White Paper

Realising the commercial potential of graphene and 2D materials in the UK

National Physical Laboratory
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Executive Summary

The UK is world-leading in the research and commercialisation of graphene and 2D materials, but without clearly-defined standards and measurement protocols it risks falling behind other graphene manufacturing centres such as China, Korea and the USA.

Graphene and 2D materials are atomically-thin crystals that may hold paradigm shifts in the design and manufacture of both new and existing products and applications. Graphene, the first of these new 2D materials to be discovered, promises to transform technology. Its remarkable properties – strong, flexible, transparent, and an excellent conductor of both heat and electricity – make it ideal for a wide range of real-world applications in areas such as electronics, biomedicine, energy and composites, to name but a few.

The lack of measurement standards that will allow the accurate, precise and rapid characterisation of these new materials leads to a lack of market confidence, as different commercial materials cannot be compared. It also stifles this technology area as end-users cannot develop application areas without first understanding how changes in their material ultimately affects their product.

The National Graphene Institute (NGI) at the University of Manchester and the National Physical Laboratory (NPL), working with key supply chain companies and other institutes in the UK, have the necessary knowledge base and intellectual capacity to develop international standards for 2D materials. By formalising the procedures for standardisation and characterisation, we aim to set the quality criteria for graphene and other 2D materials, which will also inform other key considerations such as environmental health and safety.

Heavy legislation by the European Commission sets numerous restrictions on the distribution and use of nanomaterials. If 2D materials are to be covered by such broad legislation without the correct standards in place, there is a key risk that the UK will be unable to capitalise on its world-class research as well as to establish a leading commercial position in both the supply of materials and associated equipment.

Following an initial consultation in June 2015 with over 50 key stakeholders across industry, academia and government, it was agreed that there was an urgent need to address this perceived metrology and standardisation gap in commercialisation. This resulting White Paper sets out what is required to establish a ‘one-stop-shop’ for scientific, technical and quality control expertise and services in this area.

A programme to define the technical steps and activities of measurement and standardisation of 2D materials will be needed over the next five years. This programme would be led by NPL and NGI as the two leading organisations with world-class expertise in characterisation and standardisation, creating a pathway for implementation of these materials not only in the UK, but on a global scale.

Both organisations are the leaders in their expertise – NPL in measurement and standardisation, NGI in 2D materials and together are capable of producing not simply new standards for 2D materials, but of influencing their use in applications globally for years to come. The programme will also bring in key partners and suppliers leveraging existing UK capability from the supply chain, including graphene manufacturers, end-users and equipment manufacturers.

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1 INTRODUCTION

Since the isolation and initial characterisation of graphene in 2004 at The University of Manchester, and the subsequent awarding of the Nobel Prize in 2010, there has been flurry of activity to ensure the UK and Europe can capitalise on the unique properties of this material.

This new 2D form of carbon promises to transform technology. Its remarkable properties – strong, flexible, transparent, and an excellent conductor of both heat and electricity – make it ideal for a wide range of real-world applications in areas such as electronics, biomedicine, energy and composites, to name just a few.

Graphene has been classified as an advanced material (one of the UK government's eight great technologies), and identified as an area of research where the UK has a leading position. The UK has the potential to be at the forefront of the commercialisation of this new material, and consequently the UK government has invested significant funding in graphene research[1]- notably for the National Graphene Institute (NGI) and the Graphene Engineering Innovation Centre (GEIC), both part of The University of Manchester.

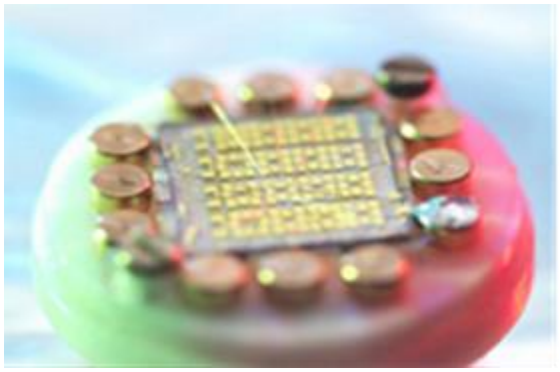

Figure 2. Primary quantum standard for resistance based on graphene first developed at the National Physical Laboratory

Figure 1. National Graphene Institute, University of Manchester

The EU has also recognised the potential of this material and has selected graphene as one of its two Flagship projects, with €1bn of funding for commercialisation-related research over a 10-year period. Countries such as China, South Korea and Singapore have already invested more than $1bn in graphene research and $2.4bn in research funding has been committed globally[2].

