Confirmed Exhibitors:
## Tuesday 20 March

<table>
<thead>
<tr>
<th>Location</th>
<th>Vincent Auditorium</th>
<th>Topic #</th>
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<tbody>
<tr>
<td>9:00 - 9:15</td>
<td>Welcome - Professor Sir Peter Gregson, Chief Executive and Vice-Chancellor Cranfield University</td>
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<tr>
<td>9:15 - 9:30</td>
<td>Opening – J L Endrino and coorganisers</td>
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<tr>
<td>9:30 - 10:00</td>
<td>Graphene Industry opportunities and challenges</td>
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<tr>
<td>10:00 - 10:30</td>
<td>Tailoring the absorption and emission properties of nanomaterials through their photonic environment</td>
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<tr>
<td>10:30 - 11:00</td>
<td>Coffee break / exhibition</td>
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<tr>
<td>11:00 - 11:30</td>
<td>Optimized properties of Al-doped ZnO thin films</td>
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<td>11:30 - 12:00</td>
<td>The catalytic potential of new MOF-based composite materials</td>
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<tr>
<td>12:00 - 12:30</td>
<td>Structure-based design and applications of closed porous coatings fabricated by magnetron sputtering</td>
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<tr>
<td>12:30 - 1:30</td>
<td>Lunch / exhibition</td>
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<tr>
<td>1:30 - 2:00</td>
<td>The Roadmap to applications of graphene and related materials</td>
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<td>2:00 - 2:30</td>
<td>Graphene and 2D materials for next generation optoelectronics and photonics</td>
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<td>2:30 - 3:00</td>
<td>Contact, friction and wear of carbon and silicon: from thin films to atomic sheets</td>
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<tr>
<td>3:00 - 3:30</td>
<td>Mo2BC thin films - a material system combining hardness and ductility?</td>
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### Topic 1 Session
**Contributed talks - Protective and tribological coatings**

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<th>Location</th>
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<tbody>
<tr>
<td>4:00 - 4:15</td>
<td>Evaluation of the corrosion performance of aluminium-based thin films as potential alternatives to cadmium coatings. Sarah Banfield, Wallwork Cambridge (UK)</td>
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<tr>
<td>4:15 - 4:30</td>
<td>Realistic lifetime estimation of protective DLC coatings for articulating biomedical implants: combined dynamic and corrosion wear test. Ainhoa Pardo, Empa (CH)</td>
</tr>
<tr>
<td>4:30 - 4:45</td>
<td>HIPIMS deposited CrN/NbN coatings to preserve the mechanical properties of the substrate material and protect against steam oxidation and water droplet erosion attacks. Papken Eh. Hovsepian, Sheffield-Hallam (UK)</td>
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<tr>
<td>4:45 - 5:00</td>
<td>Positive voltage reversal in HiPIMS discharges: widening the process window. Ivan Fernandez-Martinez, Nano4energy (ES)</td>
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<tr>
<td>5:00 - 5:15</td>
<td>Oxide coated cemented carbides for high temperature applications. Jessica Marshall, Warwick University (UK)</td>
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### Topic 6 Session
**Contributed talks - sensors and instruments based on nanofilms**

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<th>Location</th>
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<tbody>
<tr>
<td>4:00 - 4:15</td>
<td>Growth of large area conductive diamond for electrochemical and bio-sensing devices. Andrew Taylor, Institute Of Physics Cas (CZ)</td>
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<tr>
<td>4:15 - 4:30</td>
<td>NanoMIP-EIS-Sensors to detect traces of cocaine. Roberta D’aurelio, Cranfield University (UK)</td>
</tr>
<tr>
<td>4:30 - 4:45</td>
<td>Sensing of ultralow concentration of the target protein exploiting imprinted nanogels and plasmonic plastic optical fibers. Alessandra Maria Bossi, University of Verona (IT)</td>
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<tr>
<td>4:45 - 5:00</td>
<td>Large-Area Microchannel plates and photodetectors. Till Cremer, Incom Inc (US)</td>
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<tr>
<td>5:00 - 5:15</td>
<td>Gold nanoparticle-enhanced immunosensors for rapid malaria detection. Aver Hember, Cranfield University (UK)</td>
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<tr>
<td>5:15 - 5:30</td>
<td>Annealed nanostructures for biosensing applications. Rodica Elena Ionescu, Université de technologie de Troyes (FR)</td>
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<tr>
<td>5:30 - 5:45</td>
<td>Functionalized cellulose acetate membranes as coatings for biomedical applications. S.I. Voicu, University Politehnica of Bucharest (RU)</td>
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Wednesday 21 March

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<tr>
<th>Location</th>
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<tr>
<td>9:00-10:30</td>
<td>Industrial Breakfast Panel (Open to the public) Chair: Dave Rickerby, Cranfield University (UK) Confirmed guests: John Christopher, Enterprise Europe Network (UK) Laurent Espitalier, Wallwork Cambridge (UK) Trevor Walker, Thin Metal Films (UK)</td>
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<tr>
<td>10:30 - 11:00</td>
<td>Coffee break / exhibition</td>
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<tr>
<td>11:00 - 11:30</td>
<td>Electrochemical Protein, DNA and Aptamer-based Biosensors Pedro Estrela, University of Bath (UK)</td>
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<tr>
<td>11:30 - 12:00</td>
<td>Photoinduced preparation of Molecularly Imprinted Polymers (MIP) for sensor applications Olivier Soppera, French National Centre for Scientific Research (FR)</td>
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<tr>
<td>12:00 - 12:30</td>
<td>Recent advances in polymer nanocomposites based biosensors Giovanna Marrazza, University of Florence (IT)</td>
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<tr>
<td>12:30 - 1:30</td>
<td>Lunch / exhibition</td>
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<tr>
<td>1:30 - 2:00</td>
<td>Wear resistance of HVAF-sprayed hardmetal coatings Heli Koivuluoto, Tampere University of Technology (FI)</td>
<td>1</td>
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<td>2:00 - 2:30</td>
<td>Tribooxidation as a way to improve the wear resistance of cutting tools Dmitry Wainstein, Bardin Central Research Institute (RU)</td>
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</tr>
<tr>
<td>2:30 - 3:00</td>
<td>Behavior of micro-scaled composite TBCs under corrosive attack by CMAS deposits Daniel Emil Mack, Forschungszentrum Jülich GmbH (DE)</td>
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<tr>
<td>3:00 - 3:30</td>
<td>Self adaption: a solution for improving the friction behaviour of thin sputtered coatings Albano Cavaleiro, University of Coimbra (PT)</td>
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<tr>
<td>3:30 - 4:00</td>
<td>Coffee break / exhibition</td>
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**Topic 5 Session**
Contributed talks - 2D materials: graphene and beyond

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<tr>
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<tbody>
<tr>
<td>4:00 - 4:15</td>
<td>Characterisation of femtosecond pulsed laser deposited MoS2 thin films for photonic applications Chiranjeevi Maddi, University Of Leeds (UK)</td>
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<tr>
<td>4:15 - 4:30</td>
<td>Freestanding Graphene Gyroids Tomasz Cebo, Cambridge University (UK)</td>
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<tr>
<td>4:30 - 4:45</td>
<td>Electrospun Piezoelectric Thermoplastic Nanofibre Layers for Toughening and In-situ Measurement of Laminated Composites Hamed Yazdani Nezhad, Cranfield (UK)</td>
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**Topic 3**
From atoms to sheets: growth and tribology of thin films

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<tr>
<th>Location</th>
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<tbody>
<tr>
<td>4:00 - 4:15</td>
<td>Multiscale modelling of thermally sprayed particle impact and coating formation Jordan Davidson, Robert Gordon University (UK)</td>
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<tr>
<td>4:15 - 4:30</td>
<td>Influence of multiply charged ions on the adhesive strength of coatings at HV pulsed negative bias on a substrate Petr A. Tsygankov, Universidad Industrial de Santander (CO)</td>
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<tr>
<td>4:30 - 4:45</td>
<td>Nanomechanical characterisation of DLC coating systems Sam McMaster, University of Leeds (UK)</td>
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<td>4:45 - 5:00</td>
<td>Developments in nano-impact testing on optical thin films Luis Isern Arrom, Cranfield University (UK)</td>
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<tr>
<td>5:00 - 5:15</td>
<td>Determination of the mechanical properties of coatings and thin films using nanoindentation and analytical model Andrei S. Vasiliev, Don State Technical University (RU)</td>
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</tr>
<tr>
<td>Location</td>
<td>Vincent Auditorium</td>
<td>Topic #</td>
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| 9:00 - 10:30 | Special Seminar Nanofilms for Breast Cancer Detection Devices Chairs: I. Chianella, J.L. Endrino  
Heterostructure Radiation Detector Materials for the next generation of Time of Flight Positron Emission Tomography Scanners  
Field Cancerization effect in the early detection of cancer  
Zinc isotopes as a possible biomarker for breast cancer  
Development of a Point of Care Biosensor for the Detection of Breast Cancer Biomarkers |
| 9:30 - 10:00 | Coffee break / exhibition |
| 10:00 - 11:00 | Design, validation and thermal testing of durable central receiver coatings for high-temperature concentrated solar power  
Multiscale modelling of Meso-Bio-Nano (MBN) systems with MBN Explorer and MBN Studio: thermally induced morphological transitions of nanostructures  
Self-powered high performance photodetectors for ultraviolet and visible lights |
| 11:00 - 12:00 | Lunch / exhibition |
| 12:00 - 1:30 | Towards functional Copper Halide thin films for Optoelectronics  
Fracture toughness and yield strength of hard, thin coatings at high temperatures using in situ micromechanical testing |

### Thursday 22 March

#### Topic 2
Thin films for energy conversion, catalysis and related processes

<table>
<thead>
<tr>
<th>Location</th>
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| 11:00 - 12:00 | Design, validation and thermal testing of durable central receiver coatings for high-temperature concentrated solar power  
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| 12:00 - 1:30 | Lunch / exhibition |
| 1:30 - 2:00 | Towards functional Copper Halide thin films for Optoelectronics  
Fracture toughness and yield strength of hard, thin coatings at high temperatures using in situ micromechanical testing |

### 3:00 - 3:30
Characterization by TEM and Raman of high-density low-defect-concentration zinc oxide nanowires grown by afterglow  
Thierry Belmonte, Institut Jean Lamour (FR)

### 3:30 - 4:00
Coffee break / exhibition

### Topic 2
Thin films for energy conversion, catalysis and related processes

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<tr>
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</table>
| 4:00 - 4:15 | Infrared electrochromism of VO2/Ag nanowires electrodes in ionic liquid  
REACH compliant epoxides used in the synthesis of Fe2O3 aerogel monoliths for target fabrication |
| 4:15 - 4:30 | Interconnections between electronic structure and optical properties of multilayer nanolamine TiAIN/Ag and Al2O3/Ag coating  
Tuning of catalytic activity by thermoelectric materials |
| 4:30 - 4:45 | Characterization by TEM and Raman of high-density low-defect-concentration zinc oxide nanowires grown by afterglow  
Thierry Belmonte, Institut Jean Lamour (FR)  
Interconnections between electronic structure and optical properties of multilayer nanolamine TiAIN/Ag and Al2O3/Ag coating  
Dmitry Wainstein, I.P. Bardin (RU) |
| 4:45 - 5:00 | Towards functional Copper Halide thin films for Optoelectronics  
New developments in solid polymer electrolytes for electrochromic smart windows  
Plasma Printing Oxide nanoparticles by in-situ Tailoring of Oxidation State |
| 5:00 - 5:15 | The role of coatings in addressing a more cost-effective PEM electrolyser  
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**Location:** Vincent Auditorium  
**Topic:** 

**9:00 - 10:30**  
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**12:00 - 1:30**  
Lunch / exhibition

**1:30 - 2:00**  
Towards functional Copper Halide thin films for Optoelectronics  
Fracture toughness and yield strength of hard, thin coatings at high temperatures using in situ micromechanical testing

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**Location:** Vincent Auditorium  
**Topic:**

**3:00 - 3:30**  
Characterization by TEM and Raman of high-density low-defect-concentration zinc oxide nanowires grown by afterglow  
Thierry Belmonte, Institut Jean Lamour (FR)

**3:30 - 4:00**  
Coffee break / exhibition

**4:00 - 4:15**  
Infrared electrochromism of VO2/Ag nanowires electrodes in ionic liquid  
Nicolas Glandut, University Of Limoges (FR)

**4:15 - 4:30**  
REACH compliant epoxides used in the synthesis of Fe2O3 aerogel monoliths for target fabrication  
Paul Jones, Cranfield University (UK)

**4:30 - 4:45**  
Interconnections between electronic structure and optical properties of multilayer nanolamine TiAIN/Ag and Al2O3/Ag coating  
Dmitry Wainstein, I.P. Bardin (RU)

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**Location:** LR 1  
**Topic:** Thin films for energy conversion, catalysis and related processes

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Dmitry Wainstein, I.P. Bardin (RU)

**4:45 - 5:00**  
Tuning of catalytic activity by thermoelectric materials  
Zhaorong Huang, Cranfield University (UK)

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**Location:** LR 1  
**Topic:** Thin films for energy conversion, catalysis and related processes

**5:00 - 5:15**  
The role of coatings in addressing a more cost-effective PEM electrolyser  
Marlon R. Cruz Vivas, Flubetech S.L. (ES)

**5:15 - 5:30**  
New developments in solid polymer electrolytes for electrochromic smart windows  
Quentin Lonne, Cranfield University (UK)

**5:30 - 5:45**  
Plasma Printing Oxide nanoparticles by in-situ Tailoring of Oxidation State  
Avishek Dey, The Open University (UK)
Thursday 22 March

**Topic 4**
Functionalisation and characterisation of coatings and nanofilms

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<tr>
<td>4:00 - 4:15</td>
<td>Characterisation of an antimicrobial silver nanocomposite coating on orthopaedic grade cobalt chromium alloy&lt;br&gt;Liuquan Yang, Wallwork Cambridge (UK)</td>
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<td>4:15 - 4:30</td>
<td>Synthesis and deposition of polyfluoroacrylate film on anodised aluminium surface for hydrophobic and icephobic applications&lt;br&gt;Tim Barman, The University of Nottingham (UK)</td>
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<td>4:30 - 4:45</td>
<td>Characteristics of bloch surface wave resonances in total internal ellipsometry spectra: effect of adsorbed nanolayer on selected dielectric multilayers&lt;br&gt;Karl Fleury-Frenette, Université de Liège (BE)</td>
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<tr>
<td>4:45-5:00</td>
<td>High temperature nanoindentation up to 800°C for characterizing high temperature properties of coatings&lt;br&gt;Nicholas X. Randall, Anton Paar (CH)</td>
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<td>5:00 - 5:15</td>
<td>Biopolyurethane – carbon nanotubes composite for functional coating applications&lt;br&gt;Uks Piatnieks, Riga Technical University (LV)</td>
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<td>5:15 - 5:30</td>
<td>Optimization of silver nanocolumnar coatings made by glancing angle deposition with magnetron sputtering to reduce the multipactor effect&lt;br&gt;J. M. García-Martín (Instituto de Micro y Nanotecnología IMN-CNIM (ES))</td>
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<td>5:30-5:45</td>
<td>Development of smart coating for structural health monitoring&lt;br&gt;Stephen Edmondson, Direct-C (CA)</td>
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<td>5:45-6:00</td>
<td>CuS-based p-type transparent conducting thin film for all transparent electronics&lt;br&gt;Arundam Mallick, Indian Association for the Cultivation of Science (IN)</td>
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**Poster Session**

