FINAL PUBLISHABLE REPORT

Grant Agreement number	16NRM01
Project short name	GRACE
Project full title	Developing electrical characterisation methods for future graphene electronics

<table>
<tr><td colspan="2">Project start date and duration:</td><td>1 July 2017, 36 months</td></tr>
<tr><td colspan="3">Coordinator: Luca Callegaro, INRIM Tel: +39 (0)11 3919435 E-mail: l.callegaro@inrim.it
Project website address: http://empir.npl.co.uk/grace/</td></tr>
<tr><td colspan="2">Chief Stakeholder Organisation: IEC/TC113</td><td>Chief Stakeholder Contact: Akira Ono</td></tr>
<tr><td>Internal Funded Partners:
1. INRIM, Italy
2. CEM, Spain
3. NPL, United Kingdom</td><td>External Funded Partners:
4. das-Nano, Spain
5. Graphenea, Spain
6. ISC, Germany
7. UoM, United Kingdom
8. VDE, Germany</td><td>Unfunded Partners:</td></tr>
<tr><td colspan="3">RMG: -</td></tr>
</table>

TABLE OF CONTENTS

1 Overview

The overall goals of this project were 1) the development of validated protocols, to be used in the production of Good Practice Guides (GPGs), for the measurement of the electrical properties of graphene, and their implementation in order to achieve accurate and fast-throughput measurement of graphene; and 2) collaboration with international standardisation committees in order to initiate and develop dedicated documentary standards for the electrical characterisation of graphene. The project contributed towards the development of five standards from the technical committee IEC/TC113. Two of these were directly initiated by the project. Furthermore, the adoption of GPGs developed in the project and, subsequently, of such standards, has enabled industry to perform accurate measurements of the electrical properties of graphene and thereby provide customers with reliable and comparable specifications of graphene as an industrial product.

2 Need

Discovered in 2004, graphene, is a two-dimensional (2D) lattice of carbon atoms and is currently being extensively investigated by industry as a potential new material for electronics. However, the adoption of graphene as an electronic industrial product is limited by the inability to grow large areas of high-quality graphene with uniform and reproducible electric and electronic properties. Therefore, accurate and reproducible characterisation methods adapted to the 2D nature of graphene, both as test samples and in production lines, are crucial. But such electrical characterisation methods for graphene are presently underdeveloped; and guidelines for the proper implementation of such methods in an industrial environment are lacking. The issue has been highlighted by standard organisations such as the International Electrotechnical Commission (IEC) and the European Committee for Electrotechnical Standardisation (CENELEC), who have initiated working groups e.g. IEC/TC113 Nanotechnology for Electrotechnical products and systems. However, it is currently unclear how different approaches particularly high-throughput methods such as microwave resonant cavity methods and time-domain terahertz (THz) waves spectroscopy can be compared and mutually validated. Therefore, the European Committee for Standardisation and Electrotechnical Standardisation CEN/CENELEC has recognised, in a recent Standardisation, Innovation and Research (STAIR) survey, the need to initiate a metrology project at the European level on the issue of the characterisation of graphene and 2D atomic materials for electrical applications.

3 Objectives

The project aimed to develop traceable and comparable electrical characterisation methods for the electrical properties of graphene, including accurate and fast-throughput measurements. In addition, the project focussed on facilitating the uptake of such methods by standardisation bodies such as IEC/TC113. The project's specific objectives were:

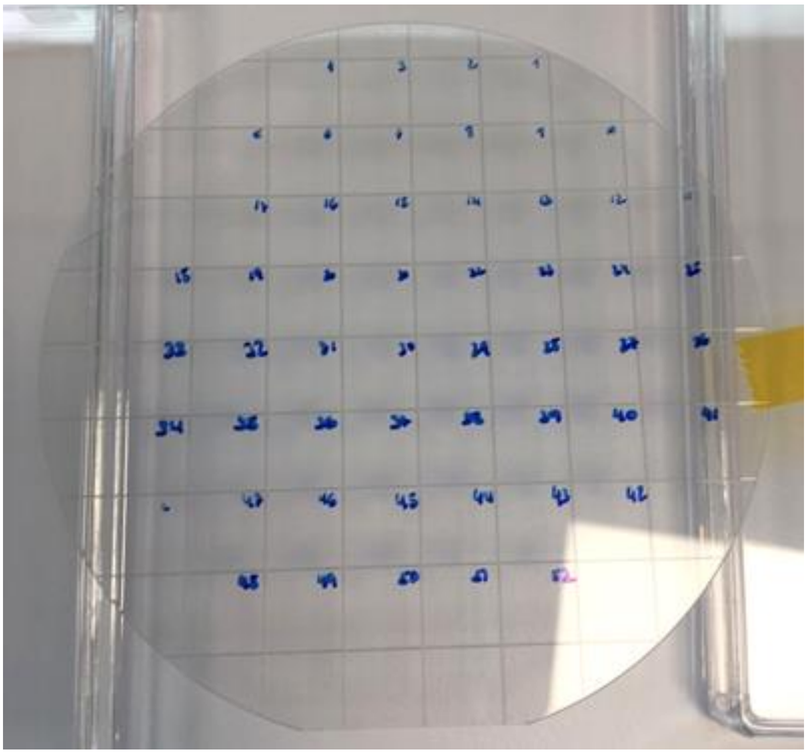
1. To develop an accurate and traceable approach for the electrical characterisation of graphene through the development and comparison of different methodologies for both contact measurement and non-contact electrical measurement of its properties, with traceability to the electrical SI units. This will include the improvement of established techniques as well as the development of new methods.
2. To develop a high-throughput approach for the electrical characterisation of graphene, with the development of novel methodologies for non-contact electrical characterisations and their validation with established techniques.
3. To disseminate the metrology and methodologies established in this project in the form of Good Practice Guides (GPGs) and input to documentary standards.
4. To contribute to the standards development work of the technical committee IEC/TC113, through the initiation of and development of new written standards for the electrical characterisation of graphene based on the GPGs developed within the project.

4 Results

This section gives an account of the project's outputs delivered against each of the project's objectives.

4.1 Objective 1

A set of 45 graphene samples was fabricated by Graphenea by transferring a CVD graphene layer on a 4" quartz substrate later diced in multiple 10 × 10 mm^2 samples. The samples were numbered and characterised by das-Nano via THz spectroscopy (see Objective 2). The sample quality was assessed by the parameters of conductivity and homogeneity and then the samples were distributed among all the partners mixing more and less uniform samples to have a variety of conditions in which the measurements will be performed. Each partner received a subset of the samples.



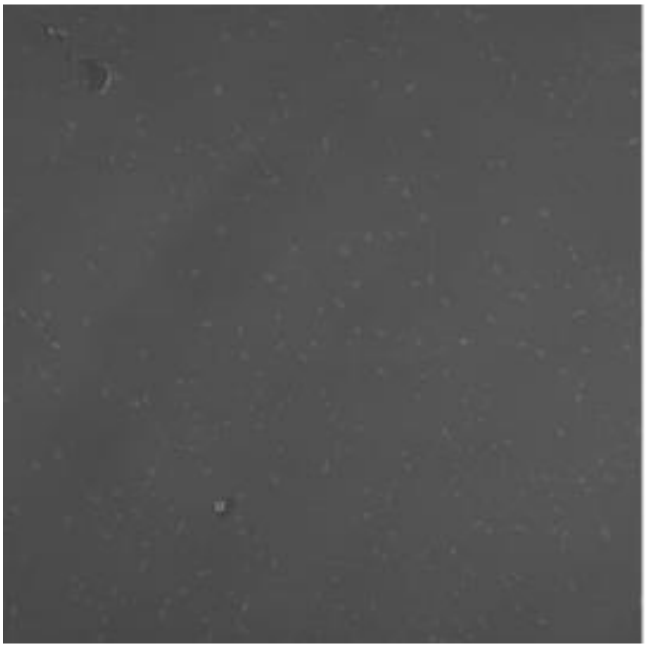
4" quartz wafer divided in 10 × 10 mm^2 regions that correspond to the individual samples.

