

D'ÉLABORATION STRUCTURALES

HIGHLIGHTS 2021





CENTRE D'ÉLABORATION DE MATÉRIAUX ET D'ÉTUDES STRUCTURALES

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We are very happy to present you the sixth edition of our yearly brochure describing the significant happenings in the year 2021, which now has a new look. As you can see, the principal change from previous editions is the decision of the lab to change to a new logo, which, while giving a more modern look than its predecessor, is still inspired by the famous "boule" that epitomizes our lab. We hope that you will like this new logo and that it will project a more attractive first impression of our laboratory.

Despite the pandemic, the year 2021 has had its fair share of important happenings. Etienne Snoeck, at whose initiative this compilation of CEMES highlights was started, has decided not to continue as the head of the laboratory. CEMES is now headed by our trio, that has greater female representation in addition to being younger. At this point, we take this opportunity to thank Etienne for all the work that he has accomplished.

Our lab has participated in a number of dissemination activities targeting the general public, such as "Velotour" or "Open Day". Activities specially designed for students include "Club CEMES" in Berthelot middle school, training of "research apprentices" and lastly, the program "DECLIC". These three actions have already been described in greater detail in the section on CEMES' support to secondary education. Several members of the lab have taken part in such actions, for example, more than 40 participated this last year. All of them have been unstinting in volunteering their time and efforts to support these actions. We would like to thank each and every one of them; without their support, none of this would have been possible.

With the aim to limit the environmental impact of its activities, CEMES is also committed to sustainable development while maintaining cutting-edge scientific research at the highest level.

Regarding scientific and technological activities, the laboratory has successfully carried out many projects—some of them have been illustrated elsewhere in this brochure. In these projects, we combined fundamental researches, industry-oriented approaches and technological advances, which have contributed to building the knowledge base and expertise of the laboratory. We congratulate the leaders of the 10 projects that have been selected for funding by ANR in 2021 and the successful selection for funding of the European project ESiM on energy storage in molecules. Last but not least, we congratulate the teams that have successfully obtained funding from industry in the form of contracts.

We hope you enjoy reading this brochure.

Alain Couret Director, CEMES

Bénédicte Warot-Fonrose Associate director CEMES

Muriel Rougalle Chief Administrative Officer, CEMES



Nous sommes très heureux de vous présenter la sixième édition des faits marquants 2021 du CEMES, qui présente un nouveau visage. Comme vous pouvez le constater, ce changement accompagne le choix du laboratoire de se doter d'un nouveau logo, plus moderne mais toujours inspiré par notre célèbre Boule. Nous espérons que vous l'apprécierez et qu'il donnera une première image attrayante du laboratoire.

Malgré la pandémie, de nombreux évènements ont marqué cette année 2021. Etienne Snoeck, d'ailleurs à l'initiative de ce recueil des « CEMES Highlights », a choisi de ne pas poursuivre son action à la tête du laboratoire. Il a donc laissé la place à notre trio, féminisé et rajeuni. C'est pour nous l'occasion de remercier chaleureusement Etienne de tout le travail qu'il a accompli.

Le laboratoire a participé à plusieurs actions d'ouverture vers le grand public, comme Vélotour ou les journées portes-ouvertes, et vers les scolaires, comme le club CEMES au collège Berthelot ou les dispositifs Apprentis-chercheurs et Déclics, qui vous sont présentés dans ce recueil. Ces actions mobilisent de nombreux personnels du laboratoire, plus d'une quarantaine cette année. Tous donnent de leur temps sans compter. Qu'ils en soient tous remerciés ; sans eux, rien n'aurait été possible.

Le CEMES s'est aussi inscrit dans une démarche de Laboratoire Durable, soucieux de limiter l'empreinte environnementale de notre recherche, tout en la maintenant au meilleur niveau.

Sur les plans scientifiques et techniques, le laboratoire a connu de nombreux succès illustrés pour partie dans ce recueil. Recherches fondamentales, activités plus orientées vers les applications et prouesses techniques s'y mêlent allègrement et font la richesse du laboratoire. Nous nous félicitons de la sélection de dix projets ANR en 2021 et de celle du nouveau projet Européen ESiM sur le stockage de l'énergie dans des molécules, sans oublier les nombreux contrats avec le monde industriel.

