EURAMET European Association of National Metrology Institutes
EMRP European Metrology Research Programme
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Publishable JRP Summary for Project T4.J04 ULQHE Enabling ultimate metrological quantum Hall effect (QHE) devices

Since 1990, in accordance with a recommendation of the *Comité International des Poids et Mesures* (CIPM) (n°2 1988), the Quantum Hall Effect (QHE) has been used as the primary reference for the electrical resistance. This physical phenomenon based on quantum electronic properties of a two dimensional conductor in presence of a high magnetic field allows the generation of an electrical resistance, the Hall resistance R_H, perfectly quantized at integer fraction of a constant R_K theoretically equal to h/e^2 (h is the Planck constant and e the electron charge). Research experiments on the R_H quantization already demonstrated that the effect is universal (so is independent of the material, its electronic properties, the geometrical shape and size of the conductor, …) and reproducible with relative accuracies as low as some parts in 10^{10}. Exactness, reproducibility, universality, intrinsically referenced character are the qualities which ensure the QHE to be the ideal candidate for the ohm metrology. In this way, the discovery of the QHE and the development of QHE based quantum resistance standards were responsible for a real breakthrough in the unit representation: the degree of equivalence of the primary resistance standards maintained in the national metrology laboratories have been reduced by three orders of magnitude. Nowadays, with demanding experimental conditions (low temperatures (~1.2 K), high magnetic field (~10 T)) and a specifically developed instrumentation (involving notably Superconducting Quantum Interference Devices (SQUIDs)) a quantum resistance standard, the value of which is $R_{K-90}/2$=12 906.4035 Ω and fabricated in sophisticated semiconductor structures, routinely allows the calibration of a material resistance with a relative uncertainty equal to some parts in 10^9. In addition to this performance, the QHE offers the possibility to link the ohm to the fundamental physical constants h and e. This ability is very crucial for the project of redefinition of the *Système International* of units (SI) based on fundamental constants of physics which is presently focusing the efforts of metrologists. For instance the QHE directly contributes to make the watt balance an experiment able to link the kilogramme to the Planck constant and that is partly this effect which makes this experiment the most promising route for a new definition of the mass unit.

The present stakes of the QHE metrology are the following:

- The improvement of the specifications and performances of the quantum resistance standards (improved accuracies ($<10^{-9}$), enlarged range of values over several decades (100 Ω to 1 MΩ), eased operation (higher temperature, lower magnetic field, …), improved handiness towards transportability …) taking the best benefits from the high potentials of the QHE with the lowest experimental implementation constraints and costs, in order to accompany the technological innovation and to anticipate the industrial demands.
- The consolidation of the confidence in the QHE physics and its experimental implementation in order to make the QHE a solid and reliable support to the establishment of a new SI.

To take up these challenges there are common needs, on one hand to deepen the understanding of the most fundamental intrinsic physical properties of the QHE and on the other hand to perfect the experimental mastery of the effect. These ambitions concerning both the knowledge and the know-how of the QHE determine the project objectives.

Indeed, the first objective of the ULQHE project is to perform new universality and quantization tests of the QHE. Two approaches to these experiments are planned. The first consists in performing comparisons of quantized Hall resistance both at accuracy levels and with uncertainties never reached so far ($<10^{-11}$) by developing instrumentation and original measurement techniques (the quantum Wheatstone bridge for instance). The second approach deals with Hall resistance quantization measurements in electronic systems which present original physics and which have never been explored so far with the metrological instruments allowing the best accuracies. Notably, graphene and two dimensional electron gases (2DEGs) in the fractional quantum Hall effect regime will be studied.

The second objective concerns the exploration of new innovating and promising materials with high potential for the QHE metrology and, for instance, to become the future improved, or even revolutionary, quantum resistance standard. Graphene will be the major subject. This material, composed of a one-atom thick layer of carbon atoms arranged in a hexagonal lattice, presents exceptional physical properties which allow, for example, the QHE to survive at room temperature, as recently observed. Thus, one can reasonably hope to use graphene to realize a quantum resistance standard operating at higher temperature or at lower magnetic field. The studies proposed include development of reliable high-quality samples fabrication process and metrological characterization of the QHE in the systems investigated (graphene and double 2DEGs).