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<tr>
<td>Charactization of bio-deterministic surfaces fabricated by photochemical machining of ai52100 steel coated with DLC and MoS2 for tribological applications&lt;br&gt;Juan C. Sanchez Gonzalez, National University of Colombia (CO)</td>
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<td>Control of the compensating defects in doped ZnO nanocrystalline thin films&lt;br&gt;Shuvraj Ghosh, Indian Association for the Cultivation of Science (IN)</td>
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<td>Large area synthesis and properties of diamond - silicon carbide composite thin films for enhanced protective coatings&lt;br&gt;Andrew Taylor, Institute of Physics CAS (CZ)</td>
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<tr>
<td>(Al, In) co-doped nanocrystalline ZnO transparent conducting thin films for rigid and flexible substrates&lt;br&gt;A Mallick, Indian Association for the Cultivation of Science (IN)</td>
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<td>Nickel-aluminide based anticorrosion coatings prepared by Plasma Spray for Concentrating Solar Power applications&lt;br&gt;Sarah Yasir, Cranfield University (UK)</td>
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<td>Manufacturing of devices using light cure resin with nanofillers of copper and copper nickel through stereolithography technique&lt;br&gt;Ely Dannier V-Niño and Andrés Díaz Lantada, Universidad Politécnica de Madrid (ES)</td>
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<tr>
<td>Electrochemical impedance spectroscopy biosensor for neuron specific enolase (NSE) biomarker for lung cancer detection&lt;br&gt;Mahdi Arabnejad, Cranfield University (ES)</td>
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<td>New DNA-based sensing platform for the detection of profenofos pesticide&lt;br&gt;Oana Hosu, University of Florence (IT)</td>
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<tr>
<td>Development of thin films for applications in piezoresistive sensors for cancer detection&lt;br&gt;James Janderson Rosero Romo, National University of Colombia (CO)</td>
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<td>Localized surface plasmon resonance in laser- deposited gold nanoparticle layers for total internal reflection based optical sensors&lt;br&gt;Juri Hastanin, Université de Liège (BE)</td>
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<td>Sharp fall of the thermal conductivity of a van der Waals heterobilayer due to interlayer sp3 bonds&lt;br&gt;Kazuhito Shintani, University of Electro-Communications (JP)</td>
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<td>Self-supporting Diamond-Like Carbon (DLC) as a material for laser-driven particle sources&lt;br&gt;David Haddock, Rutherford Appleton Laboratories (UK)</td>
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<td>Characterization of vacuum deposited thin organic layers by phase shifting interferometry and FTIR spectroscopy for space contamination study Theo Henry, Université de Liège (BE)</td>
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<tr>
<td>A simulation testbed to study the pseudoelastic behaviour of Cu-Zr binary alloys during a tensile test using molecular dynamics Fabian Duarte Martinez, Cranfield University (UK)</td>
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<tr>
<td>Optical diagnostic and formation of thin films of titanium through of the electric arc discharge F. F. Parada Becerra, Universidad Industrial de Santander (CO)</td>
<td>3</td>
</tr>
<tr>
<td>Investigation of the characteristics of cutting tools coated with mono-layer DLC and multi-layered DLC-WS2 PVD coatings Tomasz Brzezinka, Cranfield University (UK)</td>
<td>1</td>
</tr>
<tr>
<td>Plasma functionalization of Spiro-OMeTAD hole transport material for material for perovskite solar cells Paheli Ghosh, The Open University (UK)</td>
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</tr>
<tr>
<td>Carbon nanoparticles containing polymer films and coatings with EMI properties Romans Vaivodiss, Rīgas Tehniskā universitāte (LV)</td>
<td>5</td>
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Electroplated cadmium coatings are used in a wide variety of applications, primarily to provide corrosion protection to steel substrates. The dense deposits possess excellent barrier and sacrificial corrosion properties and provide galvanic protection to coated parts in contact with aluminium. These properties, amongst others, have led to the widespread use of cadmium coatings in many industries (e.g. military and aerospace). However the hazardous nature of cadmium (which is harmful to both humans and the environment) coupled with increasingly strong legislative pressure has driven research to develop safer alternatives. In this study, thick PVD AlCr(N) coatings with varying amounts of Cr were deposited on steel substrates by magnetron sputtering. The corrosion properties of the Al-based deposits were subsequently evaluated using an improved AC/DC/AC cyclic corrosion degradation test, which conventionally combines DC cathodic polarisation and periodic Electrochemical Impedance Spectroscopy (EIS) measurements.

This accelerated test was recently modified to include a pH probe in order to monitor the evolution of the electrochemical test solution pH - an important parameter which strongly affects the corrosion rate of aluminium alloys. The results reveal that, while increasing the Cr content improves the mechanical and structural properties of the Al-rich deposits, AlCr(N) coatings with lower Cr content (less than 10 at%) exhibit better corrosion resistance properties - which can be attributed to the formation of a dense, thick passive film on their surfaces. These promising results could be used to build novel graded multilayer Al-rich coatings with controlled mechanical and electrochemical properties, in which individual layers are tailored to meet specific property requirements, enabling the replacement of cadmium over a wide range of practical applications on steels and other engineering metals.
Realistic lifetime estimation of protective DLC coatings for articulating biomedical implants: combined dynamic and corrosion wear test (Contributed talk)
Ainhoa Pardo (Swiss Federal Laboratories for Materials Science and Technology- Empa), E. Ilic, P. Schmutz, R. Hauert

Current underestimated in-vitro lifetime predictions of Diamond Like Carbon (DLC) coated articulating implants lead to premature revision surgeries. The objective of the present work is the development of an advanced experimental methodology, able to achieve reliable lifetime estimations by mimicking body working conditions.

Since it was already shown that DLC coatings can overcome the wear and chemical inertness requirements when articulating in vivo joint replacements, this research focuses on the interface, which can determine the lifetime of the exposed system. Reciprocating sliding tests immersed in Phosphate Buffer Solution (PBS) electrolytes simulate time dependent corrosion and fatigue processes at the DLC/Substrate interface. In parallel the electrochemical characterization of the system reveal the key role played by an intentional oxygen contamination. Small amounts of oxygen at the interface, characterized by XPS, show an increased delamination under the dynamic load-corrosion tests. The results significantly contribute to achieve reliable lifetime estimations of carbon coated implants, which can also be applied to test the dynamic loading in corrosive media for other interfaces typically used to enhance the adhesion of DLC in contact with metallic substrates.

Positive voltage reversal in HiPIMS discharges: widening the process window (Contributed talk)
Ivan Fernandez-Martinez (Nano4energy), A. Wennberg, J-A Santiago, V. Bellido-Gonzalez, A. Mendez-Fernandez

The application of a positive voltage reversal pulse adjacent to the negative sputtering pulse has revealed unprecedented benefits for the coating industry, such as enhanced ion assistance to the growing film observed in hard carbon coatings or the increased ion incorporation in metal nitrides. The reasons for these findings are the formation of highly energetic ions in a reversed electric field with energies proportional to the cathode positive voltage reverse amplitude. If a substrate, either metallic or insulating, is placed in front of the target, an increased ion bombardment of the growing film is clearly observed. A description of this technology as well as different experimental results showing its benefits will be presented in this paper, such as:

- enhanced the ion-assisted deposition of films on glass substrates, particularly DLC and oxide layers.
- up to 20-30% deposition rate increase in hard metal nitrides, which improves the productivity of industrial HiPIMS coating machines for applications such as cutting tools, molds, dies, and metallization or decorative applications.
- deposition of hard coatings in temperature sensitive substrates.
- improved trench filling for semiconductor industries.

HIPIMS deposited CrN/NbN coatings to preserve the mechanical properties of the substrate material and protect against steam oxidation and water droplet erosion attacks (Contributed talk)

A significant reduction of CO2 emissions is expected by increasing the efficiencies of the steam turbines to $\eta > 50\%$ which can be achieved by moving from subcritical low pressure/ low temperatures, to high pressure/high temperature, ultra-supercritical regime of operation. The main challenges faced by different steel components of the power plant with this approach however, consist of material failure due to high temperature oxidation, and phenomenon such as creep, erosion and descaling after a stipulated period of time.

In the current work, 4 µm thick CrN/NbN coating utilising nanoscale multilayer structure with bi-layer thickness of $\Delta = 3.4$ nm has been used to protect low Cr content P92 steel widely used in steam power plants. The novel High Power Impulse Magnetron Sputtering (HIPIMS) deposition technology has been used to deposit CrN/NbN with enhanced adhesion (critical scratch adhesion value of Lc = 80N) and very dense microstructure as demonstrated by XTEM imaging.

P92 coated samples were oxidised at 600°C in 100% high pressure, 50 bar steam atmosphere up to 1500 h. The gas-flow velocity through the reaction zone of the test rig was 0.0133 m/s. In these conditions CrN/NbN provided reliable protection of the P92 steel. This research also revealed that unlike other state-of-the-art PVD technologies, HIPIMS does not have an adverse effect on the mechanical properties of the substrate material, which is of paramount importance in case of turbine blade applications. In high temperature (650°C) tensile strength test uncoated P92 steel showed Ultimate Tensile Strength (UTS) values of 229 MPa and Yield Strength, (YS) values of 222 MPa compared to UTS = 307 MPa and YS = 291 MPa measured for CrN/NbN coated P92 steel. Similarly in strain controlled, (0.4% strain) Low Cycle Fatigue tests at 650°C both uncoated and coated specimens failed after similar number of cycles, Nf = 1700 and Nf = 1712 respectively and showed similar half-life stress drop of -37% and -43% respectively. Finally high temperature creep tests at 650°C, tensile stress of 120 MPa revealed that the HIPIMS coating improved the creep lifetime by almost factor of two from 564 hours to 908 hours whereas the creep rate was decreased from 17.6 10⁻⁶ s⁻¹ to 13.5 10⁻⁶ s⁻¹.

The protection properties of the coating against water droplet erosion attack were tested using specialized test rig. The coating shows high resistance against water droplet erosion. After 2.4E6 impacts no measurable weight loss was detected.
A new method of retarding oxidization of cemented tungsten carbide at high temperatures in air is presented in this work. Cemented tungsten carbides (cWCs) have traditionally been used as tool tips due to their combination of high hardness and toughness from a ductile metal binder. Recent investigations have traditionally used as tool tips due to their combination of high hardness and toughness from a ductile metal binder. Recent investigations have demonstrated the potential application of cWCs as nuclear shielding materials.

This work addresses both these issues that limit the use of cWCs in high temperature hard radiation environments. Following the identification of a non-activating metal binder

1. show that cWCs are a potential candidate material for a compact radiation shielding material which is of critical importance for future nuclear power generation.

Two major factors have limited their use as a nuclear shield:

1) Co metal the most common metallic binder is an activation hazard and
2) cWCs suffer from significant degradation due to oxidization at relatively low temperatures.

This work addresses both these issues that limit the use of cWCs in high temperature hard radiation environments. Following the identification of a non-activating metal binder

2. the use of a Si-based coating on cWCs has been demonstrated to form a protective layer structure consisting of a FeSix outer crust and a WSi2 inner layer. Thin Si films were deposited onto WC-FeCr cemented carbides via the pack cementation method in a reducing atmosphere at 1000°C, forming a mixed FeSix/WSi2 layer structure. The FeSix was observed to form a SiO2 containing passivating outer layer. This significantly reduced the oxidization kinetics of coated cWCs relative to uncoated cWC materials and demonstrated the potential application of cWCs as nuclear shielding materials.

1) Windsor et al. Nuclear Fusion (3) 57 (2017)

The wear resistance of metallic components can be significantly improved by applying a thermally sprayed hardmetal coating layer on the surface. For that reason, thermally sprayed hardmetal coatings, e.g. Cr3C2- and WC-based, have established their place as the industrial standard solution for numerous demanding applications. For instance, thermally sprayed Cr3C2-25NiCr coatings are known to have good wear, corrosion and oxidation resistance. The coating properties and resulting component performance can be effectively tailored by thermal spray processing. Novel high kinetic thermal spray processes, e.g. modern high velocity air-fuel (HVAF) spray process, have proven to produce high quality coatings.

In the HVAF spray process, the particle heating and acceleration can be efficiently controlled by changing process parameters. This, in turns, affects the coating characteristics. Wear properties such as abrasion, sliding, erosion and cavitation erosion resistance of the HVAF sprayed hardmetal coatings have been extensively studied and compared to traditional high velocity oxygen-fuel (HVOF) sprayed hardmetal coatings. Higher particle velocities and lower process temperatures in the HVAF spray process produce dense coatings with improved toughness and more homogenous coating structure, resulting in enhanced wear protection and performance in several wear conditions.

Typically the streams of external energy and matter during high speed cutting are causing damage of tool materials and hard protective coatings through multiple mechanical and chemical processes. From other hand, oxidation processes could be used to improve the tools lifetime.

The talk is dedicated to mechanisms of non-equilibrium protective oxides formation under severe tribological conditions such as high speed dry cutting. Tribooxidation during friction is a non-equilibrium adaptation process. The structure and phase transformations on the wear surface in the nanostructured single layer and nanolaminated multilayer PVD coatings were investigated by XPS, AES including Auger elemental mapping, EELFS and HREELS methods. The obtained results show that the enhancement of non-equilibrium processes during friction leads to a dominating formation of protective triboceramics on base of sapphire-like, tungsten and niobium polyvalent oxides with structure that critically improve wear performance.

The dynamics of secondary phases formation on various stages of tool life revealed by electron spectroscopy will be demonstrated in the report. The role of polyvalent metals and multilayer coatings in protective oxides nanofilms formation will be discussed in the talk

Acknowledgements.

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A possible solution for coatings requiring performance in mechanical components.

**Self adaption: a solution for improving the friction behaviour of thin sputtered coatings**

Albano Cavaleiro (University of Coimbra, Portugal)

To achieve low friction in a tribological application, low shear strength conditions have to occur in the sliding contact. Whenever liquid lubrication has to be avoided (environmental concerns, high temperatures, vacuum,...), solid lubricants are the evident alternative. However, the intrinsic low friction of these materials is associated with low shear strength which, by its side, is a controversial property when high-temperature resistance is required. This has an even enhanced impact when protective coatings are concerned, knowing that their development has been usually directed for the maximization of the hardness (mechanical strength) and corrosion resistance, to achieve an extended lifetime and an optimized performance in mechanical components. A possible solution for coatings requiring an excellent mechanical resistance and, simultaneously, providing low friction during application, is to modify the chemical composition of existing coatings systems, well established in the market, with elements that can give rise to a self adaptation mechanism consisting on the formation of a lubricating tribolayer, keeping unchanged the original mechanical strength.

In this talk, several examples will be presented demonstrating the self adaption concept for hard and self lubricating coatings. For low temperature tribological applications, the focus will be on coatings based on transition metal dichalcogenides alloyed with C/N. For high temperature applications the addition of Cr and V will be explored for improving the frictional performance of transition metal nitride hard coatings.

**Behavior of micro-scaled composite TBCs under corrosive attack by CMAS deposits**

Daniel E. Mack (Forschungszentrum Jülich GmbH), O. Guillén, R. Vaßen

Facing the demands on efficiency and operational flexibility of gas turbines, thermal barrier coatings (TBCs) are being developed with considerable progress to allow increased turbine inlet temperatures. Advanced ceramics were identified in terms of the relevant materials properties as e.g. high temperature phase stability, thermal conductivity, thermal expansion, fracture toughness, etc. Based on this data, typical approaches to improve TBC capabilities comprise multi-layer coating designs where individual layers of advanced materials are intended to contribute their best-ranked functionality to withstand the harsh environments.

Since some years, in addition to the extreme thermo-mechanical loads also degradation due to meltable deposits from various sources has become a persistent challenge in the hottest part of the gas turbines. The corrosive attack from CaO-MgO-Al2O3-SiO2 related deposits (CMAS) in most cases imposes major changes on microstructure and chemistry of the outer coating layers which also affect their thermo-mechanical properties. Recent results are reviewed, where micro-scaled composites were effectively able to counter-balance unfavourable effects from corrosive degradation and provide superior performance compared to single phase materials intended for CMAS mitigation.

**Design, validation and thermal testing of durable central receiver coatings for high-temperature concentrated solar power**


Increasing central receiver solar plant’s operation temperature from 550°C to about 800°C would improve the energy conversion efficiency by 15 to 20%. Absorber coatings appropriate for such conditions have to outperform the state-of-the-art pigment paint Pyromark® that has an absorptivity $\alpha > 95\%$ but a high emittance ($\epsilon \approx 80\%$).

