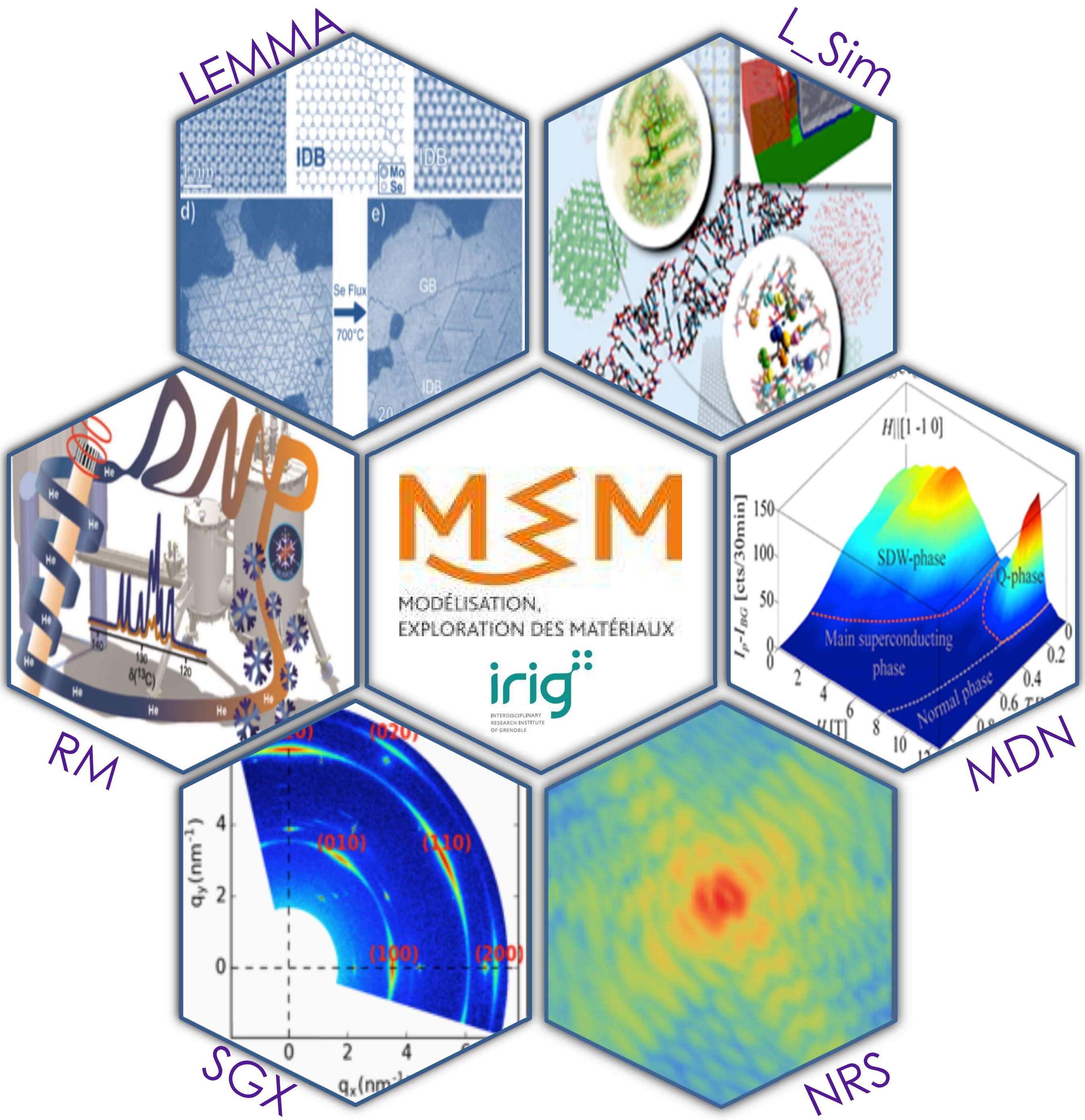
Activity Report 2014-2019



Self-assessment document
Research Unit: *MEM – UMR_E9001*
Modeling and Exploration of Materials laboratory

This activity report is the self-assessment document for the evaluation campaign 2019-2020 group A of HCERES.
The visit of the HCERES committee was on Tuesday January 7, and Wednesday January 8 2020.

Research Unit
Self-assessment document

EVALUATION CAMPAIGN 2019-2020
GROUP A

GENERAL INFORMATION

Name of the unit concerned by the current contract: Modélisation et Exploration des Matériaux

Name of the unit concerned by the next contract (if different):

Acronym of the current contract: MEM

Scientific field (name two fields if interdisciplinary evaluation): **Sciences and Technology (ST)**

Scientific sub-domains (in Hcéres' nomenclature) in descending order of importance**:**
ST2 (Physics - ST2_3: Materials, Structure and Solid-State Physics)
ST4 (chemistry – ST4_1: Theoretical and analytical chemistry)
ST6 (Information and communication technology sciences – ST6_1: computing)

Director for the current contract**: Francois Rieutord**

Director (or project leader) for the next contract**: Thierry Deutsch**

Type of application:

Identical renewal ☒ Fusion, scission, restructuring ☐ *Ex nihilo* creation ☐

Academic institutions and affiliated organisms:

List of Institutions and Organisms supervising the Research Unit **for the current and next contract**:

Current contract:	Next contract:
CEA	- CEA
UGA	- UGA
	- CNRS

Choice of the research unit's interdisciplinary evaluation (or of one or more in-house teams):

Yes ☐ No ☒

Clinical research activities:

Yes ☐ No ☒

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THE UNIT: MODELLING AND EXPLORATION OF MATERIALS (MEM)

1- Presentation of the MEM unit

History, localisation of the unit

The MEM laboratory has been created on the the first of January 2016. MEM stands for Modelling and Exploration of Materials (Modélisation et Exploration des Matériaux). The MEM laboratory ("service" in terms of CEA structure) aimed at grouping together different teams whose common point is to be centred on a given experimental technique, or class of techniques. It fits in the "recherche fondamentale et instrumentation associée" and in the "Large facilities (TGIR)" programs ("mailles") of the CEA. The initial and present mission of the teams is to be experts in their field, pushing the techniques to the forefront of current developments, for the benefit of the other labs within the institute (INAC at the beginning, IRIG now) or more generally for the CEA or the French community. Hence the MEM teams "DNA" includes collaborations with other labs and institutions, to have them benefit from the methodological developments carried out in the lab. The CEA name for laboratory (="service") hence fully deserves its name for our activity. Although MEM researchers do have specialties in certain physics or chemistry areas, or on certain class of materials, their main driver is not based on a specific theme or class of materials but rather on an experimental or modelling technique. Most researchers have a material science background.
Most of the teams were initially (ie before 2016) within the SP2M service, and their contour has not changed when included among the MEM service. The neutron team MDN came from SPSMS. Hence the two "solid state physics" labs SP2M and SPSMS were reorganized into Pheliqs and MEM services. In addition, the NMR team of the SCIB laboratory (now SYMMES) joined the MEM also.

The location of the different teams is spread over the polygone scientifique, due to the different locations of their instruments. This ranges from the EPN campus (ILL for neutrons and ESRF for synchrotron), then at C5 building for part of the NMR team, the SGX and LSIM laboratories. Service headquarters and secretaries are located in C5. Finally, part of the NMR (DNP) team and the electron microscopy team are located in Minatec to share instruments with LETI and LITEN teams. Of course, such a spread location within a research unit is not ideal for cross team collaborations, but the common preoccupations of our rather specific activities brings some intellectual proximity between team leaders and members.

Structure of the unit

As mentioned, each team is centered on a given experimental technique or class of techniques:

-the L_SIM laboratory bears the modelling part of the research unit: they are specialized in developing ab-initio simulation codes based on Density Functional Theory (DFT) or other techniques. They develop specialized codes (e.g. BigDFT) well-suited for material and device simulation.

- the LEMMA (Laboratoire d'Etude des Matériaux par Microscopie Avancée) groups researchers experts in electron microscopy and ions techniques. They use the state-of-the-art Transmission Electron Microscopes of the PFNC (PlateForme NanoCaractérisation de Minatec) to develop techniques to study at high resolution the structure, composition and strain in nanomaterials. Important to mention is the development of ion slicing technique based on FIB (e.g. FIB SEM tomography).

- the MDN laboratory groups researchers operating or using (among others) neutron diffractometers or triple axis spectrometers for investigating magnetic structures and excitations. Our instruments are operated as CRG instruments at ILL, in collaboration with the Jühlich Forschung Zentrum. Some of these instruments were initially installed on the CEA Grenoble research reactor Siloe, and then transferred (1997) to ILL as CRG (e.g. external) instruments.

- The NRS team has a similar history. It was set up at the construction of ESRF in 1993 to group CEA researchers that would operate with CNRS colleagues French CRG beamline at ESRF. Most of the researchers are specialized in the study of nanostructures and in surface and interface grazing incidence techniques.

- The RM (NMR) team groups experts in NMR for the study of materials. The techniques range from standard NMR spectroscopy (solid and liquid) to relaxometry and hyperpolarization technique such as DNP MAS NMR. The team has developed in collaboration with the low temperature service (SBT) unique setups for DNP at very low temperatures.

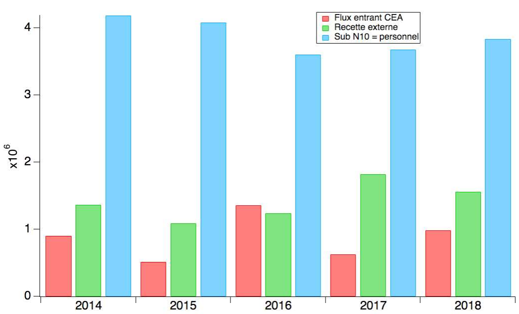
- The SGX group includes many lab X-ray instruments to be used on a daily usage by researchers from the institute – Their equipment and competence include high resolution X-ray scattering, small angle scattering, reflectometry, powder and single crystal diffraction.