Boxes containing the GRACE samples.

Some samples were investigated with well assessed non-electrical techniques employed in industry and research on CVD graphene such as confocal laser scanning microscopy and Raman spectroscopy.

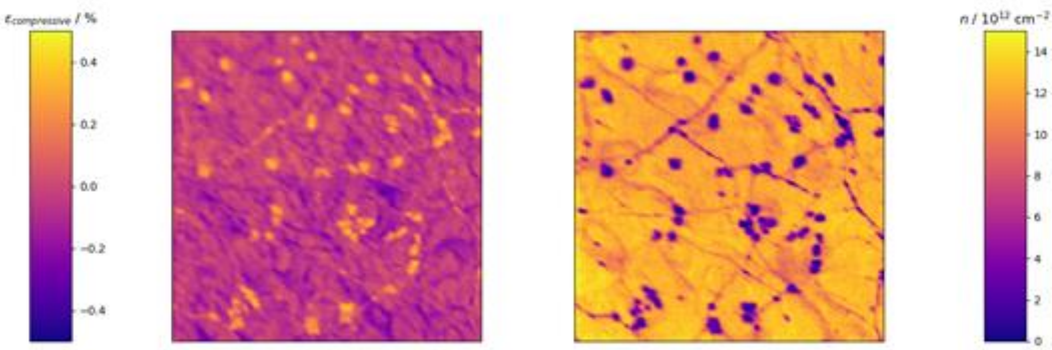
4.1.1 Optical characterisation

The samples received by NPL were investigated by the means of confocal laser scanning microscopy (CLSM) to rapidly characterise a large-area graphene and graphene nanostructures; pushing the spatial resolution beyond the optical diffraction limit and stacking individual images of different focal planes.



Confocal laser scanning microscopy image of part of a CVD graphene sample.

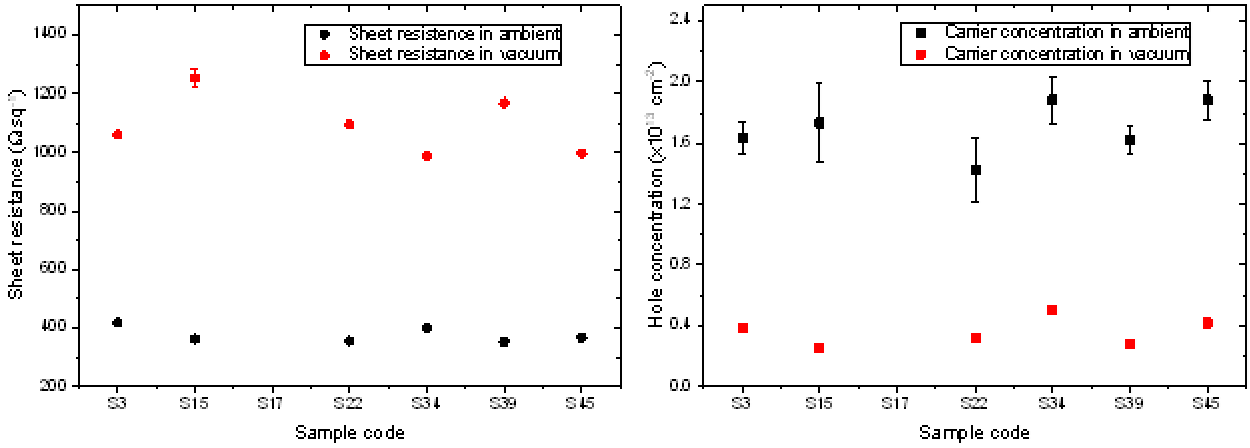
This characterisation was performed with an Olympus LEXT OLS4100 equipped with a ×100 objective, in air and did not require any sample preparation. Most of the samples showed flat and homogeneous surface morphology (consistently with the initial TDS-THz screening), features as grain boundaries, bilayer and multilayer regions and local discontinuities of the graphene film were also identified. The structural properties of the samples were also investigated through Raman spectroscopy. From the analysis of the Raman G and 2D peak ratio and position it was possible to extract the strain profile and the doping level of the graphene samples. Such an analysis revealed that the strain level did not exceed ± 0.5% and the samples were p-type doped with a carrier concentration estimated to be around 12×10^{12} cm^{-2}, with both numbers consistent with typical results for this kind of synthesis method.



Strain and carrier concentration maps of the graphene sample. Area 10 × 10 µm².

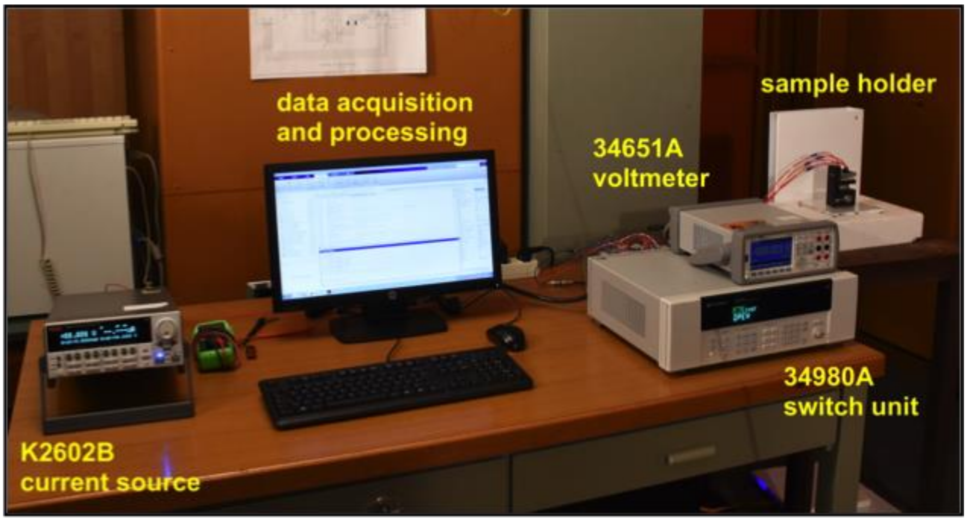
4.1.2 Electrical characterisation, contact and non-contact methods

Magneto-transport properties of the samples were investigated using the NPL's vdP (van der Pauw method) setup to determine the sheet resistance, carrier concentration and carrier mobility of each sample. VdP measurements are performed by placing contacts at the edge of the sample, with the contacts placed at the corners. Such measurements were performed either in ambient or in vacuum, in order to assess the effect of the environmental fluctuation on the measurement. Prior to the measurement in vacuum; the samples were annealed at 170°C at low pressure (for 3 hours); to promote the desorption of atmospheric contaminants that are responsible for the high doping observed in ambient conditions. The larger measurement standard deviation observed in ambient was significantly reduced in vacuum with this approach, allowing stable measurements.



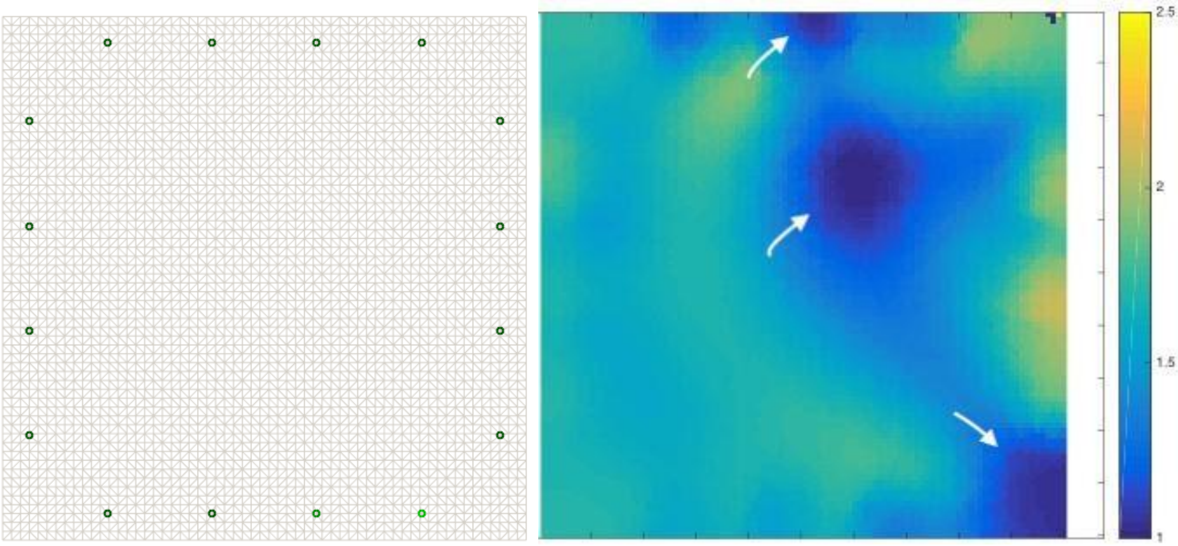
Sheet resistance (left) and carrier concentration (right) of CVD graphene samples obtained with NPL's van der Pauw setup. Both the measurements were performed in humid ambient air and in dry vacuum conditions.