This significant, yet uncoordinated, investment has allowed a fledgling graphene industry to emerge globally. However, there is a risk that worldwide investment will not be utilised efficiently because of the current lack of standardisation and quality control needed to give industry confidence in graphene's performance and reliability.

To counter this, the National Physical Laboratory (NPL), the UK's National Measurement Institute, has been supporting the development of graphene through research into the associated characterisation and measurement science (metrology) - a key enabler to the successful and efficient commercialisation of this revolutionary material.

2 PROBLEM STATEMENT

Metrology provides confidence in the characteristics of a product and enables efficient use of investment and resources. Cost benefit analysis show that a total economic cost (public and private) of £1M invested in the National Measurement System would over five years generate= an economic benefit of £3.6M[3].

The EU Technology Roadmap for Graphene, the journey from scientific discovery to applications and products, DOI: 10.1039/C4NR01600A outlines the scientific and technological roadmap over a 10 year period (2013 to 2023) to take hybrid structures from academic research to applications.

The enabling capabilities that need to be addressed to facilitate the transfer of graphene from the laboratory to the factory floor will be Characterisation, Metrology, Standardisation and Quality Control.

Regulation related to safety of nanomaterials can negatively impact on their use in innovative applications. Correct standards and regulations are essential to safeguard public safety and ultimately acceptance of this technology.

Below we describe the essential steps enabling harmonised development of graphene industrial technology.

2.1 CHARACTERISATION

Technically a material is only graphene when it is one atomic layer thick and the solely carbon atoms are arranged in a perfect honeycomb lattice. Only in this situation does the material possess the astounding properties extensively detailed in academic literature. When the number of layers is increased or the lattice is imperfect or doped with adsorbates, the properties change dramatically.

Therefore, characterisation techniques need to be developed which can determine the structure of the material itself and relate this to the final, and quantified, properties that could be used to improve products in everyday life. The required properties very much depend on the final application: for example, different material properties are desirable when producing a printable ink to those desirable when producing a high-speed transistor.

A second issue with characterisation tools is that, at the moment, these are very complex and expensive. Currently this work can only be done by experienced scientists in academic research laboratories or National Measurement Institutes like NPL.

For a real-world and global graphene industry to develop it is essential that simple and fast characterisation techniques are developed which can be operated in-situ within a fabrication setting.

2.2 METROLOGY

Metrology (the science of measurement) provides the confidence that the answer you obtain from your characterisation techniques is correct. This type of research involves comparing different characterisation techniques across institutions and establishing a scientific consensus.

For example, determining the number of layers in graphene can be done using either Raman spectroscopy or surface potential measurements, but do the answers agree? This research is typically done by National Measurement Institutes and is an essential part of a technology infrastructure.

Research undertaken by NPL found that “UK businesses report that without the National Measurement System (NMS)

their total annual sales of new products would decrease by at least £460m and that over £2bn worth of new products are at risk. Furthermore, over 1,400 business users of the NMS believe that the support they receive helps them innovate more quickly, more effectively or reduces risk."[4]

2.3 STANDARDISATION

A recent report from BSI found that standards kick-start UK innovation and enable organisations to unlock their full potential in terms of their products, processes and behaviours[5].

Standardisation builds on metrology and defines technical standards for specific measurement and characterisation techniques. In this respect, standards are very broad and include things such as vocabulary, protocols and industry norms. This needs to be done internationally so that there is compatibility, interoperability and repeatability.

Different standards bodies will be involved in this process (ISO, IEC, IEEE, etc.). A good example of the benefits of standardisation is the fact that an Airbus aircraft wing made in Ireland perfectly fits the fuselage made in Germany when assembled in France.

For graphene, standardisation would mean that material produced by company A to a certain standard will be interchangeable with material produced by company B (if it is made to the same standard).

2.4 QUALITY CONTROL

The final stage of the manufacturing process whereby industry can use the developed standards to ensure the quality of its products. It is important to stress that this is not just about handing standards to industry: the knowledge developed by academia and National Measurement Institutes like NPL needs to be disseminated to those who will use it and this will naturally involve a significant amount of training.