Nous vous souhaitons une bonne lecture de ces Faits Marquants 2021 du CEMES.

Alain Couret Directeur du CEMES

Bénédicte Warot-Fonrose Directrice Adjointe du CEMES

Muriel Rougalle Secrétaire Générale du CEMES

A RED STAR IS BORN

A LARGE STARPHENE COMPRISING PENTACENE BRANCHES

Starphenes, planar molecules comprising three acenes branches, show promising optoelectronic properties. Notably, HO-MO-LUMO energy gap presumably decreases with their increasing size, making larger homologues extremely desirable. Unfortunately, with the size, their instability and insolubility also increase, challenging classical synthetic ways. A new method has been developed, enabling synthesis of large starphenes in a quantity sufficient for their full characterization and further applications.

Researchers from the NanoSciences Group at CEMES have been able to solve this problem by designing chemically stable, non-planar and soluble precursors which allows their synthesis and characterization by classical methods of organic chemistry (in fifteen synthesis steps in total). These precursors can be converted to starphene simply by heating either in the solid phase or on an ultra-high vacuum surface or by irradiating in ultra-violet in a solvent frozen at a few degrees Kelvin. The electronic structure and optical properties of these molecules have been studied in collaboration with teams of microscopists from the DIPC in San Sebastian (Spain), photophysicists from the Academy of Sciences in Warsaw (Poland) and a theorist from the university from Hokkaido (Japan).

A Large Starphene Comprising Pentacene Branches

Jan Holec, Beatrice Cogliati, James Lawrence, Alejandro Berdonces-Layunta, Pablo Herrero, Yuuya Nagata, Marzena Banasiewicz, Boleslaw Kozankiewicz, Martina Corso, Dimas G. de Oteyza, Andrej Jancarik and Andre Gourdon.

Angew. Chem.Int. Ed.2021.60.7752-7758



Large starphenes can be accessed by thermal decarbonylation in the solid state, on a surface in ultravacuum or by UV irradiation in frozen matrices of tricarbonylated precursors (on the left, scheme of one of the precursors). On the right, the image of a single [16]starphene by Scanning Tunneling Microscopy.



(a) High-Resolution Scanning Tunneling Microscopy image (tip functionalized with a CO Molecule of a starphene). (b) dl/dV point spectra on starphene showing various resonances marked with dashed lines. The inset marks the positions at which each of the spectra are taken, two on the molecule and one on the substrate as reference. (c) and (d) Constant height dl/dV images of a starphene recorded with a Cl tip at the energy of the lowest unoccupied and highest occupied states, respectively. (e) and (f) Calculated wavefunctions of the HOMO and LUMO of free-standing starphene.

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GEARING UP NANOSCALE MACHINES

SYNTHESIS OF CHEMICALLY-LABELLED 5 NM LARGE COGWHEELS AS GEAR ELEMENTARY UNITS

Researchers from CEMES-CNRS, University Paul Sabatier and Nara Institute of Science and Technology, Japan, designed and synthesized nanoscale cogwheels that will enable visualization of rotational snapshots by scanning tunneling microscopy. Such advances are essential for monitoring and confirming the operation of intermolecular gear trains at work.

Gear systems are essential elementary mechanical units allowing the transmission of information over long distances and the conversion of the motive power of a motor through synchronized disrotatory motion of the cogwheels. They can also translate the changes in gear rotational speed into changes in rotational force. Researchers at CEMES-CNRS in partnership with research teams at University Paul Sabatier and Nara Institute of Science and Technology (NAIST), Japan, report in a new study published in Chemical Science a strategy to visualize snapshots of an ultrasmall gear train-an interconnected chain of molecular-scale gears-at work.