INRIM performed vdP measurements too (even on the same samples measured with the NPL's setup, see Objective 2 "interlaboratory comparison") with the *extended vdP and ERT setup* developed at its premises; vdP measurements were performed with multiple contact positions (corners, edge mid-points, or mixed positions). This approach allowed for a more comprehensive estimation of the sheet resistance accuracy by taking into account the effect of the sample inhomogeneity assessed with different contact configurations. INRIM also developed and performed Electrical Resistance Tomography (ERT) on the same samples. ERT is a non-invasive contact technique which allows to recover the local conductivity of an object relying on a relatively small set of trans-resistance measurements, with no need for sampling its entire volume. The basic principle of ERT is that injected current flows through an object depending on its conductivity distribution. Any voltage measurement collected at that body's boundary will carry some information about its conductivity distribution. The conductivity maps can be recovered from the trans-resistance measurements by solving an inverse problem. The boundary voltage measurements are used as input data for numerical solvers. The ERT system running at INRIM can be seen in the figure below. The experimental setup consists of a custom sample holder, a switching unit and a trans-resistance measurement setup. The sample holder is connected to the switching system with 16 terminal wires. The switching system consists of a Keysight 34480A host and a Keysight 34933 reed relay matrix module. A similar earlier version of this system was used to perform ERT on other thin film samples. Measurements were performed in a shielded environment at T = 296.0(5) K, with a constant bias current of 50µA. The measurements were highly reproducible with a relative standard deviation of $\simeq 10^{-4}$ over 100 subsequent repetitions. Each measurement run consists in 208 trans-resistance measurements, which can be collected in approximately 2 minutes.



The INRIM ERT setup developed for the GRACE project.

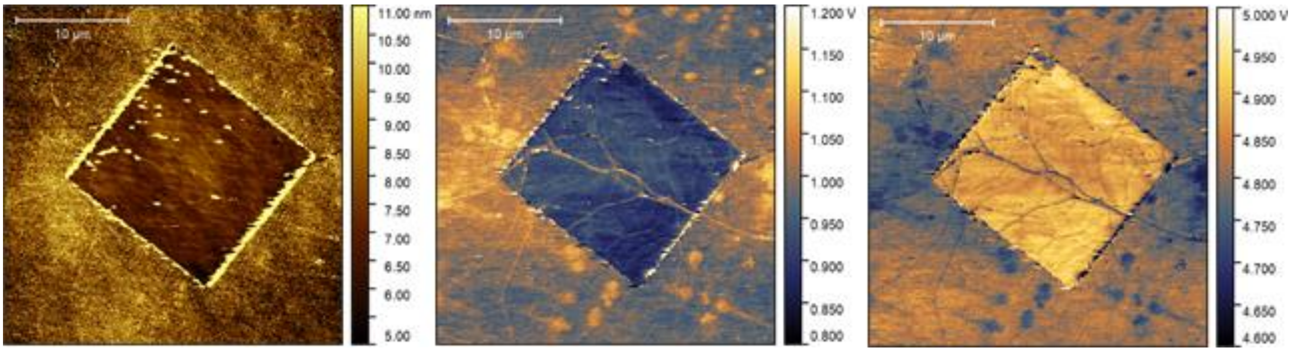
The sample is placed inside the sample holder, once the mechanical contact with the spring-mounted probes was established, then the tips were never lifted to avoid contact-area scratching which could inhibit any subsequent measurement. To recover the conductivity map, the inverse problem was solved on the mesh, shown in the next figure, with an iterative Gauss-Newton (GN) solver. This kind of solver promotes smoothness, contrarily to other "edge preserving" algorithms, and is the natural choice when the sample is not expected to have large sharp discontinuities. At the cost of a lower resolution image, GN solvers are less prone to amplitude artefacts, that is better since we are interested in reliable measurements of the conductivity values. The ERT map of sample S28 reported in the next figure. It shows some inhomogeneity in the electrical conductivity. Three lower-conductivity zones with conductivity $\sigma < 1$mS are marked by arrows. These can be due to many reasons, not yet investigated (lattice defects, contamination). The spatial resolution expected with this setup with the actual reconstruction routine is of the order of the mm. It must be noted that since the spatial resolution in ERT is limited by smoothing effects, very localised conductivity fluctuations can appear larger and weaker. We remark that one can get the same relative spatial resolution by scaling up the sample size, just maintaining the same electrode number with same relative spacing. On the quantitative side, we performed van der Pauw measurements on the same sample, resulting in a σ_{vdP} = 1.57mS, while the average value of the ERT conductivity map elements is σ_{ERT} = 1.55mS; hence the two values are in good agreement



Finite element model geometry (a) and ERT map of the graphene sample S28 (b). The colour-scale is in mS.

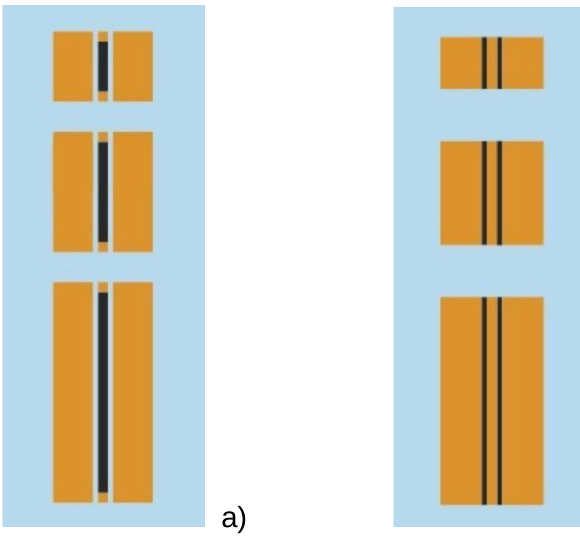
The samples were also characterised via a Scanning Kelvin Probe Microscopy (SKPM) analysis by NPL. Adversely from VdP, this technique provides a spatially resolved map of the contact potential difference between the graphene surface and a metallic coated AFM tip. Also, it is possible to determine the work function of the AFM tip by scanning a substrate of a known work function such as highly oriented pyrolytic graphite

(HOPG). The work function of the HOPG is independently determined by ultraviolet photoemission spectroscopy. In such a way it is possible to shift the contact potential difference map to produce the surface potential of the graphene sample.



AFM image of a sample. The middle area is cleaned to remove the polymer contaminants. Topography image is on the left, contact potential difference is in the middle and the surface potential map is on the right side.

The radio-frequency resistivity of graphene can be investigated with the Co-Planar Waveguide (CPW) method, applicable for graphene grown by CVD on or transferred to quartz substrates or other insulating materials as well as graphene on silicon carbide. A coplanar waveguide consists of a strip of thin metallic film on the surface of a dielectric slab with two ground electrodes running adjacent and parallel to the strip. The measurement of the S-parameters by means of vector network analysers (VNA) enables recovery of the AC conductivity of graphene, when it is partly made of the CPW itself. To apply the CPW method, the samples are prepared by lithographic geometrisation and deposition of metal contacts. The contacts are shaped as a CPW. Two different configurations of samples are possible, *series* and *shunt,* they can be realised using the same lithographic methods.



a) b)

Possible CPW configurations for graphene characterisation (series (a) and shunt (b) configurations are shown). The design of the CPW is by University of Manchester and Politecnico Torino (GRACE collaborator).

