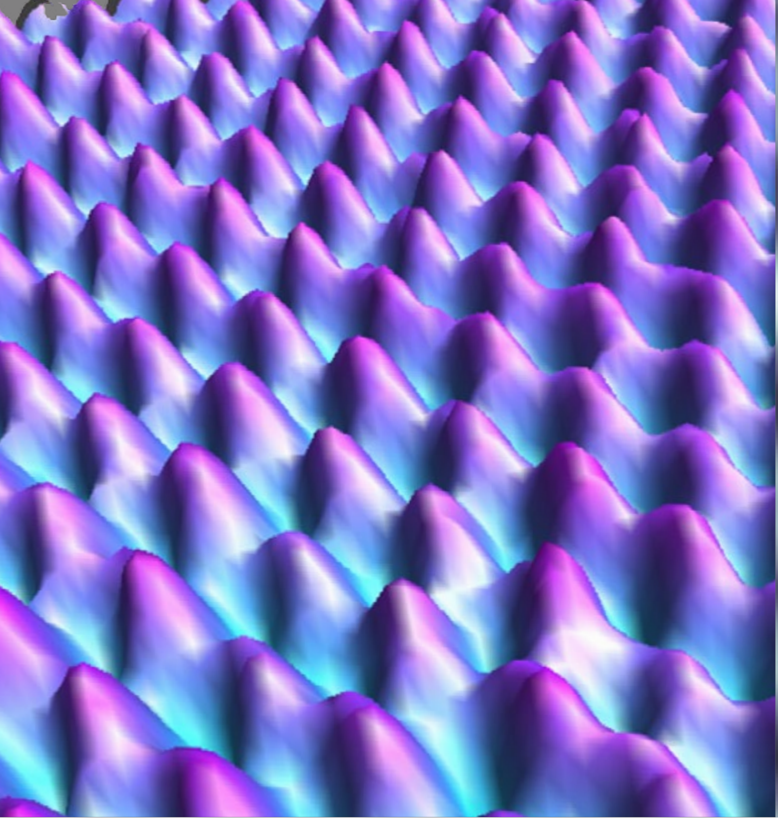
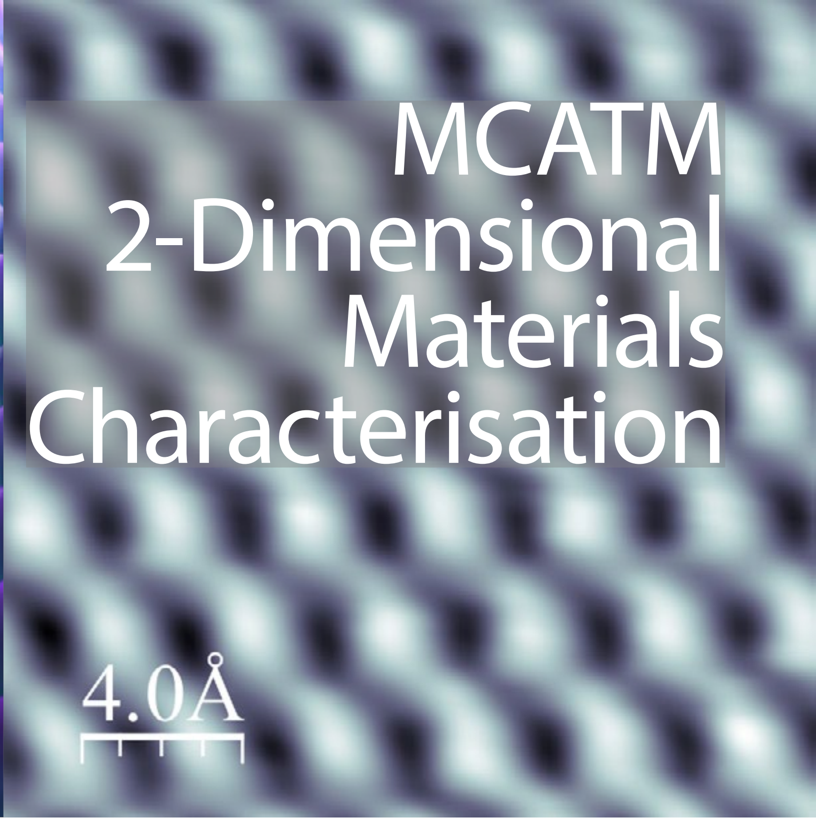


MCATM 2-Dimensional Materials Characterisation



30 MAY 2016
S4 THEATRE
MONASH CLAYTON

WORKSHOP

MICROSCOPY

Atomic Force Microscopy

Electron Microscopy

Scanning Probe Microscopy

Nanofabrication

SPECTROSCOPY

X-Ray Photoelectron Spectroscopy

Angle-resolved photoemission spectroscopy

Raman & Infra-Red Spectroscopy

Electrochemistry

VICTORIAN CAPABILITIES

Monash Centre for Atomically Thin Materials

Monash Centre for Electron Microscopy

Melbourne University Materials Characterisation & Fabrication Platform

Australian Synchrotron
ANFF-VIC MCN
CSIRO Manufacturing

About The Workshop

The Monash Centre for Atomically Thin Materials (MCATM) presents a workshop on two-dimensional materials characterisation. Experts from Monash University, The University of Melbourne, CSIRO, the MCN and the Australian synchrotron will cover a range of techniques regularly used in 2D materials research. Presentations and discussions will focus on the technique, the theory behind it and its implementation in two-dimensional materials research. We look forward to welcoming you at our inaugural **MCATM 2D Materials Workshop**.

Organising Committee: Dr. Mark Edmonds | Dr. Tich-Lam Nguyen | Prof. Michael Fuhrer

Register: 2dmaterialscharacterisation.eventbrite.com.au | More Info: monash.edu/mcatm | Contact us: mcatm@monash.edu

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PROGRAM

9:00 REGISTRATION — CHAIR: DR. TICH-LAM NGUYEN

9:30 **Welcome & Introduction to MCATM**
Prof. Michael Fuhrer, School of Physics & Astronomy and MCATM, Monash University

9:40 **Atomic Force Microscopy: Routine applications to complex methods for materials characterisation**
Prof. Ray Dagastine, Department of Chemical and Biomolecular Engineering
The University of Melbourne

10:10 **2D materials characterisation capabilities at The University of Melbourne**
Dr. Lauren Hyde, Materials Characterisation and Fabrication Platform,
The University of Melbourne

10:40 MORNING TEA & POSTER SESSION — CHAIR: DR. MARK EDMONDS

11:10 **Atomic-resolution imaging of atomically thin materials via transmission electron microscopy**
Dr. Scott Findlay, School of Physics & Astronomy, Monash University
A/Prof. Matthew Weyland, Department of Materials Science & Engineering and MCEM, Monash University

11:50 **Transmission electron microscopy of curved graphitic surfaces**
A/Prof. Laure Bourgeois, Department of Materials Science & Engineering and MCEM, Monash University

12:10 **A landmark national research facility shining light across a diverse scientific landscape**
Prof. Michael James, Australian Synchrotron

12:40 LUNCH & POSTER SESSION — CHAIR: PROF. MICHAEL FUHRER

13:40 **A Brief Status Report on Scanning Tunneling Microscopy**
Dr. Agustin Schiffrin, School of Physics & Astronomy and MCATM, Monash University
Dr. Bent Weber, School of Physics & Astronomy and MCATM, Monash University

