European Commission

THE SEVENTH FRAMEWORK PROGRAMME

The Seventh Framework Programme focuses on Community activities in the field of research, technological development and demonstration (RTD) for the period 2007 to 2013

GRAPHENE FLAGSHIP

The *Graphene-Based Revolutions in ICT And Beyond* (GRAPHENE) project is a CP-CSA supported by the European Commission Seventh Framework Programme under contract 604391

OUTLINE WORK PROGRAMME OF THE GRAPHENE FLAGSHIP CORE PROJECT

Contents

Introduction

This document outlines the scientific Work Programme of the GRAPHENE Flagship core project. It provides the objectives of the relevant Work Packages (1 to 11) and the focus of each task considered in each Work Package. It also provides contact details of GRAPHENE members in charge of these tasks.

This document is primarily provided in the frame of the GRAPHENE Flagship competitive call for consortium extension. It aims at allowing applicants to consider and assess complementarities with already planned activities and potential collaborations and cooperation with members of the GRAPHENE consortium. Such potential collaborations and cooperation should be mentioned and substantiated in part 2.3 of the proposal (see Part 4 of the Guidelines for applicants for further details.)

The GRAPHENE Flagship scientific Work Programme is composed by 11 Work Packages:

1. Materials
2. Health & environment
3. Fundamental science
4. High-frequency electronics
5. Optoelectronics
6. Spintronics
7. Sensors
8. Flexible electronics
9. Energy applications
10. Nanocomposites
11. Production

WP1 - Materials

Objectives

Objective 1: Development of scalable synthesis protocols that enable tuning of graphene electronic, structural and optical properties for different applications. Enlarging the scope of graphene applications by adding new functionalities

Objective 2: Systematic exploration of other 2D materials, as there exist hundreds of layered materials that have not been exfoliated and that could exhibit extremely interesting properties that would be useful in a range of applications. Developing hybrid structures formed by few layer graphene and other 2D materials that can be the basis of new devices.

Work Plan

- Task 1.1 Exfoliation of graphene and other layered compounds
- Task 1.2 Functionalization and doping of graphene and other layered
- Task 1.3: Graphene processing
- Task 1.4: Bottom up synthesis of graphene from molecular precursors
- Task 1.5: Synthesis of graphene on SiC and surface engineering
- Task 1.6: Synthesis of graphene on metals
- Task 1.7: UHV growth on arbitrary substrates
- Task 1.8 Growth of BN, BN/graphene heterostructures graphene and B- N-doped graphene
- Task 1.9 Modelling of growth mechanisms
- Task 1.10: Characterisation

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WP2 - Health & Environment

Objectives

Objective 1: to elucidate the mechanisms of how graphene and 2D crystals interact with cells at cellular and molecular level, with the assessment of the role of bio-corona of these nanomaterials

Objective 2: to address the effects of graphene and 2D crystals on specific tissues such as the immune system, nervous system or placenta, and to determine biomarkers for possible pathogenic risks

Objective 3: to identify any possible hazard of graphene and 2D crystals in relation to their physico-chemical properties with a special focus on the most important exposure routes (i.e. lung, skin)

Objective 4: to understand the processes that control biostability and biodegradation of graphene and 2D crystals, key issues to open the route to nano-interfacing devices

Objective 5: to investigate the potential impact of the various 2D nanoforms on aquatic species (i.e. amphibians), terrestrial organisms and microorganisms

Objective 6: to develop, at the national and international framework, a standardized and validated testing strategy for graphene and 2D crystals, to enable the regulation of these materials

Work Plan

- Task 2.1 A simple, practical, large-scale procedure to generate graphene sheets suitable for toxicity studies using mechanochemical activation by ball-milling will be developed. Commercially available melamine will be used to exfoliate graphite and generate concentrated graphene/water dispersions.