Unit's workforce and means

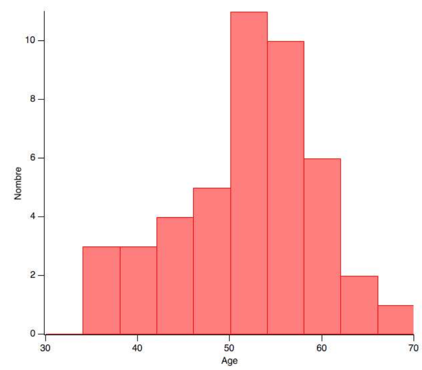
The research unit is a common CEA-UGA research unit, with a large dominance of CEA members (95%). Our unit includes only 3 assistant professors from the University (1 being presently detached to ESRF). Hence the evolution of workforce within the unit is determined by the general trend within the CEA fundamental research, i.e. little or no recruitments and consequently, a decrease of the global workforce.

Same statement can be done for the financial means. The funding at CEA is full cost, i.e. includes salaries of permanent people. Unfortunately, the constant reduction of this funding with a small decrease of the number of workers has resulted in a deficit between dotation and fixed expenses. Hence the laboratory can only rely on external resources to

1) Compensate for the lack of resources for salaries and fixed expenses (network, fluids, electricity, office building), typically 500k€/y.
2) Get some means to carry out research activity and maintain equipment (lab running, investments, etc…)



Budget evolution (revenue only). Blue is the permanent personnel budget allocation, red is the lab income managed by CEA (e.g. large facility instrument funding, contracts managed by other labs (ex: DRT) with funds transferred to the lab, or direct CEA programs) and green is direct external income (Europe, ANR, etc). Depending on the success to different calls, these numbers may vary.



Age distribution of the MEM (permanent staff). Average is 52.

Being a fundamental research lab, income possibilities to funding agencies are very competitive and this put every researcher in the lab under high pressure for funding. The present level of income (estimated on the 5 year basis) does not allow *a large enough self-financing capacity. This has been confirmed in several instances: when instruments break out after a long working lifetime, there is no or little possibility to renew or replace them, except for very specific actions that are difficult to anticipate.*

The same statement can be made for staffing. Only one person has been recruited during the 5-year period considered here (note that the recruitment of an ERC grantee is under way at the time of writing) while we experienced a few departures (4 +1[June 19]). Consequently, the age distribution shifts constantly with a 2019 average around 52y. Undoubtedly, this is a serious problem ahead.

Scientific policy

As the research unit was non-existing during the previous evaluation, we can only rely for the unit on the evaluations in individual teams. The creation of the MEM laboratory was a response to try to better organize the institute (INAC at the time), grouping together teams that, contrary to classical labs, are not centred on a given scientific issue or material but act more on technical developments to improve scientific instruments. The idea was also to share common problems and allow more easy material investigation and problem solving using multiple techniques. The easier sharing and access to large and expensive instruments to a wider community was part of the idea in the MEM creation.

The two drivers for MEM are thus to:

-provide the community (institute, CEA, France) with state-of-the-art instruments and expertise covering most of traditional material investigation techniques

-develop a few specific very advanced techniques to remain at the forefront of material investigation.

The different techniques made available to the community by the MEM lab are

Electron Microscopies where a unique suite of instruments is available thanks to the Minatec Nanocharacterization platform. It includes aberration-corrected transmission electron microscopes. The expertise developed on these microscopes concerns strain measurements, chemical analysis and 2D materials. The lab expertise includes also ion etching and analysis techniques and significant developments have been carried out using the combination of focused ion beam (FIB) and scanning electron microscopy (SEM) into a powerful tomography technique. Limited manpower and financial means have led to a shutdown of the medium energy ion scattering instrument (MEIS), which has been transferred (01/2017) to another laboratory (IN2P3, Lyon).

Ab-initio modelling with a large material expertise. The group has expertise in ab-initio code development and applications. This is used to investigate and predict material properties, such as structure, defects or excitations. Code development and implementation on HPC are still actively pursued. One new significant evolution in the group themes is the silicon CMOS spin qubit modelling, which is key for a possible quantum computer development using silicon technology.

Synchrotron and more generally X-ray scattering are also strong components of the lab with the NRS and SGX teams. These labs specialize in micro- and nanostructure characterization. The synchrotron team provides personnel for the running of CRG beamlines, essentially centred on the BM32 "interface" beamline. The completion of the new UHV surface diffractometer allowing in-situ growth investigations has been achieved thanks to an Equipex funding. In the lab, new high resolution and powder diffractometers have been installed. The new EBS source of ESRF presently under construction will set new challenges to the team.

The neutron scattering group host researchers involved in some French CRG instruments at ILL. They specialize in magnetic material studies, both for static structures (D23) and magnetic excitations and lattice dynamics (IN12 and IN22). The instruments are now fully open to the community, and many science collaborations are very active and productive around these instruments. The future shutdown of the French neutron source Orphee (end of 2019) will also put large expectations from these few French neutron instruments.

The NMR team groups experts of the technique. In addition to classical solid or liquid NMR, mostly applications- oriented, a strong development of low temperature (<100K, He cooled) Dynamic Nuclear Polarization is being carried out in the team with the help of the low temperature laboratory (SBT). A key contribution and recognition to this work is the ERC grant allocated to Gael de Paepe. A new low field relaxometer is also being developed in the lab.

The different teams of the MEM lab, although working on different themes and often in different fields share a common strategy for their development. Due to the very large cost of the instrumentation, most instruments have to be shared and financing can be born only by a rather large community. As a counterpart, the instruments have to be opened to this community with a necessary service to the community members. The range of the service to community users depends on the instruments. It is typically the institute (formerly INAC, now IRIG) for smaller instruments (lab X-ray diffractometers, NMR spectrometers, and electron microscopes) but extends to nation-wide and international service for synchrotron or neutron beamlines. The intra-institute service model follows different cost sharing agreements between teams and labs: direct cost sharing of experiments (NMR), lab cost sharing (SGX) or institute cost sharing (LEMMA). The nation-wide service at ILL and ESRF includes external funding (included in the CEA-managed part, red bars on budget graph). Beam time distribution and management in this case is done through program committees and different coordination structures: "Structure d'exploitation", "Fédération Française de diffusion neutronique" with participation of other research institutions (CNRS).

Parallel to this service part, researchers in MEM have to develop their own research usually pushing the current techniques to their ultimate limits and demonstrating the benefits of new techniques. Developing cutting edge techniques is also necessary to build successful research projects that could allow the financing of these new or better instruments.

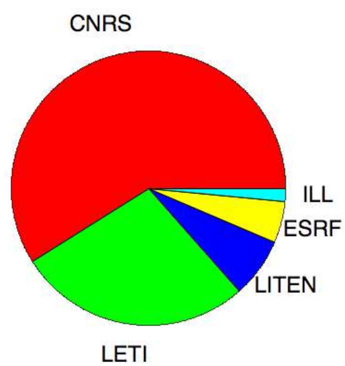
At the CEA, there is no statutory separation between engineers and researchers that could correspond to the service and research types of activities. As a result, most people in MEM carry out the two duties. Although the two activities enrich each other and bring lots of openings and collaborations, aiming at these two objectives at the same time puts generally a high level of pressure on researchers, especially in a context where the overall manpower is decreasing and most activities are understaffed. In particular, there is a lack of support personnel (e.g. technicians) considering the high technical level of most of our activities.

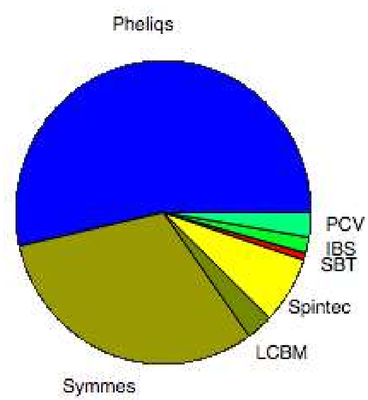
Our scientific strategy at MEM has been to favour as much as possible cross team actions to work against isolation and separation, while remaining on institute and CEA themes. Among these themes where internal interactions have taken places one can cite

- 2D materials – These materials represent *challenges* to observe them and to characterize their structure and defects. The lab is able to study these structures that include very few atoms using advanced microscopy techniques and synchrotron X-ray diffraction. The specificity of modelling these structures has also been tackled within the L_Sim lab.
- Semiconductor materials: in the institute and in the Pheliqs lab, a number of material growth instruments are available, and these are used to grow semiconducting structures. The strain inserted in these structures modifies the band structure and hence the opto-electrical properties of these compounds. Here again, modelling the property changes of the structures upon stress is a challenge.
- Batteries and specially Lithium batteries are promising material for energy storage e.g. for electric vehicles applications. Our structure and spectroscopic tools (electron microscopy (TEM, EDX, EELS) , synchrotron radiation (Small and Wide X ay scattering , X-ray Raman absorption spectroscopy, tomography) , and NMR are key tools to investigate both bulk materials and interfaces in these electrochemical systems.

2- Presentation of the MEM unit's research ecosystem

Most of our production consist of research papers. Overall both quantity (3p/y/researcher) and quality of research papers are good for the lab. Many of the papers are obtained in collaborations. The main collaborations are with other labs within former INAC (about half of our publications). As for external collaborations, Institut Néel and LETI/LITEN institutes are our main collaborators and correspond overall to also more than half our publications. (See Fig).





Collaborations with labs external from the institute as measured using common publications. A full circle corresponds to about half of our publications.

Collaborations with labs within the institute. Here again, a full circle corresponds to about half of our publications.

The MEM lab is a joint unit between CEA and UGA including only 3 assistant professors from the UGA. The lab is attached to the PEM pole of research of the University. Different LAbex are active in the Grenoble area and our lab is mainly affiliated to the LANEF and GANEX labex and to a lesser degree to the ARCANE Labex (bio-inspired chemistry). The labex LANEF have contributed to equipment financing (e.g. the high resolution SmartLab™ diffractometer for LANEF) and contribute to full- or half - PhD fellowships.