14:10 **Raman and infrared spectroscopy and imaging at the micro and nano level**
Prof. Don McNaughton, School of Chemistry, Monash University

14:40 **Graphene characterisation capabilities at CSIRO Manufacturing**
Dr. Malisja de Vries, CSIRO

15:10 AFTERNOON TEA & POSTER SESSION — CHAIR: DR. BENT WEBER

15:40 **Photoelectron Spectroscopy: Principles and Applications in 2D Materials Research**
Dr. Anton Tadich, The Australian Synchrotron

16:10 **Electrochemistry methods in 2D materials research**
Dr. Adam Best, CSIRO
Dr. Marzi Barghamadi, CSIRO

16:40 **2D Materials Characterisation and Fabrication at ANFF-Vic**
Dr. Lachlan Hyde, Melbourne Centre for Nanofabrication

17:10 CLOSING REMARKS & POSTER PRIZE PRESENTATION
Dr. Mark Edmonds, School of Physics & Astronomy and MCATM, Monash University

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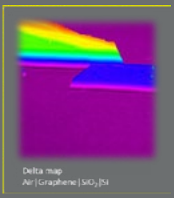
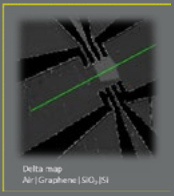
Characterization of graphene through imaging spectroscopic ellipsometry

Introduction

Graphene consists of a sp²-hybridized atomic carbon crystal. Since it was first reported by Novoselov et al. (2004) it has been of increasing interest, because of unique physical properties and promising applications. First Graphene samples were fabricated by micro mechanical cleavage of graphite. Currently, technical processes have been established to produce graphene on large areas.



To achieve a better control of thickness and properties , a characterization in the micrometer scale is required. The characterization should include the thickness and the number of layers, respectively. Also the optical properties are essential.



Imaging Ellipsometry is a fast and easy way to characterize localized exfoliated graphene

Imaging ellipsometry

Ellipsometry is a sensitive optical method, which has been used for about a hundred years to derive information about surfaces. It makes use of the fact that the polarization state of light may change when the light beam is reflected from a surface. If the surface is covered by a thin film (or a stack of films), the entire optical system of film & substrate influences the change in polarization. It is therefore possible to deduce information about the film properties, especially the film thickness.

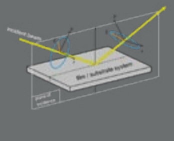


Figure 1. State of polarization of a light beam reflected at a surface.

The name ”ellipsometry” implies that it has to do something with an ”ellipse”: The elliptical state of polarization, where the electrical field vector travels along an ellipse when observed at a fixed point in space, is the most general state of polarization The basic components of an ellipsometer are: a light source, some optical components to modify the polarization and a detector. By using imaging technology, one can extend the classical ellipsometer to a new form of visualization tool or a microscope with extreme sensitivity to thin films.

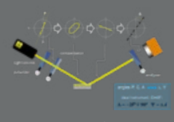


Figure 2. Principle of nulling ellipsometry.

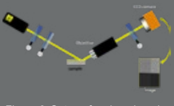


Figure 3. Setup of an imaging ellipsometer.

Contact

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Maps with the highest lateral resolution

Delta and Psi maps are based on a number of images taken at changing orientations of the polarizer and analyzer (Fig. 4 a). In general, the ellipsometric angles were obtained by analyzing the minimum in grayscale distribution for each pixel (Fig. 4b).

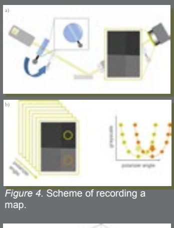

Figure 4. Scheme of recording a map.

Albrektsen et al. (2012) characterized few layer graphene by different imaging methods and showed clearly the capability of imaging ellipsometry to distinguish between single, double and few layer graphene.

Matković et al. calculated based on Delta maps at different wavelength the thickness of the graphene layer and a water interlayer independent from each other.

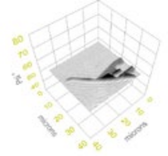

Figure 5. Psi map of a graphene flake.

Ellipsometric enhanced contrast

Based on the nulling concept, the optical setup of an imaging ellipsometers offers the option to set the optical components in a way to enhance the contrast of the micrograph. Matković published micrographs of graphene flakes on different SiO$_2$|Si substrates.

Wurstbauer et al. (2010) published ellipsometric contrast micrographs of few-layer graphene on a GaAs substrate. The contrast was optimized for the graphene layer such that the adhesive tape residues vanish, while in a second image the contrast of the immediate vicinity is enhanced.

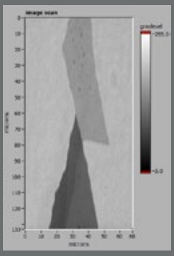

Figure 6. Ellipsometric contrast micrograph of graphene flakes.

New: Imaging NIR-ellipsometry

Based on new developments, spectra with higher spectral resolution are available in the NIR up to 1700 nm. For wavelength above 1000 nm an InGaAs cooled FPA detector was included.

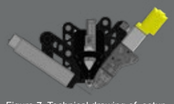

Figure 7. Technical drawing of setup with NIR camera.

Delta- and Psi maps of a graphene flake are recorded and show significant different behavior than the substrate. The technique is promising to obtain information about Drude adsorption with a lateral resolution of few micro meters. It will improve the understanding of the dielectrical properties of graphene and of the graphene substrate interactions.

Figure 8. Delta– and Psi-maps of a graphene flake at selected wavelength in the NIR.

Spectra at the smallest regions of interest (ROI)

One unique feature of imaging ellipsometry is the option of regions of interest. Unlike conventional ellipsometers that illuminate a single microspot and collect data for that one spot at a time, Imaging ellipsometers imaging the illuminated area onto a 2D array detector for a million or more simultaneous measurements. This facilitates ROI by enabling pixel selection.

The first wavelength spectra measured by the use of a ROI on single layers graphene were published by Wurstbauer et al. Matković et al. identified the Fano resonace based on spectra, measured with the ROI concept.

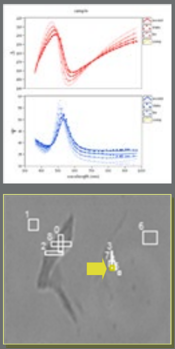

Figure 9. Wavelength spectra of Delta and Psi at different angle of incident.

The new nanofilm_ep4

The nanofilm_ep4 is based on a new frame for more free space and rigidity. It has an integrated Z-lift with higher accuracy; a new EP4 Align Sensor including the detection of the z-position and a new Instrument Alignment (instead of sample alignment) that enables the alignment on large samples like troughs, production line, etc.. Other improvements involve the new modular software concept including the optical modeling, the instrument control, and the data studio.

Figure 10. Nanofilm_ep4

The new “Ultra_Objective”-Imaging optics offers fully focused field-of-view and fast mapping in real time. Imaging spectroscopic ellipsometry is improved with a continuously tunable, high throughput monochromator and a new light source (laser-driven Xe plasma, ca. 4 times more light for better SNR) for improved precision.

Figure 11. Optional with ultraobjective

The setup offers new possibilities for the technical integration of other techniques like AFM, QCM, Terahertz spectroscopy or even customized solutions.