  The methodology paves the way for an efficient processing of these materials. Once prepared, graphene samples can be redispersed in fresh water or different cell culture media forming stable dispersions that will be used for the toxicological and environmental studies. Ball-milling techniques can also be modulated in order to achieve graphene flakes with different sizes and/or different defects, which could be useful for further studies.
- Task 2.2 Once dispersed in an appropriate solvent, graphene can be functionalized by covalent or non-covalent approaches providing multiple sites for the attachment of different classes of molecules (i.e. bioactive molecules and/or radioisotopes for targeting and pharmacokinetics studies). A series of organic reactions that have been previously successfully used to functionalize carbon nanotubes will be applied to the modification graphene and 2D crystals available from other partners of the project, particularly from WP1 and WP10.
- Task 2.3 We will study the degradation of graphene and 2D crystals and its mechanisms by candidate oxidative enzymes in test tube and in vitro (cell cultures). The oxidative and degradative activity will be analysed and correlated with degradation in primary cell cultures of interest, that: a) have been previously described to contain (or not) high levels of the candidate enzymes; and b) are the primary phagocytic cells in specific tissues of interest. Several oxidative enzymes have been identified including myeloperoxydase, cyclo-oxygenase-2 and xanthine oxydase, and

will be used to evaluate their capacity to degrade the different graphene and 2D crystals. We will evaluate the activity in test tube of isolated oxidative enzymes being selected for their strong level of expression into the cells, specific tissues or organs.

- Task 2.4 The toxicological impact of metabolites generated by the oxidative degradation of graphene and 2D crystals in test tubes, will be assessed in vitro upon incubation with several mouse-derived cell types, mainly primary immune-related cells such as macrophages isolated from different organs (i.e. spleen, lungs, brain, liver): General toxicity (cell survival/death) induced by metabolites will be assessed by measuring cell apoptosis/necrosis. Moreover, the proinflammatory potential of those molecules will be studied by analysing relevant cytokine production (IL6, TNFβ, and IL1α).
- Task 2.5 will assess the interaction of graphene and 2D crystals with innate immune cells (e.g. macrophages, neutrophils): mechanism of cellular uptake; activation of inflammasome (or other cytokine secretion); role of bio-corona for uptake and subsequent biological outcomes. We use the following methods: isolation of primary human immune-competent cells; TEM (for uptake); Luminex (for multiplex analysis of cytokine secretion); mass spectrometry-based proteomics to analyse the composition of the plasma protein corona.
- Task 2.6 Assessment of pharmacokinetics, tissue distribution and toxicokinetics. The safety profile of graphene and 2D crystals depends on tissue distribution, pharmacokinetics, blood residence time & body excretion kinetics following their in vivo administration. Tissue targeting motifs are also expected to have an impact of the biodistribution profile of targeted constructs in comparison to non-targeted constructs. The biodistribution profile will be assessed in vivo in normal animal models after administration via different routes of administration (e.g. intravenous, intranasal, oral, topical, and intramuscular). Tissue distribution & pharmacokinetic analysis using dynamic whole-body imaging techniques (NanoSPECT/CT; IVIS) are necessary to allow rapid, accurate, ethical (minimisation of animal numbers) determination of the in vivo transport kinetics & organ distribution of the material. Both radiolabeled and fluorescently labelled graphene material will be used.
- Task2.7 Biotoxicity experiments will be performed with graphene and 2D crystals tested on a variety of cells, in ex vivo experiments. We will focus on postmytotic cell (neurons and cardiomyocytes) isolated and in organ culture of 3D tissues containing fibroblasts, astrocytes, oligodendrocytes and microglia. We will investigate the biological processes influenced by nanomaterials and nanomaterials interaction with biological membranes. We will use electron microscopy (TEM and SEM) atomic force microscopy (AFM), confocal microscopy combined with immunofluorescence studies to investigate cellular and subcellular changes in morphology and cytoskeletal organization, membrane lipid raft distribution, etc., due to the interaction with nanomaterials. Single and multiple electrophysiological measures will be used in the case of neurons and cardiomyocytes to assess the electrophysiological effects of conductive nanomaterials on electrically propagating complex tissues.
- Task 2.8 We will evaluate the biocompatibility of graphene with the survival and proliferation of immortalized cell lines. To this aim, a range of cell lines derived from humans and rodents will be grown on graphene substrates. A range of tests will be

performed to evaluate cell viability and proliferation on graphene, compared to standard culture conditions. We will also evaluate the effects of graphene on the survival, development and physiology of nerve cells and glial components. To this aim, primary neuronal and glial cultures will be grown on graphene substrates. The ability of primary cells to differentiate will be evaluated by confocal and electro-microscopy. In addition, the electrophysiological properties of the neuronal network will be tested by patch-clamp techniques.