The development of environmentally stable solar-selective coatings (SSC) for these temperatures requires new concepts of design and thermal testing. Multilayer SSC based on $[\text{Al}_y\text{Ti}_1-y(\text{OxN}_1-x)]$ absorbers were designed after an extensive microstructural characterization and optical simulations. Based on excellent simulation performance values of $\alpha = 88-94\%$ and $\epsilon_{RT} = 4.8-13.6\%$, complete coating stacks were experimentally validated and tested in vacuum and in air up to temperatures of 800°C [1]. Thermal stability in vacuum up to 800°C is shown by in situ Rutherford backscattering spectrometry (RBS), Raman spectroscopy and spectroscopic ellipsometry (SE) for individual layers as well as for complete SSC. Regarding in-air stability, the most stable SSC fulfilled the standard performance criterion PC ≤ 5% for 300 symmetric, 3 hours long cyclic tests between 300°C and 600°C. Another promising and simpler coating concept to be presented is based on a metal-doped transparent conductive oxide acting as solar-selective transmitter on top of a blackbody.

The onset of the infrared reflectivity is tuneable by variation of the parameters during reactive magnetron sputtering deposition, thus matching the specific temperature requirements during solar applications. Thermal stability up to 800°C in vacuum is demonstrated by in situ RBS and SE.

Financial support by the EU, grant No. 645725, project FRIENDS2, and the HGF via the W3 program (S.G.) is gratefully acknowledged.

1) I. Heras et al., Design of high-temperature solar-selective coatings based on aluminium titanium oxynitrides $\text{Al}_y\text{Ti}_1-y(\text{OxN}_1-x)$.

Characterization of bio-deterministic surfaces fabricated by photochemical machining of Aisi 52100 steel coated with DLC and MoS2 for tribological applications (Poster)

Juan Camilo Sanchez Gonzalez (National University of Colombia), J. L. Endrino, G. Leighton, A. Toro, H. A. Estupiñan

Surface designs and new manufacturing processes for fabrication of deterministic surfaces have been developed to create different textures on several materials to improve the tribological behavior under different contact conditions. Texturing is a good option to improve the friction response of tribological systems. Deterministic surfaces can offer anisotropic friction behavior (as seen, for instance, in snake skin) by changing some geometrical parameters of the features on the surfaces and having control of this performance from the manufacturing process. Furthermore, some coatings are applied to improve the friction response and wear resistance.

In this work, samples of AISI 52100 steel commonly used for rolling bearings were texturized by photochemical machining. The texture elements selected were ellipses with different heights and aspect ratios as a function of the chemical etching time. Also, when the texturing method was carried out, two coatings were applied (DLC and MoS2) to modify the expected tribological response. The coatings were characterized by Raman Spectroscopy and XDR techniques. The results showed that the texturing method is a good option to texture samples of steel with a good lateral resolution. Also, the time elapsed for chemical etching has a marked influence on the height of the protrusions.

Nickel-aluminide based anticorrosion coatings prepared by plasma spray for concentrating solar power applications (Poster)

Sarah Yasir ( Cranfield University), A. I. Aria, E. Guillen Rodriguez, B. Shollock, J. L. Endrino

The use of solar energy for power generation provides an efficient sustainable energy solution. Among a number of technologies developed for power generation using solar energy, concentrating solar power (CSP) is encouraging because of the capability of thermal energy storage that makes it possible for the 24-hour energy production. Although the use of molten salts as heat transfer fluid and thermal storage in CSP has various advantages, they have a major disadvantage as they make the component systems highly susceptible to corrosion. Different approaches have been adopted to suppress hot corrosion including the use of high alloy steels and the use of high purity molten salts; both contribute to a substantial increase in construction and operating costs.

In this study, we investigate the use of protective coatings to enhance the corrosion resistance of the component systems against molten salts at an elevated temperature. Nickel aluminide coatings are deposited using plasma spray system with few different deposition parameters to obtain a variety of stoichiometries and crystalline structures. A low value of porosity is anticipated for corrosion resistance along with good adhesion with substrate, minimum unmelt-particles and adequate lamellar structure for optimum coatings.

Optimized coatings deposited on AISI 347H stainless steel substrate were exposed to a mixture of NaNO3 / KNO3 molten salts at 600°C for up to 1500 hours. The uncoated substrates were also included in the test for comparison. After the test, the samples were characterised by electron microscopy and x-ray diffraction to evaluate their corrosion behaviour. In my presentation, I will show the corrosion test results and discuss the potential of nickel aluminide coatings to suppress hot corrosion by molten salts in an environment that simulates that of concentrating solar power plants.

Investigation of the characteristics of cutting tools coated with mono-layer DLC and multi-layered DLC-WS2 PVD coatings (Poster)

Tomasz Brzezinka (Cranfield University) J. Rao, G. S. Fox-Rabinovich, J.M. Paiva, J. Kohlscheen, S.C. Veldhuis, J.L. Endrino

In machining, the tool life is one of the limiting criteria in the process; therefore, the development of wear-resistant material for the cutting tools is imperative. Tungsten disulfide (WS2) coatings are well established low friction coatings which have dry lubricating properties while DLC is well known from its good tribological properties such as low friction coefficients, high wear resistance, and high hardness.

The purpose of this study was to examine the impact of tribological properties of mono-layer DLC and multi-layered DLC-WS2 coatings on cutting performance of tungsten carbide KC410 milling inserts precoated with TiB2 during aluminium-silicon alloy dry machining.

DLC has been deposited by filtered cathodic vacuum arc (FCVA) technique to ensure dense, hydrogen-free tetrahedral amorphous carbon (ta-C) coating while PVD magnetron sputtering was employed to deposit WS2. A set of thin (~100nm) mono-layer DLC coatings with a different negative bias voltage applied to substrates (from −50 to −200 V) was deposited as well as multi-layered DLC-WS2 coatings (~500nm) with varying number of layers (2 to 24 in total).

A comprehensive investigation of the wear progress of the coated milling inserts was performed by measuring the flank wear during face milling of AlSi alloy on Okuma Cadet Mate CNC Vertical Machining Center at 1888 mm/min feed rate. The main properties of coatings such as thickness, surface morphology, composition and coefficient of friction were measured. The worn inserts were inspected using EDX elemental mapping as well as Alcina 3-D imaging. It has been found that the tool wear rate was greatly reduced by mono-DLC coatings compared to the benchmarked TiB2 coated only insert. Combining DLC with WS2 as a multilayered coating decreased wear rate by three times for the two-layers coating during 30 meters machining length as well as reduced the coefficient of friction down to 0.05.
Tailoring the absorption and emission properties of nanomaterials through their photonic environment


Optical design represents an essential tool to tailor the optical properties of nanomaterials. In this talk, we will discuss different approaches, such as the inclusion of metal1 or dielectric2 nanoparticles, periodically textured surfaces3 or dielectric multilayers4-6 to demonstrate that the optical properties of nanostructures can be tailored by design. Following techniques based on solution processing, we have developed porous dielectric multilayers made of different metal oxide nanoparticles in which a nanometric control over the structural parameters, and thus over their optical response, is attained over large areas. These multilayers are ideal hosts to integrate nanomaterials ranging from metal-nanoparticles, which sustain localized surface plasmons, to dye-doped polystyrene nanoparticles, which fluoresce in the visible part of the spectrum. We have proven that the absorption and emission properties of such nanomaterials can be deterministically controlled through a precise design of their photonic environment. To illustrate this, we will show that tuning the lattice constant of TiO_2/SiO_2 multilayers allows achieving a 3-fold enhancement of light absorption in gold nanoparticles along with 10-fold enhancement of the emission intensity of dye-doped polystyrene nanoparticles. These results are relevant for a wide range of fields of research such as photovoltaics,1-3 sensing4 and light emission,5,6 in which a precise control over the absorption and emission properties are sought for.

5. Fine tuning of the emission properties of nano-emitters in multilayered structures by deterministic control of their local photonic environment. A. Jiménez-Solano. Small, 2015, 11 (23), 2727-2732.
Optimized properties of Al-doped ZnO thin films

M. Mickan, H. Rinnert, M. Stoffel, U. Helmersson, David Horwat
(Institut Jean Lamour-University of Lorraine)

Aluminum-doped zinc oxide (AZO) is a transparent conductor that can be used in thin film form as a transparent electrode of electro-optical devices. For most of actual applications, a large conductivity is required on large surface areas. The rapid growth of transparent electronics and electro-optical devices on flexible supports calls for the development of methods that enable the synthesis of transparent conducting films without thermal assistance while keeping high electrical and optical performances. Magnetron sputtering has emerged as a reference method for the synthesis of AZO films. It is particularly due to its scalability to industrial scale. Unfortunately, AZO films usually produced without thermal assistance using magnetron sputtering tend to a strong inhomogeneity of the electrical properties with a large sensitivity to the process parameters (composition of the gas phase, geometry of the experiment) [1]. It has been shown that the properties of AZO films can be improved using high power Impulse Magnetron Sputtering (HiPIMS) [2]. This method uses short pulses of high electrical power density that produce a highly ionized vapor. This presentation first highlights the interest of HiPIMS to synthesize AZO films of high electronic conductivity on large surface areas and without thermal assistance. Electronic structure measurements using X-ray absorption spectroscopy evidence a correlation between the distribution of the electrical behavior and dopant activation/inactivation [3,4]. A deactivation mechanism, complementary to the well-known compensation of dopants, is proposed in the case of conventional sputtering. A model explaining the minimization of the deactivation amplitude is proposed in case of HiPIMS [5, 6]. In a second part we will show how the electrical properties of AZO films degraded after long term exposure to ambient moisture can be restored by low temperature thermal annealing and how thermal assistance during growth can prevent degradation upon exposure to ambient moisture [7, 8].

References
8. M. Mickan, U. Helmersson, D. Horwat, Effect of substrate temperature on the deposition of Al-Doped ZnO thin films using high power impulse magnetron sputtering. Accepted

The catalytic potential of new MOF-based composite materials

Andrew E. H. Wheatley (University of Cambridge)

Metal and metal oxide nanoparticles show enormous promise in (photo)catalysis. They often offer remarkable properties not available beyond the nanoscale and capable of being enhanced through careful modulation of the composition and structure of individual particles. Although nanomaterials demonstrate many desirable properties, they are difficult to retain or recover in powder form. Apart from having implications for catalyst re-use this can introduce major environmental and health issues. Immobilization offers a solution to these problems. In particular, porous metal-organic frameworks (MOFs) offer the possibility of multifunctional nanocarriers that can support nanomaterials without inhibiting the latter’s accessibility/catalytic activity. Whilst MOFs provide solid, robust, functionalizable structures capable of sifting complex mixtures, they have only recently been prepared in monolithic form without using any binder or high pressures for pelletization. Such new MOFs demonstrate excellent physical properties (higher bulk density and volumetric surface area) relative to MOF powders, and have now been prepared incorporating active nanomaterials. These new composites suggest many industrial applications depending upon the choice of nanoparticle and MOF. The design and synthesis of new systems targeting such applications as photocatalytic dye degradation, PrOx, methane activation, gas storage and chemical weapons agent destruction will be discussed alongside the possibilities for deploying these composites in flow chemistry or as thin films.

References
8. M. Mickan, U. Helmersson, D. Horwat, Effect of substrate temperature on the deposition of Al-Doped ZnO thin films using high power impulse magnetron sputtering. Accepted

Keynote talk

Keynote talk
Over the last decade, the introduction of porosity in nanomaterials has been a topic of increasing interest, and research efforts have been focused on the synthesis and characterization of newly designed porous and nanostructured materials. We have recently presented a new “bottom-up” methodology to produce highly stable amorphous porous silicon coatings with controlled refractive index using magnetron sputtering. Using He and/or Ar as the process gas, silicon coatings with tailored refractive indices, from 3.75 to 4.75 (at 500 nm), can be obtained. The use of Helium as deposition gas produces a singular microstructure with occluded porosity that can be aligned in the magnetron direction. These highly porous coatings incorporate high He amount (up to 40%) inside the closed pores evenly distributed throughout the film thickness.

The proposed approach is a very versatile methodology based on magnetron sputtering technology, and thus extensible to a wide variety of materials and substrates. This methodology enables depositing directly porous coatings over large areas on less expensive substrates like glass or even sensible substrates like polymers increasing enormously the range of possible applications of the porous coatings. Another advantage of magnetron sputtering is the easiness to produce multiple layers alternating porous and dense material just by changing the deposition gas.

In this talk, examples of different materials and possibilities will be presented from highly nanostructured thin films with applications in catalysis, fabrication of Bragg reflectors or self-supported solid He targets for nuclear reactions fabricated by this methodology.

References

Infrared electrochromism of VO2/Ag nanowires electrodes in ionic liquid (Contributed talk)

A. Masmoudi, Nicolas Glandut (University Of Limoges), C. Constantinescu, S. Vedraine, N. Papaionomou

Vanadium dioxide is a so-called ‘smart material’, capable of changing its optical transmittance and electrical conductivity when transformed from a low-temperature monoclinic phase, to a high-temperature rutile one. In addition to temperature, other stimuli can be used, and lead to the same result, without necessarily following the same transition mechanism.

Here, in this presentation, we show that it is possible to initiate a variation of the VO2 optical transmittance, especially in the infrared region, by applying an electrical potential. This phenomenon has been recently observed, and the use of an ionic liquid at the surface of VO2 is of paramount importance.

REACH compliant epoxides used in the synthesis of Fe2O3 aerogel monoliths for target fabrication (Contributed talk)

Paul Jones (Cranfield University), Alberto Valls Arrufat, Magdalena Maria Budziszewska, Clement Lopez, Aymeric Henri Nguyen, Jakub Sitek, Paul Michael Jones, Chris Paul Shaw, Ian Hayes, Gareth Cairns, and Glenn John Thomas Leighton

Metal oxide based aerogels can be used as components in high energy density laser targets as they offer low density, potentially uniform porosity and optically thick components without the problem of the relatively large pore sizes found in pure metal foams. In particular, aerogels have been identified as a future target material in the Orion laser national ignition facility at Aldermaston. A wide variety of metal oxide aerogels are required for these campaigns and therefore a versatile synthesis route is needed. Present methods for the synthesis of these materials require the use of the epoxide propylene oxide in the creation of the initial sol-gel formation, the first step in the synthesis of the Aerogel. Under EU REACH legislation this particular epoxide has been recognized as a substances of very high concern.
Interconnections between electronic structure and optical properties of multilayer nanolaminate TiAlN/Ag and Al2O3/Ag coatings (Contributed talk)

Dmitry Wainstein (I.P. Bardin Central Research Institute for Ferrous Metallurgy), A. Kovalev, V. Vakhrushev, R. Gago, J. L. Endrino

The multilayer nanolaminate TiAlN/Ag and Al2O3/Ag MIM-type coatings with different thicknesses of individual layers varied from several to several hundred nanometers were fabricated by DC–magnetron sputtering. Their optical transmittance and reflectance spectra were measured in the photons energies range from 1 to 5 eV, or from 1240 to 248 nm. The optical spectra were non-monotonous, and their transmission and reflection bands were strongly dependent on the coatings’ architecture. The electronic structure of the coatings was studied by XPS, AES, scanning Auger and plasmon microscopy, HREELS methods. The details of electronic structure transformations at metal and dielectric layers thinning are discussed. Electromagnetic waves propagation in MIMs is controlled by plasmon excitations. The spectroscopy of plasmon losses and plasmon microscopy allowed us to measure the plasmons characteristic energies and their surface distribution. The energies of plasmons peaks and their locations are strongly depending on Ag layers thickness in the MIM nanocomposite. The cross-correlations between geometrical parameters of the coatings, transmissions and reflection bands on optical spectra, parameters of electronic structure were found by statistical analysis. Particularly, the blue side of transmittance band is controlled by plasmons while the red one position is determined by the dielectric band gap. These coatings can find applications in design optical filters and photovoltaic energy conversion devices, etc.

