



 SIO GRAFEN

Pre-study Graphene characterisation and standardisation

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Summary

In order to make novel materials such as graphene certified and widely used, it is necessary to establish a reliable characterisation methodology for the material under the authorised international scheme for standardisation. This prestudy is thus divided into two parts. The first lists the graphene properties that can be characterised together with measurement methods and Swedish actors capable of performing these measurements. The second part aims to highlight areas and properties where standardized characterisation methods are of importance to Swedish industry, but that are not currently being developed internationally. This consequently describes where a potential Swedish standardisation effort is recommended.

The important properties of graphene films and flakes have been identified and the characterisation methods are listed. Identification of laboratories that are able to characterise the films and flakes accordingly have been assembled in the attached excel sheet. We recommend that this network is strengthened in order to form a future national graphene characterisation network. This would significantly facilitate Swedish graphene development.

The Graphene Flagship is mainly focusing on standards for the structural properties of graphene (such as the number of layers and defects) and the electronic properties, leaving other properties for later examination. In comparison with areas identified by SIO Grafen, this leaves a gap, for example in coatings and membranes, thermal conductivity, biological activity and toxicity. Thermal conductivity and toxicity are of huge interest globally and it is not clear which position Sweden should take in these areas. There is no unique competence in thermal conductivity of graphene within Sweden. However, Karolinska Institutet is a strong actor on toxicity studies, but a better overview of the current graphene toxicity research situation is needed before specific standardisation work can be initiated. There is less international interest in coatings and membranes, leaving this a priority area for potential future standardisation work within Sweden. Existing standardised characterisation methods for coatings and membranes without graphene should be evaluated and potential graphene related additions should be investigated.

We also identified a group of Swedish actors who potentially can engage the above suggested work and we have identified necessary steps to initiate the standardisation discussions in the areas identified by SIO Grafen, which are not currently covered by the Graphene flagship.

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1. Introduction

In order to make novel materials such as graphene both reliable and widely used, it is necessary to establish a reliable characterisation methodology for the material under the authorised international scheme for standardisation.

Standardisation is the process of developing and implementing technical standards based on the consensus of different interested parties, which can include companies (both producers and users), interest groups, characterisation laboratories, standards organisations, industry associations and governments. Standardisation aims to maximise repeatability, quality, compatibility, interoperability and safety. It also plays an important role for the company businesses where a standardised material, method or property may have an added value on the market.

When there is a lack of standardisation for measurements of any property, everyone uses their own protocols and instruments, which can lead to widely differing results for the same quoted property and thereby lead to misunderstanding by the users. Using the same protocol, specifying relevant equipment parameters, sample preparation protocols et cetera, the variation can be reduced significantly. This is a necessity in order to get quality control in the production of products including graphene materials.

Characterisation and standardisation has been mentioned and discussed at several SIO Grafen meetings. During the strategic workshops held in Västerås on 13 April 2016, it was clear that there is a need to standardise the way graphene materials are characterised, and that the role SIO Grafen and Sweden should play in this need to be addressed. This prestudy serves as a first step to address these questions and it suggests how Sweden and SIO Grafen should work.

Graphene has a huge potential in many applications, but before graphene can fulfil this potential large scale production processes and how to do it in a reproducible manner needs to be solved. Many different manufacturing techniques are being used, yielding different properties and qualities. The ability to transfer the properties from microscopic materials to macroscopic scale and in combination with other materials, is another important topic. The characterisation of graphene, both in itself and in combination with other materials, is consequently important.

Since graphene has so many remarkable properties there are many features that need to be characterised. The excel document attached to this report, lists the most important properties of graphene together with characterisation methods and laboratories who can perform these. The properties largely depend on the production method. The lists have therefore been divided into two categories, graphene films and graphene flakes. The graphene flakes are most often produced from graphite by a liquid-based technique, whereas the films are most often produced by chemical vapour deposition (CVD) or epitaxial growth on silicon carbide.

Currently, there is a large number of suppliers of graphene related materials, as well as an even larger number of companies that are currently evaluating or intending to use graphene related materials for their next generation products. The lack of standards has been identified as one of the largest threats for industrial use of graphene. It is therefore crucial to standardise the various terminologies and definitions of various graphene related materials (GRMs) as well as the various methods to evaluate its numerous physicochemical properties. The main advantage of such a set of standards would be the simplicity of a reliable comparison of the wide range of graphene products available on the market in order to select the best material for any given application.