- Task 2.9 a) Assessment of the protein corona formed around different functionalised GFNs. Coronas will be determined in reference biofluids (e.g. human serum / plasma, cell culture media with 10% and higher serum content, representing in vitro and in vivo conditions, respectively, lung lavage fluid, etc.) and following uptake / transcytosis through differentin vitro barrier models, such as for instance blood brain barrier endothelial cells and Caco-2 intestinal epithelial barriers, as well cell lines representing different organs and relevant exposure routes. Coronas will be assessed using Differential Centrifugal Sedimentation, 1D-PAGE and mass spectrometry,

  b) Assessment of potential for functionalised graphene and 2D crystals to transport / signal across model in vitro barrier models. Uptake and localization of graphene and 2D crystals in in vitro barrier models will be assessed via a combination of TEM and fluorescence imaging. Transcytosis (if occurring) will also be determined by same methods, and, where feasible, by growing the barriers on transwell systems and assessment of the particle amount in the basolateral chamber over time, as a function of size and surface functionalization of the graphene and 2D crystals. By growing the barrier models on transwell systems, the effect of paracrine signalling from the exposed barriers on cells not directly in contact with the nanomaterials, such as for instance, in the case of the BBB cells, astroctyes or glial cells grown on the bottom of the basolateral chamber, will be assessed via measurement of cytokine expression levels, and other toxicity markers (e.g. oxidative stress, DNA damage etc.)

  c) Assessment of GNF interactions with/impacts on barrier models and representative cell lines assessed by high content screening. The high screening platform allows to screen for a variety of signals activated by the GNF, such as for instance oxidative stress, calcium signalling, organellar damage, lysosomal alterations and effects on mitochondrial membrane potential, as well as cytotoxicity, cell cycle effects and apoptosis in a high throughput way. These will be correlated with size, surface functionalization and localisation of the GNF, exposure dose, duration and timing, in order to interpret the observed outcomes in a mechanistic way. By varying cell types and barrier models in order to represent main routes of exposure and by using GNF dispersions in the biological fluids relevant to that route, as for corona characterisation, a comprehensive description of likely exposure outcomes will be obtained.

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WP3 - Fundamental science of graphene and two-dimensional materials

Objectives

Objective 1: To establish the fundamental limits for functional graphene nanostructures in electronics beyond CMOS. This will be achieved via microscopic characterisation of single- and poly-crystalline graphene (G) and graphene-based nanostructures, studies of kinetic processes, investigation of the influence of defects, disorder, and influence of substrate/environment on graphene electronic properties, and multiscale modelling of graphene-based structures.

Objective 2: To explore the use of exfoliated TMDC in electronics: to characterise microscopically the electronic properties of these 2D crystals, and to investigate transport and vertical tunnelling properties of these materials and atomic layers of h-BN, in view of their later use in hybrid superstructures with graphene.

Work Plan

- Task 3.1. Optical and STM spectroscopic studies of graphene and TMDCs, including characterization of edges and defects, in single crystalline and polycrystalline graphene and TMDCs on different substrates.
- Task 3.2. Electronic and heat transport studies of graphene-based devices (including suspended layers, nanoribbons, quantum dots, and their circuits), investigation of their dynamical properties, and their interaction with substrates and environment (such as BN layers), and two-layer structures.
- Task 3.3. Theoretical mesoscale modelling of graphene-based structures, functionalised graphene, defects in graphene, and the influence of the environment. This will include density functional theory and quantum Monte Carlo modelling of materials and defects, and microscopic scattering theory and quantum transport modelling of nanostructures.
- Task 3.4. Advancement of nanofabrication technology for graphene-based devices, including development of vertically aligned multi-layered structures and devices, cleaning methods, hybrid graphene-TMDC devices, and durability and stability of graphene-based structures.
- Task 3.5. Implementation of graphene in metrology, for the development of fundamental resistance and current standards. Exploration of the operational bounds of graphene-based standards of electrical resistance based on the half-integer quantum Hall effect and of electrical current based on the single-electron pumps, identification of the underlying physical mechanisms limiting their performance, optimisation of the fabrication, cryogenic and measurement technologies.
- Task 3.6. Experimental characterisation and theoretical modelling of lateral transport characteristics of two-dimensional atomic crystals of TMDCs and vertical (tunnelling) transport properties of graphene-TMDC multilayers.

