BOOK OF ABSTRACTS Oral Presentations



SESSION ANR

Thursday March 20th 10:30 A.M. - 12:30 A.M.

Program of the session :

SALLE AB

TITLE

HOUR COORDINATOR

| 09:00 10:00 | Poster S | ession - ANR |
|----------------|--|--|
| 10:00 10:30 | Coffee break / exhibition stands session | |
| 10:30 | Gwénolé JACOPIN | Imaging carrier dynamics at the nanoscale thanks to light- and electron-matter interactions |
| 10:45 | Yan PICARD/ Clelia BASTELICA | Focused Ion Beam with correlated electron feeback: sub-nm resolution |
| 11:00 | Ludovic DESPLANQUE | In-plane nanowires with strong spin-orbit coupling for scalable mesoscopic devices |
| 11:15 | Thomas PONS | Nanowhispers – Bio-sensing using energy transfer from nanocrystal-doped whispering gallery mode microcavities |
| 11:30 | Yannick MUGNIER | Design of multifunctional lithium niobate (LiNbO3) nanoparticles for multimodal imaging and theranostic applications |
| 11:45 | Sylvie BEGIN | Phosphate capture enhancement in peritoneal dialysis process using designed iron oxide nanostructures |
| 12:00 | Estelle LEBEGUE | Electrochemistry of redox liposome nano-impacts for bacterial toxins sensing |
| 12:15 | Antoine THILL | Hybrid imogolites as tunable reactors |

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 \Box CE09 - Nanomaterials and nanotechnologies for the products of the future \boxtimes CE42 - Sensors, instrumentation

Project acronym: INDIANA

Funding instrument (JCJC, PRC, PRCE, PRCI): JCJC Keywords (max 4): Cathodoluminescence, micro-LED, nano-thermometry

Imaging carrier dynamics at the nanoscale thanks to light- and electron-matter interactions

Gwénolé Jacopin¹, Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel, 38000 Grenoble, France

To enhance performance and enable new applications, there is a strong trend toward miniaturizing electronic and optoelectronic devices. This is particularly evident in the development of micro-scale lightemitting diodes (micro-LEDs) for applications such as micro-displays and optogenetics, as well as power transistors with channel dimensions in the nanometer range. However, as device dimensions shrink, their performance is significantly impacted, resulting in efficiencies far below their theoretical limits. At these scales, two phenomena play a critical role: surface recombinations and thermal heating. Although these effects are well-known, quantifying their impact at the nanoscale remains challenging.

Within the ANR INDIANA project, we developed innovative tools to characterize the optoelectronic properties and local temperature of these devices. Our approach focuses on exploring the specificities of light-matter and electron-matter interactions at the nanoscale in order to image these phenomena. Specifically, the project achieved three main outcomes:

- (1) We quantified the influence of surface recombinations and dislocations in LEDs and micro-LEDs by combining time-correlated cathodoluminescence and time-resolved photoluminescence techniques. This allowed us to disentangle the relative contributions of surface recombinations, point defects, and dislocations to device performance [1-3].
- (2) We developed a nanoscale thermometer based on the luminescence of nanodiamonds. These were selected for their exceptional thermal conductivity, stability under electron beam exposure, and high brightness [4].
- (3) Finally, we successfully demonstrated the use of this nanothermometer to measure the in-situ heating of blue LEDs during operation.

These developments provide key insights into nanoscale heat dissipation and recombination phenomena, paving the way for optimizing the performance of next-generation miniaturized devices.

[1] S. Finot *et al.*, ACS Photonics 2022, 9, 1, 173-178 (2022)

[2] P. Sáenz de Santa María Modrono et al., ACS Photonics 11, 6, 2406–2412 (2024)

[3] P. Lottigier et al., Nanomaterials 2023, 13(18), 2569

[4] P. Sáenz de Santa María Modrono et al., Phys. Status Solidi A, 2400573 (2024)

Acknowledgement: INDIANA Project No. ANR-21-CE42-0020-01

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CE09 - Nanomaterials and nanotechnologies for the products of the future
 CE42 - Sensors, instrumentation

Project acronym: FIBback

Funding instrument (JCJC, PRC, PRCE, PRCI): PRCE Keywords (max 4): FIB, Correlations, Rydberg, deterministic source

Focused Ion beam with correlated electron feedback: sub-nm resolution

C. Bastelica¹, A. Trimeche, M. Vitteau², M. Schmidt³, C. Lopez³, D. Comparat, Y. Picard

- 1. Université Paris-Saclay, CNRS, Laboratoire Aimé Cotton, 91405, Orsay, France
- 2. Orsay Physics, 95 Avenue des Monts Auréliens, 13710 Fuveau, France
- 3. Université Paris-Saclay, CNRS, Ecole Normale Supérieure Paris-Saclay, CentraleSupélec, LuMIn, Orsay, France

Electron and ion beams have become indispensable tools in surface and material sciences, with for everincreasing demands, i.e. resolution and determinism. This project aims to develop a Focused Ion Beam (FIB), called FIBback, leveraging two innovative concepts:

- A FIB based on a cesium atoms beam cooled and collimated by lasers, excited to a Rydberg state and then ionized by field [1, 2].
- A correlated source of ions and electrons that demonstrates, among other applications, complete trajectory control of the ion using information from its correlated electron [3, 4].

We aim to enhance the resolution of our FIB by enabling the collection and detection of each electron created during the ionization process. To achieve this, we have developed several optical elements and a detector that will be incorporated into the FIB column. These additions will allow us to maintain the existing correlation between the detected electron an the ion hitting the target, both coming from the same atom. This correlation is used to enhance beam properties. The beam resolution will be improved either by using ghost imaging [3] or using the real-time trajectory control on each ion [4]. This control will also provide us a deterministic ion source. With this innovative FIB, we aim to achieve sub nanometer-scale resolution at low energy, paving the way for high-resolution non-destructive imaging applications and deterministic implantation experiments.

- [1] A. Delobbe, et al., Microsc. Microanal., vol. 24, pp. 804--805 (2018)
- [2] M. Viteau, et al., Ultramicroscopy, vol. 164, pp. 70-77 (2016)
- [3] C. Lopez, et al., Phys. Rev. Applied, vol. 11, pp. 064049 (2019)
- [4] A. Trimeche, et al., Phys. Rev. Research, vol. 2, pp. 043295 (2020)

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Acknowledgement: We acknowledge our colleagues from the technical service of LacTech. We acknowledge fundings from Agence National de la Recherche (ANR-21-CE42-0010-01 FIBBack).

