Tuesday March 18th

3:15 P.M. - 6:45 P.M.

ROOM AB

Program of the session :

Chairs: P1 Céline ELIE-CAILLE
P2 Séverine GOMEZ et Guillaume COLAS

HOUR	NAME	TITLE
15:15	Brice GAUTIER INL - INSA	Présentation du GDR Carmanano
15:30	**Myriam TAVERNA** **Inst. Galien Paris - Univ. Paris Saclay**	**Advancing Extracellular Vesicle Characterization with Capillary Electrophoresis**
16:00	James BEHAN ISCR - CNRS	Characterisation of Biogenic Nanomaterials Produced by Electroactive Bacteria using Differential Centrifugal Sedimentation
16:15	Lisa ROYER InProcess-LSP	Non-invasive and sterile nanoparticle size measurement in a broad range of containers using spatially resolved dynamic light scattering
17:00	Matias FELDMAN INSP - Sorbonne. Univ	Nanoscale control of heat flux in self-assembled ordered nanocrystal solids
17:15	François HENN L2C - Université de Montpellier	Engineering Individual SWCNT Nanofluidic Device for Enhanced Signal-to-Noise Ratio
17:30	Florant EXERTIER GPM - CNRS	Atomic scale microscopy of different materials by ultrashort THz-driven Atom Probe Tomography
17:45	Francois TREUSSART LuMIn - ENS Paris-Saclay	Polarization texture and sensing application of ferroelectric nanocrystals
18:00	Max GERIN ESRF	High pressure study of exotic hexagonal phase of Ge grown by molecular beam epitaxy on self-assisted GaAs nanowires

IDENTITY

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Short Biography

Myriam Taverna was appointed full professor in analytical chemistry and pharmaceutical biotechnology in 2005 and senior member at the Institut Universitaire de France (IUF) in 2017. She is currently the director of the Institut Galien Paris-Saclay (IGPS-CNRS) at the university of Paris-Saclay, a CNRS-research unit composed of 6 teams. She is the head of one team strongly dedicated to analytical developments with a focus on miniaturized techniques (including microsystems, biosensors and capillary electrophoresis) for the analysis of peptides, (glyco)proteins being biopharmaceuticals, drug targets or disease biomarkers. She is particularly recognized for her expertise in capillary 190 international scientific papers, a dozen of international chapters. She is a member of the editorial board of Analytica Chimica Acta. Her research work in the field of biomarkers of Alzheimer's disease, led her to co-found in 2014 the start-up Alzohis. She started exploring extracellular vesicles and in particular new miniaturized techniques to characterize them in depth in 2018. Recently her research focuses on the exploration of Extracellular vesicles for diagnostic and therapeutic applications.

Title of Oral Presentation

Advancing Extracellular Vesicle Characterization with Capillary Electrophoresis

Keywords

Characterization, capillary electrophoresis, isoelectric focusing, Taylor dispersion analysis, extracellular vesicles

Abstract of Oral Presentation

Extracellular vesicles (EVs) have recently emerged as a source of prognostic or diagnostic molecular biomarkers. Their isolation and enrichment from biological fluids remains however a challenging prerequisite prior to their exploration. To provide sufficient physical and biological information on the isolated EVs, many complementary techniques have to be carried out (e.g. microscopy-based methods, DLS, NTA, TRPS, or flow cytometry…) (1). Although some of them offer indisputable advantages, there is a great need for new techniques allowing accurate detection and characterization of EVs, in suspension, over their entire size range, with minimal calibration requirements and the ability to distinguish EV subtypes or to discriminate them from non-EV contaminants.

Over the last few years, our team has developed innovative analytical techniques for the detection and characterisation of EVs (2), most of which exploit capillary electrophoresis (CE) by using its various separation modes or the technique's ability to perform electrokinetic preconcentrations to obtain either better resolution between subpopulations or improved sensitivity for detecting EVs.

In this talk the different investigated CE-separation modes (capillary zone electrophoresis, isoelectric focusing) for characterizing EVs isolated from bovine milk or animal plasma will be presented as well as their advantages, limits and the kind of information they provide (3,4)
We have also explored Taylor dispersion analysis (TDA) conducted using CE as an alternative technique to assess the size and purity of EVs. We provided recently the first proof of concept of TDA applicability to this kind of bio-nanoparticles (5). The coupling of two separation modes into a 2D configuration (unpublished data) or of an electrokinetic preconcentration (isotachophoresis) (6) allowed us also recently to push forward the sensitivity limits of the technique for EVs.

Acknowledgement

This work has been supported as part of France 2030 program “ANR-11-IDEX-0003” (PSiNano Interdisciplinary program, University of Paris-Saclay) and has received financial support from the CNRS through the MITI inter disciplinary program 2021–2023 “Evenements Rares”. We would also thank Bio-Techne for their partnership in this project.

References

1-Alexandre L., Sun J. Taverna M, Zhong W., Anal Bioanal Chem; Advances in extracellular vesicles analysis (2023) 415(7):1235.
2-M. Morani, T.D. Mai, et al. Recent electrokinetic strategies for isolation, enrichment and separation of extracellular vesicles, Trends Anal. Chem. 135 (2021) 116179.
3- M. Morani, T.D. Mai, Z. Krupova, et al. Electrokinetic characterization of extracellular vesicles with capillary electrophoresis: A new tool for their identification and quantification, Anal Chim Acta 1128 (2020) 42.
4- D. Zohouri et al, Talanta (submitted) (2025)
5- Obeid S, Chamieh J, Mai TD, Morani M, et al. Fast, simple and calibration-free size characterization and quality control of extracellular vesicles using capillary Taylor dispersion analysis, J Chromatogr A. (2023), 705:46418
6-D. Zohouri, M. Taverna, et al. Investigation of on-line electrokinetic enrichment strategies for capillary electrophoresis of extracellular vesicles, J. Chromatogr. A 1730 (2024) 465116

Abstract

Thematic Session: Nanoscale Characterisation
Disciplinary fields involved (eg. Chemistry, Physics, Biology …)**: Chemistry, Biology**
Keywords (max. 4-5)**: nanoparticles, bio-nano interactions, electroactive bacteria, biogenic synthesis**

Characterisation of Biogenic Nanomaterials Produced by Electroactive Bacteria using Differential Centrifugal Sedimentation

James A. Behan[1], Sarra Knani[1], Riad Boughezal[1], Frédéric Barrière[1]

1 CNRS UMR 6226 Institut des Sciences Chimiques de Rennes, Université de Rennes, Rennes, France

Abstract (**no longer than 250 words** or 18 lines max. incl. figure), Calibri 11, single line spacing, black)

Electroactive bacteria including those of the genus *Geobacter* are known for their capacity to transfer electrons extracellularly to solid materials including metals and metal oxides as well as to metal ions in their environment. This capacity can be exploited for the biogenic synthesis of nanomaterials including metal oxide nanoparticles, carbon-based nanomaterials and hybrid or composite bio-nanomaterials. Biogenic synthesis of this nature is a complex phenomenon involving the direct coupling of biochemical processes at the surface of living bacteria to their environment, which poses challenges for the accurate measurement of nanoparticle sizes using standard techniques such as electron microscopy or dynamic light scattering.

In this work we present the characterization of biological nanoparticles composed of magnetite (Fe_3O_4) and amorphous carbon produced *in situ* from electroactive bacteria using the differential centrifugal sedimentation technique to follow nanomaterial synthesis and membrane-level bio-nano interactions with nanometer level resolution.

References (max. 5):

(1) Byrne, J. M.; Muhamadali, H.; Coker, V. S.; Cooper, J.; Lloyd, J. R. Scale-up of the Production of Highly Reactive Biogenic Magnetite Nanoparticles Using Geobacter Sulfurreducens. *Journal of The Royal Society Interface* **2015**, *12* (107), 20150240. https://doi.org/10.1098/rsif.2015.0240.

(2)Srivastava, S. K.; Constanti, M. Room Temperature Biogenic Synthesis of Multiple Nanoparticles (Ag, Pd, Fe, Rh, Ni, Ru, Pt, Co, and Li) by Pseudomonas Aeruginosa SM1. *Journal of Nanoparticle Research* **2012**, *14* (4), 831. https://doi.org/10.1007/s11051-012-0831-7.

(3) Behan, J. A.; Xie, Z.; Wang, Y.-F.; Yang, X.; Aastrup, T.; Yan, Y.; Adumeau, L.; Dawson, K. A. Quartz Crystal Microbalance Method to Measure Nanoparticle–Receptor Interactions and Evaluate Nanoparticle Design Efficiency. *JACS Au* **2023**, *3* (6), 1623–1633. https://doi.org/10.1021/jacsau.3c00084.

Abstract

(4)Law, N.; Ansari, S.; Livens, F. R.; Renshaw, J. C.; Lloyd, J. R. Formation of Nanoscale Elemental Silver Particles via Enzymatic Reduction by *Geobacter Sulfurreducens*. *Appl Environ Microbiol* **2008**, *74* (22), 7090–7093. https://doi.org/10.1128/AEM.01069-08.

(5) Fleming, A.; Cursi, L.; Behan, J. A.; Yan, Y.; Xie, Z.; Adumeau, L.; Dawson, K. A. Designing Functional Bionanoconstructs for Effective *In Vivo* Targeting. *Bioconjugate Chem.* **2022**, *33* (3), 429–443. https://doi.org/10.1021/acs.bioconjchem.1c00546.

Acknowledgement: *This project has received financial support from the CNRS through the MITI interdisciplinary programs*

Abstract

Thematic Session: Nanoscale characterization
Disciplinary fields involved: Physics, Biology, Chemistry
Keywords (max. 4-5)**: Dynamic Light Scattering; turbid suspensions; non-invasive; sterile; real time**

Non-invasive and sterile nanoparticle size measurement in a broad range of containers using spatially resolved dynamic light scattering.

Lisa Royer, Albert Grau-Carbonell, Carl Schuurmans, Michiel Hermes, Nick Koumakis , Rut Besseling

InProcess-LSP, Oss, The Netherlands

Nanoparticles are ubiquitous in the pharmaceutical industry. Critical parameters like average size, size distribution, or the presence of unwanted aggregates directly influence the final product's quality, safety profile, and functionality.

