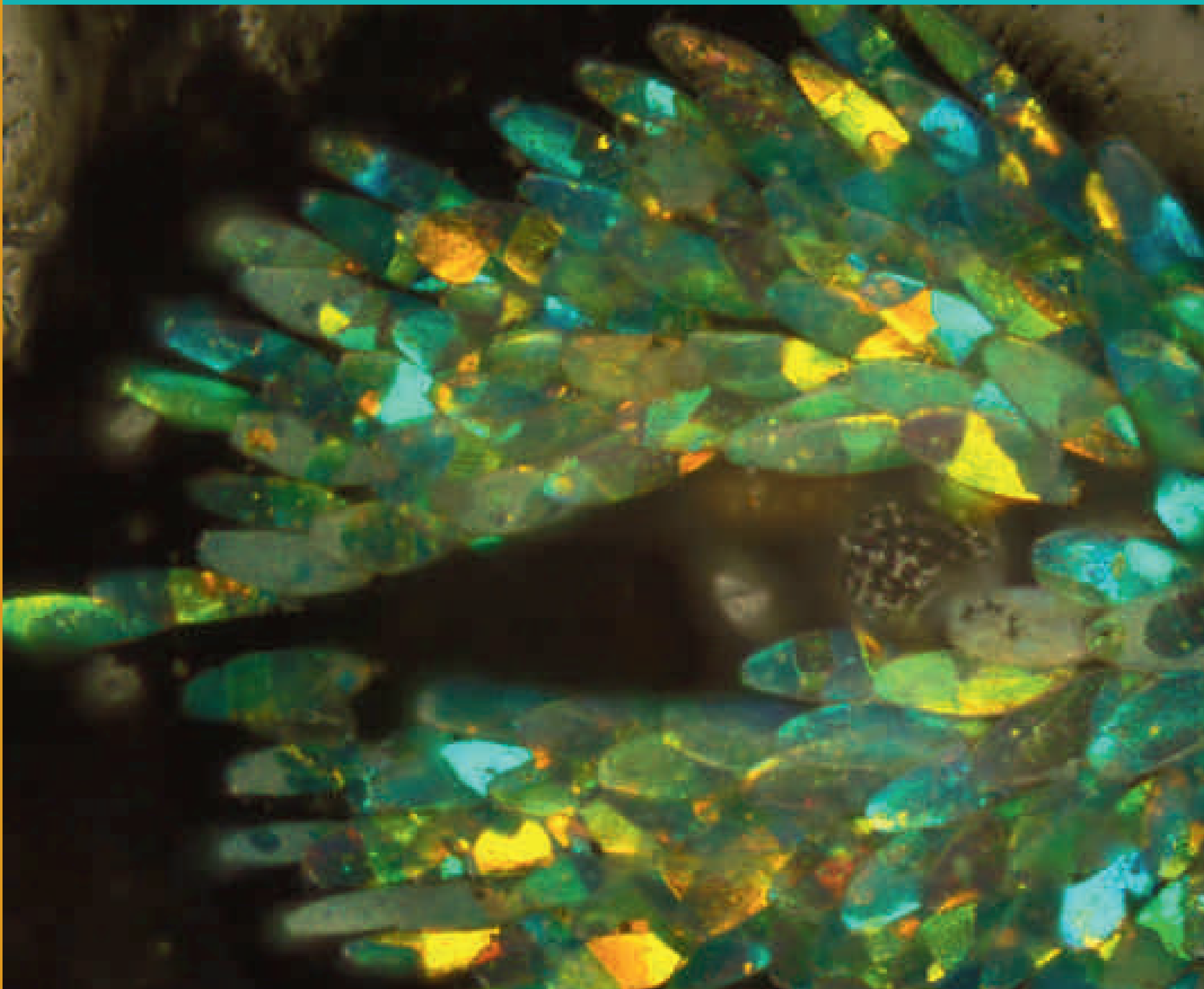
NANOTECHNOLOGIES

2011



AL Académie universitaire Louvain

NANOTECHNOLOGIES

2011

The Académie universitaire Louvain (AL) is a university consortium composed of the following four academic institutions: FUCaM, FUNDP, FUSL and UCL.

In this particular case, this brochure « Nanotechnologies » has been prepared by Research Administration Departments of the FUNDP and the UCL only, with the valuable help of a peer review committee composed of:

- Professor Bernard Nysten, UCL - Institute of Condensed Matter and Nanosciences (IMCN)
- Professor Benoît Champagne, FUNDP - Applied Theoretical Chemistry laboratory (CTA)
- Professor Luc Henrard, FUNDP - Centre de Recherches en Physique de la Matière et du Rayonnement (PMR), Solid State Physics Laboratory (LPS)
- Professor Véronique Préat, UCL - Louvain Drug Research Institute (LDRI)

With the contribution of Jean Colin (UCL), Carine Hellinckx (UCL), Dimitri Lederer (UCL), Nathalie Malengreau (FUNDP), Sandrine Tichon (UCL).

Cover: Naturally grown nanostructures found in living organisms are among the most sophisticated devices that the human mind could imagine. One of the most remarkable consequences of these elaborate architectures is the so-called structural color, which exhibits color change with viewing angle (iridescence). The reason of interest for the living organisms is that they are an inexhaustible source of inspiration for human invention, the so-called biomimetism or/and bioinspiration. The optical microscope image of the brazilian *Weevil Entimus imperialis* (a beetle from the *Curculionidae* family) is a good example showing various high-light colours delivered by the diffraction of light due to the three-dimensional photonic crystal localized in the scales irregularly implanted on its cuticle.

Foreword

Nanoscience and nanotechnology, often presented as the motors of the XXIst century industrial revolution, emerged about thirty years ago with the discovery of new nanomaterials such as fullerenes and carbon nanotubes, and the development of advanced experimental techniques allowing to see and manipulate matter at the atomic and molecular scales. The emergence was also driven by the continuous need for miniaturisation in the information and communication technologies (ICT) (Moore's law). These discoveries and technological developments put into concrete the visionary talk of Richard Feynman: "There's plenty of room at the bottom."[1]

According to the definitions proposed by the Royal Society[2], "*nanoscience is the study and manipulation of materials at the atomic, molecular and macromolecular scales, where properties differ significantly from those at a larger scale*" and "*nanotechnologies are the design, characterisation, production and application of structures, devices and systems by controlling shape and size at nanometre scale*". They are widely seen as having to bring huge potential benefits in areas such as drug delivery, water decontamination, information and communication technologies, production of stronger and lighter materials, sustainable development… They are attracting increasing investments from governments and businesses all over the world. In 2001 the US National Science Foundation (NSF) estimated that, at the horizon 2011-2015, the total annual value for all nanotechnology-related products, including ICT, will amount to $1 trillion. Today nanotechnologies and nanomaterials are already present in hundreds of commercialised products or applications such as the last computer microprocessors, nanoclay and carbon nanotube reinforced composites, alumina nanofibres for water treatment, nanoparticle containing cosmetics, drug carriers with low water solubility, etc. They also raised ethical and societal issues such as nanomaterial toxicity, economic impacts, information collection and implications for civil liberties, human enhancement, military uses…

Since the emergence of nanoscience and nanotechnologies, many research groups of the Académie universitaire Louvain have been active in those fields. The main objective of this booklet is to highlight the level of excellence they have reached. It is also aimed at enriching the existing synergies and at promoting new partnerships inside and outside the Academy. Last, but not least, it is aimed at supporting industrial innovation by raising business awareness of the Academy's competencies in nanotechnologies and by giving rise to partnerships with industrials and research centres. In particular, this booklet presents the research topics, the recent achievements and the current developments of the research teams of the Académie universitaire Louvain. From going through the booklet, it will be obvious for the reader that the research teams have developped a wide expertise in the different areas of the nanotechnology field: nanometrology, nanomaterials, nanoelectronics, and ICT, bio-nanotechnology and nanomedicine. The high level of this expertise is demonstrated through the large number of publications and patents as well as through the numerous collaborations with world renowned institutions. The booklet also highlights the partnerships already established between research groups from different disciplines (physics, chemistry, biology, medicine, engineering). Such convergence, which is one of the main characteristics of nanotechnologies, shows the quality of the research.

[1] Annual Meeting of the American Physical Society, December 1959

[2] Nanoscience and nanotechnologies: opportunities and uncertainties, The Royal Society, 2004

This booklet is arbitrarily organised in seven sections gathering the different topics according to the scientific and/or technological approach:

- nanomaterials
- surfaces and thin films
- nanoelectronics/photonics
- nanomedicine
- nanotoxicology, ethics and legal issues
- simulation and modelling techniques
- characterisation techniques

Page 9, a cross table allows positioning the research teams in the various domains of nanotechnologies.

For the editorial committee,
Bernard Nysten,
Louvain-la-Neuve, January 2011

Content

A. NANOMATERIALS

F. SIMULATION AND MODELING

G. CHARACTERIZATION TECHNIQUES

Reading notes pivot table

The pivot table below categorizes the reading notes per type of nanostrucutre geometry considered in the study (first 4 columns), per type of application (next 7 columns) and according to the nature of the investigation (4 rows). Some reading notes were classified in more than one cell, in accordance with the scope of the research topics they address.

		Geometry of nanomaterials				Applications						
		surfaces and thin films	nanopores	nanostructures (nanotubes, nanoparticles, nanoribbons, nanorods, nanocontainers, nano (bio) molecules, NEMS, nano-transistors, nanowires)	nano-composite materials	drug delivery and therapy	nano-microbiology	cell and tissue engineering	biosensors	nanoelectronics/ photonics	smart coatings	catalysis
Research type	simulation and modeling techniques	A07, B04, C09, F01, F02		A07, C02, C06, C07, C09, F01, F02, F03, F04					F04	C02, C06, C07, F01, F02, F04		
	characterization	A01, A07, B01, B04, B07, C10, G02, G03, G04, G05		A01, B07, C02, C03, C07, D01, G01, G02, G03, G04, G05	A05	A01	D01	A01, B01, B05	A01, G01, G05	A01, C02, C05, C07, C09, C10, G01	A01	
	fabrication	A01, A07, A14, B01, B02, B03, B04, B05, B06, B07, B08, C10, C12, D06	A03, A06, B06, B08	A01, A02, A04, A06, A07, A08, A09, A10, A11, A13, A14, B08, C01, C02, C03, C05, C06, C07, C08, C11, D02, D03, D04, D05, D06, D07, D08	A05	A01, A09, B04, D02, D03, D04, D05	A10	A01, A10, B01, B05, D07, D08	A01, A14, B02, C11, D06, D07, D08	A01, A14, B02, B04, B07, C01, C02, C03, C04, C05, C06, C07, C08, C09, C10, C11, C12	A01, B02, B03, B07	A09, A12, A11, A13
	nanotoxicology, ethics and legal issues			E01, E02, E03								

Functional Hybrid Nanostructures

SENIOR SCIENTISTS :

- Sophie DEMOUSTIER-CHAMPAGNE
- Etienne FERAIN
- Karine GLINEL
- Alain M. JONAS
- Roger LEGRAS
- Bernard NYSTEN
- Luc PIRAUX

Research Field and Subjects

The research group "Functional Hybrid Nanostructures" (FHYN) is one of the four research groups of the Bio- and Soft Matter pole (BSMA) of the Institute of Condensed Matter and Nanosciences (IMCN).

It gathers approximately 35 researchers whose activities are focused on the development and the characterisation of functional hybrid nanostructures and smart surfaces for spintronics, organic electronics (organic thin film transistors, organic non-volatile memories…), bio-medical applications (nano-biosensors, drug delivery, tissue engineering…), corrosion protection, nano-actuators, etc.

To achieve their objectives, the members of the group have developed expertises in the following fields:

- Synthesis/fabrication of functional nano-structures: organic, inorganic or hybrid nanowires/nanotubes, track-etched membranes, alumina templates, etc.
- Surface modification/functionalisation: nano-patterning, biofunctionalisation, layer-by-layer assembly, polymer brush synthesis, etc.
- Nanoscale characterisation of the structural, physical and chemical properties of nanomaterials and surfaces.

Representative References

- CALLEGARI V., DEMOUSTIER-CHAMPAGNE S. Interfacing Conjugated Polymers with Magnetic Nanowires. AC- Applied Materials & Interfaces, 2, 1369, **2010**.
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Patents

- Ferroelectric Organic Memories with Ultra-low Voltage Operation. GB Patent Application GB0809840.2.; European Patent Application PCT/EP2009/ 056656
- Drug-eluting nanowires array, application, number: PCT/EP2008/063803
- Method and device for high sensitivity and quantitative detection of chemical / biological molecules, application number: PCT/EP2008/059460

Awards

- A.M. Jonas: Leverhulme visiting professorship (2006-2007, University of Cambridge); Runner-up Obducat Prize 2007 for "Nanoimprint beyond lithography".

Funding

- European Union FP7: *MASTER NMP, MOMA strep, Erasmus Mundus actions…*
- UIAP-VI networks: *Functional Supramolecular Systems & Quantum Effects in Clusters and Nanowires*
- Actions de Recherche concertées: *DynanoMOVe & Hybrid metal/organic nanosystems*
- Wallonie: *NanoTIC–Feeling, NEOCERAT, DEEP, BIOSE, RAPARRY, NANOROD…*
- FNRS, FRIA, Fondation Louvain

Partnership

- Most Belgian Universities (within the frame of UIAP networks and Wallonia funded projects)
- Foreign Universities: Groeningen (NL), Cambridge (UK), Bordeaux (FR), Paris 6 (FR), Grenoble INPG & UJF (FR), Lausanne EPFL (CH), Nebraska (USA), Nevada (USA), Soochow (Suzhou, China), Nancy I (FR), Rio de Janeiro (BR), CNRS/THALES (FR), CEA- Grenoble (FR), San Luis Potosi (Mexico)
- Companies: Solvay Solexis, STMicro-electronics, IMEC, Holst, it4ip, EADS, Neurotech, Zentech, Thales

Main Equipment

- Hybrid nanostructures fabrication: electrochemical synthesis facilities, high vacuum coating systems (sputtering and e-beam), platform for surface functionalisation, electron beam nanolithography, nanoimprint lithography...
- Microscopies: Transmission and Scanning Electron Microscopies (TEM, SEM), Scanning Probe Microcopies (STM, C-AFM, LFM, FMM, Tapping™ mode, Harmonix™ mode, MFM, EFM, KPFM, PFM)
- Thin film characterisation: X-ray reflectometry, spectroscopic ellipsometry
- Investigation of physical properties: electrical and thermal measurements, SQUID magnetometer, cryogenics systems, network analysers

Products and Services

Fabrication and characterisation tools for hybrid nanostructures (thin films, nanowires, nanotubes, nano-patterned surfaces, etc.)

KEYWORDS

Biofunctionalisation
Hybrid nanowires
Functional nanotubes
Multiferroics
Nanoporous membranes and templates
Organic electronics
Smart coatings
Spintronics

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Functional Organic and Hybrid Nanotubes and Nanowires

SENIOR SCIENTIST :

- Sophie DEMOUSTIER-CHAMPAGNE

Research Field and Subjects

Recent advances in nanoscience and nanotechnology have led to the development of novel nanoparticles where size, size distribution, porosity, geometry and surface functionalisation can be controlled at the nanoscale. This opens unique opportunities to elaborate new advanced functional materials with potential applications in various areas. In this respect there is currently a particular interest into anisotropic nanostructures such as nanotubes and nanowires and more specifically into multi-component or multi-functional nanowires and nanotubes.

Among the different strategies to synthesize nanoscopic materials, template synthesis is an elegant and versatile approach. This technique consists in including the desired material(s) inside the void spaces of nanoporous host materials. For several years the team has been developing synthesis processes, based on the nano-templating method coupled with electrochemical deposition and/or layer-by-layer assembly, for producing a large range of monodispersed macromolecular (polymers, proteins, DNA), metallic and hybrid functional nanostructures presenting well-defined geometry and dimensions. The nanostructures can remain inside the pores or can be freed from the template and collected as an ensemble of free particles. Alternatively, if nanostructures are synthesized within a supported porous thin film and the template is removed, an ensemble of nanotubes or nanowires that protrude from the surface like the bristles of a brush can be obtained.
Another part of our research activities is dedicated to the development of methods for grafting bio-receptors (enzymes, antibodies and antigens) onto different types of flat or nanostructured surfaces and onto various types of nanotubes and nanowires.
From a fundamental point of view the team is interested in studying the impact of the low dimensionality of these functional nano-objects on their structure, function and properties. In collaboration with different partners, the team is also evaluating the potential applications of these various functional nanostructures in nano-electronics and in the bio-medical field (nano-biosensors, drug delivery, tissue engineering...).

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- MAGNIN D., CALLEGARI V., MATEFI-TEMPFLI S., MATEFI-TEMPFLI M., GLINEL K., JONAS A.M., DEMOUSTIER-CHAMPAGNE S. Functionalization of Magnetic Nanowires by Charged Biopolymers. Biomacromolecules, 9, 2517, **2008**.

Patents

- Drug-eluting nanowires array, application number: PCT/EP2008/063803
- Method and device for high sensitivity and quantitative detection of chemical / biological molecules, application number: PCT/EP2008/059460

Funding

- European Union: *Erasmus Mundus IDS-FunMat International Doctoral School*
- UIAP-VI networks: *Functional Supramolecular Systems*
- Wallonie: *NanoTIC–Feeling, DEEP, BIOSE, RAPARRAY*
- F.R.S.-FNRS and FRIA

Partnership

- ULg
- UGent
- Université Paris 6
- Université Joseph Fourier Grenoble
- Institut des Matériaux de Nantes
- Companies: it4ip, Neurotech, Zentech

Main Equipment

- Potentiostats/Galvanostats
- Electrochemical Quartz Crystal Microbalance (EQCM)
- Electrochemical Impedance Spectroscopy (EIS)
- UV-vis-NIR spectrometer
- Raman Spectroscopy
- Access to: TEM, SEM, AFM, X-ray reflectometry, spectroscopic ellipsometry, FT-IR, XPS and QCM-D

Products and Services

Design, elaboration and characterization of (bio-)functional nanostructures with potential applications in nanoelectronics and biomedical fields

KEYWORDS

Nanomaterials
Electropolymerization
Conductive polymer nanostructures
Multi-segmented hybrid nanowires
Bio-functional nanotubes
Surface biofunctionalisation

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Nanoporous polymers by track-etching : applications in nano-objects synthesis and diffusion control fields

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- Roger LEGRAS

Research Field and Subjects

Track-etching technology is used to make pores in polymer following a two-step process consisting in -1- the irradiation of the polymer by energetic heavy ions to create linear damage tracks, and -2- the chemical etching of these tracks to well-controlled pores.

Fundamental understanding of track-etching process is investigated, and track characterization has been considered through *in-situ* analysis of heavy ion-irradiated polycarbonate (PC), model compounds and consecutive emitted gases.

Track-etching technology has been notably extended through successive European Commission projects to new types of porous nano-templates in order to synthesize nano-objects presenting advantageous property discontinuities arising from the nano-regime, and therefore explore their potential for innovative applications. Templated growth within tracketched material was therefore efficient for production of arrays of controlled metallic or polymeric nano-objects exhibiting unexpected microwave properties or spin-dependent phenomena (Giant Magneto-Resistance, Tunnel Magneto-Resistance…).

Today, track-etched membranes are realised from polymer films like polycarbonate, polyester (PET) and polyimide; characterized by a smooth flat surface, a sharp cut-off and a precise pore size covering a large range from 10 nm to more than 30 µm, these membranes are used in the pharmaceutical, cosmetic or food industries for the detection of micro-organisms, in the diagnostic field as a control barrier in glucose sensors, the detection of cancerous cells...

Integration of such track-etched membranes into specific devices is considered through projects funded by Wallonie. Electrical insulation combined to an improved protonic conductivity expected from the anisotropic porosity of the membrane is considered in the development of an innovative 'electrode-membrane' assembly for proton exchange membrane (PEM) type fuel cell.

Track etching technology has been also adapted to a thin polymer layer deposited on a support like conductive glass or gold-coated silicon wafers. These supported porous templates are therefore suitable for the synthesis of micro- or nano-materials and can then become an integral part of new specific devices: (1) implantable device incorporating electrode with quantitative drug eluting capacity, (2) integration of supported ZnO nanowires into new photovoltaic cells, and (3) synthesis of protein biochips dedicated to SFG detection.

Other developments target notably the production of track-etched membranes with tailored surface properties, as well as the realisation of track-etched membrane from new PVDF-based and polyimide-based polymer films.

To valorise this knowledge in track etching technology, a spin-off company (i.e. it4ip) was created in early 2006.

Representative References

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- LEPRINCE L., DOGIMONT A., MAGNIN D., DEMOUSTIER-CHAMPAGNE S. Dexa-methasone electrically controlled release from polypyrrole-coated nanostructured electrodes. Journal of Materials Science: Materials in Medicine 21(3): 925-930. **2010**.
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- XAVIER S. et al. Stable field emission from arrays of vertically aligned free-standing metallic nanowires. Nanotechnology 19(21): 215601. **2008**.

Patents

- Drug-eluting nanowire array. WO 2009050168
- Process for creating pores in a thin polyimide sheet. WO 2004045750, EP 1569742, US 7419612
- Method for creating pores and microporous plastic film created thereby. WO 2001049403, EP 1242169, US 20030020024
- Method for creating pores in a polymer material. WO 2001049402, EP 1242170, US 6861006

Awards

it4ip received the biotechnology award in Nanotech 2008 exhibition in Tokyo and won the prize for technological innovation in Wallonie in 2009.

Funding

- Following European funded projects were based on the development and the use of track-etched based materials: PTM&GMR (BRITE3), NANOPTT (GROWTH), NANOTEMPLATES, BARP+, HIPERCHEM (FP6-NMP).
- Actual projects are funded by "la Wallonie" through Marshall Plan, Biowin, Solwat, Waleo3 and First Hautes Ecoles.