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■ CE09 - Nanomaterials and nanotechnologies for the products of the future □ CE42 - Sensors, instrumentation

Project acronym: INSPIRING

Funding instrument (JCJC, PRC, PRCE, PRCI): PRC Keywords: semiconductor, nanowire, selective epitaxy

In-plane nanowires with strong spin-orbit coupling for scalable mesoscopic devices

L.Desplanque¹, P.Ballet², H.Sellier³, B.Grandidier¹, W.Khelifi¹, N.Chaize², C.Barbot¹

- 1. Univ. Lille, CNRS, Centrale Lille, ISEN, Univ. Valenciennes, UMR 8520–IEMN, F-59000 Lille, France
- 2. CEA, LETI, Univ. Grenoble Alpes, 38000 Grenoble, France
- 3. Univ. Grenoble Alpes, Institut Néel, F-38000 Grenoble, France

The fabrication of quantum bits robust to decoherence could be achieved by hybridizing a strong-spin orbit one-dimensional semiconductor with a regular superconductor [1,2]. A reliable and scalable process based on this concept implies the use of high quality in-plane nanowires (NWs). In the Inspiring project, we have demonstrated that selective area molecular beam epitaxy (SAMBE) enables the growth of HgTe, InAs and InSb inside a nanoscale dielectric mask designed using electron-beam lithography and the reactive ion etching of a thin SiO₂ layer deposited on CdTe, InP or GaAs substrates [3-5]. The intrinsic conductance of the nanostructures have been measured using four-probe scanning tunneling microscopy and magneto-transport measurements at cryogenic temperatures have been performed after connecting the NWs with metal contacts, demonstrating, in the case of InAs, the benefit of protecting the NWs with a larger bandgap semiconductor shell. Current developments concern substrate engineering authorizing the tuning of the charge density in the HgTe and InSb NWs.

References:

[1]Kitaev et al. Phys.-Usp. 44 131 (2001) <u>https://doi.org/10.1070/1063-7869/44/105/S29</u>
[2]Wang et al. Nature 612, 448–453 (2022). <u>https://doi.org/10.1038/s41586-022-05352-2</u>
[3]Khelifi et al. Nanotechnology 34 265704 (2023) <u>https://dx.doi.org/10.1088/1361-6528/acc810</u>
[4]Chaize et al. Nanotechnology 35 505602 (2024) <u>https://dx.doi.org/10.1088/1361-6528/ad7ff4</u>
[5] Khelifi et al. 'Selective area molecular beam epitaxy of InSb on InP(111)B: from thin films to quantum nanostructures', under review

Acknowledgement:

This study was financially supported by the French National Research agency under the project INSPIRING ANR-21-CE09-0026-01, the French Technological Network Renatech, and the Région Hauts de France.

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□ CE09 - Nanomaterials and nanotechnologies for the products of the future X CE42 - Sensors, instrumentation

Project acronym: Nanowhispers Funding instrument: PRC Keywords: microcavity, nanoparticles, biosensing, energy transfer

Nanowhispers – Bio-sensing using energy transfer from nanocrystal-doped whispering gallery mode microcavities

Nour Alkastntini¹, Charlie Kersuzan^{1,3}, Subha Jana¹, Xiangzhen Xu¹, Céline Roux-Byl¹, Abdel Wahab Mouhamad¹, Nina Melnychuk², Andrey Klymchenko², Andreas Reisch², Sergei Celaj³, Agnès Maitre³, Thomas Pons¹

- 1. LPEM, UMR 8213, ESPCI Paris PSL, CNRS, Sorbonne Université, Paris, France.
- 2. LBP, UMR 7020, CNRS, Université de Strasbourg, Strasbourg, France.
- 3. INSP, UMR 7588, CNRS, Sorbonne Université, Paris, France.

Abstract

Fluorescent biodetection assays using pairs of fluorescent donors and acceptors interacting via Förster Resonant Energy Transfer (FRET) are appealing thanks to their ease of use, versatility and specificity. They are however limited in sensitivity due, in particular, to their limited distance range. In this project, we are developing a novel type of biodetection assay based on energy transfer using Whispering Gallery Modes (WGM) from optical microcavities excited by fluorescent quantum dots as donors to polymeric dye-loaded nanoparticles (dyeNP) as acceptors. To this aim, we have developed the synthesis and biofunctionalization of these fluorescent emitters. In particular, we have studied their interactions in a model system using streptavidin-coated microbeads labeled with quantum dots and biotinylated dyeNPs. Upon their specific biomolecular interaction, the dyeNP bind to the microcavity surface, leading to efficient energy transfer, with a typical sensitivity in the pM range, 3 orders of magnitude more sensitive than typical FRET assays. We have further developed their functionalization with antibodies and oligonucleotides in order to make them versatile biosensors for DNA sequences or protein antigens. Finally, we have developed the controlled fabrication of polymeric microcavities onto glass substrates, coated them with semiconductor nanocrystals and demonstrated their use as microlasers, which opens the way to novel, more sensitive energy transfer-based biosensors.

References:

Jana et al., ACS nano 15 (1), 1445-1453 (2020) ; Kersuzan et al., ACS Photonics 11 (4), 1715-1723 (2024).

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Acknowledgement:

We acknowledge funding from the Agence Nationale pour la Recherche (Nanowhispers, ANR-21-CE42-0029) and from Institute of Materials Science (iMAT) of the Alliance Sorbonne Universite.

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☑ CE09 - Nanomaterials and nanotechnologies for the products of the future □ CE42 - Sensors, instrumentation

Project acronym: DARE, Deep And REsolved

Funding instrument: PRCI

Keywords: multifunctional LiNbO₃-based nanocrystals, formation mechanisms, multimodal imaging, cell targeting and drug delivery

Design of multifunctional lithium niobate (LiNbO₃) nanoparticles for multimodal imaging and theranostic applications

Ameni Dhouib¹, Alessandra Spada², Paulí Figueras Llussà³, Simon Guené-Girard⁴, Isabelle Gautier-Luneau⁴, Luigi Bonacina³, Sandrine Gerber-Lemaire², Yannick Mugnier¹

- 1. Université Savoie Mont Blanc, SYMME, Annecy, France
- 2. Ecole Polytechnique Fédérale de Lausanne, Group for Functionalized Biomaterials, Lausanne, Suisse
- 3. Université de Genève, Nonlinear Bioimaging Lab, Genève, Suisse
- 4. Université Grenoble Alpes, Institut Néel, Grenoble, France

Abstract

As tracking of single cells is very challenging in terms of sensitivity, selectivity, spatial resolution and imaging penetration depth in the host organism, design of multifunctional exogeneous probes allowing multimodal contrast mechanisms and on-demand release of drug molecules is highly sought for. Inorganic nanoparticles have thus been proposed as promising candidates because of the multiple physical responses of their core structures and their cell-targeting properties after proper surface functionalization. If several proof-of-concept studies have already been demonstrated in terms of imaging/targeting/delivery, none of them make use of LiNbO₃ nanoparticles although their potential has already been evidenced in separate studies evidencing their good biocompatibility, excellent nonlinear optical properties [1] and, dual optical and MRI contrast agent capabilities [2].