The NRS and MDN teams are in close collaboration with ESRF and ILL facilities. As a matter of facts, collaborators from these teams are totally or partly hosted on the EPN campus, a few hundred meters away from the CEA campus. The relations with these large facilities are excellent and based on a long-standing mutual knowledge. However, our teams should contractually operate the instruments on an independent and stand-alone basis, and no contractual assistance from these international institutes is due to our teams. Yet, some of our instruments are unique and of interest to the international community so that exchange and collaboration between experts of both sides is frequent. Many of our PhD students have been hired by the ESRF and our teams often act as go-betweens the local academic or school system and these international large facilities. Note that the proximity of these large facilities brings some visibility to our activity, because we can benefit from their size and organization. However, their attractiveness as international facilities causes also some drain of manpower and competences from our lab.

Most of the personnel belongs to the CEA and very close collaborations exist with the other labs from the institute, mostly Pheliqs (most of the teams from MEM and Pheliqs were initially in the same lab SP2M) and also SYMMES (formerly SPraM).

We have also significant collaborations with industry, either for instrument manufacturing (Bruker, FEI) for joint development of methods, or for the applications. This collaboration takes the form of contract studies, collaborator sharing (KAO), or involvement in common labs (FEI, SOITEC...). This represents significant income for the running cost of the instruments.

It should be mentioned also that, in addition to professors and assistant professors, a few CEA researchers from the unit are involved in teaching tasks. This includes practicals in the Hercules and ESONNS schools and teaching in some classes at master level. We do also practicals in the CESIRE (L3/M1 level). Many of the lab supervisors and students are related to the doctoral schools "Physique" and "Chimie et Sciences du Vivant". Note that both the founder, director and study director of the Hercules international school are/have been members of the MEM lab. Here again, the lab members play a key role at the interface between the large facilities and the local educational university system, contributing to the international visibility of the university.

3- Research products and activities for the MEM unit

Scientific track record

The track record of our scientific activity is of good quality and quantity

Overall, the lab publishes typically 100 scientific papers per year which averages to more than 3 papers/ researcher/year not considering the separation between engineer and research work (typically the lab counts less than 30 full time researchers). The impact factor is of high quality with typically more than 20% (117/515) of publications with impact factor above 7, including Phys. Rev. Lett., JACS, NanoLett, Nature group publications etc. We refer the reader to Annex 4 and individual team reports to have a detailed view of our science highlights. The publication track record of the different teams is of course different depending on their core activity and their balance between service duties and own research. The lab has many international collaborations and about half of our publications include a member of a foreign laboratory.

Among the science themes that come often among our scientific highlights, one should cite 2D materials (studied using TEM, X-ray or Simulation), silicon or other semiconductor nanostructures (TEM, X-ray, simulation …), energy storage systems as e.g. Li batteries (TEM, FIB SEM X ray NMR, Simulation).

As mentioned, most MEM researchers do not carry by themselves a focused research activity but work within collaborations. Yet, when the experimental or simulation techniques plays a key role, several publications or research contracts are also led by MEM researchers. As an example, 2 European research contracts ERC have been obtained for the MEM lab (one by G. de Paepe on DNP, and another one by MI Richard, a former PhD student and close collaborator, under recruitment in the lab at the time). Other research contracts have been obtained, with a MEM researcher as a main proposer.

Experimental work and findings lead also to patent filing. Methodological developments have led to several patent filing (e.g. about the development of low temperature DNP NMR instrumentation) and our expertise gained on some material properties (often in collaboration with applied research) has also led to patent filing and licensing, especially in the semiconductor material area (e.g. GaN or SOI structures).

4- Organisation and life of the MEM unit

Steering, life, organisation in the unit

The direction of the unit is the interface between the lab and the institute where most personnel issues are treated for CEA employees (career, perks, bonus etc.). So, an important role of the director is to discuss with the different team leaders and institute responsibles to ensure a fairest evaluation of lab members. Regular meetings and discussions with team leaders have been carried out (1/month) and consensus decision making has always been reached, thanks to a cooperative spirit that has been set up within the lab. Being a small lab, direct interactions between unit members are easy. Formal meetings (twice a year) of the Conseil d'Unité including representatives of the different categories of workers is another place where problems can be discussed.

The different locations of the different teams, from Minatec to EPN campus is of course a factor that does not favor day-to-day "coffee-machine" interactions, or interactions between the direction and the teams. We set up "prospective" meetings within each team to review their activity and discuss the team strategy. Other team leaders or members are invited to these day-long exercises. These team meetings are organized at the location of another team (e.g. at EPN campus for Minatec based teams) allowing cross interactions between team members around instruments at the end of the day.

Parity, scientific integrity, health and safety, sustainable development and environmental impacts, intellectual property and business intelligence

Being largely a CEA structure hosted on a CEA centre, little specific actions could be undertaken at the lab level.

On gender issues, being essentially a material physics lab, we have a small number of female colleagues (12%). Yet 2 out of 6 team leaders are women, and the recruitment of an ERC-grantee Marie-Ingrid Richard should improve our statistics. We welcome of course female PhDs and Postdocs, and the ratio is higher than for permanent people.

No specific scientific integrity policy is conducted at the lab level but there are at the CEA several control steps (e.g. "autorisation de publication") that could contribute to avoid misconducts. The evaluation of personnel at the CEA includes many aspects and we try to focus on time-averaged global evaluation reducing the pressure on individuals to obtain very high impact results in short times.

We welcome PhD and postdoc students in our lab and we are committed to giving them a good training. Due to our service activity, and dependence on schedule of large instruments, this task may sometime be complicated. As a consequence, the average number of PhD fully attached to the lab is rather small.

Health and safety are essentially taken over at the institute or above level. Specific actions on RPS ("Risques Psycho-Sociaux") have been carried out with the safety engineer, medical services and management, with meetings to allow personnel to express their concerns and problems. The high workload on personnel (e.g. on large facilities) is a key factor for stress and is taken very seriously by the management.

Our lab includes disabled people and we of course take part in the specific actions carried out at the employer level on this subject (training of management, adaptation of workstation, medical equipments...)

For sustainable development and environmental impact, again, this is treated at the centre level. Financed at the lab level is the installation of closed cooling loops to avoid use of industrial water (for lab X-ray generators). The use of closed loop refrigerators for low temperature cooling is also favoured whenever possible. A number of lab and team bikes (>10) are available to facilitate displacements between the different teams, with a free maintenance of bikes at the centre level.

Most teams in the lab have industrial contracts, and are aware of problems associated with intellectual property and business intelligence. We benefit also from the action taken at the centre level. Since CEA-Grenoble is driven by the Technological Research Division, these issues are very present in the safety and security actions taken at the centre level.

Five-year project and strategy of the MEM unit

SWOT analysis

Strengths	***Weaknesses***
Good scientific production High level of complementary expertise Unique instruments Large variety of income sources (Europe, France, Industry)	Low level of support staff especially for service activity Small teams Aging instruments Aging staff (52y) Weak level of funding for running/ renewal of instruments
Opportunities	***Threats/Risks***
Central role of Grenoble in TGIR (new source EBS @ ESRF), new program at ILL Extension of the institute to new domains (Biology) Recognized importance of simulation/ data policy in new programs	Retirement of staff/loss of competence Instrument breakdown

Structure, workforce and scientific orientations

Structure

"Coping with the drought of financial and manpower resources"

The perspective of a much-improved situation for funding or staffing in the years to come seems quite unrealistic. As a consequence, we believe that strategies to cope with reduced funding and staffing should be carried on. These include pooling of equipment whenever possible, and facilitating personnel movements between structures and labs. Our long-standing collaborations with CNRS personnel in our different teams lead us to consider that having the CNRS as a supervising institution would ease a lot the common work on our instruments, avoiding situations of CNRS personnel that would find themselves isolated in their host lab. This would allow a recognition of their contribution to the common work. Most large facilities can be operated on a national basis only and having a lab that is able to smoothly integrate people from both CEA and CNRS institution (in addition to University) would certainly be a win-win operation. Note that at the moment, many of our CNRS collaborators belong either to Institut Néel or to the Spintec/Symmes CEA/CNRS labs, with whom relations are excellent.

Scientific orientation

"Coping with data deluge"

Simulation, modelling and data mining are expected to play an increased role in research and development and this is true for material research also. The creation of the L_Sim team specializing in this type of research, several years ago, has been visionary and we now have to try better benefit from their presence and knowledge in the lab. Simulations can of course be used (or are mandatory) to analyse experimental results. This is true e.g.

for NMR where DFT codes are routinely used to calculate spectra. This is true also for other spectroscopic measurements with electrons or light (e.g. Raman) where bandgap calculations are necessary to experiment interpretation (EELS or Raman).

To develop, optimize, benchmark and run its codes, the lab (within the L_SIM) has developed a recognized expertise in high performance computing. This competence should be key for other teams also: we observe on some techniques that, due to large and fast detectors, the amount of raw data produced during an experiment can be quite large. High performance computing (GPUs, neural networks...) may be needed in some instances to cope with these large data volumes and allow experiment driving in real time. This includes the use of efficient tools and algorithms which have been developed for simulation purposes and that could be used for data treatment also (e.g. wavelet transforms). An increased role of artificial intelligence and machine-learning based software is also expected (pattern recognition etc)

This may also influence the way we provide service to the community, as we may now need to include provision for specialized data treatment tools (software and hardware) probably as cloud services: SaaS IaaS PaaS[1]. Specific support for data treatment is already done today (e.g. The LaueTools suite) but this will be amplified in the future. New operating models have to be defined and proposed (Notebooks, Containers…)

Data policy of public funded institutions requires that we comply to a number of rules in our data management, including metadata supply. Research programs aiming at screening material properties using big data and IA tools are deemed to develop in the years to come. The role of aim-specific experimental research has to be maintained though and proper database feeding in material science carefully considered. Experience from biologists now in the institute (IBS) e.g. on protein structure database management and contribution may be of interest to material scientist.

"Complementarity and coherence"

Finally, we hope to pursue cross team researches where the lab can bring its complementarity of different skills.