Partnership

- Université de Liège (ULg)
- Université de Mons
- HELHa
- Certech asbl, Neurotech sa, Nanocyl sa, it4ip sa
- GANIL (Caen, FR)

Products and Services

Informations about track-etched products are available on : http://www.it4ip.be

KEYWORDS

Track etching
Track-etched membrane and template
Ion irradiation
Nanoporosity and microporosity
Polymer filter

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Nanowires and nanotubes formed in alumina nanotemplates

SENIOR SCIENTIST :

- Luc PIRAUX

Research Field and Subjects

Nanowires and nanotubes are central elements in nanoscience and nanotechnology for various applications such as nanoelectronic devices, optical components, interconnects for nanoelectronics, mechanical reinforcement, near field probes, super lubricating surfaces, biotechnology expands, chemical sensors, high-density data storage, field and light emitters…
Electrochemical template synthesis allows the realisation of high-density arrays of nanowires, nanorods, nanotubes and core/shell nanopillars, which is very difficult to obtain using conventional lithographic techniques. The methods developed by the research team based on template synthesis and on self organization seem to be the most promising for the fabrication of such nanostructures due to simplicity and low cost of this approach.

Among the most interesting nanoporous host materials used as templates, nanoporous alumina is very attractive. Porous alumina films are formed through anodic oxidation of aluminium using an acidic solution. Both self-supported alumina membranes and thin porous alumina layer supported on a silicon/glass substrate have been successfully prepared by the group at UCL. These templates contain cylindrical pores of uniform diameter that are perpendiculars to the surface. It has also been shown that a two-step anodization process can lead to highly ordered two-dimensional pore arrays with a hexagonal pattern over large areas.
The particular features of the alumina template (i.e. good mechanical stability and high-temperature resistance, the possibility of using DC electrochemical deposition inside nanopores, the easily tunable geometrical parameters such as pore diameters and interpore distances, and the possibility for a high-density ordered arrangement of nanopores) makes it an ideal candidate for further integration into large-scale fabrication of various nanowire-based devices.
In addition, the possibility of using alumina template to combine electrochemical synthesis with lithographic methods opens new ways for the fabrication of complex nanostructures.
Overall, these advantages make nanoporous anodic alumina a very promising template for the integration of the high-density nanowire-based structures in silicon processes for microelectronic manufacturing.
Work is in progress at UCL to go further both for realising specific nanostructures for fundamental studies and for applications, such as spintronics, magnetic recording, sensors and biosensors. As an example, a highly sensitive pH capacitive sensor has been recently designed by confined growth of vertically aligned nanowire arrays on interdigited microelectrodes.

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- MATEFI-TEMPFLI S., MATEFI-TEMPFLI M., VLAD A., ANTOHE V. and PIRAUX L., Nanowires and nanostructures fabrication using template methods: a step forward to real devices combining electrochemical synthesis with lithographic techniques, Journal of Materials Science: Materials in Electronics 20, S249. **2009**.
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Patents

Multiple bath electrodeposition - EP0111055.8 - 2002

Funding

- Belgium Interuniversity Attraction Pole on « Quantum Effects in Clusters and Nanowires »: PAI-IUAP P6/42 (2007-2011)
- Programme « Wallonie »: « NANOTIC » – projet « Feeling » (2005-2010)
- Action de Recherche Concertée: « Hybrid metal/organic Nanosystems », UCL (2007-2011)
- Programme « Wallonie » R&D SOLWATT: « NANOROD » (2009-2012)
- FNRS
- FRIA

Partnership

Unité Mixte de Physique CNRS/THALES THALES Palaiseau

Main Equipment

- High vacuum coating systems (sputtering and e-beam)
- Electrochemical set-up
- High Resolution Scanning Electron Microscopy & EDX probe

KEYWORDS

Nanotechnology
Nanomaterials
Nanotemplates & nanowires
Nano(bio) sensors

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http://www.nano.be/sites/default/files/PirauxL_PPT.pdf

Nanostructured materials and systems for electromagnetic absorption with multiscale architecture

SENIOR SCIENTISTS :

- Christian BAILLY
- Isabelle HUYNEN
- Thomas PARDOEN

Research Field and Subjects

Electromagnetic pollution is a matter of global concern in terms of the relationship between health and operation of a wide range of electrical devices. Indications of potential health problems due to electromagnetic pollution are indeed sometimes related. Electromagnetic interferences (EMI) can also have dramatic consequences on the functioning of electrical devices such as in medical or airspace applications.

In some situations a simple metallic foil is sufficient to reflect an incident electromagnetic wave and to preserve the electrical integrity of a system, or to avoid the electromagnetic radiations to escape the system.

In a series of applications, however, true absorption of the electromagnetic radiation, at least from one side of an interface, is recommended and sometimes mandatory. For some electronic circuits the self-reflection of the waves can be detrimental to proper operation. True electromagnetic absorption is also required in anechoic chambers for testing electronic devices, antennas … and in stealth military applications (naval or aircrafts that cannot be detected).

Composite materials emerge naturally as the solution when seemingly antagonist properties are required. Most polymers are electrical insulators such that they are transparent to electromagnetic radiations. Reinforcing polymers with carbon-based conductive loads such as carbon fillers, carbon nanotubes (CNT), carbon black, graphene platelets or a mixture thereof constitutes an attractive option to reach the required level of conductivity, around 1 S/m, responsible for electromagnetic absorption by power dissipation at high frequency. Adequate dispersion and sometimes orientation of the nanoparticles in polymers are essential to reach optimal properties. This represents a significant challenge in that there are strong interactions between the particles in the matrix. However, the dielectric constant is increased by the incorporation of carbon-based particles, which has a detrimental impact on the reflectivity of the materials. In order to benefit from the conductivity of the carbon-based conductive loads while not increasing too much the dielectric constant, different strategies are implemented. One can introduce open space to the material. Foaming is thus very effective to limit the detrimental impact of carbon loads on the dielectric. Another strategy consists in combining different layers of graded concentration in conductive nanofillers in order to induce a progressive increase of the dielectric constant and conductivity, and reduce the input reflection. Finally, the nanocomposite can be combined with other metallic fabrics conferring exceptional mechanical, electromagnetic and thermal properties to the resulting hybrid.

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Patents

- WO2008.068042.A2 “Polymer composite material structures comprising carbon based conductive loads”.
- EP10175224 “Process for preparing electromagnetic interference shielding materials”.
- EP10175887 “Hybrid material for electromagnetic absorption”.

Funding

- DGTRE/DGO6 Wallonie projects Multimasec, ATAC
- FP7 Project Harcana (http://www.harcana.eu/)
- FNRS and FRIA

Partnership

- Emerson & Cuming, Westerlo, Belgium
- Nanocyl S.A., Sambreville, Belgium
- Thales Alenia Space Etca, Belgium
- Alstom, Charleroi, Belgium
- Future Carbon, Germany
- Université de Liège, Belgium
- SEE, Belgium
- MS3, Belgium

Main Equipment

- Network analyser combined with cryogenics and magnetic field facilities
- WELCOME Measuring Facility
- Small scale nanocomposites processing
- Morphological and Mechanical characterization facilities

Products and Services

- Processing of thermoplastic and thermoset polymers and (nano)composites thereof at laboratory scale
- Rheology, TEM and SEM characterization of nanocomposites and nanofillers
- Characterization of electromagnetic properties of composite materials over a broad frequency range (DC to 100 GHz).

KEYWORDS

Electromagnetic characterization
Microwave absorbers
Nanocomposite hybrid materials
Carbon nanotubes

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Multifunctional molecular based magnetic materials: nanoparticles and nanoporosity

SENIOR SCIENTISTS :

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- Anil D. NAIK

Research Field and Subjects

The design, synthesis and study of novel supramolecular functional coordination networks constitute promising research fields in which the laboratory of molecular chemistry is largely involved. The quest of bistable magnetic molecular materials exhibiting highly cooperative thermal spin transitions is of great appeal in nanosciences since these compounds could be proposed for practical applications for display and data processing due to their reversible thermochromism and wide memory domain often occurring at room temperature. The laboratory is also concerned with the synthesis of photochromic switchable magnetic materials operating in the solid state to extend the range of external triggers. Novel routes to the synthesis of bistable nanoparticules are delineated, in particular through the use of biological membranes to seed nanoparticules of various sizes. Such deposited membranes are used to fabricate thin films using unconventional methods based on micro-contact printing. Finally, a quest for relationships between crystal structures and physical properties, in particular nanoporosity of extended polymeric coordination networks, is pursued with the aim of deriving novel multifunctional materials.

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Funding

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Partnership

- Presidency of GFSM 'Groupe Francophone de Spectrométrie Mössbauer'
- Member of GDR 'Magnétisme et Commutation Moléculaire'

Main Equipment

- Mössbauer spectrometers with cryogenic facilities (Fe, Sn, Eu)
- Differential scanning calorimeter with a liquid nitrogen cryostat
- Diffuse reflectance spectroscopy with a liquid nitrogen circular cryostat
- Solid state fluorimetry with a liquid nitrogen bath cryostat

Products and Services

Synthesis and characterization of nanomaterials.

KEYWORDS

Functional materials
Spin transition
Thermo/photochromism
Molecular electronics
Nanoparticles
Nanoporosity
Thin films
Microcontact printing

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Structural and Chemical modification of Carbon-Based Materials

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- Stéphane LUCAS
- Robert SPORKEN

Research Field and Subjects

Carbon-based nanomaterials (nanotubes (NT), graphene) have raised tremendous interest in recent years both from fundamental and technological perspectives. For example, charge and spin transport have received a great deal of attention as conventional technology for microelectronic has begun to reach its scaling limit. However, their widespread practical application could be hampered by the difficulty to grow a precise type of NT (metallic or semiconductor) or by the metallic character of all graphene sample. The low reactivity of the all-carbon materials could also curb their integration as gas sensors and electrical connects. The research focuses on fundamental investigation of the chemical and structural modification of carbon nanotubes and graphene surfaces. The final goal is a refined control of the conductivity and chemical reactivity of these systems.

Aligned nanotubes and graphene are synthesized by the exposure of transition-metal catalysts to a hydrocarbon gas at a controlled temperature in a Chemical Vapour Deposition set-up. A key challenge in this field is to tailor the thickness of the catalyst deposition.

Chemical modifications are obtained during the growth process (by changing the initial gas composition) or via a post-treatment, based on plasma activation techniques.

Graphene nanoribbons (GNR) are stripes of graphene of a few nanometre width. The electronic transport properties of a GNR strongly depend on the crystallographic orientation, the quality of its edges and the number of layers. Recently, a new Scanning Tunnelling Microscope (STM) based lithography method for cutting carbon nanoribbons has overcome some of the above-mentioned difficulties.

Many fundamental questions remain open on the structural and chemical modification of carbon nanostructures. How does the exact atomic configuration around the dopant or on the edge of a ribbon affect the physical properties of the material? What are the microscopic/spectroscopic signatures of such modifications? How does the (not modified) carbon layer below the surface one influence the physical properties or their analysis? Those questions are tackled by experimental and simulation tools.

Mainly electron microscopy, electron spectroscopy and conventional surface analysis methods are used. Amongst them, a careful analysis by STM allows to investigate the exact atomic local configuration and electronic properties.

Ab initio and semi-empirical simulations are performed to interpret the experimental results and to predict the local chemical bonding and the global physical properties (including vibrational structure and quantum transport).

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Funding

- EU - STREP BNC TUBES
- NATO linkage grants
- FNRS - HAS agreements

Partnership

- Université Paris-Diderot
- Onera
- Université de Mons
- University of Antwerp
- Research Institute for Technical Physics and Materials Science, Budapest, Hungary .
- Faculty of Physics, University of Sofia, Bulgaria.

Main Equipment

- I-SCF Scientific Computing Facilities
- UHV STM, Photoemission microscope (PEEM)
- Plasma vapour deposition (PVD)
- Chemical vapour deposition (CVD)
- Electron microscopies

Products and Services

- Lab-scale production of aligned and modified carbon nanotubes
- Characterisation of carbon nanosystems (Electron Microscopy, STM)
- Simulations of structural and electronic properties of carbon nanosystems

KEYWORDS

Carbon nanotubes
Graphene
Chemical Vapor Deposition
Functionalisation
Plasma
Electronic Structure
Quantum Transport
Scanning Tunneling Microscopy

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Carbon Nanotubes and Nanocomposites

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- Zineb MEKHALIF

Research Field and Subjects

The main objective of the research is the design and elaboration of surface and interfacial materials. Carbon nanotubes (CNTs) are nano-objects with remarkable intrinsic properties (mechanical stiffness, excellent thermal and electrical conductivity, high aspect ratio…) which are of particular interest in the fields of nanoscience and nanotechnology. However, their insolubility in most solvents and their low chemical reactivity hinder their manipulation and applications in many potential fields. Surface functionalisation is a direction to overcome some of these problems. The current activities of the laboratory in the specific field of CNTs are, as follows:

- Synthesis of carbon nanotubes (CCVD, laboratory scale).
- Functionalisation of carbon nanotubes: by ball-milling and by chemistry in solution (oxidation and silanisation).
- Incorporation of carbon nanotube into polymer (polyacrylonitrile, silicone), metallic (nickel, copper) matrices and in metal oxides (Ta_2O_5). In the case of metallic matrices and metal oxides a great deal of the research efforts is focused on electrodeposition and sol-gel processes.
- Study of the dispersion of the carbon nanotubes, pristine and chemically modified, in various solvents.

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Patents

- Nanocomposites, their manufacture and uses. EP 02447039.5 – 20/03/2002, WO 03/07/8315 A2- 25/09/2003

Funding

- Wallonie, FNRS, Europe, Industries

Partnership

- TOTAL Petrochemicals, CSL (ULG), NANOCYL, ESA, SIRRIS

Main Equipment

- Equipments for CNTs synthesis (CCVD)
- Equipments for CNTs functionalisation
- Access to equipments for CNTs characterisation

Products and Services

- CNTs synthesis (lab scale)
- CNTs functionnalisation (lab scale)
- CNTs characterisation
- CNTs buckypapers and nanocomposites fabrication

KEYWORDS
Carbon nanotubes
Functionalisation
Nanocomposites

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Production of unagregated nanoparticles with unimodal size distribution

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Research Field and Subjects

Thanks to their unique chemical, mechanical and electrical properties, nanoparticles are of great interest in many applications such as life science, nanoelectronic devices, field emission sources, scanning probes, chemical nanosensors, hydrogen storage, nanoconveyors, and so on. One of key factors for application is to master their chemical composition, size and degree of aggregation.

Thanks to numerous fundamental and applied research contracts a unique way to synthesize non aggregated nano-particules is developed . Two main objectives are being pursued: the synthesis of unagregated cold nanoparticles with unimodal size distribution, and the synthesis of radioactive nanoparticules that incorporates at least two type of radioactive atoms. Up to now, Ag, Cu, Au, Pd, Rh, TiO_2, C, Fe, Co, Cr, FeCo, FeCr nano-particules (size < 30 nm) have been produced on a regular basis.

Applications of such particles lie in material science and life science. In relation to the latter one can cite TARGAN, a project to develop a nanocluster containing several radioactive atoms, coupled to a monoclonal antibody targeting a specific marker of the tumoral endothelial cells. Radioactive nanoparticles are also functionalised *in situ* using plasma deposited polymerized allylamine or PEG films.
With regard to material science, one can cite the use of our nanoparticules as catalysts to grow aligned nanotubes and their use as a photocatalytic coating to promote photodegradation of organic polluant or material.

Representative References

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Patents

- RADIOACTIVE DEVICE, 2006, EP1838354, WO2006063418.

Funding

Wallonie (DGO6), MARSHALL Plan, FNRS, FRIA, Industry

Partnership

- IBA-Molecular (Fleurus)
- Institut des Radioéléments (IRE) (Fleurus)
- Arcelor Mittal Research Center (Liège)
- AGC (Jumet)

Main Equipment

- Nanoparticle source (PVD) with in-line mass spectrometer,
- Tandetron accelerator and associated analytical analysis equipment
- Nanoparticle measurement techniques (TEM, SEM, BET, Centrifugal disk, …)

Award

Accreditation by the European Commission (Institut des matériaux et mesures de références) for the characterization of nanoparticles.

KEYWORDS

Nanoparticles
Physical Vapor Deposition
Monte-Carlo Simulation
Functionalisation

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Living hybrid materials for environmental remediation, green energy and smart cell-therapy

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- Christophe MEUNIER
- Pierre VAN CUTSEM
- Jean-Pierre DESCY
- Carine MICHIELS

Research Field and Subjects

Living cells can be considered veritable complex molecular engines spatially enclosed and integrating the environmental factors by signal transduction pathways. Nonetheless, cells, isolated from their native environment, are generally fragile or unsuitable in the development of new technologies.

The present project proposes to take advantage both of the efficiency of living systems and of the stability of abiotic nanomaterials. In particular, this interdisciplinary research field based at the University of Namur, and including chemists and biologists, proposes a fundamental survey on the *in situ* immobilisation of cyanobacteria, algae, plant cells and animal cells within inorganic cages (SiO_2, TiO_2, Al_2O_3, etc.) in order to target innovative and sustainable functional materials. The nanostructured porous matrices molded on the shape and size of cells are formed via the sol-gel process. The control of the porosity at nanometric scale is crucial since the pores should enable the diffusion of the nutrients and the release of the metabolites, and should also provide a physical barrier to protect the encapsulated cells towards predators or harsh environmental conditions.

More than just protecting the biological species, the aim of this project is also to control the interfacial properties of cells at the molecular level. A better understanding of the cell-matrix interface will allow establishing ideal conditions of encapsulation to sustain life, the main challenge of this research field. Additionally, the integration of cells could provide a way to regulate and to functionalize cells.

Currently, the expertise developed by this research platform has led to significant progress in cell encapsulation technology. Promising applications such as photo-bioreactors, biofuel cells, depolluting devices, cell storage and artificial organs are explored and developed. For instance, the design of a material which encapsulates animal cells into an inorganic matrix could be used in a near future to treat type I diabetes.

Representative References

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Funding

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Partnership

This research platform collaborates with the LCMCP team (Paris, France).

Main Equipment

- Volumetric adsorption analyser Tristar 3000 and Autopore III *mercury porosimeter* for studying the textural properties of materials (pore size, surface area, pore volume).
- *Solid-state MAS-NMR-spectrometer.*
- *Confocal fluorescent microscope (Leica, TCS-SP)*
- Clark cell vessel (Oxy-lab) for measuring the photosynthetic or respiratory activities.

KEYWORDS

Encapsulation
Nanoporous matrix
Inorganic material
Living cell
Cell therapy
Cell storage
Photosynthesis
Photobioreactor

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Supported nanoparticles for heterogeneous catalysis derived from molecular precursors

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- Sophie HERMANS

Research Field and Subjects

At the dawn of the third millennium there is an increasing concern regarding the impact of human activities on the environment. At the same time developed countries cannot live without the material framework of modern life. Hence the chemical industry is faced with the challenge of providing the same commodities with environmental and economical savings. In this context, heterogeneous catalysis is a solution for chemical transformations at lower temperature and pressure with less waste, and ideally endless reusability. It has been demonstrated that in this field supported phases need to be in nanoparticulate form to be active. Hence, the control of the size and composition of nanoparticles supported on solid materials is a vivid area of research.

The research group has devised synthetic strategies for nano-particles heterogeneous catalysts based on molecular precursors. The advantage of this method is the possibility of controlling the characteristics of the final materials through well-defined precursors that can be prepared and characterized before incorporation on the support. The fate of the precursors during activation and applications in reactions of industrial relevance can be followed precisely by *in situ* or pseudo-*in situ* techniques.

The particular area of bimetallic catalysts has been extensively studied by this group. Additional challenges arise from the fact that it is very difficult to mix two metals at the nanoscale with perfect homogeneity of size and composition in the final nanoparticles. Molecular heterometallic cluster compounds have been thus envisaged as precursors because they contain a core of metal atoms surrounded by a sheath of ligands. The ligands allow the clusters to be handled easily, and impregnated or grafted on surfaces in solution. The metal core can be devised in such a way that it contains the desired number and type of metal atoms. A soft thermal treatment removes the ligands, leaving the active naked nanoparticles on the support.

Finally, supports and surfaces have also been modified extensively to permit optimal interactions with the molecular precursors. Indeed nano-sized particles can only be ensured if no agglomeration occurs during post-treatment. Chemical groups functioning as anchors for molecular precursors have been built on the surface of carbon materials including carbon nanofibers and nanotubes. The precursors -being metal complexes- can be fixed via ligand exchange for these functions. Depending on the type of precursor envisaged, oxygenated or phosphine-type groups have been the focus of this work.

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Awards

- *Outstanding work in precious metal research Award* delivered by the International Precious Metals Institute (IPMI).
- *Best Presentation Award* at the Symposium "Nanotechnology in Catalysis", 221st National ACS Meeting, San Diego, USA.
- *Young Scientist Prize* of the International Association of Catalysis Societies (IACS).

Funding

This research is funded by Federal Government (PAI INANOMAT 6/17) and FNRS (FRFC Convention n° 2.4574.10).

Partnerships

- The laboratory is involved in the Belgian INANOMAT network of teams working in the field of Inorganic Nanostructured Solid Materials.
- Active collaborations are sustained with:
  - the University of Liège,
  - the Meurice Institute in Brussels,
  - the University of Parma in Italy,
  - the State University of New York in Albany, USA.

Main Equipment

- FTIR-DRIFT spectrometer, UV-VIS-NIR spectroscopy (solution and solids), Fluorescence spectroscopy (solution and solids), FT-Raman spectrometer, Confocal Raman spectrometer, Atomic absorption spectrometry, Thermal analyses: combined thermogravimetric, differential thermal analysis and mass spectrometry (TGA-SDTA/1100°C), Differential scanning calorimeters, Physisorption / chemisorption analyzer for specific surface area and porosity measurements.

Products and Services

The team is competent in inorganic (molecular and solid materials) and organometallic syntheses, which means that the laboratory is equipped with standard vacuum lines for Schlenk techniques, two glove boxes and a range of ovens (including tubular).

