Report on the activities in Electricity and Magnetism within the LNE between 2017 and 2019

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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 2017 at the Laboratoire National de Métrologie et d'Essais (LNE).

1 Kibble balance

The Kibble balance (KB) experiment developed at LNE in collaboration with the CNAM and the LNE-SYRTE has obtained a value of the Planck constant with a relative standard uncertainty of 5.7 parts in 10^8 in 2017. This was the fourth best KB value of *h* taken into account by the CODATA for the calculation of the fixed value of *h* and the best value with a system operating in air. From this result, LNE has decided to continue the developments on the KB to realise the kilogram with the aim at reducing the uncertainty at the level of a few parts in 10^8. Improvements of different parts of the set-up are on-going.

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2 Capacitance metrology

Thompson-Lampard calculable capacitance standard

It has been shown that the positioning system of the electrodes allowed to construct the cavity of the calculable capacitor with the required accuracy (100 nm). It was found that the mobile guard when positioned in the standard cavity displaced the electrodes by a few micrometers. This phenomenon was reversible, when the movable guard returned to its rest position, the electrodes returned to their adjustment position within a few tens of nanometers. After many investigations, the origin of the problem was finally located in the main electrode assembly between the electrodes and their extensions. This is a machining defect on the extensions in the contact area with the central part of the electrodes. This defect prevented a correct support between the two parts, resulting in a lack of rigidity. The extensions have been modified to correct this defect. Measurements made after this last modification showed that the observed defect was no longer detectable. This change has been taken into account for the final extensions currently being manufactured. Once this problem was solved, the various tasks still to be carried out were taken over.
The external electrical screens were designed and installed on the standard. Initial electrical measurements using an automatic capacitance bridge verified the correct functioning of the standard in its current version at atmospheric pressure. The interferometer implementation scheme is currently being finalized. The polishing of the last electrodes is nearing completion.

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3 QHE metrology

In 2015, LNE reported the realization of the quantum Hall resistance standard in graphene grown by propane/hydrogen chemical vapor deposition on silicon carbide with state-of-the-art performances (Nature Nanotech, 10, 965 (2015)): accuracy to within $1 \cdot 10^{-9}$ (down to 8.2 10^{-11} (1σ)), relaxed experimental

conditions (magnetic field B down to 3.5 T, temperature up to 10 K, measurement current up to 0.5 mA). Over the last two-year period, LNE has continued the investigation of this technology.

The first objective was to test the reproducibility of the properties of these devices obtained through collaboration with CRHEA – CNRS and C2N – CNRS/Université Paris-Saclay. 10 Hall devices with various widths (20 μm, 100 μm, 200 μm) were fabricated from 4 graphene wafers grown over 4 years, in slightly different conditions. All the samples demonstrated low charge carrier density of a few 10^{11} cm^{-2}, good contacts with low resistance of about 1 Ω, and accurate quantization of the Hall resistance at $h/2e^2$ to within $2 \cdot 10^{-9}$, at $B < 10$ T, $T = 1.3$ K and current $I \geq 50$ μA (up to 0.8 mA in a 200 μm-wide device). On a given wafer, device to device reproducibility was demonstrated. Nevertheless, Hall resistance quantization was more or less robust against increasing temperature or current depending on the wafer. These results show a certain degree of reliability of the technology developed.

Besides, the devices have shown excellent stability at low temperature. A drift of the charge carrier density towards higher values was observed during storage at room temperature, without any irreversible degradation of the quantization properties. This points to the necessity of an efficient control of the carrier density to get full Hall resistance quantization at low magnetic field. Though not ideal, electrostatic doping modulation by Corona discharge was realized.