Finally, the project aims at contributing to the metrological characterization, investigation and validation of quantum resistance standards prototypes, the so-called Quantum Hall Arrays Standards (QHARS). These are 'engineered' quantum devices combining up to about one hundred of single Hall samples connected in series and/or parallel arrays in order to offer adjusted values ranging from about 100 Ω to about 1.29 MΩ, and matching then the most common values used in industry. Some of these devices have already been demonstrated to be reproducible and exact within an uncertainty of few parts in 10^9. Starting from these encouraging results, the study proposed claimed to be the last step of the development of this new generation of quantum standards preparing a future production project with industrial means.

The main impact of the project will be scientific and technical, of course. The QHE universality tests, as tests of the major and fundamental theories of the solid state physics explaining this subtle effect on one hand and as a support to the SI revision based on fundamental constants of physics, are expected to have a sound scientific impact. Since graphene is focusing the attention of the whole scientific community from the most fundamental research laboratories up to the microelectronics industry, and since its physical wealth remains largely experimentally unexplored so far, any result about graphene will have a large scientific impact. Note that the instrumentation developed for these rather fundamental experiments will benefit the calibration activity.
As reminded above, the improvement of the quantum resistance standards performances targeted is intended to answer and anticipate the needs of the industry, and more generally to support the most demanding industrial innovation. Besides, most of the studies proposed in the project contribute to the development of enabling and underpinning technology for industrially relevant devices. At time scales larger than the project duration, industrial fallouts are then expected. At the same time, an economic impact is also expected, particularly due to the progress of the quantum standards in terms of availability, and implementation costs.

During the first period of the project (1 April 2008 to 31 October 2008) several results of great importance, some of them obtained sooner than initially expected, have enabled to overpass crucial steps towards the achievements of the ambitious objectives of the project. These results have principally benefited to the QHE universality tests. On the one hand an agreement, within a measurement uncertainty of 3 parts in 10^8 in relative value, between the quantization of the Hall resistance in a high electronic mobility GaAs/AlGaAs 2DEG on the i=1/3 fractional Hall plateau (where R_H= $3R_K$) and the quantization of the Hall resistance in a more usual GaAs/AlGaAs 2DEG on the i=2 integer Hall plateau has been demonstrated by means of a comparison resistance bridge based on a Cryogenic Current Comparator (CCC) equipped with a SQUID. This experiment is the first quantization test of R_H in the Fractional Quantum Hall effect regime. On the other hand a reproducibility test of the QHE in GaAs/AlGaAs 2DEG has been performed within the record uncertainty measurement of 3.2 parts in 10^{11} using the quantum Wheatstone bridge technique and measuring the unbalance current of the device. These tests have decreased by one order of magnitude the measurement uncertainty of the best quantized Hall resistances comparisons. Efforts are maintained to improve further the QHE universality tests by means of these two experiments (FQHE and quantum Wheatstone bridge). In parallel, within the objective to investigate new innovating and promising materials with high potential for the QHE metrology, two double 2DEG (vertically stacked) devices have been fabricated from two different wafers with carrier densities of $n \sim 4.8\text{x}10^{11}\text{ cm}^{-2}$ in both channels with a very low difference for n in both layers, by using the concept of asymmetric double layer structures. Concerning the other activities of the JRP, the achievements were not so remarkable but very significant and encouraging, notably for the difficult and exploratory graphene subject. For instance, optimisation of graphene deposition has been carried out. The largest single layer flakes obtained so far by Scotch tape exfoliation have an area of about 300 μm^2, already suitable for device fabrication, and there is still much room to improve the technique in the future.

