Report on the activities in Electricity and Magnetism within the LNE between 2015 and 2017

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This report gives a brief outline of the main research and development activities in the field of electricity and Magnetism since March 2015 at the Laboratoire National de Métrologie et d'Essais (LNE).

1 Kibble balance

Measurements of the Planck constant have been performed during the first quarter of 2016 in the framework of the CCM pilot study. The Kibble balance operated then in air with a 500 g platinum–iridium mass standard. The Planck constant value obtained deviates from 2014 CODATA value by 1.9 parts in 10^7 with a standard uncertainty of 1.4 parts in 10^7. The main uncertainty components (voltage measurement, mechanical alignment, noise level, velocity measurements during the dynamic phase) have been investigated.

A new measurement campaign of Planck constant is ongoing with the Kibble balance operating in vacuum. Efforts will be geared toward further improving of voltage measurement and on maintaining alignment during air to vacuum transition.

Furthermore, a new system for the dissemination of the mass unit in France has been proposed. This relies on a primary realisation using the LNE Kibble balance with three 500 g mass standards made from platinum–iridium alloy, pure iridium and Udimet 720 (a nickel alloy) respectively, coupled with a pool of kilograms made from different materials.

Contacts: Matthieu Thomas (matthieu.thomas@lne.fr); François Piquemal (francois.piquemal@lne.fr)

2 Capacitance metrology

Thompson-Lampard calculable capacitance standard

The "Lampard" structure of the standard has been significantly modified. The two plates supporting the electrodes have been redesigned to implement a new prepositioning stage. In order to allow an optimum assembly of the different mechanical parts, position locking devices have been added. This will guarantee a better position of the electrodes after mounting, more favorable to realize the final adjustment of the cavity. The pre-positioning step makes it possible to align the electrodes on the reference shape. This operation consists first of all in centering the measuring machine on the reference shape. Then the top and the bottom of the electrodes are directly aligned with the reference shape without taking into account the machine defects. The pre-positioning settings are then locked. The first tests showed that the pre-positioning of the electrodes is achievable within 5 µm from their final positions; this point is consistent with the objective.

Regarding the submicrometric positioning, machine defects and defects in the shape of the electrodes must be taken into account. This positioning is carried out in two stages. We first use the reference shape as a

"reference" to align the top of the electrodes. The rotation of the measuring ring makes it possible to use a multi-reversal method to dissociate the positioning defects of the electrodes from that of the machine. This adjustment makes it possible to position the "top" center of the electrodes on a regular pentagon to within about 10 µm. The same method is used to align the "bottom" centers of the electrodes on the "high" centers. The alignment of the electrodes constituting the Lampard cavity is achievable within 100 nm, the position of the electrodes is then locked.

To improve the quality of the displacement of the mobile carriage supporting the measuring machine, the trapezoidal drive screws have been replaced by screws having a better straightness. The mobile guard was assembled with a temporary end (devoid of star screen). This first version was installed, and allowed to test its centering devices from the measurements made with its capacitive ring. The accessible positioning accuracy is better than 1 µm, which is a very encouraging result for the final positioning (at 100 nm) realized by the rotation of the star screen. Moreover, the various actuators associated with the mobile guard have been implanted.

Development of a programmable standard of ultra-low capacitance values

A set of ultra-low value capacitance standards together with a programmable coaxial multiplexer have been developed. The multiplexer enables the connection of these capacitances in parallel configuration and they together form the programmable capacitance standard. It is capable of producing decadic standard capacitances from 10 aF to at least 0.1 pF, which are later used to calibrate commercial precision capacitance bridges. This programmable standard has allowed one to calibrate Andeen-Hagerling AH2700A bridges with a maximum uncertainty of 0.8 aF for all the capacitances generated ranging from 10 aF to 0.1 pF, at 1 kHz. Sources of uncertainties of the programmable capacitance standard, such as parasitic effects due to stray impedances, have been evaluated and a method has been developed to overcome these hindrances. This work was a part of the EMRP AIM QuTE project.