The graphene to be measured constitutes either the whole or a part of the central conductor of the CPW (in the "series" geometry) or fills the spacing between the CPW lines (in the "shunt" geometry). The CPW is measured with a vector network analyser with a probe station, properly calibrated with wafer standards. A mathematical model allows to extract the sample resistivity at the measurement frequency from the scattering parameter matrix measured by the VNA.

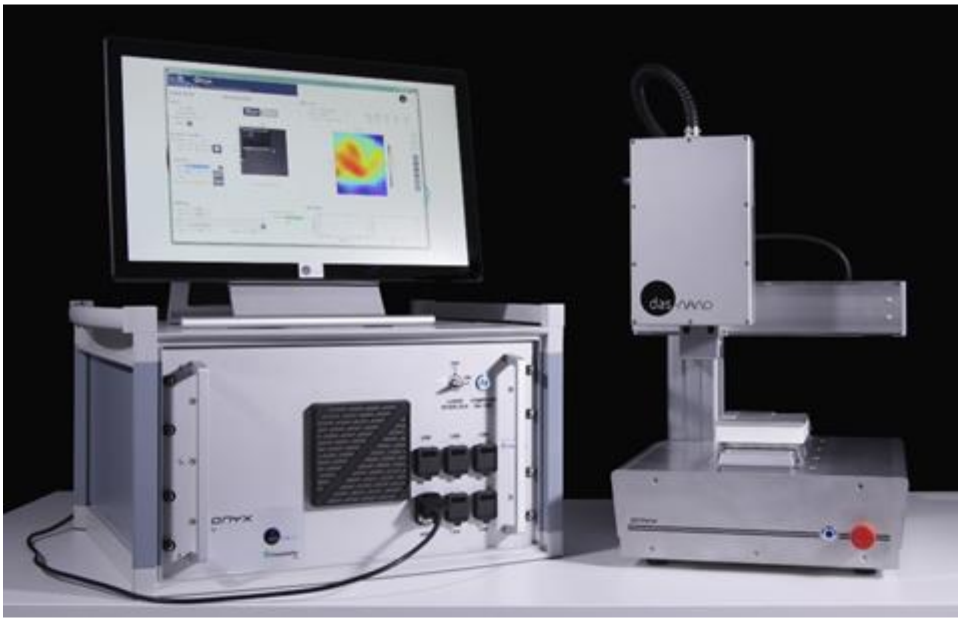
4.1.3 Key Outputs from Objective 1

This objective was fully met. Several methods for the electrical characterisation of thin films, were implemented by the partners to operate reliably on CVD large-area graphene samples. Traceability to the SI units was ensured. The implementations were thoroughly tested with graphene samples on quartz substrates, in a highly consistent comparison framework.

4.2 Objective 2

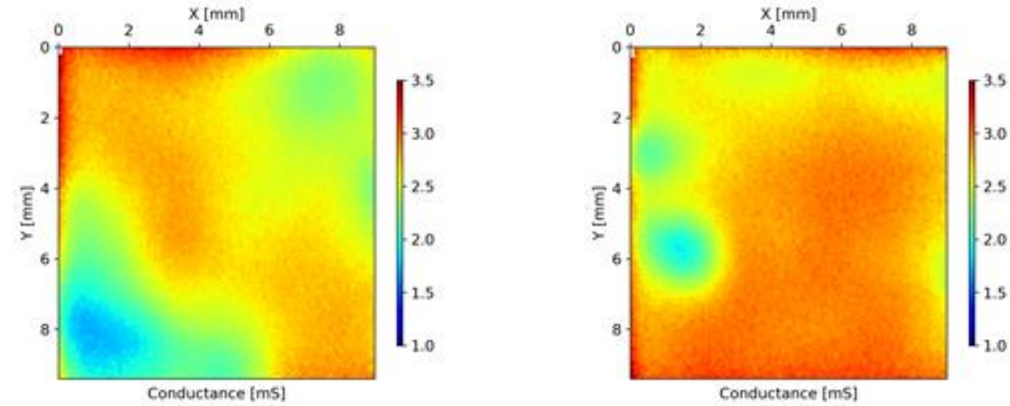
4.2.1 High-throughput electrical characterisation methods

Square 10 x 10 mm^2 samples (on quartz, PEN and PET), were analysed during the GRACE project by das-Nano, with terahertz Time-Domain Spectroscopy (TDS, system model ONYX), to verify their homogeneity and sheet conductance levels. The analysis was done before any other measurements were performed on the sample by the other partners. ONYX is a fast time-domain spectrometer for non-contact and non-destructive characterisation of large areas of graphene, 2D materials and other coatings. The TDS characterisation provides full-area maps of conductivity, allowing the assessment of the homogeneity and quality of the deposition. ONYX does not require sample preparation. The scanning resolution selected to characterise the samples was $100 \times 100\ \mu m^2$ and the measurement time was approximately 12 minutes per sample.



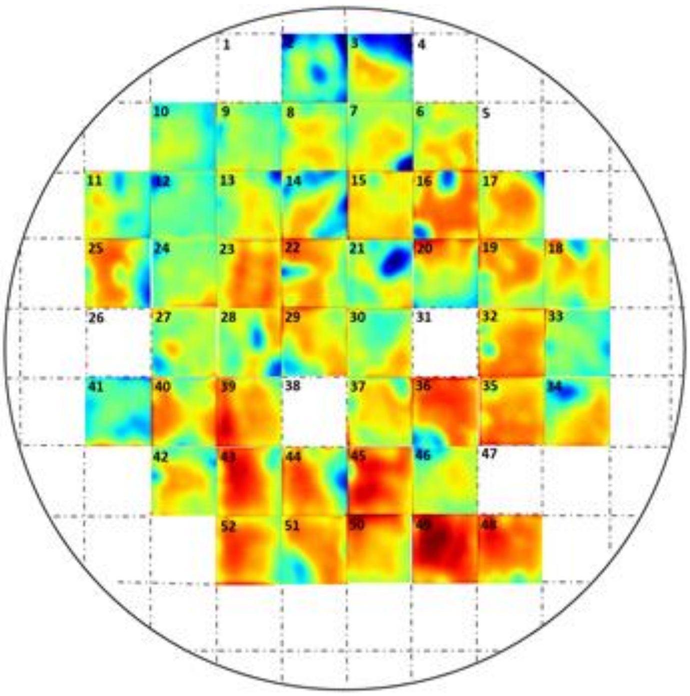
Das-Nano Onyx Graphene and 2D materials quality inspection system.

After analysing the samples, the sheet conductance map of each individual sample was obtained. Sheet resistance maps of selected samples are shown in the figure below. Every map contains approximately 7000 measurements. The colour scale represents a sheet conductance ranging between 1 mS and 3.5 mS. This colour representation allows identifying defects and heterogeneities in the electrical properties of the graphene films.