A suggestion for a standardisation network/platform for future collaboration and lobbying activities is described in the final chapter.

1.1 Definition of Graphene Materials

There is no complete consensus for the nomenclature of graphene-related materials. The word graphene refers to a one-atom-thick sheet, but is sometimes also used to describe significantly thicker materials. This can obviously lead to confusion as the properties of the material changes with the number of layers.

A few articles have discussed the topic and the recommended nomenclature can be found in Table 1. In order to avoid confusion, the term Graphene Related Material (GRM) is sometimes used, which refers to all these materials.

There are many other 2D materials apart from graphene. The most widely studied are hexagonal boron nitride and molybdenum disulphide. All these have different properties and could potentially be used in many different applications. This is a quickly growing research

area, but this document is focused on graphene-related materials as this is the most mature area.

Table 1: Recommended graphene vocabulary according to Bianco *et al.*².

Name	Property
Graphene (monolayer)	1 layer
Bilayer graphene	2 layers
Few-layer graphene	2-5 layers
Multilayer graphene	2-10 layers
Graphite nanoplatelets, graphite nanosheets, graphite nanoflakes	> 10 layers but < 100 nm
Graphene oxide (GO)	C/O atomic ratio < 3.0, closer to 2.0

2. Characterisation of Graphene

The material suppliers currently describe their products in different ways, making it difficult for purchasers to make a comparison. There are no common guidelines for describing the properties and which information the producer needs to supply in their product specification. The role of this guide is to highlight the most important parameters that are relevant for specific forms of graphene.

It would be useful to make a recommendation to all the graphene producers (at least in Sweden) to get their graphene characterised, and later on, when available, certified at accredited research institutes or industries.

Through discussions within the project group, as well as using input from the workshop at SIO Graphene's conference Svenskt Grafenforum in 2016, the most important properties of graphene that need to be characterised have been compiled. A list of these properties is shown in Table 2 and Table 3. The tables are divided into "Basic properties" and "Additional properties". The "Basic properties" are the ones that influence the functionality of most applications and are the most frequently characterised properties, while the "Additional properties" are all other important properties that can have more or less importance dependent on the specific application. Thin films and flakes of graphene are discussed separately.

It should be noted that for commercial applications the performance properties are the most important properties. These may or may not be linked to the basic/additional properties of graphene listed here. Other factors such as the preparation during the manufacturing process will influence the outcome as well as the behaviour of the material. For example, if heating is conducted during a manufacturing step, the original oxygen content of graphene oxide (GO) may be reduced.

Furthermore, from a biological activity perspective, graphene has the potential to be anti-microbial, anti-fungal or anti-fouling which could be important in many fields, and could be important for many different products, and thus Swedish industry. Sweden has a very good position to develop tests to support novel products as there already is ongoing research on the biological activity at Chalmers University of Technology.

Graphene films are typically produced either by chemical vapour deposition (CVD) or by epitaxial growth on silicon carbide. The important properties of the graphene films are listed in Table 2.

Table 2: Basic and additional properties of graphene films (in alphabetical order).

Basic properties	Additional properties
Conductivity/sheet resistance*	Chemical composition (including impurities)
Defects*	Doping
Grain size/lateral size*	Heat capacity
Number of layers*	Mobility
	Permeability of gases and liquids
	Strain
	Surface coverage
	Surface energy/contact angle
	Surface topography
	Thermal conductivity
	Transparency

* Standardisation work addressed by Graphene Flagship and IEC/ISO

Graphene flakes are typically produced in a liquid and are also often supplied as a dispersion. This introduces additional important properties of the system, as shown in Table 3. In addition, films made from dispersions can be characterised in a similar manner as those listed in Table 2.

Table 3: Basic and additional properties of graphene flakes (in alphabetical order).

Basic properties	Additional properties
C/O ratio	Chemical composition / functionalisation
Flake morphology (including conformity)	Defects*
Lateral size	Distribution of lateral sizes
Number of layers*	Interlayer distance
	Heat capacity
	Specific surface area
	Surface energy
	Thermal conductivity
Additional properties for graphene dispersions	
Concentration	Dispersants
Solvent (Aqueous or non-aqueous)	pH
Storage stability	Viscosity
	Zeta Potential

* Standardisation work addressed by Graphene Flagship and IEC/ISO

For the dispersions, we recommend that the producers state if an aqueous or organic solvent (specified) is used. If dispersants are used, it should at least be stated which ones that have been used, using the following categories: anionic, non-ionic or cationic, to avoid the use of incompatible and thus destabilising additives during use.