Within the DARE project, functionalized lanthanide-doped LiNbO₃ nanoparticles are produced with the aim to fully understand their growth mechanism, to modify their surface chemistry for cell-targeting and drug delivery, and to quantitatively assess their contrast efficiencies for a range of imaging techniques including optical microscopy, MRI and computed tomography. In this presentation, our deep understanding of the reaction pathway and formation mechanisms of the LiNbO₃ nanoparticles will first be discussed according to the non-classical nucleation and crystallization processes [3]. This sol-gel route was also proven successful for the preparation of highly-doped nanocrystals with optimal concentrations in Er and Yb, thus resulting in simultaneous emission of second harmonic and up-conversion signals under femtosecond excitation [4].

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Coating protocols making use of water-in-oil microemulsions achieved efficient silanization and covering with peptide mimics, for enhanced biocompatibility and surface display of reactive functionalities. Post-conjugation through click-chemistry based reactions allowed sequential introduction of caged therapeutic cargos and cancer cell-specific ligands targeting the overexpression of epithelial growth factor receptor (EGFR). The targeted imaging and light-induced chemotherapeutic release capabilities [5] of the resulting multifunctional NPs was evidenced on cancer cell models.

References:

[1] Campargue G., et al. *Multiorder nonlinear mixing in metal oxide nanoparticles*, Nano Letters 20(12) 2020

[2] De Matos R., et al. *Gd*³⁺-functionalized lithium niobate nanoparticles for dual multiphoton and magnetic resonance bioimaging, ACS Applied Nano Materials, 5(2) 2022

[3] Riporto F., et al. Nonclassical nucleation and crystallization of LiNbO₃ nanoparticles from the aqueous solvothermal alkoxide route, Small, 20(13) 2024

[4] Bredillet K. et al., Dual second harmonic generation and up-conversion photoluminescence emission in highly-optimized LiNbO₃ Nanocrystals doped and co-doped with Er^{3+} and Yb^{3+} , Nanoscale, 16(13) 2024

[5] Gheata A., et al. *Photoresponsive Nanocarriers Based on Lithium Niobate Nanoparticles for Harmonic Imaging and On-Demand Release of Anticancer Chemotherapeutics*, ACS Nanoscience Au 2(4)2022

Acknowledgement:

This work is supported by the French Agence Nationale de la Recherche (DARE, ANR-21-CE09-0036-01) and the Swiss National Science Foundation (DARE, grant 200021E_205754)

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■ CE09 - Nanomaterials and nanotechnologies for the products of the future □ CE42 - Sensors, instrumentation

Project acronym: PHODIA

Funding instrument (JCJC, PRC, PRCE, PRCI): PRCE Keywords (max 4): phosphate, peritoneal dialysis, iron oxide nanoparticles, dialysis set-ups

Phosphate capture enhancement in peritoneal dialysis process using designed iron oxide nanostructures

Théo Lucante ^{1,2}, Philippe Choquet ^{4*}, Ariane Zaloszyc ^{2,3*} and Sylvie Begin-Colin ^{1,2*}

- 1. University of Strasbourg, CNRS, Institut de Physique et Chimie des Matériaux de Strasbourg, UMR 7504, 67034 Strasbourg, France;
- 2. University of Strasbourg, CNRS, Institut de Chimie et Procédés pour l'Energie, l'Environnement et la Santé, UMR-7515, 25 rue Becquerel, 67087 Strasbourg, France;
- 3. Nephro-pediatry, CHU Hautepierre, Hoptaux Universitaire de Strasbourg, 1 avenue Molière, 67 098 Strasbourg.
- University of Strasbourg, CNRS and HUS, Laboratoire des Sciences de l'Ingénieur, de l'Informatique et de l'Imagerie (ICube) – CNRS/University of Strasbourg, UMR 7357 Preclinical Imaging Lab, Imaging Dpt, Hôpitaux Universitaires de Strasbourg, France.

Abstract (no longer than 300 words or 20 lines max. incl. figure), Calibri 11, single line spacing, black)

Chronic kidney disease (CKD) induces the loss of blood purification functions by the kidneys in patients. Among toxic elements, phosphates are a special species whose chronically excessive concentration in the blood can lead to life-threatening complications, notably cardiovascular problems. Peritoneal dialysis (PD) is one of two dialysis modalities for treating patients with CKD and offers advantages such as a preservation of vascular access and renal residual function, less drugs administration, a higher suitability for treating children and infants, a low cost by comparison with the more conventional dialysis mode: hemodialysis. However, its phosphate removal efficiency needs to be improved to upgrade this dialysis technique. In PHODIA, we have studied the addition of iron oxide nanoparticles (IONPs) into dialysate to enhance phosphate removal from blood and even to reduce the PD duration. Surfactant-coated IONPs have been designed to balance between a (bio)compatibility with the PD process (which requires IONPs which do not cross the peritoneal barrier) and a high efficiency of phosphate capture (lowest amount of introduced IONPs). Surfactant coated iron oxide nanostructures were synthesized through polyol solvothermal and coprecipitation approaches. Phosphate removal experiments in water and dialysate solutions have been performed in batch mode but also in two laboratory-scale dialysis set-ups. The removal efficiency was found strongly dependent on the surfactant coating and synthesis method. All these experiments have allowed us selecting the most suitable

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surfactant coated IONPs for our application. They have been then tested in a specifically designed device mimicking as close as the PD process. These experiments have confirmed the suitability of this strategy to enhance and speed up phosphate removal efficiency. These results strongly demonstrated the potential of adding stable, biocompatible IONPs in dialysate to enhance phosphate transport in PD process.

References (max. 5):

- J. Vaz-Ramos et al. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 2024, 689, 133658
- 2023: DIALYSATE COMPOSITION COMPRISING A SPECIFIC COMPOUND, Patent Europe N° 233306319.7-1112

Acknowledgement:

The PHODIA project was supported by ANR (ANR-21-CE09-0037).