,

Figure 12. Technical integration of an AFM

Literature

Matković et al. (2012) Spectroscopic imaging ellipsometry and Fano resonance modeling of graphene. J. Appl. Phys. 112, 123523

Albrektsen et al. (2012) High resolution imaging of few-layer graphene. J. Appl. Phys. 111, 064305 ellipsometry:

Wurstbauer et al. (2010) Imaging ellipsometry of graphene. Appl. Phys. Lett. 97, 231901

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SPEAKER PROFILES

PROFESSOR RAY DAGASTINE

Prof. Ray Dagastine is the initiative chair of the Materials Research Hallmark Initiative at The University of Melbourne and is the academic leader of a the Materials Characterisation and Fabrication Platform.

His research interests are in the area of particulate and droplet interfacial phenomena, emulsion stability and deformable surfaces. He is a leading researcher in developing experimental methods and theoretical analytical tools to study interaction forces between deformable liquid-liquid interfaces using both optical techniques and atomic force microscopy.

He also has research interests in structure in complex fluids, non-equilibrium surface forces, and protein interactions and nanomechanical properties of capsules and micro-particles.

DR. LAUREN HYDE

Dr. Lauren Hyde has a background in nanotechnology and a PhD in biomaterials science. Previously, she has managed the Vibrational Spectroscopy facility at the University of Sydney and was a beamline scientist on the infrared microspectroscopy beamline at the Australian Synchrotron.

Lauren has joined the MCFP team as the technical support coordinator and manager of the MCFP. She has experience with many surface topography and chemistry preparation methods as well as surface analysis experience including infrared and Raman microscopy, atomic force microscopy (AFM) and scanning electron microscopy (SEM).

SPEAKER PROFILES

ASSOCIATE PROFESSOR LAURE BOURGEOIS

A/Prof. Laure Bourgeois' main research interest lies in the determination of atomic-scale structure in inorganic materials, with emphasis on nanoscale crystals, crystalline defects and aperiodic phases (incommensurate crystalline phases, quasicrystals, but also curved graphitic structures). In addition to the fact that structure is a fundamental starting point to the science and engineering of materials and minerals, understanding the mechanisms of solid-state nucleation and growth is her main motivation for determining atomistic structure.

To achieve this, Laure uses a variety of techniques available in the great versatile tool that is transmission electron microscopy (TEM), in particular atomic resolution imaging techniques such as high-angle annular dark field (HAADF) scanning (S)TEM and high-resolution TEM in conventional and aberration-corrected transmission electron microscopes.

DR. SCOTT FINDLAY

Dr. Scott Findlay completed his PhD in Physics at the University of Melbourne in 2005. He was a Japan Society for the Promotion of Science Postdoctoral Fellow at the Institute for Engineering Innovation at the University of Tokyo from 2008 to 2010, and a QEII Fellow in the School of Physics and Astronomy at Monash University from 2011 to 2015. His research centres upon using theory and numerical modelling to develop new approaches to imaging and quantitative analysis in scanning transmission electron microscopy.

ASSOCIATE PROFESSOR MATTHEW WEYLAND

A/Prof. Matthew Weyland manages Australia's first aberration corrected TEM, the FEI Titan3, and drives research using this instrument. Matthew's research centres on the development of electron microscopy, and its application to materials problems. He has published in nearly all areas of materials science, from polymers to metals, magnetic materials to catalysts, and electronic materials to corrosion. In recent years his publications have mainly been on the atomic structure of light alloys (Al, Mg etc), semiconductor heterostructures and functional nanoparticles.

SPEAKER PROFILES

PROFESSOR MICHAEL JAMES

Prof. Michael James is Head of Science at the Australian Synchrotron. He was previously a senior principal research scientist at ANSTO, team leader of the Chemical Deuteration Laboratory and instrument scientist for the PLATYPUS Time-of-Flight neutron reflectometer, which he designed and built at ANSTO's OPAL research reactor.

Michael currently leads a team of more than 60 staff that operates the Beamlines, IT & Scientific Computing facilities and User Office at the Australian Synchrotron. His research interests include numerous areas of neutron and X-ray scattering; predominantly relating to the study atomic and molecular structures of technologically advanced materials. He has published more than 140 scientific peer-reviewed publications.

DR. AGUSTIN SCHIFFRIN

Dr. Agustin Schiffrin explores the atomic-scale structural, electronic and charge dynamics properties of solid interfaces. He uses supramolecular chemistry on surfaces for the bottom-up design of low-dimensional organic and metal-organic nanostructures with tailored electronic and optoelectronic functionalities.

He is an expert in low-temperature scanning probe microscopy and spectroscopy, x-ray absorption and photoelectron spectroscopies, and time-resolved spectroscopies.

DR. BENT WEBER

Dr. Bent Weber's research interests involve studying and controlling matter at the atomic-scale and exploring novel avenues for the atomic-scale nanostructuring of to the next generation electronic materials graphene and the layered transition metal dichalcogenides, using scanning probe and conventional nanofabrication techniques.

He has >10 years of experience investigating unique capabilities of STM to engineer a wide range of functional atomic-scale electronic devices for their application in silicon donor-based quantum information processing.

SPEAKER PROFILES

PROFESSOR DONALD MCNAUGHTON

Don McNaughton is professor of molecular sciences and director of the centre of Biospectroscopy in the School of Chemistry at Monash.

His current major program of research involves developing Raman and infrared micro and nano-spectroscopy and imaging techniques to understand and follow biological processes and disease at a molecular level in cells and tissue.

He also runs a program aimed at generating and studying, by high resolution spectroscopy techniques, species of interest in understanding interstellar and atmospheric chemistry.

DR. MALISJA DE VRIES

Dr. Malisja de Vries manages the Microscopy group in the Manufacturing Division for the past 3.5 years which also has staff at Waurn Ponds on the Deakin University Campus. The facility specialises in cryogenic preparation in SEM and TEM, as well as 3D imaging for surface metrology using SEM and laser confocal microscopy Analyse a diverse range of samples from materials (hard and soft) to biological.

She was in the Minerals Division prior to that for 7 years: firstly synthesising photoactive nanoparticles for water splitting and then developing technology to utilise biomass in anodes for use in aluminium reduction cells, via densification during pyrolysis. Anodes require graphitised, electrically conductive and highly dense carbon.

She started at CSIRO in 1999 on a PhD scholarship working on the kinetics of gas-solid reaction under pressure of ilmenite reduction in hydrogen. After which she has spent 10 years in Comalco Aluminium Research Centre.

SPEAKER PROFILES

DR. ADAM BEST

Dr. Adam Best received his Ph.D. from Monash University, Australia in 2002 before being awarded a Senior Post-Doctoral Fellowship at Delft University of Technology, The Netherlands, to work on high temperature batteries (2002 - 03).

In 2004 he returned to Australia to join the Commonwealth Scientific and Industrial Research Organisation (CSIRO), Division of Energy Technology, as a post-doctoral fellow to work on ionic liquid electrolytes for lithium metal batteries. Dr. Best was promoted to Research Scientist in 2007 and leads a number of battery research programs utilising ionic liquid electrolytes including to enabling high voltage battery cathodes and lithium-sulfur batteries.

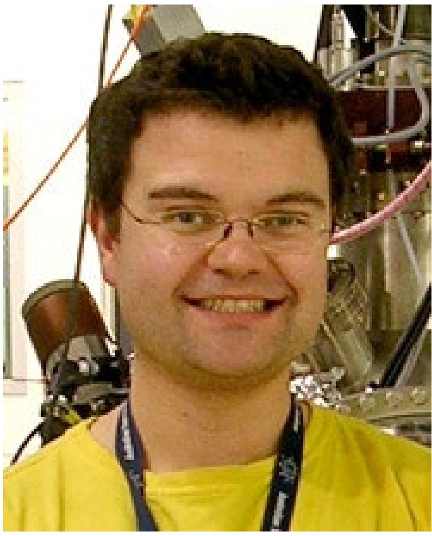
Dr. Best's other research interests include energy harvesting, capacitors and hydrogen storage. He has 52 publications and 10 patents, with a H index of 17 and over 1500 lifetime citations.