Acknowledgements.
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Tuning of catalytic activity by thermoelectric materials (Contributed talk)

A. Achour, K. Chen, M. J. Reece, and Zhaorong Huang (Cranfield University)

An innovative use of a thermoelectric material (BiCuSeO) as a support and promoter of catalysis for CO2 hydrogenation is reported here. It is proposed that the capability of thermoelectric materials to shift the Fermi-level and work function of a catalyst lead to an exponential increase of catalytic activity for catalyst particles deposited on its surface. Experimental results show that the CO2 conversion and CO selectivity are increased significantly by a thermoelectric Seebeck voltage. This suggests that the thermoelectric effect can not only increase the reaction rate, but also change chemical equilibrium, which leads to the change of thermodynamic equilibrium for the conversion of CO2 in its hydrogenation reactions. It is also shown that this thermoelectric promotion of catalysis enables BiCuSeO oxide itself to have a high catalytic activity for CO2 hydrogenation. The generic nature of the mechanism suggests the possibility that many catalytic chemical reactions can be tuned in-situ to achieve much higher reaction rates, or at lower temperatures, or have better desired selectivity through changing the backside temperature of the thermoelectric support.

The role of coatings in addressing a more cost-effective PEM electrolyser (Contributed talk)


Although hydrogen can be obtained by different ways, water electrolysis is probably one of the most environmentally friendly strategies, mainly when renewable energy sources are used. In recent years, PEM (Proton Exchange Membrane) water electrolysis (PEMWE) has become one of the most important processes of all available electrolysis technologies. The bipolar plates (BPs) are one of the most important elements for PEM electrolyser because it provide mechanical support, distribute the water inside the cell and carry the generated gases to the outlet, ensuring the efficiency of the process. From a cost-efficient point of view, materials for BPs represents a big challenge. For those reasons one of the main objectives of the Spanish project ENHIGMA are:

• Select the most suitable materials for PEM electrolysis bipolar plates, through a study of different low cost metals (mainly aluminum and steels) and protective coatings through accelerated life tests.

• Determine the most suitable coating methods for the PEM electrolysis operating conditions

In this presentation it is shows comparative results for different coated systems combinations by using PVD technologies including HiPIMS variants.
New developments in solid polymer electrolytes for electrochromic smart windows (Contributed talk)

Quentin Lonne (Cranfield University), D. Bhattacharyya, K. Lawson, T. Walker, S. Pamidi, M. Peyratout, J.L. Endrino

Using a solid polymer electrolyte (SPE) in an electrochromic (EC) window instead of a liquid or a gel offers several advantages, such as being easier to assemble with the electrodes and avoiding all chemical hazards due to a possible leakage. Moreover, polymers are light-weight, easy to process, chemically stable and cheap.

In this work, the multi-layer architecture of an EC window involving two ITO transparent conductors and two electrodes (WO3 and V2O5) separated by a novel SPE was studied. The SPE was a composite comprising a polymeric matrix, plasticisers, a lithium salt and TiO2 nanoparticles. Its formulation, synthesis parameters and coating process were studied in order to optimise the EC window assembly. Moreover, for each layer, a compromise was found between thickness and electro-optic properties to optimise the performance of the stacked structure. SEM, EDS, XRD and XPS were used to study the structure, microstructure and composition of the various layers. UV-Vis-NIR spectroscopy, 4-point probe and cyclic voltammetry were used to characterise the electro-optic properties. A proof of concept was obtained at laboratory scale on a 10 x 10 cm² EC window: a reversible colour switching, with a contrast ratio 3:1, was achieved in 2 minutes, using a bias of 2 V. Moreover, it was proved that those windows can be easily powered up, using a commercial USB solar charger (5 V, 1 A) classically employed to charge a mobile phone.

Control of the compensating defects in doped ZnO nanocrystalline thin films (Poster)

Shuvraja Ghosh (Indian Association for the Cultivation of Science), D. Basak

Concentration of the dopant impurity in degenerate metal oxide semiconductors can alter the free electron density over one order of magnitude from a few 1019 cm⁻³ to more than 1021 cm⁻³. However, the activation of the dopant is reduced when the doping level reaches degeneracy. Typical values of the dopant activation usually are known to range from 10% to 50%. The efficiency of dopant is limited because of the formation of the point defect related compensating centers. However, formation of compensating defects depends on the growth condition, nature of dopant and post-growth annealing treatments of ZnO. It is thus appealing to study the impact of dopant type on the electrical, optical and plasmonic properties of doped ZnO thin films. In this work, 1 wt% Al, In and Sn doped nanocrystalline ZnO films have been deposited using RF magnetron sputtering system under exactly similar growth conditions and subjected to post-growth annealing treatments in certain ambience. The structural, electrical, optical and plasmonic properties of the films have been investigated using x-ray diffractometry, Hall effect and UV-Vis-NIR transmittance measurements. We report a systematic experimental results showing that both trivalent as well as tetravalent dopants in ZnO nanocrystalline films have been activated by a post-growth annealing treatment in certain ambience and correspondingly, the resulting free carrier density has been increased changing the plasmonic frequencies. Interestingly, an enhanced mobility due to annealing strengthen the possible relation between this increase in free carrier density as well as mobility and the reduction of compensating acceptor defect complexes such as M³⁺Zn⁻VZn or M⁴⁺Zn⁻VZn. Detailed analyses of the electrical and optical results in the context of the influence of dopant type and annealing ambience on the improvement in the dopant activation will be presented.

(Al, In) co-doped nanocrystalline ZnO transparent conducting thin films for rigid and flexible substrates (Poster)

Plasma printing oxide nanoparticles by in-situ tailoring of oxidation state (Contributed talk)

Avishek Dey (The Open University), S. Krishnamurthy, R. P. Gandhiraman

Printing of nanostructured films with tailored oxidation state and electronic structure can have far reaching applications in several areas including printable electronics, optoelectronics, solar cells, catalytic conversion and others. Widely used inkjet/aerosol/screen printing techniques require pre and post processing for enhanced adhesion and tailoring the chemical state of the materials. The use of nanocolloidal ink in general results in oxidation of the nanoparticles. Controlled deposition of functional materials with tailored physical, chemical and electronic characteristics is needed for advanced manufacturing of flexible electronic devices. Herein we demonstrate the plasma jet printing technology’s ability to print in-situ tailor the electronic properties and surface morphology of copper oxide and graphene oxide nanoparticles. Our X-Ray absorption studies reveal that, both the oxide nanoparticles can be in-situ reduced using our direct write plasma printing technology as shown the figure. Here the printing was carried out using nanocolloidal inks of CuO and graphene oxide. Additionally, a highly conductive film can be obtained with average resistance value of 7 Ohms. The temperature and the reactivity of plasma can be varied by changing the gas mixtures, flow rates of the gasses and applied voltage. From 25C to 200C. A varied morphology of thin films can also be obtained with different gas mixtures. This system also promotes adhesion to substrate, uniformity in deposition and formation of densely packed nanoparticle structure with high throughput. The applications extend to printing on 3D objects as well. We believe this technology will prove to be revolutionary in the field of flexible printed electronics.

A proof of concept was obtained at laboratory scale on a 10 x 10 cm² EC window: a reversible colour switching, with a contrast ratio 3:1, was achieved in 2 minutes, using a bias of 2 V. Moreover, it was proved that those windows can be easily powered up, using a commercial USB solar charger (5 V, 1 A) classically employed to charge a mobile phone.
Trivalent cation doped and co-doped ZnO is found to have the potential to substitute the expensive and diminutive supply of Sn-doped In2O3 (ITO) due to comparable conductivity, optical transparency along with their low-cost and earth abundance nature. There are only a few numbers of reports on (Al, In) co-doped ZnO (AlZO), although, plentiful of studies on each Al and In-doped ZnO are available. Here, AlZO nano-crystalline thin films with a thickness 250-1100 nm are deposited at 500°C on glass substrates in pure Ar environment using Radio Frequency (RF) magnetron sputtering from a 1 at.% Al+ 1 at.% In co-doped in-house made ZnO target and the structural, optical transmission and electrical properties of the films are investigated. The film thickness is increased by increasing the deposition time and a strong c-axis (002) oriented growth of the hexagonal wurtzite phase ZnO is observed. The electrical resistivity consistently decreases with an increase of thickness and the lowest resistivity value of 9×10⁻⁴ Ω-cm (Rs=9.6 Ω/sq) is obtained for a 1100 nm thick film with an electron concentration and mobility values of 6.6×10²⁰ cm⁻³ and 10.5 cm²/Vs respectively.

All the films show very high optical transmission value >90% in 400-800 nm wavelength range. The highest Figure of merit (FOM) value of 3.4×10⁻² Ω⁻¹ is obtained for a 1100 nm thick film which is little less than the of commercial ITO or F-doped SnO₂ (5-8×10⁻² Ω⁻¹). The carrier relaxation time and mean free path of the electrons are found to increase gradually with an increase of the film thickness implying less number of impurity scattering. The 1100 nm thick film is also deposited on glass substrate at room temperature (RT) that shows a resistivity value of 1.2×10⁻² Ω-cm (Rs=118 Ω/sq) with an electron concentration and mobility values of 2×10²⁰ cm⁻³ and 2.7 cm²/Vs respectively. The lower mobility value may be due to presence of impurity defects and smaller grain size which may be improved by annealing below 150°C. This report shows that AlZO thin films derived in this study using a very small amount of dopants in contrast to earlier studies can be used as transparent conducting films on rigid as well as flexible (at RT) substrates implementing opto-electronic device applications.
Amorphous carbon, diamond and silicon are materials ubiquitously used in applications ranging from bearings to microsystem devices. Here, we describe how molecular dynamics calculations can be used to understand tribological properties of these materials: Their behavior when contacted, rubbed and worn down. Wear can be related to shear-induced amorphization in diamond-cubic Silicon and Carbon. The process is driven by shear rate with distinct atomic-scale signatures for the two materials: Upon amorphization, silicon becomes denser while carbon expands. As a result, amorphization is enhanced in silicon but suppressed in carbon as pressure increases. Calculations for tetrahedral amorphous carbon show a similar process: a dense, hard amorphous phase transforms into a softer one upon shear. Finally, we explore the suggestion of coating surfaces with the thinnest possible coating, graphene, to make them more wear resistant. Our calculations reveal that graphene reduces friction of the contacting surfaces, but that breaking and wearing graphene coatings is possible. Applications seeking to use graphene as friction-reducing coatings must therefore seek possibilities to regrow the coating during use.

Mo2BC thin films - a material system combining hardness and ductility?

R. Soler, S. Gleich, H. Fager, J-O Achenbach, C. Kirchlechner, J. M. Schneider, C. Scheu, Gerhard Dehm (Max-Planck-Institut für Eisenforschung)

Ab initio calculations predict that Mo2BC is a material with a high Young’s modulus of up to 470 GPa while simultaneously the ratio of bulk modulus to shear modulus indicates the possibility for moderate ductility [1]. Thus, from the atomic structure and bonding mechanisms Mo2BC has the potential to combine high stiffness and ductility. In this keynote talk the evolution of the nanostructure of Mo2BC coatings as a function of the growth temperature is presented and linked to the corresponding mechanical properties. X-ray diffraction studies and advanced transmission electron microscopy investigations [2] as well as nanoindentation experiments, fragmentation tensile testing and miniaturized beam bending for fracture toughness evaluations are employed to provide insights into the nanostructure dependent mechanical performance. The nanostructures under investigation range from partially amorphous to fully crystalline and provide interesting changes in their mechanical response, which will be discussed in the presentation.


Multiscale modelling of thermally sprayed particle impact and coating formation (Contributed talk)

Jordan Davidson (Robert Gordon University), A. Muir, C. Pegg, A. White, S. Z. Islam, N. H. Faisal

This work for the first time presents a multiscale modelling approach to study various thermomechanical phenomena during thermal spray of aluminium-to-aluminium and titanium-to-titanium particle-substrate interactions (e.g. Fig. 1 (a)). To evaluate importance of processing parameters during normal particle impact under high velocity oxy-fuel (HVOF) thermal spray style-conditions, topography was studied using two flattening metrics. It indicates that for specified velocity and temperature intervals, velocity is a critical parameter for splat formation of particles when analysed via flattening diameter ratio and flattening aspect ratio models. Deviating the angles from 90° to 45° was observed to produce greater flattening, due to increased momentum transfer along the substrate surface. A von Mises atomic shear strain algorithm was used to compute the shear strain and it was observed that the maximum strain is concentrated along the surface and centrally underneath the splat. Finally, phase transformations during multi-particle spray impact were studied for various impact velocities. Further work is focused on cold spray-style conditions for titanium-to-titanium coatings and aims to extend the atomistic scale simulations of lattice disorder, atomic shear strains and topography to mesoscale explicit dynamics and computational fluid dynamics simulations, studying high strain-rate deformation, residual stresses and cooling rates (e.g. Fig. 1 (b) – (d)). This simulation approach could help define spray parameters for tailored coating and enhanced tribological properties.

Fig. 1.

a. Molecular dynamics (MD) simulation using LAMMPS during HVOF thermal spray coating formation: (i) model snapshots while spraying various powder size and multiple-particle spraying and (ii) cross-section of simulations, highlighting phase transformations during spray impact using common neighbourhood analysis (CNA) algorithm,

b. Explicit dynamics simulation showing stress distribution during impact,

c. MD simulation for cold spray clustered particle impact showing shear stress,

d. Thermal simulation showing cooling and temperature distribution post-impact.
Influence of multiply charged ions on the adhesive strength of coatings at HV pulsed negative bias on a substrate (Contributed talk)

P. A. Tsygankov (Universidad Industrial de Santander), F.F. Parada Becerra, R. I. Chelmodeev, E. D. V. Niño, V. D. Dugar-Zhabon

The problem of obtaining the proper adhesive strength is solved every time when applying the coating. The main role in its provision is played by the initial stage of growth of a thin film - atoms interaction with surface and the formation of an interface with a substrate. Such sources as a vacuum arc, PLD, HIPIMS are generators of ionized vapour and therefore it is possible to regulate the energy parameters of the flow coming to the substrate by the bias potential on it. The principal issue of the quality of the coating being formed is the initial energy composition and the degree of ionization of the vapors.

In this paper, the results of measuring of the energy composition and the degree of ionization of the flows which are generated by a vacuum-arc evaporator are presented.

Diagnosis is carried out by a multi-electrode grid retarding potential probe. It is noted that a high proportion of two, three and four-charged ions in the flow with a total degree of ionization of up to 20% is detected. To the substrate with a growing film, a pulsed negative potential with a voltage of up to 20 kV is applied, which ensures ion implantation of the ionized atoms of target. The results of studying the surface of silicon wafer with a deposited titanium film using this technology are presented. Comparative tests of the adhesive strength of the obtained films on the substrate of HSS are carried out.

Nanomechanical characterisation of DLC coating systems (Contributed talk)

Sam McMaster (University of Leeds), T. Liskiewicz, A. Neville, B. Beake

Diamond-like carbon is a metastable form of amorphous carbon with varying ratios of sp2/sp3 bonding. These coatings possess attractive mechanical, optical, chemical and tribological properties [1]. DLC coatings are becoming increasingly popular in the automotive and aerospace industry due to their high hardness, resistance to wear and low friction coefficient [2]; well characterised coatings will allow for them to be tailored to applications where these properties are favourable [3]. They can suffer from poor adhesion at high loads [4,5]. DLC often displays high levels of intrinsic stress due to deposition by sputtering [5].

The DLC coating systems have been varied in this study by changing the composition (a-C:H, Si-doped and W-doped), coating thickness (1 micron and 3 microns), substrate material (316L stainless steel and hardened M2 Tool Steel) and substrate roughness (0.01 microns and 0.08 microns). All DLC coatings in this study have a gradient interlayer present (Cr/W-C/W-C:H).

Mechanical properties have been measured by nanoindentation using a partial loading technique. Variations in hardness and elastic modulus have been mapped through the multilayer coating structure. Macro-scale and nano-scale coating adhesion has been tested though scratch testing at both macro and nano-scales respectively. Impact and erosion testing has been used as part of a comparative study equating the energies dispersed in the substrate surface and characterising the wear scars produced by each method. DLC impact fatigue resistance requires improvement for more demanding applications [6]. DLC failure mechanisms have also been investigated using backscatter SEM due to its sensitivity to crack detection.