KEYWORDS

Catalysis
Characterization of solids
Clusters
Materials
Nanoparticles
Nanostructured solids
Nanotubes
Surface Functionalization

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Non conventional methods for the preparation of multicomponent nanostructured oxides

SENIOR SCIENTISTS :

- Michel DEVILLERS
- Eric GAIGNEAUX

Research Field and Subjects

The research groups have acquired a strong expertise in the synthesis of bulk or dispersed oxides. Beside mastering classical procedures (impregnation, co-precipitation, 'citrate method', deposition-precipitation etc) - some of them being also achieved at the pilot scale, non conventional approaches are being developed based on the use of specially designed molecular precursors, organic-inorganic hybrids, and modified sol-gel approaches. These approaches are actually based on a molecular level chemistry and thus constitute a real field of nano-engineering. The aim is to prepare materials with dimensional features in the nanometer range : films with thickness up to 100 nm, pores in bulk materials and films with mouths smaller than 20 nm, and supported active metal/oxide particles smaller than 10 nm.

Most studies deal with oxides containing transition metals of groups 5 (V, Nb, Ta) and 6 (Mo, W), sometimes combined with other elements. These oxides often display complex formulations and a polymorphism which is difficult to master. These materials generate a broad interest due to their numerous outstanding functional properties, such as ferroelectrics, ion conductors or heterogeneous (photo)catalysts. In this area there is clearly a demand for developing new chemical routes able to produce these materials with high purity under various forms : bulk phases with adequate morphologies and textural properties, highly dispersed supported phases for catalytic applications, or thin films.

Intensive research has been dedicated to the systems based on niobium and tantalum. Namely, to compensate for the poor availability of water-soluble Nb and Ta compounds, new stoichiometrically well-defined Nb/Ta coordination compounds (oxo- or peroxo-complexes with high denticity carboxylate or polyamino-carboxylate ligands) were developed, which open new perspectives as molecular precursors for Nb-M mixed oxides, with M = V, Mo, Ta, Y or Bi. Efforts were also made to prepare heterometallic compounds associating Nb and Ta in which peroxo groups are present together with other multi-dentate carboxylate ligands. They were used as single-source precursors in solid materials synthesis. Phases that are difficult to prepare could be stabilized under moderate conditions and solid solutions were obtained in many cases over a wide composition range. The nature of the precursor used was found to allow the control of the morphology of the oxide particles and of their micro- and mesoporosity (pore mouth below 2 nm and between 2 and 20 nm, respectively).

Another route to prepare mixed oxides (V-Mo-Ti, and Mo-Al) was developed via a modified non-hydrolytic sol-gel approach. It starts with the sol-gel preparation of a homogeneous multimetallic solid precursor. The proper adjustment of the parameters of the subsequent thermal treatment allows controlling the eventual enrichment of the surface of the solid in one of the involved elements. Supported phases are thus advantageously obtained in a one-pot procedure, as monoatomic (sub-nanometer) species dispersed on the support.

Finally, a promising and widely applicable pathway to prepare M-M-O systems was based on homogeneous hybrid inorganic-organic mixtures in which specially-designed ampholytic copolymers (associating monomers with carboxylate groups and others with amine moieties) are used as matrices to stabilise inorganic species in aqueous solutions. These hybrid systems were then used as novel precursors for a wide variety of inorganic materials under soft conditions. Hybrid materials are prepared from these copolymers and stoichiometrically well-defined inorganic salts or coordination compounds. This new route has been applied successfully in different model systems such as bismuth vanadates ($BiVO_4$, $Bi_4V_2O_{11}$) as bulk phases or thin films, and bulk $(Nb_{1-x}Ta_x)_2O_5$ and $Nb_2Mo_3O_{14}$ phases. Currently this investigation is extended to mixed (Bi or Ni or Co)-Mo oxides with as objective the control of their textural properties towards dedicated heterogeneous catalytic applications. Obtaining features with a periodicity in the range of 1 nm is the objective.

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Funding

Financial support is provided by the Federal Government (PAI INANOMAT n°6/17), the Communauté française (ARC 08/13-009), and F.R.S.-FNRS (FRFC Conventions n° 2.4540.05 and 2.4508.08).

Partnerships

‣ The laboratories are involved in the Belgian INANOMAT network working in the field of Inorganic Nanostructured Solid Materials.
‣ Active collaborations in this field are sustained with other UCL teams,
‣ The University of Montpellier, France,
‣ The University of Parma, Italy

Main Equipment

‣ FTIR-DRIFT spectrometer, UV-VIS-NIR spectroscopy (solution and solids), Fluorescence spectroscopy (solution and solids), FT- and Confocal Raman spectrometers, Atomic absorption spectrometry, X-ray (thermo)diffraction and fluorescence, X-ray photoelectron spectroscopy, Thermal analyses: combined thermo-gravimetric, differential thermal analysis and mass spectrometry, Differential scanning calorimeters, Physi- and chemisorption analyzers for specific surface area and porosity measurements.
‣ Equipment for pilot scale preparation and shaping (extrusion and pelletization).

Products and Services

The groups have an expertise for the synthesis and characterization of coordination compounds and inorganic solids. The laboratories are equipped with a range of ovens (including tubular), standard analytical equipment, and spectroscopic equipments for solutions and solid state measurements, including under working conditions (high temperature, under gas flow). Beside, an expertise in the pilot scale preparation and the shaping of the cited materials is available.

KEYWORDS

Coordination compounds
Characterization of solids
Hybrid materials
Nanostructured solids
Transition metal oxides
Scaling up
Thin films

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Thin films of (mixed) oxides with controlled composition, homogeneity, crystallinity and nanoscale porosity

SENIOR SCIENTIST :

- Eric GAIGNEAUX

Research Field and Subjects

Films with thicknesses ranging from a few nanometers up to a few hundreds of nanometers are often used as coatings on steel, glass and polymers in order to give these substrates various properties such as: resistance against corrosion, biofouling and scratches, antibacterial properties, optical properties, thermo-, electro- and hygrochromism, etc. Such films are also sometimes used as materials to simulate the surface of heterogeneous catalysts.

For all of these applications, beside the thickness of the films, it is often necessary to control their composition and homogeneity, as well as their crystallinity – should the films be amorphous or crystalline, and if they should be crystalline, of which crystalline symmetry they should be –, and/or their porosity – should the films be dense or porous, and if they should be porous, what should the size of the pore mouths be –, should they be connected, what should be their total volume, etc.

The research group has acquired great expertise in the preparation of thin films of (mixed) oxides with controlled crystallinity, composition and homogeneity, and/or porosity in the nanometric range. Thin films are produced by spin-coating adequate precursors solutions, and applying further heat treatments. Combining the sol-gel approach and spin-coating is also sometimes applied.

In the context of MoO_3 crystalline films, the group demonstrated that it was possible to obtain films no more than one crystal thick. In fact, their thickness can be tuned down to 30 nm. Beyond this threshold, voids appear, leaving some parts of the substrate uncovered. By varying the nature of the spin-coated Mo precursor (heptamolybdate, citrate, oxalate, peroxopolymolybdate), it was demonstrated that the size of the crystals forming the films can be adjusted, as well as their morphology, which could be tuned as 'isotropic' or 'platelet'.

In the next step, the group proved that thin films of mixed oxides can be produced. Bismuth, cobalt, nickel and iron molybdate films were prepared by spin-coating mixed citrate complexes. When it was applied, it was shown that the proper adjustment of the composition of the spin-coated solution and the heat treatment applied subsequently, allowed the preparation of homogenous films with well-defined stoichiometry (for instance concerning bismuth molybdates: $Bi_2Mo_3O_{12}$, $Bi_2Mo_2O_9$ or Bi_2MoO_6) and/or crystalline symmetry (for instance concerning nickel molybdates : alpha or beta $NiMoO_4$).

Beside transition metal oxides, the group extended its expertise to silica, titanium and alumina, for which it was required to generate some porosity. To do so, the sol-gel approach was used. It was shown that, playing with the sol parameters, and the nature and concentration of the surfactants added to the sol, it was possible to generate micropores (pore mouth less than 2 nm) or mesopores (pore mouth between 2 and 50 nm) in typically 100 nm thick films. The use of porogens (as for instance latex beads, starch grains, etc) was also shown to allow the generation of macropores (pore mouth greater than 50 nm). Finally, when multilayers were applied to the substrates, the combination of adequate surfactants and porogens led to interconnected porous networks.

All the films mentioned above were developed by the group as representative of their respective families, but are in no way exclusive. The expertise can easily be extended to other compositions.

Representative References

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Funding

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(ARC 08/13-009), and the 'Hygrochrom' Winnomat-2 project funded by the DGO6 (Direction générale des Technologies, de la Recherche et de l'Energie) (Convention n°716676).

Partnerships

- The laboratory is involved in the Belgian INANOMAT network of teams working in the field of Inorganic Nanostructured Solid Materials.
- Active collaborations in this field are also sustained with other teams of the Université de Mons.
- Besides, the laboratory is involved in the European Multifunctional Materials Institute (EMMI) initiated from the former EU-FP6-Network of Excellence FAME.

Main Equipment

- Equipment for the preparation of the films : spin-coaters, UV-ozone chamber, clean furnaces and drying ovens (including under controlled atmospheres and vacuum).
- Equipement for the physico-chemical characterization of the films : X-ray photoelectron spectroscopy (surface composition, oxidation state of elements present, mapping), confocal FT-Raman (including mapping) and FTIR spectrometers, X-ray (thermo)diffraction (nature of crystalline phases present, crystal size), scanning probe microscopes (AFM, STM), accesses to UV-Vis spectrometer, electron microscopes (SEM-EDX), and contact angle measurements (hydrophili-phobicity).

Products and Services

The laboratory proposes its expertise and equipment for the preparation and the physico-chemical characterization of thin films made of oxides, with controlled crystallinities and porosity (micro, meso and macropores). The laboratory is equipped with a range of ovens (including tubular), analytical equipment, and spectroscopic solid state measurements, including under working conditions (high temperature, under controlled atmosphere and vacuum).

KEYWORDS

Alumina
Characterization of thin films
Coatings
Micro- and mesoporous films
(Mixed) (transition) oxide films
Multilayer films
Silica
Titanium

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WINFAB – Technological Platform – Micro- and Nano-Fabrication

SENIOR SCIENTISTS :

- Romain DELAMARE
- Mathieu VANDEN BULCKE
- Denis FLANDRE
- Jean-Pierre RASKIN

Research Field and Subjects

WINFAB (Wallonia Infrastructure Nano Fabrication) is the UCL technological platform active in micro and nano-fabrication. It is an inter-institute platform in the Sciences and Technology Sector. Main research activities include SOI (Silicon-on-Insulator)-CMOS integrated circuit processing, Micro(Nano) Electromechanical Systems (MEMS/NEMS), nanoelectronics, organic electronics, photovoltaics and biosensors.
Besides processing, activities are also carried out on materials development and characterization.
The infrastructure occupies about 1000 m^2 of clean environment (ISO 5), over two levels and 13 zones.
Overall process line is based on 3 inch silicon wafers, but some equipments can accommodate smaller and larger wafer sizes, as well as glass slides. The panel of available equipment allows users to experiment standard and non standard micro-nano-fabrication process steps, applied to various substrates, but also more applications oriented processing aiming at modifying or adding properties to selected materials.

Funding

Funding from la Wallonie and the European Union, through several programs or projects :

- NANOTIC – Programme d'excellence de la Wallonie, 2005-2010
- FEDER, programme compétitivité, portefeuille MINATIS
- SKYWIN and MECATECH, Pôles de compétitivité
- NANOSIL, European Network of excellence
- TRIADE, FP7 Aero project …

Funding from the Belgian National Fund for Scientific Research (FNRS)

Partnership

- As technological platform, WINFAB opens its infrastructure to external users from university or industry.
- Technical training for teachers, workers, students, and unemployed via Technifutur (www.technifutur.be/formation_entreprise.asp).

Main Equipments

- Cleanroom Environment
- Characterization (microscopy, ellipsometry, profilometry…)
- Wet Benches (substrate cleaning, wet etch…) and dry etching (RIE, DRIE)
- UV Lithography (Spin coating, Exposure, Development)
- E-beam nanolithography
- Nano-imprint lithography
- Furnaces (Oxidation, LP CVD Nitride and PolySi, Anneal…)
- Thin film (reactive sputtering, evaporation, Atomic Layer Deposition, PE CVD Nitride and Oxide…)
- Ion Implanter
- Glove boxes
- MEMS release (CPD)
- Pre- / Post processing (wafer grinding, CMP…)
- Packaging (Dicing, wire bonding…)

Products and Services

- Clean work environment
- Available process steps (cleaning, etching, lithography, characterization…)
- MEMS, Sensors and SOI CMOS Processing
- Education and training

KEYWORDS

Cleanroom
CMOS
MEMS
Microelectronics
Nanoelectronics
Organic electronics
Photovoltaics
Sensors
SOI

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Nanostructured surfaces for cell engineering

SENIOR SCIENTIST :

- Christine DUPONT

Research Field and Subjects

Creating environments mimicking the extracellular matrix, which is highly organised on micro- and nanoscales, is an important challenge for biomaterials science and tissue engineering.

The general aim of ongoing research projects is the design of biomimetic surfaces through controlled protein assembly. This includes the following working lines:
(i) creation and characterisation of surfaces with a nano-architecture which can organize adsorbed proteins,
(ii) design of methods to induce protein assembly,
(iii) development of analytical tools to probe the nanoscale organisation of interfaces, and in particular the orientation, conformation and supramolecular organisation of adsorbed proteins.
The biomimetic surfaces obtained are then further used to trigger particular cell behaviours, in collaboration with cell biologists. This could hopefully lead to the design of biomaterials with new properties or to the development of new strategies for tissue engineering.

The techniques and methods involved in the research include the preparation of nanostructured organic surfaces and protein layers, surface characterisation by atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) and wetting measurements, *in situ* monitoring of adsorption events by quartz crystal microbalance (QCM-D), and cell culture experiments.

Representative References

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Patents

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- Method for controlling the morphology of a polymer surface and said obtained polymer surface. PCT International Application 2001-EP14862 20011217.

Funding

- National Foundation for Scientific Research (FNRS)
- Foundation for Training in Industrial and Agricultural Research (FRIA)
- Wallonie
- Université catholique de Louvain
- Federal Office for Scientific, Technical and Cultural Affairs (Interuniversity Poles of Attraction Programme)
- European Community
- Industrial partners

Partnership

- Industrial partners:

AGC Flat Glass Europe
Cardiatis
DiaSource
GSK Biologicals
THT

- Member of:

Wallonia Network for Nanotechnologies
(NANOWAL)

Main Equipment

- X-ray photolelectron spectrometers (XPS)
- Atomic force microscopes (AFM)
- Quartz crystal microbalance (QCM-D)
- Wetting measurements

Products and Services

- Expertise in surface characterization and modification at the nanometer-scale
- Surface chemical analysis (XPS)
- Wetting properties (surface tension, contact angle, wetting dynamics)
- Surface nanostructure (AFM)
- *In situ* monitoring of adsorption (QCM-D)

KEYWORDS

Biosurfaces
Nanostructured surfaces
Protein adsorption
Surface characterisation
Cell culture
Biomaterials science
Biocompatibility
Biomimetism

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Functional Thin Films and Patterned Surfaces for Applications in Biosensing, Organic Electronics, and Smart Coatings

SENIOR SCIENTIST :

- Alain JONAS

Research Field and Subjects

The development of hybrid systems such as organic non-volatile memories, nanowire-based biosensors or responsive smart surfaces critically rests on our ability to assemble organic functional macromolecules in thin films or in nano-objects of controlled size at specific locations on a substrate, preferably with some degree of control being achievable on the orientation of the molecules for optimal performance.

Over the last 10 years, the research group has focused on developing a general-purpose toolbox allowing them either to coat surfaces with a range of functional organic layers, patterned or not, or to produce nanostructures of functional polymers over inorganic and organic surfaces. Emphasis is given to the simplicity and speed of the procedures. Selected examples of methods, systems and targeted applications are given in the table on the below, which contains the expertise linked to this set of projects. Associated with these systems are advanced characterization tools, which belong to the UCL Bio- and Soft Matter Division of the Institute of Condensed Matter and Nanosciences (IMCN/BSMA) and are operated in cooperation with a wide range of senior scientists of the division. Among these techniques, the research group is more specifically (co)managing tools such as X-ray reflectometry, ellipsometry, transmission and scanning electron microscopy, electron beam nanolithography, nano-imprint lithography, and a platform for the chemical modification of surfaces by (bio)organic components.

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- JONAS A. M., HU Z., GLINEL K., HUCK W. T. S. Effect of Nanoconfinement on the Collapse Transition of Responsive Polymer Brushes. Nano Letters 8, 3819-3824, **2008**.

Methods	Systems	Applications
Layer-by-layer assembly (LbL)	‣ Nanostructured coatings of synthetic and bio-polymers ‣ Nano-containers made by LbL templating	‣ Super-hydrophobic surfaces ‣ Nano-compartmentalized films ‣ Corrosion protection ‣ Coupling layers for proteins
Grafting from/to surfaces (ATRP)	‣ Polymer brushes ‣ Silane monolayers	‣ Smart surfaces responding to photons, pH, temperature ‣ Coupling layers for biomolecules or functional molecules
Nanolithography	Nanopatterned surfaces: ‣ topography ‣ chemistry	‣ Templating surfaces for self-assembly or cell growth
Nano-imprint lithography (NIL)	Nanostructures of functional polymers: ‣ conducting ‣ semiconducting ‣ ferroelectric ‣ …	‣ organic FeRAMs ‣ biosensors ‣ nanowire transistors

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Patents

- Method and device for high sensitivity and quantitative detection of chemical/biological molecules, European Patent Application EP-07014037.1; US Patent Application US 12/669,191.
- Ferroelectric Organic Memories with Ultra-low Voltage Operation. GB Patent Application GB0809840.2.; European Patent Application PCT/EP2009/ 056656.

Awards

- Leverhulme visiting professorship (2006-2007, University of Cambridge).
- Runner-up Obducat Prize 2007 for "Nanoimprint beyond lithography".

Funding

<u>As principal investigator (2010):</u>
- EU FP7 Strep MOMA, *Embedded Organic Memory Arrays*.
- Fondation Louvain, *Soft ferroelectric memories*.
- PAI-VI *Functional Supramolecular Systems*.
- ARC DYnanoMOVe, *Controlling Motion and Dynamical Phenomena at the Nanometer Scale*.

<u>As associated investigator (2010):</u>
- RW NANOTIC–FEELING Excellence Program. *Development of new sensing systems for biological applications*.
- EU FP7 Erasmus Mundus Action IIB. *International Doctoral School IDS-FunMat – Materials for Energy, Health and Information Technology*.

Partnership

- Most Belgian Universities (within the frame of PAI FS2)
- Foreign Universities : Groeningen (NL), Cambridge (UK), Bordeaux (FR), Paris 6 (FR), Grenoble INPG (FR), Lausanne EPFL (CH), Nebraska (USA), Nevada (USA), Soochow (Suzhou, China)
- Companies: Solvay Solexis, STMicro-electronics, IMEC, Holst, it4ip, EADS.

Main Equipment

- Transmission and scanning electron microscopes
- X-ray reflectometry
- Spectroscopic ellipsometry
- Platform of surface functionalization (surface polymerization, self-assembled monolayers, robotized layer-by-layer assembly)
- Electron beam nanolithography
- Nanoimprint lithography

Products and Services

Characterization and fabrication tools for thin films, coupling layers, nanopatterned surfaces, etc.

KEYWORDS

(Nano)patterning
Smart coatings
Biosensor
Organic electronics
Biofunctionalisation
Superhydrophobic
Self-cleaning surfaces

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Self-assembled nanofilms on metal and metal oxide surfaces : fundamental research and applications

SENIOR SCIENTISTS :

- Zineb MEKHALIF
- Joseph DELHALLE

Research Field and Subjects

Self-assembly is a modern bottom-up approach used to construct materials on a nanoscale. Molecular self-assembly on surfaces has become a remarkable method for tailoring surface properties. It is largely of interest because of its simplicity, adaptability, reproducibility and the possibility of achieving monolayers with a high level of molecular organization.

The expertise developed by the laboratory deals with self-assembly on active metals of a large family of molecules with different anchoring groups. While self-assembly on noble metals is easy, building such molecular systems on oxidizable metals is a challenging process because of the oxide on the surface which is detrimental to adsorption. Benefiting from the power of electrochemistry to monitor the surface of such metals and combining this with surface chemistry, the team has been able to develop monolayers and multilayers of organoselenols and organothiols on different surfaces such as copper, nickel, zinc and their alloys.

The comprehensive studies developed by the laboratory these last 10 years regarding the interfacial phenomena between such active surfaces and organothiols and selenols has given the laboratory expertise in surface modifications with control of the interface at molecular level as well as organisation of the monolayers.

Another family of self-assembled monolayers on metal surface, retaining and reinforcing their oxide layers is based on organosilanes and organophosphonic acids on metals oxides such as alumina, tantalum and titanium oxides.

This research opens doors to the study of new interfacial phenomena and applications in different fields. Among the applications developed, self-assembled monolayers as a new generation of corrosion inhibitors, molecular lubricants, molecular connectors for polymer or metal adhesion, initiator of ATRP polymerisation as well as applications in biomaterials.

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Patents

- Contact for an electrical connector protected by a polymer film and its production process, US05350323 (issued 09/27/1994), EP00500415A1 (issued 08/26/1992).
- Process for the production of chemical reactions in a polymer film, EP00715944A1 (issued 06/12/1996).
- Two-layerfluoroalkane-perfluoroether lubricant coatings for lubrication of electric contacts in automobiles and other vehicles, FR 2839316 A1- 07/11/2003, WO 03/09/2914-13 november 2003.

Funding

FUNDP, Wallonie, FNRS, Europe, Industries

Partnership

- TOTAL Petrochemicals, THERMPHOS, SOVITEC
- Several academic laboratories and research groups (national and international)

Main Equipment

- Potentiostats-Galvanostats
- Electrochemical Impedance spectrometer (EIS)
- Electrochemical Quartz Microbalance (EQCM)
- Scanning Vibrating Electrode Technique (SVET)
- Scanning Kelvin Probe (SKP)
- Scanning Electrochemical Microscope (SECM)
- FTIR PM-IRRAS
- Infrared microscope
- Spectroscopic ellipsometer
- Contact angle measurements
- Access to: XPS, ToF-SIMS, SEM, TEM, XRD, NMR

Products and Services

Surface modifications and characterisation (XPS, PM-IRRAS, ellipsometry, classical and local (SECM, SVET, SKP) electrochemistry

KEYWORDS

Self-assembly
Surfaces
Active metals
Oxides
Functionalisation
Nanofilms

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Elaboration of thin coatings by physical vapor deposition

SENIOR SCIENTISTS :

- Stéphane LUCAS
- Guy TERWAGNE

Research Field and Subjects

Through numerous fundamental and applied research projects, the research team is active in the following fields:

- Photocatalytic coatings,
- Nanoelectronic coatings,
- Tribological coatings,
- Corrosion protection coatings,
- Functionalisation coatings,
- Thermochromic coatings,
- Photovoltaic coatings
- …

The team's involvement in numerous fundamental and applied research programs has helped to develop unique expertise in coating substrate with various thin layer combinations: monolayer, multilayer, alloys, radioactive…

The coating material ranges from Plasma Polymers (PPAA) to ceramics (SiO_2, Al_2O_3, AlN) including semiconductors and very reactive material such as Li.