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 \Box CE09 - Nanomaterials and nanotechnologies for the products of the future \boxtimes CE42 - Sensors, instrumentation

Project acronym: ELIPOX

Funding instrument (JCJC, PRC, PRCE, PRCI): JCJC Keywords: redox liposomes, nano-impacts, bacterial toxins, electrochemical sensing

Electrochemistry of redox liposome nano-impacts for bacterial toxins sensing

Estelle Lebègue¹

1. Nantes Université, CNRS, CEISAM, UMR 6230, F-44000 Nantes, France

The most recent advances in single liposome electrochemistry are the single-vesicle electrochemical events with the exocytosis process, the electrochemical single impacts of synthetic redox liposomes, and the electroanalysis of single giant unilamellar vesicle [1]. About single-impact of synthetic redox liposomes, two main different methods are used for characterizing liposomes at single-cell scale: the blocking impact method and the electrolysis method. Blocking impact electrochemistry is very versatile and efficient for detecting various insulating particles in a short analysis time and with a high sensitivity. It consists to detect a "current step" in the chronoamperometry (i-t) curve when a liposome collides with the ultramicroelectrode (UME) polarized at the redox potential of the aqueous electroactive probe [2]. The current step magnitude in blocking impact experiments depends on the size of the adsorbed liposome, the size and the shape of the UME, the type and the concentration of the redox probe in solution, the applied potential and the location of the liposome adsorbed onto the UME surface [3].

redox probe because it consists to detect the electrolysis of this redox content during the release at the UME after the single impact [1]. This sensitive technique is extended to sensing virulence factors from pathogenic bacteria, where redox liposomes are based on 1,2-dimyristoyl-*sn*-glycero-3-phosphocholine (DMPC) as a pure phospholipid and potassium ferrocyanide as an encapsulated redox probe [4].

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- [3] Langlard, A.; Smida, H.; Chevalet, R.; Thobie-Gautier, C.; Boujtita, M.; Lebègue, E. ACS Meas. Sci. Au 2024, 4 (5), 585–592. <u>https://doi.org/10.1021/acsmeasuresciau.4c00046</u>
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Acknowledgement:

ANR-21-CE42-0007-01

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x CE09 - Nanomaterials and nanotechnologies for the products of the future □ CE42 - Sensors, instrumentation

Project acronym: BENALOR Funding instrument (JCJC, PRC, PRCE, PRCI): PRC Keywords (max 4): nanoreactor, imogolite, adsorption, synthesis

Hybrid imogolites as tunable reactors

Antoine Thill¹, Bénédicte Prélot², Lorette Sicard³, Fadwa Al Fadel Raad^{1,3}, Ali Dhaini², Zhang Ye³, Delphine Schaming³, Pierre Picot¹, Jerzy Zajac², Pierre-Marie Gassin², Gaelle Gassin-Martin², Sophie Le Caër¹, Frédéric Gobeaux¹, Fabienne Testard¹, Philippe Trens².

- 1. Université Paris-Saclay, CEA, CNRS, NIMBE, 91191 Gif-sur-Yvette, France.
- 2. ICGM, University of Montpellier, ENSCM, CNRS, Montpellier, France
- 3. Université Paris Cité, CNRS, ITODYS, F-75013 Paris, France

Hybrid aluminosilicate nanotubes are able to encapsulate small molecules within their polarized cavity. The possibility to modify the internal surface and tune the interaction with the nanotube and the encapsulated molecules makes these hybrid imogolites potentially interesting chemical nanoreactors. In the BENALOR project, we explored the bottom-up synthesis of hybrid imogolite nanotubes and managed to improve their yield and purity. We studied the adsorption properties of the hybrid nanotubes combining gas adsorption, calorimetry and Small Angle X-ray Scattering². We explored the encapsulation of dyes and redox active molecules inside the nanotubes. For some favorable systems, we used second harmonic scattering to decipher the precise organization of the dyes within the nanometric cavity^{1,3}. The reactivity of the encapsulated molecules was explored under UV irradiation to monitor the role of the encapsulation in the photoreactivity of the molecules. It is shown that hybrid imogolite can either stabilize a dye or improve its photodegradation. We also coupled the hybrid nanotubes with external catalytic centers (gold nanoparticles)⁵. This coupling enables a strong increase of the photoreactivity. Finally, the charge transfer within the encapsulated molecules has been explored using model redox active molecules (ferrocene, ferrocene methanol). Using cyclic voltammetry, we show that the encapsulated molecules can reversibly exchange electrons with an electrode. A charge transfer mechanism is proposed. Finally, we show that the chemical stability of ferrocene molecules is affected by the encapsulation and the degradation mechanism is explored.

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1) Dhaini et al., Second harmonic scattering reveals different orientational orders inside the hydrophobic cavity of hybrid nanotubes, *Journal of Chemical Physics*, 2024, 161, 13, 134707, DOI: 10.1063/5.0226364

2) Dhaini et al., Dried hybrid imogolite nanotubes as solids with a changeable surface area: an insight into textural properties based on the correlation between nitrogen gas adsorption, immersion calorimetry into water, and small angle X-ray scattering, *Physical Chemistry Chemical Physics*, 2024, 26, 36, 23835-23845 DOI: 10.1039/d4cp02396j

3) Dhaini et al., Hydrophobic dye solubilization <i>via</i> hybrid imogolite nanotubes probed using second harmonic scattering, *Physical Chemistry Chemical Physics*, 2023, 37591824, DOI : 10.1039/d3cp02780e

4) Patra et al., UV-Visible photo-reactivity of permanently polarized inorganic nanotubes coupled to gold nanoparticles, *Nanoscale*, 2023, 15, 8, 4101-4113 DOI:10.1039/d2nr05796d

5) Patra et al., Inorganic nanotubes with permanent wall polarization as dual photo-reactors for wastewater treatment with simultaneous fuel production, *Environmental Science-Nano*, 2021, 8, 9, 2523-2541 DOI: 10.1039/d1en00405k

Acknowledgement:

The SWAXS lab platform of NIMBE and LLB is acknowledged for the SAXS facility. We want to thank O. Taché and E. Cournède for their help with the SAXS experiments in the SWAXS Lab. This work was funded by the ANR grant BENALOR (ANR-20-CE09-0029).

SESSION ANR

Thursday March 20th 12:30 A.M. - 16:30 P.M.