Mastering intermolecular gearing is crucial for the emergence of complex functional nanoscale machineries. However, achieving correlated motion within trains of molecular gears remains highly challenging. The most straightforward way to monitor the motion of molecular gears adsorbed on a surface is through static scanning tunneling microscopy images. For these purposes, one of the teeth of the cogwheel subunits has been labelled through chemical modification. The teeth are made from porphyrins, a fragment already used in Nature for many processes like oxygen transportation (hemoglobin) or photosynthesis (chlorophyl). The electronic properties of one single tooth were modulated by varying the porphyrinic substituents or the nature of the central metal (nickel or zinc). Such tailored properties are important because one can observe the labelled teeth as differences in submolecular contrast by using scanning tunneling microscopy, thus facilitating imaging of cogwheels and monitoring of their rotation. The pentaporphyrinic cogwheel prototypes are very complex molecules made up of at least 824 atoms (chemical formula C391H395BN28Ni5S-3Ru) linked together in a very controlled manner. With their unique design and complexified chemical structure, these cogwheels will be used to build trains of gears on a metallic surface. In this arrangement, it will be possible to rotate a series of cogwheels in a coordinated way as in a conventional macroscopic gear train with a means to visualize such rotations.



S. Abid, Y. Gisbert, M. Kojima, N. Saffon-Merceron, J. Cuny, C. Kammerer, G. Rapenne.

Chem. Sci., 2021, 12, 4709

gears remains highly challenging.

Cogwheel molecule as elementary unit of intermolecular gears on surface. The top arm (in red) has a different chemical structure to follow the rotation.

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OPTICS NEAR SURFACESAND AT THE NANOMETER SCALE

THIS NEW BOOK EXPLORES THE PHYSICAL PHENOMENA UNDERLYING THE OPTICAL RESPONSES OF NANOSCALE SYSTEMS

This book explores the physical phenomena underlying the optical responses of nanoscale systems. Important aspects of wave optics on surfaces and at small scales are discussed, such as the optical interference near surfaces, how imaging optical fields can be used to enhance resolution, the consequences of the finite size of the focal spot and the physical origin of the index of refraction based on an ensemble of discrete scatterers.

The finite width of a beam incident on a reflecting surface creates a zone where the incident and reflected beams overlap. In this overlap zone, the two beams interfere, giving rise to a surface standing wave and we can take advantage of surface standing waves to enhance the optical response of adsorbed nanoparticles or molecules.

In chapter three we describe how interference effects can be exploited to improve image resolution in optical microscopy. Using an optical scanning probe in collection mode, lateral standing waves can be recorded at variable distances from the surface. We then show how an intermediate field region outside the near field region can be defined. In the intermediate field region, amplitude variations across the image are considerable at distances smaller than five times the wavelength of the incident beam. This enables a smooth transition between indirect (diffraction) and direct images. Furthermore, we explain how the image of the particle can be reconstructed. Resolution can be as high as that observed in near field optics, with the added advantage that no feedback signal between the probe and surface is required.

Chapter four considers the consequence of the finite size of the focal spot in optical spectroscopy. Changing the position of the nanotube within the focal spot leads to spectral shifts when displacing in a particular direction but not in the direction perpendicular to it. This means that when using a grating spectrometer, apparent shifts in spectral positions can occur if the nano emitters are smaller than the size of the focal spot. The associated spectral shifts are small but cannot be ignored when studying strain or doping induced effects in optical spectra of nano-objects.

The final chapter goes into details of how the index of refraction can be related to scattering by an ensemble of discrete scatterers. We start by considering a plane of scatterers after which we introduce the third dimension. We then go on to show how the phase shift is related to the finite speed of light and the physical dimension of the interacting medium.

Optics Near Surfaces and at the Nanometer Scale

Bacsa, Wolfgang, Bacsa, Revathi, Myers, Tim.

Springer Briefs in Physics ISSN: 2191-5423



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SINGLE ORGANIC MOLECULES FOR PHOTONIC QUANTUM TECHNOLOGIES

ORGANIC MOLECULES UNVEIL A BRIGHTER FUTURE FOR QUANTUM TECHNOLOGY!!

The control of light emission by single molecules diluted in solid organic matrices allows for the fabrication of quantum photonic devices such as sensors, transducers, and elements for optical quantum computing.

Beyond fundamental physics, quantum mechanics is nowaday a fertile ground for breakthrough technologies in information science and high precision measurements, evidenced by the huge investments being made across the globe in this area. But what are the ideal material platforms providing such technologies? This is still an open question for both private and public investors, while also for researchers and engineers.