On 2D materials, the collaboration that was initiated within the "2D factory" CEA program, could be pursued. Emphasis will now be put on the material processing and use of 2D membranes. Some actions have been taken in collaboration with DRT, since these materials may now be ready to enter practical usage. Research has been engaged on the transfer of these layers between substrates, based on the bonding and transfer expertise gained in silicon. The use of 2D layers as membranes for the study of confined fluid flow is also a perspective of collaboration with other labs, in parallel to pores in biological membranes that are studied in the new institute.

The NMR X-ray complementarity has been demonstrated in several instance (battery material, hydrogen storage, perovskites…), with standard short range order/ long range order complementarity. The gain in NMR sensitivity brought by DNP may allow this type of studies to be done on interfacial layers, in complement to grazing incidence X-ray techniques. The complementarity in the methodology may also be developed around experimental techniques like ptychography, where oversampling is used to ease inversion of coherent scattering patterns. New instruments (ESRF EBS and Low voltage aberration corrected TEMs) are the triggers for these developments.

The recent inclusion of the lab inside a bigger institute together with biologists from the fundamental research division of the CEA may bring new opportunities to our lab, as our techniques may be key assets to contribute to biology subjects. Already demonstrated is e.g. the key interest of FIB-SEM tomography techniques to study micro-algae and their role in carbon cyle. Expertise in NMR and electron microscopy is already present in other labs, but new techniques like low T DNP may bring revolutionary input to their disciplines by allowing e.g. Studies of biological objects at natural isotopic abundance.

[1] SaaS,PaaS, IaaS stand for **S**oftware, **P**latform, **I**nfrastructure **a**s **a** **S**ervice.

We hope to be able to recruit new collaborators in the years to come. Recruitment of the ERC consolidator grantee Marie-Ingrid Richard is underway and confirms the lab attractiveness. This will be an additional momentum in the development of new techniques making use of new beam properties (coherence) and new acquisition scheme (ptychography), in between direct and reciprocal space. This will be of interest to both X-ray and electron microscopy teams.

Critical is the situation for service at neutron and synchrotron, with a need of young collaborators to contribute to user assistance and technical developments. This demand is also put to the university, as our lab has played a key role in the interface between university and large facilities.

Other teams are also in dire need for additional manpower. Hiring a data scientist/architect would be useful for the whole unit to help bearing the numerical transition to come. Reinforcing the technical capability of the NMR team for both service and development is also necessary, which would allow e.g. the general spread of low temperature DNP NMR in the community.

TEAM1: ATOMISTIC SIMULATION (L_Sim)

1- Presentation of the L_Sim team

Introduction

The Laboratory of Atomistic Simulation (L_Sim) is mainly located at the C5 building except for the group of YM Niquet (3 persons) which is hosted in the Laboratory of Simulation and Modelling (CEA-LETI) in order to facilitate strong collaborations with the technological research direction (DRT) of CEA. The aim of L_Sim is to develop new computational methods to calculate the electronic structures and properties of materials and devices at the atomic level and to study, in collaboration with some experimentalist teams, material structures and properties as defects in semi-conductors, grain boundaries, 2D materials, and organic molecules.

A first team of 5 researchers (DC, TD, LG, FL, and PP) develops and uses the BigDFT code for their studies. Started in 2005, BigDFT is based on wavelet to solve the Kohn-Sham equations in the Density Functional Theory (KS-DFT) in order to calculate the electronic structure and the total energies of molecules.

Since 2011, I. Duchemin, in strong collaboration with X. Blase, G. d'Avino (Néel Institute, CNRS) and D. Jacquemin (Nantes University) have been developing Fiesta and now beDeft to calculate charged and neutral excitations in molecules using GW and Bethe-Salpeter equations (BSE) from the Many-Body Perturbation Theory (MBPT).

Since 2004, in strong collaboration with LSM(CEA-DRT) and IEMN Lille, YM. Niquet, has been developing a multi-scale multi-physics tool, TB_Sim mainly oriented to the study of transport and electronic properties of CMOS and photonic devices based on non-equilibrium Green functions. Since 2017, his research topic was switched to the modelling of silicon qubits in tight collaboration with experimentalists from CEA (PHELIQS laboratory and LETI) and Néel institute (ERC Synergy quCube).

Unit's workforce and means

Permanent staff (01/2019)	Non-permanent staff
Damien Caliste, CEA researcher Thierry Deutsch, CEA researcher, team leader Ivan Duchemin, CEA researcher, main developer of Fiesta and beDeft Luigi Genovese, CEA researcher, main developer of BigDFT Frédéric Lançon, CEA researcher (retired 07/2019, scientific adviser) Yann -Michel Niquet, CEA researcher, main developer of TB_Sim Pascal Pochet, CEA researcher, team deputy	**Post-doctoral researchers** L Ratcliff (2012-2014), S Mohr (2013-2015), M Bronsgeest (2013-2014), A Cerioni (2014-2015), E Vandyukova-Zvereva (2014-2016), J Li (2013-2015, 2018-2020), JM Escalante Fernandez (2014-2015), Z Zeng (2015-2017), B Videau (2016-2018), JC Alvarez Quiceno (2018-2020), A Degomme (2018-2021), S Selvaraj (2018-2020) **PhD students** C Faber (2011-2014), D Timerkaeva (2012-2015), G Brenet (2012-2016), S Mathur (2013-2016), M Morinière (2013-2016), L Bourdet (2015-2018), B McGuigan (2013-2018), K Moratis (2016-2019), B Venitucci (2017-2020), E Annevelink (2017-2021), S Gupta (2018-2021)

The laboratory budget is of about 200 k€ per year, which is shared into non-permanent salaries (approx. 80 %), running costs (approx. 10 %), and investments (approx. 10%). Most of the funding is coming from European and ANR projects.

Scientific policy

Methodology and software development, within a network of partner laboratories, are the strength of the laboratory. The award-winning HPC codes, BigDFT, TB_Sim and Fiesta, are recognized mainly in the ab initio community as some innovative approaches to study, faster and more accurately, the structure and properties of materials.

Thanks to these predictive and powerful tools, we study defect engineering applied to semi-conductors and 2D materials. P. Pochet and D. Caliste studied the formation energies of different neutral and charged defects in different materials such as HgCdTe with a contract with the Sofradir company specialist in the infrared vision business. In the European EXTMOS project, using BigDFT and the optical properties with Fiesta, the electronic structure of OLED films was determined. Different architectures of CMOS as FDSOI or nanowire transistors were modelled using TB_Sim to explain the behaviours and to predict their characteristics for smaller geometries.

2- Presentation of the L_Sim team's research ecosystem

The team collaborates strongly for the development of the codes especially around the HPC (High Performance Computing) and algorithms. We have tight collaborations with computer scientists (INRIA, LIG, RIKEN, BSC) to develop massively parallel algorithms or using accelerated platforms (Graphics Processing Units) but also mathematicians (CERMICS) and physicists (Néel Institute, LETI). Because we have no permanent computer scientists in our group, these collaborations are very important to improve the quality or our programming thanks to the software engineering. We have been participating to different networks as the European Centre of Excellence EoCoE (2015-2018) and the European centre of excellence MaX (2019-2022) which groups the main European DFT developer groups (SISSA, EPFL, Jülich, ICN2).

We are involved into the federative structure of UGA, GRICAD, and are contributing to the CECAM local node (European network organizing high level scientific workshops). T. Deutsch is the head of the Centre forPredictive Simulation (CSP) which regroups the laboratory of simulation from IRIG (MEM/L_Sim and SPINTEC), LETI (LSM), and LITEN. Strong Interactions with experimentalists of MEM (mainly LEMMA and NRS), PHELIQS laboratory (NPSC, SINAPS, and LATEQS laboratories), CNRS (Néel Institute), LETI, LITEN, and with foreign laboratories (Urbana-Champaign, Montreal University, Boston College, Imperial College, Basel University, and EPFL) allows for high research impact in domains where our innovative approaches provide a competitive benefit.

3- Research products and activities for the L_Sim team

Software development activities

This software legacy represents a very large effort in programming (TB_Sim 500,000 code lines roughly 30 man.years, BigDFT 500,000 lines roughly 30 man.years, Fiesta, 100 000 lines 10 man.years, beDeft 50,000 lines 3 man.years) with some development overlapping. BigDFT uses the Gain library from Fiesta. TB_Sim was also accelerated in GPU as BigDFT. Hiring also computer scientists in our group (B. Videau and A. Degomme) is important to have a computer background and to interact strongly with the computer scientists.

During the period 2014-2019, we improved considerably the software quality of our codes based on version control as git, non-regression tests and continuous integration. We developed the linear scaling approach and extended our developer network around BigDFT. beDeft is a rewriting of Fiesta to enlarge its capabilities based on C++ and Lua scripts. TB_Sim was considerably extended in order to simulate the physics associated to qubits. Python associated with Jupyter notebooks is widely used fro pre- and post-processing of our simulation results.

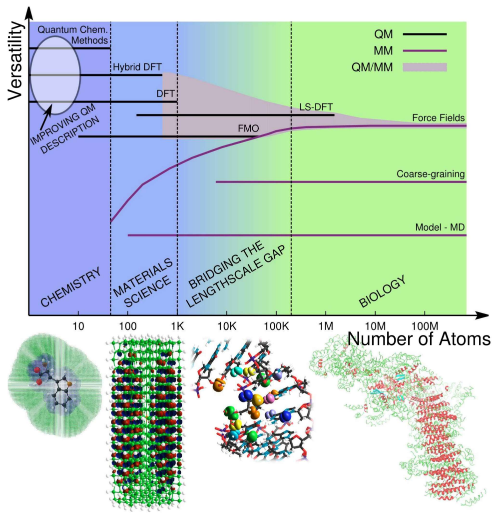
Scientific activity

Four main scientific themes:

- Developments of high-performance computing methods in the field of DFT for large systems and for linear response. We use mainly our BigDFT tool to explore new formalisms or new possibility to simulate large systems with different kinds of approximations.
- Determination of the structure, defects (point, line and surface defects) and elementary mechanisms, using ab initio methods, mainly for semi-conductors and 2D materials.
- The calculation of optical spectroscopy of molecules based in GW/BSE in a solvent via an embedding scheme related to the OLEDs
- The electronic transport properties for devices as CMOS and qubits.