It is expected that a combination of basic and additional properties can be used as an initial guide to find the most interesting graphene materials for each type of application or product. Companies are expected having to perform complimentary testing in addition to the properties listed by manufacturers, to fully capture the particular requirements of their industrial application. Nevertheless, they may be helped by the introduction of “generic” application

tests, for example, how well dispersed the graphene flakes are in standard oils or in polymers of different polarities, the thermal and electrical conductivity, viscosity and/or mechanical properties of these composites et cetera.

In summary, users of graphene-related materials would be helped by having both basic and additional properties characterised by the supplier. Characterisation services for these kinds of measurements could also be of high value.

There are many different characterisation methods of GRMs. The most frequently used ones are listed in the attached excel sheet, along with which properties each method can characterise.

3. Standardisation of Graphene

Characterisation of novel materials is prone to variability. This is usually due to the developing nature of the underlying physical models used to simulate/extract useful parameters/values from the measured data. This leads to a situation where different actors performing the same measurements of the exact same material can obtain significantly different values. The variability arises from the differences in sample preparation protocols, instrument settings and variations in models used for data interpretations. Different applications of graphene-related materials originate from different physicochemical aspects, such as conductivity, transparency, gas diffusion barriers or novel applications arising from functionalised graphenes, such as corrosion protection, catalyst supports, amongst others. Thus, it is important to have standardised methods for a reliable and repeatable evaluation of the various parameters.

According to the World Bank, one of the most important economic benefits of standards is the increased productivity and innovative efficiency. Innovation is supported by standardisation by providing an important basis for developing solutions as standards increase safety, interoperability, competition and help to remove any trade barriers.

Standards are developed either at a national level (SIS) or international level, either through organisations that work globally (i.e. the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC)) or just within Europe (i.e. [CEN](#), [CENELEC](#) and the European Telecommunications Standards Institute (ETSI)). The workflow for producing standards is similar in these organisations. Figure 1 describes the workflow within ISO.



Figure 1: Workflow of standardisation within the International Organization for Standardization (ISO).

The main working committees that should tackle the standardisation of graphene are the ones related to nanotechnology, in particular, SIS/TK 516 Nanoteknik at Swedish level and ISO Technical Committee (TC) 229 at an international level. It is also important to highlight that ISO TC 229 has several working groups (WG) and joint working groups (JWG) that cover different areas of nanotechnology, as highlighted in the scheme below:

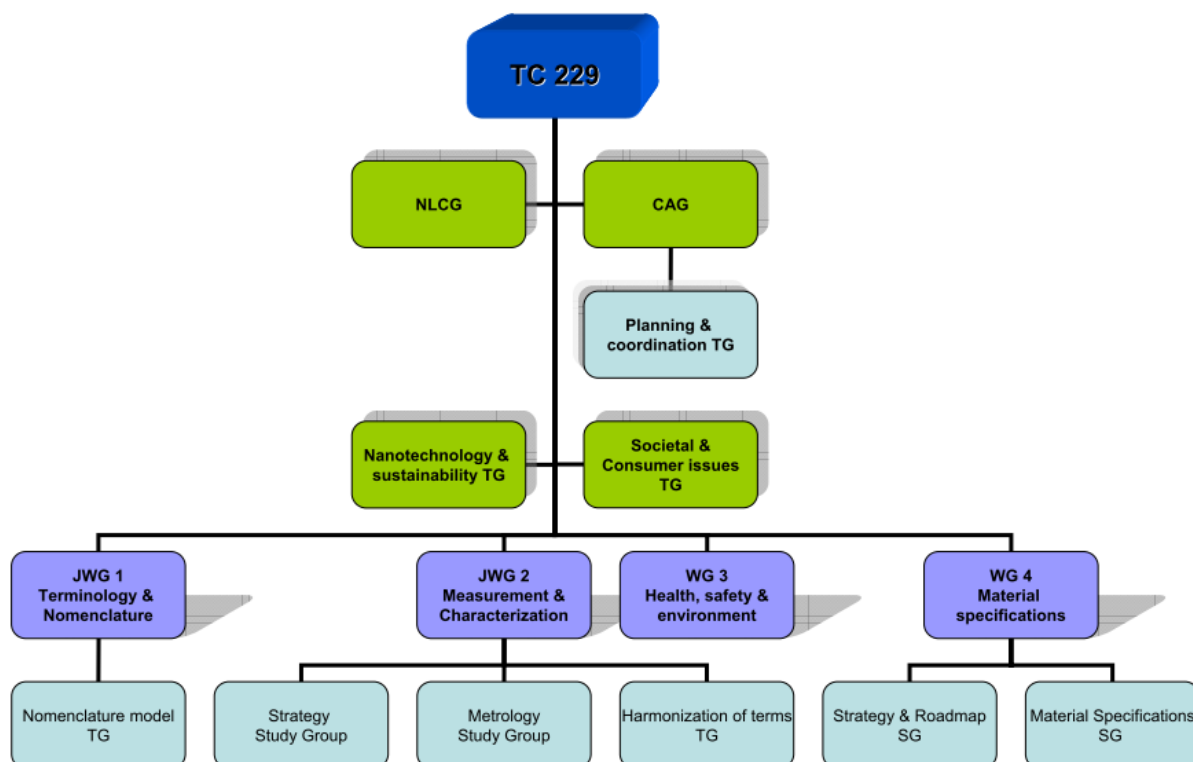


Figure 2: Structure of the ISO/TC 229, which is focused on nanotechnology.

Obviously, there is a need to collaborate with most working groups to cover all aspects related to the standardisation of graphene, from terminology to characterisation, from health and safety to material and product specifications.

3.1 Status of Ongoing International Standardisation Work

The status of the standardisation work in the Graphene Flagship – as described below, is from a short report provided by the Graphene Flagship and from interviews with Katarina Boustedt, head of administration and part of the team that is leading the standardisation work in the Graphene Flagship.

The main consortia that are working on the standardisation of graphene are the Graphene Flagship, [the Graphene Council](#) and the China Innovation Alliance of the graphene industry (CGIA). There is no direct collaboration between the Graphene Flagship and the CGIA. Other important actors that are involved in the standardisation work are the National Graphene Metrology Centre of NPL (the National Physical Laboratory in the United Kingdom), the [Graphene Stakeholder association](#), and the National Metrology Institute of Germany ([PTB](#)).

The metrology research program at the European level (EMPIR) is currently financing very few projects in the broad nanotechnology area.

The Graphene Flagship is collaborating with the International Electrotechnical Commission (IEC TC113) and the International Organization for Standardization (ISO TC229). The Graphene Flagship is for the time being focusing on the definition of vocabulary, characterisation methods and standardisation of graphene¹.

Prior to focusing on the actual standardisation, the nomenclature has to be determined and agreed upon. The current situation is that different people and companies use their own definitions (for example the distinction between graphene and graphite) to best suit their own purposes. One report (ISO/TS 80004-13) and a few publications are available^{2,3}. A short summary with a recommended vocabulary can be found in Table 1.

The Graphene Flagship is developing a database with key control characteristics (KCCs) of graphene. This database is intended to collect measurements of the samples produced within the Graphene Flagship. They have identified three different user types of this database:

- The first user type are the sample producers who submit product datasheets and get information back about the samples which can be used for production optimisation.
- The characterisation labs, which are analysing these samples and are entering test methods and results into the database.
- Finally, the end users that are using the database for specific tests, target applications and use the data to design future products.

This database can be used for the standardisation work since all (or at least most) relevant properties should be included in the database. The idea is to focus on a limited number of properties and characterisation methods and the standardisation of these, before continuing with other properties. The aim is to develop these standards very fast. One reason is that the standards are urgently needed for enabling the next step in the graphene technology development. The other reason is to set standards that are agreed upon within Europe before a conflict potentially appears with any Chinese standards.

Most of this is work in progress and is for the time being only available for members of the Graphene Flagship. However, one standardisation document (IEC TS 62607-6-4:2016) has been published and is entitled “Nanomanufacturing - Key control characteristics - Part 6-4: Graphene - Surface conductance measurement using resonant cavity”⁴. More information about surface conductance measurements using the resonant cavity method can be found elsewhere⁵.