Contacts: Olivier Thévenot (olivier.thevenot@lne.fr); Gaël Thuillier (gael.thuillier@lne.fr); Mohammad-Saïf Khan (mohammad-saif.khan@lne.fr)

3 QHE metrology

LNE has continued the investigation of the quantum Hall effect (QHE) in Hall bar devices made of graphene grown by propane/hydrogen chemical vapor deposition on silicon carbide. In a large sample (100 µm x 420 µm) with low electron density (1.8 10^{11}cm^{-2} at low temperature), the complete scanning of the experimental conditions (magnetic flux density B, temperature T and current I) required for quantization of the Hall resistance R_H, at Landau level filling factor of 2 and with accuracy to within 1 10^{-9} or below (down to 8.2 10^{-11}) was achieved. This study has revealed a broad range of conditions where the graphene device operates as a reliable and accurate resistance standard, largely surpassing a typical GaAs/AlGaAs device (LEP 514): minimum operation magnetic flux density of 3.5 T (three times smaller than in LEP 514), maximum operation temperature of 10 K (six times higher than in LEP 514), maximum operation current of 0.5 mA (ten times higher than in LEP 514). Interesting working points (5 T, 5 K, 50 µA) or (6 T, 5 K, 160 µA) compatible with simple and compact helium-free cryomagnetic systems are accessible for this versatile graphene-based standard. Moreover, the device has shown all the properties of an ideal quantum Hall resistance standard (low contact resistance <1 Ω, homogenous low dissipation R_{xx}<30 µΩ (down to a few µΩ), spatial homogeneity of R_H within 1x10^{-9}, invariance of R_H with B-field direction within 1 10^{-9}). Up to around ten devices obtained from two distinct growths were tested, which all show excellent contacts, and R_H quantization with accuracy to within 10^{-9} in experimental conditions similar or relaxed as compared to those required for typical GaAs/AlGaAs devices. The graphene devices have also demonstrated very robust properties over three-year investigation with many thermal cyclings. Attempts to reduce the operation magnetic flux density by reducing the carrier concentration using Corona

discharge have revealed a degradation of Hall quantization properties, notably lower breakdown current values. This suggests the improvement of the material quality and graphene devices fabrication to reach lower operation magnetic flux density around 1 T. Finally, ongoing studies focus on identifying the low-dissipation mechanism at play in the tested graphene devices and the structural key parameter for accurate quantization.

Contacts: Wilfrid Poirier (wilfrid.Poirier@lne.fr); Félicien Schopfer (felicien.schopfer@lne.fr)

4 Quantum ampere

The ampere, once defined from *e*, can be realized using quantum effects, either by using single electron tunnelling devices or by applying directly Ohm's law to the quantum voltage and resistance standards. In this context, the LNE has developed a novel programmable quantum current generator (PQCG) by applying Ohm's law in an original circuit combining the Josephson voltage and quantum Hall resistance standards with a cryogenic current comparator (CCC). More precisely, an accurate reference quantized current is defined by polarizing a multi-connected quantum Hall resistance standard with the Josephson voltage. Detected by the CCC, it is then used to servo-control an external current source. We have demonstrated that the current generated in the milliampere range is quantized in terms of ef_J (f_J is the Josephson frequency) within one part in 10^8. Able to deliver current down to the microampere range with such an accuracy by changing the CCC amplification gain, the PQCG can improve the calibration and measurement capabilities (CMCs) declared by national metrology institutes in the current field by two orders of magnitude. This new current generator has all the properties of an ideal quantum standard: it benefits from the universality and reproducibility of the quantum Hall effect and the Josephson effect, as well as of the existence of quantization criteria that can be quickly checked. It was efficiently used to calibrate digital ammeters with record uncertainty only limited by the apparatus under test. This research constitute a first step towards new applications in metrology: an AC current quantum standard, a quantum ammeter and even a compact quantum calibrator (voltage, resistance, current) if combining the Josephson standard with a graphene-based resistance standard operating in relaxed experimental conditions.

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

5 Electrical nanometrology

The eSPM based activity dedicated to the traceable measurement of electrical quantities at the nanoscale was focused on the coordination and developments of SolCell EMRP project and 3D Stack EMPIR project. The first is aiming to develop accurate and spatially resolved metrology to determine the complete material properties layer by layer of multi-junction solar cells (MJSC) based on III-V materials. The second is addressing the measurement challenges related to the 3D integration of heterogeneous semiconductor technologies which uses copper Through Silicon Vias (TSV) to electrically connect a stack of chips-bonded semiconductor wafers and dies.

In Solcell project, a GaAs staircase dopant density sample developed by LAAS (Toulouse, France) has been successfully measured by LNE, METAS and Keysight-Austria using SMM techniques. A clear contrast between the different layers is visible. Value of plateaus corresponds well both with the expected dopant densities and was found in good agreement with SIMS measurements carried out at CEA-LETI in Grenoble. Furthermore an improved model for non-local band-to-band tunneling carrier transport has been proposed and compared to experimental measurement from GaAs tunnel junction devices. The model suggests that elastic band-to-band tunneling instead of trap-assisted-tunneling is the predominant mechanism in GaAs tunnel junctions, which is of great interest for better understanding and improving III–V multi-junction solar cells.

