Progress report on electrical metrology at MIKES Metrology between 2013 and 2015

for the 29th meeting of the Consultative Committee for Electricity and Magnetism (CCEM), March 2015

Organizational changes

In the beginning of 2015, MIKES Metrology amalgamated with VTT Technical Research Centre of Finland, and they together formed a new, fully state-owned not-for-profit-company VTT Technical Research Centre of Finland Ltd. At the same time, the accreditation department (FINAS) of the former Centre for Metrology and Accreditation (MIKES) became an operationally and economically independent department within the organisation of the Finnish Safety and Chemicals Agency (Tukes). VTT Ltd has about 2600 employees, and it is Finland's biggest applied research organisation and the Nordic countries' leading research and technology company. According to Finnish legislation, VTT Ltd is Finland’s National Metrology Institute since the beginning of 2015. In practice, national metrology activities are still concentrated on MIKES Metrology, which is now one of the Research Areas in the organization of VTT Ltd. MIKES Metrology’s Electrical Metrology Team with about 20 researchers is responsible for Finland’s national metrology activities in fields of electricity, time and frequency, and acoustics.

Quantum current standard and other applications of single-electron tunneling

MIKES has continued efforts for the development of a quantum current standard based on the SINIS hybrid turnstile. The work is done in close collaboration with the O.V. Lounasmaa Laboratory of Aalto University. As a part of these efforts, MIKES had an active role in writing a thorough review article on single-electron current sources in collaboration with Aalto University, NEC, and Stony Brook University [4]. Much of the work on the quantum current standard is done in the framework of joint research project Qu-Ampere of the European Metrology Research Programme (EMRP) [24]. In that project, a major goal of MIKES is parallelisation of hybrid turnstiles to increase the quantized current level above 100 pA. The progress has been slower than anticipated due to delayed delivery and other problems with the new electron beam lithography writer (EBL) and other essential equipment needed in sample fabrication in the clean room of Aalto University. However, most of the problems have been solved now, and prototype samples of parallel SINIS turnstiles have been fabricated using the newly-developed three-layer process including a hard germanium mask [35]. Meanwhile, understanding of physics of the SINIS turnstile, including Andreev tunneling [7], the effect of high-temperature environment [6], and quasiparticles in the superconductor [5, 8], has been improved considerably in experimental and theoretical studies with Aalto University and other collaborators [44]. In addition to metrology applications, work on single-electron tunneling has also contributed to understanding of some interesting topics of fundamental physics, such as Maxwell demon and Szilard engine [10, 11]. MIKES has also participated studies on a single-electron pump based on a silicon quantum dot with tunable tunnel barriers, in which generation of 80 pA current with uncertainty below 50 ppm was demonstrated [12].

As a step towards a quantum metrology triangle -type experiment, MIKES has built a traceable measurement setup based on a room-temperature large-value resistor and voltage source, but so far the system has been tested only at current levels below 1 pA [46]. As an alternative, a SQUID-based null detector with equivalent input current noise of 14 $fA/Hz^{1/2}$ has been developed in collaboration with VTT [3].

Single-electron tunneling can also be applied in detection of microwave radiation. In EMRP, MIKES coordinates a joint research project MICROPHOTON, whose main aims are to develop novel microwave detectors and sources ultimately on single-photon level [34]. Technical activities at MIKES focus mainly on developing SINIS SET devices as microwave photon detectors, and on improvement in the performance of cryoelectronic quantum devices by understanding and eliminating the detrimental effects caused by microwave radiation. For this purpose, a cryogenic measurement setup consisting e.g. of an RF-SET-based charge detector and a sample chamber with variable level of filtering and shielding has been constructed. Experiments with double SINIS photon detectors from MIKES and SINIS traps from PTB are in progress.

MIKES also develops thermometry applications of single-electron tunneling [23]. In joint research project InK of EMRP, the main aim of MIKES is to develop primary Coulomb blockade thermometry (CBT) for temperature range between 50 mK and 1 K with uncertainty below 0.3%. Comparisons against other primary cryogenic thermometers are in progress.

Applications of Josephson AC voltage standard

MIKES has successfully commercialized a two-channel highly stable audio-frequency voltage source DualDAC with a Finnish company Aivon Oy [36]. The roots of the development are in the joint research project JOSY of the first phase of the EMRP. In that project, a more stable sine wave generator was needed for the AC voltage standard based on using square waves from a Josephson junction array. The source has been further developed based on experience obtained in EMRP projects AIMQuTE and Q-Wave. The stability of the amplitude ratio of the two channels approaches 10^{-8} which is very useful for fully digital impedance bridges, for example. Even for a single channel, the short-term amplitude and phase stability of the new source have been observed to be better than those of best commercial calibrators. This claim is based on testing the source with the AC Quantum Volt Meter (AC-QVM) at PTB. The unforeseen stability of the source proved to be useful in developing the details how the QVM could be used best [37]. The source will be used in 2015 as a transfer standard of simple arbitrary periodic waveforms for which several partners of Q-WAVE are developing new measurement techniques.