References:
Developments in nano-impact testing on optical thin films
(Contributed talk)

Luis Isern Arrom (Cranfield University), D. Bhattacharyya, K. Lawson, J. L. Endino and B. Beake

The resistance of thin films to single impact and fatigue damage can be characterised by nano-impact testing. Previously, this method had been optimised for the analysis of high-resistant coatings, often requiring sharp indenter geometries to produce coating fracture. However, the method has not been optimised to test brittle coatings designed for optical applications. This paper aims to optimise the testing and analysis of the nano-impact method to evaluate the mechanical properties of optical coatings. The response of two different indenter geometries, sphero-conical and cube-corner, has been tested on two thin ZrO2 films of thickness 270 and 790 nm.

Determination of the mechanical properties of coatings and thin films using nanoindentation and analytical model
(Contributed talk)

Andrei S. Vasiliev (Don State Technical University) E.V. Sadyrin, B.I. Mitrin, L.I. Krenev, S.M. Aizikovich

A statistical analysis of the curves of indenter position depth over time was performed and identified a number of parameters to describe them. Inspection and damage quantification of the individual failure locations show that the blunt spherico-conical geometry is more suited for nano-impact testing of optical coatings, as a load threshold can be established between no coating delamination and 100% coating delamination. On the contrary, the sharp cube-corner geometry is not suited for nano-impact testing as it produces 100% coating delamination for all loads and repetitions, thus comparison between samples is not possible.

“Multiscale modelling of Meso-Bio-Nano (MBN) systems with MBN Explorer and MBN Studio: thermally induced morphological transitions of nanostructures

Andrey V. Solov’yov (MBN Research Centre)

MesoBioNano (MBN) Explorer [1] is a multi-purpose software package for advanced multiscale simulations of complex molecular structure and dynamics developed by MBN Research Center. It has many unique features and a wide range of applications in Physics, Chemistry, Biology, Materials Science, and Industries. A broad variety of algorithms and interatomic potentials implemented in the program allows simulations of structure and dynamics of a very broad range of systems with the sizes from the atomic up to the mesoscopic scales, see [2] and references therein. MesoBioNano (MBN) Studio is a special multi-task software toolkit with graphical user interface developed for MBN Explorer [3].

The talk will give an overview of the main features of the packages and will highlight a number of recent case studies devoted to thermally induced morphological transitions [4, 5] of nanostructures (nanowires, nanofoils, etc) investigated by means of MBN Explorer and MBN Studio [1, 2, 3]. Attention will also be devoted to the modelling of tribological transformations of materials and phase transitions studied by means of novel algorithms and multiscale simulation techniques [5, 6].

References

** Keynote talk
Large area synthesis and properties of diamond - silicon carbide composite thin films for enhanced protective coatings

Andrew Taylor (Institute of Physics CAS), R. Ctvrtlik, L. Klimša, Z. Remeš, M. Vronka and V. Mortet

This is due to the fact that microwaves are delivered into the growth chamber in a linear form by four pairs of antennas enclosed in quartz envelopes enabling a surface wave to be sustained linearly along the antennas, therefore the size of the microwave discharge (and hence plasma discharge) is not limited by the wavelength of the inputted microwave. Layers deposited on various substrates (silicon, glass, fused silica and titanium) with sizes up to 6 inch in diameter have been characterized by scanning electron microscopy, transmission electron microscopy, Raman spectroscopy and Fourier transform infrared transmission. Mechanical and tribological properties were explored via nanoindentation and micro-scratch tests performed with diamond indenters. At optimal deposition conditions, deposited layers exhibit superior scratch resistivity compared to standard diamond coatings. Chemical and structural characterization of these layers confirm the presence of not only NCD but also a silicon carbide (SiC) phase. The enhanced scratch resistivity of these new nanocomposite NCD/SiC layers may be attributed to the possible improved stress redistribution and accommodation mechanics coming from the nanocomposite structure combining SiC and NCD. Finally, TEM together with EDX and EELS have been used to closely investigate the nature of the deposited nanocomposite NCD/SiC layers in order to understand the origin of the mechanical properties of these new composite layer.

Optical diagnostic and formation of thin films of titanium through the electric arc discharge

F. F. Parada Becerra (Universidad Industrial de Santander), P. A. Tsygankov, A. Plata Gómez, C. Mendoza Luna, C. Chacón, E. D. V. Niño, V. Dugar-Zhabon

The vacuum cathodic arc systems are a font of high performance that generating metallic vapor of ionizes species and it is technique widely used for fabricating coating of thin films. In this research work is presented the optical spectrum obtained, by means of HR 4000 spectrometer, during the evaporation of a titanium cathode where through of the ions of grade 1 and 2, is achieve obtaining a value plasma temperature approximately of 8800K, and on the surfaces, is evidence the presence of micro drops during the coating deposition which is generated by the behaviour of the spot cathodic. Besides is found that deposition rate of titanium thin film is range between 2nm/s to 10nm/s dependence of current provided to the electric arc.

A simulation testbed to study the pseudoelastic behaviour of Cu-Zr binary alloys during a tensile test using molecular dynamics

Fabian Duarte Martinez (Cranfield University), S. Goel

New frontiers in the field of biomaterials are becoming the core in advancing our understanding of the biomedical sciences required for efficient healthcare. One of the main materials leading the field of biomedical discipline are shape memory alloys (SMA).

A wide range of alloys such as NiTi and AgCd have shown shape memory behaviour; however, only those that can recover from a large amount of strain have been of high commercial interest in biomaterials. One of the material properties that allow a large amount of strain recovery is known as pseudoelasticity and involves a reversible austenite to martensite phase transformation during its deformation process. The study of pseudoelasticity is key in furthering our fundamental understanding of shape memory alloys.

Copper-Zirconium binary alloys is considered as a potential candidate in biomedical commercial applications due to their pseudoelastic behaviour and due to their mechanical and biocompatible properties. For this reason, the aim of this work was to study the stress-induced structural transformations in the stable alloy composition of Cu50Zr50 and CuZr2 during nanoscale tensile and shear tests. This work provides a fresh understanding on the sites of martensitic nucleation during its deformation process, maximum recoverable strain, changes in local microstructure and the reverse phase transformation. These deliverables will aid in developing a fundamental understanding of the pseudoelastic behaviour of Cu-Zr binary alloy and elevate the applications of Zr based BMG’s and binary alloys in the field of biomaterials.
Self-powered high performance photodetectors for ultraviolet and visible lights

Durga Basak (Indian Association for the Cultivation of Science)

Photo-detectors are used in a wide range of applications from everyday consumer electronics to sophisticated applications like environmental monitoring, binary switches, memory storage, space research and optical communication. In the present scenario of energy crisis, self-powered photo-detectors that need no external voltage to detect light have recently attracted considerable attention among researchers and technologists. A multispectral photosensitivity feature of a self-powered photodetector provides an additional powerful quality. This talk will present an account of experimental results describing an unprecedented high performance dual wavelength self-powered ZnO@CdS/PEDOT:PSS core–shell nanorods’ array film photodetector synthesized by a simple aqueous chemical method. The detector shows a very sharp and prominent dual wavelength photosensitivity of two orders of higher magnitude than that of pristine ZnO with a temporal response faster than 20 ms. On the other hand, organic semiconductors possess the advantage of solution process-ability with high flexibility and functional tunability but has the drawbacks of low carrier mobility. Hence, organic-inorganic hybrid junctions have the unique advantage that integrates functional advantage of each component while overcoming their respective drawbacks. The talk will have a component that will provide an idea of designing a high quality organic-inorganic hybrid p-n junction self-powered photo-diode by simply spin coating spiro-OMeTAD over solution processed Sb2S3 thin film as primary light absorbing materials. The device shows a photo-responsivity over 0.3 A/W and a value of 102 A/W at operating voltages of 0 V and ±1 V respectively at an incident light of wavelength 470 nm and intensity 0.45 mW/cm2. It also shows a fast photoresponse time of <25 ms. More interestingly our device is capable of detecting visible light at very low intensity of the order of 1 μW/cm2 with a photo-to-dark current ratio more than 8. The values of responsivity, short circuit current and open circuit voltage of the photodetector can be improved significantly using a thin layer of TiO2 hole-blocking layer.

**Keynote talk

Topic Lead: S. Krishnamurthy and V. K. Thakur

This topic will include all the presentations more centred in properties and advanced characterization of films: either from a performance point of view or fundamental analysis (microstructure, crystallography, composition, spectroscopies, etc.).
Towards functional copper halide thin films for optoelectronics

Aidan Cowley (European Space Agency)

The Cuprous Halides (CuCl, CuBr, Cul) have received considerable interest over the years, primarily for their interesting excitonic properties, but also due to their potential practical applications towards functional optoelectronics. Owing to their interesting band structure and excitonic features, theoretical and spectroscopic studies on CuCl and CuBr have been performed giving a valuable insight into the complexities of this group of I-VII semiconductor materials.

For optoelectronics, CuCl in particular has been extensively studied, initially as a potential electro-optic modulator. Initially this drove the need for the growth of high quality CuCl single crystals, which were achieved by various bulk crystal growth techniques applied over the years: growth by the traveling heater method, Czochralski growth, vertical Bridgeman and top seeded solution growth as well as gel growth. Following on from these early single crystal growths, practical demonstration of thin film deposition has been achieved for CuHa materials, targeting functional UV/Blue light emission applications. CuCl in particular, due to its close lattice match with Si (< 0.4% mismatch), has been the target of much of this development. Deposition on Si via magnetron sputtering as well as vacuum evaporation have resulted in polycrystalline γ-CuCl on Si with notably strong UV excitonic emission. CuCl on Si and GaAs has also been demonstrated via MBE wherein single crystal CuCl was successfully grown.

Prototype electroluminescent devices (ELD) based on γ-CuCl and γ-CuBr active layers have been produced, with reported light emission at 380 nm and 387 nm for CuCl devices. Work on developing epitaxially matched γ-CuBr/γ-CuCl on SiGaAs substrates respectively via Liquid Phase Epitaxy (LPE), as well as nanostructured KCl/KBr doped CuCl/CuBr thin films, show that there still remain many novel thin film methodologies to be explored with this material.

Fracture toughness and yield strength of hard, thin coatings at high temperatures using in situ micromechanical testing

Jeff Wheeler (Swiss Federal Institute of Technology)

In order to evaluate the performance of hard coating materials used in aggressive cutting operations such as end milling or high speed machining, it is necessary to investigate the materials properties at the high temperatures generated in service. However, the small length scale of the coatings limits the utility of conventional high temperature techniques. The recent extension of nanoscale techniques such as nanoindentation and nanoimpact to elevated temperatures has recently made this a possibility, and recent work using these techniques has demonstrated the utility of these techniques [1, 2].

The development of vacuum and in situ SEM techniques for high temperature nanoindentation [3, 4] has recently also allowed measurement at temperatures where oxidation of the indenter or sample may be problematic. Furthermore, in situ testing allows direct visualisation of the deformation mechanism changes at elevated temperature, so that buckling, fracture, or plastic deformation can be immediately observed. The development of micropillar compression techniques and other new micromechanical fracture testing geometries has expanded this envelope even further to allow direct measurement of yield strength [5] and fracture toughness [6] of hard coatings at elevated temperatures.

Micropillar compression at high temperature offers several attractive advantages over hardness testing. Indentation is highly dependent on tip geometry for accurate measurements, and indenter geometry variation due to erosion by oxidation and abrasive wear or plastic deformation of the tip is a serious concern at high temperatures. Using a flat punch indenter avoids any such geometric variation. Also, the micropillar geometry offers a uniaxial stress state in contrast to the triaxial stress state of indentation. This provides a direct measurement of the yield stress; rather than an indirect method such as indentation where the relationship between the yield stress and hardness is complex.

Fracture toughness at the micro-scale can be evaluated using a number of geometries [7]: pillar splitting, and notched single and double cantilever beams. The effectiveness of these geometries and the effects of the ion beam used to machine these structures have both been evaluated. Procedures for successfully measuring the yield strength and fracture toughness of hard coatings at elevated temperature have now been established, and these are demonstrated on a range of CrN-based hard coatings.

References


“Keynote talk
Integration of nanotechnology and advanced manufacturing processes presents an attractive route to produce devices for adaptive biomedical device technologies. However, tailoring biological, physical, and chemical properties often leads to complex processing steps and therefore to high manufacturing cost impeding future scalability. Herein, novel laser-based approaches are introduced to manufacture low-cost biocompatible polymer substrates functionalized with ultrapure nanoparticles. Laser direct writing was performed to create micron-sized patterns on 188 m cyclic olefin polymer substrates using a picosecond pulsed 1064 nm Nd:YAG laser. The pulsed laser ablation in liquids (PLAL) process was exploited in this work to prepare colloidal solutions of ultrapure nanoparticles to impart bio-functionality onto laser patterned surfaces. Combining the laser patterns and their modification with PLAL nanoparticles resulted in a functional and biocompatible substrate for biosensing applications. Our in vitro cell viability studies using a model cell line (human skin keratinocyte, HaCaT) suggest that these nanoparticles immobilize on the surfaces function as a biomimetic platform with the ability to interact with different biological entities (e.g., DNA, antibodies, etc.)

Characterization by TEM and Raman of high-density low-defect-concentration zinc oxide nanowires grown by afterglow

ZnO has gained substantial interest in different research fields due to its attractive properties and its wide range of applications. For instance, ZnO is a promising material in photocatalysis applications for water treatment and disinfection [1]. Furthermore, photocatalysis efficiency would increase by reaching quantum confinement sizes. The observation of such a phenomenon in 1D structures requires highly crystalline nanostructures with low dimensions. In this study, the synthesis of ultrathin single-crystalline zinc oxide nanostructures was achieved by treating, in a flowing microwave plasma oxidation process, zinc films coated beforehand by a sputtered thin buffer layer of copper. The morphology of synthesized nanostructures can be controlled by the experimental parameters: treatment duration, furnace temperature, oxygen concentration, pressure. Ultrathin nanowires, nanoribbons and nanocombs are successfully synthesized for very short treatments, typically less than 5 minutes. For example in the case of nanowires, an average diameter of 6 nm (for a mean length of 750 nm) can be reached with a fairly high surface number density. The oxidized samples are characterized by means of SEM, XRD, SIMS, HRTEM and EDX techniques. Nanostructures are only composed of ZnO without copper particles inside or at the end of nanowires or nanoribbons. Temperature-dependent photoluminescence measurements confirm that ZnO nanowires are of high crystalline quality and thin enough to produce quantum confinement [2].

References

Characterisation of an antimicrobial silver nanocomposite coating on orthopaedic grade cobalt chromium alloy

A hard-wearing silver nanocomposite coating has shown antimicrobial effect and can be a good barrier for the release of cobalt ions from the conventional orthopaedic grade cobalt chromium molybdenum (CoCr) alloy. Therefore, such a coating family may lower the risk of post-operation infection which accounts for up to 4% of all procedures. The controlled release of silver and the overall tribological friction/wear performances are critical for the safety and longevity of orthopaedic implant research. This is a study focusing on the optimisation of electron beam physical vapour deposition (EBPVD) coatings deposited on the MatOrtho Ltd, UK, medial rotation knee (MRKT) with different levels of silver content in the coating structure. Four levels of silver were tested in this novel ‘smart’ coating using physical and chemical surface characterisation and various clinically relevant in vitro models. The application of ‘smart’ chromium nitride silver coatings which have improved tribological properties and antimicrobial action hold a lot of promise for orthopaedics in the future.
Synthesis and deposition of polyfluoroacrylate film on anodised aluminium surface for hydrophobic and icephobic applications

(Contributed talk)

Tamal Barman (The University of Nottingham), H Chen, J Liu, W Zhao, C Peng, S Li, K So Choi and X Hou

Ice formation and accumulation causes great concerns for many industries including infrastructure, transport, powerlines, wind turbines and offshore oil platforms etc. as it reduces the operational efficiency and may also lead to disaster. Applying icephobic coatings on the substrate surface is believed to be a promising solution as it can minimize the accumulation of ice and prompt the removal of the ice from the surface. Many polymeric coating have been proposed for icephobic application but it is still a challenge to identify a suitable coating system to provide satisfactory ice protection. In the present work, a modified polyfluoroacrylate (PFA) has been synthesized with a fluorine content of 33 weight % and deposited on anodised aluminium surface which can substantially reduce the surface energy and improve the durability. The anodised surface of Al alloy provides the desirable surface roughness with average Ra value of 1.77 µm and offers better adhesion with the PFA film.