Linear scaling algorithms for Kohn-Sham (KS) Density Functional Theory (DFT), developed already some time ago[2][3], have recently become accessible to a broader community thanks to the introduction of reliable and robust approaches (see [4] and references therein). This fact has important consequences for the interpretation

[2] S Goedecker, "Linear scaling electronic structure methods," Rev. Mod. Phys. 71, 1085 (1999).
[3] D R Bowler and T Miyazaki, "O(N) methods in electronic structure calculations." Rep. Prog. Phys. 75, 036503 (2012)
[4] LE Ratcliff, S Mohr, G Huhs, T Deutsch, M Masella, L Genovese, Wiley, Interd. Rev. Comp. Mol. Sci. 7, e1290 (2017).

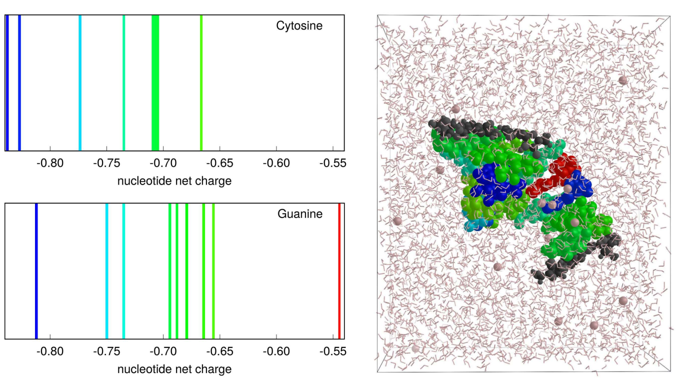


Overview of atomistic simulation methods, showing the length-scales over which, they are applied and the transferability, i.e. the extent to which they give accurate results with no retuning.

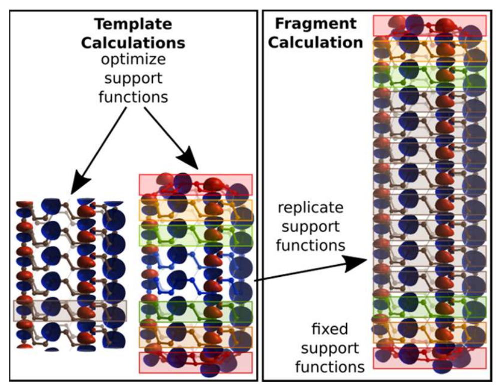
and the design of first-principle approaches, as the possibility to tackle systems of unconventionally large sizes allows to address new problems and questions. For a system containing many thousand atoms, it is likely that the fundamental constituents (or "moieties") of the system are of O(1), i.e. their size does not increase with the total number of atoms of the system. It appears therefore interesting to single out such moieties, and to try to model their mutual interactions with a less complex description. Thanks to linear scaling DFT, the full quantum-mechanical (QM) calculation of the original system can be used as an assessment of the quality of the simplified description. When linking together various length scales, such considerations are not just optional, but they rather become compulsory. Performing a set of production QM simulations with an approach being unnecessarily costly would provide a study of poor quality, as the simulation scheme entangles interactions with different length scales and couplings. Taking these considerations into account allows one to focus on the region of the system that require a high level of theory, providing a better understanding of the fundamental mechanisms and avoiding an unnecessary waste of computational resources.

H1: Reducing the complexity of Large-Scale Density Functional Calculation

Permanent staff: L. Genovese, T. Deutsch



Example of Fragmentation of a small piece of DNA in solvent investigated by BigDFT code.



Seven pseudo-fragments for a SiC nanotube generated from a combination of a six unit periodic NT template and an eight unit finite NT template.

The fundamental principle of such a reduction of the complexity of the calculation lies in the identification of the essential moieties (i.e. "fragments") of a system out of an atomic description. These fragments should then, in turn, be treated with an adequate methodology depending on the specific needs. A less complex description of a system might contribute to decreasing the computational cost of the calculation; however, this is not the sole advantage. Within such a scheme the observable quantities that can be extracted for the system as a whole can also be assigned separately to each of the fragments. Such a procedure enables a better understanding of the relevant mechanisms which govern the interactions among the constituents of the system, together with the design and validation of coarse-graining models, that are adapted for systems of length scales for which atomistic QM models would be unnecessarily costly or even out of reach.

In a publication[5], we derived a simple quantitative way of determining whether a chosen fragmentation is reasonable. If this is the case, the fragments become "independent" of each other and can be assigned "pseudo-observables", i.e. quantities with an interpretable physico-chemical meaning. We have also introduced[6] in this context a pseudo-fragment method based on LS-DFT which is able to treat extended systems containing some degree of repetition, e.g. a periodic system comprising many repeat units.

[5] S Mohr, M Masella, LE Ratcliff, Li Genovese, J. Chem. Theory and Computation 13, 4079 (2017)
[6] LE Ratcliff and L Genovese J. Phys.: Condens. Matter 31 285901 (2019)

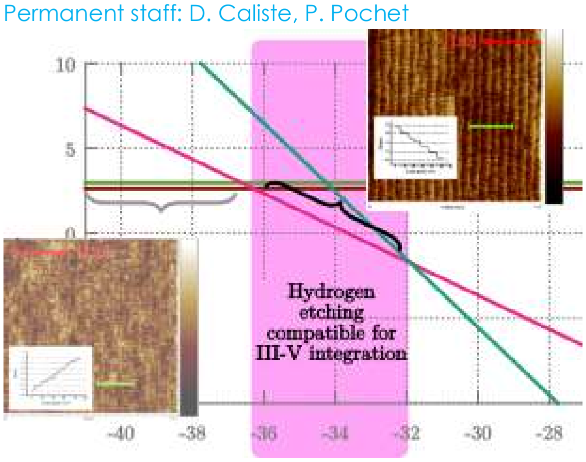
Such an approach might easily be defined if, instead of taking an isolated template fragment, one embeds the pseudo-fragment in a representative environment. As with the molecular fragment approach, this can result in considerable computational savings. More interestingly, it also provides the opportunity to explore in details the errors introduced by different levels of approximation.

This approach to large scale calculations extends the range of possible applications to new fields, and to communities traditionally focused on larger systems. In a similar manner to the uptake of DFT in the Quantum Chemistry community, things are progressing as if we are entering a "second era" of DFT calculations, where DFT and, more generally, large-scale quantum mechanical treatments, are susceptible to wide diffusion in other communities. Work is ongoing to extend this formalism in communities like electrochemistry and biology.

Point defect engineering involves three researchers in the group (DC, FL & PP). The activity has been settled more 15 years ago as a support to the microelectronic field (Leti), mainly in silicon. It has been extended during the period to 2D materials and 3D semiconductors compounds to explain their structure, growth mechanism or doping ability. A part of the work is a support to experimental work through ANR projects (ANR Emouvan, 2D transformers, and Mechaspin), industrial grants (SOFRADIR) or local informal collaborations (LEMMA, PHELIQS, SPINTEC, LTM). This leads to several experimental/theoretical papers on both 3D and 2D semiconductors. Another part of the work is more theoretical. We implemented a QM/MM approach in BigDFT, in collaboration with UdeM, to treat surface reconstruction. The exploration of the Potential Energy Surface (PES) is also another bottleneck as we have to find a defect reconstruction. We have started to use point defects themselves in the PES exploration (collaboration with UIUC). The concept was demonstrated for a fullerene molecule but should be applied to surface or bulk systems. Finally, we also develop with UIUC a framework based on van der Waals dislocations to explain some properties of vertical heterostructures of 2D materials.

H2: Preferential hydrogen etching on Si(001) explained by surface Density Functional Theory calculations

Permanent staff: D. Caliste, P. Pochet



Formation energy of di-vacancy rows (DVR) and lines (DVL) versus hydrogen chemical potential. Hydrogen induces a bias between DVL and DVR.

The integration of III-V semiconductors on silicon is still a hot topic as it will open up a way to co-integrate Si CMOS logic with photonic devices. Nevertheless, anti-phase boundaries are created when growing III-V materials on low-miscut Si(001) surfaces. They act as non-radiative recombination centres and degrade the photonic performances of devices. Their existence is due to single-layer steps (SL) on the silicon surface during growth. To inhibit their formation, commonly used strategies are: tilted substrates or Si(211) substrates. Both techniques don't integrate well with Si CMOS industry standards. Another possibility is to increase the miscut angle by converting single-layer steps into double layer (DL) steps. Unfortunately, DL are thermodynamically less stable in usual growth conditions. We propose an etching process that is applied on low miscut (0.15°) (CZ)-grown silicon (001) wafers of 300mm[7]. In an MOCVD reactor designed for III-V growth, the wafer is annealed in presence of H_2. From AFM measurements, it is clear that such process is converting the standard Si surface into a DL one. We have conducted atomistic simulations to understand the role of hydrogen etching into the conversion of SL into DL.

The reconstructed Si(001) surface is creating dimers. Removing a dimer from the Si(001) surface creates a so-called dimer-vacancy. These surface defects tend to aggregate into lines (DVL) or rows (DVR) [2]. DVR are natural precursors to create steps, since atoms of the first layer naturally rearrange into a $\pi/2$ rotated 2×2 reconstruction. As extended defects, they induce important elastic deformations that propagate up to the 15th layer from the surface. We have used a QM/MM approach based on BigDFT and its ability to properly treat surface boundary conditions. The above Illustration shows the formation energies of DVR (brown and pink curves) and DVL (green and blue curves) with respect to hydrogen chemical potential. In a hydrogen-poor regime, both defects have a similar formation energy. DVRs are not favoured and we don't expect the creation of DL steps. Indeed, the AFM image of such surface features SL steps in (110) and (1-10) directions. When hydrogen content is increased, there is a partial pressure of hydrogen span where the DVRs (pink curve) will dominate over DVLs. DVRs can then migrate and aggregate to form DL steps, as seen on the AFM image.