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WP4 - High frequency electronics

Objectives

This work package is devoted towards the long term perspective of establishing graphene based high-frequency electronics technologies, which are capable of significantly outperforming state-of-the-art technologies. Therefore the key objectives of this WP are:

Objective 1: Optimise process technologies critical towards meeting the requirements of specific applications, including work on contact resistance, gate stack, passivation, band gap engineering and integration of different 2D materials.

Objective 2: Identify further key technological bottlenecks for realising graphene based high frequency integrated circuits and develop solutions for solving them.

Objective 3: Develop novel concepts for realising graphene-based high-frequency devices including ballistic devices.

Objective 4: Design and realisation of graphene-based integrated circuits capable of fully utilising the unique electronic properties of graphene.

Objective 5: Assess the developed technologies towards the current state-of-the-art and define target parameters for ensuring long-term relevance.

Objective 6: Define means of standardisation, both for materials, processes and devices.

Objective 7: Realise different kinds of integrated circuits in order to verify the performance of the developed devices and to benchmark these against established IC technologies.

Objective 8: Based on this work, establish a clear and detailed roadmap for developing graphene as the next-generation of high-performance electronics.

Workplan

- Task 4.1: Development and optimization of process technology, realisation of device components
- Task 4.2: Circuit design, realisation and testing
- Task 4.3: Ballistic devices B
- Task 4.4: Technology assessment, definition of specification and standardisation

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WP 5 - Optoelectronics

Objectives

The WP5 goal is to establish a new field of graphene photonics and optoelectronics, sustained by the convergence and co-integration of graphene-based electronic and photonic components such as lasers, switches, optical waveguides, optical frequency converters, amplifiers, cavities, modulators, photodetectors, nano-photonics components, metamaterials and solar cells. These objectives are supported by the development of different production methods both for graphene and related 2D layered materials, and comprise:

Objectives

Objective 1: Photodetectors for broadband spectral range (covering UV, visible, near-IR and THz frequencies) will be developed for (i) ultra-fast devices for optical communications and (ii) ultra-low-intensity or single-photon detection. Broadband and ultra-fast graphene photodetectors will be integrated in opto-electronic networks, taking into account the influences of processing and “real world” environments. These devices will exploit the ultra-high carrier mobility and unique wide spectral range of graphene.

Objective 2: Fabrication and testing of mid-infrared and plasma-wave based THz photodetectors and integration of these elements into detector arrays of large areas of order cm^2. Improved performance in terms of responsivity and bandwidth, and operation limits well above 1 THz.

Objective 3: Development of graphene-based ultra-broadband tuneable lasers, such as mid-infrared fibre lasers, high-power solid-state lasers, wide-band semiconductor lasers, high-repetition rate waveguide lasers, etc., and full testing of these devices.

Objective 4: Implementation of plasmon-based nanoscale optical routing and switching networks for optoelectronic interfaces and interconnects, for ultraviolet, visible, near-infrared and mid-infrared frequencies. These components will exploit graphene as an active plasmonic material with ultra-strong and gate-tuneable optical field localisation.

Objective 5: Development of integrated opto-electronic networks consisting of high-speed electro-optical modulators and photodetectors coupled to waveguides and resonators.

Objective 6: Development of fast-switchable, frequency tuneable, ultrathin, graphene-based THz optical polarizers and magneto-optical isolators for novel THz applications

Objective 7: Design, fabrication and testing of a whole new generation of gate-tuneable plasmonic metamaterials with optical properties electrically controlled by graphene.

Objective 8: Development of ultrafast and small active optical elements for light harvesting and sensing, by combining graphene with metallic plasmonic metamaterials.

Objective 9: Development of semiconducting 2D materials for ultra sensitive photo-detection, and active gain materials for light amplification.

Objective 10: Development of multi-layered 2D hybrid structures for photo-voltaics and photo-detection

Objective 11: Development of graphene as an electrode for OLEDs, optimised for band alignment with semiconducting layers is achieved.

Objective 12: Development of LEDs with integrated plasmonic structures

Objective 13: Identification of the graphene technology within WP5 would be most effective for the realisation of a proof-of-concept system driven by industrial needs. In addition a portfolio of ideas aiming to capitalise on specific figures of merit shall be realised.