Spatially Resolved Dynamic Light Scattering (SR-DLS) can provide real-time measurement of nanoparticle size and distribution in undiluted suspensions both in static and flow conditions [1]. It can also perform non-invasive and sterile measurements across different container types, including IV bags, prefilled syringes [2], and IVLE bottles [3]. Recently, two new analytical modes to enhance SR-DLS were developed: PhaSR-DLS which improves sensitivity and operational range [4] and Large Particle Detection (LPD) which enables visualization of scattering signals from larger particles [5].

In this talk, we study the formation of Bovine Serum Albumin (BSA) and Polyethyleneimine (PEI) transfection complexes, a model system that mimics the formation of non-viral vectors for use in gene therapy. The influence of parameters influencing the growth and kinetics such as BSA:PEI mass ratio, PEI molecular weight is studied in real-time (figure 1A). PhaSR and LPD modes are also used to study BSA particle size and size distribution over time and temperature, displaying important aggregation (figure 1B). Finally, propofol emulsions' mean droplet size and the volume-weighted percentage of large diameter (>5µm) droplets were studied in situ and non-destructively, maintaining sterility [3].

Abstract

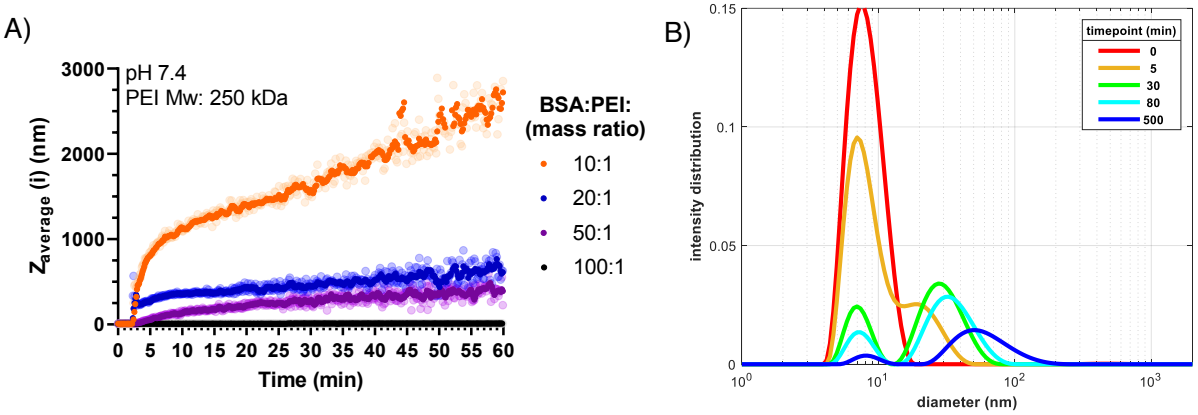

Figure 1: A) Real-time complex hydrodynamic diameter (Z_{av}) using SR-DLS. Different ratios of model protein were mixed with 250 kDa PEI. B) Intensity-based particle size distribution of BSA over 500 minutes at 65°C.

References:

[1] R. Besseling, M. Damen, J. Wijgergangs, M. Hermes, G. Wynia, and A. Gerich, "New unique PAT method and instrument for real-time inline size characterization of concentrated, flowing nanosuspensions," *Eur J Pharm Sci*, vol. 133, pp. 205–213, May 2019, doi: 10.1016/j.ejps.2019.03.024.

[2] "How can you measure particle size CQAs in (sterile) pharmaceutical packaging? – non-invasive measurements using SR-DLS," News-Medical. Accessed: Nov. 25, 2024. [Online]. Available: https://www.news-medical.net/whitepaper/20241106/How-can-you-measure-particle-size-CQAs-in-(sterile)-pharmaceutical-packaging-e28093-non-invasive-measurements-using-SR-DLS.aspx

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Abstract

Thematic Session: Nanoparticles, Nanomaterials for energy, Nanoscale characterization
Disciplinary fields involved: Physics, Chemistry
Keywords: thermal transport, thermoreflectance, nanocrystals, supercrystal, anisotropy

Nanoscale control of heat flux in self-assembled ordered nanocrystal solids

Matias Feldman[1], Charles Vernier[2], Rahul Nag[3], Juan J. Barrios-Capuchino[4], Sébastien Royer[1], Hervé Cruguel[1], Emmanuelle Lacaze[1], Emmanuel Lhuillier[1], Danièle Fournier[1], Florian Schulz[4], Cyrille Hamon[3], Hervé Portalès[2], and James K. Utterback[1]

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2. *MONARIS, Sorbonne Université, CNRS, Paris, France*
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4. *Institute for Nanostructure and Solid-State Physics, University of Hamburg, Hamburg, Germany*

Nanocrystal based solids are a promising class of materials whose emergent properties are highly tunable as a function of constituent shape, size, material composition and surface-capping ligands. They are of particular interest for the development of plasmonic, optoelectronic and thermoelectric devices. Understanding and controlling heat flow in these materials is fundamental to all such applications as heating due to optical excitation or current leads to performance degradation, instability and unwanted chemical activity. I will present recent results on the thermal properties of supercrystals of gold nanospheres, nanorods and nano-bipyramids. Thanks to correlative SEM and spatio-temporally resolved thermoreflectance we were able to access sub-micron structural and nanosecond dynamical thermal information. In superlattices of gold nanospheres capped with polymeric ligands, we found that thermal transport is faster in monolayers than in multilayers. Quasi-ballistic Monte-Carlo simulations suggest that this effect arises as a consequence of the combination of a long phonon mean free path with ligand interdigitation. In supercrystals of gold nanorods and nano-bipyramids, we demonstrated that heat flow predominantly follows the orientation of the elongated nanoparticles and does so even in curved assemblies. In ordered superlattices, heat transport is anisotropic flowing faster along the particles' long axis. Our measurements together with finite element simulations and effective medium modelling show that this anisotropy can be finely tuned through the nanoparticles' aspect ratio, shape and packing. Leveraging this anisotropy opens the way to enhanced thermal dissipation and thermal routing directly using the device's active material while maintaining control over size-dependent optical and electronic effects.

References:

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Abstract

Thematic Session: Functional thin films, Nanostructures & 2D materials
Disciplinary fields involved: Physics
Keywords (max. 4-5)**:** Single-Wall Carbon Nanotubes,

Engineering Individual SWCNT Nanofluidic Device for Enhanced Signal-to-Noise Ratio

L. Bsawmaii[1], C. Delacou[1], V. Kotok[1,2], S. Méance[1], K. Saada[1], A. Kribeche[1], S. Tahir[1], C. Roblin[1], A. Louiset[3], H. Okuno[3], M. Manghi[4], J. Palmeri[1], F. Henn[1], A. Noury[1] & V. Jourdain[1]

1. Laboratoire Charles Coulomb (L2C, UMR CNRS 5221), Université de Montpellier, Montpellier, France;
2. Processes, apparatus and general chemical technology department, Ukrainian State University of Chemical Technology, Dnipro, Ukraine;
3. IRIG-MEM, Univ. Grenoble Alpes, CEA, Grenoble, France
4. Laboratoire de Physique Théorique (LPT UMR CNRS 5152), Université Toulouse III – Paul Sabatier, Toulouse, France

Despite extensive investigation spanning 15 years, the fabrication and study of nanofluidic devices incorporating a single carbon nanotube (CNT) remains a significant experimental challenge [1]. In this study, we present the fabrication of nanofluidic devices that integrate an individual single-walled CNT (SWCNT), demonstrating a remarkable reduction in noise by 1-3 orders of magnitude compared to conventional devices [2]. This advancement was achieved by utilizing high-dielectric-constant materials for both the substrate and the CNT-covering layer. Furthermore, we provide a detailed analysis of the critical factors influencing the successful fabrication of SWCNT-based nanofluidic devices that are reliably leak-free, plug-free, and long-lasting. Key considerations include the quality of the substrate-layer interface, the nanotube opening, and the effective removal of photoresist residues and trapped microbubbles. We demonstrate that these devices, characterized by a high signal-to-noise ratio, enable spectral noise analysis of ionic transport through an individual SWCNT, revealing that SWCNTs adhere to Hooge's law in 1/f at low frequencies [3]. Beyond advancing our fundamental understanding of ion transport in SWCNTs [4,5], these ultralow-noise measurements pave the way for harnessing SWCNTs in nanopore sensing applications for single-molecule detection, offering high sensitivity and identification capabilities.

References:

[1] P. Robin & L. Bocquet, J. Chem. Phys. 2023,158, 160901
[2] L. Bsawmaii et al. Nanoscale, 2024, 16, 21970
[3] P. Robin et al. Faraday Discussion 2023, 246, 556
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Acknowledgement: V. Kotok is grateful to CNRS and to the French PAUSE, ANR-Ukraine funding for refugees.

Abstract

Thematic Session : Nanoscale characterization**,** Nanophotonics & nano-optics

Disciplinary fields involved: Physics, Biology

Keywords (max. 4-5)**:** microscopy, nanoneedles, THz radiation

Atomic scale microscopy of different materials by ultrashort THz-driven Atom Probe Tomography

Florant EXERTIER, Matteo DE TULLIO[1*], Ivan BLUM,[1] Emmanuel CADEL,[1] Laurence CHEVALIER, Martin ANDERSSON,[3] Gustav ERIKSSON,[3] Jonathan HOUARD,[1] Marc ROPITAUX,[2] Angela VELLA[1]

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Terahertz (THz) radiations with low energetic photons (meV) are used today in a wide range of applications such as imaging, sensing, or spectroscopy. This low photon energy has the advantage to induce low damages on fragile materials, or avoid strong heating encountered when a visible wavelength is used [i]. Our research builds on integrating THz radiation with atom probe tomography (APT) to investigate its effect on ion-field evaporation across a range of materials, including metals [ii], semiconductors (crystalline silicon), and insulators (amorphous silica). Atom Probe Tomography (APT) is an imaging technique based on controlled field evaporation of atoms from a nanometric needle-shaped sample under a strong electric field. It provides three-dimensional compositional mapping with sub-nanometric resolution, adding high chemical sensitivity throughout the whole periodic table of elements [iii]. THz radiation has been proven to be beneficial for the reduction of thermal effects in field evaporation in the case of metals [2]. Further tests have also been performed on pure silicon nanotips, where the thermal effect are visible and strongly dependent on the spectrum of the THz radiation. Finally, we analyzed nanotips made of sol-gel amorphous silica [iv], which is chosen as matrix for bio-molecules embedding for future studies.