Program of the session :

SALLE AB

HOUR COORDINATOR

18:00

TITLE

| 12:30 14:00 | Lunch | |
|----------------|--|--|
| 14:00 | Guillaume VIAU | Nucleation, Growth and Integration of Magnetic Nanorods |
| 14:15 | Vincent GARCIA | Tailoring topological states in multiferroics |
| 14:30 | Pascal RUELLO | THz dynamics in MUltiFerrolc Nanostructures and Superlattices |
| 14:45 | Jonathan AMODEO | SASHA: Surface state and mechanics of nano-objects |
| 15:00 | David BABONNEAU | Intelligent real-time manipulation of metal nanostructure growth |
| 15:15 | Vincent FOURNEE | New ultrathin oxide films on metal substrates |
| 15:30 | Rodrigue LESCOUEZEC | Electronically-Active Thin Films for New Concept of Nano-Devices |
| 15:45 | Jerome LAGOUTE | Defect engineering in 2D materials |
| 16:00 | Beniamino SCIACCA | Designer metasurfaces from colloidal building blocks |
| 16:15 | Damien VOIRY | 2D nanolaminate - nanofluidic ionic diodes hybrid membranes for desalination and water purification (2D-MEMBA) |
| 16:30 17:00 | Coffee break / exhibition stands session | |
| 17:00 | Poster | Session - ANP |

Poster Session - ANR

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☑CE09 - Nanomaterials and nanotechnologies for the products of the future □ CE42 - Sensors, instrumentation

Project acronym: NIMRod Funding instrument: PRC Keywords (max 4): Seed-mediated growth, Nanorods, Magnetophoresis, SAXS

Nucleation, Growth and Integration of Magnetic Nanorods

R. K. Ramamoorthy¹, L.-M. Lacroix¹, S. Cayez¹, N. Ratel¹, K. Soulantika¹, T. Blon¹, G. Viau¹, R. Parmar², I. Rodriguez-Ruiz², S. Teychené², J. Marcone³, C. Goldman³, C. Hamon³, P. Davidson³, M. Imperor³

- 1. Laboratoire de Physique et Chimie des Nano-Objets, UMR 5215 INSA CNRS UT3, Toulouse, France
- 2. Laboratoire de Génie Chimique, UMR 5503, INP CNRS UT3, Toulouse, France
- 3. Laboratoire de Physique des Solides, UMR 8502 CNRS Université Paris Saclay, Orsay, France

The aim of the NIMRod is to develop new routes for the synthesis of metallic magnetic nanorods (NRs) through heterogeneous nucleation (WP1) with designed seeds (WP2), to control their alignment, densification (WP3) and integration to fabricate performant NR based submillimetric permanent magnets. The different steps, nucleation, growth, alignment and assembly, were followed by small angle X-ray scattering (SAXS) and electron microscopy.

The synthesis approach requires to control the structure of ultra-small nanoparticles (NPs) that are used in the seed-mediated growth of the second metal. Experimental parameters controlling the structure of ultra-small Au and Ag NPs were identified [1]. The crystallization of the different polymorphs (truncated octahedra, icosahedra, decahedra) was followed by time-resolved in situ SAXS using a homemade microfluidic set-up designed for the project [2]. The role of confinement of the nucleation stage seems to be the key point that orients the crystallization of icosahedral NPs. The seed-mediated growth of nickel NPs on decahedral Au seeds was carried out. Conditions of Ni epitaxy on Au seeds were determined and Ni decahedra or NRs were obtained depending on the growth conditions.

Depletion induced self-assembly and magnetic field induced assembly were studied on model NRs (pentagonal cross-section Au NRs and hcp-Co with different diameters and aspect ratio) by in situ SAXS and U-SAXS at SOLEIL (Swing) and ESRF (ID2). Self-assembly of nanorods with a pentagonal cross-section is giving rise to a rich polymorphism [3]. Modelling of the SAXS required to derive the form factor of nanoprisms with any regular cross-section and aspect ratio [4]. Alignment in liquid phase under increasing magnetic fields revealed different behaviors depending on the starting suspension of Co NRs (ID2). The mean distance between the NRs was followed all along the drying (ID2). Finally, a mapping of Co NRs alignment inside magnets was established thanks to a microfocus set-up (SWING).

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Acknowledgement:

This study has been supported through the French national project NIMRod (ANR-21-CE09-0019-01)

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➡ CE09 - Nanomaterials and nanotechnologies for the products of the future
 □ CE42 - Sensors, instrumentation

Project acronym: TATOO

Funding instrument (JCJC, PRC, PRCE, PRCI): PRC Keywords (max 4): multiferroics, antiferromagnetism, topology, nanostructures

Tailoring topological states in multiferroics

Vincent Garcia¹, Stéphane Fusil¹, Michel Viret², Jean-Yves Chauleau², Vincent Jacques³, Aurore Finco³, Brahim Dkhil⁴, Nicolas Jaouen⁵, Arthur Chaudron¹ (PhD), Amr Abdelsamie¹ (Post-doc), P. Dufour¹ (PhD)

- 1. Laboratoire Albert Fert, CNRS, Thales, Université Paris-Saclay, Palaiseau, France
- 2. SPEC, CEA, CNRS, Université Paris-Saclay, Gif-sur-Yvette, France
- 3. Laboratoire Charles Coulomb, Université de Montpellier and CNRS, Montpellier, France
- 4. Université Paris-Saclay, CentraleSupélec, CNRS, Laboratoire SPMS, 91190, Gif-sur-Yvette, France
- 5. Synchrotron SOLEIL, Gif-sur-Yvette, France

Topological solitons have been widely investigated in ferromagnetic materials over the last fifteen years, as exemplified by skyrmions. Similar objects are now emerging in ferroelectric materials with potential advantages of low-energy electric-field control and small sizes. In antiferromagnetic materials, skyrmions are foreseen as the ultimate goal for spintronics due to their intrinsic ultrafast THz dynamics and high velocities along the driving forces, but they remain to be demonstrated. As antiferromagnets are insensitive to external magnetic fields, one must find alternative ways to control them. In the TATOO project, we use magnetoelectric antiferromagnetic multiferroics to promote a low energy electric-field control of antiferromagnetism. We take advantage of the room-temperature magnetoelectric coupling in multiferroic BiFeO₃ to deterministically control antiferromagnetic spin textures via the ferroelectric domains. We first show that the surface of single crystals shows an unexpected continuous rotation of the antiferromagnetic cycloid propagation vector with the presence of antiferromagnetic topological defects [1]. Using anisotropic strain in epitaxial thin films, we design model systems containing a single ferroelectric domain coupled to a single antiferromagnetic domain [2], opening further opportunities for investigations of the interplay between non-collinear antiferromagnetic orders and magnon excitations. In standard striped ferroelectric domains in BiFeO₃ thin films, we use epitaxial strain to finely tune the asgrown spin textures going from cycloidal to collinear states [3, 4]. Finally, in submicron devices based on BiFeO₃, we stabilize topological center polar states using a radial electric field. We show that such polar textures can contain flux closure of antiferromagnetic spin cycloids or quadrant of canted antiferromagnetic domains, depending on the epitaxial strain [5]. These results open the way for electrically-reconfigurable antiferromagnetic topological objects.

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Acknowledgement:

We are thankful for support from the French Agence Nationale de la Recherche (ANR) through the project TATOO (ANR-21-CE09-0033)

CE09 - Nanomaterials and nanotechnologies for the products of the future
 CE42 - Sensors, instrumentation

Project acronym: THz-MUFINS

Funding instrument (JCJC, PRC, PRCE, PRCI): PRC Keywords (max 4): THz photoacoustics, THz emission, ferroelectrics, domains engineering.