DR. MARZI BARGHAMADI

Dr. Marzi Barghamadi has recently completed her PhD on lithium-sulfur batteries at Swinburne University of Technology in collaboration with CSIRO, Energy, Victoria. During her Bachelor's studies, she worked on magnetic nanoparticles synthesis.

After finishing her Master's degree in Chemistry in 2008, she was appointed a research position on nanofibers synthesis. Currently, she is a Research Assistant at CSIRO in the field of lithium batteries, which is her major expertise and interest.

SPEAKER PROFILES



DR. ANTON TADICH

Dr Anton Tadich received his PhD in Physics from La Trobe University in 2008 for his work on the development of a novel instrument for synchrotron-based Angle-Resolved Photoelectron Spectroscopy (ARPES) at the German synchrotron BESSY2.

Immediately following the completion of his PhD, Anton commenced a position as a Scientific Support Officer at the soft x-ray beamline at the Australian synchrotron, where he continues to work today as a Senior Beamline Scientist.

In recent years, Anton has worked toward increasing the capabilities of the beamline and endstation for detailed x-ray photoemission and absorption measurement of the surface electronic and chemical structure of contemporary condensed matter systems including surface functionalised diamond, graphene, topological insulator materials and more.

DR. LACHLAN HYDE

Lachlan holds a BSc (Hons) in Nanotechnology from Flinders University and a PhD in Applied Science (Minerals and Materials) at the University of South Australia where he customised his own apparatus for Plasma Enhanced Chemical Vapor Deposition.

In addition, Lachlan has research proficiencies using the techniques of X-ray Photoelectron Spectroscopy (XPS), Variable Angle Spectroscopic Ellipsometry (VASE), Atomic Force Microscopy (AFM), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM).

At MCN Lachlan is available as the expert in Atomic Layer Deposition (ALD) and VASE and has used these techniques to develop processes for leading scientific organisations such as NASA. When he is not depositing Angstroms of material by ALD, Lachlan can be found depositing microns of ultra pure or boron doped diamond in MCN's world class diamond deposition suite.

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ATOMIC FORCE MICROSCOPY : ROUTINE APPLICATIONS TO COMPLEX METHODS FOR MATERIALS CHARACTERISATION

Prof. Ray Dagastine, Department of Chemical & Biomolecular Engineering, The University of Melbourne

Atomic Force Microscopy (AFM), invented by the same group that received the Nobel Prize for the invention of the scanning tunnelling microscope, was originally developed as a tool to obtain 3-D topography information on the nano-scale for insulating surfaces.

Since its invention thirty years ago, the number of application and uses of AFM has enabled an explosion in the amount of research focused on the visualization of materials at the nano-scale. The extension of the use of AFM to imaging under liquid, atomic scale resolution imaging, conductive AFM, nano-mechanical property mapping and measurement and force spectroscopy methods have driven the uptake and evolution of these methods in a range of fields around material science, soft matter and self assembled systems, polymer physics, biology focusing on biomolecules and living cells, energy materials, semiconductor processing, 2D materials and many more.

Many have expected AFM measurements to become as routine as using a toaster, and this talk will give an overview of the types of capabilities that are often routine in the lab with some of the possible pitfalls for users in analysis. In addition, many applications that use AFM are still more attune to playing a violin, where atomic resolution imaging, some biological applications, sophisticated nano-mechanical methods and a force spectroscopy methods often vary from routine measurements to quite advanced techniques that can require a great deal of expertise.

This talk will highlight a number of these methods, some with more detail based on specific research examples in measuring cell stiffness, material property mapping of complex surfaces, compression of micro-capsules, single molecule unfolding, and force spectroscopy between drops and bubbles.

INVITED ABSTRACTS

2D MATERIALS CHARACTERISATION CAPABILITIES AT THE UNIVERSITY OF MELBOURNE

Dr. Lauren Hyde, Materials Characterisation and Fabrication Platform, The University of Melbourne

The Materials Characterisation and Fabrication Platform (MCFP) supports materials research through advanced instrumentation, analysis and characterisation. The platform facilitates access to a range of complementary technologies, focusing on atomic force microscopy, super-resolution microscopy, nanofabrication, mass cytometry and X-ray diffractionComprised of 4 nodes; Nanomaterials Characterisation, Advanced Fluorescence Imaging, X-Ray Diffraction and Nanofabricaiton, our team of dedicated technical specialists can provide training, advice on experiment design, as well as fee-for service measurements and analysis.

The MCFP welcomes all users across the scientific community, from universities and research institutes to industry. This talk will highlight the many MCFP operated materials characterisation capabilities available to researchers, with a particular emphasis on how they can be applied to characterise 2D materials.

ATOMIC-RESOLUTION IMAGING OF ATOMICALLY THIN MATERIALS VIA TRANSMISSION ELECTRON MICROSCOPY

Dr. Scott Findlay, School of Physics & Astronomy, Monash University
A/Prof. Matthew Weyland, Department of Materials Science & Engineering and Monash Centre for Electron Microscopy, Monash University

Transmission electron microscopy (TEM), in both conventional and scanning modes and making use of aberration-corrected optics, can image the structure of atomically-thin materials with atomic resolution. This enables direct, atomic-level visualization of defect and dislocation structures, stable sites for individual atoms and nanoclusters suspended on the atomically-thin material's surface, charge redistribution around dopants, and measurement of strain. It can also, via spectroscopic techniques, offer local information on the chemistry, electrical properties and bonding of the specimen. However, atomically-thin materials are often beam-sensitive, meaning that their structure may be modified or damaged by the probing electron beam, and care is needed to distinguish intrinsic structures from those induced by beam damage.

This talk will present the capabilities of advanced TEM for exploring the structure of atomically-thin materials. It will overview some of the main TEM imaging modes applied to imaging atomically thin materials and the information they contain, using examples from the recent literature. It will also cover practical aspects of preparing specimens for TEM imaging and the appropriate experimental conditions for imaging these often beam-sensitive specimens.

TRANSMISSION ELECTRON MICROSCOPY OF CURVED GRAPHITIC SURFACES

A/Prof. Laure Bourgeois, Department of Materials Science & Engineering and Monash Centre for Electron Microscopy, Monash University

Layered crystal structures can display an astonishing variety of topologies, from spheres, tubes and cones to the more exotic negatively curved surfaces. The best-known family of curved surfaces is carbon, with “flat” graphite able to curve into an atomically thin sphere (C60), tube (carbon nanotube), cone or saddle.

This talk will present an overview of past and present work on the characterisation of such structures by transmission electron microscopy. This technique has been an important tool in the structural determination of curved nanoscale materials. We will focus on carbon and boron nitride, but will also briefly introduce lesser known “curved crystals”.