Sheet conductance maps of graphene samples. Left: sample 29. Right: sample 32

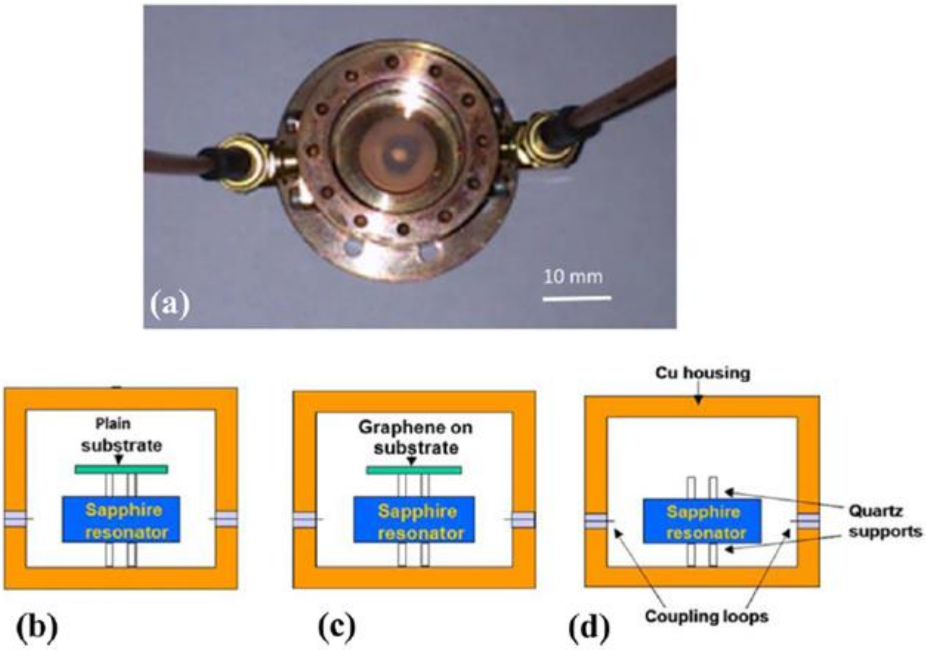
The complete set of sheet conductance maps is collected as a mosaic of the wafer based on the location of each sample; the mosaic is shown in Figure 5. The results indicate that there is a large variation of conductance across the samples and the presence of defects. The presence of such variations and features could be attributed to the cutting process and did not affect the outcome of the project related investigations (i.e. the applicability and comparison of different methods to the same set of samples).



Reconstructed sheet conductance of the wafer.

After the inspection of the samples with the THz-TDS ONYX equipment, the samples were sent in groups to the project partners in order to characterise their electrical properties with other contact and non-contact methods.

Additionally, a set of samples was investigated with another high-throughput method, which is the closed cavity microwave resonator. The measurement principle behind this technique is the perturbation of a well characterised high-Q dielectric microwave resonator (sapphire) induced by the presence of a graphene sample within the resonator's cavity. A vector network analyser (VNA) is used to measure the S-parameters of the two-port resonator. The measurements were performed in three steps: once with the empty resonator, then with a bare quartz substrate and finally with the graphene loaded sample. An algebraic equation enabled us to extract the sheet resistance value of the samples from the knowledge of the net shift in the resonant frequency and linewidth of the resonator.

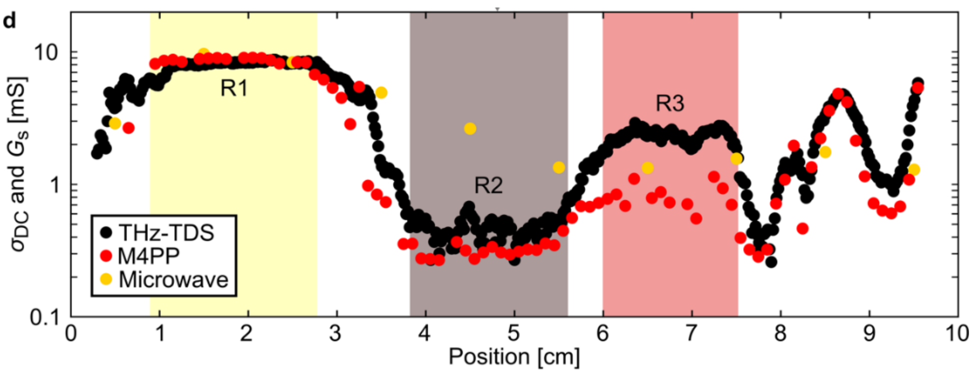


NPL setup for the microwave resonant cavity method. (a) Photo of sapphire puck and quartz spacer tubes inside copper housing (lid removed). (b)-(d) Schematic diagram of the high-Q sapphire dielectric resonator for measurement of the surface impedance of graphene samples. (b) A plain substrate, (c) an identical substrate with graphene film, and (d) with neither substrate, just the dielectric resonator and support structures. Adapted from Hao2013.

The results of the measurements of sheet resistance by these two high-throughput methods were compared with the results of other techniques based on contact and non-contact methods performed by the GRACE consortium. All the results indicated a good agreement of the data obtained by the different methods and constitute the inter-laboratory comparison reported at the end of this section.

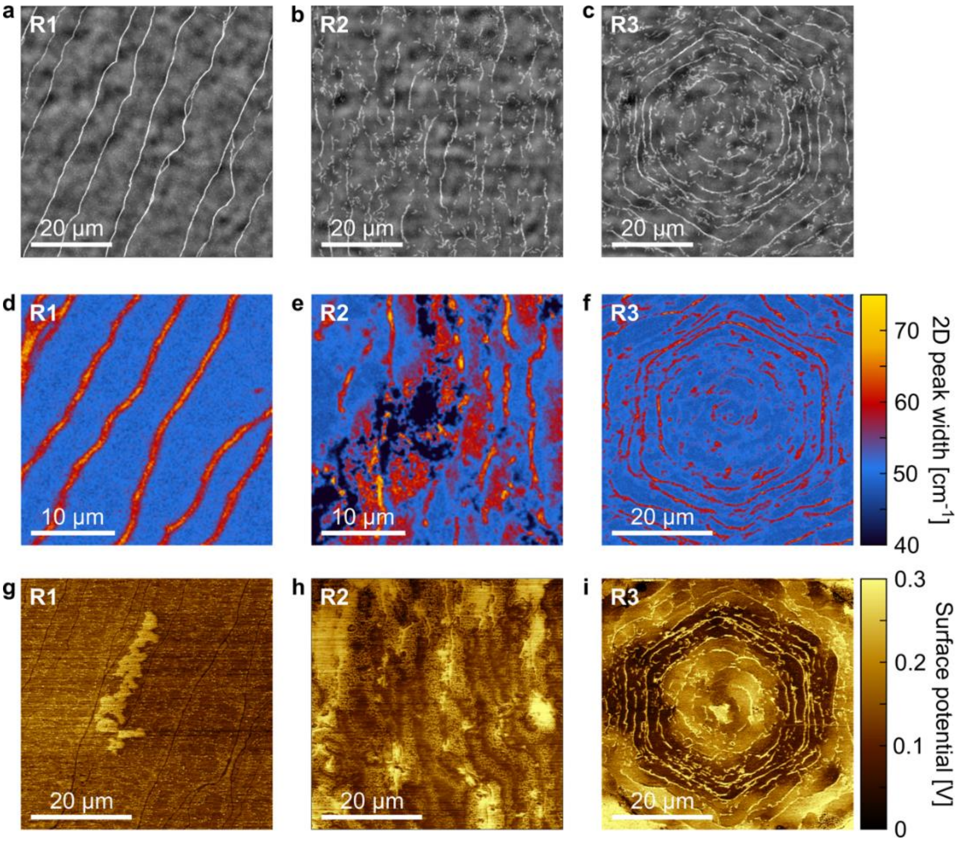
4.2.2 Wafer scale graphene characterisation

Different optical methods (THz-TDS, RAMAN), scanning probe microscopies (CLSM, KPFM) and electrical characterisation methods (4PP and MW cavity) were used to characterise the quality of graphene layer on a Silicon Carbide (SiC) substrate. The large-scale methods employed (THz-TDS and MW cavity) identify a variation of more than an order of magnitude of the sheet conductivity of a bilayer graphene across a 4“ SiC wafer. Remarkably the results of the three methods are in excellent agreement between each other.



Line plot of electrical conductivity σ_{DC} from THz-TDS, M4PP, and microwave measurements.