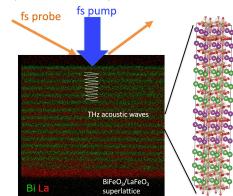
THz dynamics in MUltiFerrolc Nanostructures and Superlattices

A. Poirier¹, R. Gu¹, G. Vaudel¹, M. C. Weber¹, R. Xu², F. Delodovici², C. Paillard^{3,2}, B. Dkhil², L. Bellaiche³, V. Garcia⁴, S. Fusil⁴, M. Khiary⁵, C. Laulhé⁶, L. Yedra⁷, H. Bouyanfif⁵, V. Juvé¹, P. Ruello¹

- 1. Institut des Molécules et Matériaux du Mans, UMR 6283 CNRS, Le Mans Université, 72085 Le Mans, France
- 2. Laboratoire Structures, Propriétés et Modélisation des Solides, CentraleSupelec, UMR CNRS 8580, Université Paris-Saclay, 91190 Gif-sur-Yvette, France
- 3. Smart Ferroic Materials Center, Institute for Nanoscience and Engineering and Department of Physics, University of Arkansas, Fayetteville Arkansas 72701, USA
- 4. Laboratoire Albert Fert (UMR137) CNRS, Thales, Université Paris-Saclay
- 5. Laboratoire Physique de la Matière Condensée, Université Picardie-Jules Vernes, Amiens, France
- 6. Laboratoire de Physique des Solides, CNRS UMR 8502, Université Paris Saclay, 91405 Orsay, France
- 7. Laboratory of Electron Nanoscopies (LENS) MIND Department of Electronics and Biomedical Engineering & Institute of Nanoscience and Nanotechnology (IN2UB), University of Barcelona, Barcelona, Spain

In this project, we have investigated the light-induced THz dynamics in BiFeO₃ ferroelectric-based nanostructures by combining experimental [1-3] and theoretical methods [4,5]. The ferroelectric nanostructures have been grown with state-of-the-art pulse laser deposition techniques [1-3].

First of all, different strategies were validated to light-induce sub-THz-THz acoustic waves in BiFeO₃ thin films and superlattices [1,2,4]. We also demonstrated that, submitted to an ultrafast visible optical excitation, these ferroelectric nanostructures can emit THz radiation. In both cases, we have shown that we can tune the spectrum and polarisation of sub-THz-THz acoustic waves and THz radiation by tuning the ferroelectric orders thanks to different controlled parameters (domains, superorders, superlattices, screening) [3,5]. These original results pave the way for the development of sub-THz-THz acoustic transducers and open new avenues for the engineering of THz sources.



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Fig 1 : sketch of light-induced THz acoustic waves in ferroelectric superlattices

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Acknowledgement:

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agence nationale de la recherche

X CE09 - Nanomaterials and nanotechnologies for the products of the future□ CE42 - Sensors, instrumentation

Project acronym: SASHA Funding instrument (JCJC, PRC, PRCE, PRCI): JCJC Keywords (max 4): nanomechanics, nanoparticles, surface state

SASHA: SurfAce State and mecHanics of nAno-objects

Jonathan Amodeo¹

1. Aix-Marseille Université, Université de Toulon, CNRS, IM2NP, Marseille, F-13013, France

When compared to their bulk counterparts, nano-objects are known to benefit of particularly augmented mechanical properties related to their high surface/volume ratio. Nevertheless, this "smaller is stronger" trend is characterized by severe data scattering, merely induced by stress concentration during loading, that impedes quantitative prediction. This behavior cannot be predicted by the current modeling methodology that calls for improper sample geometry, increasing the gap between simulations and experiments. SASHA aimed at the development and application of a new nano-by-design methodology to better characterize the mechanical properties of nano-objects (e.g., nanoparticles, nanowires) with more realistic shapes [1,2]. For the very first time, nano-objects with optimized and realistic surfaces, including surface roughness and chemical alteration (oxidation), were designed and probed within molecular dynamics and finite-element simulations, via a statistical and quantitative approach [3]. This modeling study was carried in adequacy with top-of-the-art experiments. The realistic description of nano-objects surfaces is very critical and might bring a new paradigm in the understanding of mechanical properties at the nanoscale.

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 \Box CE09 - Nanomaterials and nanotechnologies for the products of the future \boxtimes CE42 - Sensors, instrumentation

Project acronym: IRMA

Funding instrument (JCJC, PRC, PRCE, PRCI): PRCI Keywords (max 4): nanostructured thin films; growth dynamics; optical and electrical properties; *in situ* and real-time characterizations

Intelligent real-time manipulation of metal nanostructure growth

D. Babonneau¹, G. Abadias¹, A Michel¹, F. Pailloux¹, K. Solanki¹, A. Coati², Y. Garreau^{2,3}, A. Resta², A. Vlad², B. Voisin², M. Kamiński⁴, B. Krause⁴

- 1. Institut Pprime, Dpt. Physique et Mécanique des Matériaux, CNRS, Université de Poitiers, Poitiers, France
- 2. Synchrotron SOLEIL, Saint-Aubin, France
- 3. Laboratoire Matériaux et Phénomènes Quantiques, Université Paris Cité, CNRS, Paris, France
- 4. Karlsruhe Institute of Technology, Institute of Photon Science and Synchrotron Radiation, Karlsruhe, Germany

Ultrathin silver films with thickness below a few nanometers are interesting candidates for use in various applications (*e.g.*, flexible electronics and low-emissivity glazing). However, early growth stages of Ag films deposited by magnetron sputtering on weakly interacting substrates are dominated by a natural tendency to form disconnected 3D islands, which manifests itself in high electrical resistivities and broad absorption bands in the visible range. Growth strategies to produce continuous and ultrathin Ag films without compromising their electrical conductivity and optical transparency have lately been deployed. Among them, the use of gaseous additives or buffer layers appears to be an effective means of promoting wetting of Ag on the substrate surface [1-3].

Overall, there is a need for a thorough understanding of the nanoscale mechanisms of thin film formation, which requires implementation of real-time techniques during growth [1,4,5]. In particular, the widely used *ex situ* diagnostics can provide misleading information, as the films evolve even under high vacuum conditions. In the present work, we study nitrogen-mediated growth of Ag on SiO_x and SiN_x surfaces as well as growth of Ag on amorphous Ge seed layers. We employ a simultaneous combination of real-time techniques during Ag deposition by magnetron sputtering, including grazing incidence small-angle x-ray scattering (GISAXS), grazing incidence diffraction (GID), substrate curvature measurements, and surface differential reflectance spectroscopy (SDRS). In particular, GISAXS reveals changes in nanoscale morphology, GID gives insight into the crystallinity of thin films, while substrate curvature measurements and SDRS provide information about the average intrinsic stress and optical properties, respectively. Using the information from all four techniques, we will discuss the impact of nitrogen additive and of buffer layers from the very first stages of growth (island nucleation, growth,

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and coalescence) up to formation of percolated and continuous films, including the evolution of the film after growth interruptions.