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[7] M. Martin et al., APL 109, 253103 (2016)

This atomistic investigation has demonstrated the peculiar role of hydrogen passivation of the step precursors, favoring the DVRs in a specific hydrogen partial pressure regime.

Over the recent years, the scientific production of I. Duchemin has been largely focussed on both methodology and numerical developments, with the idea to develop efficient algorithms that lead to high performance implementation of Many Body Perturbation related quantities[8]. As a milestone in this direction, the first Bull-Fourier award was obtained in 2014, recognizing his effort as the main developer of the Fiesta HPC initiative.

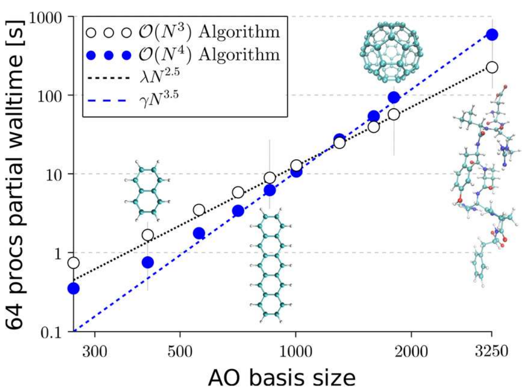
This algorithmic and coding effort is complemented by numerous studies on systems of interest with a particular focus set on Organic Photovoltaics and OLED devices[9]. Most of these studies are the fruit of a collaboration between I. Duchemin, X. Blase from CNRS Neel Institute and D. Jacquemin from Nantes University. In particular numerous benchmarks assessing the accuracy of both GW and Bethe Salpeter methodologies serves now as reference in the literature[10].

This interest for organic materials and organic devices has led to the integration of embedding technique such as the polarizable continuum model (PCM) withing the GW and Bethe Salpeter Formalism[11], demonstrating the importance of the inclusion of the environment electrostatic response in order to access quantities that can be confronted to experience. These developments have been continued within the EXTMOS European project and undertaken by Gabriele D'Avino and Jing Li from CNRS Neel Institute.

Finally, recent developments around the Resolution-of-the-Identity technique[12] have opened a path for an efficient MBPT calculations, with a first demonstration on cubic scaling RPA total energies[13] and an ongoing development on cubic scaling GW quasiparticle energies.

H3: Hybrid and Constrained Resolution-of-Identity Techniques for Coulomb Integrals

Permanent staff: I. Duchemin, X. Blase (CNRS/I. Néel)



These recent years have been dedicated to an important effort on the comprehension and development around the so-called Resolution-of-the-Identity (RI) technique, in application to computation within the Quantum Chemistry (QC) and Many Body Perturbation Theory (MBPT) frameworks.

The Resolution of the Identity technique stems from Gaussian basis QC community and was initially dedicated to the speed up of Hartree and latter Fock Exchange energies by replacing electronic co-densities by a linear combination of Gaussian auxiliary functions. This powerful technique has nowadays been extended well beyond the boundary of Gaussian basis and Hartree Fock electronic quantities. In particular, it has been at the Heart of our O(N^4) GW framework Fiesta since 2011.

Our recent development ranged from the mathematical foundation of the RI[14] to its application for the description of environment electrostatic effect on MBPT energies for solvated molecules[15], while demonstrating O(N^3) low scaling order algorithm for Random Phase Approximation (RPA) of molecular total energies[16].

This latter development stems from the understanding of the mathematical foundation of the resolution of the RI technique, with very promising application in particular to a cubic scaling GW quasiparticle energies algorithm that is currently being investigated. All the work mentioned so far have been implemented in the new c++ code beDeft that targets massively parallel environment, opening the possibility of large-scale calculations for a more realistic description of complex systems.

TB_Sim is a high-performance platform for the modelling of the electronic, optical and transport properties of semiconductor nanostructures. It has been used in the first half of the reporting period for the modelling of strained Germanium lasers and for the simulation of advanced silicon devices for micro-electronics, in tight

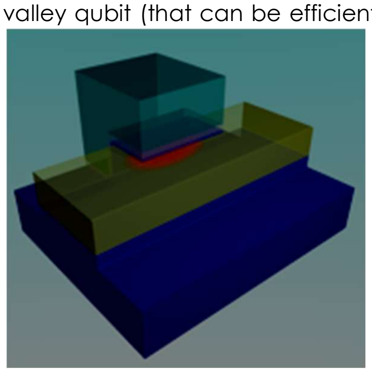
[8] X Blase, I Duchemin, D Jacquemin, Chemical Society Reviews 47 (3), 1022 (2018)
[9] C Faber, P Boulanger, C Attaccalite, I Duchemin, X Blase, Philosophical Transactions of the Royal Society A (2014)
[10] D Jacquemin, I Duchemin, X Blase J. chemical theory and computation 11 (11), 5340-5359 (2015)
[11] I Duchemin, CA Guido, D Jacquemin, X Blase, Chemical science 9 (19), 4430-4443 (2018)
[12] I Duchemin, J Li, X Blase, J. chemical theory and computation 13 (3), 1199-1208 (2017)
[13] I Duchemin, X Blase, J. Chem.Phys. 150, 174120 (2019)
[14] I Duchemin, J Li and XBlase, JCTC, 13, 3, 1199-1208 (2017)
[15] I Duchemin, C.A. Guido, D.Jacquemin and X.Blase, Chem. Sci.,9, 4430 (2018)
[16] I Duchemin and X Blase, J. Chem. Phys. 150, 174120 (2019)

collaboration with CEA/LETI and ST Microelectronics (through, in particular, the ANR projects QUASANOVA and NOODLES). We have notably achieved the first Non-Equilibrium Green's Functions (NEGF) simulation of an industrial CMOS technology, the 28 nm silicon-on-insulator node from ST Microelectronics. We have answered pending questions about the physics of the devices, and we have de-embedded the different mechanisms limiting the mobility of the carriers and the contact resistances. Taking advantage of these extensive comparisons with experimental data, we have then been able to make reliable predictions for the ultimate nanowire nodes developed, in particular, at CEA/LETI. In the second half of the reporting period we have leveraged on the experience gained on classical CMOS devices to move to silicon quantum bits. We have explored different aspects of the physics of silicon spin qubits, such as spin-orbit interactions (see highlight below). We are now tightly connected to the different teams fabricating and characterizing these qubits at LETI and PHELIQS, and help them designing the qubits and interpreting the experimental data through microscopic simulations down to the atomic scale when needed.

H4: Modeling spin-orbit interactions in silicon spin qubits.

Permanent staff: Y. M. Niquet, PhD students: L. Bourdet, B. Venitucci, post-Doc: J. Li

Spin-orbit coupling is a relativistic effect that couples the spin and orbital motion of a particle. In semiconductor qubits, a strong enough spin-orbit coupling allows for the electrical manipulation of electron and hole spins (through the so-called Electric Dipole Spin Resonance or EDSR). Following experimental demonstrations at INAC/PHELIQS[17],[18], we have modelled EDSR in realistic silicon qubits using atomistic tight-binding and multi-bands **k.p** Hamiltonians within the TB_Sim code. We show that the EDSR of electrons results from a subtle interplay between spin-orbit and valley physics. This interplay is strengthened in the silicon-on-insulator (SOI) qubits fabricated at CEA/LETI by the low symmetry of the "corner" quantum dots that form in these devices (see figure). We have proposed an original manipulation protocol based on the use of the SOI substrate back gate in order to transform reversibly a spin qubit (pretty immune to charge noise but hardly controllable electrically) into a valley qubit (that can be efficiently manipulated by EDSR)[19]. As for holes, we have introduced a versatile "*g*-matrix" formalism to describe the electrical response of the spins[20]. This formalism provides a very efficient framework for the analysis of the experiments, and for analytical as well as numerical modelling. In this framework, we demonstrate that silicon shows the best opportunities (among conventional semiconductors) for fast electrical hole spin manipulation thanks to its very large valence band anisotropy[21].



A SOI qubit as modelled with the TB_Sim code. The silicon nanowire channel is coloured in yellow, the SiO_2 oxide in dark blue, the HfO_2 oxide in purple, and the control gate in light blue. An iso-surface of the squared wave function of the electron trapped under the gate is outlined in orange. The electron is localized in a highly asymmetric "corner" dot at the edge of the nanowire covered by the gate.

4- Organisation and life of the L_Sim team

Steering, life, organisation in the unit

We have few team meetings (3 per years) but discuss actively thanks to the Slack collaborative tools. This collaborative tool is also used to exchange information about code developments, discussion about new articles and experimental and simulation results with our collaborators. Slack is well adapted for the exchange of scientific work, can create easily thread about a specific topic and has the advantage to keep history about a subject. Coffee around a whiteboard, each morning is also a way to exchange about new ideas. For 3 years, we started to use repositories (github, gitlab and also NOMAD) and Jupyter notebooks to share our data from our articles.

[17] A. Corna, L. Bourdet, R. Maurand, A. Crippa, D. Kotekar-Patil, H. Bohuslavskyi, R. Laviéville, L. Hutin, S. Barraud, X. Jehl, M. Vinet, S. de Franceschi, Y.-M. Niquet and M. Sanquer, npj Quantum Information 4, 6 (2018)

[18] A. Crippa, R. Maurand, L. Bourdet, D. Kotekar-Patil, A. Amisse, X. Jehl, M. Sanquer, R. Laviéville, H. Bohuslavskyi, L. Hutin, S. Barraud, M. Vinet, Y.-M. Niquet and S. de Franceschi, Physical Review Letters 120, 137702 (2018)

[19] L. Bourdet and Y.-M. Niquet, Physical Review B 97, 155433 (2018)

[20] B. Venitucci, L. Bourdet, D. Pouzada and Y.-M. Niquet, Physical Review B 98, 155319 (2018)

[21] B. Venitucci and Y.-M. Niquet, Physical Review B 99, 115317 (2019)

We also participate in the Centre for Predictive Simulation (CSP, 2 meetings per year). This centre is virtual but permits to have a quite large computer cluster in the CEA centre (3,000 cores) and to set up internal, national or European projects. We have the goal to have a common place to facilitate scientific discussions.

