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	Nathaly CHAARAOUI ITheMM - URCA	Investigation of local thermal properties in nanostructured materials using Scanning Thermal Microscopy
17:15	Matias FELDMAN INSP - Sorbonne. Univ	Nanoscale control of heat flux in self-assembled ordered nanocrystal solids
17:30	François HENN L2C - Université de Montpellier	Engineering Individual SWCNT Nanofluidic Device for Enhanced Signal-to-Noise Ratio
17:45	Florant EXERTIER GPM - CNRS	Atomic scale microscopy of different materials by ultrashort THz-driven Atom Probe Tomography
18:00	Francois TREUSSART LuMIn - ENS Paris-Saclay	Polarization texture and sensing application of ferroelectric nanocrystals
18:15	Max GERIN ESRF	High pressure study of exotic hexagonal phase of Ge grown by molecular beam epitaxy on self-assisted GaAs nanowires

IDENTITY

Myriam TAVERNA (Univ. Paris Saclay - Inst. Galien Paris-Saclay, Orsay)

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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

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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]

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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.

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(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

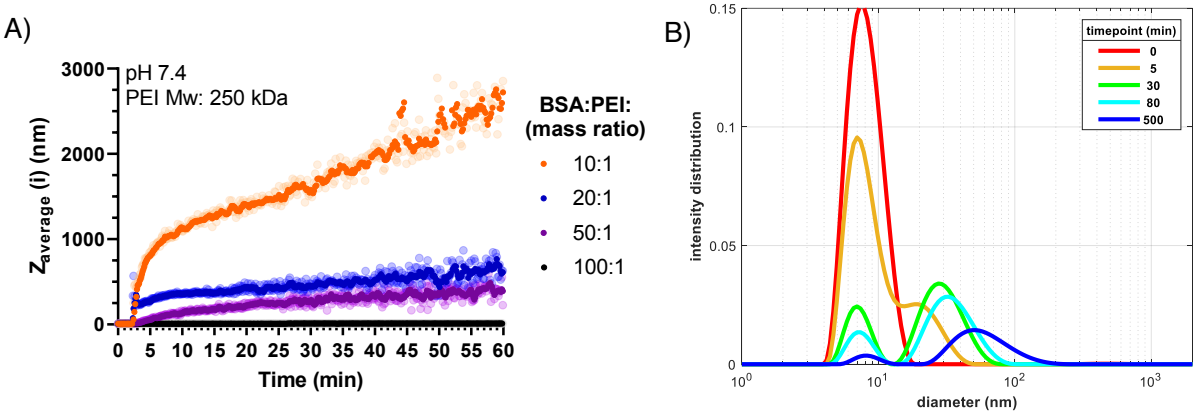

Figure 1: A) Real-time complex hydrodynamic diameter (Z_{av}) using SR-DLS. Different ratios of model protein were mixed with 250 kDa PEI. B) Intensity-based particle size distribution of BSA over 500 minutes at 65°C.

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.

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[3] T. Rooimans *et al.*, "Development of a compounded propofol nanoemulsion using multiple non-invasive process analytical technologies," *International Journal of Pharmaceutics*, vol. 640, p. 122960, Jun. 2023, doi: 10.1016/j.ijpharm.2023.122960.

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[5] "A New Method for Non-invasive or Inline Detection of Aggregates and Oversized Particles in Nanosuspensions," ResearchGate. Accessed: Nov. 28, 2024. [Online]. Available: https://www.researchgate.net/publication/385515333_A_New_Method_for_Non-invasive_or_Inline_Detection_of_Aggregates_and_Oversized_Particles_in_Nanosuspensions

Abstract

Thematic Session: Nanomaterials
Disciplinary fields involved: Physics
Keywords: Nanostructured materials, Scanning thermal microscopy (SThM), thermal probes, thermal conductivity, inverse methods.

Investigation of local thermal properties in nanostructured materials using Scanning Thermal Microscopy

Nathaly Chaaraoui[1*], Nathalie Trannoy[1] and Thierry Duvaut[1]

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Nanotechnologies require specific characterization methods for nanosystems and nanomaterials. Advanced understanding of heat transfer at the nanoscale and the thermal properties of nanostructured materials is crucial for the innovation of new materials. The thermal properties of thin films have been extensively studied for various materials, and scanning thermal microscopy (SThM), based on atomic force microscopy (AFM), proves to be a reliable tool for locally investigating the behavior of these materials and heat transfer mechanisms at the micro/nanoscale.
This project aims to evaluate the local thermal properties of nanostructured materials. For this purpose, two samples with buried nanostructures were specially designed. The first features a subsurface triangular step in SiO2 deposited on a silicon substrate, covered with polished CVD SiO2. The SiO2/Si interface is linear, with a thickness varying between 400 and 2150 nm. The second sample consists of three SiO2 steps with a thickness of 260 nm, covered with a SiO2 layer deposited on a silicon substrate. These two different geometries aim to study the microprobe's response in terms of the internal structuring of the sample and thus evaluate the local thermal conductivity of SiO2.
To obtain thermal information on nanostructured materials, the thermal microscope (SThM) must be equipped with a thermal sensor, enabling simultaneous acquisition of topographic and thermal images. A thermosensitive micrometric Wollaston probe was selected for this purpose. To interpret the SThM experimental results, a heat transfer model using the finite element method (FEM) was developed to (a) study the impact of the sample structure on the probe's thermal signal and (b) characterize the local thermal conductivity.
Using the numerical model, an inverse technique was implemented with the Levenberg-Marquardt algorithm, combining two inverse methods: the Gauss-Newton method and the conjugate descent method, to evaluate the local thermal conductivity from the power dissipated by the probe. The numerical model, based on the electrothermal coupling of the probe/sample system, evaluates the heat flux dissipated by the thermoresistive element and towards the sample. Comparison between simulation and experimental measurements shows that the model reproduces the experimental thermal profiles obtained from thermal images and for the two variable SiO2 thickness. It is worth noting that the sample's design ensures same contact for every measurement point. Our results indicate a decrease in thermal conductivity with the thickness of the SiO2 layer. These findings are confirmed with a differently organized nanostructure having the same material composition. The methodology is ongoing with the palladium probe, as the experimental profiles have been reproduced.