In this review article published in Nature Materials, we discuss the prominent impact of organic molecules in quantum technologies as a result of over 20 years of active investigation. This work, authored by 16 scientists from 7 different countries, analyses the close synergy between molecules and photons. The recent advances in controlling this interaction at the level of single quanta will be the key transduction mechanism for optically reading the charge of a single electron or a single quantum of mechanical vibration. Light generated by single organic molecules can carry intrinsically secure information, one photon at a time, propagating unperturbed over long distances. Considering the extreme versatility and scalability of organic synthesis, which can provide, at low costs, a wide palette of properties, a new flexible way of building hybrid quantum devices is possible. The authors envision small hybrid optical networks on microchips that carry light instead of electricity, where photons and molecules together can perform certain tasks that are computationally hard for classical machines.

Single organic molecules for photonic quantum technologies

C. Toninelli, I. Gerhardt, A. S. Clark, A. Reserbat-Plantey, S. Götzinger, Z. Ristanović, M. Colautti, P. Lombardi, K. D. Major, I. Deperasińska, W. H. Pernice, F. H. L. Koppens, B. Kozankiewicz, A. Gourdon, V. Sandoghdar and M. Orrit.

Nature Materials 20, 1615-1628 (2021)



Single organic fluorophores can be used as transducers in sensors, single photon sources for communication, and non-linear elements in optical computing.

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NANO-SILICON ANTENNAS TO CAPTURE INFRARED

ORDERED ARRAYS OF DOPED SILICON NANODISKS EXTEND PLASMONICS TO INFRARED

The appearance of localized surface plasmon resonances in ordered arrays of doped silicon nanodisks allows plasmonics to be extended to the infrared range. The resonance frequency is simply adjusted with the free charge concentration given by dopants. Up to now restricted to noble metals, plasmonic antennas are now made of silicon, a non-toxic and abundant material. These results are essential for the detection of molecules or thermal imaging.

Plasmonics, using the collective oscillations of free electrons located in metallic nanostructures (plasmon resonances) allows light to be manipulated at a much smaller scale than its wavelength, down to nanometric dimensions. This opens the field towards nano-optics, making it possible to bridge the dimensional gap between electronic and optical devices.

In a study, researchers from CEMES associated with colleagues from LAAS and in collaboration with CEA-LETI, have produced for the first time a plasmonic antenna array made up of nano objects in doped silicon, a material non toxic and abundant of the semiconductor family, at the base of microelectronics. With their extensive knowledge in materials science, nanotechnology and infrared optics, they have used a top-down approach consisting first in optimizing the doping of thin silicon on insulator (here silica) layers beyond the usual doses by means of pulsed laser thermal annealing. Then, dense hexagonal arrays of identical disks of nanometric sizes are formed by electron lithography etching. These doped nanostructures exhibit intense localized surface plasmon resonances, measured from the medium to the near infrared by Fourier Transform Infrared Spectroscopy. This wide spectral window can be covered by simply tuning the free carrier concentration, hence the active phosphorus dopant concentration.

Numerical simulations have made it possible to study the optical properties of a single nanodisk as well as the metasurface, thus identifying the collective effects and near-field coupling in the metasurface. These results open up very promising prospects for the development of all-silicon integrated plasmonic devices targeting applications requiring broadband and high-resolution infrared detection, such as micro-integrated bolometers or miniaturized infrared cameras.

Hyper-Doped Silicon Nanoantennas and Metasurfaces for Tunable Infrared Plasmonics

Jean-Marie Poumirol, Clément Majorel, Nicolas Chery, Christian Girard, Peter R. Wiecha, Nicolas Mallet, Richard Monflier, Guilhem Larrieu, Filadelfo Cristiano, Anne-Sophie Royet, Pablo Acosta Alba, Sébastien Kerdiles, Vincent Paillard and Caroline Bonafos.

ACS Photonics 2021, 8, 5, 1393–1399



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BIOINSPIRED TIME-TEMPERATURE INDICATORS

BEETLES, LIQUID CRYSTALS AND SMART LABELS

Researchers from CEMES-CNRS have manufactured structural-color-based, smart labels that record the history of the product conservation, for example a vaccine. A qualitative information reports that the product was kept outside of the specified storage temperature, and a quantitative information gives an indication of the time elapsed since then.