We have obtained encouraging results also for non-metallic samples, but further investigation will be necessary to find out the optimal parameters (THz amplitude, DC field) to improve the accuracy of compositional analyses, thus to increase the signal to noise ratio and the mass resolution power of the mass spectra.

References (max. 5):

[i] J. Houard et al., *APL*, Volume **15**, (2020).
[ii] A. Vella et al., *Sci. Adv.,* Volume **7**, (2021).
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[iv] G. Sundell, et al., *Small*, Volume **15**, (2019).

Abstract

Thematic Session : Nanoscale characterization / Nanophotonics & Nanooptics
Disciplinary fields involved (eg. Chemistry, Physics, Biology …)**: Physics, Chemistry**
Keywords (max. 4-5)**: ferroelectric nanocrystals, polarization texture, rare-earth ions, up-conversion, sensor**

Polarization texture and sensing application of ferroelectric nanocrystals

A. K. Muraleedharan[1], K. Co[2], J. Zou[2], M. Vallet[2], A. Zaki[2], C. Bogicevic[2], F Karolak[2], C Paillard[2,3], K. Perronet[1], C. Fiorini-Debuisschert[4], F. Treussart[1]

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Ferroelectric materials offer unique polarization textures at the nanoscale, with potential applications in improving energy efficiency and enabling advanced sensing technologies. In this work, we explore the polarization properties and electric field sensing capabilities of barium titanate ($BaTiO_3$) nanocrystals (NCs), emphasizing their intrinsic ferroelectric properties and functional potential.

First, we investigated the polarization textures of $BaTiO_3$ NCs using piezoresponse force microscopy (PFM). Experimental results, complemented by theoretical simulations, revealed a core structure composed of 180° up-and-down domains, surrounded by surface layers exhibiting 90° domain rotations [1]. These findings establish PFM as a valuable tool to assess the potential of ferroelectric nanostructures in advanced sensors.

Building on this, we developed a nanosensor for detecting electric fields with submillisecond response times. Using rare-earth-doped $BaTiO_3$ NCs capable of upconversion (UC) luminescence, we demonstrated optical detection of rapid changes in electric potential. Surface charge variations induced by external fields alter the polarization via the converse piezoelectric effect, modulating the UC spectrum. These NCs exhibit a response time of 100 µs and a sensitivity of 4.8 kV/cm/√Hz, enabling detection of fields comparable to the one of neuronal action potentials [2].

Together, these studies highlight the dual promise of $BaTiO_3$ NCs: understanding fundamental polarization phenomena and advancing nanoscale electric field sensing for applications in neuroscience and beyond.

[1] Muraleedharan, A. K., Co, K., Vallet, M., Zaki, A., Karolak, F., Bogicevic, C., Perronet, K., Dkhil, B., Paillard, C., Fiorini-Debuisschert, C. & Treussart, F. Ferroelectric Texture of Individual Barium Titanate Nanocrystals. *ACS Nano* **18,** 18355–18367 (2024).

Abstract

[2] Muraleedharan, A. K., Zou, J., Vallet, M., Zaki, A., Bogicevic, C., Paillard, C., Perronet, K. & Treussart, F. Submillisecond Electric Field Sensing with an Individual Rare-Earth Doped Ferroelectric Nanocrystal. *ACS Appl. Mater. Interfaces* **16,** 60800–60810 (2024).

Acknowledgement:

This work was financially supported by CNRS MITI interdisciplinary program, the French National Research Agency (ANR, grant numbers ANR-21-CE09-0028 and ANR-21-CE09-0033). GENCI–TGCC computing resources, through grant AD010913519.

Abstract

Thematic Session: Nanophotonic
Disciplinary fields involved: Physics
Keywords: large instruments, extreme conditions, nanowires, phase transitions, mechanical properties

High pressure study of exotic hexagonal phase of Ge grown by molecular beam epitaxy on self-assisted GaAs nanowires

M. Gerin[1], V. Pischedda[2], D. Machon[3], Nicolas Chauvin[4], Philippe Regreny[4], José Penuelas[4]

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3. *Laboratoire Nanotechnologies et Nanosystèmes (LN2), CNRS UMI-3463, Université de Sherbrooke*
4. *Institut des Nanotechnologies de Lyon (INL), CNRS 5270, 69622 Villeurbanne Cedex, France*

In silicon photonics, the critical missing device is a monolithic light source (LEDs and laser) compatible with complementary-metal-oxide-semiconductor (CMOS) technologies. This limit is a direct consequence of the indirect bandgap of the cubic diamond structure of silicon.

Other crystallographic forms of type-IV materials have been proved to exhibit a direct bandgap. This was first discovered in Germanium (hex-Ge) and extended to SiGe alloys (hex-SiGe). In addition to solving the bandgap problem, tunability of the emission with composition was made possible in SiGe alloys[1]. Those exotic phases consist in distorted version of the original cubic diamond structure and are known to appear in high-pressure high-temperature experiments under specific conditions[2].

However, the thermodynamic pathways toward these hex-(Si)Ge phases remain complex, preventing the measurement of their most fundamental mechanical properties such as bulk modulus and thermodynamical stability, needed for both simulation and experimental progress.

In this talk, I will address this issue by reporting the high-pressure behavior of hex-Ge phase grown on GaAs nanowires (GaAs@hex-Ge NW) by molecular beam epitaxy (MBE)[3]. Using X-ray synchrotron *in situ* diffraction at the nano-focus ID27 beamline (ESRF), investigation of hydrostatic and non-hydrostatic effects on hex-Ge in two independent experiments was performed up to 20 GPa. Promising results were obtained not only in understandin the dependence of hexagonal structure parameters on pressure but also in uncovering the associated transition pathway. Notably, a reversible transition from the hexagonal to a metallic phase was observed for the first time (fig1. c).

Abstract

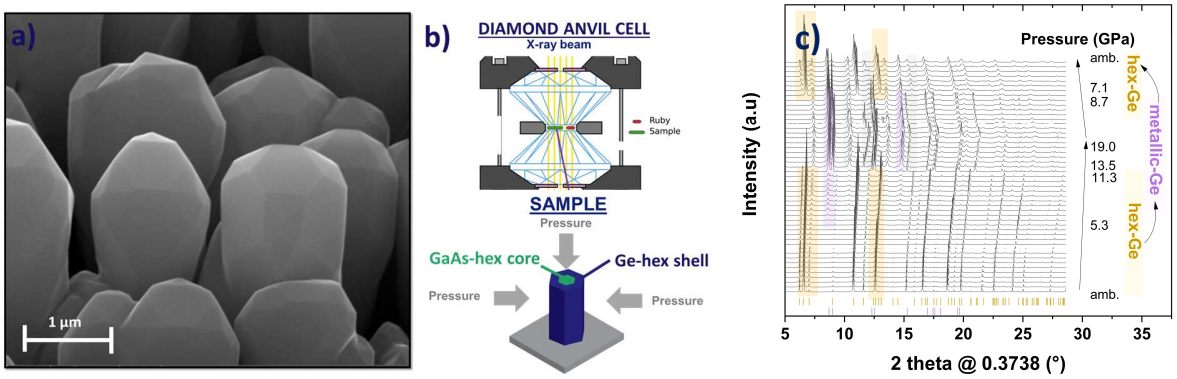

Figure. a) SEM image of GaAs@hex-Ge NW b) schematics of the diamond anvil cell and nanowire c) X-ray diffraction patterns observed during pressure cycling up to 20 GPa.

(1) Fadaly *et al.*, Direct-Bandgap Emission from Hexagonal Ge and SiGe Alloys. Nature 2020, 580 (7802), 205–209. https://doi.org/10.1038/s41586-020-2150-y.
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(3) Dudko *et al.* Hexagonal Ge Grown by Molecular Beam Epitaxy on Self-Assisted GaAs Nanowires. Crystal Growth & Design 2022, 22 (1), 32–36. https://doi.org/10.1021/acs.cgd.1c00945.

Acknowledgement:

The authors thank the Nanolyon platform for access to the equipment and J. B. Goure for technical assistance. This work was supported by the LABEX iMUST of the University of Lyon (ANR-10-LABX-0064), created within the « Plan France 2030 » set up by the French government and managed by the French National Research Agency (ANR). We acknowledge the European Synchrotron Radiation Facility (ESRF) for provision of synchrotron radiation facilities and Mohamed Mezouar for the allocation of beamtime for in-house research.

Thursday March 20th

10:30 A.M. - 12:30 A.M.

ROOM CD

Program of the session :

Chairs: Brice GAUTIER et Rosine COQ GERMANICUS

HOUR	NAME	TITLE
10:30	**François PIQUEMAL**	**ELENA project – electrical nanoscale metrology in industry: Review of the main results**
11:00	Jose MORAN LNE	Calibrated measurements of dopant concentration on vertical nanowires by scanning microwave microscopy
11:15	José ALVAREZ GeePS - CNRS	Understanding and Optimizing Local Electrical Measurements on Cross-Sectional devices Using Conductive Atomic Force Microscopy (C-AFM)
11:30	Hugues GIRARD NIMBE - CEA	In situ photoemission spectroscopies to reveal surface transfer doping on hydrogenated milled nanodiamonds
11:45	Emma AOUSTIN Lab. Albert Fert - CNRS	Towards switchable magnetic tunnel junctions based on polyoxometalates monolayer.
12:00	Anthony SZYMCZYK ISCR - Rennes	Electrokinetic Leakage: Danger and Opportunity for Advanced Materials Characterization
12:15	Bertrand BOUDART GREYC - Univ. Caen Normandie	Time-resolved self-heating temperature measurements of GaN-based HEMTs using nanoparticles as Raman thermometers

IDENTITY

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Short Biography

Francois Piquemal received the PhD degree in condensed matter physics from the Université de Jussieu, Paris (1988) and the habilitation degree in Sciences for Engineer from the Ecole Normale Supérieure ENS-Cachan (2013). He has more than 30 years of experience in fundamental and quantum electrical metrology. His current research interests include the electrical metrology at the nanoscale based on scanning probe microscopy, in particular Scanning Microwave Microscopy (SMM) and Conductive probe Atomic Force Microscopy (C-AFM).