Within 3DStack project, first tests on TSV with conductive AFM (Resiscope) have been carried out and a 150 mm probe station fitted with four positioners (1 µm resolution) and four coaxial probe arms for I-V and C-V measurements has been installed.

Contact: François Piquemal (francois.piquemal@lne.fr)

6 Power and Energy

Since June 2014 LNE is involved in the development of "Measurement Tools for Smart Grids Stability and Supply Quality Management" in the framework of the 2013 EMRP Energy call. In this framework, the former developments for the reference PMU are being evolved in order to deal with real exploitation conditions of actual smart electrical grids. New algorithms dealing with the requirements of IEEE C37.118-2011 standard for dynamic conditions have been implemented and tested against a PMU calibrator developed by METAS for both static and dynamic tests conditions. The complete metrological characterization of the reference PMU is now under way.

More recently, LNE contributes in two other European projects:

- The first one corresponds to the EMPIR project "TracePQM": Traceability routes for Power Quality Measurements" within 2015 EMPIR call. This project aims at developing a modular metrological system for power and power quality measurements on a frequency domain ranging from DC to 1 MHz. It relies on the implementation of sampling methods for achieving these goals. A control software platform is also developed for the management of individual devices and data processing tools. LNE is then in charge of transferring its knowledge on the measurement of power and power quality parameters owing to sampling techniques in the low frequency domain.
- The second project (untitled MEAN4SG) is supported by H2020 programme and has started in March 2016. The objective is to educate 11 young researchers in the smart grids metrology field by constructing a training network gathering the whole innovation value chain. LNE is particularly involved in the development of tools for accurate and reliable measurements of Smart Grid Power Quality.

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

7 High Voltage/High Current metrology

The LNE activities in this field rely on several European and national projects.

LNE participates to the EMRP project "Future Grid" with the aim of supporting wider application of novel sensor technologies in future power networks. LNE is involved in the development and characterization of precision optical sensors. The key challenges are to achieve traceable calibration with a measurement uncertainty of better than one part in 104 for 0.1 % to 120 % of a rated current of 10 kA under field conditions. The existing Fiber Optic Current Sensors (FOCS) are used for much higher current levels (hundreds of kA) in order to achieve the maximum performances and sensitivities. The project approach aims at increasing the sensitivity of the designed FOCS by working at lower wavelength (632.8 nm is chosen). LNE accomplished the characterization of the selected sensing fiber. The custom spun optical fiber was developed especially for the project by the University of Southampton. Measurements of the Verdet constant of the single mode spun low birefringence optical fiber are reported. The experimental setup to study the temperature influence on the Verdet constant was designed and characterized. The temperature dependence of the Verdet constant from -20 °C up to +60 °C was determined.

In the framework of the EMPIR prenormative research project focused on the development of techniques for ultra-high voltage and very fast transients, LNE, with support from other institutes (CEA-Saclay, GE Medical Systems and LCOE) will develop the traceability of the voltage and the current of pulsed X-ray

generators used in diagnostic applications such radiology, mammography, dental, fluoroscopic and others. The voltage is up 150 kV, the current under high voltage is up to few amperes and the duration of the pulses could be from 200 µs to few seconds. High voltage dividers and low current probes have been selected and purchased by LNE. Their characterisations are in progress by LNE, CEA, GEMS and LCOE.

Within a national project, an accurate method has been developed for the calibration of high voltage transformers for the frequencies between 15 Hz and 400 Hz including the railway frequency of 16 Hz2/3. It consists of using a resistive divider for voltage up to 100 kV or a capacitive divider for voltage up to 300 kV as standards to determine the ratio error and the phase displacement of high voltage transformers. The measurements are based on sampling and Discrete Fourier Transform processing techniques. Because of the industrial needs, the targeted voltages are limited to 300 kV for 50 Hz, 150 kV for 60 Hz and 35 kV for (15- 400) Hz. The uncertainties of measurement are better than 25 µV/V for ratio error and (0.35×f + 5) µrad for phase displacement (f is the frequency of the signal in Hz).

A new LNE project has been started in 2016 to elaborate the traceability of high impulse voltages. The first objective is to develop a new standard, according to the new requirement of IEC 60060-1 and IEC 60060-2, for the calibration of fast transients including lightning impulse (1.2/50 µs) and switching impulse (250 /2500 µs) for voltage up to 500 kV with targeted uncertainties of less than 0.3 % for peak value and less than 1 % for time parameters. The second objective is to develop a new standard for the calibration of very fast transient with rise time in the range of 100 ns for voltage up to 400 kV, the targeted uncertainty is less than 5 % for the peak value and less than 5 % for time parameters. For this purpose, a new sampler has been characterized and its performances fulfil the expectations. The on-going acquisition of a high voltage impulse generator will enable to characterize soon the LNE high voltage divider (GARY) for the calibration of very fast transient.