The second objective was to investigate the underpinning physics of the devices and identify the (structural) key control parameter for robust and accurate Hall quantization as well as associated simple electrical criteria. i) Carrier density dependence of the carrier mobility suggests that short-range scattering is dominant. ii) Hall resistance at zero magnetic field, expected to be zero, is finite in those samples and can be explained by anisotropic scattering at SiC step edges. iii) In addition the carrier density dependence of this Hall offset suggests an effect of the inhomogeneity of the carrier density. iv) At constant Landau level filling factor, in a given sample, the maximal quantum Hall breakdown current decreases with the density decreasing and equally points towards an effect of inhomogeneity. v) In two different samples, at a given density, the higher the mobility is, the higher the breakdown current. vi) The magnetic field dependence of the Hall resistance plateau at $h/2e^2$ together with that of the breakdown current can be explained with a model of charge transfer through the interface layer ($6\sqrt{3}x6\sqrt{3}$- R30° reconstruction layer) between the graphene layer and the SiC substrate. vii) To end, the usual variable range hopping (VRH) models expected to describe the temperature and current dependences of the longitudinal conductivity are questioned possibly by particular short-range scattering and/or dielectric environment. viii) The observation of a transition between two VRH models in certain samples suggests a change in the screening properties correlated to charge transfer through the interface layer. This bundle of experimental clues suggests that the key control parameter is the quality of the interface between the graphene layer and the SiC substrate. Additional morphology characterization of this interface is required to confirm this assumption. Already, a low value of the Hall resistance at zero magnetic field and high carrier mobility appear to be simple electrical criteria for robust and accurate Hall resistance quantization.

In the wake of the development of the quantum Hall resistance standard, LNE carries on exploiting the quantum Hall effect in graphene for quantum metrology experiments through the development of an avalanche detector based on the breakdown of the quantum Hall state for sensing single electrons or more generally elementary excitations. This includes measurement of the electronic noise at high frequency (~100 MHz). Such a detector, with expected easier operation in graphene, is intended to be utilized in single-electron interferometers based on quantum optics concepts, within the perspective of ultra-sensitive electromagnetic quantum sensors. This work is carried out in the frame of the EURAMET project SEQUOIA 17FUN04.

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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. The quantum standard is able to generate 10^{-8}-accurate currents in the range from 1µA up to 10 mA. In 2018, new experiments have been carried out to improve the quantum current generator in order to achieve relative uncertainties lower than 10^{-8} and extend the current range. Optimization of the ground reference position and cooling of the damping circuit of the CCC have allowed reducing not only the noise peak around 1 kHz caused by the damping circuit by a factor of ten but also the low-frequency noise below 10 Hz by a factor of three, at a level close to that of the CCC unconnected. Taking advantage of these improvements, calibrations of digital ammeters, HP3458A and Fluke 8508A, were performed. They allowed comparing the accuracy and stability of current measurements of these devices over several ranges. In the frame of the EMPIR project e-SI-Amp, a comparison of measurement of currents between LNE and PTB was carried out. The LNE quantum current generator was used to supply an ultra-low noise current amplifier (ULCA) developed and calibrated by PTB. Preliminary data analysis shows an agreement of current measurements to within relative uncertainties below 10^{-6}.

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5 Electrical nanometrology

The LNE has recently launched an internal project aimed at developing metrological tools especially for scanning microwave microscopes (SMM) which are commonly used for local impedance measurements at nanoscale. It is planned to manufacture shielded tips and reference structures, to implement a Mach Zender interferometer in the electronic circuit connecting the tip and the VNA, and to develop calibration methods taking into account more complete models than at present. The reduction of local capacitance measurement uncertainties to a few % is possible under optimal conditions for a value range from a hundred attofarads to ten femtofarads. After having developed and validated a calibration method, first repeatability tests of capacitance measurements were carried out on a reference structure composed of 48 nanocapacitors. The results show a repeatability characterized by a standard uncertainty lower than 1% for capacitance values higher than 1 fF and between 1% and 10% for capacitance values between 1 fF and 100 aF.

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6 Power and Energy

Since 2016, LNE actively participates to H2020-Metrology Excellence Academic Network for Smart Grids (MEAN4SG) project. Aiming at the quantification and the reproduction of the harmonic emissions between 2 kHz and 150 kHz in smart grids, LNE developed a metrology grade calibration platform. This platform can be used for the generation, acquisition and real-time processing of distorted waveforms (up to 250 V and 50 A with harmonics and interharmonics up to 150 kHz in voltage and current signal) and for PQ measurements system in severe conditions.

LNE contributes also to the TracePQM EMPIR project which aims at developing and validating a modular metrology grade system for the measurement of power and power quality (PQ) parameters using digital sampling techniques. These techniques can ensure both the lowest possible uncertainties and the highest possible bandwidth using commercially available components. LNE collaborates in designing of the

measurement setups for low and high frequency and in developing the open software tool for instrumentation control, data acquisition and the calculation of PQ parameters with full uncertainty estimation.