Parity, scientific integrity, health and safety, sustainable development and environmental impacts, intellectual property and business intelligence.

There is no woman in the group but we had two female PhD students (C Faber and D Timerkaeva) and two female postdocs (L Ratcliff and E Zvereva). Damien Caliste is a first aider for the C5 building. 4 patents were deposited during the period with experimentalists. We prioritize to use an open licence as GPL in order to share and spread our developments. For some case, proprietary licence is more adapted for some domains where large groups are leader.

We have a contract with the SOFRADIR Company. All our European projects are in collaboration with companies and some ANR projects also.

Five-year project and strategy of the L_Sim team

L_Sim SWOT analysis

Strengths	***Weaknesses***
Large panel of own developed codes Experienced staff about physics, materials, HPC and algorithms Large networks of collaborations on DFT development with the main codes (Quantum Expresso, ABINIT, CP2K, Fleur, Yambo), on point defect engineering with theoretical and experimental groups.	No computer scientist in the group and no possibility to hire one. No specialist about the exploration of potential energy surface (PES) in the group, the topic is only developed through collaborations (Basel, UdeM, UIUC) Short duration of the funded projects which are not compatible with a long-term development of research topic Collaborations with experimentalists fluctuates with rules of funding agency that do not favour local or long-term collaborations.
Opportunities	***Threats/Risks***
Collaboration with biologists with the new created institute IRIG.	Difficulty to fund developments of codes. Short duration of post-doc contracts is a lack of attractivity.

Structure, workforce and scientific orientations

Our four main scientific themes will be pursued during the five next years based on the current projects and the networks of our partners.

The mechanistic models based on Linear scaling DFT are able to extract specific physical observables that may be related to the distribution of the electrons. Such observables are routinely employed in Material Science and Quantum Chemistry Communities, but are seldom used in life science or biology. This research will be fundamental for the identification of a common dictionary that may translate the quantities which are accessible from mechanistic approaches to the typical indicators that are of interest in the context of enzymatic activity, more generally in bioremediation. The future activity would be to identify the grounds for the establishment of a research paradigm that integrate electronic structure calculation techniques with approaches in bioinformatics.

More specifically, we will test such a research paradigm in the laccase-aflatoxin example. We will employ the outcome of the present investigation to try to design the optimal features that a hypothetical enzyme should have in view of de-toxification. Such an approach will be interesting in designing a “function provider” out of a mechanistic-based investigation. We are presently proposing a mechanistic approach to improve the

efficiency of microbial laccase, a promising environmentally-friendly enzyme, for mycotoxin degradation in view of de-toxification, in collaboration with Boston College and RIKEN. We will utilize recent advances in computational power and quantum mechanical (QM) approaches to perform full QM mechanistic modeling of the enzyme. We outline a ground-breaking multidisciplinary effort to formulate and validate first-principle models and capitalize on the resulting insights to rationally design enzymes optimized for mycotoxin degradation. Developing a know-how for rational enzyme design will have huge implications for health, the environment, and industry, beyond the case of mycotoxin degradation studied here.

The activity on point defect engineering will be pursue through the different ongoing and future experimental collaborations depending on the funded grants. For example, we are involved in an ANR proposal to study the impact of vacancies in Hybrid-organic-inorganic-Perovskite materials. This proposal was not funded in the two last calls. Besides, we will continue to work through collaborations (UIUC, UdeM) on the exploration of the PES using point defects. The framework of van der Waals dislocations will be used to predict interesting vertical heterostructures of 2D materials. In particular we aim to apply this framework in the growing field of 2D superconductors.

The recently developed Resolution of the identity based on a real-space dual formalism allows not only to decrease the computational complexity of some of the MBPT electronic quantities such as RPA total energy or GW quasiparticle energies, it also facilitates the calculation of other quantities that have a simpler expression in their real-space formulation rather than the usual Kohn Sham or auxiliary basis counterparts. This is in particular the case of RPA forces for which an $O(N^3)$ expression can be achieved, a step toward accurate description of relaxation effect in the excited state.

The years to come should thus logically be dedicated to explore the possibilities opened by these new developments. In parallel, the work on embedding coupled with MBPT approach will be pursued, in particular the exploration of a formulation based on fragments, that should also decrease the complexity of such calculation for large systems.

Most of the activity related to TB_Sim code will be focused on the microscopic modelling of silicon qubits. This work will be carried out in the frame of the ERC synergy project quCube (2019-2025), which is led by CEA/LETI (M. Vinet), CEA/IRIG (S. De Franceschi) and Tristan Meunier (CNRS/Néel). We are and will be involved in the design of the qubits, in the interpretation of the experiments, and in the exploration of the physics of these devices (spin manipulation, readout, decoherence, etc...). We will, in particular, set-up a comprehensive microscopic framework for the modelling of semiconductor qubits, able to account for material properties at the atomistic level, and to make reliable predictions about the dynamics of the qubits, the variability of the devices, and the mechanisms limiting the coherence. We will endeavour to develop our network of collaborations with the partners of CEA in Europe, and will pay attention to the rapid development and evolution of this field, in particular in terms of options for materials and devices.

Research products and activities of the L_Sim team

Articles

Scientific articles (total number) 136

20% most significant articles

2019

1. M D'Alessandro, L Genovese, Locality and computational reliability of linear response calculations for molecular systems; Physical Review Materials 3 (2), 02380 (2019).
2. B Venitucci and YM Niquet, Simple model for electrical hole spin manipulation in semiconductor quantum dots: Impact of dot material and orientation, Physical Review B, vol. 99, no. 11, 115317 (2019).
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38. C Tavernier, FG Pereira, O Nier, D Rideau, F Monsieur, G Torrente, M Haond, H Jaouen, O Noblanc, YM Niquet, MA Jaud, F Triozon, M Casse, J Lacord, JC Barbe, TCAD modeling challenges for 14nm FullyDepleted SOI technology performance assessment, 2015 International Conference on Simulation of Semiconductor Processes and Devices (SISPAD)

39. G Mugny, D Rideau, F Triozon, YM Niquet, C Kriso, FG Pereira, D Garetto, C Tavernier, C Delerue, Full-zone k p parametrization for III-As materials, 2015 International Conference on Simulation of Semiconductor Processes and Devices (SISPAD)

2014

40. J Li, C Delerue, YM Niquet, F Triozon, L Genovese, A computational model for the investigation of phonon-limited charge transport in 2D systems, 17th International Workshop on Computational Electronics, IWCE 2014

41. J Pelloux-Prayer, M Cassé, S Barraud, P Nguyen, M Koyama, YM Niquet, F Triozon, I Duchemin, A Abisset, A Idrissi-Eloudrhiri, S Martinie, J-L Rouvière, H Iwai, G Reimbold, Study of the piezoresistive properties of NMOS and PMOS Ω-gate SOI nanowire transistors: Scalability effects and high stress level, 2014 IEEE International Electron Devices Meeting, 20.5. 1-20.5. 4 2014

42. B Voisin, VH Nguyen, J Renard, X Jehl, S Barraud, F Triozon, M Vinet, I Duchemin, YM Niquet, S de Franceschi, M Sanquer, Edge-states at the onset of a silicon trigate nanowire FET, 2014 Silicon Nanoelectronics Workshop (SNW)

43. YM Niquet, VH Nguyen, F Triozon, I Duchemin, J Li, O Nier, D Rideau, Modeling of FDSOI and trigate devices: What can we learn from Non-Equilibrium Green's Functions?, 2014 International Workshop on Computational Electronics (IWCE)

44. G Mugny, D Rideau, F Triozon, YM Niquet, FG Pereira, A Soussou, I Duchemin, D Garetto, C Tavernier, H Jaouen, C Delerue, Image and many-body effects in ultra-thin gate insulator MOSFET with In0. 53Ga0. 47As channel material and their influence on gate leakage current, 17èmes Journées Nationales du Réseau Doctoral en Micro-Nanoélectronique, JNRDM 2014

45. S Guarnay, F Triozon, S Martinie, YM Niquet, and A Bournel, Monte Carlo study of effective mobility in short channel FDSOI MOSFETs, in 2014 International Conference on Simulation of Semiconductor Processes and Devices (SISPAD), p. 105–108, IEEE, 2014.

46. D Rideau, F Monsieur, O Nier, YM Niquet, J Lacord, V Quenette, G Mugny, G Hiblot, G Gouget, M Quoirin, et al., Experimental and theoretical investigation of the 'apparent'mobility degradation in Bulk and UTBB-FDSOI devices: a focus on the near-spacer-region resistance, in 2014 International Conference on Simulation of Semiconductor Processes and Devices (SISPAD), p. 101–104, IEEE, 2014.

Other products presented in symposia / congress and research seminars 18

Electronic tools and products

Softwares

1. TB_Sim (propriatory licence, 500,000 code lines roughly 30 man.years), multi-scale, multi-physics tool to simulate electronic transport and properties of nanoscale devioces (CMOS transistors, nanowires, photonics, qubits)
2. BigDFT (GPL licence, 500,000 lines roughly 30 man.years), Daubechies wavelet based to calculate the electronic structure in the Kohn-Sham-DFT formalism using psuedopotenetials, documentation in the web site http://bigdft.org, the repository in https://gitlab.com/l_sim/bigdft-suite
3. GW/BSE Fiesta (100 000 lines 10 man.years), Many-body perturbation theory using a Gaussian basis set, http://perso.neel.cnrs.fr/X. .blase/fiesta/
4. BeDeft (50,000 lines 3 man.years), rewritten code using some components of Fiesta using c++ language and lua scripts with a new resolution of identity.
5. V_Sim (100,000 lines, 15 man.years),visualisation tool, https://gitlab.com/l_sim/v_sim
6. Gain, library to compute integrals for Gaussian basis set used in Fiesta,beDEFT and BigDFT
7. BOAST, automatic generation of optimized kernels, developed by Brice Videau in collaboration with INRIA (repository https://github.com/Nanosim-LIG/boast)

Databases

- Results added in the database NOMAD repository https://nomad-repository.eu/

Other products

Theorised artistic creations, staging, movies

The studies by F. Lançon on incommensurate interfaces and by L. Genovese on electronic-structure calculations were presented in an original and humorous way by the troupe "Les n + 1" of the company "Les ateliers du spectacle" from 2012 to 2016. Their play "L'apéro mathématiques" included a show on "what's in the head of Éric Frelançon and Gino Giuvelese" (see http://www.ateliers-du-spectacle.org/spectacle/lapero-mathematiques/).

