vol 29 ; n°2 ; April May June ; p22-31 ; 10p

N. S. RAJPUT, A. KOTBI, K. KAJA AND M. JOUIAD, "Long-term aging of CVD grown 2D-MoS2 nanosheets in ambient environment (NPJMATDEG-00597R)" 2022 *npj Materials Degradation* 6:75; https://doi.org/10.1038/s41529-022-00288-4

D.-H. CHAE, M. KRUSKOPF, J. KUCERA, J. PARK, N. THANH MAI TRAN, D. B. KIM, K. PIERZ, M. GÖTZ, Y. YIN, P. SVOBODA, P. CHROBOK, F. COUËDO AND F. SCHOPFER, "Investigation of the stability of graphene devices for quantum resistance metrology at direct and alternating current", 2022 *Measurement Science and Technology*; Vol 33 ; art n°065012 ; 11p

M. NASR, L. BENHAMOU, A. KOTBI, A-I. LAHMAR, K. HOUMMADA, K. KAJA, "Photoelectrochemical Enhancement of Graphene@WS2 Nanosheets for Water Splitting Reaction", 2022 *Nanomaterials*; 12(11); art n°1914; 14p; https://doi.org/10.3390/nano12111914