Since September 2017, LNE is involved in 16ENG04 project “Metrology for smart energy management in electric railway systems” (MyRailS). LNE and FFII are developing the calibration systems of the Energy Measurement Functions working under AC supply system. The LNE focuses mainly on the development of a fictive power source to generate voltage of 15 kV-16.7 Hz and 25 kV-50 Hz and current waveforms of 500 A with harmonics (only in the current) up to 5 kHz. Solutions are implemented to generate high level harmonics (more than 50% of the fundamental) and high order harmonics (up to the order 100) reproducing the harsh on-board train conditions. The results up to now allow generating the phase-fired waveform defined in the standard EN 50463-2 up to 100 A with an harmonic content up to 5 kHz. The uncertainty target for laboratory and on-board calibrations is 0.5 % for AC energy measurements under distorted waveforms.

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7 High Voltage/High Current metrology

X-ray HV measurements

LNE has developed a reference system for the calibration of the Practical Peak Voltage (PPV) which is the quantity adopted by IEC 61676 for the qualification of X-ray generators in radiology diagnostic. The PPV is based on the concept that the radiation generated by a high voltage of any waveform produces the same air kerma contrast behind a specified phantom as a radiation generated by an equivalent constant potential. In X-ray diagnostic application, the voltage level is up to 150 kV, the time duration is varying between 50 µs (fast transient overvoltages) to several seconds (temporal overvoltages), the rise time is varying between 10 µs and 500 µs, the waveforms are usually superposed by DC voltage, spikes and a high frequency ripples up to 200 kHz. Two paired and frequency-compensated invasive resistive dividers, for anode and cathode voltages, connected between the x-ray generator and the x-ray high voltage tube and combined with a storage digitiser with a minimum of 12 bits resolution, are used as a reference for the evaluation of the high voltage pulses. The linearity of the system has been checked with lightning impulse, switching impulse, chopped switching impulse and DC chopped impulse according to the IEC 60060-2. The step response and the frequency linearity have been also checked at low voltage to ensure the correct measurement of ripples and spikes in the voltage waveforms. Temperature and proximity effects have been performed for in-situ calibrations to occur a best accuracy. The frequency non linearity could be corrected by using a deconvolution technic (step response, FFT and Inverse FFT). Finally a technical procedure to measure with accuracy this large type of pulses using one reference system has been proposed.

Impulse HV testing

New reference systems for the calibration of high voltage impulses have been developed at LNE to reach the new requirements of the new IEC60060-1&2. It is composed by two high voltage dividers; the first one is a damped homemade resistive divider for lightening impulse measurement for voltage up to 420 kV. The second one is a mixed resistive capacitive divider for switching impulse measurements for voltage up to 500 kV. Each divider is associated with a matched 75 Ω transmission cable of 20 meters lengths, a high precision digitizer and validated software for signal processing. For calibration purpose, LNE has purchased a high voltage generator 400 kV/20 kJ. Each high voltage divider has been fully characterized at LNE. Both dividers present excellent metrological characteristics. The digitizer has a 75 MHz bandwidth, 200 MHz sampling frequency and 14 bits vertical resolution. It is composed by two channels; each one has 24 ranges for voltage up to 2000 V. It has been fully validated and calibrated at

LNE according to IEC 61083-1 for all the ranges and channels. The software has been validated at LNE according to IEC 61083-2 for all the typical wave forms. A test procedure has been developed at LNE for accurate measurements especially for in-situ calibrations in 5 successive steps; measurements of radiofrequency disturbances, step response of voltage dividers, measurement the scale factors of voltage dividers, calibration of digitizer's channels and ranges, measurement the linearity of voltage divider under calibration up to maximum voltage.

Calibration of combined high voltage and high current sensors with very low output voltages