Title of Oral Presentation

ELENA project – electrical nanoscale metrology in industry: Review of the main results

Keywords

Calibration methods, conductive probe AFM, metrology, reference standards, scanning microwave microscopy

Abstract of Oral Presentation

The measurement of electrical properties at the nanoscale allows evaluating the performance of nanomaterials developed for consumer electronics, innovative quantum technologies, and IoT applications. Local DC resistances and high frequency (HF) impedances are among the most prominent properties to measure for nowadays-advanced devices. Currently, Conductive probe Atomic Force Microscopy (C-AFM) and Scanning Microwave Microscopy (SMM) are two main techniques used for the characterization of these properties. Although powerful, these two techniques suffer from major drawbacks: costly, complicated implementation, and lack of traceability. Measurements are thus unreliable.
The European project ELENA (1st September 2021 - 31st August 2024) was aimed at pioneering the traceability of such measurements, with stated uncertainties (targeting in the order of 10% or less), increasing the affordability of these methods by developing and testing cost effective instrumentation and reference standards spanning the range from DC to GHz.

Elaboration of robust calibration methods and good practice guides using simplified uncertainty budgets was planned to underpin this effort. This required the quantification of uncertainty contributions due to influencing factors (samples design, tip-sample interactions, measurement's instrument in the laboratory environment) and the development of reliable 3D multi-physics models to evaluate in particular the effect of the water meniscus (at the tip-sample interface) as well as the effects of the tip's real shape and composition on electrical measurements. We will review the key results obtained in this project and give some perspectives.

Acknowledgement

The project (EMPIR 20IND12 ELENA) has received funding from the EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

References

Elena Project website: http://projects.lne.eu/jrp-elena/

Abstract

Thematic Session: nanomaterials
Disciplinary fields involved: Physics
Keywords: Dopant profiling, nanowire, calibration method, scanning microwave microscopy, uncertainty

Calibrated measurements of dopant concentration on vertical nanowires by scanning microwave microscopy

José Morán-Meza[1], José Penuelas[2], Philippe Régreny[2], and François Piquemal[1]

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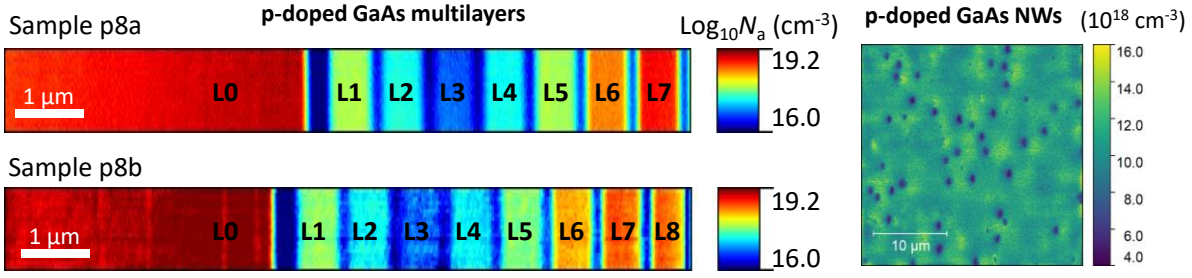
2. *Institut des Nanotechnologies de Lyon (INL), FR-69100 Villeurbanne, France*

Abstract

Arrays of vertically aligned semiconducting GaAs nanowires (NWs) with radial and axial p-n junctions constitute the core element for novel photovoltaic cells for enhanced efficiency [1]. Nevertheless, the accurate characterization of their junctions' dopant concentrations (N_a) is crucial for screening defective NWs and analyze their effects on solar cell performance. To this end, high-resolution measurements are performed using electrical scanning probe microscopy techniques with reference N_a values for traceable measurements on NWs.

Scanning microwave microscopy (SMM) is a powerful technique for measuring impedances at the nanoscale with sub-50-nm spatial resolution. A conductive tip is in contact with the sample surface and connected to a RF source in the GHz range. Measuring the impedance of doped semiconductor with a native oxide layer enables the determination of its dopants concentration levels [2]. To perform quantitative N_a measurements, SMM was calibrated using a reference sample based on p-doped GaAs multilayers with different doping levels N_a ranging from $6{\cdot}10^{16}$/cm^3 to $1{\cdot}10^{19}$/cm^3, measured by secondary-ion mass spectrometry.

The SMM calibration on doped GaAs multilayer samples show a good agreement for N_a values with a combined uncertainty of 10%. We have also found a deviation of 20% for N_a values on another similar GaAs multilayer sample, which corresponds to two standard deviations. The SMM calibration allowed us to extract the doping levels of p-doped GaAs NWs, with preliminary N_a values of $(5.0 \pm 1.2){\cdot}10^{18}$/cm^3 and $(4.6 \pm 1.1){\cdot}10^{18}$/cm^3, which are in the same order of magnitude as the estimated values of about $3.3{\cdot}10^{18}$/cm^3 and $1.8{\cdot}10^{18}$/cm^3, respectively.



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Acknowledgement:

This research project is supported by the European Union and is funded within the scope of the European Metrology Programme for Innovation and Research (EMPIR) project 19ENG05 NanoWires entitled 'High throughput metrology for nanowire energy harvesting devices'.

Abstract

Thematic Session : Nanoscale Characterization

Disciplinary fields involved : Physics

Keywords (max. 4-5)**: C-AFM, cross-sectional analysis, resistivity profile, electrical modelling**

Understanding and Optimizing Local Electrical Measurements on Cross-Sectional devices Using Conductive Atomic Force Microscopy (C-AFM)

Mattia da Lisca[1,2], James P. Connolly[1,2], Pascal Chrétien[1], Jean-Paul Kleider[1,2], Oleksandr Bilousov[2], Amadéo Michaud[2], Maxime Levillayer[2], Stéphane Collin[2,3], Philippe Regreny[4], José Penuelas[4], José Alvarez[1,2]

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The conductive tip atomic force microscopy (C-AFM) technique is widely used for the local electrical characterization of micro- and nanomaterials. Among its experimental approaches, cross-sectional analysis is particularly powerful for examining active device layers and their interfaces.

Our study focuses on this method, emphasizing the experimental conditions, particularly the electrical bias voltage applied between the tip and the sample. This will be demonstrated through the characterization of a cleaved p-AlGaAs:Be / n-GaInP:Si photovoltaic (PV) structure, comprising a stack of eight layers with thicknesses ranging from 20 nm to 1900 nm.

The experimental C-AFM analysis shows that the electrical measurement allows us to spatially resolve the different layers that compose the device. A key observation from this initial analysis is the rectifying behaviour occurring between the AFM tip and the sample, particularly during the transition from a p+ region to an n+ region. To investigate this rectification effect in detail, we conducted an in-depth analysis of III-V multilayer structures with staircase n- or p-type doping, with doping levels calibrated using Electrochemical Capacitance Voltage (ECV).

These analyses will enable us to extract a local resistivity profile of the p-AlGaAs:Be / n-GaInP:Si structure, which will then be compared to a resistivity profile obtained through 2D finite-element electrical modelling, including surface state defects that are rarely investigated. Electrical modelling shows that the experimental methodology is consistent with the expected resistivity profile. It also demonstrates the strong influence of surface states which can modulate the surface resistivity over several orders of magnitude.

Abstract

Thematic Session: Nanochemistry, nanoparticles and assembly
Disciplinary fields involved: Chemistry, Physics
Keywords: Nanodiamonds, Surface transfer doping, Electron spectroscopies

In situ photoemission spectroscopies to reveal surface transfer doping on hydrogenated milled nanodiamonds

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Nanodiamonds (ND) exhibit exceptional chemical, electronic, thermal, and optical properties, making them valuable for applications in nanomedicine, energy, quantum technologies, advanced lubricants, and polymer composites. ND inherits semiconducting properties from bulk diamond. The surface chemistry strongly influences their electronic properties leading to a large energy shift in their band structure (valence and conduction bands) and turning the electronic affinity from positive to negative for oxidized and hydrogenated diamonds, respectively [1]. Surface analysis techniques such as X-ray photoemission spectroscopy (XPS), ultraviolet photoemission spectroscopy (UPS) and reflection electron energy loss spectroscopy (REELS) are critical for understanding the surface properties of nanomaterials, including nanodiamonds [2].

This study investigates hydrogenated milled nanodiamonds (H-MND) by integrating UPS and XPS measurements with REELS. Through *in situ* annealing within an ultra-high vacuum (UHV) chamber, we examine the impact of surface termination on surface conductivity, focusing on the role of adsorbates. Our findings reveal that a surface transfer doping mechanism, akin to that observed in bulk diamond, governs a pseudo p-type conductivity in H-MND. The conductivity dependence on ambient exposure, water, and subsequent annealing demonstrates its reversibility. The study also discusses the nature of electron acceptors and the influence of ND facets on conductivity [3].

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Abstract

Acknowledgement:

This work was supported by a public grant overseen by the French National Research Agency (ANR) as part of the “Investissements d’Avenir” program (Labex NanoSaclay, reference: ANR-10-LABX-0035; Labex Charmmat, reference: ANR-11-LABX-0039-grant).

Abstract

Thematic Session: Nanoelectronics, nanomagnetism & spintronics
Disciplinary fields involved: Physics
Keywords: Magnetic tunnel junction, Interfaces, 2D material

Towards switchable magnetic tunnel junctions based on polyoxometalates monolayer

Emma Aoustin[1], Julie Lion[1], Florian Godel[1], Sophie Colin[1], Aymeric Vecchiola[1], Abdourahim Hammani[2], Florence Volatron[2], Anna Proust[2], Marie-Blandine Martin[1], Frederic Petroff[1], Pierre Seneor[1], Richard Mattana[1]

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2. *Institut Parisien de Chimie Moléculaire, Sorbonne Universités, Paris, France*

Spintronics has revolutionized modern electronics by introducing new possibilities for data storage and magnetic sensing technologies. As its core, are magnetic tunnel junctions (MTJs) consisting of two ferromagnetic electrodes separated by a thin insulating barrier. It has been shown that molecules could be integrated into MTJs giving rise to molecular spintronics that combines the principles of spintronics with the properties of organic molecules. Thanks to the spin-dependent hybridization at ferromagnetic electrode/molecule interfaces, spin polarization and thus tunnel magnetoresistance can be tailored [1]. However, up to now, mainly passive molecules such as alkane chain or aromatic rings have been integrated into MTJs [2-3].