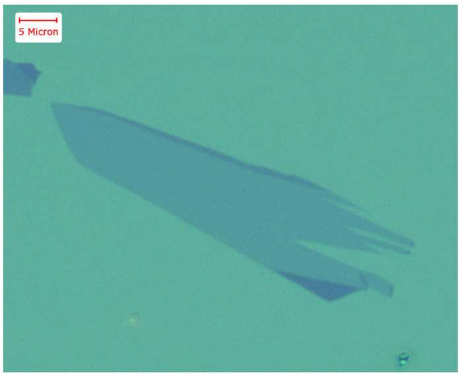

Fig. 1
Optical microscope image of a graphene single layer obtained by Scotch tape exfoliation from Highly Oriented Pyrolitic Graphite

Finally a test plan for the QHARS exchange inside the consortium accompanied with technical guidelines for the handling and operation of these peculiar quantum devices has been elaborated. The document should ensure the success of the exchange, notably the enrichment of knowledge and know-how of these devices. A first quantization measurement of a high value QHARS (1.29 MΩ) within the uncertainty of 2 parts in10^8 confirms our expectations about the metrological quality of QHARS and their ability to be the new generation of quantum resistance standards.

During the second period of the project (1 November 2008 to 31 October 2009), the achievements were still significant. The most intensive and exciting activity was on the burning issue of graphene. The work of sample fabrication and preliminary characterization measurements is arid, as expected for this novel and exploratory topic. Nevertheless significant progress has been realized with the successful demonstration of the feasibility of graphene microdevices with Hall bar geometry (typical size of 10 µm) (see Fig. 2) using micromechanical exfoliation of graphite, deposition of graphene layers on doped silicon substrate covered by silicon oxide, electron-beam lithography, photolithography and standard microfabrication techniques. The use of such a substrate has the advantage to enable the adjustment of the carrier denity in the graphene flakes by electric field effect tuned in applying a voltage between the flake and the doped silicon substrate (which acts as an electrostatic back gate). A fine optical contrast analysis technique has been developed to control of the quality of graphene deposited. Preliminary electrical characterizations, with field effect and magnetotransport measurements of the Hall and longitudinal resistances, have also been carried out. Carriers mobilities ranging from 5000 to 8000 cm^2/V/s at room temperature and contact resistance of the order of a few kΩ were deduced from these measurements in several samples. In addition to these characterizations, transport measurements have been performed at low temperature (1.5 K): the "anomalous" quantum Hall effect, resulting from the very peculiar physics of graphene, has been observed. This is encouraging for the ultimate objective of the project: the metrological measurement of the Hall resistance quantization in a graphene-based Hall device. Collaborative efforts among the partners will be absolutely needed to overcome the next difficulty along the hazardous route towards this goal (high contact resistance, inhomogeneity of the carrier density for instance,…).

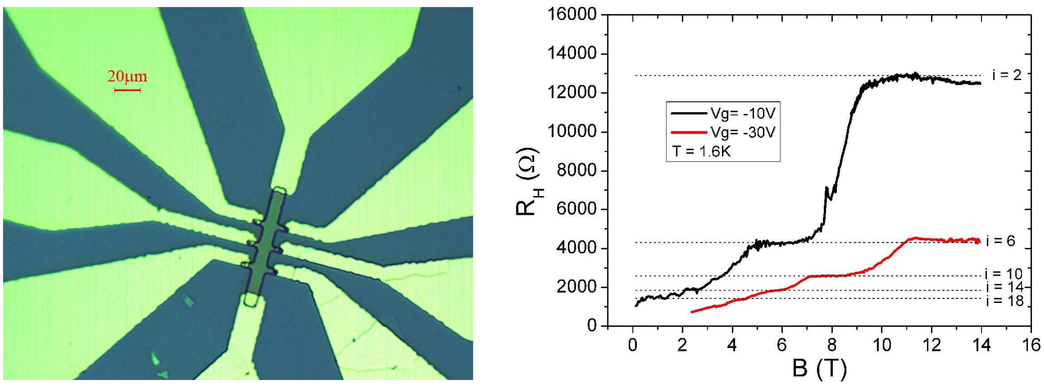

Fig.2 Optical microscope image (left) and magnetoresistance measurements of a graphene-based device fabricated and characterized at INRIM which reveal the half-integer anomalous QHE. The Hall bar is covered by a photoresist layer.