The substrate may be polymer, metal, ceramic, tissue or composite. Complex substrate shapes as carbon nanotubes have also been coated with polymeric layers to improve nanotube dispersion in different matrices.

Applications range from fundamental research (determination of diffusion coefficient, adhesion studies, electronic properties…) to coating for life sciences (Li for Boron Neutron Capture Therapy), coatings for tribological applications (DLC coatings to prevent wear on engine parts), photocatalytic coatings, and coatings for photovoltaic applications.

The best way to produce them is investigated thanks to our own Monte-Carlo simulation software and our deposition equipment: cluster deposition sources equipped with DC-Pulsed-RF magnetron sputtering guns, Ion beam Assisted Deposition capabilities and high temperature substrate heating systems. Some of the equipment has load-lock to ensure very good vacuum and reduce downtime.

Finally, the in-house analytical capabilities associated to the nuclear reactions induced by particle accelerator ALTAIS allow to investigate very quickly the synthesised products. Other conventional investigation tools like TEM, SEM, Ellipsometry, Pin-on-disk bring other view to get the complete picture of a coating system.

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Funding

DGO6 (Wallonie), MARSHALL Plan, Belspo, FNRS, Communauté française, Industry

Partnership

- Université de Liège, Laboratoire LCIS – GreenMAT
- CERN-Genève
- Université de Poitiers, Lab. de Métallurgie Physique
- Université de Franche-Comté, FEMTO, Besanson
- INRS-EMT, Université du Québec, Varennes, Canada

Main Equipment

- One in-line three chambers PVD system,
- Three single chamber PVD system,
- Plasma diagnogtis systems (Langmuir, OES, RFA, …)
- Pin-On-Disk, Dektak,
- Tandetron accelerator and associated analytical analysis equipment (RBS, PIXE, ERD, …),
- Ion Implantation facility.

KEYWORDS

Nanoparticles
Coatings
Physical Vapor Deposition
Magnetron sputtering
Evaporation
Monte-Carlo Simulation
Functionalisation

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Thin Biofunctional Coatings for the Control of Cell/Surface Interactions

SENIOR SCIENTIST :

- Karine GLINEL

Research Field and Subjects

The interactions between cells and material surfaces is a critical issue in diverse fields ranging from medicine to industry. A smart way of controlling such interactions consists of depositing a thin function layer on the material surface that provides a new functionality without affecting the bulk properties of the material. Depending on the application and the nature of the cells (microbial or animal), coatings are prepared to promote or avoid cell adhesion.

For instance, the development of antibacterial surfaces is of major interest for biomedical or industrial applications. Indeed, bacteria show a strong tendency to adhere to solid surfaces. Once adhered, they grow to form microcolonies and subsequently biofilms which then act as reservoirs for the development of pathogenic infections. They also cause persistent biofouling and corrosion problems in industrial water delivery systems and other industrial processes.

In contrast, tissue engineering applications are essentially based on the adhesion and proliferation of mammalian cells on biomaterial surfaces acting as implants.

In this context, the research group focuses on the development of new thin and ultrathin organic coatings able to promote the adhesion, proliferation and even differentiation of mammalian cells or, to avoid the adhesion of bacterial cells. Different parameters are explored to control cell/surface interactions such as nano-stiffness, lateral structure (chemical, mechanical and/or topographical patterning) and bio-functionalization of the deposited films. Approaches based on the self-assembly or grafting of macromolecules to solid substrates are used to prepare thin films showing surface properties close to natural surfaces such as living tissues.

The research group has gained expertise in the fabrication of thin biofunctional coatings as well as skills in the fabrication of nano/micro-structured surfaces by a series of lithography methodologies.

The research performed in this group thus aims at engineering the materials' surface properties in order to control cell/surface interactions and develop new coatings which are of interest for industrial, cosmetic or biomedical applications.

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Awards

- Dr. K. Glinel is a CNRS researcher currently benefiting from a FNRS MIS-ULYSSE grant to work at UCL (2009-2011).

Funding

- FNRS MIS-ULYSSE
- OSEO project (France) in collaboration with a French startup company
- Réseau interrégional Matériaux Polymères Plasturgie (France)
- CNRS (France)

Partnership

- International doctoral school IDS-FunMat

Products and Services

- Preparation and characterization of biofunctional surfaces to promote or avoid cell adhesion.
- Development of micro/nanopatterned surfaces showing a lateral variation of their properties for biological applications.

KEYWORDS

Surface chemistry
Biofunctionnal surfaces
Smart coatings
Nano/micropatterned surfaces
Antibacterial coatings
Cell adhesive coatings
Polymer surfaces
Lithography

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Functionalized nanoporous thin films from self-assembled block copolymers

SENIOR SCIENTISTS :

- Charles-André FUSTIN
- Jean-François GOHY

Research Field and Subjects

Block copolymers have attracted increasing interest due to their ability to self-assemble into well-ordered periodic structures. The size of those structures is determined by the molecular weight of the polymer blocks therefore lies in the nanometer range. Different morphologies, e.g. spheres, cylinders and lamellae, can be obtained depending on the block length ratio and interaction parameter. The cylindrical morphology is of particular interest since it can be transformed into an array of nanopores after elimination of the minor component. Those porous materials can in turn be used as membranes, sensors or templates for functional nanostructures, such as catalyst support or high density nanorod arrays for information storage applications. Most methods reported up to now for the creation of nanopores, rely on the degradation of the minor block by ozonolysis, UV etching, or hydrolysis for example, thus providing rather poor control over the chemistry of the pore walls.

This project aims at developing methods to create polymeric nanoporous thin films while controlling the chemical functions taking place at the pore walls. The building blocks used for this approach are block copolymers where the two blocks are linked by an addressable junction. Two main types of junction are exploited: a photocleavable bond or a supramolecular bond of the metal-ligand type.

The general process consists in self-assembling block copolymers into thin films (30-300 nm thick) while controlling the process in order to obtain cylinders, made of the minor block, perpendicular to the film surface. A stimulus is then applied to cleave the block copolymer at the junction and release the minor block. The stimulus can be irradiation at a given wavelength in the case of a photocleavable junction, or chemical (redox, or competitive ligand) for the metal-ligand junction. Since cleavage occurs only at the junction of the two blocks and via a defined chemical process, it is thus possible to control the functional groups (carboxylic acid amine, free ligand…) that will be present at the pore walls after removal of the minor block. It is particularly important to control pore wall chemistry for applications such as sensors, or selective membranes.

The team involved in this project has gained expertise in all the different steps of the process, from the synthesis of block copolymers, to their self-assembly into thin films, and the creation and characterization of nanoporous materials.

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Main Equipment

- GPC system (PSS) with triple detection (DRI, UV diode array, MALS).
- Atomic Force Microscope (Nanoscope V)

Funding

This research is supported by the EU FP7 project SELFMEM

Partnership

Members of the SELFMEM project (www.selfmem.eu)

Products and Services

Synthesis of (functionalized) block copolymers by controlled radical polymerization methods
Self-assembly and characterization of block copolymers in thin films (30-300 nm thick)

KEYWORDS

Nanomaterials
Membranes
Polymer synthesis
Self-assembly
Stimuli responsive materials

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Nano-engineered thin films and surface characterization techniques

SENIOR SCIENTISTS :

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- Laurent HOUSSIAU
- Jacques GHIJSEN
- Jeremy BRISON

Research Field and Subjects

LISE Laboratory has two long-developed objectives and expertises: (1) engineering (thin) layers, with a control of composition and structure for specific applications; and (2) improving and mastering characterization techniques capable of quantifing this type of composition and structure.
During the last decade, this expertise has been directed at objects and ultra-thin films in nano-dimension(s). New materials are currently produced using physical deposition methods (evaporation, sublimation, vacuum RF plasma…), while X-ray, electron and ion-based experiments are developed to study their properties on a nano-scale.

Current research projects are aiming for example to:

- engineer a sub-monolayer of organic molecules on a metal electrode to boost the efficiency of an organic light-emitting diode;
- tailor metal nano-clusters on carbon nanotubes with control of cluster size and size distribution
- optimize layer composition to increase the efficiency of organic photovoltaic cells
- develop and optimize metal oxide layers, for functionalization and grafting of biomolecules
- develop organic-inorganic nanostructured hybrid layers to control corrosion of a metal substrate
- master Scanning Transmission X-Ray Microscopy (STXM) in order to study surface chemistry and structure of isolated nano-objects (dimension above 20 nm), such as carbon nanotubes or metal clusters
- develop a new depth profiling technique in Time-of-Flight Secondary Ion Mass Spectroscopy to analyse thin organic layers, disclosing all elemental, chemical, and molecular information through the successive interfaces.

Representative References

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Patents

- Method for depositing nanoparticles on a support, WO 2009/021988.
- Sensor for benzene, P200930969
- Nanoparticles on substrates, EP 0174984.4 (with a specific contribution from Dr Michal Gulas).

Funding

EC (FP6, FP7), Wallonie, Belspo (PAI, PAT)

Partnership

- Arcelor-Mittal
- AGC

Main Equipment

LISE is equipped with:

- X-Ray Photoelectron Spectroscopy (XPS),
- Time-of-Flight Secondary Ion Mass Spectroscopy (ToF-SIMS),
- HREELS high resolution electron energy loss spectrometer,
- optical spectroscopies,
- five low pressure plasma chambers.

It has access to synchrotron radiation-based experiments in different countries.

Products and Services

Potential applications –and patents– are oriented towards catalysis, gas sensing, biosensors, membranes for fuel cells, organic electronics…
Analysis: Elemental, chemical and molecular composition of (thin) layers, materials surfaces or interfaces;
Synthesis: deposition of layers/coatings by evaporation, sputtering, sublimation, RF plasma

KEYWORDS

Surfaces
Interface
Analysis
Nanoparticles
Thin films
Depth profile

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Supported noble metal nanoparticles for heterogeneous catalytic applications, and their physico-chemical characterization

SENIOR SCIENTISTS :

- Eric GAIGNEAUX
- Patricio RUIZ

Research Field and Subjects

Noble metals are highly demanded as heterogeneous catalysts for several important chemical reactions either in the liquid or in the gas phase. For such applications it is required to have the metals as nanoparticles dispersed at the surface of supports as oxides (silica, alumina, titania, magnesia, etc) or active carbons. The metal particles are required to be as small as possible, typically in the range of 2-5 nm (or less), so that the metal dispersion (i.e. the ratio between the number of atoms exposed at the particles surface vs the number of atoms forming the entire particles) be maximized. This is the key to maximize the quantity of exposed active metal and thus the catalytic performances. In addition, it is required that the nanoparticles are accessible to the chemical reactants. Thus they should be properly located with respect to the porosity of the support). They should be also resistant to sintering (i.e. when heated they should not agglomerate to bigger particles) and to leaching (i.e. when submitted to mechanical or corroding stresses they should remain on the support).

The research group possesses a strong expertise in the preparation of supported metal nanoparticles presenting the requirements for heterogeneous catalysis. Briefly, 'reverse microemulsion' is a method that allows the obtention of metal particles smaller than 1 nm at the surface of a support. 'Deposition-precipitation' is based on the precipitation of metal hydroxides using the hydroxyls moieties present at the surface of the supports. This method has proven to efficiently lead after reduction to metal particles firmly anchored to the support, thus preventing them from sintering and leaching. A similar result is obtained by the 'grafting' of alkoxide or chloride metal precursors on the support hydroxyls, followed by a reduction step. The 'onion method' is bio-inspired. It consists in bringing the nanoparticles on the support via multilayer vesicles in which the metal is pre-reduced thanks to the action of a proper surfactant. When a partial elimination of the surfactant is performed, the metal nanoparticles are nonetheless made accessible for the chemicals, but they are also protected against sintering as the remaining surfactant residues serve as mechanical barriers hindering particles agglomeration. The present methods are complementary to those developed in 'Supported nanoparticles for heterogeneous catalysis derived from molecular precursors'.

Besides this expertise, the group possesses the equipement and the expertise to fully characterize metal nanoparticles in terms of composition, size, location, etc. It proposes also a certain expertise in the scaling-up of the procedures mentioned, as well as in the modifications of the supports, as often recommended in order to improve the efficiency of the metal supporting methods.

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Funding

This expertise was and is still acquired thanks to the support of the Federal Government (PAI 'Supramolecularity' n°5 and 'Inanomat' n°6/17), several projects funded by the Wallonie ('Comcat', 'Epurcat', 'Hydrogen', 'Cocagnes' and 'Hypharco'), by the Fonds Spécial de la Recherche of the UCL, by the FRIA , and via industrial partnership.

Partnerships

- The laboratories are involved in the Belgian INANOMAT network of teams working in the field of Inorganic Nanostructured Solid Materials.
- Active collaborations are also sustained with the University of Bordeaux 1, France and the Royal Institute of Technology, Stockholm, Sweden.
- Besides, the laboratory is involved in the European Multifunctional Materials Institute (EMMI) initiated from the former EU-FP6-Network of Excellence FAME.

Main Equipment

- Equipment for the preparation of supported metal nanoparticles, and for the required modifications of the supports, including pilot scale preparation and shaping (extrusion and pelletization).
- Equipement for the physico-chemical characterization of the modified supports and of the supported metal nanoparticles : X-ray photoelectron spectroscopy (particle size, surface chemical moeities and composition, oxidation states), X-ray diffraction (nature of phases present, nanoparticles size) and fluorescence (metal content), specific surface area and porosity measurements (location of nanoparticles with respect to the support), probe molecules adsorption followed by IR spectroscopy (nature of supported metal species), chimisorption of CO and H_2 (dispersion of the metal, particles size), accesses to electron microscopes.
- Equipment for catalytic performances measurements in several liquid and gas phase reactions requiring metal nanoparticles.

Products and Services

The group proposes its expertise for the preparation of supported (noble) metal nanoparticles. The expertise also includes the modifications of the supports sometimes required for the obtention of nanoparticles with the desired size, location, resistances to leaching and sintering. The group also proposes its equipment and expertise for the characterization of the supported nanoparticles (size, location, accessibility, etc) via different physico-chemical methods, microscopies, and spectroscopies including under working conditions (high temperature, under gas flow). The group also proposes to measure the catalytic performances of the supported nanoparticles in several heterogeneous catalytic reactions in the liquid or in the gas phase. Beside, an expertise and the equipment in the pilot scale preparation and the shaping of the cited materials is available.

KEYWORDS

Characterization of metal nanoparticles
Heterogeneous catalysis
Leaching (resistance to)
Microemulsion
Noble and semi-noble metals
Scaling up
Sintering (resistance to)
Support

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Micro- and nano-electro-mechanical-systems

SENIOR SCIENTISTS:

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- Denis FLANDRE
- Laurent FRANCIS
- Thomas PARDOEN
- Joris PROOST

Research Field and Subjects

Micro- and nano- electro-mechanical systems (MEMS/NEMS) as well as integrated sensors and electronics circuits play an ever increasing role in the development of new information systems from telecommunication to human body, machine or process monitoring applications.

A major effort has been made at UCL in recent years to build a multidisciplinary group of researchers combining expertise in materials science, micro/nano-fabrication technology, integrated systems, physics, chemistry, continuum mechanics and electronics to support the development of robust MEMS technology as well as novel electronic devices on micro and nanometer scales.

Major emphasis is put on :

- improving deposition methods,
- depositing new thin film materials (functional ceramics),
- integration of new materials in classical MOS processes for improving electrical performance and extending the functions of micro/nano-devices, sensors and circuits,
- wideband modeling and characterization of advanced MOS devices,
- monitoring internal stresses during and after deposition,
- optimizing etching methods to release structures,
- improving wafer bonding techniques,
- making use of internal stresses to assemble structures,
- measuring and modeling the elastic and (visco-) plastic properties of thin films using nanoindentation and microbending,
- measuring and modeling the adhesion of wafers and films inside multilayers or on substrates,
- accurately determining displacements using optical methods,
- characterizing thin film microstructure.

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Patents

- Method for filling an opening in an insulating layer. European Patent Number 99870058.7, International Patent Number 60/083,822.
- Selective etching for semiconductor devices, WO/2007/082745
- Internal stress actuated micro- and nanomachines for testing physical properties of micro and nano-sized material samples, WO/2007/093018
- Imposing and determining stress in sub-micron samples, WO/2008/098993

Awards

Best Student Paper Award - International Conference on Fracture 2009, M. Coulombier, A. Boé, A. Safi, S. Gravier, J.-P. Raskin, T. Pardoen, "Fracture of thin freestanding ductile metallic films investigated using a nanomechanical lab on chip device", 12th International Conference on Fracture, Ottawa, Ontario, Canada, July 12-17, 2009, paper # T44.002.

Funding

- Convention RW no. 31/5574 "CAVIMA – Capteurs Vitaux Intelligents, Miniaturisés et Autonomes"
- Action Concertée, « Innovative technologies for physical and (bio)chemical nano-sensors »
- Projet DGTRE-RW, NANOTIC – CITE, "Essaims de Senseurs Intelligents - Composants intégrés de transduction électronique"
- DGTRE-RW, First Spin-off, SENSOI, "Micro-senseurs en technologie SOI"
- DGTRE-RW, Plan Marshall, HM+, "L'Avion plus Intelligent : Health Monitoring et Maintenance Prédictive"
- FEDER, DGTRE-RW, MINATIS, "micro et nanofabrication, caractérisation et fiabilité de composants miniaturisés"
- DGTRE-RW, Plan Marshall – Open Innovation, MEMSVIT, "Développement d'un projet d'innovation ouverte centré sur le transfert de capteurs miniatures sur vitrage"
- ARC – "Propriétés optiques des structures naturelles et artificielles"

Partnership

- Laboratoire Européen Associé with IEMN, Lille, France.
- IMEC, Leuven, Belgium.
- Partner of Network of Excellence – METAMORPHOSE (MetaMaterials Organized for radio, millimeter wave, and PHOtonic Superlattice Engineering).
- Partner of the Wallonie's projects "Nanotic" and "BIOSE". Leader of the FEDER/Wallonie projet "MINATIS".

Main Equipment

- The largest research-oriented clean room facilities (800 m2) in Wallonie with all key technologies required for micro/nano-systems and micro/nano-electronics fabrication (WINFAB).
- Deposition chambers equipped with *in-situ* monitoring of internal stresses.
- Atomic layer deposition tool with in-situ ellipsometry and annealing (850°C).
- Micromechanical tests: nano-indentation, micro tensile testing stage for *in-situ* testing in a SEM, microwedge tests.
- Characterization techniques: optical interferometry, SEM, TEM, OIM-EBSD.

Products and Services

- Measurement and extraction of electrical properties (conductivity, permittivity, permeability) over wide frequency band and temperature range (WELCOME).
- Deposition (LPCVD, PECVD, ALD, electrochemical) and etching (chemical, plasma) techniques for thin film coatings on solid substrates (silicon, glass…)
- Optical non-destructive characterization. Laser and low-coherence interferometry for high precision shapes and residual stress measurement, vibrometry and thermoreflectivity.
- Expertise in micromechanical testing, and internal stress measurements.

KEY WORDS

Mechanical characterization
MEMS/NEMS
Thin film deposition
Microelectronics
Microfabrication
Optical characterization
Surface Functionalization

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Nano-electronics: advanced silicon devices and circuits

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- Jean-Didier LEGAT
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- François-Xavier STANDAERT
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Research Field and Subjects

Overall study of Silicon-on-Insulator (SOI) technologies, devices and circuits, for low-voltage low-power, radiation-hardened, microwave, high-temperature, telecommunication, cryptographic and quantum applications.

Characterization, simulation and modeling of advanced devices, eg. :

- Measurement and extraction of static and dynamic behaviours and parameters of semiconductor devices, e.g. statistical digital/analog/RF figures-of-merit, floating-body and substrate time constants, distortion, matching, 1/f noise, reliability etc.
- DC to RF wideband behaviors of e.g. advanced SOI MOSFETs and nanowires.
- 3-D semiconductor simulation of e.g. double- and multiple-gate MOSFETs.
- Compact modeling of SOI ultra-thin-body, fully-depleted single-gate, double-gate and graded-channel MOSFETs and RF macro-modeling.
- 3D simulation for microwave active and passive integrated devices

Subsequent analog, RF and digital circuit design in many SOI CMOS processes down to sub-100nm generations, e.g.

- Ultra-low-power analog, digital and memory circuit families for e.g. RFID and biomedical applications.
- Secure cryptography functions.
- Analysis of SOI substrate crosstalk in mixed-signal circuits.
- Microwave circuits design (LNA, VCO, UWB pulse generator) for low power telecommunication front ends

Fabrication of advanced SOI MOSFETs and quantum nanoscale devices, e.g. :

- Critical fabrication steps for Double-Gate SOI MOSFET's and nanowires (patterned wafer bonding, self-aligned buried mask…).
- Thin silicides on SOI: Er-Pt (for low Schottky barriers), Ni (for low ohmic and RF contacts), Co (for high-temperature contacts), etc.
- Quantum wires, Single-Electron-Memories, Single-Electron-Transistors, etc.

Representative References

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Patents

- *ULP basic blocks and their uses.* Continuation in Part, filled in USA on June 23rd 2003 under reference no. 10/602,016.
- *High resistivity poly-SOI substrate.* PCT/BE2004/000137, September 2004.
- Ultra-low-power circuit, European application 30.04.2007, Pub. No.: US 2009/0273356 Al (43), Nov. 5, 2009
- Double gate floating body memory device, WO 2009/087125 A1, 16/07/09
- Network Architecture For Wirelessly Interfacing Sensors At Ultra Low Power, Pub. No.: WO/2009/092771, PCT/EP2009/050734, 30.07.2009.