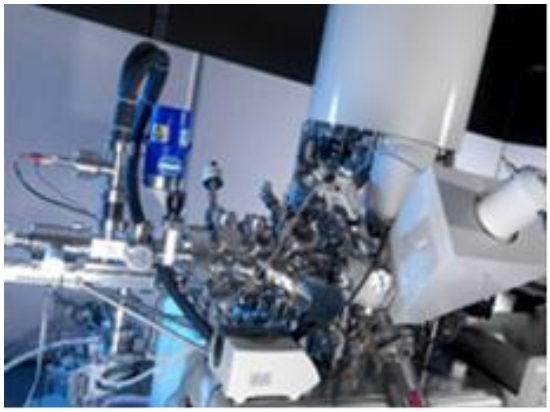

Figure 3 NPL's X-ray photoelectron spectroscopy (XPS) instrument, used to characterise the surface chemistry of graphene

Traditionally, the standardisation process is a linear progression and the complete process could take many years. For a fast moving field like graphene this is far from ideal as companies need standards now to encourage confidence in the products they manufacture.

On the other hand, graphene, with its unique properties, requires radically new measurement and standardisation approaches rather than straightforward adaptation of existing methods, the establishment of new measurement and characterisation facilities, and equipment to enable accelerated development of graphene standards.

NPL and NGI held a workshop in June 2015 to consult with the key stakeholders from industry. The outputs from the workshop confirmed the requirement for significant investment to remove the barriers to commercial exploitation of graphene that arise from issues with metrology, characterisation, standardisation and quality control. The summary from the workshop is attached in Appendix B.

3 PROPOSED SOLUTION

As outlined above, for the UK to leverage government investment in graphene and realise its potential through the creation of new products and industries, measurement infrastructure must be established, bridging the gap between scientific discovery and viable commercial products. Significant progress has been made in the UK at NPL and at The University of Manchester, with the NGI and the future Graphene Engineering Innovation Centre.

NPL has applied existing measurement techniques and created new approaches to better understand graphene, and is regarded as the world leader in the metrology of 2D materials. The NGI has invested in state-of-the-art facilities for graphene characterisation and is driving forward the knowledge to support graphene-related science and the development of new applications. Both organisations are very active in progressing standardisation, both in the UK and internationally.

Figure 4. Inside the NGI

The proposed solution is to establish a joint centre of excellence for graphene characterisation, metrology, standardisation and quality control; building on the knowledge, investment and capability already developed at NPL and The University of Manchester.

The centre would act as the UK's focus for these enabling activities, working with other UK graphene centres providing the necessary support to industry and academia.

The centre intends to coordinate existing NPL and NGI activities while:

- Developing new measurement science and metrology
- Expanding the knowledge and capability for characterisation
- Working nationally and internationally to develop new industry standards
- Creating the protocols and instrumentation techniques for quality control

The centre of excellence will work closely with all the key 2D material organisations in the UK and Europe to utilise their knowledge and capability. This will include establishments such as BSI, the Centre for Process Innovation (CPI) and the Graphene Flagship.

Ultimately, the joint centre will be a one-stop shop offering graphene-related technical services. Alongside the science and technical developments will be an outreach programme, ensuring that the knowledge created reaches those who need it, including training programmes for industry and students.

5 REQUIREMENTS

While both NPL and the NGI have existing programmes of work, the development and delivery of a joined-up solution to enable the UK to lead the establishment of a new industry would require additional support and funding from the UK government. Individual companies and organisations tend to under-invest in metrology because of the high costs involved and the fact that everybody (including competitors) will benefit from it.

Therefore there is good argument for the government to bridge this gap [Economic Impact of the NMS: Evidence Paper July 2010]. The funding for the NGI has provided a world-class facility and infrastructure located within the knowledge base of The University of Manchester, but there is no specific funding to develop the underpinning metrology for graphene because the current remit is to focus on discovery and development of prototype applications.

Whilst the GEIC is focused on higher Technology Readiness Levels and industry partners are readying themselves to exploit this new capability, this will only happen when a suitable standards infrastructure has been developed and is in place.

NPL has made funding available within existing research programmes to address the immediate need for measurement science required for 2D materials but given the wide scope of opportunities and applications in this area, this funding is insufficient to address the requirements for industry to realise the potential of graphene. NPL, NGI, GEIC (and other key institutes and organisations) will require funding for:

The establishment of a graphene 'one-stop-shop' for scientific, technical and quality control expertise and services, related to measurement and standardisation:

- Coordinated development of novel, dedicated characterisation equipment (to be based primarily at NPL and NGI, but also at other centres of excellence)
- Provision of characterisation and metrology services, providing the confidence to commercial UK exploiters that materials meet specification
- Setting up of new measurement facilities to provide state-of-the art quality assurance and control of both graphene material and products. This will significantly aid and speed up product development, innovation and exploitation
- Address measurements related to toxicology studies and consultation with experts.
- Coordination of the UK requirements for graphene standardisation, inputting and influencing into the development of international standards so they best represent the UK's interests
- Promotion, to UK industry and academia, of best practice in the measurement and characterisation of graphene, as well as provision of related policy advice to UK government
- Training for university researchers and industry

6 ANTICIPATED RESULTS

Technologies and products based on graphene have the potential to be disruptive in markets such as:

- Energy – solar cells, energy storage, supercapacitors
- Advanced materials – aerospace components, coatings, composites
- Healthcare – sensors, functional materials
- Information technology – flexible electronics, touch screens, transistors
- Utilities – membranes

The market size for graphene products is estimated to be £400m per year by the year 2024. The UK has world-leading capability and is in prime position to take a meaningful share of this market.