Superhydrophobicity with water contact angle (WCA) of 162.5° and contact angle hysteresis (CAH) of 8° can be achieved on the deposited film. De-icing tests are performed at environmental temperature of -5 °C by electrical heating method. And 20% decrease in energy consumption during the de-icing process is observed with the use of synthesized PFA film. It is suggested that the modified PFA coated anodised Al surface demonstrates superhydrophobicity and better energy efficiency in ice removal, showing good potential for icephobic applications.

Acknowledgement

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Characteristics of bloch surface wave resonances in total internal ellipsometry spectra: effect of adsorbed nanolayer on selected dielectric multilayers

(Contributed talk)

Karl Fleury-Frenette (Université de Liège)

Total internal reflection ellipsometry (TIRE) provides enhanced sensitivity compared to standard ellipsometry in monitoring surface adsorption or desorption. When combined to surface resonance effects excited in total internal reflection conditions, this sensitivity can even be increased by orders of magnitude. Here we present the amplitude and phase spectral characteristics of Bloch surface wave resonances (BWSR) in TiO2/SiO2, Nb2O5/SiO2 and Ta205/Si02 multilayers in presence of an adsorbed nanolayer. Multilayers with different total optical thickness each optimized to support Bloch surface wave resonances are investigated. The resonance height, full width at half maximum and shift in wavelength are used to extract a figure of merit for each considered configurations as function of the adlayer thickness.

High temperature nanoindentation up to 800°C for characterizing high temperature properties of coatings

(Contributed talk)

Nicholas X. Randall (Anton Paar), M. Conte, G. Mohanty, J. Schwiedrzik, J. Michler

One of the primary motivations for development of instrumented indentation was to measure the mechanical properties of thin films. Characterization of thin film mechanical properties as a function of temperature is of immense industrial and scientific interest. The major bottlenecks in variable temperature measurements have been thermal drift, signal stability (noise) and oxidation of/condensation on the surfaces. Thermal drift is a measurement artifact that arises due to thermal expansion/contraction of indenter tip and loading column. This gets superimposed on the mechanical behavior data precluding accurate extraction of mechanical properties of the sample at elevated temperatures. Vacuum is essential to prevent sample/tip oxidation at elevated temperatures.

In this talk, the design and development of a novel nanoindentation system that can perform reliable load-displacement measurements over a wide temperature range (from -150 to 800 °C) will be presented emphasizing the procedures and techniques for carrying out accurate nanomechanical measurements. This system is based on the Ultra Nanoindentation Tester (UNHT) that utilizes an active surface referencing technique comprising of two independent axes, one for surface referencing and another for indentation. The differential depth measurement technology results in negligible compliance of the system and very low thermal drift rates at high temperatures. The sample, indenter and reference tip are heated/cooled separately and the surface temperatures matched to obtain drift rates as low as 1nm/min at 800 °C without correction. Instrumentation development, system characterization, experimental protocol, operational refinements and thermal drift characteristics over the temperature range will be presented, together with a range of results on different coating materials.

Biopolyurethane – carbon nanotubes composite for functional coating applications

(Contributed talk)

Oskars Platnieks (Riga Technical University), S.Gaidukovs, U.Cabulis

Polyurethanes (PU) are one of the most versatile type of polymers with wide range of applications and variable properties. PU-carbon nanoparticles nanocomposites are of great deal of interest due to differing from conventional composite materials with the exceptionally high surface to volume ratio of the reinforcing phase, which allows to significantly change material properties. In the present investigation, we aim to increase PU mechanical, thermal, chemical, biological and electrical properties with additional of the very small amounts of multiwall carbon nanotubes. The nanoparticles content was varied in the range (0.01-1 wt.%). Hybrid additives strategy was applied; thereto, in addition, nanoscale zinc oxide, nanoclay, silica were chosen for hybrid filler preparation. Composites were prepared by casting of two-component biobased liquid polyurethane; their hardening was realized at room temperature and ambient conditions. Finally, it can be concluded that the properties of the biopolyurethane – carbon nanotubes composites can be significantly enhanced.
Optimization of silver nanocolumnar coatings made by glancing angle deposition with magnetron sputtering to reduce the multipactor effect. (Contributed talk)

(Instituto de Micro y Nanotecnología IMN-CNMT, CSIC)

In this work, glancing angle deposition with magnetron sputtering has been used to fabricate nanocolumnar coatings made of silver. We have found that a highly collimated flux of atoms is important to achieve a well-defined columnar structure. Depending on the deposition parameters, several morphologies can be obtained, such as tilted or vertical nanopillars with different lengths. The secondary electron emission yield (SEY) from these nanostructured coatings has been measured, and it is lower than that obtained from flat silver, especially for those coatings with higher aspect ratio. The search of low-SEY coatings fabricated by simple techniques is of interest for high power RF devices in vacuum (for instance spacecraft equipment or particle accelerators apparatus), the main goal being the reduction of the resonant avalanche electron discharge that is known as the multipactor effect.

Development of smart coating for structural health monitoring (Contributed talk)

K. Parmar, Stephen Edmondson (Direct-C Ltd), A. Banica

This study describes the design and fabrication of smart coatings or smart films based on polymer nanocomposites where surface coating is actively used for continuous structural health monitoring. The smart coating comprises of polymer admixed with conductive carbon nanoparticles such as graphene nanoplatelets which improves the electrical conductivity of the nanocomposite significantly and provides the piezo-resistive capability. The smart coating is sensitive to changes in the external disturbance (i.e. stress/force/pressure or strain) and temperature, which helps to determine the structural dynamics. Based on the information gathered from the smart coating, the failure of a structure due to corrosion and/or other external factors may be predicted and/or detected before any substantial damage to the structure. The detection of disturbance is performed by detecting a change of the electrical conductivity of the smart coating. The coating is applied to a sample structure/surface using the spray coating method. The spray-coated samples are tested by performing various compression tests to determining its sensitivity towards external stress/strain and applied force.

CuS-based p-type transparent conducting thin film for all transparent electronics (Contributed talk)

Arindam Mallick (Indian Association for the Cultivation of Science), D. Basak

Lack of competent p-type transparent conducting films (TCFs) as compared to its n-type conjugate hinders the practical realization of transparent electronics and transparent p-n junction devices. The existing p-type TCFs are either highly resistive or less transparent or require high preparation temperature. CuS, a highly p-type conducting narrow band-gap semiconductor (~2.4 eV) has been found to be a promising candidate provided the transmission value in the visible solar light can be raised in CuS-based nano-composite thin films. In order to achieve high transparency in the visible-light, a wide band-gap (<3.5 eV) component should be chosen to prepare CuS-based nano-composite thin films. In an attempt to introduce a wide band-gap semiconductor like ZnS (band-gap=3.5-3.8 eV) into CuS, a series of CuS-ZnS nano-composite thin films of ~50 nm thickness with various Cu/(Cu+Zn) value have been deposited on glass substrates at 80-90 °C from an aqueous solution containing CuSO4, 5H2O, Zn (CH3COO)2, 2H2O and Na2EDTA, 2H2O in an appropriate ratio using chemical bath deposition (CBD) technique. The structural, electrical and optical properties of the deposited thin films have been investigated. The transmission value found to increase with the increase of ZnS component although at the cost of electrical conductivity. The highest figure of merit (FOM) value of 7.3×10-4 Ω-1 paired with resistivity value of 8.55×10-4 Ω-cm and optical transmission value of 85% at 550 nm has been obtained for a film deposited from the solution where Cu/(Cu+Zn) is 0.6. Although the FOM value is two orders less than that of commercial n-type TCFs, still it is among the greatest for the p-type TCF to the best of our knowledge. A p-n junction has been fabricated using n-GaN on SiC as n-type substrate and best-configured p-TCF i.e. Cu/Cu+Zn=0.6 shows excellent rectification ratio of ~3×104 at 2V. Detailed structural, electrical and optical results will be discussed during the presentation. The results show that the CuS-based p-type TCF may find some application in the all-transparent p-n junction in near future.
Self-supporting Diamond-Like Carbon (DLC) as a material for laser-driven particle sources

**David Haddock (Rutherford Appleton Laboratories), C. Spindloe, M. Tolley**

Laser driven ion sources potentially offer a compact alternative to conventional sources for scientific, technological, and health-care applications.

High-energy ion beams can be created by an ultra-intense laser focused onto a thin (nanometres-microns thickness) target foil. Ions are accelerated from the interaction and current research is focussed on refining the ion beam for use in applications. A robust, consistent, fully characterised target solution is required for the continuous development of laser-driven ion sources.

Of particular interest is a low-Z self-supporting film variable between 5 - 50 nanometres supported over an array of 400 micron diameter apertures. One suitable material is Diamond-Like Carbon and presented is its use as a laser target material. Its characterisation and processing parameters are discussed with the aim of making the foil suitable for laser irradiation.

The preparation of targets for laser experiments at the Central Laser Facility is the responsibility of the Target Fabrication group, who have coating capabilities including: CVD, PVD and PECVD. As well as, characterisation methods: AFM, Interferometry and Raman microscopy.

The Science and Technologies Facilities Council maintain and provide access to the UK's large-scale facilities at Rutherford Appleton Laboratories including ISIS neutron source, Diamond Light Source and the Central Laser facility.

characterization of vacuum deposited thin organic layers by phase shifting interferometry and FTIR spectroscopy for space contamination study

**Theo Henry (Université de Liège), K. Fleury-Frenette**

Although satellites and spacecrafts are exposed to very small amounts of organic contamination (typically 10-9 to 10-5 g.cm-2 cumulated over their lifetime) from the outgassing of some of their own structural components, fixation and photo-degradation of organic compounds can significantly alter the thermo-optical properties of critical surfaces involved in their passive thermal regulation. The study of surface molecular contamination under conditions representative of space environment is therefore of prime interest in order to define mitigation strategies. The Centre Spatial de Liège has recently initiated research work on this topic and first experimental results are presented here.

Organic thin films have been deposited using vacuum thermal evaporation/sublimation and their topology and equivalent surface concentration have been characterized using a combination of phase shifting interferometry and spectroscopic ellipsometry. Furthermore, FT-IR spectroscopy in reflection configuration have been tested in different conditions in order to monitor the IR signature of contaminated surface samples this in the perspective of implementing an in situ monitoring system on a deposition chamber with UV exposure capability.

Plasma functionalization of Spiro-OMeTAD hole transport material for material for perovskite solar cells

**Paheli Ghosh (The Open University), A. Ivaturi, T. Nixon, J. Bowen, N. S. Braithwaite and S. Krishnamurthy**

Spiro-OMeTAD [2,2',7,7'-tetrakis(N,N-di-p-methoxyphenylamine)-9,9'-spirobifluorene] is the most commonly used small organic molecule for hole transport material in perovskite solar cells (PSCs). Spiro-OMeTAD suffers from low hole conductivity and doping with Li-TFSI and tBP and exposure of the doped films in ambient conditions is required. This doping procedure might cause detrimental problems to the stability and resilience of PSCs [1]. A new route to improve the conductivity of spiro-OMeTAD without dopants is by using atmospheric pressure plasma jet (APPJ) to functionalize the films [2]. A systematic study of oxygen APPJ with different exposure time is thoroughly investigated. We observe change in morphology, electronic, electrical and optical properties of spiro-OMeTAD films due to plasma functionalization. The sheet resistance decreases substantially on plasma exposure. The surface conductivity mapping undertaken with electrostatic force microscopy (EFM) shows improvement in conductivity post plasma treatment. Change in the electrical properties can be attributed to the changes in the chemical structure of the carbon, nitrogen and oxygen as observed with x-ray photoelectron spectroscopy (XPS), in particular to difference in nitrogen bonding.

Change in band gap from 2.72 eV to 1.99 eV after plasma exposure is measured with Ellipsometry. These observations demonstrate that the excited species of ions and electrons in oxygen APPJ can be used as a low-cost and effective means of doping and surface functionalization of spiro-OMeTAD. This approach shows great promise for using plasma-doped spiro-OMeTAD as hole transport material for perovskite solar cells, that would assist easy scaling up process for commercialization. Further research needs to be undertaken to understand the mechanism behind the functionalization of these novel systems.

References:


2. Paheli Ghosh1, Aruna Ivaturi2, Tony Nixon1, James Bowen1, N.S. Braithwaite1, and Satheesh Krishnamurth1, Plasma functionalization of Spiro-OMeTAD hole transport material for perovskite solar cells (manuscript under preparation).
**Graphene: industry opportunities and challenges**

*Amaia Zurutuza (Graphenea S.L.)*

Graphene and 2D materials have attracted a huge amount of interest from the research community due to their exciting new properties and their potential industrial applications. The list of new 2D materials that are being isolated and investigated keeps growing everyday. However, applications using a new material can take many years to arrive to the market and considering graphene is the first of the 2D materials to be discovered, it is the one that is farther ahead in that race. Graphene in combination with other materials such as quantum dots or silicon have shown to provide enhanced performance in many applications. However, in order to integrate graphene in semiconductor devices in a cost effective manner, it will require combining it with CMOS technology. Indeed graphene has been monolithically integrated with CMOS technology. In addition, the fabrication of graphene at an industrial scale will require to overcome numerous challenges such as wafer scale uniformity with a high charge carrier mobility. In this sense, wafer scale CVD graphene growth and transfer with a high quality and uniform properties is key in order to ensure a uniform device performance. During this talk, I will cover current challenges related to graphene transfer, device fabrication\(^2,3\) and integration while providing some insight into potential solutions.\(^4,5\)

**References:**


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**The roadmap to applications of graphene and related materials**

*Andrea C. Ferrari (University of Cambridge)*

Disruptive technologies are usually characterised by universal, versatile applications, which change many aspects of our life simultaneously, penetrating every corner of our existence. In order to become disruptive, a new technology needs to offer not incremental, but dramatic, orders of magnitude improvements. Moreover, the more universal the technology, the better chances it has for broad base success.
Graphene and 2D materials for next generation optoelectronics and photonics

Anna Baldycheva (University of Exeter), A. Neves, M. F. Craciun, S. Russo

Graphene is an emerging material for electronics, photonics and optoelectronics due to unique physical properties such as high electrical conductivity, optical transparency and mechanical flexibility. These properties can be further enhanced or tailored to fit specific device functionalities by means of chemical functionalization. A recent example of the potential of chemical functionalization is the intercalation of few-layer-graphene with FeCl3 (dubbed GraphExeter)1, developed at the Centre for Graphene Science at Exeter. In the past years, our Graphene Centre demonstrated that this material is the best performing carbon-based transparent conductor1, with resilience to extreme conditions2 and potential for transparent photo-detectors3, flexible photovoltaic and organic light emitting devices4 and foldable light-emitting devices5. Among other emerging optoelectronic materials, fluid-dispersed atomically thin 2D nanocomposite materials demonstrate great promise for the next generation of multi-functional optoelectronic systems with a wide range of important applications, such as renewable energy, optical communications, bio-chemical sensing, and security and defence technologies.

The most recent highlight in this new research is the first demonstration of 2D materials optofluidic systems- a novel device concept for graphene and 2D materials optoelectronics and integrated photonics11 developed at the Opto-Electronic Systems Laboratory at the Centre for Graphene Science. In this talk I will review our latest developments in the use of graphene and functionalized graphene for electronics, photonics and optoelectronics. I will present our recent studies on the use of high quality graphene for next generation light emitting devices and for flexible, wearable touch-sensors6,7. I will review our recent demonstration of 2D heterostructures for video-frame-rate imaging applications8, intelligent design of 2D devices9 and GraphExeter photodetectors for high-definition sensing and video technologies10. I will also present our most recent results on dynamically controlled three-dimensional self-assembly of suspended 2D liquid exfoliated nano-flakes, which provides a breakthrough route for technological realization of 2D material based 3D meta-architectures11.