The Graphene Flagship is working on several standardisation projects together with their partners and these are listed in Table 4. Most of the standardisation work has been done on electrical measurements. Other areas include structural properties, such as the determination of a number of layers of the graphene, the defect density and strain uniformity. There is also work done on graphene-based flexible electrodes and nano-inks.

It is clear that Raman spectroscopy is seen as an important tool, as it is the chosen technique in several of these standardisation projects (for characterising the number of layers, defect density and strain uniformity).

The Graphene Flagship wants consensus on the developed standards and is welcoming new suggestions. According to Katarina Boustedt, the easiest way for Sweden to influence the standardisation work in the Graphene Flagship is through the partner companies. The Swedish partners are ABB, Chalmers University of Technology, Chalmers Industrial Technology, Ericsson, Karolinska Institutet, Linköping University, Nanosc AB and Umeå University whereas Graphensic, Lund University and SP are associated members.

Table 4: Standardisation projects in the Graphene Flagship and IEC/ISO.

Title	Number
Matrix of characterization and measurement methods for Graphene	ISO/TR 19733
Nanomanufacturing - KCC - Part 6-1: Graphene - Measurement of sheet resistance of commercial graphene powders by the Four Probe Method	IEC/TS 62607-6-1
Nanomanufacturing - KCC - Part 6-2: Graphene - Evaluation of the number of layers of graphene	IEC/TS 62607-6-2
Nanomanufacturing - KCC - Part 6-3: Graphene – Characterization of graphene domains	IEC/TS 62607-6-3

Nanomanufacturing - KCC - Part 6-4: Graphene - Non-contact conductance measurement using resonant cavity	IEC/TS 62607-6-4
Nanomanufacturing – KCC – Part 6-5: Graphene – Sheet resistance and contact resistance measurement using the transmission line method	IEC/TS 62607-6-5
Nanomanufacturing – KCC – Part 6-6: Graphene – Uniformity of strain in graphene analyzed by Raman spectroscopy	IEC/TS 62607-6-6
Nanomanufacturing - KCC - Part 6-8: Graphene - Measurement of sheet resistance by the four-point probe method	IEC/TS 62607-6-8
Nanomanufacturing - KCC - Part 6-9: Graphene - Measurement of sheet resistance by the non-contact Eddy current method	IEC/TS 62607-6-9
Nanomanufacturing - KCC - Part 6-10: Graphene - Measurement of sheet resistance by terahertz time-domain spectroscopy	IEC/TS 62607-6-10
Nanomanufacturing - KCC - Part 6-11: Graphene - Defect level of graphene analysed by Raman spectroscopy	IEC/TS 62607-6-11
Nanomanufacturing - KCC - Part 6-13: Determination of content of functional group of graphene materials using Boehm's titration method	IEC 62607-6-13
Nanomanufacturing - KCC - Part 6-14: Graphene-defect level analysis of graphene powder using Raman spectroscopy	IEC 62607-6-14
Nanomanufacturing - Material specifications - Part 3-1: Graphene - Blank detail specification	IEC/TS 62565-3-1
Nanomanufacturing - Material specifications - Part 3-2: Graphene - Detail specification for nano-ink"	IEC/TS 62565-3-2
Nanomanufacturing - Material specifications - Part 3-3: Detail specification for single layer graphene	IEC TS/62565-3-3
Nanomanufacturing - Material specifications - Part 3-4: Detail specification for bi-layer graphene	IEC TS/62565-3-4
Nanomanufacturing – Reliability assessment - Graphene – Bending test for graphene-based flexible electrodes	RA01
Nanomanufacturing – Reliability assessment - Graphene – Basic reliability qualification for graphene layers, temperature and humidity	PWI
Nanotechnologies -- Structural characterization of graphene	ISO/PWI 21356
Nanotechnologies - Vocabulary - Part 13: Graphene and other two dimensional materials	ISO/TS 80004-13

Sample preparation for resistance measurement of edge contacts for graphene and two-dimensional materials	PWI 113-95
Surface chemical analysis — Characterization of nanostructured materials	ISO/TR 4187:2011

4. Future

4.1 Characterisation of Graphene

Most of the industries that are developing graphene-based products are not equipped with highly specialised characterisation tools such as Atomic Force Microscopy (AFM), Raman spectroscopy, Scanning and Transmission Electron Microscopies (SEM and TEM), X-ray Photoelectron Spectroscopy (XPS) et cetera. Therefore, universities and institutes in Sweden are key partners in the innovation chain by providing the necessary infrastructure for characterisation.