MIKES has also started building a compact cryocooler-based Josephson impedance bridge for audio frequencies in EMRP-project AIMQuTE. The work builds on the experience obtained in collaboration with PTB on Josephson bridges excited with square waves. Programmable 1 V arrays provided by PTB have been mounted and work is going on to optimize the setup for optimal voltage steps.

Resistance, impedance, and applications of graphene

The main efforts were concentrated on investigating and developing of graphene for resistance metrology based on quantum Hall effect (QHE). The work has been done in close collaboration with Aalto University. A set of graphene QHE devices, based on epitaxial graphene films grown on SiC, fabricated in Aalto University, were investigated in MIKES and suitable devices for accurate resistance measurements were chosen. Adjustment of the carrier density to the optimum values using photochemical and chemical functionalization of graphene, allowed observation of half-integer quantum Hall effect with $\nu = 2$ plateau starting at relatively low (3 T) magnetic field. Selection of the devices has been made according to the technical guidelines for reliable QHR measurements. Accurate measurements of a 100 Ω resistance standard against $R_H(2)$ on graphene QHR device (at $B = 7$ T, $T = 1.5$ K, and $I_{sd} = 33$ µA) and on GaAs device, performed with CCC bridge, showed an agreement within $(6\text{-}10)\cdot 10^{-9}$. Non-homogeneity of carrier density distribution and variation in carrier mobility at different areas of the graphene devices were the main factors limiting the precision measurements [2, 20, 26]. Graphene QHE device fabricated in Aalto University and characterized at dc in MIKES was also used in first ac QHR measurement which was performed in ac QHE setup at PTB [13, 29]. The graphene device shows only weak frequency dependence similar or even better than for plain GaAs devices without any complex shielding technique applied. The results are promising for the realization of a graphene-based quantum impedance standard operating at lower magnetic field and higher temperature.

In project GraphOhm of EMRP [27], the main role of MIKES is to develop a current comparator for operation at low frequencies (LFCC) in the 0.1 Hz to 1 Hz range and at room temperature. Two prototypes have been realized [28]. Experimental tests include the determination of the frequency-dependent sensitivity, measurement of the ratio error for different windings and frequencies and a comparative measurement of the ratio of standard resistors with nominal values of 10 kΩ, 1 kΩ and 100 Ω. The measured sensitivity of both LFCCs varied from 3 V / A turn up to 124 V / A turn in the frequency range 0.14 Hz - 1.00 Hz. The ratio error measured at 0.7 Hz with increasing of the tested number of turns from 1:1 up to 1024:1024 was reduced from $5\cdot 10^{-8}$ to values below 10^{-9}. Deviation from 1:1 measured at frequencies 0.72 Hz, 0.52 Hz, 0.37 Hz, 0.22 Hz and 0.14 Hz using 1024 : 1024 turn coils was within $1\cdot 10^{-9}$. The comparison of one of the developed RTCC and the current comparator of BIPM at 10:1 ratio has been started using BIPM 1 Hz bridge electronics. Preliminary results of the measurements the ratios of 1 kΩ / 100 Ω and 10 kΩ / 1 kΩ using developed RTCC and the equipment of PTB and BIPM showed agreement in measurement results within parts in 10^8. It has been decided to continue the comparative measurements of LFCCs of different design at frequencies 0.5 Hz and 1 Hz taking into account the improvements in developed measurement equipment. The realized current comparator is the main ratio standard in a precision ac resistance bridge operated without need of liquid helium which will be one of the main measurement instruments for a future graphene-based quantum Hall resistance standard. The developed LFCC can replace the CCC in commercial bridges, if the software and control electronics are appropriately modified. This would reduce the expenses on liquid helium and simplify the calibration of resistors.

Joint research project MACPoll (Metrology for chemical pollutants in air) of EMRP started in June 2011 and finished in May 2014. During 2013-2014 the work on further improvements of the graphene based gas sensor has been conducted [1]. It was found that an optimization of the graphene/metal contact configuration and complementary annealing of graphene sensor after each gas exposure led to significant improvement in the sensing performance [25, 21, 45]. The response of the annealed sensor to nitrogen dioxide (NO_2) was tenfold higher than that of an as-fabricated graphene sensor. NO_2 concentration as low as 0.2 parts per billion (ppb) was easily detectable. The detection limit of the graphene sensor was estimated to be 0.6 ppt (parts per trillion) [21]. The present technology with additional annealing improves the performance of the graphene based sensor and makes it suitable for the environmental nitrogen dioxide gas monitoring.

Applications of MEMS

As a part of MIKES activities in joint research project MetNEMS of EMRP, work on applications of microelectromechanical systems (MEMS) for electrical metrology has been continued. Prototypes of a sensitive

MEMS-based voltage detector and a stand-alone temperature and pressure stabilised MEMS-based AC voltage reference with an automatic tracking of the operating point have been designed and constructed in collaboration with Aivon Oy and VTT [39]. Tests of the voltage detector with a non-optimal MEMS device indicate its great potential e.g. as a sensitive null detector [22].