References:
5. Torres Alonso et al. (2016), Homogeneously Bright, Flexible, and Foldable Lighting Devices with Functionalized Graphene Electrodes. ACS Applied Materials and Interfaces 8, 16541.
Two-dimensional transition metal dichalcogenides (TMDCs) are the focus of intense experimental and theoretical research, because of intriguing physical structure which offers unique device potentials [1-3] that are complementary to graphene. For example, TMDCs show strong electron-photon coupling owing to band nesting, strong quantum confinement effects, spin orbit coupling provide promising platforms for fundamental studies with potential applications in optoelectronics and valleytronics [1,2]. Recently, the 2D layered materials grown by pulsed laser deposition technique is attractive in wide range of device applications [3].

Here, we present an ultrashort pulsed laser deposition of un-doped and rare-earth Yb3+ ion doped MoS2 films. The femtosecond laser with a wavelength of 800 nm, pulse duration of 100 fs and repetition rate of 1 kHz were used to deposit the layered MoS2 films. Different deposition parameters were used to form layered structures of MoS2 by controlling the laser fluence at 3 J/cm2. Ar pressure of 10 mTorr and the substrate temperature was maintained at 500°C. The deposited films were characterized by Raman spectroscopy which clearly showed the characteristic A1g and modes. The separation between the two Raman modes is 23 cm-1, which translates it into the ~4-5 number of layers. Strikingly, when these few layer MoS2 are doped with Yb3+ ions, the Raman modes were shifted to higher frequencies, implying the possible effect of Yb3+ doping and/or defects on 2D MoS2 structure. Further confirmation of layer number and doping, the films were studied by transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS). Nonlinear optical properties of un-doped and doped MoS2 films were studied using open aperture Z-scan technique. For this measurements, 7 ns pulses from the second harmonics of the Nd-YAG laser centered at 532 nm with a repetition rate of 10 Hz were used to excite the sample. In Yb3+ doped films, enhancement in saturable absorption (SA) at room temperature was observed, which is highly advantageous for applications in nano-photonic devices such as passive modelocking and optical switches. Our results provide important fundamental insight into the nonlinear optical response and are of crucial importance in designing novel multifunctional rare-earth doped 2D materials.

References:
assessing the quality of the part achieved. The study has been supported by carrying out mechanical and phase characterisations (XRD, FTIR, EDS and SEM), whereas pulsed thermography and ultrasonic C-scanning were used for the identification of the IDE embedded within the composite laminate. The research has shown promising results in terms of piezoelectricity after co-processing. The findings of the research provide recommendations for such in-situ monitoring enabled composites particularly via looking into development of an optimum process cycle that does not degrade the crystal phases of the PVDF, properly cures the laminate, and maintains the accuracy of the IDE post-curing.

Sharp fall of the thermal conductivity of a van der Waals heterobilayer due to interlayer sp3 bonds (Poster)

T. Iwata, Kazuhiro Shintani (University of Electro-Communications)

Graphene and van der Waals (vdW) heterobilayers are applicable to energy conversion as thermoelectric devices owing to their large Seebeck effects [1][2]. Since the figure of merit of such devices is inversely proportional to the thermal conductivity (TC) of the constituting material, reduction of its TC may lead to enhancement of the figure of merit. It was reported the TC of bilayer graphene is drastically reduced by interlayer sp3 bonds which can be introduced between the two layers via femtosecond laser excitation [3]. Such a report prompts one to expect that the TCs of the other vdW bilayers can also be tuned by interlayer sp3 bonds. In the present work, the TC of a graphene/hexagonal boron nitride (g/hBN) heterobilayer with interlayer sp3 bonds is addressed by means of nonequilibrium molecular dynamics (NEMD) simulation. It is found that if just a small fraction of sp3 bonds are introduced between the two layers, the TC of a g/hBN heterobilayer sharply decreases, and continues to gradually decrease with increasing their fraction up to 0.25. If the fraction exceeds 0.25, the TC starts to gradually increase, viz., there appears the minimum of the TC. Its existence can be understood by examining the local phonon density of states. Interlayer sp3 bonds act as phonon scatterers analogously to defects. As far as their fraction is small, the two layers constructing the heterobilayer are bound primarily by vdW interactions, viz., it is a vdW heterobilayer, and the contribution of the interlayer sp3 bonds to its structural unification is small. In such a situation, the TC decreases because the phonons of in-plane modes are scattered by the interlayer sp3 bonds. However, with increasing their fraction, they contribute more and more to binding the two layers, and the heterobilayer structure approaches a quasi-three-dimensional one whose rigidity is greater than those of vdW bilayers. Thus, the TC of the heterobilayer with interlayer sp3 bonds starts to increase if their fraction exceeds a critical value. Such a characteristic behavior of the TC is also verified by examining the overlap phonon energy in each of the two layers. These findings will be useful to tuning the TC of the heterobilayer and hence to enhancing the figure of merit of a thermoelectric device consisting of vdW bilayers.


Carbon nanoparticles containing polymer films and coatings with EMI properties (Poster)

Romans Vaivodiss (Rīgas Tehniskā universitāte), L. Klava, A. Barkane, S. Gaidukovs

Nowadays, very intensive use of the smartphone, laptops and other telecommunication devices led to tremendous electromagnetic pollution. Motivated by the human health, there is huge demand to develop novel materials, for example, polymer nanocomposites, which can mitigate electromagnetic interference (EMI). This presentation is connected with the preparation and characterisation of the bulk and layered polymer/graphene/carbon nanotubes/iron oxide nanocomposites.

We propose that the conductive nanofillers of different shape and physical properties can effectively improve EMI shielding and mechanical properties. As polymer matrix were used ethylene-vinyl acetate (EVA) copolymer. Nanocomposite were prepared by melt mixing method with nanofiller content varying from 0-40 wt%. Layered sandwich structures were designed by combining layers of polymer composite with layers of graphene, carbon nanotubes and iron oxide nanofillers. The dielectric, calorimetric and electrical conductivity properties were tested. Results showed remarkable improvement in material conductivity, dielectric permeability and also see minor changes in the thermal properties for layered polymer nanocomposite films.
Along with its excellent biocompatibility properties boron-doped diamond is acknowledged as one of the best electrodes for electrochemical applications, however its industrial application is restricted by the limited coating area of MW deposition systems. Large area deposition of boron-doped nano-crystalline diamond (B-NCD) layers using a Microwave Plasma Enhanced Chemical Vapour Deposition system with linear antenna delivery was reported (MW-LA-PECVD) in 2014 [1]. This synthesis is complicated by the addition of oxygen species, which are known to limit boron incorporation [2] and therefore reduce electrical conductivity in comparison with layers deposited using conventional MW systems.

In this work [3] we report on the optimisation of precursor gas composition for the repeatable preparation of large area highly conductive B-NCD layers with low sp2 content using MW-LA-PECVD apparatus. The precursor gas composition parameter space was probed by varying the boron, oxygen and carbon atomic ratios whilst fixing all other parameters. By radically increasing the B content and careful consideration of the B and O content in the gas phase, thin B-NCD layers (~ 300 nm) were prepared over large areas (6 inch diameter), repeatable, with high boron concentrations (~ 2×1021 at/cm3), electrical conductivity levels akin to B-NCD layers prepared in conventional MW PECVD systems (> 35 S.cm-1) and with electrochemical properties suitable for industrial applications.

We report on preliminary results of the use of such layers for monitoring of neuron electrical activity, using B-NCD based micro-electrode arrays (MEAs) and neural chemical activity, using B-NCD based electrodes. Rat hippocampal neurons were cultivated on B-NCD layers and their functionality successfully tested by micro-spectrofluorimetric imaging of changes of intracellular calcium using a calcium sensitive fluorescent probe (FURA2) and by electrophysiologic methods using B-NCD coated MEAs. Fast electron transfer kinetics for selected outer and inner sphere redox markers [Ru(NH3)6]3+/2+, [Fe(CN)6]3−/4− and dopamine along with fouling resistance was confirmed by cyclic voltammetry of B-NCD electrodes.

Finally, we report on the use of electrodes coated with B-NCD for water treatment using the electrochemical advanced oxidation process.

1. A. Taylor, DRM 47 (2014) 27;
2. A. Taylor, PSS A 212 (2015) 2418;
Among all drugs of abuse, cocaine is the most commonly illicit stimulant used in European Countries. Several societal implications are linked to the abuse of cocaine. From security outlook, cocaine illicit market is worth around 6 billion Euros yearly. In order to prevent illicit cocaine trafficking and their use, it is vital to identify suspected items/substances and to screen them promptly. Currently, on-site screening methods rely on ion mobility spectroscopy (IMS), on competitive inhibition immunoassay in lateral flow format, and on colorimetric tests. Nevertheless, both methods provide only qualitative or semi-quantitative results and required trained users. Presently, electrochemical impedance spectroscopy (EIS) biosensors are gaining popularity, due to the outstanding performance in detecting target molecule at very low concentration. On the other hand, nanoscale molecularly imprinting polymer (nanoMIP) are considered highly selective receptor. This work has combined the two technologies in order to develop NanoMIP-EIS-Sensors able to detect cocaine in trace amount. Performance of the resulting NanoMIP-EIS-Sensors in term of affinity, sensitivity (limit of detection) and cross-reactivity has also been investigated. The NanoMIP-EIS sensors showed to be able to detect cocaine in very low amount (pg mL-1), with low cross-reactivity towards both other drugs and the most common cocaine’s cutting-agents. This makes the technology proposed here a viable candidate for the production of devices suitable for fast and reliable on-site screening of illicit drugs.

**Sensing of ultralow concentration of the target protein exploiting imprinted nanogels and plasmonic plastic optical fibers**

*N. Cennamo, L. Zeni, M. Pesavento, Alessandra Maria Bossi (University of Verona, Italy)*

Molecularly imprinted polymers (MIPs) are a class of tailor-made biomimetic materials, prepared by template assisted synthesis. Being characterized by the robustness of the polymeric materials and the recognition properties of natural antibodies, MIPs are promising recognition elements for sensors. Moreover the high fidelity of imprinting achieved with protein templates opens up the use of MIPs in targeted proteomics methods. Here with the aim to provide a sensitive platform for the analysis of targeted proteins, molecularly imprinted nanogels (nanoMIPs) were synthesized using as template the model protein human transferrin. NanoMIPs were then coupled to a thin gold layer on a D-shaped plastic optical fiber (nanoMIP/POF) and the performance of the Plasmonic nanoMIP/POF sensor was studied. Acrylamido-based nanoMIPs, prepared by precipitation polymerization, resulted in swellable nanomaterial of about 45 nm in diameter. The nanoMIPs were chemically coupled to the optical fiber sensor to form a quasi-monolayer sensing surface. The response of the nanoMIP/POF to the target protein showed a Kd = 35 fmol/L with a LOD = 550 attomol/L. The selectivity was tested with competitor proteins and in diluted sera. So far the Plasmonic nanoMIP/POF method give an excellent sensitivity and is foreseen as a quasi-single molecule probe.

**Gold nanoparticle-enhanced immunosensors for rapid malaria detection**

*Aver Hemben (Cranfield University) and Ibtisam E. Tothill*

Malaria is a disease of global importance cause by an Apicomplexan Plasmodium parasite and transmitted by adult female Anopheles mosquitoes. Malaria affects approximately 50% of the world’s population causing millions of deaths every year. Mostly affected are pregnant women and children under 5 years of age. Despite control efforts the disease continues to affect productivity. This can be minimised by early detection of the disease. Methods for malaria detection include blood film microscopy, immunochromatographic, serological and molecular tests. Blood film microscopy shows the highest sensitivity and specificity when used by trained personnel with reliable instruments. It is however time-consuming and cannot be applied as a point-of-care diagnostic method.
Therefore a simple, fast and reliable method of detection such as biosensors is needed.

Two electrochemical biosensors (immunosensors) for malaria biomarkers Plasmodium falciparum histidine rich protein 2 (PFHRP 2) and parasite L-Lactate dehydrogenase (LDH) were developed in this work for the detection and quantification of Plasmodium species. The methods were based on screen-printed gold electrodes (SPGEs) and sandwich assay comprising of a ‘capture antibody’ immobilised on the SPGEs and a detector monoclonal antibody conjugated to the enzyme HRP for signal generation. Enhancement of the sandwich assays by the use of gold nanoparticles (AuNP) was also investigated. The biosensors developed for PFHRP 2 were capable of detecting sub-microscopic Plasmodium infection with a limit of detection (LOD) as low as 2.14 ng mL\(^{-1}\) and 2.95 ng mL\(^{-1}\) in buffer and serum assays respectively. When AuNP were used to enhance the assays, the LODs were lowered to 36 pg mL\(^{-1}\) and 40 pg mL\(^{-1}\).

The biosensors developed for the parasite Lactate dehydrogenase (LDH) showed LODs of 1.80 ng mL\(^{-1}\) and 0.70 ng mL\(^{-1}\) in buffer and serum assays. By the used of AuNP, the two LODs were lowered to 19 pg mL\(^{-1}\) and 23 pg mL\(^{-1}\).

Sensitivity of the immunosensors for PFHRP 2 and LDH was compared against commercially available Plasmodium immunochromatographic (ICT) kits: OptiMAL-IT and BinaxNOW Malaria kits. The optimised immuno-electrochemical biosensors were able to detect PFHRP 2 and LDH at much lower concentration than the commercial ICT tests.

The immunosensors developed here are therefore recommended for field trial.

### Annealed nanostructures for biosensing applications (Contributed Talk)

**Rodica Elena Ionescu, (Université de technologie de Troyes)**

Gold/silver nanostructures (NPs) are obtained on glass substrates by using high temperature annealing of metallic pre-coated solid surfaces. Such supports have stable plasmonic properties being very good candidates for designing ultrasensitive localized surface plasmon resonance (LSPR) immunosensors and genosensors.

For optical investigations, several areas containing nanostructures are analyzed and compared thanks to a grafted TEM micro-grid on glass supports. Moreover, “bar-code” multiplexing samples containing nanoparticles are created on glass by using a scotch – tape approach to delimit different NPs-areas. (Bio) functionalization of nanostructures for specific and non-specific LSPR detection of proteins, herbicides and DNA strains from Brettanomyces Bruxellensis will be presented.

Finally, a novel approach to fabricate highly sensitive Surface Enhanced Raman Spectroscopy (SERS) substrates by using E. coli bacteria and high temperature annealing for the creation of extremely well-organized nanoparticles within a simple and cost-effective manner will also be discussed. As an important substrate application, a standard molecule named trans-1, 2-bis(4-pyridyl)ethylene (BPE) is investigated for its SERS specific signature over a large range of concentrations from 10-5 M down to 10-12 M.

### Functionalized cellulose acetate membranes as coatings for biomedical applications (Contributed Talk)

**Stefan. I. Voicu (University Politehnica of Bucharest) V.K. Thakur, A.M. Pandele**

This work describe a new method for obtaining functionalized cellulose acetate membranes with resveratrol, respectively sercin for improving osseointegration at the interface between bone and metallic implants. For this purpose, cellulose acetate membrane is hydrolyzed in the presence of potassium hydroxide, followed by covalent immobilization of aminopropyl triethoxy silane (APTS). Sercin, respectively resveratrol were immobilized onto membranes using glutaraldehyde as linker. Scanning electron microscopy revealed morphological changes (due to the crosslinking effect of APTS and glutaraldehyde reactions), correlated with water flows through synthesized membranes and proteins retention.

Covalent immobilization of resveratrol, respectively sercin was proved by FT-IR, Raman, XPS and thermal analysis. The obtained membrane materials were also evaluated for osseointegration potential and cytotoxicity using MC3T3-E1 cells, proving their biomedical potential.

**Acknowledgement:**

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### Electrochemical protein, DNA and aptamer-based biosensors

**Pedro Estrela (University of Bath)**

There is a great need for low-cost intelligent biochips capable of massive parallel detection to be used in portable instrumentation. Electrochemical methods are inherently low-cost, miniaturizable and easily integrated into multiplexed systems for the parallel screening of a panel of biomarkers.