In this presentation, we will present molecular MTJs integrating complex molecules, called “active” molecules, which can be switched by an external stimulus (light, pressure, electric field…). MTJs properties are expected to be tuned by switching the molecule. Here, we focus on polyoxotungstate $(PW_{11}O_{40}(SiC_3H_6SH)_2)$ redox switchable molecules, from the polyoxometalate (POM) family [4]. We will first present the multiple challenges we have faced to fabricate MTJs (Fig.1). Next, we will show the characterizations of the interface which confirm the grafting of molecules onto the electrodes. We will then focus on the investigation of the electrical switching of the molecules by conductive-tip AFM. Finally, we will present the preliminary electrical characterization which demonstrate that switchable polyoxometalate monolayer can be successfully integrated in MTJs. This opens the way to develop multifunctional spintronic devices.

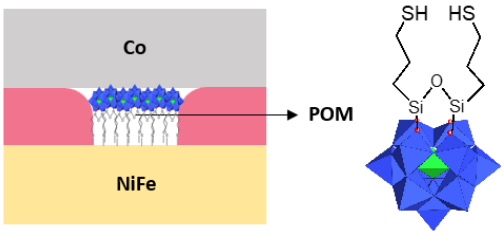

Figure 1: Representation of a MTJ integrating POM molecules

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Abstract

Thematic Session: Caractérisation à l'échelle nano, Couches minces fonctionnelles & Nanostructures (1D, 2D)
Disciplinary fields involved: Chemistry
Keywords (max. 4-5)**: electrokinetics, surface characterization, zeta potential, membranes, polyelectrolyte monolayers**

Electrokinetic Leakage: Danger and Opportunity for Advanced Materials Characterization

Agathe Lizée[1], Patrick Loulergue[1], Anthony Szymczyk[1]*

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Electrokinetic techniques such as streaming current measurements have become increasingly popular in material science and are now part of the toolbox for material surface characterization [1]. They make it possible to determine the zeta potential of a solid in contact with a liquid phase, which is a reliable indicator of the electrostatic interaction between the surface of the solid and the charged species in the surrounding solution. The streaming current technique is based on measuring the ionic current generated by the displacement of excess counterions in the electrical double layer when a hydrostatic pressure gradient is applied along the surface to be characterized. However, when such tangential electrokinetic measurements are applied to porous materials, an additional current, referred to as "electrokinetic leakage", may occur within the porosity of the material, thus adding to the surface ionic current. By considering the example of polymer membranes modified by assemblies of polyelectrolyte monolayers by the well-known layer-by-layer method, we demonstrate that neglecting this parasitic phenomenon is likely to lead to dramatic quantitative errors in the zeta potential determination and even qualitative misinterpretations of the raw experimental signals. An advanced protocol based on measuring the streaming current between two surfaces of the same material, but changing the distance between them, has made it possible (i) to correct the raw data for the electrokinetic leakage phenomenon and thus to correctly and accurately determine the zeta potential of the membrane surface and (ii) to determine whether polyelectrolyte monolayers have been deposited only on the outer surface of the membrane or whether they also penetrated into the membrane pores [2].

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Abstract

Thematic Session: GDR CarMaNano Mesures thermiques
Disciplinary fields involved: electronics
Keywords: temperature measurement, Raman spectroscopy, time-resolved, GaN transistors

Time-resolved self-heating temperature measurements of GaN-based HEMTs using nanoparticles as Raman thermometers

Yannick GUHEL[1], Raphael STRENAER[1], and Bertrand BOUDART[1]

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Abstract

GaN-based HEMTs are attracted increasing interest in recent decades due to their exceptional properties such as wide bandgap, high thermal conductivity and radiation hardness. They are therefore suitable for a wide range of civil and military applications in telecommunications, power electronics, etc. The electrical performance and reliability of these transistors depend on their thermal behavior. Raman spectroscopy is an optical technique used to determine the temperature of electronic materials and components.
This is a well-known technique for studying semiconductors, but not for studying metals, apart from surface oxidation phenomena. Component manufacturers tend to add metal to the surface, for example to create a field plate. This makes conventional Raman spectroscopy measurements more difficult. To avoid this problem, we have developed a new technique using CeO_2 nanoparticles that act as Raman thermometers [1, 2].
But in view of the applications for these components, it was necessary to develop a new Raman system enabling self-heating measurements to be carried out in pulsed mode. By probing the nanoparticles deposited on the surface, we can determine the surface temperature in the pulse regime and thus follow the self-heating of the transistors during a voltage pulse, as well as tracking this temperature during the cooling phase [3].
Thus, the experimental GaN volumetric and surface self-heating temperatures measured for biased GaN-based HEMTs in both DC and pulsed regime are reported with a sub micrometer spatial resolution and a temperature resolution of about 5 °C.

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Abstract

Acknowledgement:

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Poster Session

FUNCTIONAL THIN FILMS, NANOSTRUCTURES & 2D MATERIALS/ NANOSCALE CHARACTERIZATION

N° POSTER	TITLE	NOM	Prénom
64	OVERCOMING SAMPLE PREPARATION CHALLENGES IN NANOPARTICLE CHARACTERIZATION BY SEM	AMBERT	Stéphane
65	THE HREELM Project – The High Resolution Electron Energy Loss Microscope is coming to probe the surface vibrations at the microscopic scale	AMIAUD	Lionel
66	Development of measuring protocols and data processing methods for reference samples designed to calibrate electrical measurements at nanoscale	CHRETIEN/PIQUEMAL/HOUZE/MORAN-MEZA/HAROURI	Pascal/François/Frédéric/josé/Abdelmounaim
67	AI-Machine Learning models for conductive electrical modes on AFM: maps prediction and material clustering	COQ GERMANICUS	Rosine
68	Unravelling complex mixtures at the nanoscale: the power of coupling field flow fractionation and electron microscopy (FFF-EM)	CROUZIER	Loïc
69	Boron Phosphide Nanocrystals from the Viewpoint of Pair Distribution Function Analysis	DOISNEAU	Clara
70	Combined Study of Casimir-Polder Interactions and Patch Potentials on SiNx Nanogratings	FABRE	Nathalie
71	Nano-architecture of mixed organic layers on a silver surface	GUAN	Yimin
72	CARBON NANOTUBE MECHANICAL MASS SENSOR WITH SUB-YOCTOGRAM SENSITIVITY AT ROOM TEMPERATURE	HENN	François
73	Nanoscale characterization of ZnS:Cu Phosphor Powder	HERNANDEZ	Roberto
74	Fluorescence properties of mixed-dimension heterostructures	LE BALLE	Juliette
75	In-rich InGaN/GaN nanowires for red light emitting diodes	TCHOULAYEU POSSIE	Nidel Dilan
76	Design of efficient nanocatalysts for H2 release from boranes and silanes	THIBAULT	Maxime
77	THE HREELM Project – The High Resolution Electron Energy Loss Microscope is coming to probe the surface vibrations at the microscopic scale	AMIAUD	Lionel
78	Enhanced Light Absorption through Nanostructuring of Titanium Nitride (TiN)	BEN MOUSSA	Nizar
79	Study of physical properties of antiphase boundaries in III-V epitaxial layer on silicon with conductive tip atomic force microscopy (C-AFM) and with Kelvin Probe Force Microscopy (KPFM) techniques.	BERNARD	Rozenn
80	Interfacial self-assembly of polydiacetylene and graphene oxide for organic photovoltaics	BISTINTZANOS	Alexia
81	TMD Engineering of 2D-Magnetic Tunnel Junctions – From Barriers to Electrodes	DANIEL	Jane
82	Study and Characterization of TzDA Langmuir Films for Polydiacetylene-Based Sensors	KANDYLI	Maria
83	Charge transfer between plasmonic PdAg nanoparticles and C60 molecules	LI	Xingtong
84	Développement et caractérisation de cristaux magnoniques sur substrats flexibles pour la straintronique	MNASRI	Walid
85	Direct CVD graphene integration for Spintronics	PERRIN	Jérémy
86	Design and Synthesis of Bioactive Materials Using Two-Photon Polymerization and Thiol-Ene Click Chemistry	PINON VASQUEZ	Ana Karen
87	Engineering Spin Wave dispersion and Surface Acoustic Wave-driven FMR in Fe thin films by N-implantation	SHARMA	Anupam

Abstract

Thematic Session (eg. Nanophotonics & nano-optics, nanomaterials, nanobioscience …)**:** **Caracterisation at nano scale**
Disciplinary fields involved (eg. Chemistry, Physics, Biology …)**: Analytical, Physics**
Keywords (max. 4-5)**: Nanoparticles, regulatory, Scanning Electron Microscopy**

OVERCOMING SAMPLE PREPARATION CHALLENGES IN NANOPARTICLE CHARACTERIZATION BY SEM

S. Ambert[1], L. Crouzier[1] T. Lucante[1]

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Abstract (**no longer than 250 words** or 18 lines max. incl. figure), Calibri 11, single line spacing, black)

The analysis of complex nanomaterials (e.g., polydisperse, mixtures of chemicals, prone to agglomeration, etc.) used in cosmetics presents a significant analytical challenge. Ensuring product safety and regulatory compliance requires precise characterization [1], yet conventional methods such as scanning electron microscopy (SEM) are difficult to apply effectively to these complex materials. This research, conducted on specifically prepared mixtures of certified size latex dispersions, investigates the difficulties in achieving a uniform deposition of the particle dispersions on SEM substrates, a prerequisite for accurate analysis. Focusing on the impact of capillary forces, "coffee-ring" effects [2], and particle interactions, the study explores a systematic approach using a spin-coater [3], controlling parameters such as solvent properties, substrate surface charge, and rotation speeds to optimize deposition quality. Results demonstrate improved dispersion homogeneity and particle size accuracy, particularly for single-size particle systems. However, analysis of polydisperse samples revealed discrepancies in size distribution. This study identifies three key areas for future investigation: optimized substrate preparations (chemical and physical treatments), enhanced sample homogenization/stabilization, and advanced deposition methods combining dynamic and static techniques. In the long term, this comprehensive approach aims to develop a robust and universal protocol for reliable and accurate SEM analysis of diverse nanoparticle systems, essential for the industry.