A LANDMARK NATIONAL RESEARCH FACILITY SHINING LIGHT ACROSS A DIVERSE SCIENTIFIC LANDSCAPE

Prof. Michael James, The Australian Synchrotron

The Australian Synchrotron is one of Australia’s premier research facilities and, at about $300 million, represents one of the biggest single investments in scientific excellence in the nation’s history. Operated by the Australian Nuclear Science and Technology Organisation, the Australian Synchrotron produces intense beams of X-ray and infrared light at its ten experimental endstations, providing unique research capabilities. The Australian Synchrotron is not only an experimental facility, staffed by ~135 scientists, engineers, technicians, and support staff. At its heart are two highly tuned electron accelerators: a 100 million volt (MeV) linear accelerator that sets packets of electrons moving at relativistic speeds just under the speed of light, before injecting then into the 3 billion volt (3 GeV) booster synchrotron that increases their mass by a factor of ~60. Since commencing user operations in 2007 the Australian Synchrotron has become an integral part of the Australian and New Zealand research landscape. The facility has now supported over 31,000 user visits resulting in scientific research that has already had a significant and lasting impact.

As these high-energy packets of electrons interact with specialised magnetic devices that steer and focus the electron beam around the synchrotron storage ring they generate intense beams of synchrotron light that are used for a huge range of research applications. Access to the world-class facilities at the Australian Synchrotron creates an opportunity for research that can’t be done any other way. As a result some of the best in research from Australia, New Zealand and beyond has been facilitated by access to the Australian Synchrotron. This presentation will touch upon the basics of synchrotron science, and present a diverse range of examples of the facility’s impact across the scientific landscape.

INVITED ABSTRACTS

A BRIEF STATUS REPORT ON SCANNING TUNNELING MICROSCOPY

Dr. Agustin Schiffrin, School of Physics & Astronomy and MCATM, Monash University
Dr. Bent Weber, School of Physics & Astronomy and MCATM, Monash University

The invention of the scanning tunneling microscope (STM) in the early 1980's by Rohrer and Binnig – who soon after won the Nobel Prize in Physics in 1986 – revolutionised the fields of microscopy, advanced materials science and nanotechnology.

This technique relies on the quantum mechanical tunneling effect, according to which a small electric current can flow between two conducting materials separated by a distance on the order of a nanometer. The measurement of such tiny currents allows for the reconstruction of the real-space topography of surfaces with unprecedented atomic-scale resolution.

This tool further opened the door to the manipulation of single atoms and molecules, as well as to the study of the local electronic, magnetic and vibrational properties of materials. In particular, it has been extensively utilised in the study of two-dimensional materials.

Here, we will summarise the theory behind STM, as well as recent advances made in the field, including studies on two-dimensional materials, nano-device fabrication, local ultrafast dynamics and light-assisted STM.

RAMAN AND INFRARED SPECTROSCOPY AND IMAGING AT THE MICRO AND NANO LEVEL

Prof. Donald McNaughton, School of Chemistry, Monash University

A brief introduction to the theory and instrumentation of Raman and Infrared spectroscopy and imaging together with methods of Raman spectral enhancement will be presented. Examples of diffraction limited molecular imaging techniques will be used to show the power of these techniques as well as to point out their limitations.

Recently both of these techniques have been coupled to AFM microscope systems to provide spatial resolution for molecular spectroscopy and imaging down to 20nm. The instrumentation now installed and to be installed at Monash will be described and results on both of these systems will be used to outline their capabilities.

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GRAPHENE CHARACTERISATION CAPABILITIES AT CSIRO MANUFACTURING

Dr. Malisja de Vries, CSIRO Manufacturing

The microscopy facility lies within the Biophysics group at CSIRO Manufacturing with capability and expertise in a diverse range of fields including X-ray science, spectroscopy, structural biology, spectrometry and surface characterisation. The group specialises in holistic characterisation of a vast range of materials, ranging from semi-conductors to protein crystals.

An overview of these complimentary capabilities will be discussed with emphasis in the microscopy area. The microscopy facility specialises in 3D imaging and surface metrology, cryogenic preparation for analysis and ultra-high resolution processing in SEM, TEM and optical microscopy. Case studies will be presented showing the expertise in graphene, spinnable carbon nanotubes and 3D characterisation of polymer bank notes.

PHOTOELECTRON SPECTROSCOPY:
PRINCIPLES AND APPLICATIONS IN 2D MATERIALS RESEARCH

Dr. Anton Tadich, The Australian Synchrotron

Photoelectron spectroscopy (PES) is a surface sensitive photon-in/electron-out experimental technique for investigating the chemical composition and electronic structure of the surfaces of materials to a depth of around 10nm. The technique can be performed using a fixed photon energy source (x-ray or ultraviolet), or alternatively performed at a synchrotron light source where several advantages are offered.

A variant on the technique, Angle Resolved Photoelectron Spectroscopy (ARPES), is one of the most powerful techniques for measuring the electronic bandstructure of condensed matter systems. For 2D materials in particular, ARPES is able to yield the complete bandstructure of the material; results from ARPES experiments feature regularly in research on cutting edge 2D condensed matter systems such as graphene, phosphorene, topological insulators, high Tc superconductors and more.

This presentation will outline the basic principles of PES and its extension to ARPES. Some key examples from recent research using PES at the soft x-ray beamline will be presented, as well as some examples of ARPES from the literature. The talk will also cover the practical issues of performing PES and ARPES experiments, such as sample preparation, vacuum systems, and other in-situ techniques often needed for a complete analysis. Finally, an outline of forthcoming ARPES capability at the Australian Synchrotron will be given.

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ELECTROCHEMISTRY METHODS IN 2D MATERIALS RESEARCH

Dr. Adam Best, CSIRO Manufacturing
Dr. Marzi Barghamadi, CSIRO Energy

Graphene has been found to be a technologically interesting material for use in energy storage on devices. To-date, Graphene has been heavily researched as a material to improve the electronic conductivity of electrodes for lithium-ion batteries, as a barrier for polysulfides in Lithium-Sulfur batteries and as the major constituent for super-capacitors. Of these applications, supercapacitors has attracted the most attention to-date, due to the fact that the effective surface area of graphene material(s) does not depend on the distribution of pores as is found in common carbon materials.

But how do we characterise the electrochemical properties of these materials? There are many common challenges when setting up electrochemical experiments such as cyclic voltammetry. For example, the use of appropriate reference electrodes matched to the electrolyte of choice is critical to properly evaluating the potentials at which electrochemical reactions occur. The preparation of devices, such as battery and capacitor electrodes also poses substantial challenges in ensuring that the materials remain as graphene and don’t collapse to graphite during the slurry mixing and coating process.

This presentation will discuss simple electrochemical experiments and the appropriate set-up, characterisation of capacitors and batteries including the terminology that is commonly used to describe the results. We will show examples from literature to demonstrate the types of experiments and results that may be expected.

2D MATERIALS CHARACTERISATION AND FABRICATION AT ANFF-VIC

Dr. Lachlan Hyde, Melbourne Centre for Nanofabrication

The Melbourne Centre for Nanofabrication (MCN) is Australia’s largest open-access cleanroom. It is host to a comprehensive array of state of the art micro and nanofabrication capabilities and serves as the flagship facility — and national headquarters — within the Victorian node of the Australian National Fabrication Facility (ANFF).

Among its offerings, the MCN houses a variety of instrumentation for performing 2D material characterization. In this talk, we will highlight some of those capabilities as well as discuss some of the FAQs regarding facility access, induction, pricing and training.