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Acknowledgement:

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☑ CE09 - Nanomaterials and nanotechnologies for the products of the future □ CE42 - Sensors, instrumentation

Project acronym: NOUS

Funding instrument (JCJC, PRC, PRCE, PRCI): PRC Keywords (max 4): Thin films, interfaces, 2D oxides, quasicrystals.

New ultrathin oxide films on metal substrates

C. Ruano Merchan^{1,3}, V. K. Singh^{1,3}, T. T. Dorini^{1,3}, F. Brix^{1,3}, W. Ma^{1,3}, C. Chatelier^{1,3}, L. Pasquier^{1,3}, M. Jullien^{1,3}, D. Pierre^{1,3}, S. Andrieu^{1,3}, K. Dumesnil^{1,3}, S. S. Parapari^{2,3}, S. Sturm^{2,3}, J. Ledieu^{1,3}, M. Sicot^{1,3}, O. Copie^{1,3}, E. Gaudry^{1,3}, V. Fournée^{1,3}.

- 1. Institut Jean Lamour UMR 7198, Université de Lorraine CNRS, Nancy, France.
- 2. Jožef Stefan Institute, Jamova Cesta 39, Ljubljana 1000, Slovenia
- 3. International Research Project Materiomics, CNRS-Université de Lorraine, Nancy, France and Jožef Stefan Institute, Ljubljana, Slovenia

Abstract (no longer than 300 words or 20 lines max. incl. figure), Calibri 11, single line spacing, black)

Two-dimensional oxide quasicrystals have been discovered in Ba-Ti-O [1] and Sr-Ti-O [2] ultra-thin films supported on hexagonally close-packed metal substrates. In the NOUS project, an all-thin-film approach has been adopted where the metal single crystal was replaced by a 10 nm thick Pt-(111) buffer layer grown on an Al₂O₃(0001) substrate by molecular beam epitaxy, whereas various perovskite oxide thin films were grown by pulsed laser deposition [3]. Several new 2D oxide phases have been discovered, including a new square approximant with a giant unit cell as well as a hexagonal phase with a large surface unit cell. A model has been proposed to describe these phases based on DFT calculations, with a broad applicability [4]. A continuous transformation of such Sr-Ti-O quasicrystalline approximant thin films into Sr decorated Ti₂O₃ honeycomb lattices upon annealing under ultra-high vacuum conditions has been observed [5]. These results are consistent with a model describing the transformation of two-dimensional quasicrystals/approximants based on square-triangle-rhombus tilings into Sr-decorated Ti₂O₃ honeycomb low-energy defects identified as Stone–Wales transformations, typical of hexagonal 2D materials. Other systems were also investigated, including SrRuO₃ and DyVO₃ oxide films supported on metal upon ultra-high vacuum thermal treatments. The presentation will propose a summary of these results.

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 C. Ruano Merchan, T. T. Dorini, F. Brix, L. Pasquier, M. Jullien, D. Pierre, S. Andrieu, K. Dumesnil, S. S. Parapari, S. Sturm, J. Ledieu, M. Sicot, O. Copie, E. Gaudry, V. Fournée, Phys. Chem. Chem. Phys. 24, 7253-7263 (2022).

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X CE09 - Nanomaterials and nanotechnologies for the products of the future CE42 - Sensors, instrumentation

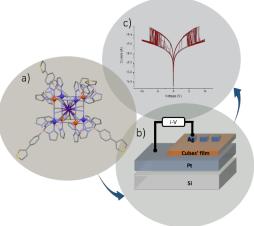
Project acronym: Cub'ic Funding instrument (JCJC, PRC, PRCE, PRCI): PRCE Keywords : ultra-thin film; molecular switch; memristor

Electronically-Active Thin Films for New Concept of Nano-Devices

Sofia Russi¹, Thomas Petenzi², Amina Benchohra¹, Camille Jube², Laure Fillaud², Rodrigue Lescouëzec¹, J. Landoulsi³, Gabor Molnar⁴, Volodymyr Malytskyi,¹ Yanling Li¹, Lise-Marie Chamoreau¹, Emmanuel Maisonhaute¹⁻², Candice Botuha¹, David Kreher¹, Christophe Méthivier,² Azzedine Bousseksou⁴

- 1. Sorbonne Université, Institut Parisien de Chimie Moléculaire, UMR 8232, Paris, France
- 2. Sorbonne Université, Laboratoire Interfaces et Systèmes Electrochimiques, UMR 8235, Paris, France
- 3. Sorbonne Université, Laboratoire de Réactivité de Surfaces, UMR 7197, 4 place Jussieu, Paris, France
- 4. CNRS Toulouse, Laboratoire de Chimie de Coordination, UPR 8241, Toulouse, France

The deposition of molecular magnetic switches onto surface is actively investigated with the aim of designing original electronic devices that would have no counterpart in the current Si-based technology. In this work, we took profit of the remarkable solution stability of a recently designed family of dual redox/magnetic cubic switches,¹⁻² to propose a soft chemistry approach for the deposition



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- a) Cube structure before polymerization.
- b) Device configuration. c) i-V curve.

of these dual switches directly onto conductive substrates. Our goals were: (i) to check in which conditions the switchable properties could be maintained once the molecules are processed as ultrathin films; (ii) to investigate the transport properties through these thin-films in two-electrode device configuration. To these ends, we thus developed a simple strategy based on electro-polymerization of functional cubic units bearing thiophene or EDOT peripheral groups.³ This has permitted the growth of thin-films with controlled thickness (2-30 nm). By investigating these nanomaterials using different techniques (CV, AFM, XPS and XAS), we demonstrated the stability of the cubic molecules after deposition and we showed in which conditions the photo magnetic and multi-level redox switching

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could be maintained. Finally, a top electrode was deposited to measure the electric activity of the film, which showed memristive-like effect, reminiscent of that of molecular films.⁴

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☑ CE09 - Nanomaterials and nanotechnologies for the products of the future □ CE42 - Sensors, instrumentation

Project acronym: DEFINE2D

Funding instrument : PRCI

Keywords : 2D materials, defect engineering, scanning tunneling microscopy

Defect engineering in 2D materials

Jérôme Lagoute¹, Cyril Chacon¹, Yann Girard¹, Yannick Dappe², Alexander Smogunov², Wei Bin Su³, Woei Wu Pai⁴