7 CONCLUSION

The potential of graphene and other related two-dimensional materials to be disruptive technologies in many applications across a range of sectors over both the near- and long-term is very real. However, bridging 'the valley of death' between research and its commercialisation is never easy.

The often overlooked areas of characterisation, metrology, standardisation and quality control are key enablers for a technology to go from the research laboratory to large-scale commercial adoption. NPL has shown in many other areas of industry that metrological expertise is viewed as an essential commodity, particularly by countries in Asia.

By developing and leading in this area, the UK will have an essential advantage and, coupled with the already significant scientific knowledge base in the UK through previous and current investment, the UK can be a world leader in the future graphene market.

8 APPENDIX A

8.1 THE NATIONAL PHYSICAL LABORATORY

NPL is the UK's National Measurement Institute, and is a world-leading centre of excellence in developing and applying the most accurate measurement standards, science and technology available. For more than a century NPL has developed and maintained the nation's primary measurement standards. These standards underpin the National Measurement System infrastructure of traceability throughout the UK and the world that ensures accuracy and consistency of measurement.

NPL ensures that cutting-edge measurement science and technology have a positive impact in the real world, and delivers world-leading measurement solutions that are critical to commercial research and development, and support business success across the UK and the globe. www.npl.co.uk

8.2 THE GRAPHENE ENGINEERING INNOVATION CENTRE

The Graphene Engineering Innovation Centre (GEIC) is a £60m facility which is critical to exploit and maintain the UK's world-leading position in graphene and related 2D materials. Based at The University of Manchester the GEIC will allow the acceleration of application research and development in partnership with other research organisations and industry. The GEIC will complement Manchester's existing National Graphene Institute, with the two facilities situated in the heart of the globally leading knowledge base at Manchester, where currently over 230 scientists and engineers work directly on graphene.

The GEIC will fill a critical gap in the ecosystem for graphene and 2D material research, development, and application in the UK. It offers the UK the unique opportunity to establish a leading role in a disruptive technology which spans a number of industrial sectors. Linking into existing national expertise the GEIC will focus on pilot production and characterisation, together with application development in composites, energy, solution formulations and coatings, electronics, and membranes. The research and development within GEIC will focus on all applications and manufacture of materials within the Technology Readiness Levels (TRL) 4-6. The GEIC is set to be completed in 2017.

8.3 THE NATIONAL GRAPHENE INSTITUTE

The £61m National Graphene Institute (NGI) is the world's leading centre of graphene research and commercialisation. Opened in March 2015, it is not only home to graphene scientists from The University of Manchester but also from across the UK, partnering with leading commercial organisations interested in producing the applications of the future. Currently the NGI has more than 40 industrial partners, working on a range of applications across a wide variety of disciplines. With 7,800 square metres of collaborative research facilities and 1,500 square metres of cleanroom space, the NGI is the largest academic space of its kind in the world for dedicated graphene research. The NGI is funded by £38m from the Engineering and Physical Sciences Research Council (EPSRC) and £23m from the European Regional Development Fund. www.graphene@manchester.ac.uk

8.4 THE UNIVERSITY OF MANCHESTER

The University of Manchester, a member of the prestigious Russell Group of British universities, is the largest and most popular university in the UK. It has 20 academic schools and hundreds of specialist research groups undertaking pioneering multi-disciplinary teaching and research of worldwide significance. The University of Manchester is one of the country's major research institutions, rated fifth in the UK in terms of 'research power' (REF 2014), and has had no fewer than 25 Nobel laureates either work or study there. The University had an annual income of £886 million in 2013/14.www.manchester.ac.uk