The twisted structures of the chitin-based carapace of insects confer on them specific optical characteristics. Intrigued by the observation of Bragg gratings with a depth-dependent periodicity in the carapace of Chrysina beetles, researchers from CEMES-CNRS have determined the experimental conditions leading to their transcription into cholesteric liquid crystal oligomers. They correlate the optical properties of such reflectors with their internal morphology, as observed by transmission electron microscopy, performed at the Center for Integrative Biology in Toulouse (CNRS, University Paul-Sabatier). With the use of a single parameter, the thermal annealing time, the reflection color is made time-tunable. Different spectral bands, from golden yellow to near-infrared are available and irreversibility of the final color is reached. On the basis of the design concept and these properties, these hybrid chiral-achiral materials inspire the fabrication of smart reflective labels. When encapsulated in the package of a product to be kept in cold conditions, the label records the history of the product conservation. Two kinds of information based on color changes are recorded: qualitative information reporting that the product was kept outside of the specified storage temperature and quantitative information giving an indication of the time elapsed since then.

Bioinspired, Cholesteric Liquid-Crystal Reflectors with Time-ControlledCoexisting Chiral and Achiral Structures

Cécilia Boyon, Vanessa Soldan, and Michel Mitov.

ACS Appl. Mater. Interfaces 13, 30118 (2021)



0 20 40 60 80 100

NIC

100

100

Structural analysis of samples for different annealing times. Transmission electron microscopy transverse views accompanied by the half pitch (distance between two bright stripes) as a function of the normalized depth (local depth over the total thickness of the sample). Scale bar = 2 micrometers.

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CRYSTALLIZATION KINETICS OF NITROGEN DOPED Ge2Sb2Te5

REINFORCING THE NUCLEATION-DOMINATED CRYSTALLIZATION OF Ge₂Sb₂Te₅ ALLOYS BY N DOPING

N doping significantly improves some key characteristics of PCM devices, but the origin, at the atomic scale, of these alterations was elusive. Using a combination of ex-situ and in-situ TEM techniques, we show that the observed higher crystallization temperature and finer grain morphology result from the bonding of N with Ge which renders amorphous Ge₂Sb₂Te₅ more viscous and impedes Ge diffusion in both the amorphous and crystalline phases during annealing.

Researchers at CEMES-CNRS, supported by Léti/CEA engineers, have reported the results of a combination of in situ and ex situ transmission electron microscopy (TEM) investigations carried out on specifically designed samples to evidence the influence of N concentration on the crystallization kinetics and resulting morphology of the alloy. Beyond the known shift of the crystallization temperature and the observation of smaller grains, we show that N renders the crystallization process more "nucleation-dominated" and ascribe this characteristic to the increased viscosity of the amorphous state. This increased viscosity is linked to the mechanical rigidity and the reduced diffusivity resulting from the formation of Ge–N bonds in the amorphous phase. During thermal annealing, N hampers the coalescence of the crystalline grains and the cubic to hexagonal transition.

Making use of AbStrain, a TEM-based technique recently invented at CEMES, we evidence that the nanocrystals formed from the crystallization of N-doped amorphous Ge₂Sb₂Te₅ are under tension, which suggests that N is inserted in the lattice and explains why it is not found at grain boundaries. Globally, all these results demonstrate that the origin of the effect of N on the crystallization of Ge₂Sb₂Te₅ is not to be attributed to the formation of a secondary phase such as a nitride, but to the ability of N to bind to Ge in the amorphous and crystalline phases and to unbind and rebind with Ge along the diffusion path of this atomic species during annealing.



Left, Dark Field TEM image of the sample after annealing at 180°C for 30 min showing the difference in crystal sizes in the N implanted (top) and in the unimplanted (bottom) regions. Middle: differences in nucleation and growth probabilities in pure and N implanted GST-225. Right: Strain experienced by the Ge₂Sb₂Te₅ nanocrystals in the N implanted and unimplanted regions.

Effect of Nitrogen Doping on the Crystallization Kinetics of Ge₂Sb₂Te₅

Minh Anh Luong, Nikolay Cherkashin, Béatrice Pecassou, Chiara Sabbione, Frédéric Mazen and Alain Claverie.