In order to respond to a new industrial need, LNE has developed a new reference system for the precise calibration of combined high voltage and high current sensors at 50 Hz and 60 Hz with very low output voltages. The lowest measurable voltage is 100 µV and the highest is up to few volts. The phase shift between the output and the input could take any value from 0° to ± 180 °. New Fully automated method has been proposed and developed. The calibration of the combined transformers is performed in controlled temperature chamber by comparison to a standard current sensor and a voltage transformer. Both of them are calibrated in LNE with uncertainty of 3 parts in 10^5 for the scale factor and 30 µrad for the phase-shift. High voltage up to 35 kV is supplied using a step up voltage transformer 110 V/ 35 kV. High current up to 1000 A are supplied using a step-up current transformer 130 V/ 1000 A. The needed energy is supplied by a controlled power sources and could supply power up to 6 kVA. The output voltages coming from the combined transformer and the standards are sampled using two sampling voltmeters using DC reads with at least 20 bits of vertical resolution. The samplers are processed by a DFT algorithm allowing the possibility to have access to the amplitude and the phase of the signals. Because, the number of samples could not be infinite, a PLL is used to avoid the truncation errors. Software has been developed for fully automated procedure. It includes a validated algorithm for the processing and the calculation of combined transformer parameters. Switches have also been developed and implemented before the sampling voltmeters in order to perform in the same time measurements of current and voltage. The uncertainties of measurement are 0.5 % for the scale factor and 0.5 mrad for the phase shift when the output voltage is equal to 100 µV. They are better than 0.01 % for the scale factor and 0.01 mrad for the phase shift when the output voltages are equal to 10 mV.

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8 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 based on theoretical basis and innovated design: at 1 MHz the phase shift and transposition deviation are -0.01 mrad and 15 ppm respectively. 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 analyzer, allows the AC-DC deviation and impedance phase of a shunt to be measured simultaneously with relative uncertainties less than 1.10^{-3} at 1 MHz.

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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 EMPIR programme.

Vector SAR: SAR measurement using vector probes

The objective is to establish traceability of vector SAR measurement systems, especially systems using vector probe arrays in line with the very soon publication of the new IEC 62209-3 standard. In collaboration with the project partners, work has been carried out for developing in-situ measurement of the complex permittivity of sealed phantoms and providing high accuracy reference antennas with usable bandwidths exceeding 30 % with computed field distributions (magnitude and phase) and 1 g and 10 g SAR values for head (SAM) and body (box phantom) that will be verified by measurements on different facilities. A test protocol has been prepared and based on a set of measurands which are able to describe the complex field distributions on a 2D plane resulting from a given device under test (DUT) and its similarity to other distributions, in order to verify the reliability of different principle and technology measurement systems. Finally, a set of waveguide based calibration systems covering the main telecommunications frequencies between 400 MHz and 6 GHz is being set up for both scalar and single vector probe calibration, together with an automated probe positioning system for phantom embedded 2D EM field scans.

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ADVENT: Metrology for advanced energy-saving technology in next generation electronics applications

A first objective is to develop the techniques and systems which allow the traceable calibration of silicon embedded power sensors to be used typically in 5G ICs. Two schemes of traceability are set up; using a connectorised power comparison system, traceable to calorimetric techniques and using a direct on-chip calibration, using an embedded power comparison system, where the first scheme of traceability validates the second one for which the embedded power standard is designed and fabricated on chip. Consequently, an on-chip thermal RF power sensor has been designed and near to be fabricated, for use as an on-chip RF power standard in order to demonstrate the feasibility of such traceable mm-wave power measurement at the chip level. A second objective is to develop a non-invasive technique to traceably monitor the power consumption in a PCB. This technique is based on an electromagnetic (EM) near-field scanner that is used for non-invasive contactless measurement of the PCB and estimation of the location of unexpected power losses by heat or radiation, for production tests or prototype development. Several reference PCBs (connectorised transmission lines and simple circuit including passive components) have been designed and fabricated and for which RF power can be traceably assessed in terms of power consumption, using both the EM modelling and calibration that have been carried out and that rely on geometry, permittivity, conductivity.

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Metrology for mixed-mode on-wafer S parameter

With the objective to develop traceable on-wafer mixed-mode S-parameter measurements, multimode TRL calibration kits have been designed and fabricated using coupled coplanar waveguide (CCPW) calibration standards on SiO_2 and GaAs substrates, in both ground-signal-ground-signal-ground (GSGSG) and ground-signal-signal-ground (GSSG) configurations. Validation measurements were performed using two methods based on one-tier and two-tier calibrations, i.e. direct on-wafer calibration and indirect (from coaxial to wafer reference plane) calibration, respectively. Very good agreement was obtained on the calibrated measurement data between the two approaches, using on-purpose developed verification standards like CCPW based matched and mismatched transmission lines and T-attenuators and for which the consistency of the results validates, up to 40 GHz, the use of the true-mode stimulus option available on four-port vector network analysers (VNA) allowing less time consuming calibration thanks to the far

reduced number of standards and connection. A full uncertainty analysis is being finalized to validate the whole process within established confidence levels.

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10 List of publications

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