9 APPENDIX B

9.1 OUTCOMES FROM NPL – NGI GRAPHENE WORKSHOP:

9.1.1 Are characterisation /metrology/standardisation/quality control issues a barrier to commercialisation

- Among the graphene community there is a very clear understanding that an agreed vocabulary is an essential starting point. There is too much confusion about what certain terms mean and how they are used. This needs to be progressed urgently.
- The other unanimous point is the need for urgency. There is a clear perception that a full-blown metrology / characterisation / standardisation process would take far too long and that solutions are needed now if the UK wants to exploit its leading position.
- The drive for standards is coming from two opposite ends. One from material manufacturers and users of base material - what is actually graphene and what does it look like, is it reproducible, are company's claims correct? The other from application of graphene in end products (what is the performance I get from graphene in my product, display, composite, barrier, filter etc.).
- Defining standards too early (without the appropriate metrology and measurement science) could stifle the market or just be useless. However, be the first and you'll set the standard and can potentially block competitors out. I.e. standards can be strategic tool. This has a European dimension with the Flagship being a significant operator. At the same time, the UK may end up not being in prime position if they do not actively pursue this area. Standards need input from range of stakeholders (industry, standards bodies, etc) otherwise won't reflect the needs of the market.
- There is a perception that standards and QC are expensive and a barrier. Therefore there needs to be a cheap and freely available provider for this. It would be extremely important to have an organisation who can police the claims of manufacturers?
- Companies are trying to get round these issues by going it alone, but this will cause problems later, either for the company who has to change its processes, or because the industry will have very different and competing processes that can't be harmonised.

9.1.2 What do we require in terms of Characterisation?

- The key requirement is that we have confidence (reproducibility) through the value chain from production of source material through to performance of end application with relative uncertainties. Issues with this can be that some of the primary properties are not representative for the functioning of the end product. Testing samples of a batch might not be representative. Characterisation needs to be done in-line during production and manufacture. Many properties need to be characterised to make sure final product is consistent – e.g. the lateral size can stay the same but if the number of layers changes product will be very different.

9.1.3 What do we require in terms of Metrology?

- Metrology is an enabler. Helps you take things to market.
- Brings consistency and confidence in measurement techniques.
- There is a need for a materials (properties) database – 'database of producers materials matched to application'

9.1.4 What do we require in terms of Standardisation?

- Recurring comments are: Consistency in how properties are measured and reported. Different application areas will need different standards. Terminology. Urgency.
- The lack of a standards infrastructure will lead to ad hoc and local standards. Then lead to Process failure.
- Hard to standardise as no direct funding.
- For a lot of (existing) applications the standards already exist, for example there are lots of standards for displays and graphene will need to adhere to these standards, however without material standards this can't happen.
- Something like the ITRS is needed which brings the whole value chain together.
- Raw material needs standards – also things like the substrate or the production process affects the quality of the material (complex).

9.1.5 What do we require in terms of Quality Control?

- No large-scale QC techniques exist.
- Needed for different stages of the manufacturing process rather than just one point in the process.
- Range of techniques required which are affordable, easy to use (i.e. don't need PhD scientists).

9.2 BENEFITS OF METROLOGY AND STANDARDISATION INFRASTRUCTURE

- Confidence in materials and properties, judge if claims are correct or if legal issues.
- Ultimately save time and money for business with the infrastructure in place.
- Protect market share of small businesses and in case of legal issues (tackle the snake oil salesman).
- Some comments around measurement infrastructure being too expensive, not everything need a standard. Sometimes benchmarks and relative methods are enough rather than precise and absolute measures.
- The standardisation process tells you something about the variability of the material and so this can actually be used to improve the material.
- Standardisation can give you an idea of the type of material, rather than whose stuff is better, as different material needed for different products, so all the producers may benefit.
- Don't spend too much time working out what is the best standard to develop etc. etc. Just start with something and get momentum going.

9.3 GENERAL COMMENTS FEEDBACK

- Nobody has made any money yet out of graphene.
- It makes sense if someone wants to pay for it. Would be beneficial if this full characterisation (rather than QC) can be done cheaply or freely, by some kind of service provider as producers themselves will not be able to do it.
- Need for meetings like the Manchester meeting to bring the different stakeholders in the value chain together.

[1] c£95m of UK Govt Investment - https://www.gov.uk/government/news/national-graphene-institute-officially-opens

[2] Reports of graphene market analyses: (i) The World Market for Graphene, Future Markets Inc., Report No. 59, Sept 12. (ii) Graphene: The Global Market, Future Markets Inc., Aug 13. (iii) Graphene: Analysis of Technology, Markets and Players, IDTechEx Report Oct 12. Graphene production estimates, lists of companies & news of production scale-ups

[3] Capital Spending Template for the NMS, 2015

[4] Study of users and non-users for the National Measurement System, 2015

[5] http://www.bsigroup.com/LocalFiles/en-GB/standards/BSI-standards-research-report-The-Economic-Contribution-of-Standards-to-the-UK-Economy-UK-EN.pdf