Funding

- SINANO / NANOSIL, Silicon based nano-devices, EU Networks of excellence, FP6 / 7 (STM, IMEC, CEA-LETI, AMO, IEMN,…).
- EUROSOI, SOI technology EU Thematic Networks, FP6 / 7 (CISSOID, Philips, VTT, SOITEC,…).
- GRADIO, T206, MARQUIS and WITNESS, MEDEA+ projects investigating Low-power / RF SOI circuits for different telecom applications (STM, CISSOID, Philips, FPMS, Agilent,…).
- LEMM**,** Laboratoire européen associé, CNRS-FNRS with IEMN, Lille, France.
- NANOTIC Project (Wallonie)
- TELECOM / S@T Projects of SKYWIN, Walloon aeronautical pole.

Products and Services

- Integrated device fabrication from micro-dimensions (optical lithography, thin layer deposition and etching) down to nano-dimensions (defined by e-beam lithography and controlled oxidations).
- Devices and circuits design (finite element simulations, electrical simulations with Cadence, ELDO, VHDL-AMS, ADS…)
- Devices and circuits characterization (ICCAP, S-parameters…)

Main Equipment

- Complete fabrication line for the rapid prototyping and validation of new fabrication steps and integrated devices on silicon/SOI substrates (3-inch).
- Electrical measurement set-ups from DC (up to 110 GHz) and from a few mK up to 400°C on wafer-scale (semi-automatic prober) as well as packaged circuits levels.
- Semiconductor simulation tools (ISE, Avanti and Silvaco). Electro-magnetic and circuit simulations (IE3D, ELDO, VHDL-AMS, ADS).

KEY WORDS

Cryptography
Integrated circuits
Micro-nanotechnology
Microwave circuits
Nanoelectronics
Semiconductor devices
System-on-chip
Telecommunication front ends
RF

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Spintronics and nanomagnetism

SENIOR SCIENTIST :

- Luc PIRAUX

Research Field and Subjects

For most of the twentieth century it was known that the electrons which mediate electrical current in electronic circuitry possess angular momentum called "spin": yet no practical use has been made of this. Since the discovery of "giant magnetoresistance (GMR)" in 1988 by Albert Fert's group at Orsay (winner of the 2007 Nobel Prize for Physics), a novel technology has emerged called "spin electronics" or « spintronics » that is based on the up or down spin of the carriers rather than on electrons or holes as in traditional semiconductor electronics. The giant magnetoresistance (GMR) has led to a dramatic increase in the sensitivity of modern magnetic hard drive read heads. This has led to the manufacture of hard disks with much greater storage density. Almost all hard drives are equipped with such heads today.

The template strategy has been successfully used by the research group to first produce arrays of magnetic multilayered nanowires by electrodeposition in nanoporous template. This is the context in which strong collaboration has developed over the last 17 years between the research group at UCL and the Unité Mixte de Physique CNRS/THALES. The nanowires system has provided an important opportunity in exploring GMR effects in the current perpendicular to the planes geometry and in testing theoretical models within various limits. Extensive giant magnetoresistance measurements have been performed on various magnetic multilayered nanowires such as Co/Cu and NiFe/Cu nanowires.

More recently, it has been established that a spin polarized current can induce torque on the magnetization of a ferromagnet. Spin transfer torque in magnetic nanostructures has generated huge interest in recent years for its potential application in the fields of data storage and current driven microwave oscillators. The research group was able to investigate spin transfer torque phenomena in spin valve multilayered nanowires grown by electrodeposition on nanoporous alumina templates. Spin transfer torque experiments were performed by injecting a high density spin-polarized current in a single nanowire connected using a nanolithography based contacting method. Magnetic excitation was detected on single Co/Cu/Co spin valves as well as on several microwave oscillators connected in series along the same nanowire for phase-locked experiments.

Since the discovery of the giant magnetoresistance effect, nanomagnetism has also been at the centre of a great deal of research interest and a considerable effort has been made to develop fundamental research and move new materials and effects toward applications. For example, two-dimensional arrays of bistable magnetic nanowires are a class of systems which have been considered by the research group for studying magnetic interactions and switching behaviour and have been proposed as candidates for ultrahigh storage densities.

Representative References

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Patents

« Multiple bath electrodeposition » -EP0111055.8 - 2002

Awards

Invited talks at the « Gordon Research Conferences », Invited talk at the International *Symposium* " 20 Years of *Spintronics*: *Retrospective* and Perspective", Doctor Honoris Causa – University of Bucarest – Romania (2008)

Funding

- Belgium Interuniversity Attraction Pole on « Quantum Effects in Clusters and Nanowires»: PAI-IUAP P6/42 (2007-2011)
- Small or medium-scale focused research project FP7-NMP-2007-small-1 : «Microwave Amplification by Spin Transfer Emission Radiation» (2008-2012)
- FNRS, FRIA

Partnership

- Unité Mixte de Physique CNRS/THALES THALES Palaiseau
- CEA- Grenoble
- Institut NEEL - Grenoble
- SAN LUIS POTOSI (Mexico)

Main Equipment

- High vacuum coating systems (sputtering and e-beam)
- Electrochemical set-up
- High Resolution Scanning Electron Microscopy & EDX probe
- Helium-4 cryogenics systems for electrical measurements
- SQUID magnetometer
- Network analyser (up to 65GHz)
- Electromagnets & superconducting magnets

KEYWORDS

Nanotechnology
Spintronics
Magnetic recording
Magnetic characterization
Nanotemplates & nanowires
Magnetic multilayers

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Nanoscale superconductors

SENIOR SCIENTIST :

- Luc PIRAUX

Research Field and Subjects

The research activity developed in the group concerns the electrical transport properties of superconducting thin films and nanowires. The aim of the work is to improve superconducting properties through the nanostructuring of thin films, as well as the investigation of non-equilibrium phenomena in superconducting microstrips.

Nanostructuring of thin films is of technological and scientific interest. Particularly, the introduction of defects in superconducting thin films has proved to enhance their functional properties. Generally, lithographic techniques are used to create periodic vortex pinning center lattices. This leads to a global increase in critical current and also periodic enhancement of vortex pinning at the so-called matching fields (i.e. the magnetic fields at which the vortex lattice matches the pinning centers lattice). However, lithographic techniques are time-consuming and expensive, which strongly limits the size of the samples.

The group at UCL has recently developed a promising, alternative process to lithographic techniques, based on ordered nanoporous alumina templates, in order to create periodic vortex pinning center lattices. This approach is suitable for preparing a much higher density of pinning centers on a larger surface and at low cost. The templates, obtained by a two-step anodization of aluminum, present triangular arrays of long channels closed at the end by a barrier layer which can be revealed by a selective etching of the aluminum. Using these highly ordered templates, different routes to imprint a pinning center lattice into superconducting thin films were investigated. First, the porous surface of the template was used as a substrate for the vapor deposition of antidot array superconducting thin films. Second, the barrier layer of the anodized aluminum oxide templates was used for fabricating hexagonally close-packed nanobump arrays. Again, such nanobump arrays were used as a substrate for producing superconducting thin films with a higher number of pinning centers. A third approach was to take advantage of the interaction between an ordered ferromagnetic nanowire array and the superconducting film to promote magnetic pinning of the vortices. By this template strategy, the research group has prepared superconducting nanostructures with different designed morphologies and has demonstrated matching effects at much higher fields than using lithographic techniques.

On the other hand, heat dissipation is known to impact the electrical behavior of superconducting devices, especially for mesoscopic samples. The main challenge is to determine the real influence of Joule heating on the voltage-current characteristics. For the last few years much attention has been devoted in the UCL group to investigation of the non-equilibrium phenomena that compete in superconducting nanowires and niobium-based microstrips. The distinction between phase-slippage and the propagation of normal hotspot domains has been particularly studied.
In addition such lithographically defined structures are well suited to better understanding the perfectly (and non dissipative) superconducting state. Another topic of the research deals with the impact of fluxoïd quantization on the instability current of superconducting loops.

Representative References

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Patents

« Multiple bath electrodeposition » -EP0111055.8 - 2002

Funding

- Belgium Interuniversity Attraction Pole on « Quantum Effects in Clusters and Nanowires»: PAI-IUAP P6/42 (2007-2011)
- FNRS
- FRIA

Partnership

- LPN Marcoussis
- KUL
- UA (Universiteit Antwerpen)

Main Equipment

- High vacuum coating systems (sputtering and e-beam)
- Electrochemical set-up
- High Resolution Scanning Electron Microscopy & EDX probe
- Helium-4 cryogenics systems for electrical measurements
- SQUID magnetometer
- Electromagnets & superconducting magnets

KEYWORDS

Superconductivity
Nanophysics
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Thin films
Vortex matter

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Microwave devices based on magnetic nanowired substrate

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Research Field and Subjects

On the road towards size reduction of microwave devices, ferromagnetic nanowires embedded into porous templates are an interesting alternative route to ferrite based materials. The work of the research groups is at the leading edge in this field. Over the last decade, they have successfully prepared a variety of ferromagnetic nanowired substrates and demonstrated their ability to build planar reciprocal and non-reciprocal devices for very high frequencies, as are now requested for wireless communication and automotive systems. Indeed, such templates were recently used to design various microwave devices, such as or circulators, isolators and phase shifters.

The main advantages of ferromagnetic nanowired substrates are that they present a zero-field microwave absorption frequency thanks to the very high aspect ratio of the nanowires. The zero-field microwave absorption frequency can be easily tuned over a wide range of frequencies by an appropriate choice of materials. In contrast, conventional ferrite devices need to be biased by a magnetic field provided by a permanent magnet in order to operate, which constitutes a negative aspect with respect to volume reduction. Other advantages of such materials compared with classical ferrites are higher operating frequency and higher saturation magnetization. Prototypes of (nonreciprocal) microwave circulators and phase shifters have already been successfully demonstrated on ferromagnetic substrates.

The presence of ferromagnetic material is also responsible for an increase in permeability at low frequency and frequency tunability when an external DC magnetic field is applied. Tunability performance of integrated inductances and quality factors predicted by simulations were successfully validated by experiment.
Tunability is also of great interest for filters and and novel compact devices using the metamaterial concept, combining negative permittivity and permeability of substrates.

Finally, such ferromagnetic nanowired substrates are low cost and fast to produce over large areas compared with standard ferrite devices.

Representative References

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Funding

- Belgium Interuniversity Attraction Pole on « Quantum Effects in Clusters and Nanowires» : PAI-IUAP P6/42 (2007-2011) FR
- FRIA and FRS-FNRS, Belgium

Partnership

- THALES
- SAN LUIS POTOSI (Mexico)
- Institut NEEL (Grenoble)

Main Equipment

- High vacuum coating systems (sputtering and e-beam)
- Electrochemical set-up
- High Resolution Scanning Electron Microscopy & EDX probe
- Network analyser combined with cryogenics and magnetic field facilities
- WELCOME Measuring Facility

KEYWORDS

Nanotechnology
Nanowired substrates
High frequency characterization
Thin film vacuum deposition
Electrodeposition
Microwave devices

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Molecular nano-electronics and plasmonics

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Research Field and Subjects

Today, conventional microelectronics uses smaller and smaller devices and increasingly complex lithography procedures in a top-down approach to produce semiconductor circuits. When this strategy comes to a dead end – because of device physics or the cost of lithography – it can be replaced by a bottom-up approach building complete information-processing units via self-assembly of functional molecular nanostructures. New technologies, namely *molecular nanoelectronics* and *plasmonics*, would not only provide the solution to current limitations in reducing the size of electronic devices, but could also make a breakthrough in developing a simpler and much cheaper production technology.

A multi-disciplinary team is actively working on new methods to design, synthesize and characterize novel archetypes of low-dimensional hybrid organic-inorganic systems for nano-electronics and plasmonics. For instance, polymer nanostructures (e.g. polyaniline) were patterned on micro- and nanoelectrodes - used as catalytic active areas, so that manifold circuit elements and devices can be built besides traditional electronic lab-on-chip nanosensors.

While research activities traditionally concern the synthesis and modeling of materials, a vigorous study of the propagation, manipulation and harvesting of light at nano- to macro-scale levels has been recently initiated. Sub-wavelength resolution imaging, focusing and lithography (defeating Abbe's law), photonic band gap and metamaterials on polymer-based structures have been proposed (e.g. polystyrene micro-architectures).

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Products and Services

Synthesis and characterization of active molecular species, hybrid organic-inorganic nanowires, highly ordered polymer architectures, high resolution electron beam lithography, colloidal lithography, Si micro- and nano-ribbons.

Detailed measurements of macroscopic and local optical and electronic properties of various materials and devices (*in situ* or in a controlled environment, when necessary).

An expertise in *ab initio* simulations is provided to companies.

Main Equipment

Synthesis facilities are principally located at IMCN Institute. The nanofabrication tools are part of the UCL WINFAB cleanroom. The characterization instruments are mainly situated in the UCL WELCOME platform and notably include low temperature setups (*e.g.* scanning gate and scanning tunneling microscopy) and precision electronic equipment for DC and low AC measurements. The access to the UCL computers for intensive scientific calculations is provided by the CISM Institute.

Partnership

- NANOWAL, Belgium
- Académie Universitaire “Wallonie-Bruxelles”, Belgium
- Chalmers University of Technology, Sweden

Funding

The molecular nanoelectronics and plasmonics program is supported by the ARC research convention n°09/14-023, the Belgian FRS - FNRS and the NANOTIC Program of Excellence, Wallonie, Belgium. Support has been received from COST Chemistry D35, ETSF and MC2 Access EU programmes.

KEYWORDS

Ab initio simulations
Atomic technology
Block copolymers
Low temperatures
Molecular electronics
Nanolithography
Plasmonics
Synthesis

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Molecular optoelectronics and spintronics

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- Sorin MELINTE

Research Field and Subjects

The drive towards superior nanomaterials for applications at the frontier of knowledge is usually hampered by their stability and the difficulty in accurately controlling and manipulating their structural and physical properties. Novel *molecular tinkertoys*, easily tunable, and featuring exceptional optoelectronic and spintronic properties are thus urgently needed.

The expertise of the team addresses: the synthesis of new supramolecular components with pre-programmed functions and properties, the development of circuit design strategies that can incorporate such supramolecular architectures, the development of ultra-clean production tools to build a large number of these devices with atomic precision and regularity, and theoretical design and modeling.

The main results of this research effort include (a) preparation of a novel generation of highly-ϖ-conjugated organic molecular tinkertoys featuring enhanced opto-electronic and spintronic properties (e.g. porphyrin derivatives), (b) characterization of monomolecular optoelectronic and spintronic devices by a new scanning tool with varying wavelength capabilities and liquid pulsed deposition, and (c) the development of quantum chemical methods to predict and interpret the optical and magnetic properties of large closed- and open-shell organic ϖ-conjugated species.

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Funding

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Partnership

- NANOWAL, Belgium
- Académie Universitaire "Walonie-Bruxelles", Belgium
- SMEAGOL Team, Ireland
- Institut des Sciences Moléculaires, Université de Bordeaux
- Osaka University, Department of Materials Engineering Science

Main Equipment

- Synthesis is carried out at the Laboratory of Supramolecular Organic Chemistry at FUNDP.
- The nanofabrication tools are mainly part of the UCL cleanroom (WINFAB). The characterization instruments include low temperatures facilities down to 100 mK and precision electronic equipment for DC and low AC measurements. The UCL experimental group also operates a set of glove boxes for controlled fabrication of devices and a ultra-clean cluster tool for surface science techniques and ultra-high-vacuum methods (LEED, AES, liquid molecular epitaxy, thermal deposition of molecules…) as well as a low temperature scanning tunneling microscope. (STM). The development of a spintronic and optoelectronic STM is the main thrust of this research.
- The theory group develops its activities at the interuniversity Scientific Computing Facility (see reading note F1).

Products and Services

- Synthesis and characterization of active molecular species.
- Detailed measurements of macroscopic and local properties of various organic materials and devices (*in situ* or in a controlled environment, when necessary).
- Elaboration and implementation of quantum chemistry methods, *ab initio* simulations of the structural, electronic, optical, and vibrational properties of molecules and supramolecules.

KEYWORDS

Atomic technology
Low temperatures
Molecular electronics
Nanochemistry
Optoelectronic tunneling microscopy
Quantum chemistry
Spintronics
Synthesis

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Fabrication of Si-based nano-electronic devices

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- Denis FLANDRE
- Jean-Pierre RASKIN

Research Field and Subjects

To meet requirements of low power consumption, fast access speed and high integration density, electronic device dimensions are reduced to nanometer scale. Single-electron devices, such as single-electron transistors and single-electron memory, are the ultimate electronic devices. To produce quantum effects (quantum confinement and Coulomb blockade) at room temperature, device dimensions must be less than 10 nm. Nowadays, it is not difficult to produce nanodots and nanowires with a width of less than 10 nm, but the difficult task is to use them as device components and place them at the desired locations. Therefore, new and tricky fabrication methods, but compatible with complementary metal oxide semiconductor (CMOS) technology, must be proposed and developed.

Based on Silicon-on-Insulator (SOI), $Si/Si_{1-x}Ge_x/Si$ heterostructure and other materials, room-temperature operation of single-electron transistors and single-electron memories with a single floating gate or double floating gates have been demonstrated. The production of these devices makes multi-bit memory cells and qubit quantum computers more realistic. The following specific technologies are available: SOI wafer thinning by wet etching (down to 10-nm-thick), e-beam lithography with PMMA and HSQ as hard masks, selective etching by Reactive Ion Etching, atomic layer deposition (Al_2O_3, HfO_2, other oxide layers, nitrides and pure metals), dopant-enhanced oxidation, Pt or Er silicidation.

In addition, nano-biosensors have the potential to increase sensor sensitivity to single-molecule detection level. Three kinds of nano-biosensors have been implemented: MOSFET sensor with an Au nano-interdigitated array gate, Si single-nanowire sensor, and partial PtSi multiple-nanowire sensor. They can be used to detect proteins, DNA strands, bacteria, spores or virus targets, as well as gas chemical molecules. The manufacturing process is reproducible, reliable and valid for large-scale products. These sensors can be directly included on a unique SOI chip with previous micro-biosensors, impedance transducers and CMOS circuits. This gives opportunities for selective and multiple detection of chemical/biological molecules.

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Patents

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Awards

« Raith Micrograph Award », 4th place in 2007.

Funding

This research is supported by FRS-FNRS Belgium.

Partnership

- Institut d'Electronique, de Microélectronique et de Nanotechnologie, France
- University Stuttgart, Inst. Halbleitertechnik, Germany
- Tyndall National Institute, University College Cork, Ireland

Main Equipment

A complete fabrication line for the nano-electronic devices, nano-biosensors and integrated devices on Si or SOI substrates (3-inch), high resolution e-beam lithography tool and atomic layer deposition system.

Products and Services

Nano-electronic devices and nano-biosensors fabrications

KEYWORDS

Nano-electronic devices
Single-electron transistor
Single-electron memory
Nano(bio) sensors
Si
SOI
Si1-xGex heterostructure and CMOS.

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Nanophotonics : From plasmonic particles to nanostructured photonic devices

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Research Field and Subjects

In recent years, understanding the optical properties of matter on a nanoscale has opened new and diverse routes of research, which are based on the theoretical study of the optical (plasmonic) response of individual metallic particles as well as nanostructured photonic media. Applications are numerous: sensors, phototherapy, solar cells, ultra-sensitive optical spectroscopy (SERS), filters ... The complex behaviour of nanophotonic systems can not however be handled or predicted without the support of dedicated numerical simulations. The work done in the development of new and efficient simulation tools at the University of Namur (FUNDP) is described briefly here.
Metallic nanoparticles have optical properties which depend on the size, shape and local environment of the nano-object. Numerical analysis of the optical response and the electromagnetic field distribution can be probed by electron spectroscopy with nanoscale spatial resolution (Electron Energy Loss Spectroscopy) with the aim of understanding the local response of individual or coupled metallic nanoparticles to global excitations.
Through electromagnetic coupling of a plasmonic nano-object and a molecule, the fluorescence properties of the latter can be tuned. Such promising properties are studied for applications in biosensing.
Periodic structuring of dielectric materials at wavelength scale creates photonic crystals. The effect of structural disorder is often only considered as perturbation. However, the resulting intrinsic properties are important for the control of light propagation or scattering in such disordered photonic media, e.g. for light filtering as in the eye cornea.
Nanocomposites made of plasmonic nanoparticles embedded in glass are also disordered materials with peculiar linear and non-linear optical properties. The distribution in particle size and the short range correlation of particle interdistance are investigated with a view to developping applications in sensing.
Finally, nanostructuring of thin films solar cells is studied with the aim of enhancing light trapping and therefore increasing photovoltaic efficiency.

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Funding

- PHC Tournesol 2010 (WBI)
- AGC FlatGlass Europe

Partnership

- Université de Paris-Sud
- University of Southampton

Main Equipment

I-SCF Scientific Computing Facilities

Products and Services

Expertise in the simulation of the optical properties of nanostructured materials, including homemade softwares

KEYWORDS

Nanophotonics
Plasmonics
Electron-Energy Loss Spectroscopy
Metallic nanoparticles
Nanocomposites
Disordered optical media
Photovoltaic devices

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Semiconductor nanostructures: fabrication and electronic properties

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Research Field and Subjects

Semiconductors are almost ubiquitous in today's world: computing, consumer electronics, light emitters or detectors, photovoltaic panels, etc. Modern electronic devices often rely on the reduced scale of semiconducting regions and the combination of different types of semiconductors.

The Laboratory of Electronic Materials (LPME) specializes in producing semiconductor heterostructures in a well-controlled environment, allowing for the control of composition and thickness of the structures at atomic level. Materials under investigation are mostly semiconductors that have recently attracted interest for special applications. They include, but are not limited to: GaN (for blue and (ultra)violet light emitters or detectors), ZnO (for spintronics or high power or high temperature electronics), CdTe and related compounds (for solar cells and light detectors.)