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CURRENT TRENDS IN MICROSCOPIC CHARACTERIZATION OF 2D MATERIALS WITH SPECTROSCOPIC IMAGING ELLIPSOMETRY (SIE)

Sebastian Funke, Matthias Duve, Peter H. Thiesen, Accurion GmbH, Stresemanstraße 30, 37079 Göttingen, Germany

Current developments in the field of 2D-Materials are focused on microscopic devices based on stacking different 2D-materials into hybrid-structures, discovering new material configurations by exfoliating unconventional layered materials, optimising CVD processes by minimizing crystallite boarders or using ideal shaped microcrystals as the starting point of a growth process and many more. These developments have in common that they require microscopic non-destructive characterization technologies.

Conventional ellipsometry is well established in the field of thin film metrology due to the exceptionally high resolution in the z-axis, enabling very accurate thickness measurements for nano- and microfilms. However, conventional ellipsometry does not have a sufficient lateral resolution for a number of the described developments nor a direct microscopic visualization with highest contrast. The approach of spectroscopic imaging ellipsometry (SIE) differs from conventional ellipsometry in that the measurements are based on a series of micrographs taken at dedicated orientations of the optical components. The primary measurements are microscopic maps of the ellipsometric angles Delta and Psi (Fig. 1 a, b) or micro maps of dedicated elements of the Müller Matrix. Areas can be condensed by binning, averaging of selected pixels data (Region of interest, ROI, Fig. 1c) of equivalent areas and by using histogram data of inhomogeneous regions.

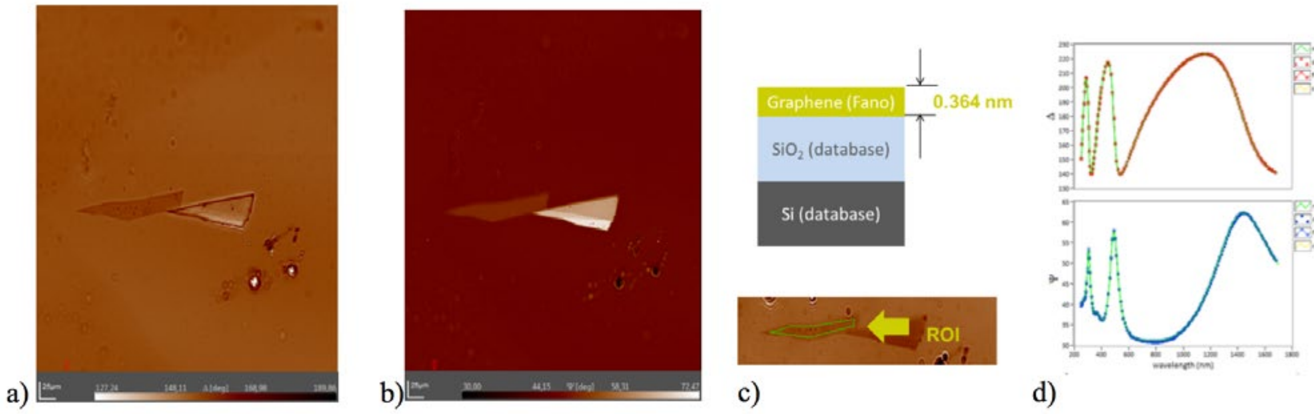

Fig. 1 Spectroscopic imaging ellipsometric measurement of a Graphene flake: Delta (a) and Psi (b) map – examples of a graphene flake, selected Region of Interest and Optical model (c) and wavelength spectra of Delta and Psi, (d) extracted for the ROI from Delta and Psi maps recorded for wavelengths in between 250 and 1700 nm (measured points and model, line).

The data can be transferred into optical properties and metrological parameters like thickness by optical modelling (Fig. 1d). Matkovic et al. [1] for example characterized monolayers of graphene by SIE. The resulting Fano resonance modelling for the dispersion of Graphene can be used to localize flakes of Graphene on different substrates. The flake search algorithm compares the measured ellipsometric values of micro maps of Delta and Psi recorded at a dedicated number of wavelengths with the values for the targeted thickness calculated with the appropriate optical model.

The approach is less dependent on the substrate compared to e.g. conventional light-microscopy. In the talk we will present the localization and characterization of Graphene Monolayer flakes and similar thicknesses regions of insulating hexagonal boron nitride (HBN).

Further investigations on semiconducting transition metal dichalcogenide (TMD) Molybdenum disulphide (MoS_2) are done [2]. To avoid disturbing backside reflections of the substrate the beam-cutter technique is used. From the UV to VIS characterization anisotropy in the dispersion for the out-of-plane component is found.

To conclude, imaging ellipsometry is a technology, based on the same physical concepts as conventional ellipsometry, but because of the microscopic imaging capability and the 2D-detector, the technique offers new paths in metrology and optical characterization of ultrathin films and micro objects.

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AIR-STABLE DOPING OF BI_2SE_3 BY MOO_3 INTO THE TOPOLOGICAL REGIME

M. T. Edmonds[1], J. T. Hellerstedt[1,2], A. Tadich[3], A. Schenk[4], K. M. O'Donnell[3], J. Tosado[1,2], N. P. Butch[2], P. Syers[2], J. Paglione[2], M. S. Fuhrer[1,2]

1. Monash Centre for Atomically Thin Materials and School of Physics and Astronomy, Monash University
2. Centre for Nanophysics and Advanced Materials, University of Maryland
3. Australian Synchrotron, Clayton
4. Department of Physics, La Trobe University

Topological insulators, such as Bi_2Se_3, are a new class of material that possess topologically protected Dirac surface states that hold great promise for future nano-electronic devices including spin generation and detection without ferromagnetism, and dissipationless electronic transport in the quantum anomalous Hall state. However, major challenges exist in realizing Bi_2Se_3 devices that operate in the topological regime in air. The first is that as-prepared Bi_2Se_3 is invariably n-type doped due to selenium vacancies, such that the Fermi level resides in the bulk conduction band, not within the Dirac surface states necessary to realize these electronic devices. The second is that Bi_2Se_3 is unstable upon exposure to atmosphere and becomes further n-type doped and degrades over time [1].

Here we report high-resolution photoemission spectroscopy (PES) of lightly-doped Bi_2Se_3 crystals (surface Fermi energy relative to Dirac point EF – ED ~ 270 meV) [2] cleaved in vacuum and exposed to the molecular dopant MoO_3 [3]. Coverage-dependent PES of Bi core levels allows tracking of the Fermi energy shift, while Mo PES reveals the fraction of charged MoO_3 molecules, and the low-energy cutoff measures the work function. We find efficient acceptor doping by MoO_3, saturating at EF – ED = 100 meV, well within the topological regime. We observe an induced interface dipole of 0.74 eV, corresponding to ~1013 cm^{-2} electrons transferred from the Bi_2Se_3. We implement a doping model based on Fermi-Dirac statistics to accurately describe the doping behavior including saturation. Furthermore, we demonstrate that a 100 nm passivation layer of MoO_3 can be used to acceptor dope a Bi_2Se_3 thin film, as well as prevent degradation of Bi_2Se_3 when it is exposed to atmosphere with carrier density remaining unchanged after two days.

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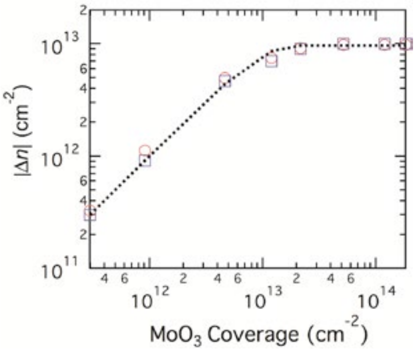

Fig.1 Change in electron density as a function of MoO_3 coverage, where squares and circles are experimental data and the dashed line corresponds to the doping model.