Nanomaterials 2021,11,1729

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COMPLEX THREE-DIMENSIONAL MAGNETIC CONFIGURATIONS

A 3D STUDY AT THE NANOSCALE OF MULTI-VORTEX MAGNETIC STATES

CEMES researchers have studied the 3D magnetic configuration of longitudinal and transverse multi-vortex states of different chiralities in CoNi magnetic nanowires using a unique holographic vector-field electron tomography method. This study has revealed the complex distribution of the magnetization and highlighted an unexpected domain wall separating two vortex domains of opposite chirality.

Cylindrical magnetic nanowires in which the direction of easy magnetization is perpendicular to the wire axis (or close to), exhibit peculiar magnetic configurations resulting from the competition between the exchange and anisotropy (magnetocrystalline and shape) energies. In a collaboration with the IFW in Dresden, the University of Berlin, ICMM Madrid, and University del Valle de Cali, CEMES researchers used the unique holographic vector-field electron tomography (VFET) method to study the 3D magnetic configurations of hcp/fcc CoNi nanowires in the remanent state. The 3D structure of magnetic domains has been revealed with a spatial resolution of 10 nm. By tuning the applied magnetic field direction perpendicular or parallel to the wire axis before the observations, they could stabilize respectively a transverse-vortex chain with a zig-zag modulation of the vortex lines or a longitudinal vortex configuration separated by an unexpected chiral domain wall (CDW). This CDW exhibits a complex 3D shape characterized by the expulsion of the opposite vortex lines from the nanowire (see figure). It is demonstrated to reduce both the exchange and the anisotropy energies compared to conventional DWs. By combining the experimental results and the micromagnetic simulations carried out on both perfectly cylindrical nanowires, the "ideal" case, and of the real shape nanowire obtained experimentally by VFET, they demonstrated the impact of the crystalline properties and real shape real at the nanometric scale on resulting magnetic configurations of the nano-objects.

Field tunable threedimensional magnetic nanotextures in cobaltnickel nanowires

I. M. Andersen, D. Wolf, L. A. Rodriguez, A. Lubk, D. Oliveros, C. Bran, T. Niermann, U. K. Rößler, M. Vazquez, C. Gatel, and E. Snoeck.

Phys. Rev. Research 3 033085



3D reconstruction of the magnetic B-field inside CoNi NW by holographic VFET and comparison with micromagnetic simulation after magnetizing the NW in axial x-direction. The z-direction corresponds to the electron beam trajectory. Central slices in axial (xy) and (xz) direction through the 3D B-field inside the NW visualized by arrow-plots and color-coded volume rendering of Bx and By reveal a complex configuration in the hcp region with the appearance of Chiral Domain Walls (CDW).

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PyMoDAQ AN OPEN-SOURCE SOFTWARE FOR SIMPLE DATA ACQUISITION FROM COMPLEX EXPERIMENTAL SETUPS

PyMoDAQ, Modular Data Acquisition with Python is an open-source software for automated data acquisition as a function of various experimental parameters. Its generic graphical interfaces, that can be run without any programming skills, allow instrument controls while specific extensions make it very versatile.

Driving experimental setups and acquiring data is often made using either commercial software or home made ones. As the experiment evolves, adding controls to these solutions can be cumbersome and time consuming. Simple, open-source and free tools devoted to this task is therefore a must for the scientific community. Various projects, developed by scientific communities, are existing either focusing on a specific task or on the contrary to no predefined thematic application. PyMoDAQ has been developed with this second objective in mind but also to be as simple to use as possible. PyMoDAQ is opensource and written in Python language with a graphical interface allowing its use without any programming skills. It can be adapted to any application with the help of its modular structure and its various extensions. Its characteristics and comparison with other project have been published in Review of Scientific Instruments.

Born four years ago, PyMoDAQ allows now the control of any instruments using two generic interfaces: one for detectors (for instance to record signals from an oscilloscope or from a camera) and one for actuators (for instance to control experimental parameters such as applied current or laser polarization...). These interfaces are grouped together within a dashboard allowing their configuration (number, types, settings...) for a particular experimental setup. Several extensions are complementing the dashboard allowing for instance the automated data acquisition as a function of one or more variable parameters (DAQ_Scan).