Contacts: Mohamed Agazar (mohamed.agazar@lne.fr); Daniela Istrate (daniela.istrate@lne.fr)

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

Electrical current shunts are widely used as a resistance standard in metrology laboratories and precision instruments with the aim of measuring high level of alternative and direct currents (AC and DC) with high accuracy. These AC current sensors are characterized by a broadband operating frequency bandwidth and are commonly calibrated up to 100 kHz. The use of a current shunt requires the preliminary knowledge of its magnitude deviation from DC and phase angle according to the frequency. The determination of these parameters in high frequency, typically beyond 100 kHz, is challenging and existing current shunts exhibit a strong variation in magnitude and phase. The best magnitude performances of standard shunts used in National Metrology Institutes are limited to 2 µΩ/Ω at 100 kHz. Since 2015, LNE has developed a completely calculable current shunt standard based on theoretical basis and innovated design suitable for current measurements at 10 A up to 1 MHz. All the work done so far has been focused on the design as well as the thermal and electromagnetic modelling of the shunt. The frequency variations as well as the temperature coefficient of the developed shunt are completely calculable. Theoretically, the relative deviation from DC is less than $2 \cdot 10^{-6}$ µΩ/Ω and $2 \cdot 10^{-4}$ µΩ/Ω in magnitude at 100 kHz and 1 MHz respectively and the temperature coefficient is below 10 parts in 10^6. This new device makes possible the measurement of high levels of currents up to the megahertz range and the calibration of current sensors.

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

9 RF and MW metrology

The LNE R&D activities in the RF and MW domain mainly include contributions in three 3-years projects, the two first being funded by EMRP programme and were completed in 2016.

HF-Circuits: "Metrology for new electrical measurement quantities in high-frequency circuits"

This project was focused on the traceable characterization of Vector Network Analysers (VNA) and calibration standards for S parameters measurement in order to extend the European capability in terms of calibration with special focus on frequency extension (up to 1.1 THz in waveguide and up to 110 GHz in coaxial line), on mixed mode, extreme impedance and nonlinear measurements and on update of uncertainty evaluation procedures. Since 2015 LNE worked on establishing traceable measurement in 1 mm coaxial line up to 110 GHz, by characterizing dimensionally a full set of offset shorts and propagating their uncertainties to the device under test through the VNA calibration process. LNE worked also on the estimation of mixed mode S parameter calibration standards uncertainty by performing 3D EM simulated sensitivity study.

MORSE: Metrology for Optical and RF Systems

This project was dedicated on metrology for the wireless, optical and satellite communication parts of the network, covering all aspects from the network-edge to the network-core like the development of measurement of radiated RF power and Over-The-Air testing for 4G with several and even reconfigurable antennas configurations and the development of applications for improved efficiency in terms of time and accuracy, of systems such as the work on test-time reduction using modelling and interpolation and the development of a non-invasive E-field sensor to test small antennas at high frequencies. Over the last two years, LNE worked on developing a measurement standard for Long Term Evolution (LTE) signals, and on developing an improved antenna test using combined measurement and modelling. LNE worked also on developing a compact antenna test range defect compensation algorithm and on developing a measurement system of antennas over a controllable temperature range.

Metrology for mixed-mode on-wafer S parameter

This national project has started in 2016 with the objective to develop the calibration standards and the methods to establish traceable measurement of mixed mode S parameters on wafer, including the differential and common modes and the two corresponding mode conversions. 3D EM simulations have been carried out to design the planar calibration standards for both the ground-signal-signal-ground (GSSG) and ground-signal-ground-signal-ground (GSGSG) configurations. The calibration standards are basically dual lines of different lengths for both configurations. The set of standards includes also different reflect standards, in order to perform the multimode thru-reflect-line (TRL) calibration. A program performing the multimode TRL calibration procedure has been written in Matlab. A capacitive model of both configurations, together with self-determination of the propagation constant have also been established to allow for the determination of the common and differential mode characteristic impedances that will provide the traceability of the measured S parameters to SI units.

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

10 List of publications

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[16] Istrate D, Etienne R, Dubard J, Litwin A, and Enouf O 2016 Determination of the Verdet Constant of Low Birefringence Single-Mode Optical Fiber *Proceedings of Conference CPEM'2016*, Ottawa, 11-15 July, 2016

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