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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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:

(1) Feldman M.; Vernier C.; Nag R.; Barrios J.; Royer S.; Cruguel H.; Lacaze E.; Lhuillier E.; Fournier D.; Schulz F.; Hamon C.; Portalès H.; Utterback J. K. Anisotropic Thermal Transport in Tunable Self-Assembled Nanocrystal Supercrystals. *ACS Nano* 2024.

(2) Utterback, J. K.; Sood, A.; Coropceanu, I.; Guzelturk, B.; Talapin, D. V.; Lindenberg, A. M.; Ginsberg, N. S. Nanoscale Disorder Generates Subdiffusive Heat Transport in Self-Assembled Nanocrystal Films. Nano Lett. 2021, 21 (8), 3540−3547.

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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

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2. Processes, apparatus and general chemical technology department, Ukrainian State University of Chemical Technology, Dnipro, Ukraine;
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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
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[4] M. Manghi et al. J. Phys. Chem. C, 2021, 125, 22943

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).
[iii] B. Gault, et al., Nat Rev Methods Primers, Volume **1**, (2021).
[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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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

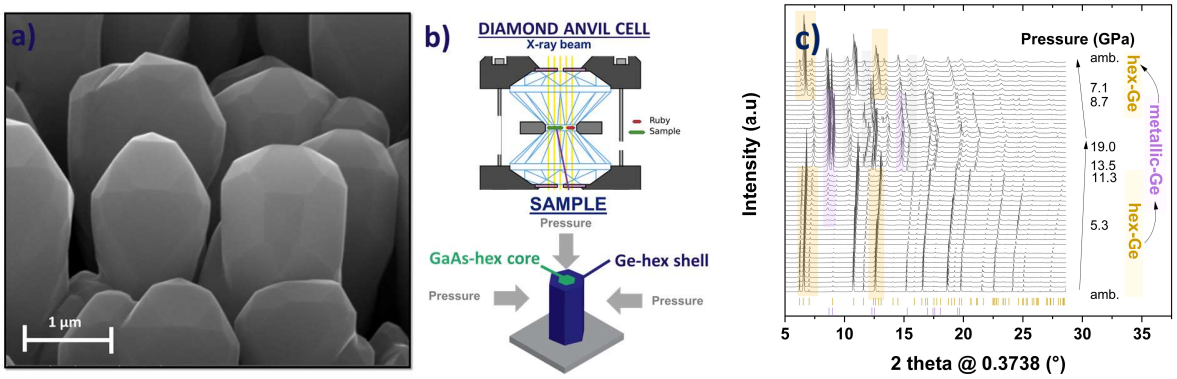

Figure. a) SEM image of GaAs@hex-Ge NW b) schematics of the diamond anvil cell and nanowire c) X-ray diffraction patterns observed during pressure cycling up to 20 GPa.

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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

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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

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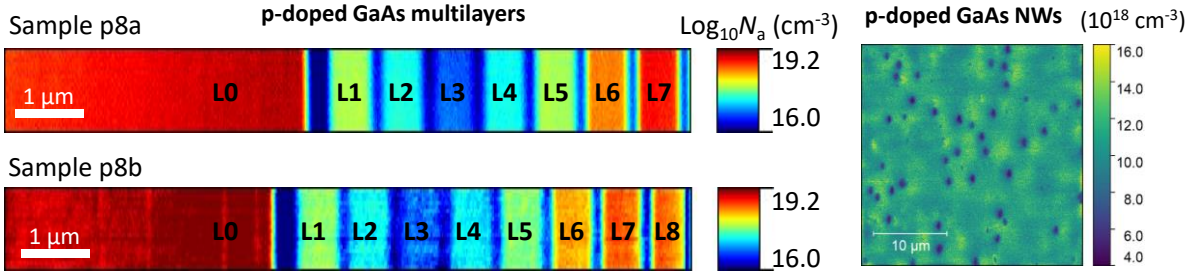
Abstract

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.



Abstract

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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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2. *Institut Photovoltaïque d'Ile de France (IPVF) – Palaiseau, France*
3. *Centre de Nanosciences Et de Nanotechnologies (C2N), CNRS, Université Paris-Saclay, Palaiseau, France*
4. *Ecole Centrale de Lyon, CNRS, INSA Lyon, Université Claude Bernard Lyon 1, CPE Lyon, INL, Ecully, France*

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

C. Njel[1,2], H. A. Girard[2], M. Frégnaux[1], D. Aureau[1], J. C. Arnault[2]

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[2] *Université Paris-Saclay, CEA, CNRS, NIMBE, 91191, Gif sur Yvette, France*

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.

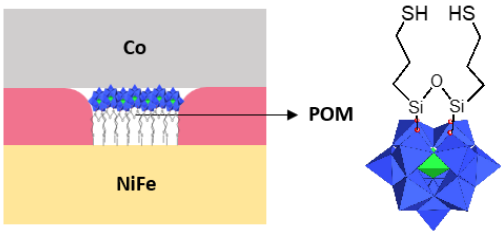

Figure 1: Representation of a MTJ integrating POM molecules

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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:

The authors would like to thank the European project “ETHNOTEVE” which was funded by the European Union within the framework of Operational Program ERDF/ESF 2014-2020 and the Communauté d’Agglomération du Cotentin for its financial support.