ELECTRONIC PROPERTIES OF HIGH-QUALITY EPITAXIAL TOPOLOGICAL DIRAC SEMIMETAL THIN FILMS

J. T. Hellerstedt[1,2], M. T. Edmonds[1,2], N. Ramakrishnan[3], C. Liu[1,2] , B. Weber[1,2], A. Tadich[4], K. M. O'Donnell[4], S. Adam[3], M. S. Fuhrer[1,2]

1. Monash Centre for Atomically Thin Materials, Monash University
2. School of Physics and Astronomy, Monash University
3. Department of Physics and Centre for Advanced 2D Materials, National University of Singapore
4. Australian Synchrotron, Clayton

Topological Dirac semimetals (TDS) are three-dimensional analogues of graphene, with linear electronic dispersions in three dimensions. Electrical measurements so far have been on bulk TDS crystals and have revealed unusual axion magnetoresistance [1].

Yet, the ability to grow and characterise TDS thin-films on thickness scales ranging from several monolayers to tens of monolayers with transport and scanning tunnelling microscopy (STM) opens up numerous new possibilities, including studying the conventional-to-topological quantum phase transition (QPT) as a function of layer thickness or incorporating gate electrodes to enable an electric field-tuned QPT, realizing a topological transistor [2].

We combine molecular beam epitaxial growth with a low-temperature STM capable of magnetotransport at 5K to study the electronic properties of Na_3Bi thin films. Thin films (20 nm) of Na_3Bi on α-Al_2O_3(0001) substrates are found to possess low temperature charge carrier mobilities exceeding 6000 $cm^2V^{-1}s^{-1}$ with n-type carrier densities below 1 x 1018 cm^{-3} that are comparable to the best single crystal values [3]. Perpendicular magnetoresistance at low field shows the perfect weak-antilocalization behaviour expected for Dirac fermions in the absence of intervalley scattering.

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CHARACTERISATION OF GRAPHENE-BASED ASSEMBLIES: LINKING MORPHOLOGY AND TRANSPORT PROPERTIES

Ashley Roberts, Department of Materials Science and Engineering, Monash University

Reports have shown that graphene-based assemblies consisting of chemically converted graphene (CCG) are tunable structures with unique membrane properties that also exhibit responsive behavior.

Currently, no definitive models exist to describe the structure of these multi-layer graphene-based materials, and results presented here are discussed in relation to a porous carbon, lamellar system. Because the chemical composition, carbon bonding, and planar structure of each graphene sheet varies, multiple 2D and 3D characterization methods must be applied in order to determine key morphological features extending across a very broad length scale.

While XPS, Raman, and XRD provide information about the carbon bonding, X-ray and neutron scattering techniques are able to provide valuable insight on the variation in surface corrugation and interlayer spacing for these materials with the added advantage of performing in-situ experimentation to reveal dynamic behavior. Disorder induced by bonded functional groups along the graphene lattice and sheet corrugation are the main determinants of the surface forces, pore size, and pore size distribution and thus control the transport properties of these assemblies.

MITIGATING FORWARD OSMOSIS MEMBRANE BIOFOULING BY GRAPHENE OXIDE AND GRAPHENE OXIDE-SILVER NANOCOMPOSITE SURFACE FUNCTIONALIZATION

Dr Ming Xie, Institute for Sustainability and Innovation, College of Engineering and Science, Victoria University

We propose the fabrication of anti-biofouling thin-film composite membranes functionalized with graphene oxide and a graphene oxide-silver nanocomposite. In our membrane modification strategy, a crosslinker-mediated reaction is used to bind the carboxyl groups on graphene oxide and graphene oxide-silver sheets to the amine-terminated surface of thin-film composite membranes.

The modified membranes exhibited an 80% inactivation rate against attached Pseudomonas aeruginosa cells as well as the reduced accumulation of microbial biomass on graphene oxide modified membranes. In addition, confocal microscopy demonstrated that cells deposited on the membrane surface are inactivated, resulting in a layer of dead cells on GO-TFC that limit biofilm formation. These findings highlight the potential of GO to be used for biofouling mitigation in FO membrane design.

2D MATERIALS CHARACTERISATIONS: RAMAN, PL, WAVEGUIDES AND PHOTOCURRENT IMAGING

Zaiquan Xu and Qiaoliang Bao, Department of Materials Science and Engineering, Monash University

Two-dimensional (2D) transition metal dichalcogenides (TMDs) and graphene are important building blocks for next-generation miniaturized electronic devices due to their extremely small size, exceptional electronic and optical properties.

The preparations and processing of these materials towards the commercialization of such devices has been one of the main motivations of the research todate. One of the exotic phenomenon in 2D materials is their layer dependant electrical and optical properties, therefore, it is vital to determine the thickness and characterize the optical properties of TMDs.

In this poster, we shall present the capabilities of our confocal Raman microscopy on characterization of 2D materials, i.e., Raman, PL, contrast and waveguides. Beside, energy harvest devices, such as solar cells and photodetectors are key technology that converts optical power into measurable electrical signals, and they are extremely important components in optoelectronic devices. Examples about photocurrent mapping on photodetection and photovoltaics are also given.

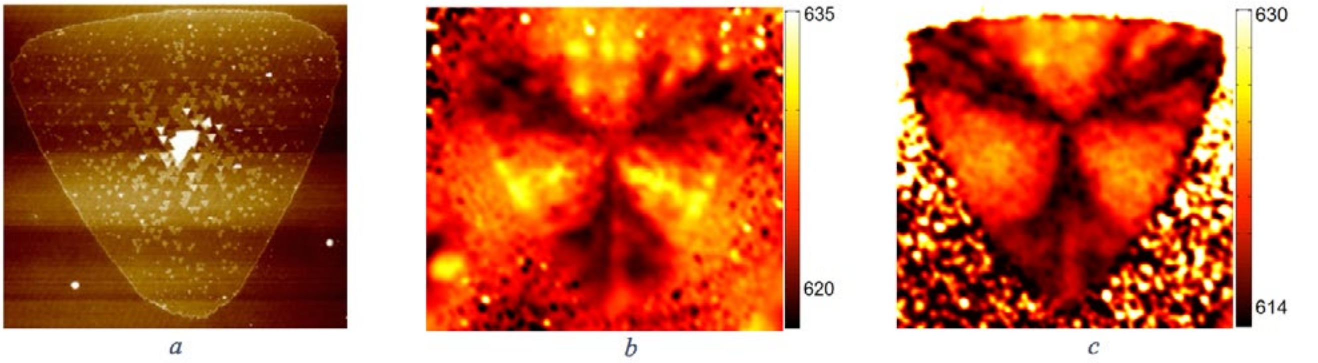
PHOTOLUMINESCENCE AND DIFFERENTIAL REFLECTANCE MAPS OF MONOLAYERS OF WS_2 REVEAL BRANCHED PATTERN

Pavel Kolesnichenko and Jeffrey Davis, Swinburne University of Technology

Monolayers of tungsten disulphide WS_2 have been intensively studied recently by the researchers all over the world for several reasons. First, they have a large direct band gap, which is good for electronics and optoelectronics applications. Second, the lack of inversion symmetry leads to the strong spin-orbit coupling, rendering them as potentially successful candidatures for spintronics applications. Further, the possibility of breaking time-reversal symmetry in WS_2 allows removing the degeneracy of the spin-orbit coupled bands, making the materials useful for valleytronics applications.