Objective 14: Benchmarking of performance of all the proposed and realised optoelectronic devices with conventional technologies.

Furthermore, extensive theoretical support will be provided within WP5 for the understanding of the optical behaviour and optoelectronic response of graphene and 2D materials, with emphasis on charge-carrier generation, in order to develop predictive tools that can assist us in the design of optoelectronic devices. The following levels of theory will be developed:

- Microscopic description of graphene plasmonics and carrier interactions, starting from first principles;
- Macroscopic electromagnetic theory,
- Integral description of the electro-optical environment of actual devices incorporating graphene and other optical and electronic elements such as gates and structured substrates.

Work Plan

- Task 5.1. Development of graphene photodetectors for visible and IR
- Task 5.2. Development of graphene-based ultra-broadband tuneable lasers. Integration into mode-locked lasers (fibre, semiconductor, waveguide and solid state lasers) to achieve ultra-broadband tuneability in the telecommunications and mid-IR range
- Task 5.3. Implementation of graphene-based nanoscale optical routing and switching devices and networks.
- Task 5.4. Development of graphene-based tuneable metamaterials including plasmonic nanoresonators based on metallic/graphene composite materials tuneable by low voltages. The combination of graphene with plasmonic metamaterials will be exploited for ultrafast and small active optical elements for light harvesting and sensing, in connection to Tasks 5.1 and 5.5.
- Task 5.5. Long-wavelength photodetectors
- Task 5.6. Integration of selected components into a proof-of-concept system implementation (POCSI)

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WP6 - Spintronics

WP6 aims at establishing the ultimate potential of graphene for spintronics, targeting efficient room temperature spin injection and detection but also spin gating and spin manipulation in graphene spintronic devices. Spin transport mechanisms in graphene devices will be explored using different materials (provided by WP1), and by comparing the results with realistic theoretical simulations, we will work towards the demonstration of novel types of functional graphene spintronic devices. The research objectives are supported by a strong collaboration between experimental and theoretical efforts, and comprise:

- Optimisation of materials and devices characteristics for graphene spintronics
- Investigation of induced magnetism in graphene and its interaction with spin transport
- Clarification of spin transport and spin relaxation in low-dimensional graphene devices
- Design of spin sensors and spin gating graphene devices
- Fabrication of practical (room-temperature) graphene spintronic devices

This includes the clarification of the physical mechanisms dictating the spin relaxation times and spin relaxation lengths in (high quality) graphene devices, together with the development of spintronic devices with tuneable magnetism or spin gating functionality, by evaluating the (spin)-transport response of gated devices to induced magnetic moments induced by hydrogenation, defects or other chemical or structural treatments (nanomesh design). Other devices where graphene is in proximity with a ferromagnetic insulator or a magnetic insulator will be studied. The effect of (quantum) confinement on spin relaxation and spin dephasing will be scrutinised, together with the modification of Dyakonov Perel (or Elliot-Yafet) mechanisms for spin relaxation when reducing the lateral and longitudinal sizes of graphene samples (graphene nanoribbons and graphene quantum dots). The structural characterisation and the role of graphene edges on spin scattering and relaxation will be clarified. Finally, the potential of graphene where the injected spin-polarised charges flow in close proximity (and interact) with other extrinsic spins (in localised or more extended charged states, located below or on top of the graphene) will be analysed in details.

Finally, all practical technological aspects (device optimisation, upscalability, and reproducibility, room temperature operability), as well as the introduction of optimised material and device geometries (two-terminal) will be explored, working towards prototype applicable graphene spintronics devices. Attention will be given to the realisation of large values of the magnetoresistance in two-terminal devices.

By tackling these issues, one anticipates on the development of external ways to control (gate) the long distance propagation of spin currents, achieving operational reliability at room temperature and architectural compatibility with silicon technologies. The achievement of the following tasks will pave the way towards all-graphene based integrated MRAM and Spin information processing based circuits.