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Abstract

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Acknowledgement:

(*calibri 10)*

Abstract

Thematic Session (Functional thin films, Nanostructures & 2D materials)
Disciplinary fields involved (Physical Chemistry, Instrumentation
Keywords (max. 4-5)**:** New Instrument, Microscopy, Spectroscopy, Surface Science

THE HREELM Project – The High Resolution Electron Energy Loss Microscope is coming to probe the surface vibrations at the microscopic scale

Lionel Amiaud[1], Daniel Comparat[2], Nick Barrett[3], Anne Lafosse[1], Raphaël Hahn[4], Florent Vallée[2]

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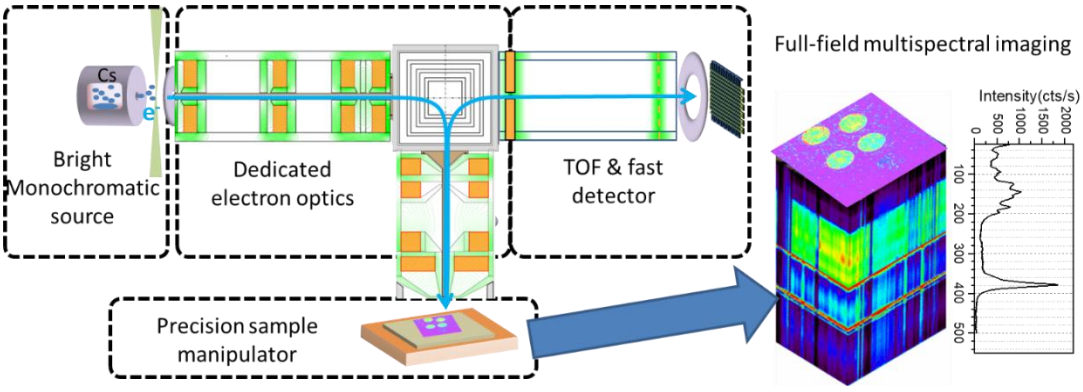
4. *Department of Chemistry and Applied Biosciences, ETH Zurich, CH-8093 Zurich, Switzerlan*

The HREELM will be a fully innovative instrument dedicated to analyzing the vibrational states on surfaces. It combines spectral analyses, spatial resolution and surface sensitivity that are inaccessible simultaneously with any other state-of-the-art technique. The patented design[1] allows for full-field imaging of low energy electron energy losses (in real and in reciprocal space) by combining nanometric spatial (20 nm) and high spectral resolution (10 meV, 80 cm-1). It features an highly monochromatic electron source[2] and a new low energy electron optics, specially designed to preserve spectral resolution[3]. These key elements will be presented, along with the first case studies we project to investigate once the instrument is operational in 2029.



Schematic of the HREELM prototype. HREELM data is in the form of a 3D stack (x,y,energy) in real space or (k_x,k_y, energy) in reciprocal space, allowing pixel by pixel extraction of the vibrational spectra.

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Acknowledgements:

European Research Council (ERC) for the (ERC CITRON 2021), the Region Île-de-France for the SESAME HREELM 2024, the ANR for the TPX4 ANR and the LABEX PsiNano for the TOF4HREELM project.

Abstract

Thematic Session: nanoscale characterization
Disciplinary fields involved: engineering sciences, physics
Keywords: conductive probe atomic force microscopy, reference sample, resistance, current, calibration

Development of measuring protocols and data processing methods for reference samples designed to calibrate electrical measurements at nanoscale

Pascal Chrétien[1,2], François Piquemal[3], Frédéric Houzé[1,2], José Morán-Meza[3], Christian Ulysse[4], Abdelmounaim Harouri[4]

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2. *Laboratoire de génie électrique et électronique de Paris - GeePs, Sorbonne Université, CentraleSupelec, CNRS, Gif-sur-Yvette (France)*
3. *Laboratoire national de métrologie et d'essais - LNE, Trappes (France)*
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Abstract:

Conductive tip atomic force microscopy (C-AFM) has become a widely used technique in many fields for measuring resistance and current at nanoscale. Despite the variety of existing approaches, until very recently the community had no means of calibrating this type of measurement, nor to compare instruments with each other. To fill this gap, we have lately proposed [1,2] reference samples specifically designed to calibrate the entire C-AFM measurement circuit over a wide range of 9 decades of resistance and current, from 1 kΩ to 1 TΩ and from 10 fA to 10 µA, respectively. The aim of our poster is to shed light on the operating conditions and data analysis methods that have been elaborated to make the most of these samples, in both imaging and *I-V* spectroscopy modes. The studies presented here were carried out using a C-AFM setup based on a home-made electrical measurement module called WiCMD (Wide range Current Measuring Device), which provides real-time resistance or current maps, as well as *I-V* curves, over a range of 11 decades. Here, the recommendations we are formulating are intended to be applicable by any C-AFM user: samples and their associated best-practice guide could form a kind of "universal kit", paving the way for reliable and comparable resistance/current nanoscale measurements.

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Acknowledgement:
This research work was carried out in the framework of the ELENA project (EMPIR 20IND12), which is supported by the European Metrology Program for Innovation and Research (EMPIR). Part of this work was done at the C2N micro nanotechnologies platforms and was partly supported by the RENATECH network and the General Council of Essonne.

Abstract

Thematic Session: Characterization at the Nanoscale
Disciplinary fields involved: Physics
Keywords: AFM, Machine Learning, C-AFM, SSRM

AI-Machine Learning models for conductive electrical modes on AFM: maps prediction and material clustering

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Nanoscale characterization is essential for advancing material research and optimizing the performance of functional systems, particularly in microelectronic and energy-related applications. For electrical, electrochemical and mechanical investigations, Atomic Force Microscopy (AFM), combined with the hyperspectral Data Cube [1-2] techniques, provides a powerful route for accessing and analyzing local properties at nanoscale resolutions. This work highlights the development of advanced data analysis tools with supervised AI machine learning methodologies to handle the big data AFM measurements and predict AFM maps. For local electrical properties, a conductive AFM tip enables one to record current-voltage spectroscopy at each pixel of the scanned area, providing insights into the localized electrical behavior under applied voltage. The MultiDAT-AFM [3], a Python-based tool, enables efficient processing of multi-dimensional data with features such as detailed visualizations, animated mappings, and 3D representations of hyperspectral Data Cube modes. Through the evaluation of Machine Learning algorithms, the Random Forest Regressor emerged as the most effective model, achieving high prediction accuracy and rapid execution times. These methodologies were demonstrated on silicon microelectronic devices for RF applications using the Data Cube Scanning Spreading Resistance (SSRM) method, as well as on cathode material for Lithium-ion batteries with the TUNA conductive mode. For the cathode material, which is a nano-size metal-oxide agglomerated structure, unsupervised Machine Learning clustering methods are presented. By combining advanced AFM techniques and predictive machine learning models, this approach paves the way for deeper insights into material performance and the design of optimized systems, with implications spanning microelectronics, energy storage, and transport.

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Acknowledgement:
The authors also would like to thank CARNOT ESP Institute for supporting the SiC Ageing project

Abstract

Thematic Session: **Nanoscale characterization**

Disciplinary fields involved: Chemistry, Physics, Material

Keywords: Boron Phosphide, PDF, defects, local organization, molten salt

Boron Phosphide Nanocrystals from the Viewpoint of Pair Distribution Function Analysis

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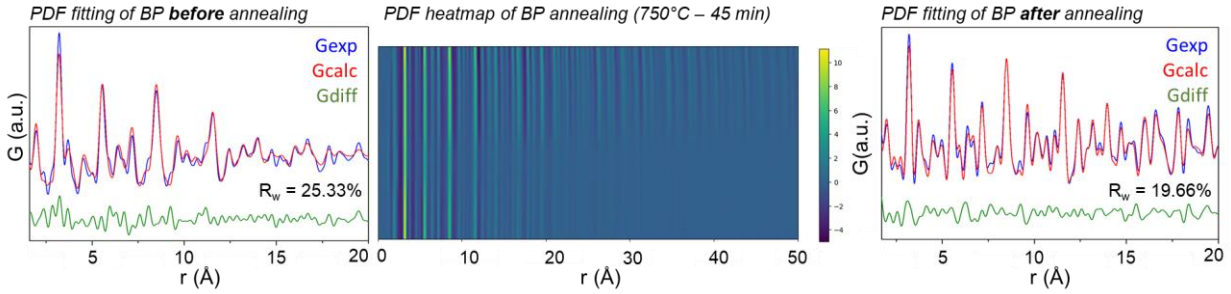
Boron phosphide is known as a super-hard material, potential photolectrocatalyst and p-type transparent conductor.[1,2] We recently developed the first synthesis of *c*-BP nanocrystals with the sphalerite structure and controlled size of ca. 5 nm, using inorganic molten salts as liquid reaction media at 550°C. Elemental composition measurements (X-ray fluorescence, XPS, ICP-OES) and the study of the local structure by ^{31}P and ^{11}B NMR show that these nano-objects exhibit, additional atomic environments for P and B.

We engaged in an in-depth study using total X-ray scattering to probe all distances between atoms and identify the nature of this unreported local environment, irrespective of the crystalline or amorphous nature of the solid.[3,4] Pair Distribution Function (PDF) analysis shows indeed significant deviations to the ideal sphalerite structure and enable identification of specific inter-atomic distances impacted by these deviations. PDF analysis carried out *in situ* during annealing (synchrotron radiation) shows that the structure undergoes reorganisation before grain growth at 750 °C. During annealing, the overall structure evolves towards ideal sphalerite. Few defects reappear during cooling.

We will discuss PDF data treatment and the possible origins of the new atomic environments, especially related to the surface states of the nanocrystals.



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Acknowledgement:

This project has received funding from the European Research Council (ERC) Consolidator Grant GENESIS (grant agreement n°864850). Experiments at the ID11 beamline of ESRF has been funded by ESRF as the User Proposals MA4506, MA4760, and MA5188. C.D. thanks the doctoral school ED397 for funding her Ph.D.

Abstract

Thematic Session : Nano characterization
Disciplinary fields involved : Physics, Chemistry
Keywords : Casimir-Polder, SiNx nanogratings, patch potentials, atomic diffraction

Combined Study of Casimir-Polder Interactions and Patch Potentials on SiNx Nanogratings

Ayoub Hadi [1], Matthieu Bruneau [1,2], Julien Lecoffre [1], Nathalie Fabre [1], Quentin Bouton [1], Gabriel Dutier [1]

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Silicon nitride (SiNx) nanogratings are an essential tool for atomic interferometric experiment aiming at studying short-range interactions, such as Casimir-Polder (C-P) forces. Such forces play a fundamental role for the miniaturization of quantum technologies. However, electrons implanted in the materials, as well as clusters on the surface, cause additional interactions, which are referred as patch potentials. This work investigates the impact of patch potentials and atomic interactions at the nanoscale using a combined experimental and numerical approach.