The main interest within LPME is in understanding the formation of interfaces between these semiconductors and other materials, such as silicon, oxides or metals. More specifically, one wants to find out how the growth conditions of thin films of these materials affect their nano- or microstructure and how this influences their electronic, optical or mechanical properties. By carefully adjusting substrate preparation and experimental conditions, nanostructures can be produced during growth of a thin film, without the need to remove unwanted parts of the deposited material by lithography.

The main asset of LPME is its experience with a wide range of semiconductors and other inorganic materials. Combining evaporation, DC sputtering and molecular beam epitaxy, thin films of virtually any material can be produced, as well as multilayers combining different materials, as long as they are compatible with high or ultra-high vacuum. The structure of these materials is then analyzed on a nanoscale with scanning tunneling microscopy and photoelectron microscopy, without exposing the samples to air. Many other possibilities for characterization exist through collaborations with other laboratories.

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Patents

Nanocomposites, their manufacture and uses, EP 02447039.5 – 20 mars 2002, WO 03/07/8315 A2- 25/09/2003

Funding

Belgian National Fund for Scientific Research

Partnership

- University of Illinois at Chicago
- Novasic SA
- Universität Hamburg

Main Equipment

- Scanning Tunneling Microscopy in UHV
- Photoemission Microscope (PEEM)
- Molecular Beam Epitaxy
- Vacuum deposition (evaporation, sputtering)

KEYWORDS

Semiconductors
Scanning Tunneling Microscopy
Thin films
Surfaces

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Bio and Soft Electronics for Sensors and Implantable Electrodes

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- Sami YUNUS

Research Field and Subjects

The development of new synthetic approaches and methods for hierarchical assembly is becoming essential, especially when structuring materials down to micro and nanometer scale for real applications. In this context, one research expertise concerns the development of new micro and nano-devices for bio-sensing and/or neuro-stimulation. Besides micro and nanofabrication, a large part of the research is also devoted to the development of new electrochemical methods adapted to these new devices. As a consequence of these two approaches, the laboratory has gained strong experience in the production of complete real working devices such as USB portable biosensors that take advantage of cutting edge technologies such as the use of electro-conductive polymer materials. Today, the laboratory includes many new facilities allowing rapid PCB and electrode prototyping, micro-controller programming (MSP430) and electrochemical testing.

These research fields include many other related subjects such as surface characterization, thin organic films and supramolecular assemblies, biomaterials and hybrid materials, plasma treatments, chemical grafting, conducting polymer film growth, electrospinning, electrochemistry, thin (organic/metallic) layer adsorption, soft lithography, breath figure imprinting, micro-fluidics, etc.

Representative References

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Patents

- Stretchable conductor and method for producing the same, PCT n°PCT/EP2007/053159 on 02/04/2007 (2007)
- Smart Sensor System using Polyaniline as a Transducer, EP10150280.5 (2010)

Funding

NANOTIC - Wallonie.

Partnership

- The Progenosis company
Bifunctionnal Hybrid Protein Development
(Belgium)

- Université de Liège (Belgium)

- Consiglio Nazionale delle Ricerche (CNR-Italy)

- The Neurotech S.A. company
Neuro-Stimulation Devices Development
(Belgium)

Main Equipment

- Chemistry Laboratory for materials synthesis and functionnalization
- Electronic Laboratory for PCB manufacturing and electrical characterization
- Process Laboratory for microfluidics fabrication and plasma treatment facilities
- Home-made USB transportable potentiostats and disposable electrodes for easy and fast biosensing kit development

Products and Services

Home-made USB transportable potentiostats and disposable electrodes for easy and fast biosensing kit development

KEYWORDS

Implantable electrodes
Polydimethylsiloxane (PDMS)
Biosensor
Enzymes
Electrochemistry
Micro-fluidics
Electro-conductive Polymers

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Expanding the limits of (Thin Film) Photovoltaic Technologies

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- Jean-Pierre RASKIN
- Sorin MELINTE
- Laurent FRANCIS
- Sébastien MICHOTTE
- Romain DELAMARE

Research Field and Subjects

Major advances have occurred in the past several years as thin film photovoltaic (PV) technologies continue to enter the market for various applications. Critical production issues, such as standardization of absorber- and active layer deposition, still need to be addressed to help lower the unit cost of module production in large-scale thin film PV manufacturing. Once that has been achieved, the applications that can be pursued using thin film PV technologies include building-integrated photovoltaics (BIPV), roof-top applications and utility-scale applications.

Skills:

- passivating and anti-reflective large-bandgap window layers for Solar devices
- Transparent and conductive oxide layers for solar applications
- development of hetero-junctions for high-efficiency solar cells
- Organic bulk hetero-junction for the third generation of solar cells

Representative References

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Funding

SUNTUBE project (Solwatt Wallonie)

Main Equipment

- WINFAB: microelectronics clean Room class 100 with CMOS SOI prototype line (Implanter, annealing and diffusion furnaces, wet etching, dry etching, UV lithography).
- Custom-built batch-type reactive sputter deposition tool with in-situ optical diagnostics (located WINFAB)
- Thin film deposition and characterizations: CVD techniques (3 LPCVD furnaces, 3 plasma CVD, 1 ALD), PVD techniques (2 evaporators, 2 sputterings), characterizations by ellipsometry, profiling and microscopy.

Products and Services

Deposition of ultra-pure, custom-specified (ultra-)thin films and multilayers of metals and metallic alloys, (doped) metallic oxides and nitrides.

- Ultrathin silicon films and substrates
- Micro and nanostructuration of surfaces and thin films
- Ion implantation
- Thin film bonding

KEYWORDS

Thin film photovoltaics
TCO
Reactive and pulsed sputtering
Solar cells co-integrated with electronic circuits
Ultra-thin monocristalline Si pv cells
Nanostructured Si films
Organic solar cells bulk heterojunctions

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Nanomicrobiology

SENIOR SCIENTISTS :

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- Vincent DUPRES

Research Field and Subjects

Understanding the surface properties of microbial cells is a crucial challenge of current microbiology, and a key theme in biomedical research for understanding the molecular bases of microbial infections. Today, much is known about the structure and biosynthesis of microbial cell envelope constituents, but the three-dimensional organization and interactions of the individual components remain poorly understood.

With its ability to observe and force probe the cell envelope down to molecular level under physiological conditions, atomic force microscopy (AFM) has recently offered new opportunities in microbiology. While high-resolution AFM imaging is a powerful tool for visualizing the architecture of live cells in buffer solution, force spectroscopy offers a means to analyze the localization, interactions and elasticity of individual constituents such as receptors and sensors. These nanoscale experiments complement microscopy and biochemical methods traditionally used to analyse the microbial envelope, and open new avenues in nanomedicine for developing new antimicrobial strategies.

During the past 10 years, the "Nanomicrobiology" team at UCL has developed strong expertise in analyzing microbial cells with AFM-based techniques. This expertise is strengthened by numerous national and international collaborations with top-notch microbiologists. These nanoscale analyses provide key insights into the molecular bases of cell surface interactions, including mechanosensing and pathogen-host interactions.

Representative References

- ANDRE G., KULAKAUSKAS S., CHAPOT-CHARTIER M.P., NAVET B., DEGHORAIN M., BERNARD E., HOLS P. and DUFRENE Y.F., Imaging the nanoscale organization of peptidoglycan in living *Lactococcus lactis* cells. Nat. Commun. 1:27, **2010**.
- DUPRES V., ALSTEENS D., WILK S., HANSEN B., HEINISCH J.J. and DUFRENE Y.F., The yeast Wsc1 cell surface sensor behaves like a nanospring *in vivo.* Nat. Chem. Biol. 5, 857-862, **2009**.
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Funding

- National Foundation for Scientific Research (FNRS)
- Foundation for Training in Industrial and Agricultural Research (FRIA)
- Wallonie
- Federal Office for Scientific, Technical and Cultural Affairs (Interuniversity Poles of Attraction Programme)
- Research Department of the Communauté française de Belgique (Concerted Research Action)

Main Equipment

Nanoscope V Multimode and BioScope Catalyst AFMs

Products and Services

Nanoscale imaging of biosurfaces by AFM

KEYWORDS

Nanomedicine
Atomic force microscopy
Nanoscale imaging
Living cell
Microbes
Pathogens

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Micellar nanocontainers from block copolymers

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Research Field and Subjects

The self-assembly of amphiphilic block copolymers in selective solvents is intensively investigated. This sustained interest stems from the numerous potential applications of the resulting micelles in various fields such as drug or gene delivery, catalysis and separation techniques.

Amphiphilic block copolymers can self-assemble in a selective solvent into micellar aggregates showing a large variety of different morphologies such as spheres, rods, vesicles, platelets, toroids, and even more complex structures. Control over micellar morphology and size is generally achieved by adjusting the copolymer composition and architecture.

One of the aims of this project is to find new strategies for controlling the morphology of block copolymer micelles. In this respect, the effect of parameters such as solvent nature, the presence of additives (such as salts), use of mixed solvents, and the preparation method is investigated.

Another key issue in the design of block copolymer micelles lies in imparting stimuli-responsive properties to them. This project also deals with polymer blocks that can modify their solubility properties as a function of a stimulus such as pH, temperature, ionic strength or light. Inserting such a polymer block in to block copolymer micelles leads to the formation of so-called smart micellar nanocontainers that can either assemble or disassemble or modify the solubility of one of their compartments upon application of a stimulus. Such nanocontainers are particularly interesting for controlled encapsulation or release of molecules of interest.

Besides stimuli-responsive systems, multicompartment block copolymer micelles are also investigated. These nanocontainers have received special interest because they contain distinct immiscible non-soluble microdomains. Indeed, such micelles have some similarities with complex natural systems such as cell organelles, because both of them contain several miniaturized compartments of different character, properties, and mutual accessibility with exchange processes allowing selective transport. In order to produce distinct compartments in block copolymer micelles, the micellization of (block) copolymers containing non-soluble hydrocarbon and fluorocarbon segments is studied.

Mixtures of mutually interacting block copolymers to build up micelles with controlled morphologies and properties are also considered in this project. To reach this goal, non-covalent interactions such as ionic interactions and hydrogen bonds between polymer blocks are introduced. The resulting complexes may be insobuble in the used solvent and then aggregate into micellar cores stabilized by uncomplexed segments. Such micellar systems also exhibit stimuli-responsive properties.

Finally, some applications of block copolymer micelles are evaluated in this project. This includes the use of micelles as templating nanoreactors for the synthesis of noble metal nanoparticles, that are further used as catalysts, the use of micellar nanocontainers for drug release and the formation of smart micellar gels exhibiting self-healing properties.

Representative References

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Funding

2008-2011: Projet RW WINNOMAT II "Emulfive"
2008-2011: Projet RW WINNOMAT II "Chitopol"
2008-2013: ARC 08/013-010 "Supratune"
2005-2010: Réseau European Science Foundation "STIPOMAT"
2007-2011: IAP Phase VI "FS2"

Main Equipment

- Dynamic and static light scattering (Malvern-ALV CGS-3)
- GPC system (PSS) with triple detection (DRI, UV diode array, MALS).
- Atomic Force Microscope (Nanoscope V, Digital Instrument)
- Transmission electron microscopy (LEO922)

Products and Services

- Synthesis of (functionalized) block copolymers by controlled radical polymerization methods
- Self-assembly and characterization of micellar nanocontainers
- Characterization of colloids and nanoparticles

KEYWORDS
Nanomaterials
Micelles
Vesicles
Polymer synthesis
Stimuli responsive materials

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Pulmonary delivery of nanomedicines

SENIOR SCIENTIST :

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Research Field and Subjects

The large molecular size, hydrophilicity, chemical and enzymatic liabilities of peptides and proteins exclude the use of traditional oral dosage forms and injection is currently the most common method for their administration. The research team aims to optimize pulmonary delivery of proteins for local action in the lungs or systemic absorption, as an alternative to injection. The proteins studied include therapeutic proteins or vaccine antigens.

The research team optimizes the pulmonary administration of proteins by :

1. designing dry powder aerosols with elevated deep lung deposition,
2. selecting appropriate excipients,
3. understanding the biological losses encountered by inhaled macromolecules in the lung. In this respect, we demonstrated in rats that a primary source of elimination of macromolecules following delivery to the lung and prior to absorption into the bloodstream is throught clearance by alveolar macrophages.
4. targeting the appropriate site of deposition within the respiratory tract. For instance, we showed in mice that the deeper the antigen deposition within the lungs, the stronger the immune response.
5. chemically modifying the protein.

Products and Services

- Formulation of inhalation dry powders by spray-drying.
- Analysis of aerosols deposition in cascade impactors.
- *In vivo* studies of pulmonary drug absorption and deposition.
- *In vitro* studies of drug transport and uptake in monolayers of pulmonary epithelial cells and in macrophages.

Main Equipment

- Spray-dryer
- Sympatec laser diffraction system for particle sizing
- Pharmacopoeia cascade impactors
- HPLC with radioactivity detection

Representative References

- FERNANDES C.A. and VANBEVER R., Preclinical models of pulmonary drug delivery (invited review). Exp. Opin. Drug Deliv. 6 (11) : 1231-1245, **2009**.
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- MINNE A., HUAUX F., JAWORSKA J., RHA R.D., HAMELMANN E. and VANBEVER R., Safety evaluation of pulmonary influenza vaccination in healthy and "asthmatic" mice, Vaccine, 26: 2360-2368, **2008**.
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Awards

- V. Codrons, AAPS Graduate Award in Biotechnology, 2002.
- R. Vanbever, Named one of the world's top young innovators by Technology Review, Massachusetts Institute of Technology's magazine of innovation, 2003.
- A. Minne, Student Research Award of the International Society for Aerosols in Medicine, 2007.
- A. Minne, Award National Prize of the Belgian Society of Pharmaceutical Sciences, 2009.

Funding

FNRS, Wallonie

Partnership

- Scientific Institute of Public Health, Uccle
- UCB Pharma
- Université de Liège

KEY WORDS

Airway hyperactivity
Antigens
Drug delivery, non-invasive
Dry powder aerosols
Lung
Monoclonal antibody
Polymer
Proteins
Vaccine adjuvant

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Nanomedicine for tumor targeting

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Research field and subject

Recent advances in nanotechnology have offered new opportunities for cancer detection, and treatment to solve several limitations of conventional drug delivery systems such as nonspecific biodistribution and targeting, poor water solubility, poor oral bioavailability, and low therapeutic indices.

Nanomedicine (a nanometer scale system consisting of at least 2 components, one of which is the active ingredient) can target tumors via a passive or active process. Passive targeting implies that nanomedicines smaller than endothelial fenestrations can enter the interstitium and be trapped in the tumor (Enhanced Permeability and Retention effect). Active targeting involves drug delivery to a specific site based on molecular recognition. One approach is to couple a ligand to nanomedicines which can then interact with its receptor at the target cell site.

Of the nanomedicines, polymeric nanocarriers are particularly promising. Polymeric micelles (25 nm) and nanoparticles (150 nm) have been developed to deliver anticancer drugs, mainly poorly soluble drugs into tumors and siRNA.

Paclitaxel and cyclin-dependent kinase inhibitor loaded in micelles and PEGylated PLGA-based nanoparticles enhanced *in vivo* anti-tumor efficacy in experimental models compared to the reference formulation.
The preferential expression of αv integrins (especially $\alpha v\beta 3$) on endothelial cells lining tumor blood vessels (and tumor cells themselves) was exploited to target the nanocarriers to the tumors. New specific peptidomimetic ligands based on the RGD motive endowed with high affinity for αv integrins were developed. PLGA-based nanoparticles grafted with these ligands target tumor blood vessels and lead to a net tumor growth delay and prolonged survival of treated mice (*vs* non-targeted nanoparticles).

Besides expertise in the design and evaluation of targeted and untargeted nanoparticles loaded with anticancer drugs, novel nanocarriers for the delivery of siRNA to tumors are also evaluated.

Representative References

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- DANHIER F., LECOUTURIER N., VROMAN B., JÉRÔME C., MARCHAND-BRYNAERT J., FERON O., PRÉAT V., Paclitaxel-loaded PEGylated PLGA-based nanoparticles: In vitro and in vivo evaluation, J Control Release, 133: 11-17, **2009**.
- DANHIER F., VROMAN B., LECOUTURIER N., CROKART N., POURCELLE V., FREICHELS H., JÉRÔME C, MARCHAND-BRYNAERT J., FERON O., PRÉAT V., Targeting of tumor endothelium by RGD-grafted PLGA-nanoparticles loaded with Paclitaxel, J Control Release, 140: 166-173, **2009**.
- DANHIER F., MAGOTTEAUX N., UCAKAR B., LECOUTURIER N., BREWSTER M., PRÉAT V., Novel self-assembling PEG-p-CL-co-TMC) polymeric micelles as safe and effective delivery system for Paclitaxel, Eur J Pharm Biopharm, 73: 230-238, **2009**.
- BOUZIN C, FERON O., Targeting tumor stroma and exploiting mature tumor vasculature to improve anti-cancer drug delivery. Drug Resist Updat. 10:109-20, **2007**.
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Funding

FNRS, Télévie, Wallonie (Biowin TARGETUM), IWT

Partnership

- Université de Liège
- Kitozyme
- UCB
- Johnson & Johnson

Main Equipment

- Nanosizer ZS
- IVIS Xenogen
- Intravital microscopy set-up

Products and Services

- Formulation of anticancer drug loaded nanoparticles
- Biodistribution and efficacy of anticancer drug loaded nanoparticles
- Photografting of ligands on polymers

KEYWORDS

Nanoparticles
Nanomedicine
Tumor targeting
Tumor vasculature
Integrins
SiRNA

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Nanoparticles for oral vaccine delivery

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- Anne DES RIEUX

Research field and subject

Many efforts have been directed towards the development of patient-friendly, needle-free vaccine administered by the mucosal route, including the oral route. To overcome the drawbacks associated with oral delivery of vaccine, the strategy of encapsulating the antigen(s) in polymeric nanocarriers is particularly attractive. Polymeric carriers can protect antigens against degradation and inactivation in the harsh gastro-intestinal environment, have the ability to enhance their transmucosal transport and act as adjuvant for mucosal immunisation.

M-cells in the follicle associated epithelium (FAE) of intestinal Peyer's patches and isolated lymphoid follicles are gatekeepers of the mucosal immune system. Given their unique features to transcytose particles, M-cells are an interesting target in oral vaccine delivery to transport antigen to underlying immune cells. Using *in vitro* models of human FAE (Caco-2 cells cocultured with Raji cells) the contribution of M-cells to the transport of nanoparticles was demonstrated. Grafting new ligands of M cells identified by phage display or RGD that targets integrin specically expressed by M cells, significantly increased nanoparticles in vitro transport.
Intraduodenal immunization with nanoparticles grafted with ligands targeting intestinal M cells or antigen, presenting cells and loaded with a model antigen elicited a higher production of IgG antibodies than intramuscular injection of free antigen or intraduodenal administration of non-targeted nanoparticles. Targeted formulations were also able to induce a cellular immune response.

Bioadhesion could also enhance antigen delivery. Chitosan (CS) particles have been described as potential oral vaccine carriers. Nanoparticles made from chitosan (CS) and its N-trimethylated derivative, TMC, loaded with a model antigen were prepared by ionic gelation. Intraduodenal vaccination with nanoparticles led to significantly higher antibody responses than immunization with atigen alone. TMC nanoparticles but not CS or PLGA nanoparticles had intrinsic adjuvant effect on human monocyte derived dendritic cells.

Representative References

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- DES RIEUX A., FIEVEZ V., GARINOT M., SCHNEIDER Y.J., PRÉAT V., Nanoparticles as potential oral delivery systems of proteins and vaccines: a mechanistic approach, Journal of Controlled Release, 116:1-27, **2006**.

Awards

Laurence Plapied, Prix Sanofi-Aventis, GTRV 2009

Funding

FNRS, Wallonie

Partnership

- Leiden Amsterdam center for drug research
- Université de Liège
- Kitozyme

Main Equipment

- Nanosizer ZS (Malvern)
- Cell culture

Products and Services

- Caco-2 permeability studies
- Nanoparticle formulation

KEYWORDS

Nanoparticles
M cell
Vaccine
Immune responses
Bioadhesion
Chitosan

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Electrical Detection of DNA and Pathogens

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- Jean-Luc GALA
- Benoît MACQ
- Sorin MELINTE
- Jean-Pierre RASKIN

Research Field and Subjects

Rapid, real-time detection of pathogenic microorganisms is an emerging and fast evolving field of research, especially with respect to microorganisms that pose a major threat to public health.

Electonic DNA analysis also appears to be of major interest in view of the miniaturisation of bioanalytic systems such as labs-on-chip for low-cost diagnosis or point-of-care applications. This still presents numerous scientific and technical challenges, especially with respect to problems of compatibility between integrated electronic circuits and biochemical species in aqueous and ionic solutions, as well as to the extreme levels of sensitivity and specificity to be achieved.

In this context, the research group has developed interdigitated capacitive microsensors that are used both for the detection of micro-organisms and DNA hybridization. The interdigitated fingers are covered by metal oxides that are synthesised by anodising thin metal films.

In the case of micro-organism analysis, the sensors have been tested for bacterial detection by coating them with an anti-Staphylococcus aureus monoclonal antibody (MoAb), which provides a significant capacitance shift with very good selectivity between a positive sample, Staphylococcus aureus, and a negative one, Staphylococcus epidermidis. Furthermore the capacitance variation appears to be proportional to the number of bacteria grafted on to the chip and is compatible with the specific detection of less than 100 bacteria.

At the same time, micro-organism detection is also being carried out using engineered phages that are placed on the surface of the metal oxide layer. The use of phages is believed to be a much more selective and durable alternative to conventional surface functionalisation techniques, for both diagnostic and therapeutic purposes.

Concerning DNA hybridization detection, the sensors have been successfully tested with an ultimate limit of detection down to atto-mole level. Measurements of DNA/RNA concentration in assay tubes by UV light and specific photodiodes have been performed with greater sensitivity than existing lab equipment. Optical measurements in microfluidic platforms are also targeted.

Finally, in addition to sensor production, the research group carries out its biological experimentation and statistical data processing of the electrical results.

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Patents

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- *Insulated Substrate Impedance Transducers.* EP06018835.6, **2006**.
- *Genetically modified bacteriophage, biosensor containing same, and method of use*, US Patent Number US 61/186 538, 2009.