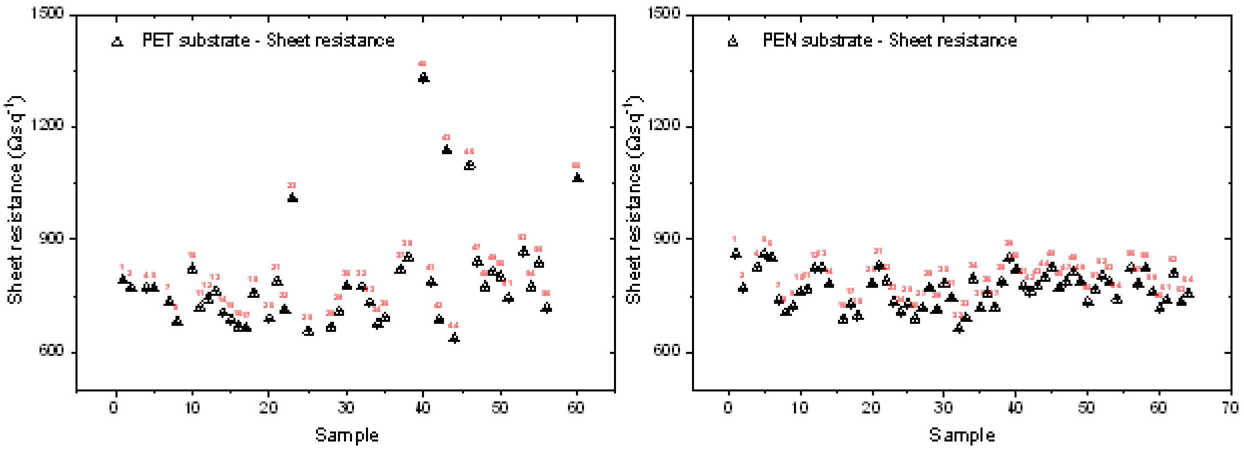
Three different regions on the sample surface were identified and analysed with the high-resolution methods (CLSM, KPFM and RAMAN) the result of this analysis is that the quasi freestanding graphene is affected by the imperfections on the SiC surface which are present prior the graphene growth. This has been proved by analysing different wafers originated by the same ingot that show the same features.



Graphene on 4" wafer SiC: (a−c) CLSM, (d−f) Raman 2D peak width, and (g−i) KPFM surface potential [Whelan2018]

Two 4" wafers of CVD graphene on PEN and PET were also fabricated by Graphenea and characterised via THz-TDS by das-Nano and with the vdP method by NPL. The wafer was diced and each individual 10 × 10 mm^2 sample was investigated. The results indicated a better homogeneity of the electrical properties, when using a PEN substrate rather than PET. All in all, the main outcome of the investigation was the agreement of a traceable method and a high-throughput method on the same samples.

Graphenea synthesized 10 × 10 mm² graphene samples on PEN and PET substrates.



Sheet resistance of 10 × 10 mm² graphene samples on PEN and PET substrates obtained with the vdP method.

4.2.3 Interlaboratory comparison

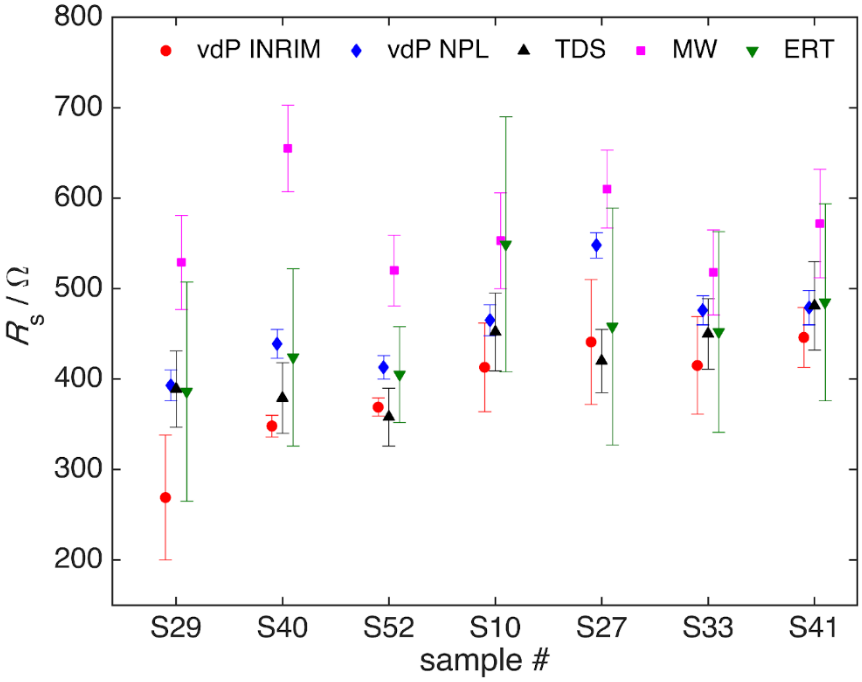
The consortium performed an interlaboratory study of contact- and non-contact electrical measurements with the aim to validate the latter. The comparison involved the following techniques:

- van der Pauw method (vdP)
- Electrical Resistance Tomography (ERT)
- Time-Domain terahertz spectroscopy (THz-TDS)
- Microwave Resonant Cavity (MW)

A subset of 7 samples from the batch was measured. The table below reports the measurement techniques that were used by the partners:

Technique	**Partner**	**Environment**
vdP	INRIM	laboratory
vdP	NPL	laboratory
THz-TDS	das-Nano	Industrial
ERT	INRIM	laboratory
MW	NPL	laboratory

This figure reports the outcome of the comparison in graphical format:



Sheet resistance of several samples of the same batch of CVD graphene on quartz, measured by means of many of the methods validated for graphene during the GRACE project.

4.2.4 How to compare vdP and TDS maps

The vdP method [Pauw1958] returns a global conductivity value of the sample, σ_{vdP}, while the TDS returns a map of local conductivity values $\sigma_{i,j}$. To compare these two methods is necessary to properly average the values $\sigma_{i,j}$. One of the basic assumptions of the vdP method is the sample's conductivity. This because the value σ_{vdP} is an average of the local conductivity, with some regions of the sample being weighted more than others. So σ_{vdP} is representative of the sample only under this hypothesis. This matter is discussed in depth in a series of papers [3, 4, 5, 6]. In presence of conductivity inhomogeneities, these will affect the global result as a function of their position. This fact implies that the conductivity calculated from the TDS map should be a certain weigthed average of the local $\sigma_{i,j}$ values, in order to take into account, the nature of the vdP measurement and properly compare the two methods.

In general, the outcome of a vdP measurement can be written as

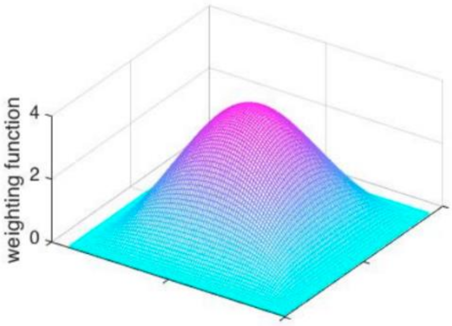
$$\sigma_{vdP} = \int S\ \sigma(r) w_{vdP}(r) dS,$$

where σ(r) is the local conductivity and w(r) is a weighting function which accounts for the sensitivity of the vdP measurement to the local conductivity. The function w(r) depends on the sample shape, the contacts position and emerges naturally from the uneven distribution of the electric field inside the sample when performing the required vdP trans-resistance measurements. In the linear limit of weak perturbations, w(r) can be considered independent of σ(r). For each vdP measurement configuration, the weighting function w_{vdP} can be calculated using the method in Sec. IV of the following paper: D. Koon and C. Knickerbocker, "What do you measure when you measure resistivity?" Rev. Sci. Instrum., vol. 63, no. 1, pp. 207–210, 1992. The vdP method requires two different trans-resistance measurement configurations, to which correspond two weighting functions w1 and w2. In order to obtain the vdP weighting function These are averaged as:

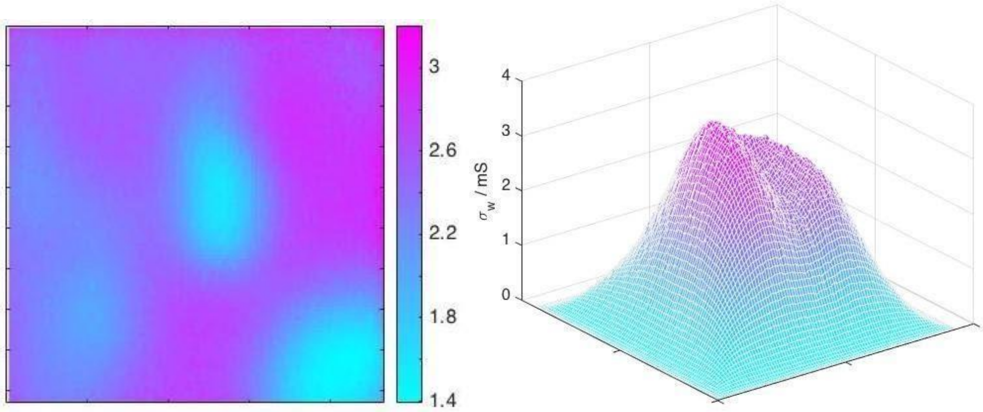
$$w_{vdP} = w_1 + w_2$$

The weighting function w_{vdP} shown here was obtained with a numerical solution of the problem in (2) on a

100 × 100 elements square grid (of the same density of the TDS map). The weight of the conductivity value in the centre is ≃ 3.2 while on the border it is 0.



The map below shows the TDS map (Das-Nano, Oct. 2017) of the sample 28 (batch 1, Sept. 2017).The weighting function w_{vdP} (calculated above) was applied to this map in order to obtain the *weighted* map. Using the *weighted* map, both the simple average $\langle\sigma_{map}\rangle$ and the weighted average were calculated to simulate the ideal result of a vdP measurement.



Finite element model geometry (a) and ERT map of the graphene sample n. 28 (b). The conductivity colour-scale is in mS.

4.2.4 Key Outputs from Objective 2

High-throughput methods for the characterisation of graphene in industrial / in-line environment, were implemented, tested and validated by comparison with the traceable methods developed in Objective 1. This objective was fully met.

4.3 Objective 3

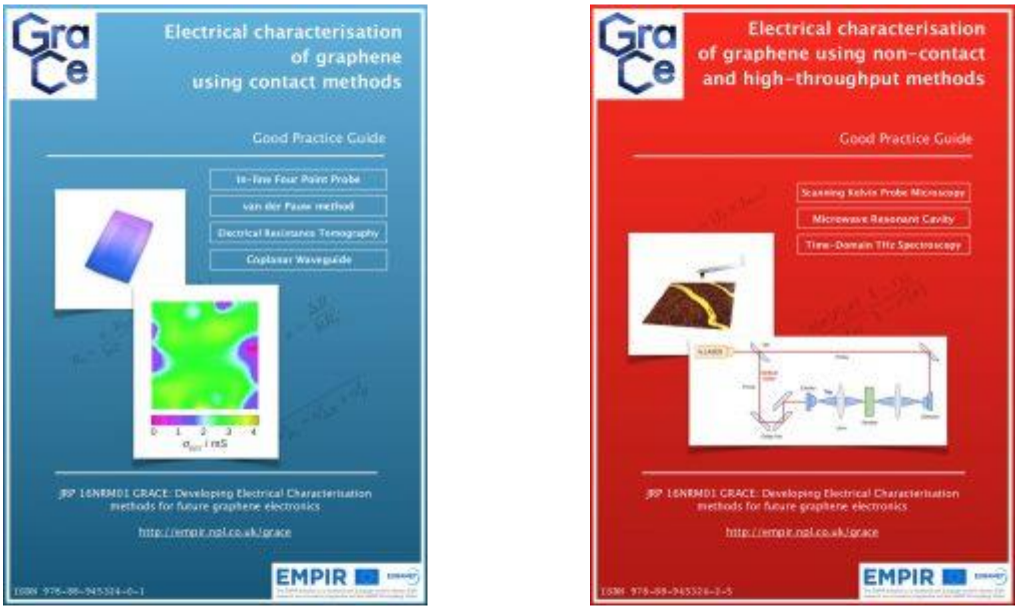
4.3.1 Good Practice Guides

The measurements performed in Objectives 1 and 2 have been documented as written measurement protocols, which formed the basis of the two GPGs. These are currently published as open access on the project's website http://empir.npl.co.uk/grace/. The GPG for contact methods on graphene has been written by CEM, INRIM, NPL, Graphenea, UoM, ISC and VDE. The GPGs are entitled:

- "Good Practice Guide on the electrical characterisation of graphene using contact methods", 2020, Edited by A. Fabricius, A. Catanzaro and A. Cultrera, ISBN: 978-88-945324-0-1.
- "Good Practice Guide on the electrical characterisation of graphene using non-contact and high throughput methods", 2020, Edited by A. Fabricius, A. Cultrera and A. Catanzaro, ISBN: 978-88-945324-2-5.

These guides provide protocols for determining the electrical properties of graphene sheets, on insulating substrates, using contact methods: that is by physical contacting the graphene surface with metallic electrodes.

Depending on the methodology the properties that can be measured are the electrical sheet conductivity, the concentration and mobility of electrical charge carriers. Each protocol gives advice to understand the measurement principle, how to implement it to perform reliable measurements traceable to the International System of units, and hints to express the corresponding measurement uncertainty. The guide is for producers and users of graphene who need to understand how to measure the electrical properties of graphene.



Cover pages of the two GPGs published by the GRACE consortium in 2020.

Such information is essential in a host of technology application areas where graphene may be used. This guide provides a detailed description of how to determine the key electrical properties of graphene, so that the graphene community can adopt a common, metrological approach that allows the comparison of commercially available graphene materials. The guide is intended to form a bedrock for future interlaboratory comparisons and support the direct collaboration with international standardisation organisations. The guide is for users in research and industry who have experience with and access to the advanced techniques described herein. It is targeted at analytical scientists and professionals who have a bachelor's degree in science.

4.3.2 Key Outputs

Two good practice guides were published and distributed among IEC/TC 113 as inputs for new and revised documentary standards. Therefore, this objective was fully met.

4.4 Objective 4

In the last few years standardisation activities regarding graphene were one of the major topics in IEC/TC 113 "Nanotechnology for electrotechnical products and systems". This includes the development of standardised specifications, the related measurement techniques for graphene as well as terminology (in liaison with ISO/TC 226 "Nanotechnology"). From the beginning of the project, the committee was involved in EMPIR GRACE as the major stakeholder. An important step in the interaction between TC 113 and GRACE was establishing a C liaison between the two on the TC meeting in Busan in October 2018. Category C liaison organisations have the right to participate as full members in a working group or project team of IEC. Category C liaison experts act as the official representative of the organisation by which they are appointed. This allowed the GRACE consortium to propose the development of new standards and nominate technical experts and project leaders for the related project teams in IEC/TC 113.

Due to this liaison, IEC/TC 113 has been regularly updated by the GRACE consortium on their progress. GRACE reported the scientific status of the project at all meetings which were held during the EMPIR project duration. Furthermore, GRACE and IEC/TC 113 organises a Joint Workshop "Electrical Characterisation of Graphene" during their spring meeting in Madrid, Spain, in May 2019.

IEC/TC 113 has several liaisons with other committees in IEC and other Standard Developing Organisations to avoid double work and contradicting standards. Through these liaisons the committees share documents and participate on each other's meetings. Liaison organisations of IEC/TC 113 interested in graphene are ISO/TC 229 "Nanotechnologies", IEC/TC 119 "Printed Electronics", CEN/TC 352 "Nanotechnologies" and the Graphene Flagship Standardisation Committee (GFSC).

The GRACE consortium has developed and validated a significant number of measurement techniques for the electrical characterisation of graphene. Early adoption of the need of the IEC technical committee ensured that GRACE was able to make normative contributions to one standard at the CD stage and working drafts for two new work item proposals. After discussion of the documents at several IEC/TC 113 meetings they were circulated to the IEC member countries for comments and voting. The IEC/TC 113 liaison organisations are invited to comment as well. The GRACE Good Practice Guides were distributed as informative documents for discussion at the next meeting of IEC/TC 113 in October 2020.