Monte Carlo simulations (CASINO) were employed to model charge implantation effects caused by electron beam lithography, which significantly influence electrostatic properties and force measurements [2,4]. Concurrently, atomic diffraction experiments using laser-cooled argon atoms enabled precise measurements of the C-P interaction parameter. The analysis revealed a strong correlation between the geometrical properties of nanogratings and CP potentials, limiting the measurement accuracy at 17% level [1]. Additionally, the analysis indicated that patch potentials are negligible at this level of precision, but highlights the challenges associated with systematic characterization of nanostructures [1,3].

This work demonstrates the importance of surface properties and fabrication processes in understanding fundamental interactions between atoms and nanostructures. The methodology paves the way for further exploration of electrostatic interactions and non-Newtonian short-range forces.

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Acknowledgement:

This work was supported by the RENATECH network and the facilities at IEMN.

Abstract

Thematic Session: nanomaterials
Disciplinary fields involved: Physics
Keywords: Perylene, Fullerene, Ag (110), Organic Layers, Surface Science

Nano-architecture of mixed organic layers on a silver surface.

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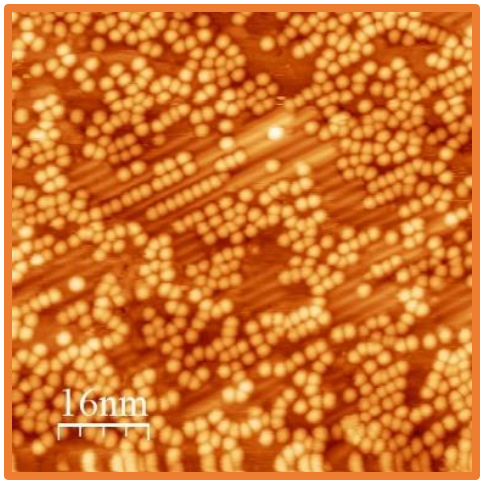
Corresponding author email: laurent.guilemot@universite-paris-saclay.fr

We present a study of the formation of mixed organic molecular layers on a Ag (110) surface.
We focus on layers of an Aromatic Polycyclic Hydrocarbon (APH) molecule, namely perylene, with added fullerenes (C60). We use surface science techniques such as STM/STS, UPS/XPS and others to investigate the structure of these molecular assemblies and to study the link with their electronic, optical and vibrational properties.
We focus on these particular molecules because we demonstrated that the 3D structure of perylene multilayers on Ag (110) shows exciting structural properties [1]. This overlayer has the unique ability to adapt to the morphology of the underlying substrate preserving its lateral order and maintaining an epitaxial relationship with the various surface terraces. Moreover, in the domain of photosynthesis perylene along with fullerene has proved to be efficient in an important process like "Triplet-Triplet Annihilation – Up Conversion" (TTA-UC) [2,3]. TTA-UC finds useful applications in OSC (Organic Solar Cells), OLED (Organic Light Emitting Diodes), and Photolysis cancer therapy.
We present a study on the ability of the perylene multilayer to be used as a template for forming composite layers with fullerenes (C60).



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Abstract

Thematic Session: Nanophotonics & Nanooptics
Disciplinary fields involved: Physics
Keywords: Carbon nanotube, mechanical resonator, mass sensing

Carbon Nanotube Mechanical Mass Sensor with Sub-Yoctogram Sensitivity at Room Temperature

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Carbon nanotube are exceptional mechanical resonators. Indeed, they are extremely light, with masses in the range of ~ag (1 ag = 10^{-18}g). In addition, as bottom-up materials, they do not suffer from surface defect as their top-down counterparts as MEMS. From both features stems their exquisite mass sensitivity as demonstrated by Chaste et al. [1] with a sensitivity of 1.4 yg in a cryogenic environment. This sensitivity was reported to be much worse at room temperature, about 25 zg[2]. This has hindered the use of CNTs as sensors in real-life applications. In this work, we propose a unique set-up to track in real time the vibrations of an individual single-walled CNT at room temperature. It allows us to demonstrate a record mass sensitivity of 0.2 yg +/- 0.17 yg better than the proton mass. We investigated the noise mechanisms that could be limiting the sensitivity: set-up noise, interaction with the gas phase and Brownian motion to name only the most important ones. We found that our device operates closely to the Brownian noise limit and is not limited by external sources of noise. Surprisingly, increasing the pressure up to 35 mbar does not seem to degrade the sensitivity, which might be preserved up to ambient pressure. Finally, yet importantly, the exquisite sensitivity observed here is similar for several devices, making it a reliable and reproducible feature. This opens up the possibility to perform single-molecule sensing, in various contexts such as biological applications, mass spectrometry or surface science [1,3].

Abstract

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Thematic Session: Nanoscale characterization

Keywords (max. 4-5): AC-Electroluminescence, Transmission Electron Microscopy (TEM), Cathodoluminescence.

Nanoscale characterization of ZnS:Cu,Cl Phosphor Powder

Roberto Hernandez[1] [2], Ludovic Largeau[1], Stefano Pirotta[1], Etienne Herth[1], Frédéric Mahut[1], Nathaniel Findling[1], Krishnendu Sarkar[1], Nidel Tchoulayeu[1], Sidahmed Beddar[2], Maria Tchernycheva[1]

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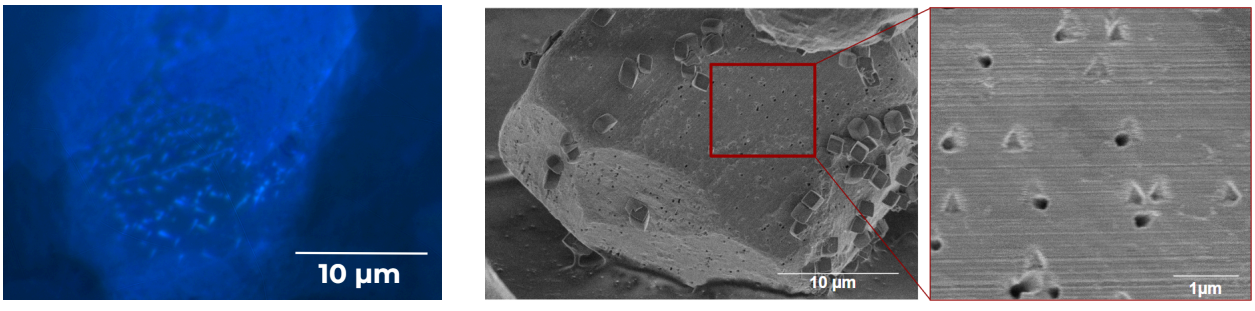
2 Valeo Vision, 34 rue Saint André - 93012 - Bobigny, France

Electroluminescent (EL) powders have been used for more than 50 years, but the mechanism of operation is still debated. When a high-frequency AC-voltage is applied to ZnS:Cu,Cl grains (≈ 15 µm) electroluminescence is observed. It is assumed that EL emission is caused by concentrating the E-field at Cu_xS defects, which inject carriers into the host lattice (ZnS), radiative recombination takes place through donor–acceptor pair transitions [1]. This technology is appealing for its fabrication simplicity and low power consumption, but its short lifespan limits widespread adoption.

Fabrication of ZnS:Cu,Cl involves firing at high temperatures (1,000-1,024◦C) where the hexagonal phase predominates, when cooled there is a cubic phase transition that creates a high density of planar stacking faults [2]. Those are believed to be necessary for EL, as copper preferentially precipitates on defects formed in the hexagonal-to-cubic transformation resulting in nanoscale luminescent lines. The luminescence spectra of a full grain consist of a broad emission from 2.15 to 3.15 eV, spectral shape and intensity distribution are not fixed but vary depending on the excitation method.

In this contribution, we performed nanoscale analysis of phosphor grains by transmission electron microscopy, photoluminescence, cathodoluminescence, and electroluminescence to provide a relationship between stacking faults, luminescence spectra and excitation methods. Hydrofluoric acid etching allowed us to reveal the internal crystal structure, we identified triangular etchpits that allowed us to image the phase transitions and to perform a detailed CL mapping to visualize spectral intensity variations across different sample regions.

a) b)



c)

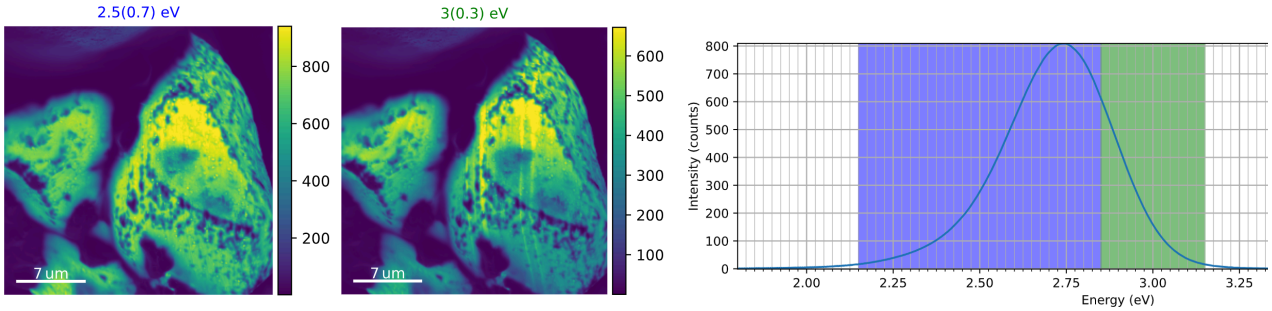


Fig. a) Optical image of ZnS:Cu grain under EL excitation, b) Hydrofluoric acid etch on ZnS:Cu grain with a zoom at triangular etchpits, c) Cathodoluminescence mapping showing different spectral contributions along a grain.

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Abstract

Thematic Session : Nano-optics and nano-materials
Disciplinary fields involved : Physics and chemistry
Keywords : nanomaterials, heterostructures, fluorescence, energy transfers

Fluorescence properties of mixed-dimension heterostructures

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Since the discovery of graphene, the assembling of 2D semiconductors in van der Waals (VdW) heterostructures results in the emergence of fascinating properties[1] with potential applications in photonics and optoelecrtronics. However, the presence of intrinsic structural defects and inhomogeneities, associated to indirect band gaps and low quantum yields in their bulk form, still hamper their study and use in nanophotonics as pure photon emitters. In contrast, 0D materials such as luminescent organic molecules can act as cooperative quantum emitters with strong light/matter interaction and single photon emission. However, these molecules are fragile and difficult to arrange and position at the nanoscale.