Funding

- NANOTIC – Programme d'excellence de la Wallonie, 2005-2010.
- DNA-SIP – MNT-ERA Programme, funding from the Wallonie

Partnership

- Coris Bioconcept
- ULg-Microsys
- IMT-Bucharest
- Genetic Lab en Roumania

Main Equipment

- The largest research-oriented clean room facilities (1000 m^2) in Wallonie with the all key technologies required for micro/nano-systems and micro/nano-electronics fabrication.
- Characterization techniques : optical and electrical in a large range of frequencies and temperature.
- Technological platform for molecular genetic analyses

Products and Services

Fabrication and characterization of electrical and optical bio-micro-nano-sensors :

- Variable capacitive structures
- Microwave detectors
- Inorganic nanowires
- Microfluidic channels
- Silicon-on-Insulator photodiodes

Optimization of pre-analytical procedures
Validation in clinical setteing
Statistical data set analysis
Bioinformatic analysis

KEYWORDS

Electrical detection
Bacteries, Phages
Micro-sensors
UV photodiode
DNA hybridization
DNA/RNA concentration

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Engineering enzymes for biosensors

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Research Field and Subjects

Research efforts are devoted to the genetic engineering of enzymes as alternatives to antibodies. Artificial binding sites are created by extending loops on the surface of a chosen enzyme. Using phage display technology, chimerical enzymes that specifically bind to potentially any kind of target molecule or material can be selected from large libraries of mutants. In most cases, binding of such a selected enzyme to a target is accompanied by modulation of its catalytic activity. These regulation effects can be very useful for detection purposes when these enzymes are incorporated into biosensor devices. Compared with antibodies, these engineered enzymes feature similar binding properties but with many advantages: they are smaller, easier to produce or refold, easier to genetically manipulate and, last but not least, endowed with catalytic activity that can be used for detection.

Using this strategy, enzymes that specifically bind to monoclonal antibodies, other globular proteins, small organic compounds and metallic ions have been successfully selected and characterized.

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Patents

- Chimeric enzyme molecules having a regulatable activity for use in assays, PCT Int. Appl., WO 9823731
- Method for the selective survival or selective growth of a target cell by the use of a conjugate, its use in therapeutics and/or diagnostics and the preparation of the said conjugate, PCT Int. Appl. WO 0197854

Partnership

- Partner in the program *Pôles d'attraction interuniversitaires* (PAI) on "Protein function and supramolecular assemblies"
- Partner of the European Integrated Training Network (ITN) : European network on directed evolution of functional proteins
- Partner of the Nanotic program (RW): "Essaims de senseurs intelligents"
- DelphiGenetics, Braine l'Alleud, Belgium

Main Equipment

- High sensitivity UV-Vis spectrophotometer
- Molecular biology equipment

Products and Services

- Libraries of phage displayed enzymes

KEY WORDS

Diagnostic tools
Directed evolution
Enzyme engineering
Immunoassays
Protein engineering
Biosensor

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Engineering bacteriophages as programmable nano-objects

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Research Field and Subjects

Bacteriophages are nanoscopic supra-molecular assemblies containing genetic material (DNA or RNA) encapsidated into a proteic coat. Using molecular biology techniques, they can be easily manipulated and evolved to give them unnatural properties. Research efforts are devoted to the engineering of new properties on filamentous bacteriophages. Controlling the length and binding properties along the rod and at both tips of the particle transforms these phages into attractive autoassembling nano-wires for bioelectronic devices.

Specific research projects are focusing on the simultaneous functionalization of 2 or 3 different coat proteins of the filamentous particles and on the engineering of particles that specifically bind to metal oxide surfaces.

As a first example, the genetic selection of filamentous phages that specifically bind to anodic alumina was demonstrated and is opening a new biological route to surface functionalization that could offer several advantages over classical chemical functionalization (robustness, safety).

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Patent

- Genetically modified bacteriophage, biosensor containing same, and method of use, US Patent Appl. US 61/186 538.

Partnership

- Partner in the program *Pôles d'attraction interuniversitaires* (PAI) on "Protein function and supramolecular assemblies"
- Partner of the European Integrated Training Network (ITN) : European network on directed evolution of functional proteins
- Partner of the Nanotic program (wallonie): "Essaims de senseurs intelligents"
- DelphiGenetics, Braine l'Alleud, Belgium

Funding

- Nanotic program (Wallonie): "Essaims de senseurs intelligents"

Products and Services

- Libraries of phage displayed peptides and proteins.

Main Equipment

- High sensitivity UV-Vis spectrophotometer
- Molecular biology equipment

KEY WORDS

Diagnostic tools
Directed evolution
Enzyme engineering
Immunoassays
Protein engineering
Biosensor

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Nanotoxico : Effect of manufactured nanoparticles on human health

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- Joseph DELHALLE
- Bernard MASEREEL

Research Field and Subjects

In the near future, products based on nanotechnology will impact most industrial sectors and enter the consumer markets in large volumes. However the unique physicochemical properties of manufactured nanomaterials raise concern about their potential adverse effects on human health and the environment.
Currently, there is limited understanding of the correlation between the physicochemical properties of the nanomaterial and their biointeractions. Research is needed to determine the effects of physicochemical properties on nanomaterial behaviour.
Nanotoxico is an interdisciplinary research platform based at the University of Namur that includes chemists, physicists, biologists, and pharmacists whose joint efforts lead to the development of toxicity assays for nanomaterial safety assessment. Particular attention is given to *in vitro* testing in order to comply with EU policies on cosmetics (*Council Directive 2003/15/EC*) and chemicals (REACH) that promote the use of alternative methods to animal testing. Since the current *in vitro* test methods are likely to be influenced by nanoparticle-specific properties, extensive characterization of nanoparticle properties and validation of test systems are performed.
The chemists and physicists working on the platform have gained outstanding expertise in nanosciences and nanotechnology through their participation in various high level research programs leading to the set up of appropriate methods of nanoparticle characterization (e.g. size, size distribution, shape, elemental composition, crystallography, surface area and surface chemistry).
One of the assets of the Nanotoxico platform is the use of engineered tissues mimicking the design and function of normal human tissues, an important step forward for regulatory toxicology (and much more realistic than cell cultures). These *in vitro* tissue models mimick important functions of the skin, lung and gut that constitute the main potential routes of contact with nanoparticles. In addition, animal studies are carried out to perform subchronic or chronic exposures that monitor the fate of nanomaterials : their entry into the body, possible modifications and excretion or storage in the body.

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Patents

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EP 02447039.5 – 20/03/2002, WO 03/07/8315 A2-
- Radiotherapy device and method 2006, WO2006063419 -
- Radioactive device 2006, WO2006063418

Funding

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Partnership

The FUNDP-Nanotoxico platform is a validated supplier of the Institute for Reference Materials and Measurements Reference Material Unit (IRMM-European Commission-Joint Research Centre) for nanoparticle characterisation via centrifugal liquid sedimentation and electron microscopy.

Main Equipment

- Applied Biosystems 7900 HT real time RT-PCR for studying gene expression.
- 2 MeV Tandetron linear accelerator (Altaïs) for nuclear reactions based spectroscopy.
- Field Emission Gun - Scanning Electron Microsocpe JSM-7500F /Jeol (resolution 0.6 nm) with EDX detector.
- CPS 2400 Disc Centrifuge for nanoparticle size analysis.
- *In vitro* models of skin, small intestine and lung suitable for nanotoxicology

Products and Services

- Trace element detection or biopersistence studies by Particle Induces X-ray emission (PIXE) analysis.
- Nanoparticle size distribution measurements using the differential sedimentation method.
- *In vitro* nanotoxicological testing

KEY WORDS

Health safety
Nanomaterials
Toxicity
Physico-chemical properties

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Nanoparticles in food production: an *in vitro* evaluation of risks and toxicity

SENIOR SCIENTISTS:

- Yves-Jacques SCHNEIDER
- Alexandra BAZES

Research Field and Subject

Nanoparticles (NPs) are used in a wide range of applications in science, technology and medicine. For instance, the research group, in collaboration with the Louvain Drug Research Institute (LDRI), has shown its interest in drug and mucosal vaccine delivery. Nevertheless, it is also of great concern to assess the potential negative impacts that nanomaterials, currently present in food and feed as well as in our environment, may have on biological systems. Toxicity testing of NPs requires that the end dose-response relationships can be described, for either *in vitro* or *in vivo* tests.

Caco-2 cells are a human intestinal cell-line, which mirrors the absorptive epithelium of the intestine. Hence the "Nanomaterial Toxicity Screening Working Group", from the Risk Science Institute recommended the use of this cell line for *in vitro* testing of the ingestion of NPs. Using this model, the studies should focus on the evaluation at intestinal level, of: (i) direct toxicity; (ii) possible pro-inflammatory stimulation with e.g. disruptions in tight junctions, which would increase paracellular permeability, cytokine secretions,…; (iii) transport of NPs through the intestinal epithelium. In particular, it should be taken into account that NPs would probably cross the intestinal epithelium *via* the M cells of the follicle associated epithelium. An *in vitro* model of this barrier was previously developed by the research group. Furthermore, the intestinal hydrophilic mucus layer physiologically covering, the epithelium would also play a key role in the adsorption of NPs onto epithelial cells and their transport across the intestinal barrier. *In vitro* models also exist in our lab to mimic this important aspect.

To work as closely as possible to an *in vivo* situation using *in vitro* methodology, upon oral exposure to nanoparticles, the physical and chemical properties of the material are characterized in the form delivered to humans or animals using appropriate physico-chemical methods. Four complementary experimental steps are under investigation in close collaboration with the CERVA within a Federal Public Service, Public Health, Food Safety, funded project. (i) The NPs are first characterized in a matrix as close as possible to the food product. (ii) The NPs bioaccessibility is assessed and *in vitro* tools have been developed to mimic the different steps of food digestion. (iii) The absorption of NPs is evaluated by different models, *i.e.* Caco-2 cells, as model of enterocytes, Caco-2 cells converted into M like cells, both in the presence or absence of mucus secreted by goblet cells. (iv) The possible interactions with the intestinal barrier are investigated, with special emphasis on direct (necrosis) or indirect toxicity, with particular attention focused on the possible activation of intestinal inflammation and related transduction cascades, as well as biotransformation and efflux activities.

Representative References

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Products and Services

- Digestion of food matrices.
- Evaluation of the absorption of NPs in 3 different models (enterocytes, M like cells, mucus layer).
- Study of the activation of intestinal inflammation, biotransformation and efflux activities.

Main Equipment

Infrastructure for animal cell technology, molecular cell biochemistry, confocal microscopy …

Funding

SPF/Public Health: RT 10/05 NANORISK

Partnership

- CERVA, Tervuren
- it4ip, Seneffe

KEYWORDS

Nanoparticles
Food, toxicity
In vitro cell culture systems
M cell
Mucus
Inflammation

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Respiratory toxicity of nanomaterials

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Research Field and Subjects

The research group has long experience in investigating the mechanisms of toxicity of inhalated particles (cobalt-containing alloys, metals, Indium Tin Oxyde (ITO), silica, nanosilicas, carbon nanotubes …) on the respiratory tract.

Models and methods (experimental animals and cell cultures) are available to assess inflammatory and fibrotic reactions of the lung, as well as the genotoxic and carcinogenic potential.

The main focus is to investigate how these reactions are affected (possibly abrogated) by modulating the physico-chemical characteristics of the particles (surface reactivity, size, doping…).

Recent achievements:

- demonstration of the unique toxicological properties of ITO particles,
- characterization of the toxic responses of the respiratory tract to carbon nanotubes and identification of the role of surface defects as underlying mechanisms,
- development of silica nanoparticle models to investigate the importance of surface area and porosity in toxic reactions.

Representative References

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Funding

- Wallonie, EU DG Research: BINANOCO project
- Federal Public Service for Science Policy (BELSPO), Health and Environment: S²NANO project

Partnership

- Università degli Studi di Torino
- KULeuven

Products and Services

Assessment of the respiratory toxicity and identification of the determinants of toxicity.

Main Equipment

Laboratory of experimental toxicology: *in vivo* (rats, mice) and *in vitro* (cell culture).

KEY WORDS

Inhalation
Lung
Particle
Nanoparticles
Nanotubes
Toxicity
Toxicology

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Interuniversity Scientific Computing Facility

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- Philippe LAMBIN
- Daniel P. VERCAUTEREN
- Frédéric WAUTELET
- Johan WOUTERS

Research Field and Subjects

The interuniversity Scientific Computing Facility (iSCF) was created in 1985 to help Belgian scientists working in the fields of theoretical physics and chemistry to access powerful computing resources in a constructive spirit of know-how sharing. Since then, the computing resources have roughly doubled every two years, reaching a theoretical performance of 9 teraFLOPS in the current cluster configuration.

The organization of the center is still thematic and oriented towards solving problems in computational physics and chemistry, bringing in the simplification of a coherent tool optimized for a focused objective. At the nanoscale quantum chemistry and numerical solid state physics merge and share common numerical tools.

The iSCF allows one to perform quantum chemistry calculations with growing complexity and sophistication, in that the systems under study today are clearly in the scale range of macro-molecules, supramolecules, and nanostructures. On the other hand, physics of solid materials extend into the less symmetric cases of structured materials and nanoparticles. Present investigations tackling chromophores, proteins and DNA strands in interaction with drugs, carbon (nano)clusters, nanotubes and (nano)sheets (including graphene), and inorganic mesoporous materials address their structural, thermodynamical, electronic, optical, magnetic, and vibrational properties.
Over recent years the iSCF project has also included theoretical developments in the field of photonics, as needed for the control of light fluxes in transport, photovoltaic devices, logical devices. In this direction, new methodologies are proposed to address biomimetic materials and plasmonic systems. For example, the reverse engineering of natural structures implies developing large-scale simulations of electromagnetic waves propagation in highly complex structures.

Representative References

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- LIÉGEOIS V., RUUD K. and CHAMPAGNE B., An analytical derivative procedure for the calculation of vibrational Raman optical activity spectra, J. Chem. Phys. 127, 204105, **2007**.

Awards

- Triennial Prize of the Belgium Royal Society of Chemistry (2003: Benoît CHAMPAGNE, 2009: Johan WOUTERS)
- In 2009, Luc HENRARD received a ‘Special recognition diploma’ by the World Cultural Council

Funding

FRS-FNRS, European (FP6, FP7), Wallonie, Communauté française (ARC), IAP 6/27

Partnership

- University of Mons (The iSCF is a shared high-performance computing center with UMons)
- University of California in Santa Barbara (USA)
- University of Bordeaux (France)
- University of Sherbrooke (Canada)
- KULeuven, UCLouvain, UHasselt, UGent, ULB
- Université Pierre et Marie Curie – Paris 6 (France)
- Université Diderot – Paris 7 (France)
- Natural History Museum – London (UK)
- Linköping University (Sweden)
- University of Stockholm (Sweden)
- Moscow State University (Russia)

Main Equipment

- iSCF cluster consists of 85 computing nodes for a total of 792 cores with a peak performance of 9 TFLOPS, 6 interactive nodes from which jobs can be submitted to the job scheduler to run on the nodes and two file servers with about 45 TB of disk space available. There are two main types of computer nodes:
- 58 nodes with 16 or 64 GB of memory, dual Quad-Core Intel Xeon X5460.
- 25 nodes with 36 or 72 GB of memory, dual Six-Core Intel Xeon X5650.

Products and Services

- Elaboration and implementation of methods of theoretical chemistry and physics as well as of optics.
- Simulations of structural, electronic, optical, magnetic, and vibrational properties of molecules, supramolecules, clusters, polymers, surfaces, and crystals.

KEYWORDS

Theoretical and Quantum chemistry
Theoretical Physics
Scientific Computing
Electronic properties
Optics
Photonic crystals
Biophotonics
Plasmonics

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First-principles simulations of nanostructures

SENIOR SCIENTISTS :

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- Xavier GONZE
- Gian-Marco RIGNANESE

Research Field and Subjects

The research activities concern the *ab initio* modelling of the structural, dynamical, electronic, dielectric, magnetic and optical properties of nanostructures. In particular, such first-principle simulations allow to predict these properties at the atomic scale in the framework of the density-functional theory (without empirical data).

The scientific research focuses on nanoscale materials used in conventional silicon-based technology (Metal/Oxide/Silicon, in particular with new high-k dielectrics), in photovoltaïcs, and in light-emitting diodes (LED), as well as on novel materials to be used in more prospective technologies such as quantum electronics (in particular carbon nanotubes, graphene-based systems and organic molecules).
The studies address properties directly related to industrial and experimental needs such as electronic properties (e.g. band offsets), dielectric constants, transport properties (e.g. quantum conductance and I-V curves) and optical properties.

The numerical simulations are based on the ABINIT code. The ABINIT code allows one to find properties of systems made of electrons and nuclei (periodic solids and nano-systems) on the basis of quantum mechanics and electromagnetism (first-principle calculations). Atomic geometries are optimized according to the forces and stresses, molecular dynamics simulations can be done and vibrational or dielectric properties can be also addressed. Excited states can be computed within the Time-Dependent Density Functional Theory (for molecules) or within Many-Body Perturbation Theory (the GW approximation). The development of ABINIT, to which several dozen researchers contribute worldwide, is synchronized at the UCL.

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Funding

EU (FP6,FP7), PAI, ARC, Wallonie, FNRS, IWT

Main Equipment

Our intensive scientific calculations are performed on a serie of UCL computers including:

- LEMAITRE (256 Procs - AMD Opterons 252 / 2.6GHz - 1330 GFlops)
- GREEN (824 Procs - DUal Quad core Intel Xeon / 2.5GHz - 8240 GFlops)
- Apple Cluster Xserve under MacOS X 10.3 (18 Xserver Nodes, XRaid 1.7TB)

Products and Services

An expertise in first-principles simulations is proposed to interested industrials and experimentalists in order to answer their specific demands and to help them to solve nanotechnological issues.

Partnership

- Université de Liège (Belgium)
- Commissariat à l'Energie Atomique - Grenoble (France)
- Commissariat à l'Energie Atomique - Arpajon (France)
- Institut Néel - Grenoble (France)
- Ecole Polytechnique Fédérale de Lausanne (Switzerland)
- Massachusetts Institute of Technology - Cambridge (USA)
- Rensselaer Polytechnic Institute - Troy (USA)
- University of Dalhousie (Canada)
- CINVESTAV Queretaro (Mexico)
- PICyT (Mexico)

Within networks:

- ETSF: European Theoretical Spectroscopy Facility (E-infrastructure)

KEYWORDS

Ab initio simulations
Condensed matter theory
Electronic and dielectric properties
Transport properties
High-k materials
Semiconducting nanowires
Carbon nanostrustrures
Molecular electronics

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http://www.etsf.eu
http://www.uclouvain.be/cism

Open-shell Organic Molecules with Outstanding Electrical, Magnetic, and Optical Properties

SENIOR SCIENTISTS :

- Benoît CHAMPAGNE
- Edith BOTEK

Research Field and Subjects

Though most applications in electronics and optoelectronics are based on closed-shell π-conjugated systems, open-shell compounds turn out to be serious alternatives in that they display outstanding electrical, magnetic and optical properties. This research focuses on the elaboration of new stable open-shell π-conjugated compounds by adopting a joint theoretical-experimental approach that combines synthesis with characterization and modelling. The theoretical emphasis is put on the development and application of quantum chemistry methods to characterize these systems properties as well as to deduce structure-property relationships. That will contribute to design new and more efficient compounds and materials.

The main targeted compounds are diradicals and multi-radicals, a class of open-shell compounds with an even number of electrons, whose electronic structure – and its variations – are closely related to the chemical bond itself, its formation and its breaking, in other words its instability. These compounds are ubiquitous in Chemistry, including in thermal sigmatropic migrations such as Cope rearrangement, efficient singlet fission chromophores for dye-sensitized solar cells, polynitrenes and nitreno-radicals, spiro-fused oligo(triarylamine)s, and diarylethene with nitronyl nitroxide radicals.

The research focuses mostly on the specific linear and nonlinear optical properties of these diradicals and multi-radicals, addressing not only the isolated molecules (or molecules in solution) but also aggregates where intra- and inter-molecular covalent bonds coexist originating from the open-shell character of the constitutive molecules. Another direction of investigation concerns the effects of spin multiplicity, directly linked to their magnetic properties and to spintronics. Currently this research has considered a broad range of compounds based on phenalenyl units and graphene nanoflakes as well as compounds containing transition metal atoms.

A fundamental understanding of the electronic structures of these systems is expected to generate novel properties originating from the multi-diradical electronic structure.

Representative References

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Funding

- F.R.S.-FNRS/JSPS – "Joint Research Projects – FY 2009".
- F.R.S.-FNRS

Partnership

- Osaka University, Department of Materials Engineering Science, Toyonaka, Osaka 560-8531, Japan
- Osaka University, Department of Chemistry, Toyonaka, Osaka 560-8531, Japan,
- Research Institute for Ubiquitous Energy Devices, National Institute of Advanced Industrial Science and Technology (AIST), Ikeda, Osaka 563-8577, Japan.

Main Equipment

Interuniversity Scientific Computing Facility

KEYWORDS

Ab initio simulations
Diradicals and multi-radicals
Hyperpolarizabilities
Nonlinear optics
Open-shell systems
Two-photon absorption

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NEGF Quantum Simulations of Ultra Scaled Silicon Devices for the Advanced Nano- and Bio-Electronics Era

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- Denis FLANDRE

Research Field and Subjects

In a continuous effort to increase current drive and better control short-channel effects, electron device dimensions are shrunk. For ultra-scaled devices with cross-section dimensions smaller than 10 nm and channel length, L, of a few nanometers to a few tens of nanometers, quantum effects are playing a crucial role on device performances and parameters. At these scales, properties that were used to be considered as material properties in bulkier devices, like bandgap and band structure for example, are now becoming device properties and really depend on the size and shape of the device. This potentially opens a full new range of perspectives and applications. However, in order to fully benefit from the scaling new device architectures and new simulations tools are needed to exploit at their best these new effects. According to many experts, future scaling below 16 nm node (i.e. in about 10 years) will be based on a total change of paradigm, which directions have not even been established yet!