Several Swedish actors have expressed an interest in establishing a centre capable of certifying graphene characterisation. We have seen an interest both from actors wanting help from such a centre and from actors that are offering to perform the measurements.

A future formation of such a ‘national graphene characterisation centre’ might be possible through collaboration between important Swedish actors such as RISE, Chalmers University of Technology, Chalmers Industriteknik and Karolinska Institutet. The major responsibility of the graphene characterisation in Sweden can then be undertaken by the ‘National graphene characterisation centre’. Not only graphene research, but the general characterisation of carbon materials could be undertaken by the centre. This kind of centre would also be in a good position to develop Standard Operating Procedures (SOPs) and other standards. These SOPs are important as when testing new materials with established techniques, incorrect and misleading results may frequently be observed, unless the methods are developed using a full understanding of the unique properties of the material being tested.

It is important that the organisations which are offering the characterisation service have experience in working with external projects. Actors that are not able to provide professional services (for example some universities) but have an expertise in graphene characterisation can be valuable advisory members of such a centre and could help establishing proper characterisation procedures. An initial list of potential members is shown in Table 5. World-renown graphene experts in graphene/carbon materials characterisation (and standardisation) may be called to sit as members of the board and yearly meetings can be organised by the centre.

This would also put Sweden in a central position worldwide in graphene characterisation and standardisation along with other famous graphene research centres around the world (Table 6). This would provide Swedish industries with a competitive advantage.

Table 5: Key Actors for Characterisation in Sweden.

Chalmers is an important graphene research centre with has many graphene activities, including the coordination of the Graphene Flagship and the Graphene Centre at Chalmers.

Chalmers Industriteknik (CIT) is coordinating the national strategic innovation programme SIO Grafen, has a wide established network of Swedish industry companies and is involved in graphene innovation projects with many Swedish actors. CIT is coordinating innovation in the Graphene Flagship.

The Myfab network offers processing and characterisation services in the four Swedish clean room facilities at Chalmers, KTH/Acreo, Lund University and at Uppsala University.

Besides the services, it is also possible to access the cleanroom facilities and operate them yourself and use many process and measurement tools.

RISE is a stronghold for graphene characterisation and standardisation due to the easy availability of specialised instruments operated by RISE instrument specialists. AFM, XPS, HRSEM, TEM, Raman spectroscopy etc. All services are easily accessible through currently available infrastructure. RISE not only undertakes the graphene characterisation at materials level, but also performs the characterisation on a product level.

The Institute of Environmental Medicine (IMM) is a department at Karolinska Institutet, a world leader in nanotoxicity research and is a partner in the Graphene Flagship.

Researchers at the Luleå University of Technology have expertise in Raman spectroscopy of GRMs.

Researchers at Umeå University have expertise in graphene oxide and the university is a partner in the Graphene Flagship.

Researchers at Linköping University have expertise in graphene grown by epitaxial growth from silicon carbide and the university is a partner in the Graphene Flagship.

Table 6: Research centres known for graphene characterisation outside Sweden.

Centre	Country
The Carbon Materials Innovation Centre (CMIC) at BASF	Germany
Centre for Multidimensional Carbon Materials	Korea
Graphene Research Centre at NUS	Singapore
National Graphene Institute (NGI), UM	UK

4.2 Potential Future Graphene Standardisation in Sweden

There are already actors working on the standardisation of graphene (i.e. the Graphene Flagship and partners) as described in chapter 0.1. The Graphene Flagship has identified the most basic properties and is working on the standardisation of these together with the properties they consider to be very important. Since the focus in Sweden and Europe may be partly different, this might leave gaps in the standardisation work in application areas not covered by the Graphene Flagship. Sweden should not try to compete with this work, but instead identify areas that are important for Swedish industry, but get less attention internationally. SIO Grafen has identified six areas of strength for graphene:

- Printed electronics
- Coatings and membranes
- Manufacturing (including composites)
- Components and sensors
- Energy
- Life science

SIO Grafen has also identified composites and coatings to be two areas of a large interest from Swedish industry. A Swedish roadmap for these areas is currently being produced.