The main interest of PyMoDAQ is its simplicity of use with its graphical interfaces while still being usable for any kind of experimental setup. PyMoDAQ aims to be a collaborative project giving access to the community of multiple base elements allowing the construction of other specific extensions. A broad range of instruments are already taking care of existing plugins and this number is increasing with the help of other contributors.

PyMoDAQ: An opensource Python-based software for Modular Data AcQuisition

S. J. Weber.

Rev. Sci. Instrum. 92, 045104 (2021)



PyMoDAQ's schematic structure showing control interfaces grouped within a dashboard and the existing extensions.

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TIME-RESOLVED NANOCATHODOLUMINESCENCE IN A TEM

A PIONEER WORK, RESULT OF A COLLABORATION BETWEEN RESEARCHERS FROM CEMES AND LPS-ORSAY

A collaboration between researchers from CEMES and LPS-Orsay has allowed for the first time to perform a time-resolved cathodoluminescence experiment in a transmission electron microscope. This work demonstrates the possibility to map the fluorescence lifetime of emitters with an unprecedented spatio-temporal resolution.

Light emission, also known as luminescence, provides access to valuable information about the physical properties and dynamics of atomic, molecular or excited solid systems. Among these properties, the lifetime of an excited state is of fundamental as well as applicative interest. Thus, its measurement has stimulated for decades the development of new instrumental developments. The techniques used have long been based on optical excitation of the sample. However, the limited spatial resolution of optical spectroscopies and the characteristic length scales of the main relaxation processes often below the optical wavelength have motivated the search for alternative strategies.

Historically, light emission measurements have been performed in scanning electron microscopes (SEM). However, despite the significant gains in spatial resolution com-

80 40 Sto 30 d) $T_1 = 23.4 \pm 0.7 \text{ ns}$ (us) 25 ifetime $T_2 = 15.8 \pm 0.8 \text{ ns}$ 20 20 40 60 80 100

pared to optical systems, SEM technologies are intrinsically limited, and do not allow, for example, to access atomic structural information in parallel with optical information.

This is what transmission electron microscopy (TEM) technologies allow. Collaboration between researchers from CEMES in Toulouse and LPS-Orsay has made it possible to achieve such a measurement in an ultrafast transmission electron microscope (UTEM).

These time-resolved nanocathodoluminescence experiments in a TEM will provide information at the nanoscale on the connection between structural properties and charge carrier dynamics in semiconductor nanostructures. They will allow for example to study the ultralocal modification of the internal quantum efficiency of semiconductor nanostructures linked to the modulation of the dopant concentration, to stress variations, to electric fields (quantum confined Stark effect), to interfaces or to structural defects such as dislocations. They will deepen our understanding of the physics of excitons in these confined systems, a physics that is at the heart of many applications in photodetection, light emission, single photon sources...

TEM images of a nanodiamond cluster, the white square represents the scanned area (insert image). Intensity (b) and lifetime (c) map of the light emission detected at each pixel. d) Time-resolved cathodoluminescence signal detected at the locations represented by the red and blue squares in c).

Time-resolved cathodoluminescence in an ultrafast transmission electron microscopes

Meuret, L. H.G. Tizei, F. Houdellier, S. Weber, Y. Auad, M. Tencé, H.-C. Chang, M. Kociak and A. Arbouet.

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CEMES SUPPORTS SECONDARY SCHOOLS

Since several years, scientists along with nonscientific staff working at the French National Institute for Scientific Research (CNRS) have been participating in a variety of scientific and cultural activities in schools and other allied educational institutions of the Toulouse academy, the regional education district.

Be it through individual or collective initiatives, with the help of associations or centers of scientific interest, researchers and professors have proposed a series of actions to introduce high school students at all levels to the different possibilities to have a career in scientific research and to awaken their interest in the different scientific disciplines.

In this context, on November 15, 2021, CEMES hosted the ceremony where a framework agreement for the promotion of industrial scientific, technological culture in schools was signed between CNRS (west Occitanie) and the Toulouse academy. Currently, CEMES is actively engaged in contributing to this action: given below are the three ways in which CEMES collaborates with secondary schools.