The research here is based on the building of efficient quantum simulation tools for device simulations, (using the NEGF (Non-Equilibrium Green's Function) method which has shown a real possibility to capture the essential physics at these scales). The simulator is then used to understand, predict and design electronics properties in nowadays and next generation state-of-the-art nano-electronics devices, such as silicon quantum nanowires. Emphasis is placed on exploratory research using the dependency of quantum effects to the device structure at the nanoscale to create disruptive device architectures and paradigms. Effects of structural modification have been explored and have led to new fully CMOS-compatible nanowires using tunnel effects with performances that push beyond the limits of classical devices and theory.

In addition, nanowires are very promising as sensors, in particular for the sensing and detection of biological systems (e. g. DNA or cells) and chemical gases. Nanowires indeed combine a very high sensitivity with a size comparable to many molecules of interest allowing one to sense biological and chemical phenomena with an unpreceded level of precision. In some cases single molecule detection and / or coupling can be achieved (e. g. coupling a nanowire to a neuron which allows one to sense the neuron activity very precisely and in real time). The resulting bio-electronics field is a new but rapidly growing field because of the huge and revolutionary potential for applications and scientific progress it can bring in the sector of medical and life science with social benefits (e.g. rapid disease diagnostic). The quantum simulator built here uses general finite-element multiphysic (FEM) softwares: Comsol multiphysics and Matlab. It brings the possibility to solve easily together coupled problems including not only electrical variations but also temperature, illumination, fluxes… One of the goals of the research project undertaken here is to build a co-integrated quantum bio-electronics simulator, reusing the code to simulate the nanowires for the electronic part and developing new codes to simulate the electrical interaction of the biological system on these nanowires.

Representative References

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Patents

Tunnel nanowire transisitor PCT/EP2009/005317, 22.07.2009, WO/2010/009873, 28.01.2010.

Funding

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Partnership

- Tyndall National Institute
- NANOSIL /SINANO networks

Products and Services

Expertise in physics and simulation techniques of advanced nanoscale transistors including quantum effects, scattering and ballistic transport, tunneling and resonant tunneling mechanisms, Schottky barriers… Advanced NEGF simulationtools

KEYWORDS

Nanoelectronics
Bio-electronics
Quantum NEGF Simulation
Si
SOI
Nanowire
Resonant Tunneling
MOSFET
Chemical sensors

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Wallonia ELectronics and COmmunications MEasurements : a facility making a bridge from molecules to signals

SENIOR SCIENTISTS :

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- Pascal SIMON
- Pierre GERARD

Research Field and Subjects

Benefiting from the expertise developed by UCL for over 25 years in electrical measurements, the WELCOME facility is a state-of-the-art technological platform providing multidisciplinary tools in the field of electrical and electromagnetic characterization. For over 10 years, an expertise in the field of nanosciences and nanotechnology has been jointly developing with the Institut de la Matière Condensée et des Nanosciences (IMCN) of UCL.

Available tools and techniques in the WELCOME facility result from various research axes including micro- and nanotechnology (materials and devices), Silicon-on-Insulator technology, Radiofrequency (RF) and microwave circuits, digital systems and VLSI architectures, Micro/Nano Electro Mechanical Systems (MEMS/NEMS), cryptography, ultra low-power wireless (bio)sensors, molecular electronics, wireless communications between sensors. WELCOME intends to cover and integrate in a broadband and rather unified approach various aspects of electrical and electromagnetic characterization of nanomaterials and sensors, going from the physical behaviour of materials and electron devices to systems architectures and ultra-wideband communications protocols between them.

Research in nanoscience and nanotechnology field covers in WELCOME the following fields:

- nanoelectronics, through the characterisation of active nano-devices over a broad frequency range (from DC to 110 GHz);
- nanospintronics, through the characterisation of ferromagnetic nanowires at microwave frequencies for the design of novel RF devices;
- electromechanical characterisation of MEMS/NEMS sensors for RF and biomedical applications;
- carbon-based nanocomposite materials (carbon nanotubes and graphene) for tailoring the propagation of electromagnetic waves;
- molecular electronics and Scanning Tunneling Microscope imaging of nanostructures

Representative References

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Patents (since 2005)

- EP2151056 (A1) 2010-02-10 ultra-low-power circuit
- WO/2009/092771 network architecture for wirelessly interfacing sensors at ultra low power
- WO2009087125 (A2) 2009-07-16 double-gate floating-body memory device
- WO2008068042 (A2) polymer composite material structures comprising carbon based conductive loads
- WO2008098993 (A2) 2008-08-21 imposing and determining stress in sub-micron samples
- WO/2007/093018 internal stress actuated micro- and nanomachines for testing physical properties of micro and nano-sized material samples
- WO/2005/031842 method of manufacturing a multilayer semiconductor structure with reduced ohmic losses

‣ WO/2005/031853 process for manufacturing a multilayer structure made from semiconducting materials
‣ US 2005/0227373 method and device for high sensitivity detection of the presence of dna and other probes

Funding

‣ EC FP6 and FP7 programs (Nanosil, Sinano, Harcana, Metamorphose, METACHEM…)
‣ ESF COST actions
‣ FRS-FNRS and FRIA
‣ IWT
‣ FEDER (Project Minatis)
‣ PAI
‣ Wallonie, DGO6 projects (Nanotic, Plan Marshall "PICOM", Winnomat "Multimasec" and "Dinosaure", PIT "ATAC"…)

Partnership

The WELCOME facility is complementary in many aspects to resources and skills available in nearby research centers active in similar fields. This complementarily favors fruitful collaborations in the frame of numerous projects funded at the regional and federal level and ensures its international competitiveness.
‣ Belgian collaborations (non exhaustive list): *Academic:* Cenaero, Certech, Cetic, Sirris, Materia Nova, IMEC, KULeuven, Multitel, UGent, University of Liège, ULB/VUB (Brussels), Univ. MonsHainaut&FPMS, FUNDP (Namur), IEMN (F)…
Industrial: BER, Melexis, nSilition, Nanocyl, CISSOID, IMEC, AGC-Flat Glass, Solvay, On Semi Belgium, Arcelor Mittal, Thalès Alenia Space Etca, Thalès Communications Belgium.
‣ International collaborations:
Academic: partners of collaborative projects listed above
Industrial: SOITEC (F), RFMD (USA), ST-Microelectronics (F), IBM (USA), OKI (J), EADS (F, D), Thalès TRT (F)…

Main Equipment

‣ Various coaxial setups, on-wafer probe stations and Vector Network Analyzers configurations to achieve a multi-port (up to 4 accesses) and multi-parametric characterization of (nano) materials, electron devices and sensors (IV, CV, temperature-microwave, electro-mechanical, and magneto-electrical sweeps…), in small-signal and nonlinear regime, from DC to 110 GHz, in the temperature range 4K-500K.
‣ low current probes, logical analyzers, analog waveform and digital pattern generators, digital oscilloscopes, vector signal generators with I/Q or digital modulation for analog/digital circuits and systems-in-package (smart cards, RFIDs, FPGAs) interfacing with (nano)sensors
‣ Scanning Tunnelling Microscope
‣ climatic rooms (for measurements in controlled humidity and temperature environment)
‣ Polytech vibrometer for interferometric measurements of Micro and Nanosystems
‣ anechoic chamber for electromagnetic testing of nano-composite materials and sensors
‣ RF hardware for wireless and ultra-wideband communications between (nano)sensors

see full catalog list on: http://sites-test.uclouvain.be/welcome/WebBooking/help/facilities.pdf

Products and Services

WELCOME already answers needs of industry in Belgium and abroad, in the frame of (sub)contracts and consultancy. With some regulation policy, trainings for R&D engineers from industry are also possible.
‣ DC and RF characterization of active and passive (nano) devices and sensors, under various bias, and in temperature range 4°K-500K
‣ 3-D dynamic characterization of MEMS and MOEMS microstructures (in and out-of-plane vibrations, surface topography under pressure stimuli)
‣ Electrical characterization of various bulk and composite materials (liquid, solid, film, powder), from DC to 75 GHz
‣ Evaluation of EMI shielding and radiation performances of materials and sensors in anechoic chamber

KEYWORDS

Nanoelectronics
Nanospintronics
Nanocomposites
Molecular electronics
MEMS/NEMS
Nano(bio) sensors
Multi parametric characterisation

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Nanomechanical testing of thin films, coatings, microsystems and heterogenous materials

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- Thomas PARDOEN
- Joris PROOST
- Jean-Pierre RASKIN

Research Field and Subjects

Thin films and coatings have become ubiquitous in modern technologies, with applications ranging from microelectronics and and microsystems such as MEMS to surface functionalization and protection. In many instances, mechanical reliability, coupled to other phenomena, is the main limiting factor for industrialisation. Now, nanomechanics of thin films or heterogeneous systems is also a field of fundamental scientific interest involving phenomena that do not exist in macrosystems, providing near ideal system to investigate elementary mechanisms, or opening to new couplings with electrical, magnetic and chemical effects. In the present context, by "thin", it is meant "smaller than 1 micrometer".

The research group has developed a range of testing techniques and procedures, some of them being patented, to address the mechanical properties of thin films and/or systems involving at least one nanoscopic dimension with the goal not only to measure properties but also to unravel the underlying mechanisms. Measuring the mechanical response of nanosamples is extremely challenging, from the preparation and manipulation of specimens to the application of small loads and extraction of accurate stresses and strains.

1. A new concept of nanomechanical lab-on-chip involving suites of thousands of different test structures has been developed to measure the elastic modulus, strength, ductility, fracture stress, internal stress, crack and creep resistance of thin films and beams. The idea is to use nanofabrication techniques to repeat elementary test structures on a Si wafer involving the layer of interest.
2. The nanomechanical lab-on-chip has been extended to allow direct in situ TEM analysis.
3. The nanomechanical lab-on-chip has been extended to allow coupled electromechanical measurement in order to address phenomena like piezoresistivity.
4. High resolution *in-situ* monitoring of the internal stress evolution during thin film growth has been set up both in solution for electrochemical process (e.g. anodizing) or in vacuum for physical deposition processes (sputtering). The evolution of internal stress is interesting *per se*, but also to unravel various chemical and physical phenomena.
5. Nanoindentation and nanoscratch expertise is available to supplement the information gained with the other techniques in order to characterize thin films and for measuring properties of submicron-sized constituents in heterogenous materials.
6. Adhesion testing of thin films is performed by different extensions of the so-called superlayer test, using again the lab-on-chip technique, and by the wedge opening method.
7. A significant effort is placed on coupling all these analyses with characterization methods in order to link the measured properties to the nano/micro-structure and mechanisms of deformation and fracture. SEM, TEM, EBSD, x-Ray diffraction and chemical analysis are used for that purpose, as well as electrical measurements.

In addition to the effort in setting up these test methods, data reduction schemes are built in order to extract the relevant information. Among others, multiscale models involving size dependent mechanical laws constitute an important part of the research effort.

Representative References

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Patents

- Internal stress actuated micro- and nanomachines for testing physical properties of micro and nano-sized material samples, Pub. No.: WO/2007/093018, International Application no.: PCT/BE2007/000017, Publication Date: 23.08.2007, Priority Data: 14.02.2006
- Imposing and determining stress in sub-micron samples, Pub. no.: WO/2008/098993, International Application no.: PCT/EP2008/051810, Publication Date: 21.08.2008, Priority Data: 14.02.2007.

Funding

- Wallonie projects: Winnomat II (Dinosaure), First doctorat (Jadhere), PPP (Griffe)
- ARC-UCL, Communauté française de Belgique
- PAI, Belspo

Partnership

- Université de Lille 1
- Grenoble INP
- Newcastle University
- ArcelorMittal Liège, Belgium
- AGC Belgium

Main Equipment

See in the project description

- WINFAB clean room facilities (800 m²) with all key technologies required for microsystems and thin films;
- Deposition chambers equipped with *in-situ* monitoring of internal stresses;
- Micromechanical tests: Nano-indentation, micro tensile testing stage for *in-situ* testing in a SEM, microwedge tests;
- Characterization techniques: optical interferometry, SEM, TEM, OIM-EBSD

Products and Services

Access to experimental facilities and possibility to perform test campaigns (see contacts below)

KEYWORDS

Thin films
Nanomechanical testing
Internal stress
Strength
Ductility
Cracking
Adhesion
Scratch

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3D molecular characterization at the sub-micrometer scale

SENIOR SCIENTISTS :

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Research Field and Subjects

The development of new surface treatments and modifications in nano-technologies requires a fine control of the surface molecular composition and structure at the sub-micrometer scale. As far as characterization is concerned, the main area of expertise of the group is the development and use of surface analytical methods based on the ion-solid interaction, in combination with other surface techniques such as AES, XPS and the near field microscopies (AFM, STM).

More specifically, for over the past fifteen years, the team has been contributing to the development of secondary ion mass spectrometry (SIMS) for the molecular characterization of surfaces, with emphasis on organic materials such as polymers or proteins. The SIMS technique is inherently a nanoscale chemical characterization technique because of its very limited information depth (~1 nm). Owing to recent developments of the method, including better focusing of ion beams down to 50nm in size, the technique has now reached the level required for nanoscale molecular imaging. With the advent of cluster ions sources such as C_{60}, sub-micrometric 3D molecular imaging has even been made possible, because the reduced sample degradation induced by these primary ion probes allows one to maintain the integrity of the molecular signals upon depth profiling of the surface. The combination of the molecular information of SIMS with the topographical and mechanical information obtained in AFM opens the door to a full characterization of organic and inorganic samples at the sub-micrometer scale.

The application fields of the characterization methods developed in this group include thin organic films and supramolecular assemblies, biomaterials and hybrid materials, biosensors, organic and molecular electronic devices, catalysts, etc. In addition to the characterization of samples and materials obtained through collaborations, the group also uses different approaches to modify the surface of materials at the micro- and the nanoscale, including chemical and physical treatments: plasma treatments, ion beam irradiation, chemical grafting, conducting polymer film growth, electrospinning, electrochemistry, thin (organic/ metallic) layer adsorption, soft lithography, breath figure imprinting.

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Patents

- Biomaterial and method for obtaining it, Belgian Patent n° 09401022, Nov. 14; International Patent Application PCT / BE95/00104, Nov. 14, 1995; United states Patent n° 5,962,136
- Membrane pour chambre d'encapsulation de cellules produisant au moins une substance biologiquement active et organe bio-artificiel comprenant une telle membrane, patent FR2820057.

Funding

- 3D nanochemiscope - FP7 European community; COOPERATION "NMP-2007-SME-1".
- NANOTIC - Wallonie.
- Hybrid metal-organic nanosystems - Concerted Research Action program (ARC n°01/06-269).

Partnership

- ION-TOF gmbh, Munster (Germany).
- CRP-Gabriel Lippmann, Belvaux (Luxembourg).
- Penn State University (USA).

Main Equipment

Equipments for surface characterization:

- Secondary Ion Mass Spectrometry:

3(*) static imaging time-of-flight mass spectrometers (ToF-SIMS)

- Scanning Auger Microprobe (AES–SAM)
- Access to AFM, STM, XPS-ESCA, SEM, TEM, XRD, Ellipsometry, static and dynamic contact angles, IR, Raman
- Access to clean room facilities.

(*) including a new instrument with automated 3D imaging capabilities.

Products and Services

Service provided to companies for practical surface characterization and imaging

KEYWORDS

3D molecular imaging
Mass spectrometry
Ion spectrometries
Surface characterization
Surface modification

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Scanning Probe Microscopies: from physical to biological characterisation of hybrid and functional nanostructures

SENIOR SCIENTIST :

- Bernard NYSTEN

Research Field and Subjects

It is generally recognised that the development of the Scanning Tunnelling Microscope (STM) and the Atomic Force Microscope (AFM) were, along with de discovery of new nanomaterials like the fullerenes and the carbon nanotubes, the main triggers for the rapid development of nanotechnologies.

Scanning Probe Microscopies (STM and AFM) became essential tools for the characterisation of materials at the nanoscale. This is not only due to their extraordinary high resolution that allows the mapping of surfaces and objects down to the molecular and atomic scale. It is mainly linked to the possibilities they offer to characterise mechanical, physical, chemical and biological properties down to the single molecule level.

The research group has developed a broad expertise in that field. Methodologies have been developed to characterise, i.e. to map and measure, mechanical, physical, chemical and biological properties of surfaces and nanostructures with the atomic force microscope and its derived techniques: phase detection microscopy, force spectroscopy, HarmoniX® mode, magnetic force microscopy, electrostatic and Kelvin probe force microscopy, piezoresponse force microscopy, current-sensing force microscopy …

These techniques allow
- the quantitative measurement and mapping of the elastic modulus of polymer surfaces, organic and inorganic nanowires,
- the characterisation of the magnetic properties of metallic alloys and metallic nanowires,
- the measurement and the mapping of the surface charges and potential on surfaces and nanostructures such as nano-fibres, organic thin film transitors…,
- the characterisation of organic piezoelectric nanostructures,
- the measurement of molecular interaction forces acting between host/gest couples such as antigens and antibodies,
- …

The use of cantilevers functionnalised with reactive polymer brushes also allows the development of chemical nano-sensors and of chemical nano-lithography procedures.

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Funding

- FRS-FNRS
- UIAP: IAP-PAI 6/27 FS2 Functional Supramolecular Systems
- Actions de Recherche concertées (ARC): DyNanoMove
- Wallonie: “Etiquel” and “Neocerat” projects
- Fondation Louvain: Partenariat Solvay

Partnership

- ULB
- Umons
- ULg
- Université de Nancy, France
- Federal University of Rio de Janeiro, Brasil

Main Equipment

Five multimode scanning probe microcopes working in air, liquid, controlled environnement, or vacuum, at ambiant temperature or between -15°C up to 250°C, comprising the following options: STM, C-AFM, LFM, FMM, Tapping™ mode, Harmonix™ mode, MFM, EFM, KPFM, PFM.

Products and Services

Characterisation of the morphological, mechanical, physical and chemical properties of surfaces and nanomaterials with scanning probe microscopies.

KEYWORDS

Scanning Tunneling Microscopy
Atomic force microscopy
Nanomechanics
Nanophysics
Nanochemistry
Surfaces
Functional hybrid nanostructures

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Vibrational, electronic, and nonlinear optical properties of interfacial nanosystems

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- Francesca CECCHET
- André PEREMANS

Research Field and Subjects

A primary goal of nanotechnology is the conception of organized nanostructured interfaces presenting tailored and tunable functions. The efficiency and the operation of many devices indeed rely on the physico-chemical properties of such nano-interfaces.

The research activity of the Lasers and Spectroscopy Laboratory (LLS) in the field of nanotechnology deals with the characterization of the fundamental vibrational, electronic, optical and nonlinear optical properties of interfacial nanosystems. The objective is to understand their unique physico-chemical characteristics, and to contribute to the elaboration of organized functional nanostructures. Topics include:

- carbon-based nanomaterials adsorbed on surfaces, such as fullerenes, graphene and carbon nanotubes;
- metallic nanoparticles and nanostructures to tailor materials optical properties;
- structure and molecular orientation of self-assembled (SAMs) and Langmuir-Blodgett monolayers, nanoparticle organization at surfaces, surface patterning by fast micro-contact printing, molecular recognition at bio-inspired surfaces;
- dynamics of molecular processes at surfaces.

To study nanoscale and nanostructured interfacial systems, the LLS uses its long experience of optical spectroscopy/microscopy, vibrational spectroscopy using photons or electrons, and of local probe microscopy. In particular, the team has developed a strong expertise in using surface nonlinear optical spectroscopy based on lasers tunable in the visible and the infrared frequency range (sum-frequency generation, second harmonic generation, coherent anti-Stokes Raman scattering).

These techniques provide precise vibrational, electronic, vibronic, and morphological information on nanostructured interfaces probed *in situ* (in solid, liquid and gas/vacuum phases). They give access to the chemical composition, to the structural characteristics (e.g. molecular orientation), to the physico-chemical interactions (e.g. adsorption, chemical bonding, intermolecular interactions, biological recognition), and to the surface organization (e.g. surface roughness, domains, patterning) of the investigated system. The use of tunable lasers allows us to selectively excite chosen vibrational or electronic properties of materials. This versatility can also be exploited to enhance the sensitivity of the spectroscopic response.

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Patents

United States Patent 6928090, issued on August 9, 2005, Device and process for mode-locking a laser.

Awards

Economics price of the Namur Province, 1999.

Funding

- F.R.S-FNRS
- Wallonie
- Wallonie-Bruxelles International
- EU-COST Action

Partnership

- ULg (Belgium)
- University of Sherbrooke (Canada)
- University of Bologna (Italy)
- University of Groningen (The Netherlands)
- CLIO Free Electron Laser Facility (France)

Main Equipment

- Sum-frequency generation (SFG) spectrometer and microscope.
- High-resolution electron energy loss spectrometer (HREELS).
- Atomic force microscope (AFM) and scanning tunnelling microscope (STM).

Products and Services

- Surface and interface analysis by optical (SFG, IR) and electronic (HREELS) spectroscopies
- Surface and interface analysis by optical (SFG) and local probe (AFM-STM) microscopies
- Picosecond tunable infrared (2.5–10 μm) and visible (410–710 nm) lasers.

KEYWORDS

Nonlinear optics
Sum-frequency generation
Vibrational spectroscopy
Microscopy
Surfaces and interfaces
(Bio)-molecular self-assembly
Metallic nanoparticles
Carbon-based nanomaterials

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Key Words Index

The Académie universitaire Louvain (AL) is a university consortium composed of the following four academic institutions: FUCaM, FUNDP, FUSL and UCL.
The Research Administration departments of the Académie universitaire Louvain aim at promoting and facilitating academic collaboration with outside partners (companies, research centres and other bodies).

The Research Administrations mission is multiple:

- to inform the researchers (dissemination of the information on available funding sources) and to advise the researchers (legal and financial expertise);
- to assist the researchers at several stages of their research project (identification of the available funding sources, application for the funding best matching their needs, contract negotiation, appropriate management of the obtained funds during the project lifetime);
- to ensure the dissemination and monitoring of research contracts inside the academy;
- to initiate and stimulate the relations between research laboratories and enterprises;
- to facilitate technology transfer and contribute to bringing academic skills and competencies at the forefront of industrial development;
- to contribute to the regional economic development through cooperation with local firms and research centres;
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