All in all, five standards in the work program of IEC/TC 113 are directly supported by EMPIR GRACE results. These are:

- 62607-06-04: Nanomanufacturing - Key control characteristics - Part 6-4: Graphene - Surface conductance measurement using resonant cavity (Development of Ed. 2.0)
- 62607-06-07: Nanomanufacturing – Key control characteristics – Part 6-7: Graphene material – Sheet resistance: van der Pauw method (New Work Item Proposal)
- 62607-06-08: Nanomanufacturing – Key control characteristics – Part 6-8: Graphene material – Sheet resistance: In-line four-point probe (New Work Item Proposal)
- 62607-06-10: Nanomanufacturing – Key control characteristics – Part 10: Graphene film – Sheet resistance: Terahertz time-domain spectroscopy (New Committee Draft)
- 62607-06-25: Nanomanufacturing – Key control characteristics – Part 6-25: Two-dimensional materials – Doping concentration: Kelvin Probe Force Microscopy (Technical input).

The activities based on the New Work Item Proposal will be supported by GRACE scientists even after the project duration as the project leader.

4.4.1 Key Outputs from Objective 4

This objective was fully met. Five standards in the work program of IEC/TC 113 are directly supported by EMPIR GRACE input, ranging from validated measurement protocols to experimental results.

5 Impact

The project has prepared 12 open access peer-reviewed papers (10 have been published and 2 have been submitted to journals for publication. The scientific outcomes of the project have also been presented at 27 conferences and events, giving focus to the status of graphene standardisation and its contribution to the commercialisation. Key events have included: 9th annual Recent Progress in Graphene and Two-dimensional Materials Research Conference (RPGR2017), Graphene2018, GraphChina 2018 and 2019, the 15th International Conference on Nanosciences & Nanotechnologies, CPEM 2018, Graphene Week 2018 and 2019, E-MRS 2020 and various other conferences and workshops. Substantial scientific outreach was also delivered to the general public through mainstream media.

Impact on industrial and other user communities

The project stakeholder committee consisted of 12 members from 7 countries. The inaugural meeting of the stakeholder committee took place during the mid-term meeting of the project which was organised in concomitance of the Graphene Week conference, 10-14 September 2018 in San Sebastian, Spain. A second stakeholder meeting was held on May 2019 in Madrid, Spain. In addition, a "industry friendly workshop" was held on September 2019 in Helsinki, Finland within the Graphene Week and at the Graphene Industrial Forum 2020.

The GPGs produced during the project, will help industrial end users in the practical implementation of different methods for the electrical characterisations of graphene and as part of the project's research such methods will be tested under industrial environmental conditions. The GPGs content has been presented to the stakeholder community at the stakeholders' events above listed. In order to incorporate the needs of industrial end users, input from the project's stakeholders committee was considered during the drafting process.

Several companies have expressed interest in the exploitation of the techniques developed in the project. LG Electronics Inc.- Materials & Production engineering Research Institute and the National Institute for Standards and Technology (NIST) are already using the project's Conductivity mapping by Electrical Resistance Tomography facility for the characterisation of CVD and epitaxial graphene samples (respectively).

Impact on the metrology and scientific communities

The project's GPGs were based on the measurement protocols developed by the consortium. These protocols include descriptions that can enable scientific users to understand and implement the electrical characterisation methods, and explain how to express a measurement uncertainty, check the assumptions underpinning the method, and validate it. The content of these protocols has been disseminated by presentations at scientific workshops and conferences, and during the project mid-term meeting and the stakeholder meetings. Two major training events were organised by the consortium in connection with international meetings and conferences: a "Joint Workshop of IEC/TC 113 and EMPIR project 16NRM01 GRACE on the electrical characterisation of graphene - Meeting for stakeholders" (Madrid, May 2019), and the workshop "GRACE — Methods for the Electrical Characterisation of Graphene" (Helsinki, Sept. 2019).

Furthermore, the project partners addressed the issues related to the comparability of results achieved with different measurement methods for the electrical characterisation of graphene. An interlaboratory comparison study confirmed the reliability of the implementation of the different methods for the electrical properties of graphene measurement, in particular for non-contact and high-throughput methods.

Impact on relevant standards

At the IEC/TC113 committee meeting held on November 2017 in Shenzhen, China, the project was presented to the working group dedicated to graphene. IEC/TC113 has accepted from the consortium the drafts of four potential new technical specifications, that are now at various stages of consideration and approval by the Committee. Additionally, project consortium provided regular updates on the project's progress; in particular on the drafting process of the validated measurement protocol; at relevant IEC/TC 113 meetings. A further discussion of the inputs provided by GRACE will take place at the next IEC/TC 113 meeting planned for October 2020. Also, presentations of the project and its results were given to the EURAMET Technical Committee on Electricity and Magnetism (TC-EM).

Longer-term economic, social and environmental impacts

In the longer term, the project outcomes will contribute to legislation related to graphene and its specifications as an industrial product, at the European level. The high-throughput methods developed in the project; will lead to improved industrial characterisation, quality and efficiency; which will have an impact on the whole graphene supply chain, from producers to end-users. The development of enhanced products based on graphene will also have a positive impact on energy efficiency of lighting and electrical energy storage. Furthermore, applications of graphene are predicted to be viable for healthcare monitoring; the potential toxicity of graphene-based products is closely related to their long-term stability, that could be monitored with the methods developed in this project.

6 List of publications

[1]. Melios, Christos, et al. "Detection of Ultralow Concentration NO_2 in Complex Environment Using Epitaxial Graphene Sensors." *ACS sensors* 3.9 (2018): 1666-1674. https://doi.org/10.1021/acssensors.8b00364; https://arxiv.org/abs/1808.09776

[2]. Whelan, Patrick R., et al. "Electrical Homogeneity Mapping of Epitaxial Graphene on Silicon Carbide." *ACS applied materials & interfaces* 10.37 (2018): 31641-31647. https://pubs.acs.org/doi/10.1021/acsami.8b11428

[3]. Melios, Christos, et al. "Water on graphene: review of recent progress." *2D Materials* 5.2 (2018): 022001. https://arxiv.org/abs/1804.09518; https://arxiv.org/abs/1804.09518

[4]. Panchal, Vishal, et al. "Confocal laser scanning microscopy for rapid optical characterization of graphene." *Communications Physics* 1.1 (2018): 83. https://www.nature.com/articles/s42005-018-0084-6

[5]. Cultrera, Alessandro et al. “Mapping the conductivity of graphene with Electrical Resistance Tomography.” Scientific Reports 9 .1 (2019), 10655. https://doi.org/10.1038/s41598-019-46713-8

[6]. Marzano, Martina et al. “A correlation noise spectrometer for flicker noise measurement in graphene samples.” Measurement Science and Technology 30.3 (2019). https://doi.org/10.1088/1361-6501/aafcab

[7]. Melios, Christos et al., “Towards standardisation of contact and contactless electrical measurements of CVD graphene at the macro-, micro- and nano-scale,” Scientific Reports, 10, 3223 (2020) https://doi.org/10.1038/s41598-020-59851-1

[8]. Fabricius, Alexandra, Catanzaro, Alessandro and Cultrera, Alessandro (Ed.), “Electrical characterisation of graphene using contact methods”, (2020), ISBN: 978-88-945324-0-1. https://iris.inrim.it/handle/11696/63312; https://arxiv.org/abs/2007.13348

[9]. Fabricius, Alexandra, Cultrera, Alessandro, and Catanzaro, Alessandro(Ed.), “Electrical characterisation of graphene using non-contact and high-throughput methods”, (2020), ISBN: 978-88-945324-2-5. https://iris.inrim.it/handle/11696/63314; https://arxiv.org/abs/2007.14047

[10]. Cultrera, Alessandro and Callegaro, Luca, “A Simple algorithm to find the L-curve corner in the regularisation of ill-posed inverse problems”, IOP SciNotes, 1, 025004 (2020) https://doi.org/10.1088/2633-1357/abad0d