CEMES Club in Berthelot Middle School

In 2021, for the second year in a row, three teachers, two in physics and chemistry and one in biology-geosciences, started the CEMES club at Berthelot middle school in Toulouse. A group of around 20 eighth grade students from different sections came together during off class hours between 12 and 2 every Thursday for 10 weeks. Working together, the group prepared an agenda for their visit to CEMES. The students prepared detailed reports and made presentations; marks awarded were counted in the overall grade for those students who appeared for the secondary school certificate exam. This work, which comprised lab visits, dialogues with different members of CEMES (interviewees included research scientists, associate professors, and also technical administrative staff) to get to know the different career options in science. The students also took part in small research projects based on on-going research work at the lab. Not less than 18 members of CEMES took part in this initiative.

'Apprentis Chercheurs'

For the first time, our laboratory has committed to the national 'Apprentis Chercheurs' (research apprenticeship) program of the association 'l'Arbre de la Connaissance'. Under this action, students work in pairs in research laboratories (an eighthgrade student partnering with a twelfth grader) and are mentored by researchers in various laboratories. Each pair works with a researcher every Wednesday afternoon for 8 weeks around a research project specially designed, taking into consideration their level of competence and aptitude. The students then present their results to the general public during a special event in spring. During the year 2021-2022, our laboratory will host two pairs of students who will work on the optical properties of materials.

Plan DECLICS

At the heart of the innovative plan 'DECLICS' (that essentially translates to promoting dialogue between scientists and high school students to motivate them to build their knowledge base) is a speed-dating type meeting: scientific personnel (doctoral student, tenured researcher, technician, laboratory director ...) sit at a table together with five high school students, converse with them for twelve minutes, and then change tables. This information exchange is repeated several times, during which each student gets to meet seven different scientists. In 2021, CEMES participated for the first time in this national initiative led by the Cercle FSER in which, 5 members of the laboratory met up with first year high school students from Lycée (High school) Berthelot.







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MECHANICAL SERVICE UNIT

In CEMES, we have a mechanical service unit which has a technical design center and a workshop where both conventional and computer-controlled machines are available. This service employs three persons and pools its machine tools with INSA Toulouse.

Our mechanical service designs and fabricates individual pieces or fitted ensembles and assists in the development of sophisticated instrumentation for the various research groups and service platforms in addition to providing overall technical assistance to the lab. Such activities are mainly related to microscopy techniques (near field, electron, or optical). To efficiently assist researchers, our technical team is qualified in vacuum techniques, cryogenic or high temperature mechanics and also in the ultra-high precision machining of a wide range of materials, some of them novel or unconventional.

Among our ongoing projects, several deal with the development of innovative devices to enable light injection in different types of equipment. We describe here, these rather special devices.

One particular example is that of Project FEMTOTEM, where we were called upon to fabricate a one-of-its-kind mirror holder that allows to focus a laser beam on an electron emitting filament of an electron microscope, allowing to study the interaction between photons and electrons in an unprecedented manner. For the first prototype, we had to adapt our mirror holder to commercially available mirrors, which turned out to be a particularly complex operation. The excellent results obtained led to the development of a second instrument where the mirrors were custom-made in the laboratory.

This integration of mirrors is also required for an ongoing project on optical microscopy for which we chose to not only design but also fabricate the mirrors ourselves. Our aim here is to develop expertise and inhouse technology to cater to the needs of researchers in their projects and give them the opportunity to develop devices with more robust performances.

Our learning experience in the fabrication of these innovative objects also led us to procure special monocrystalline diamond-based tools. Such tools are used in turning or in milling, for which our 5 axis CNC milling machine is ideally suited. For the mirrors, we are currently in the process of trying out two materials (copper and aluminum) for which, a surface roughness less than 100 nm (less than 1/10 the wavelength of light) is needed, according to the specifications given to us. Depending of the roughness of the mirror surface, as revealed from AFM and laser interferometry, an additional gold or silver layer will be deposited if necessary.

CAD design - Left: mirror holder designed for a standard mirror (already made and working); right: mirror directly machined on the support (in progress)





5-axis CNC (computer numerical control) milling machine used to fabricate the mirror



Machined mirror in which the diamond tool is reflected



Machined mirror

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