Of particular interest are biologically sensitive field-effect transistors (BioFETs) and impedance-based sensors.

We here exemplify the use of synthetic molecules such as DNA aptamers and peptide aptamers (Affimers), as alternatives to antibodies, as well as oligonucleotide-based approaches in impedance and BioFET sensors for the detection of a range of biomarkers in medical diagnosis and for environmental monitoring.

The use of nanomaterials, in particular nanoparticles, can help improve sensitivity and limits of detection, as well as provide new routes for creating surface chemistries with enhanced anti-fouling properties.

_Nanoparticles can also be used for signal enhancement in DNA sensing and on-chip validation of biomolecular interactions, such as protein phosphorylation on BioFETs, through nanoplasmonic effects._
"Photoinduced preparation of Molecularly Imprinted Polymers (MIP) for sensor applications"

Olivier Soppera (CNRS – Université de Haute-Alsace)

In the context of chemical microsensors or microbiosensors, Molecularly Imprinted Polymers (MIPs) are particularly well adapted as synthetic biomimetic recognition materials. Recent examples of MIP have demonstrated their interest for chemical sensors associated to a wide range of chemicals for which these materials exhibit both specificity and sensitivity.

However, their development in devices has been confronted to the difficulty to interface the functional material with the transducer or with a microchip. The photochemical route that we proposed significantly simplifies the integration of the functional material into the sensor device. Specific MIP precursors formulations were prepared to fulfill the requirements for micropatterning and molecular imprinting.

Photoinduced polymerization can be used to achieve the preparation of the MIP and at the same time spatially controlled irradiation allows shaping the material. Such route significantly simplifies the integration of MIP in sensors. Advanced methods of photopatterning were used including interference (holography) 3,4, optical near-field2, lithography on optical fibers5 and two-photon stereolithography6.

Photopatterning appears thereby as one of the most suitable methods for patterning MIP at the micro and nanoscale, directly on the transducer surface. Demonstration of use of these strategies for sensor application is presented. Such route opens new opportunities for low cost, highly sensitive and highly specific sensors.


"Recent advances in polymer nanocomposites based biosensors"

Giovanna Marrazza (University of Florence)

In the last decade, polymer nanocomposites have brought about a revolution among many new hybrid materials and consequently their application in chemical sensors and biosensors. Since these polymer nanocomposites facilitate combinations of unusual properties with enhanced flexibility in structural design, they appear to be more promising than conventional materials in terms of their applications, cost, properties, shelf life, handling, and thermo-mechanical properties.

Metal nanoparticles have been often incorporated in conducting polymer-based biosensor configurations with two main purposes: the fabrication of nanostructured supports and their use as signal enhancers. They possess high electrocatalytic activity, stability and biocompatibility, and they can be easily functionalized. They favor electron transfer and, as a result, higher sensitivities and lower limit of detections are attained by metal nanomaterial-modified electrochemical biosensors. The embedding of metal nanoparticles into polymer matrices represents a simple way to use the advantages of nanoparticles. This technique is one of the most efficient strategies to avoid the aggregation of nanosized metal and conserve their properties. Therefore, polymer-metal nanocomposites in which polymer phase acts, as a stabilizer, template or protecting agent show many important attributes.

Numerous biosensors have been realized incorporating oligonucleotides, enzymes, aptamer, or antigen/antibodies into the polymer-metal nanocomposites. Thus, a comprehensive overview of polymer nanocomposites based biosensor along with polymer synthesis routes, surface treatment strategies and biosensors approaches will be presented.

"Field cancerization effect in the early detection of cancer"

David Alejandro Miranda Mercado (Universidad Industrial de Santander)

Early detection of cancer increases the probability of successful treatment dramatically. The traditional cancer detection techniques imply the study of tissues to detect anomalies. However, it is tricky to detect these anomalies in the early stadiums of cancer by traditional detection techniques. Molecular, genetic and epigenetic techniques could be used to detect anomalies associated with cancer in the early stadiums.

New biochemical techniques for early detection of cancer are studying, but, what is the biochemical fundament of all of these new technologies? In this work, an interpretation of field cancerization effect as an amplification of biochemical anomalies is proposed to explain the biochemical fundamentals of new technologies to the early detection of cancer.
“Heterostructure radiation detector materials for the next generation of time of flight positron emission tomography scanners

Gregory Bizarri (Cranfield University)

Since the introduction of early ring systems more than 40 years ago, Positron emission tomography (PET) instrumentation has become increasingly sophisticated and successful, driven to a large degree by the rapidly growing needs across tumour imaging; initial diagnosis, detection of recurrent tumour, and evaluation of response to therapy. However, the demand is still high for advanced scanners allowing to minimize radiation dose while maintaining image quality, and in fine diagnostic power. This effort has supported a steady and multifaceted improvement of PET instrumentation with the implementation of new hardware (i.e. Time of Flight, multi-modality PET/CT and PET/MRI scanners) and new reconstruction methods (i.e. compensation for respiratory and cardiac patient motion).

Of the many areas explored in detector design, the scintillators per se have seen moderate evolution since the implementation of Lu2SiO5:Ce3+ (LSO) crystals as scintillator of choice. This incremental progress is deeply rooted in the historical approach of using a unique material as energy conversion medium which has to answer to the extremely tight and time to time averse requirements imposed by the application. As a result, even LSO was selected as a trade-off between application needs (extremely high stopping power, good light output and ultrafast sub-nanosecond timing) and material availability. After a short introduction on PET technology, the presentation will focus on the recent concept of heterostructure radiation materials and how they could theoretically supersede LSO as high performance Time of Flight PET radiation detectors. The presentation will underline the multi-disciplinary material challenges which, if bypassed, will enable heterostructure radiation detectors to surpass LSO performance by sharing the application requirements across multiple materials. This would result in a detector material with similar stopping power and light output to LSO while introducing a sub-nanosecond component in the scintillator time response. Special attention will be given to the latter by discussing the primordial role of surface engineering in managing light transport and extraction from the heterostructure, which in turn will allow to maintain an ultra-fast timing across the entire detector assembly; from light production to detection. This is key to eventually enable new operat us modi in tumour imaging and establish non-invasive medical procedure.

“Zinc isotopes as a possible biomarker for breast cancer

Rebekah Moore (Imperial College), M. Rehkämper, F. Larner, R. C. Coombes

Humans use zinc and other trace metals in metabolic processes that are fundamental to life. High-precision isotopic analyses are routinely used in geosciences to study the cycling of trace elements in natural systems. The application of such methods in medical research is just emerging and shows potential to access previously elusive metabolic processes and improve understanding of illnesses. In particular, it was shown that isotopic data for Zn and Cu can support investigations of cancers and should be explored as a potential biomarker for early detection.

The high Zn concentrations of breast tumour tissue have been known for over a decade but the underlying cause for the altered Zn metabolism remains elusive. To investigate further, we carried out the first high precision measurements of Zn isotope status on various tissues from breast cancer patients and controls. Our results confirm previous findings of higher Zn contents for tumour relative to healthy breast tissue. They also reveal, for the first time, that tumours have a significantly lighter Zn isotope composition than the blood, serum and healthy breast tissue of both cancer patients and healthy controls. The isotopically light Zn of tumours suggests that sulphur-rich metallothionein dominates the isotopic selectivity of breast tissue whilst the significant and systematic change in Zn isotope status might provide a new biomarker for early breast cancer detection.

Manufacturing of devices using light cure resin with nanofillers of copper and copper nickel through stereolithography technique

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The present research focuses on the additive manufacturing using the stereolithography technique different geometric shapes and their mass functionalization, with and without additives of copper nanowires, copper-nickel alloy nanowires and a mixture of copper nanowires with copper nanoparticles at different concentrations (among the 5% and 20% in w/w). The substrates were designed in a computer aided design software and later were manufactured, layer by layer with a resolution of the layer thickness of 100 microns, in photosensitive resin, were the polymeric matrix contains Cu NWs (70 μm, 70 nm wide, aspect ratio of 1000) or CuNi NWs (about 26 μm long and 88 nm wide, aspect ratio about 300), which have been used to manufacture nanofilms used as transparent electrodes, in search of an adequate mass functionalization. The manufacture of devices, test probes, and the consequent characterization results show the possibility of mass-functionalizing resins for additive manufacturing towards final components with special functionalities such as manufacturing of flexible sensors or the building of circuits/devices using self assembly approaches.
New DNA-based sensing platform for the detection of profenofos pesticide (Poster)

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Herein, we propose an electrochemical DNA-based sensor for the sensitive detection of an organophosphorus pesticide, namely profenofos. The sensor is based on a competitive assay where the graphite-based screen-printed electrodes were electrochemically modified with polyaniline film and gold nanoparticles by cyclic voltammetry [1][2]. Then, the gold nanoparticles were functionalized with thiol-tethered DNA oligonucleotide sequence complementary to the DNA aptamer. Different profenofos solutions containing a fixed amount of biotinylated aptamer by DNA-based arrays were analysed. Streptavidin-alkaline phosphatase conjugate was then added to trace the affinity reaction and catalyse the hydrolysis of 1-naphthyl phosphate to 1-naphthol. The enzymatic product was detected by differential pulse voltammetry. A decrease of the signal was obtained when the pesticide concentration was increased, making the sensor work as signal off assay. The parameters involved in the DNA sensor development were studied and optimised using optical and electrochemical techniques. Under optimised conditions, a dose-response curve was obtained in the concentration range of 0.05-10.0 µM profenofos solutions. The selectivity of the DNA-based sensor was also tested in the presence of non-organophosphorus pesticides.

References

Acknowledgements

Development of thin films for applications in piezoresistive sensors for cancer detection (Poster)

J.J Rosero Romo (National University Of Colombia), H.A. Estupiñan Duran, J L Endrino

Portable devices are increasingly used and often used, as they give us advantages such as the ease of having the information we want at hand, either from a simple device to listen to music, a laptop or even more sophisticated machines of industrial analysis or field exploration for industrial and medical applications. The advance in micro-nanotechnology and wireless communication mediums over large and short distances in the last decades has allowed us to advance more in the improvement and development of new applications of these devices. One of the current areas and of greater attention due to its usefulness is in the field of health. The construction of portable sensors for the monitoring of physical and physiological changes in daily life is a promising field of research that provides benefits in improving the quality of life for the user who has access to this new class of sensors. Some of these sensors have already been created and are of great help in medical applications, as is the monitoring of physiological phenomena, such as cardiovascular diseases. In the last 50 years, cancer has become the main cause of death as can be seen in associations dedicated to keeping a record of these statistics and a great increase in the appearance of cancer has been evidenced, especially in developed cities. The need for a rapid, sensitive, reliable, easy to use and non-invasive diagnosis is necessary. Several research groups worldwide are focusing on these issues. Microelectromechanical systems (MEMS) are candidates for medical applications and require the creation of specialized thin films. Therefore, these films have been developed with composite materials. The research focuses on the characterization and finds suitable parameters such as the dispersion of the nanostructures and percolation threshold, on which the properties of the created film depend. This paper presents the results of the manufacture of thin films with conductive polymers such as polypyrrole (PPY), Poly (3, 4-ethylene dioxythiophene) (PEDOT) and multi-walled carbon nanotubes (MWCNT), graphene Nano
platelets (NpGr) electrodeposited on a silicon / silicon oxide substrate. By varying the concentration of the components, the films obtained were characterized by electrochemical impedance spectroscopy (EIS), morphologically by scanning electron microscopy (SEM), topographically and mechanical properties by atomic force microscopy (AFM). The results obtained showed that by means of this electrodeposition method it is possible to obtain carbon nanostructures embedded in slightly ordered PPY and PEDOT and with important variations in the morphology that improving the manufacturing process can be applied in the construction and development of piezoresistive sensors and that present adequate mechanical, electrical properties to be a promising element in portable medicine applications and a high level of sensitivity and be able to create light, economical and portable devices.

Localized surface plasmon resonance in laser- deposited gold nanoparticle layers for total internal reflection based optical sensors (Poster)

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The localized surface plasmon resonance (LSPR) of laser-deposited gold nanoparticle layers has been investigated using spectral transmision, total internal reflectometry and total internal reflection ellipsometry (TIRE). The LSPR characteristics depend on the size and density of nanoparticles within the layers which in turn can be adjusted to a certain extent through the laser process parameters and that with the high spatial resolution provided by the laser beam. Moreover it appears that the use of total internal reflection (TIR) as a readout configuration can significantly enhance LSPR- based sensors sensitivity to refractive index variations in probed liquid samples in comparison to the transmission read-out configuration. The laser implementation of LSPR-supporting nanoparticles in a lab-on-a-chip device integrating a TIR-based multiplexed optical read-out system is discussed here in the frame of biopesticides detection.

Development of a Point of Care Biosensor for the Detection of Breast Cancer Biomarkers

Shayalini Wignarajah (Cranfield University), I.E.Tothill

Breast cancer is the fifth highest cause of death around the world and the highest cause of mortality in women in the UK. It is a disease affecting the tissues of the human breast and it can quickly spread to other parts of the body resulting in an acute disease that cannot be cured. To prevent high mortality of breast cancer early detection of the disease is vital.

Biosensors can provide a promising rapid and easy-to-use method for breast cancer detection as an alternative to the currently available tools. A highly sensitive electrochemical immunosensor based on a screen-printed electrode (SPGE) with on board carbon counter and silver/silver chloride pseudo-reference electrode was developed in this work for the detection and quantification of the breast cancer biomarker Human Epidermal Growth Factor Receptor (hEGFR) and serum human epidermal growth factor receptor 2 (HER-2). Both EGFR and HER-2 are usually overexpressed during breast cancer and can be used as indicators of the disease. The sensor used an indirect sandwich ELISA format with an optimised monoclonal antibody against EGFR and HER-2 immobilised on the gold working electrode using passive adsorption. A polyclonal antibody against EGFR and HER-2 was applied and finally a secondary antibody conjugated to the electroactive enzyme horseradish peroxidase was applied for signal generation. The electrochemical measurements were conducted using TMB/H2O2 as the enzyme substrate system at a potential of -200mV.

The developed EGFR biosensor was capable of detecting EGFR in buffer with a linear range of 0.5- 75 ng mL-1 and limit of detection as low as 1.06 ng mL-1. While the HER-2 biosensor was able of detecting HER-2 in buffer with a linear range of 5- 200 ng mL-1 and limit of detection as low as 0.95 ng mL-1. Benchmarked against the commercially available EGFR and HER-2 ELISA kits, both the optimised immuno-electrochemical biosensor showed a strong correlation of the quantified samples (R2=0.99).

Secondly, gold nanoparticles labelled with secondary antibody-HRP conjugate at a dilution factor of 1:10 were employed as a strategy for signal enhancement and increased sensitivity. The developed AuNP electrochemical immunosensor was able of detecting EGFR in buffer with a linear range of 0.5-0.05 ng mL-1 and limit of detection as low as 30 pg mL-1. While the developed gold nanoparticle electrochemical immunosensor was able of detecting HER-2 in buffer with a linear range of 0.25 – 50 ng mL-1 and limit of detection as low as 30 pg mL-1. Both optimised immuno-electrochemical biosensor showed a strong correlation of the quantified samples (R2=0.99).

Finally the immunosensors were tested with serum. Thus, the developed electrochemical immunosensor was able to detect EGFR in spiked 100% serum with a linear range of 0.5 -75 ng mL-1 and limit of detection as low as 1.17 ng mL-1. While the developed electrochemical immunosensor was able of detecting HER-2 in spiked 100% serum with a linear range of 5- 80 ng mL-1 for the normal assay, and a linear range of 0.25- 25 ng mL-1 for the AuNP assay. A limit of detection of 1.47 ng mL-1 in the HER-2 normal serum assay and 50 pg mL-1 in the AuNP 100% serum assay was achieved. The HER-2 AuNP electrochemical immunosensor showed high specificity with very low cross-reactivity to EGFR. Both optimised immuno-electrochemical biosensor showed a strong correlation of the quantified samples (R2=0.99). Both developed sensing systems have shown a potential use for future early detection in breast cancer using specific biomarkers.