In this presentation we show that boron nitride nanotubes (BNNTs) can be used as a template for integrating a 1D chain of luminescent molecules[2-5] onto 2D materials in the van der Waals regime. Different heterostructures are fabricated by modifying either the nature of the 2D material (MoS_2, WSe_2, WS_2). We present various methods for fabricating mixed-dimension heterostructures and preliminary results of their structural characterization and fingerprints of optical interactions between molecules inside BNNTs as well as between molecules and the 2D semiconductor.

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Acknowledgement:
The authors thank the French Aerospace Lab (Onera), French National Research Center (CNRS) and the Naquidis Center for financial support.

Abstract

Thematic Session : Nanophotonics & Nano-optics
Disciplinary fields involved : Physics
Keywords : InGaN/GaN NWs, MBE, light emitting diode, doping

In-rich InGaN/GaN nanowires for red light emitting diodes

Nidel Tchoulayeu, Martina Morassi, Krishnendu Sarkar, Quang-Chieu Bui, Laurent Travers, Julien Chaste, Jean-Christophe Harmand, Maria Tchernycheva, Noëlle Gogneau

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Indium-rich $In_XGa_{1-X}N$ alloys are very interesting for applications such as blue-to-red light emitting diodes. The particular choice of these direct-gap materials comes from their ability to cover the large spectral range from the infrared of InN (0.65eV) to the ultraviolet of GaN (3.4eV) as a function of the Indium content, and therefore cover the visible spectrum with the RGB colours of interest for displays.
Today, InGaN's commercial LEDs are fabricated from 2D epitaxial layers on Sapphire substrates, which work well for the blue spectral range. However, when we tend to increase the indium content above 20%, the formation of dislocations and indium clusters leads to a deterioration in the material quality. To overcome these problems, one approach is to grow LEDs in the form of nanowires, using GaN nanowires as pseudo-substrate for growing InGaN. Due to their small footprint, nanowires are free from dislocations and allow easier stress relaxation through their free lateral surface [1].

In this contribution, we describe the growth of InGaN/GaN nanowires on Si (111) by molecular beam epitaxy. Earlier, we achieved In-rich defect free heterostructures with photoluminescence wavelength up to 600 nm [1]. Here, we discuss the key parameters for their growth, especially the In/Ga flux ratio defining NWs morphologies, and substrate temperatures allowing to control the In content and in particular to further shift the wavelength towards the red color. We also studied here the influence of doping, using Mg as p-dopant, and Si as n-dopant in view of the optimization of the electrical injection in the p-GaN/InGaN/n-GaN LED. We performed SEM / EDX analysis of InGaN/GaN heterostructures in nanowires, which show that the InGaN insertion tends to form a core-shell structure, with an InGaN core surrounded by a GaN shell. The red InGaN/GaN nanowire LED fabrication is underway.

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Abstract

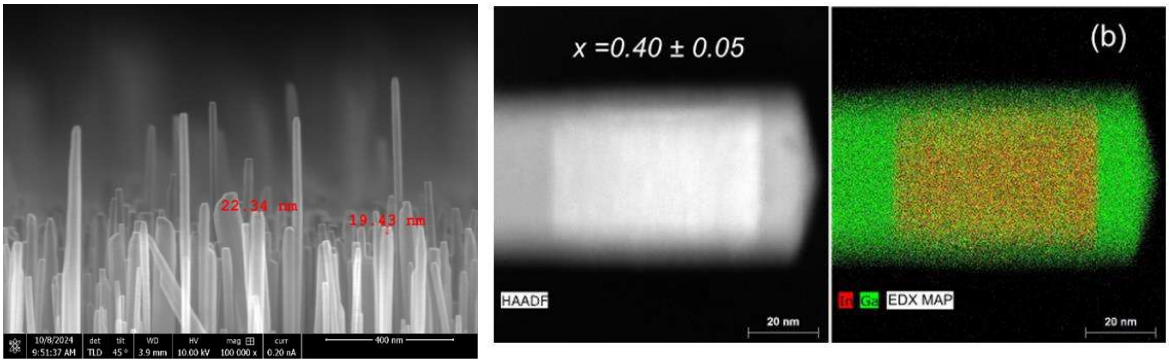
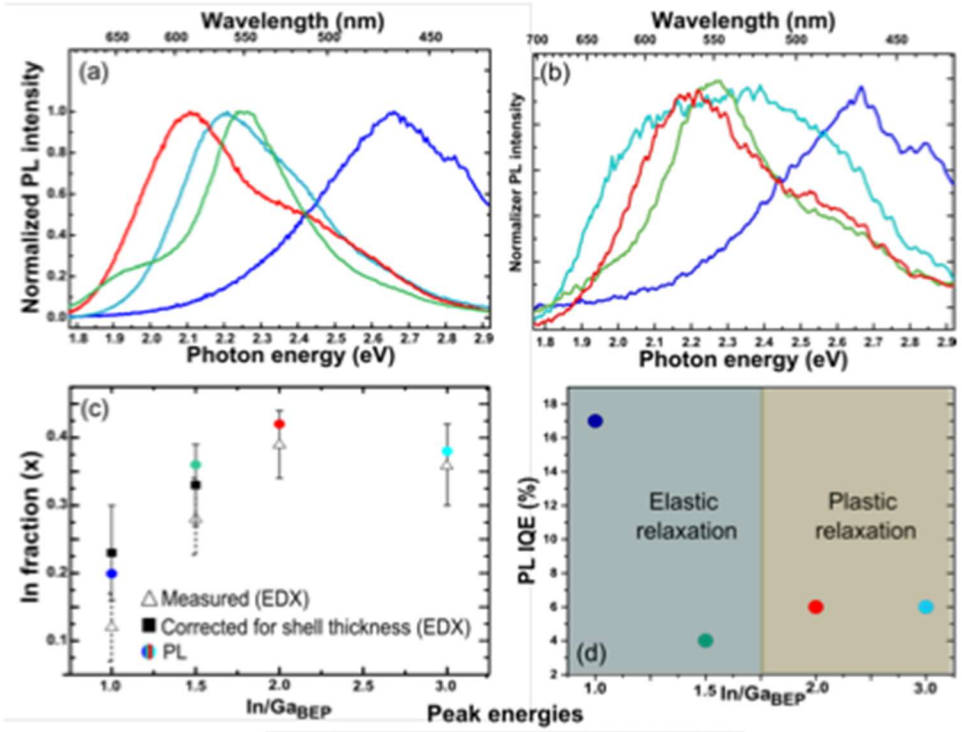


Fig. 1 Left – SEM imae of InGaN/GaN nanowires. Right – TEM image and the EDX map of the $In_xGa_{1-x}N$ insertion.



In/Ga	Sample name	4 K (eV)	300 K (eV)
1	#C016	2.65	2.7
1.5	#C028	2.25	2.3
2	#C017	2.2	2.2
3	#C041	2.1	2.3

Figure 2: Photoluminescence of InGaN nanowire ensembles at (a)10 K and (b) 300 K. (c) In fraction estimated from the PL peak and measured by EDX. (d) Optical internal quantum efficiency. Summary table.

Abstract

Thematic Session: Nano-synthesis & Nano-catalysis
Disciplinary fields involved: Chemistry, Materials
Keywords: Organometallic, heterogeneous nano-catalysts, hydrogen release

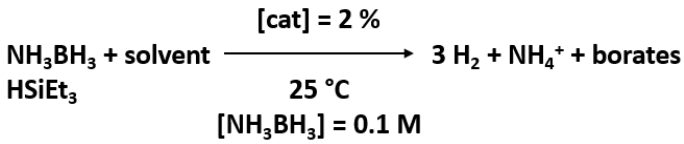
Design of efficient nano-catalysts for H_2 Release from Boranes and Silanes

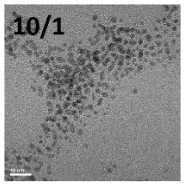
Maxime THIBAULT[1], Gizem KARACAOGLAN[1], Emmanuel LERAYER[1], Julien ROGER[1], Nadine PIRIO[1], Myrtil L. KAHN[2], Jean-Cyrille HIERSO[1]

1. *Institute of Molecular Chemistry of the University of Burgundy (ICMUB UMR CNRS 6302), University of Burgundy, Dijon, France*
2. *Laboratory of Coordination Chemistry (LCC UPR CNRS 8241), CNRS, Toulouse, France*

Abstract

The combustion of fuel produces deleterious gas (CO_2) for which the capture and recycling is mostly not achieved. Conversely, the use of chemical hydrogen storage materials, releasing H_2 –for combustion engines or fuel cells– and liquid or solid by-products, gives the important opportunity to easily capture and handle this co-produced waste[1]. On one hand, ammonia-borane NH_3BH_3 (AB, 19.5 wt% H) and $NaBH_4$ (10.8 wt% H) are able to release H_2 by hydrolysis and alcoholysis, in the presence of a metal catalyst in ambient conditions[2]. These reactions have attracted considerable attention, with special focus on the catalysts for fast H_2 delivery[3]. In comparison, critical aspects for the industrial implementation of this approach, like the nature of the solvolysis by-product(s) have been much less investigated, while their identification and recycling will be needed in a righteous circular approach[4]. On the other hand, organosilicons and specially hydrosilanes –as widely manufactured commercial products– may also be attractive as potential sources of H_2. This, following various catalyzed chemical roads, including hydrolysis and alcoholysis of silanes ($R_3Si–H$, R_2SiH_2, *etc.*), or their dehydrogenative oligomerization ($PhSiH_3$ dehydrocoupling)[5]. Here we will present the development of ruthenium, and iridium NPs for AB and hydrosilane (Et_3SiH) solvolytic dehydrogenation (H_2O, MeOH). The synthesis of Ru, and Ir nanocatalysts stabilized by bulky, rigid, σ-donating functionalized adamantane and diamantane ligands will be described and their performances in H_2 production discussed.





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[1] Anderson, J. et al., *Int. J. Hydrogen Energy*, **2019**, 11901-11919. [2] Xu, Q. et al., *ACS Catal.,* **2016**, 6, 6892. [3] Hierso, J.-C., Kahn, M., et al., *Small*, **2021**, 2102759. [4] Mboyi, C. D. et al., *Small* **2021**, *17* (44). [5] Huertos, M. A. et al., *Angew. Chem. Inter. Ed.,* **2022**, 61.

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