The Graphene Flagship is mainly focusing on standards for the structural properties of graphene (such as the number of layers and defects) and the electronic properties, leaving

other properties for later. In comparison with SIO Grafen's identified areas, this leaves a gap in for example the areas of coatings and membranes, composites and life science.

We believe that it is advantageous to focus and put in a larger effort towards standardisation in a very limited number of areas rather than making a smaller effort in many areas. The following areas which are not covered by the current work in the Graphene Flagship, have accordingly been identified as important from a Swedish perspective:

- Coatings and membranes

One complexity here could be that it is not only the properties of the graphene flake itself that are important, but rather the macroscopic properties of for example the graphene-enhanced coatings or composites. These macroscopic properties depend also on the concentration of graphene and the dispersion and orientation of the flakes compared to each other and the matrix. Standardised methods for the preparation and the characterisation of samples in for example standardised composites could be developed.

One important aspect of coatings and membranes is the barrier properties. The permeability of a film to molecules and ions are important in many applications such as in printed electronics, packaging for food and drinks, anti-corrosion, composites and many more. There is a great interest in this area in Sweden, but it is not included in the international focus. Thus, it could be important for Sweden to focus on this area. A comparison with general standards for permeability should be made.

- Thermal conductivity

The thermal conductivity is very important in many applications in most of the identified areas, also internationally. Similarly to the discussion about coatings and membranes, it is the thermal conductivity on a macroscopic scale which is important, rather than the microscopic level. However, according to our knowledge, there is not yet any significant standardisation work in this area. Sweden could start working towards this, but in contrast to the situation with the toxicity area, Sweden does not have a unique position to drive the work towards standards developed for thermal conductivity of graphene.

- Toxicity

Toxicity is in an enormous area since a significant amount of tests (cytotoxicity, genotoxicity and ecotoxicity) need to be performed to regard a material to be safe for humans and for the environment. How to safely handle any new material is obviously extremely important to everyone coming into contact with the material, not the least the industries planning to use larger quantities in the production.

The toxicity of graphene is often discussed globally, but there is no consensus. There are even contradictory results in the literature, which highlights that a larger effort is needed in this area.

There are already international projects dealing with the safety of nanomaterials in different ways. For example Prosafe, Nanoreg and Nanoreg2 which address the Safety by design (SbD) concept. These should be further consulted before any potential specific graphene standardisation project is started in this field.

Furthermore, it is most likely that graphene can also soon be successful in for example the packaging market where these questions are critical. In summary, it makes sense to perform standardised toxicity tests to support the development of graphene-based products.

5. Suggestions for future work

The international standardisation work has so far been focused on the properties of the actual graphene material, mainly structural and electronic properties. Initially, we should focus on how samples are prepared for testing, as much as the testing methodology itself. This is a very important area during the characterisation step, especially if users need to choose between graphene from different suppliers, and different characterisation laboratories have been used to perform the tests. Thus, it is recommended that standard operation procedures (SOPs) are developed for each characterisation method. This would strengthen the position of Sweden as a centre for Characterisation excellence. To this end, there are possibilities to establish procedures to produce and investigate dispersions with graphene, for example using standardised oils or plastics, and dispersing/compounding procedures et cetera in order to establish the dispersion and alignment related properties of graphene. The Graphene Flagship, IEC and ISO are working on documents related to inks and flexible electrodes, but this aspect is otherwise largely absent in other areas.

A half-day workshop on standardisation is proposed with invited participants, but the workshop is open to all. The aim is to gather actors that are willing to participate in the standardisation of graphene. The workshop should be held in conjunction with another SIO Grafen event.

Once the topics for standardisation have been fully established, appropriate lobbying to the Graphene Flagship should be performed through a flagship partner (unless there are strong reasons to do otherwise). An increased impact will be achieved if several partners collaborate with support from actors who are not members in the flagship. Actors outside of Sweden with an interest in the same standards should be approached to strengthen the cause.

The current situation in Sweden is that Chalmers, Linköping University, RISE, SIS and Karolinska Institutet seem to have unique infrastructures to support future graphene standardisation work. The nanotechnology committee (SIS/TK 516) of SIS is an important organisation in Sweden that needs to be included in any standardisation effort by Swedish actors. In addition to these organisations listed above, there are groups at universities who are experts in graphene characterisation. These could be important members in future standardisation work, and are included in the list in Table 5.