The activities related to the other topics handled in the project were also thankless but fruitful during this period. Considerable developments and improvements have been undertaken by several partners which concern both the instrumentation and experimental setup (new cryogenic current comparators (CCC)-based resistance bridge, installation of 19/20T superconducting magnet fitted with a helium 3 refrigerator) on one hand and new micro-nanostructured semiconductor devices (high electronic mobilities GaAs/AlGaAs based two dimensional electron gas (10^7 cm^2/V/s), quantum Hall circuits based on III-V (GaAs) and II-VI (HgTe) semiconductor heterostructures) on the other hand. Both of these types of developments are intended to prepare the next QHE quantization tests and are expected to enable major achievements in this field: investigation of the fractional quantum Hall effect regime with relative uncertainties better than some parts in 10^8, quantized Hall resistance comparison with record accuracies of some parts in 10^{12} in relative value and/or in rarely explored electron systems by means of the quantum Wheatstone bridge.

In parallel, fine measurements on the QHARS have been carried on using both CCC based resistance comparison bridge and more common commercial bridges. The very high-quality results obtained confirm the compatibility of these quantum standards with commercial equipments – what makes the high value (MΩ range) quantum standards all the more interesting because of the lack of very stable material standards in this resistance range -, and besides their usefulness for the characterization tests of metrological resistance comparison bridge, as well as for international comparisons.
Finally, measurements of the Hall resistance R_H realized in Hall bars etched in GaAs based double vertically stacked 2DEG fabricated in the first period of the project, have been performed on the $i = 2$ Hall plateau where $R_H = R_{K-90}/4 \approx 6453.2\ \Omega$ within an uncertainty of 1 part in 10^8. This result confirms the high-quality of these sophisticated semiconductor heterostructures and thereby their potential for the fabrication of Quantum Hall Array Resistance Standards.

During the third period of the project (1 November 2009 to 30 April 2010), the activity has been still mainly concentrated on graphene. After the very encouraging and exciting observation of the QHE in the first samples, the time has come for the optimization of the graphene-based devices for high-precision measurements of the QHE quantization and the detailed analysis of the problems inherently arising when investigating and exploring very novel systems. This was the occasion of a strong interplay between the electrical characterization measurements and the refinement or development of new fabrication process steps. Among the major difficulty faced was the bad quality of graphene-metal contacts which often appeared to present high electrical resistance or which were responsible for "junction-like" behavior observed when varying the voltage on the graphene sample backgate. An "ageing" of the titanium/gold contacts was also reported. New strategies for minimizing such effects and improving the quality of metal-graphene contacts are envisaged. A strong carrier density spatial inhomogeneity has also been identified as a recurrent problem. One positive point is that large flakes (3µmx40µm typically) have been commonly obtained. Otherwise, preliminary studies have been undertaken on graphene deposited on different prepared substrates (functionalized SiO_2 substrates, on mica and on GaAs substrates), with the intention to improve the electrical performance of the devices. Plasma treatments of graphene have also been explored. This period of the project has also been the occasion of an extensive work for adapting the cryomagnetic experimental setup to the specificity of the field effect magnetotransport measurement in graphene, which has been confirmed to be very sensitive to the electrical shocks. In the light of these first experiences the goal of a very accurate QHE quantization measurement in graphene has confirmed to be a great challenge. Anyway, this gives rise to an exciting scientific adventure.
In parallel, quantization measurements on QHARS have brought still very interesting results, allowing, for instance, the characterization of a new CCC based resistance bridge developed for refined QHE universality tests in the fractional quantum Hall effect regime and the calibration of a commercial resistance bridge error when using high resistances. New measurements of the QHE quantization in GaAs double 2DEG based Hall bars have been performed which confirm the quality of the devices for metrology (agreement of the Hall resistance with the expected value within 1 part in 10^8).
To end, major progress has been reported in the fabrication of sophisticated semiconductor devices for new ultimate QHE quantization tests (high electronic mobility III-V GaAs/AlGaAs Hall bar samples, fully integrated GaAs/AlGaAs quantum wheatstone bridges, II-VI HgTe/HgCdTe Hall bar samples).

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