- 1. Laboratoire Matériaux et Phénomènes Quantiques (MPQ), Université Paris Cité, CNRS, Paris, France
- 2. SPEC, CEA, CNRS, Université Paris-Saclay, CEA Saclay, France
- 3. Institute of Physics, Academia Sinica, Taïwan
- 4. Center for Condensed Matter (CCMS), National Taïwan University (NTU), Taipei, Taïwan

The DEFINE2D project explores defect engineering in 2D materials like graphene, black phosphorus, and transition metal dichalcogenides TMDs. By introducing defects such as heteroatoms, intercalation, and domain boundaries, we aim to tailor their properties for applications in nanoelectronics and catalysis through defect engineering. We combine scanning tunneling microscopy and spectroscopy (STM/STS) with ab initio calculations to reveal the impact of the generated defects on the properties of the materials at the atomic scale. We combined molecular layer deposition and nitrogen doping to realize band engineering and reveal the profile of pn junctions in graphene at the atomic level [1]. In black phosphorus, we identified the nature of native point defects which was under debate in the literature and realized their local manipulation with the STM tip [2]. We investigated metallic TMDs exhibiting charge density waves (CDW). We showed that the intercalation of alkali atoms in bulk VSe₂ leads to the transition of the CDW from the bulk phase to the monolayer phase [3]. More recently we revealed the possibility to realize and observe all elementary excitations of CDWs in VTe₂ that can switch from a 4x4 phase to 4x1 phase, and that can rotate or slide under the local excitation by the STM tip [4]. We are now increasing the complexity of defect engineering by combining two type of atomic scale point defects, namely vacancies and substitutional nitrogen, in graphite allowing to control the energy position of electronic resonant states which can be used to reach different chemical functionalities [5].

This project has bring new knowledge on defect engineering in 2D materials, and showed how electronic band engineering, chemical functionality, electronic phase manipulation can be realized in these materials.

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Acknowledgement:

ANR-20-CE09-0023

□ CE09 - Nanomaterials and nanotechnologies for the products of the future

Project acronym: MeMeNtO Funding instrument: JCJC Keywords (max 4): Nanocube assembly, plasmonic metasurfaces, nanocube epitaxy

Designer metasurfaces from colloidal building blocks

Beniamino Sciacca¹

1. Aix-Marseille Univ., CNRS, CINaM

Abstract

High-quality monocrystalline materials and nanostructures are key to high-efficiency optoelectronic devices, plasmonic materials and metasurfaces. A large variety of materials can be synthesized at low temperature in solution as colloidal single crystals, combining the advantages of high-quality material and low-cost fabrication, but the potential for large area integration into nanophotonic and plasmonic devices via nanoparticle assembly techniques still remain elusive. The two major bottleneck are:

1) Difficulty to achieve defect-free colloids self-assembly in truly arbitrary patterns

2) Sub-optimal optical and electronic properties due to the discreetness of individual building blocks.

I will present recent breakthroughs from our team tackling both challenges with a new approach that we have introduced based on nanocube epitaxy. In the first part I will focus on advances on directed-assembly with detailed insight into the mechanisms. I will show the fabrication of complex and truly arbitrary organisation of nanocubes on a large surface, with a single nanocube resolution. Examples includes 1D linear assembly [1], 2D nanocube grid arrays for transparent electrodes [2], highly dense split-ring resonator absorbers and Pancharatnam–Berry metasurfaces [3].

Next, I will focus on nanocube epitaxy, showing a general methodology to transform an arbitrary assembly of metal nanocubes into defect-free continuous nanostructures and *operando* mechanistic insights [4, 5]. This approach enables to obtain monocrystalline plasmonic and nanophotonic surfaces that can be readily printed on any substrate, but also to make nanocrystals with unconventional (à *la carte*) geometries, to be used as colloids beyond Platonic solids.

Finally, I will show optical characterisation of nanocube-enabled optical metasurfaces.

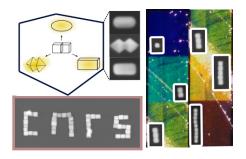
I believe that this constitute a major advancement in the field, can inspire many scientists and lead the way for a new generation of bottom-up metasurfaces and their spread in industrially-relevant products.

References (max. 5):

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2D nanolaminate - nanofluidic ionic diodes hybrid membranes for desalination and water purification (2D-MEMBA)

Damien Voiry¹, Sébastien Balme¹, Chrystelle Salameh¹, Carlos Drummond², Jean-Christophe Gabriel³

- 1. Institut Européen des Membranes, IEM, UMR 5635, Université Montpellier, ENSCM, CNRS, Montpellier 34000, France
- 2. Centre Paul Pascal, UMR 5031, CNRS, avenue Schweitzer, 33600 Pessac, France
- 3. LICSEN, NIMBE, UMR CEA/CNRS 3685, Université Paris-Saclay, Gif-sur-Yvette, 91191 France

Abstract:

The limitations of conventional polymer membranes, dictated by the permeability-rejection trade-off, have driven research into next-generation separation technologies [1]. Among emerging solutions, twodimensional (2D) materials have attracted significant interest due to their large lateral surface areas, high charge surface densities, and abundant active sites, making them ideal for constructing laminar membranes with sub-nanometer channels [2]. These channels offer unique opportunities for nanofluidic studies and precise molecular sieving.

In 2D-MEMBA, we explore an innovative hybrid membrane design combining 2D materials (graphene and exfoliated MoS_2 nanosheets, ionic nanosheets: V_2O_5 , $H_3Sb_3P_2O_{14}$) with track-etched polyethylene terephthalate (PET) membranes, which function as ionic diodes, or PVDF commercial substrates. The PET membranes are fabricated through a two-step process: heavy swift ion irradiation with a control of latent track density, followed by chemical etching to create asymmetric, bullet-shaped pores that induce non-linear ionic current responses [3]. The resulting hybrid membranes integrate the high selectivity of 2D nanolaminates with the tunable nanofluidic behavior of track-etched membranes.

These hybrid membranes were evaluated under forward osmosis for salt and nanopollutant separation, while I-V characteristics were recorded in various electrolyte solutions. Results demonstrate water fluxes as high as 10 L m⁻² h⁻¹ with precise molecular cut-off below 200 Da for the nanolaminated membranes, confirming the efficacy of the nanolaminate structure for fast and precise molecular sieving. Additionally, preliminary findings on the hybrid structure points out to a synergistic effect between the nanolaminate structure and the track-etched support.

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Our presentation will highlight the potential of nanolaminated hybrid membranes as versatile platforms for water purification, metal cations separation and nanofluidic research, paving the way for sustainable and efficient separation technologies[4,5,6].

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