The next steps for standardisation

A generalised chart of the workflow for developing standardisations within ISO/EN is shown in Figure 3. The current project has been working on Point 1 and 3.

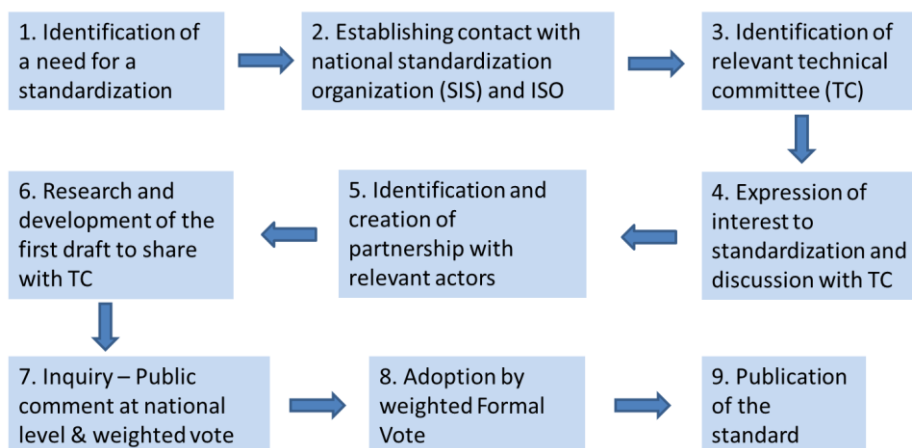


Figure 3: A generalised chart of the workflow for developing standardisations within ISO/EN

Standardisation of sample preparation is expected to be at least as challenging as the standardisation of measurement procedures. The next step is to contact the relevant technical committee to get feedback from relevant actors about the proposed areas for standardisation and identification of relevant partners (step 4 and 5). A feasibility study including the 3 proposed areas (Thermal conductivity, Biological activity and toxicity, Permeability of a film to molecules and ions) is suggested. The feasibility study should include the following tasks:

1. Lead half-day workshop in Standardisation in conjunction with a SIO Grafen event.
2. Investigate which current standards that could provide useful input for standardisation of the three chosen properties of graphene. For example standards on material characterisation or measurement technology could be useful.
3. Literature searches for methods used for the preparation of samples (powders, films, dispersion) for the different tests, including an evaluation of which ones that seem to be the most interesting ones to be used in standardisation.
4. Identify which laboratories have published, and may be available for future method development and Round Robin testing?

If needed, ask SIO Grafen partners for specific advice for any of the steps in Standardisation.

Budget: 400 000 SEK (100 000 SEK for each area, and 100 000 SEK for workshop and project management and meetings etc.)

The next steps for Characterisation

The long term aim could be a “national graphene characterisation centre”. However, initially strengthening of the network identified in this project (Table 5) through networking activities is needed. The following is suggested:

1. Physical meetings for all partners in the network to demonstrate their capabilities, so that all partners are fully aware and can take advantage of each other’s competence.
2. A contact point for queries concerning characterisation, initially working with up-keeping of the network, excel sheet on characterisation, answering questions concerning where characterisation of graphene can be performed, and project leading the networking activities.
3. The Network should evaluate the possibility to become a centre, look for funding possibilities, and describe further steps necessary.

Budget: 300 000 SEK (100 000 SEK for Contact point and 200 000 SEK for workshop meetings etc.)

6. References

1. Paillet, M. & Fabricius, N. Graphene Flagship - Deliverable 15.3. at <<http://graphene-flagship.eu/Lists/Deliverables/D15.3.pdf>>
2. Bianco, A. *et al.* All in the graphene family – A recommended nomenclature for two-dimensional carbon materials. *Carbon N. Y.* **65**, 1–6 (2013).
3. Wick, P. *et al.* Classification framework for graphene-based materials. *Angew. Chemie - Int. Ed.* **53**, 7714–7718 (2014).
4. Standard - resonant cavity. at <<https://webstore.iec.ch/publication/25950>>
5. Obrzut, J., Emiroglu, C., Kirillov, O., Yang, Y. & Elmquist, R. E. Surface conductance of graphene from non-contact resonant cavity. *Measurement* **87**, 146–151 (2016).