Power and energy

As a partner of a Finnish research programme Smart Grids and Energy Markets, MIKES has developed a multichannel sampler for the most accurate three phase power and power quality measurements. The PCB was carefully designed in order to fully utilize the good characteristics of the selected ADC converter (AD 7690, TC ±0.3 ppm/K) and voltage reference (LTZ1000, TC ±0.05 ppm/K) [32]. Blackfin evaluation kit is used to interface the ADCs with a PC. Currently the software performs power calculations according to IEEE1459, and algorithms according to DIN 40010 have been prepared and are waiting for merging to the package. Currently the characterization of the first six channel prototype is under way. The development of the systems will continue on domestic funding by adding support for digital input and output in line with IEC 61850. Reservation has also been made for using a GPS module and/or Ethernet connection for time stamping the samples.

High voltage metrology

In EMRP project HVDC lasting from 2010 to 2013, MIKES was responsible for coordinating of the design, manufacture, and calibration of a modular 1000 kV direct voltage divider. Altogether 9 modules were built in joint effort of 5 NMIs: MIKES, SP, VSL, PTB, and TUBITAK. The performance of the modular divider was studied in a month long measurement session at Aalto University, Finland in May/June 2013. A number of influences - e.g temperature and voltage dependence, self-heating, leakage current and corona - were studied. The estimated uncertainty of measurement of 1000 kV dc is 16 µV/V. The −3 dB bandwidth of the divider extends beyond 100 kHz. [15, 16, 19, 31, 42, 43]

In addition to traditional voltage divider approach, using a massive series connection of semiconductor based two-terminal low-voltage references was studied up to 20 kV. Series connection of cheap LM4040 references is temperature compensated by Zener diodes, and a precise current sink is used to stabilize the current through the references. An uncertainty of 3 µV/V was achieved. [17, 41]

Other high voltage related activities include development of analysis algorithms for impulse voltage measurement [40] and study of non-linearities of a high-voltage current comparator based capacitance bridge [33].

High frequency metrology

In EMRP project Ultrafast (2011 - 2014), MIKES has developed traceable communications software. To apply the full traceability through various communications calibration standards requires the usage of covariance matrix. The problem until now has been the size of the covariance matrix as the sample count increases. In the project a compressed covariance method was developed [18]. This methodology was applied in the Vector Signal Analyser (VSA) software development [30]. This software is written in C++ language in the Qt application framework. Both the compiler and the Qt framework use object oriented constructs. Most of the libraries used in the software are open source. The waveform and the associated covariance matrix are embedded in the same structure and in this way they form a usable object. The object oriented approach in C++ is programmed to automatically select the right kind of arithmetic to be performed to the covariance matrix according to what kind of operation is performed to the waveform itself. In sampling the symbol data from the modulated waveform the covariance matrix is reduced also in size.

Other research activities

MIKES has collaborated with the Fraunhofer Institute for Physical Measurement Techniques (IPM) to develop a new way of simultaneously measuring the key physical properties of thermoelectric materials. Feasibility study of fabricating a microstructure on top of sample surface to allow measurements of electrical conductivity, thermal conductivity and Seebeck coefficient in the same sample area has been started [38]. First measurements suggest that our state-of-the-art bias source (the DualDAC, see above) enables measuring reliably very small Seebeck voltages. Aided by numerical simulations, even highly anisotropic materials could be characterized. In addition, an apparatus for performing measurements between 4 K and room temperature has been designed and built [47].

As an application of traceable measurement of fA-range DC currents, MIKES has participated in developing a particle number concentration standard for nanometre-sized aerosol particles, so-called single charged aerosol reference (SCAR). As a part of that work, a comparison of aerosol electrometers (AE) has been arranged between several national metrology institutes and expert laboratories in the framework of EMRP project PartEmission [14]. The comparison covered the particle size and charge concentration ranges 20 nm to 200 nm and $0.16 \cdot 10^{-15}$ Ccm^{-3}

to $2.7 \cdot 10^{-15}$ Ccm^{-3}, respectively. The obtained results agreed to within about ±3%, which was within stated uncertainties, with only a few exceptions.

Comparisons

MIKES has participated the following key or supplementary comparisons whose final or draft report appeared during the reporting period: EUROMET.EM-S24 (Ultra-low DC current sources), EURAMET.EM-S32 (Resistance standards at 1 TΩ and 100 TΩ), EUROMET.EM-S33 (AC up to 200 kV), EUROMET.EM-S34 (Capacitance and dissipation factor up to 200 kV). The results of MIKES are good and support CMC claims. In addition, results of a comparison of a number of 800 kV to 1000 kV dc systems within EMRP HVDC project were published [19, 43].

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