EMPIR Call 2015 – Health, SI, Normative and Research Potential

Selected Research Topic number: **SRT-n01**
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Title: Characterisation of graphene and 2-D materials to enable commercial applications

Abstract

The accurate and reproducible characterisation of different types of graphene materials, grown or exfoliated using various production routes, is now a crucial barrier that must be overcome for this emerging industry to flourish. Commercialisation and the uptake of this material by end-users is currently being hindered as industrially-produced graphene is not correctly characterised and thus may not be suitable for specific applications, or has the same properties as bulk material. The required structural, chemical and electrical measurement techniques and related protocols for graphene and other 2-D materials must be identified, developed and standardised at the European and International level to overcome the gap in metrology in this area.

Keywords

Graphene, structural, chemical, electrical, characterisation, beyond CMOS, layered materials, graphite, standardisation, nanotechnology

Background to the Metrological Challenges

Graphene – a single-layer of carbon atoms – was first isolated in a laboratory only eleven years ago but is already being investigated by industrial entities, ranging from small-to-medium enterprises (SMEs) to multinational corporations, for a large range of applications such as membranes, composites, sensors, transistors, Li-ion batteries, transparent conductive films and supercapacitors [1]. Thousands of academic publications and patents related to graphene are now published every year [2] and the future world-wide graphene market is estimated to be worth about 400 M$ in 2024 [3], which equals a growth of more than 300 % each year. Currently there is no legislation at the national or European level directly linked to graphene, and the standardisation of the area will be critical for industry regulation in the future. There are currently over 100 commercial ‘graphene’ producers worldwide [4], including leading graphene producers in Europe, with an offering of materials with vastly different properties.

A graphene-specific business-plan has been initiated within CENELEC Workshop SGRM [5] and development of CEN/TC352/WG1/PG1 ‘Guidance on measurands for characterising nano-objects and materials that contain them' is currently underway within CEN/TC 352 ‘Nanotechnologies’, which includes graphene and other 2-D materials. Meanwhile, the first standards related to graphene are now under development within the ISO/TC 229 - ‘Nanotechnologies’ and IEC/TC 113 - ‘Nanotechnology standardisation for electrical and electronic products and systems’ international standards committees, with eight activities now approved for development.

The main requirement in this area is the development of the pre-normative standardisation work related to the structural, chemical and electrical characterisation of graphene and other 2-D materials to enable commercialisation. This requirement has already been identified as a priority in the EMPIR TP “Pre- and co-normative research (2015)” call, through a formal consultation process within CEN and CENELEC, defining an objective to “develop pre-normative standardisation related to characterisation and commercial exploitation of graphene”. The recent activity in the development of International Standards shows that this is the opportune time to start directly investing in pre-normative research related to graphene standards.

The current state-of-the-art characterisation tools for graphene and 2-D materials are techniques such as optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), Raman spectroscopy, ellipsometry, X-ray photoelectron microscopy (XPS), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), scanning Kelvin probe microscopy (SKPM), spread resistance microscopy, the van der Pauw method, microwave resonant cavity

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methods and Terahertz time-domain spectroscopy. These methods have already been extensively used to measure the structural, chemical and electrical properties of graphene and 2-D materials, and a substantial knowledge base has been established over the last decade of graphene research. This capability (both equipment and expertise) typically resides in NMIs and Universities, but there are no established measurement standards in this area that allow the cross-comparison of even a few basic properties of graphene and other 2-D materials. Crucially, although few of the (typically more affordable) techniques are used within industry, the level of expertise required to develop traceable and comparable methodologies does not currently reside within industry. SMEs, particularly, only have a limited ability to compete in this commercial area until usable and standardised methods are available to them.

Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the protocol.

The JRP shall focus on the characterisation of graphene and other related 2-D materials for use in standards being developed in ISO/TC 229 'Nanotechnologies' and IEC/TC 113 'Nanotechnology standardisation for electrical and electronic products and systems' and related groups.

The specific objectives are

1. To develop standardised, scalable and transferable methods for the structural characterisation of graphene and other 2D materials, where the main measurands of structural quality are number of layers, thickness, domain size, structural defect density. The methods should be applicable for graphene and other 2D materials produced by different methods, e.g. CVD, epitaxial growth, liquid-phase exfoliation, and existing in different forms, e.g. films, powders, liquid dispersion.
2. To develop characterisation techniques for the accurate and precise measurement of the chemical composition of graphene, functionalised graphene and other 2-D materials, to quantify parameters down to 0.1 atomic %. The hierarchy of suitable methods and the methodologies required to enable the chemical composition of materials to be characterised and decoupled from the level of contamination due to environmental or processing conditions, whether contained within a graphene powder or as a surface contaminant on a graphene film, should also be developed.
3. To develop methodologies for the contact and non-contact electrical characterisation of graphene and other 2D materials. Properties such as carrier density and mobility, sheet and contact resistance, work function should be addressed.
4. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain, ensuring traceability of measurement results to graphene producers and end users and to contribute to the development of standards by international standards committees concerning graphene and other 2-D materials. Dissemination of project results in the form of Good practice Guides should take place as early as possible to establish a standardised approach.

The proposed research shall be justified by clear reference to the measurement needs within strategic documents published by the relevant Standards Developing Organisation or by a letter signed by the convenor of the respective TC/WG. EURAMET encourages proposals that include representatives from industry, regulators and standardisation bodies actively participating in the projects.

Proposers should establish the current state of the art, and explain how their proposed project goes beyond this. In particular, proposers should outline the achievements of the EMRP project SIB51 GraphOhm and how their proposal will build on those.

EURAMET has defined an upper limit of 1.0 M€ for the EU contribution to this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 30 % of the total EU Contribution to the project. Any deviation from this must be justified.

Any industrial partners that will receive significant benefit from the results of the proposed project are expected to be unfunded partners.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community, describing how the project partners will engage with relevant communities during the project to facilitate knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the “end user” community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

- Address the SRT objectives and deliver solutions to the documented needs,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the graphene and 2-D materials sector.

You should detail other impacts of your proposed JRP as specified in the document “Guide 4: Writing Joint Research Projects (JRPs)”.

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include the best available contributions from across the metrology community. Specifically the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work

Time-scale

The project should be of up to 3 years duration.

Additional information

This topic is in response to needs identified by CEN/CENELEC published at http://msu.euramet.org/pre_norm_2015/index.html#stage1-orientation (priority 14: Characterisation of graphene and 2D atomic materials for electrical applications).

The references were provided by PRT submitters; proposers should therefore establish the relevance of any references.

[1] Science and technology roadmap for graphene, related two-dimensional crystals, and hybrid systems, European Graphene Flagship, 2014
[2] Graphene: The worldwide patent landscape in 2013, Intellectual Property Office, UK, 2013
[3] Graphene Markets, Technologies and Opportunities 2014-2024, IDTechEx, 2014
[4] www.graphene-info.com
[5] CENELEC WS SGRM Business Plan, CENELEC, 2014
[6] Materials for Key Enabling Technologies, E-MRS, ESF & MatSEEC, 2011
[7] Molecular Precision Manufacturing - An Emerging Paradigm for the Commercialization of Graphene and Advanced Nanomaterials, SNS Special Letter, 2015
[8] Key Enabling Technologies: Final Report, European Commission, 2011