Work Plan

- Task 6.1: Optimising materials and devices for graphene spintronics
- Task 6.2: Magnetism in graphene and its interaction with spin transport
- Task 6.3: Spin transport and spin relaxation in low-dimensional graphene devices
- Task 6.4: Spin sensors and spin gating graphene devices
- Task 6.5: Towards practical graphene spintronic devices

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WP7 - Sensors

Objectives

To develop sensing devices based on graphene membranes. In particular, sensitive read-out for a variety of sensor applications will be developed up to the proof-of-principle stage, supported by modelling activities.

Work Plan

- Task 7.1: Sample fabrication and basic testing
- Task 7.2: Demonstration of sensor working principles
- Task 7.3: Technology and feasibility assessment supported by modelling

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WP8 - Flexible Electronics

Objectives

WP8 investigates the application of graphene to key enabling technologies required for the realisation of flexible electronic devices and systems. WP8 will have a technology integrator role between other WPs and thereby this work will have connections to the major part of initiative.

Work Plan

- Task 8.1 Materials and fabrication processes

- Task 8.2 Flexible energy solutions
- Task 8.3 Flexible RF electronics and wireless connectivity solutions
- Task 8.4 Flexible sensors
- Task 8.5 Flexible passive electronics
- Task 8.6 System-level platform for flexible electronics

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WP9 - Energy applications

Objectives

Taking advantage of availability of various forms of graphene within the consortium, the objective of WP9 is to assess their interest in various daily life energy applications. By focusing on specific functions involved in applications including photovoltaics, energy storage, fuel cells and hydrogen storage, WP9 intends to "connect" the fundamental and technological graphene expertise to the designers and developers of energy conversion and storage devices. This investigation into energy applications will enable to:

- Define the applicative graphene specifications on the basis of experimental/modelling approaches.
- Achieve proofs of concept of graphene-related materials in different energy conversion and storage devices.
- Explore novel research routes for future graphene-based material which could better match the energy application needs

Work Plan

- Task 9.1. Photovoltaics

- Task 9.2. Energy storage
- Task 9.3. Fuel Cells
- Task 9.4. H2 storage

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WP10 - Nanocomposites

Objectives

Objective 1: Transfer the ideal properties of single graphene sheets from the atomic scale to the meso-macroscopic level (continuous layers or bulk materials).

Objective 2: Understand the failure mechanisms of graphene composites by studying their behaviour at the macro/micro/nanoscale, while the material undergoes electrical and/or mechanical stress.

Work Plan

Task 10.1 Production of graphite nano-platelets as low cost filler and standardized starting material

Task 10.2 Production of graphene sheets by exfoliation in solution with organic molecules

Task 10.3 Production of large, soluble, monoatomic sheets of graphene oxide for further processing and functionalisation.

Task 10.4 Production of graphene sheets and graphene nano-ribbons by bottom-up assembly of nano-graphenes

Task 10.5 Production of Aerographite

Task 10.6 Production of other 2D materials

Task 10.7 Production of structural composites with commercial polymers

Task 10.8 Production of functional composites with organic semiconductors

Task 10.9 Production of 2D composites

Task 10.10 Composites for high-performance structural applications

Task 10.11 Composites as smart gas barriers

Task 10.12 Composites for heat transport and management applications

Task 10.13 Composites for energy storage applications

Task 10.14 Composites for flexible electronics

Task 10.15 Spectroscopic characterisation (Raman, UPS, etc.)

Task 10.16 Microscopic characterization (Atomic Force, SEM, TEM, SAED, Etc.)

Task 10.17 Macroscopic electric and mechanical characterization (Current-Voltage, Time Of Flight, Etc.)

Task 10.18 Modelling of graphene interactions and graphene failure in composites

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WP 11 - Production

Objectives

Objective 1: Implementation of growth technology and commercial exploitation of bulk graphene/graphene films at the equipment and material level. To evaluate production scalability, reproducibility and cost of production/ownership.

Objective 2: Supply graphene to various WP's for characterisation and integration into devices

Objective 3: Being established companies in the graphene industry, the partners here have access to a large network of international customers for bringing in external requirements and providing routes of exploitation to the projects results.

Objective 4: Align graphene production requirements with projects in "NMP.2013.4.0-1 Graphene production technologies"

Work Plan

Task 11.1: Wafer scale graphene equipment

Task 11.2: Graphene Films

Task 11.3: Bulk Graphene

Task 11.4: Alignment with NMP.2013.4.0-1

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