In order to fully reveal their potential, first they need to be produced in a confident and controllable way, which requires using different quality estimation techniques, two of which are photoluminescence (PL) and differential reflectance (DR) mapping spectroscopies. PL and DR signals coming from the monolayers are very sensitive to any deviation in the crystal structure from the perfect arrangement of atoms, which may come from defects, micro strains, grain boundary formations, intercalation of water beneath a monolayer, etc.

Our samples were produced at Melbourne Centre for Nanofabrication during a CVD process, assumingly, in conditions of relatively high hydrogen gas flow rates. Outcome of the process is shown on the Fig.1a, revealing multiple second-layer “islands” on top of the first layer. We scanned our samples for PL (Fig.1b) and DR (Fig.1c) signals, using 100x objective with numerical aperture NA = 0.8, implying the maximum achievable resolution to be ~0.4 um for the wavelength of excitation laser beam λ = 433 nm. One of the reasons for appearing of these patterns could be the competition of the growing processes originating from different nucleation centres, forming branches. Another reason could be the way we obtain the images, making the observed pattern no more than optical effect. The nature of the presence of this pattern is still yet to be clarified



a b c

Fig.1. (a) AFM image of WS_2 flake. (b) Wavelength distribution of the PL spectrum peak. (c) Wavelength distribution of A-exciton absorption peak.

FABRICATION AND CHARACTERISATION OF GRAPHENE ON NANO-POROUS ALUMINA

H. Zhan[1], David J. Garrett[1,2], and Jiri Cervenka[1,3]

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3 Institute of Physics ASCR, v.v.i., Cukrovarnická 10, 16253 Praha 6, Czech Republic

Three-dimensional (3D) graphene has been successfully fabricated by graphitization of a diamond-like-carbon (DLC) precursor thin film on nanoporous anodic alumina. This graphitization process as well as the deposition of the DLC film was done by plasma enhanced chemical vapor deposition.

Characterization of this material has been conducted using several techniques, such as Raman Spectroscopy, SEM, TEM, Electron Energy Loss Spectroscopy (EELS), Electrochemical Impedance Spectroscopy (EIS), HF corrosion test, and two-point probe resistance measurement. SEM and TEM analyses show higher quality monolayer to few-layer graphene (FLG) films were obtained, in comparison with 3D graphene produced by thermal graphitization.

The resulting nanoporous graphene/amorphous carbon/alumina structure has a very high surface area, good electrical conductivity and exhibits excellent chemically stability, providing a good material platform for electrochemical applications. Consequently very large electrochemical capacitance values, as high as 2.1 mF for a sample of 1cm×1cm×0.1mm, were achieved. The electrochemical capacitance of the material exhibits a dependence on bias voltage.

DIAMOND QUANTUM SENSORS AS TOOLS FOR REAL-SPACE CHARACTERISATION OF 2D MATERIALS

Dr. Jean-Philippe Tetienne, School of Physics, University of Melbourne

In recent years, there has been tremendous progress in the development of nanoscale quantum sensors based on optically active spins localised at point defects in diamond, and their use to characterise and image a range of solid-state and biological systems. Here we show that they can be a valuable tool to characterise 2D materials, focusing on graphene as a model system.

Our approach consists in fabricating graphene devices directly on a diamond chip containing an array of active spins. The graphene ribbons are visualised directly via fluorescence quenching of the sub-surface spins. Using optically detected magnetic resonance of the diamond spins, we then map the magnetic field induced by the charge current flowing in the electrically driven graphene ribbons, thus providing a way to directly visualise the current path.

We find that the current distribution in our devices is highly inhomogeneous, being channelled away from high resistivity areas. These areas correspond to cracks in the graphene layer, which can be seen both in situ in the fluorescence quenching images, or independently by scanning electron microscopy. With the ability to operate as electric field, magnetic field or temperature sensors, diamond spin sensors open new opportunities for real-space characterisation of 2D materials, including devices operating under ambient conditions.

EFFECT OF CARBON PRECURSOR FLOW RATE ON THE BARRIER PROPERTIES OF A CVD GROWN GRAPHENE COATING

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Graphene coating developed via chemical vapour deposition (CVD) has been well known to improve the corrosion resistance of various metals. However, considerable variations in the barrier properties due to CVD graphene coating in different studies are reported, which is generally attributed to the variations in the uniformity of the coating.

Although it is well known that coating synthesis parameters determine barrier properties of a coating, there are few investigations on the influence of various CVD process parameters (such as, carbon precursor flow rate, growth temperature, hydrogen flow rate etc.) on the uniformity, and hence, the barrier performance of graphene coatings.

In fact, optimization of these parameters would enable a systematic evaluation of the potential of graphene as an effective corrosion barrier for metallic substrates. In the present study, the influence of carbon precursor flow rate on the corrosion resistance of a graphene coating has been analysed. Graphene was grown on Cu at various flow rates (0.4 0.8 and 1.6 SCCM) of the carbon precursor (n-hexane) maintaining other parameters identical in each case such as growth temperature, annealing temperature, hydrogen flow rate etc..

The uniformity, quality, thickness and post corrosion morphologies of the graphene coatings were evaluated by optical microscopy and Raman spectroscopy. Potentiodynamic polarisation and electrochemical impedance spectroscopy (EIS) were performed on the coated and uncoated Cu specimens in 0.1 M sodium chloride (NaCl). Interestingly, the barrier properties of the graphene coating increased with increasing flow rate of the precursor.

EXFOLIATION OF BI$_2$S$_3$: A NEW 2D MATERIAL

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Bismuth sulphide is a group V-VI semiconductor with a band gap of 1.3 eV. The highly anisotropic, orthorhombic crystal structure favours formation of one-dimensional materials such as nanowires or nanotubes. These 1D forms have been shown to have useful properties for applications in thermoelectrics, photodetectors and solar cells. Although, there have been no previous reports of exfoliation of bulk Bi$_2$Se$_3$ in an attempt to form two-dimensional structures.

Success with liquid phase exfoliation of graphene and transition metal dichalcogenides has prompted interest in the investigation of exfoliation of other layered materials. In this chemical exfoliation process ultrathin sheets of Bi$_2$Se$_3$, with lateral dimensions up to 30 μm, are formed. AFM characterisation shows that the sheets are as thin as 2 nm when lying flat. Low voltage SEM was performed using a stage bias and concentric backscatter (CBS) detector, for detailed information about the surface morphology and wrinkling of the sheets.

This new dimensionality of an already established material provides increased surface area and altered electronic properties. Direct synthesis of uniform thin films, by side stepping the assembly of intertwined one-dimensional particles, provides a more reliable option for device fabrication. These new 2D sheets have the potential to be used for applications such as thin film electronic devices and sensors.

DYNAMIC ELECTROSORPTION ANALYSIS: A VIABLE LIQUID-PHASE CHARACTERISATION METHOD FOR POROUS CARBON?

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Characterisation is one of the most fundamental aspects in porous carbon research. Due to the complex structure and diversity in crystallinity, morphology, texture, and porosity of traditional porous carbon materials, revealing their structure both on microscopic and bulk levels remains challenging.

Using recently developed multilayered graphene membranes as a model porous carbon, this article highlights the potential and challenges of using dynamic electrosorption analysis as a characterization tool for probing the structure of porous carbon in the solution phase. With further theoretical and in situ experimental investigation falling into place, it is expected that dynamic electrosorption analysis could be developed into a standardized technique that complements and covers up the shortfall of the traditional gas adsorption methods.

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