Reviewing activities

Reviewing of articles

- Physical Review Letter, Physical Review B, Physical Review Materials, Physical Review Applied, Applied Physics Letters, Nanoletters, Journal of Physical Physics, Computer Physics Communication

Reviewing of research institutes

- Head of the HCERES committee of Laboratoire des Solides irradiés (LSI), TD, 11/2019

Academic research grants

European (ERC, H2020, etc.) and international (NSF, JSPS, NIH, World Bank, FAO, etc.) grants – partnership

1. HiC FP7, Novel in situ and in operando techniques for characterization of interfaces in electrochemical storage systems, 2013-2017, PP
2. EXTMOS FP8, multi-scale modelling for OLEDS, 2015-2019, ID, TD, LG
3. EoCoE, FP8 Centre of Excellence about high performance computing towards exascale in the field of Energy, 2015-2018, TD, LG
4. MaX, H2020 Centre of Excellence about HPC in the field of Materials, 2018-2021, DC, TD, LG
5. MOS-QUITO, MOS-based quantum information technology, 2016-2019, YMN
6. ERC Synergy quCube to realize Si qubits 2019-2023, YMN

National public grants (ANR, PHRC, FUI, INCA, etc.) – coordination

1. ANR MAQSI, Modeling and Assessment of Qubits on Silicon, 2018-2021, YMN
2. ANR 2D Transformers, Two-dimensional Phase Transformation Materials, 2014-2020, PP

National public grants (ANR, PHRC, FUI, INCA, etc.) – partnership

7. ANR NOODLES, NanOdevice mODeling for Low powEr applicationS, 2015-2017, YMN
1. ANR ESPADON, Anisotropic quantum dots for better spin and photon control, 2016-2018, YMN
2. ANR EMOUVAN, UV light emission with nanowires, 2016-2019, DC
3. ANR Mechaspin, Positioning at the atomic scale and coherent mechanical control of the spin of a magnetic atom, 2017-2021, PP, DC
4. Ancre MACMA (M. Saubanère, University of Montpellier), improve DFT to study electorchemical interfaces, 2019-2020, LG

Local grants (collectivités territoriales) – coordination

1. Wage AGIR UGA project to have visitors about wavelets Mathematics, 2016-2017, LG

PIA (labex, equipex etc.) grants – partnership

1. LANEF, L_Sim team
2. S. de (PhD grant of Maxime Morinère), TD, LG

Grants from foundations and charities (ARC, FMR, FRM, etc.) – partnership

1. 2D factory DSM-DRT CEA project, PP
2. CEA project PHOTONIQUE (about Ge laser), ID, YMN
3. ZeroPOVA DSM-DRT CEA project, Zero Power and Zero Variability for sub 5 nm Heterogeneous Single Atom Devices, 2013-2014, YMN
4. SITEQ DSM-DRT CEA project, Silicon Technologies for Quantum Information, 2017-2020, YMN

International grants – coordination

- "Moire Engineering in 2D Materials Beyond Graphene" (ARO USA 2017-2020) PI: H Johnson, Co-PI: PPochet

International grants – partnership

- "Brazilian student exchange" (CAPES, Brazil 2019-2024) PI: G Dalpian

Visiting senior scientists and post-doc

Post-docs (total number) 12

1. Brice Videau (EoCoE, 2016-2018), automatic generation of optimized kernels, BOAST
2. Augustin Degomme (EXTMOS, MAX, 2018-2021), GPU containers, optimizations, convolutions library

Foreign post-docs

3. Laura Ratcliff (2012-2014), linear scaling method, fragment approach, constrained DFT
4. Stephan Mohr (2013-2015), linear scaling method
5. Alessandro Cerioni (2014-2015), Poisson solver
6. E. Vadnyukova-Zvereva (HIC, 2014-2016), solid electrolyte interface model for battery
7. Jing Li (ZeroPOVA 2013-2015, 2018-2020)
8. José Maria Escalante Fernandez (PHOTONIQUE 2014-2015), Laser Ge
9. Zaiping Zeng (NOODLES 2015-2017)
10. Merijntje Bronsgeest (CROISIC, 2012-2014), Croissance de 2D
11. Shuze Zhu PRDA at UIUC (ARO 2017-2019) 2D materials and Moiré
12. Juan Camillo Alvarez Quiceno (2D Transformer, 2018-2020), transformation de phase dans 2D
13. Selva Selvaraj (2018-2020), SOFRADIR contract about defects in HgCdTe

Contracts for a post-doc in a foreign university

14. Paul Boulanger (one year) Montréal University
15. Maxime Morinière (two years), Montréal University

Visiting scientists (total number) 5

Foreign visiting scientists

1. Pr. Harley Johnson, University of Illinois at Urbana-Champaign (UIUC), invited six-month UJF professor (2016)
2. Pr. Gustavo Dalpian, University of ABC, Sao Paulo (initial visit to settle a Brazilian student exchange grant running 2019-2023)
3. Marco d'Alessandro, Roma (several weeks 2018-2019), linear response methodology
4. William Dawson, RIKEN (1 week, 2019), fragment approach and aflatoxine calculation
5. Anders Bergman, Uppsala University (one year, 2016-2017), magnetism
6. Giuseppe Fisicaro, Catania University (several weeks, 2016-2019)
7. Laura Ratcliff (several weeks, 2018-2019)
8. Boris Nectoux (one week, 2016)

Scientific recognition

Prizes and/or distinctions

- 2014 Bull-Atos Fourier prize (FIESTA, I Duchemin and X. Blase)

Chair of learned and scientific societies

1. Athanasios Dimoulas (LANEF chair of Excellence 2016-2017) about two-dimensional (2D) transition metal dichalcogenide (TMD) materials

Organisations of meetings and symposia (out of France)

1. Symposium "Materials by design for Energy applications" co-organized by P. Pochet within E-MRS spring meeting 2016,
2. International CECAM workshop "Multiscale modelling of organic semiconductors: from elementary processes to devices", September 2017, Grenoble

Interaction of the L_Sim team with the non-academic world, impacts on economy, society, culture or health

Patents

Accepted patents 3

1. Procédé de recristallisation d'un matériau carboné, tel que du graphène ou des nanotubes de carbone, Jean Dijon, H. Okuno, P. Pochet, Raphael Ramos, WO2017037396A1 (2016)
2. Method for manufacturing solar cells, attenuating lid phenomena, P Pochet, S Dubois, US Patent 9,412,896 (2016)
3. Single-electron transistor and its fabrication method, S Barraud, I Duchemin, L Hutin, Y Niquet, M Vinet, US Patent 9,911,841

Filed patent 1

4. Procédé de contrôle d'un dispositif à qubit de spin, L Bourdet, L Hutin, YM Niquet, M Vinet, submitted in June 28. 2017, France

Socio-economic interactions

Industrial and R&D contracts

- 2 R&D contracts with SOFRADIR (2016 and 2018)

Expertise

Participation in expert committees (ANSES etc.)

1. President of Material committee for the national computational allocation (TD)
2. User representative in CCRT national supercomputer centre (DC)

3. Member of the evaluation committee of GENCI (PP)
4. Director of the Centre for Predictive Simulation, CSP, T Deutsch, 2017-present

Involvement of the L_Sim team in training through research

Educational outputs

E-learning, MOOCs, multimedia lessons, etc.

1. TP Cesire with F. Lançon between 2 and 3 groups per year, one day training (2014-2018)
2. Cours CUDA/OpenCL école doctorale UJF, L Genovese and B. Videau (March 2015)
3. MOOC UJF, À la recherche d'autres planètes habitables, M Morinière(June-July 2016)

Training

Habilitated (HDR) scientists

1. Thierry Deutsch (2008)
2. Frédéric Lançon (1984)
3. Y. -Michel Niquet (2013)
4. P. Pochet (2012)

HDR obtained during the period 0

PhD students (total number) 12

1. Carina Faber (2011-2014), supervisors X Blase (Néel) and T Deutsch, defense 26/11/2014
2. Dilyara Timerkaeva (2012-2015), supervisors P Pochet and D Caliste, defense 10/4/2015
3. Gilles Brenet (2012-2016), supervisors P Pochet and D Caliste, defense 10/5/2016
4. Shashank Mathur (2013-2016, Exp.), supervisors J Coraux (Néel) and P Pochet, defense 16/9/2016
5. Maxime Morinière (2014-2016) supervisors T Deutsch and L Genovese, defense 15/12/2016
6. Huong Pham (IFPEN collaboration), supervisors V Perrier (IMAG) and L Genovese, defense 30/6/2017
7. Léo Bourdet (2015-2018), supervisor YM Niquet, defense 22/11/2018
8. Brian McGuigan (PhD UIUC, 2013-2018) supervisors H Johnson (UIUC) and P Pochet, defense 10/12/2018
9. Kimon Moratis (2016-2019), supervisor YM Niquet
10. Benjamin Venitucci (2017-2020), supervisor YM Niquet
11. Emil Annevelink (PhD UIUC, 2017-2021) supervisors E Ertekin (UIUC), H Johnson (UIUC) and P Pochet,
12. Sameer Gupta (2018-2021), supervisors P Pochet and D Caliste

Defended PhDs 8

Mean PhD duration 3 years

Internships (M1, M2)

1. Raivo Andrian (BSc MIT)
2. Corentin Nélias (M2, INSA-Lyon)
3. Alex Cornic (M1, UGA)
4. James Diamond (BSc, UdeM)
5. Louis Gravel (MSc, UdeM)
6. Ganesh Ananthakrishnan (Msc, UIUC)
7. Tawfiqur Rakib (Msc, UIUC)
8. Adrien Foueillessar (M1)
9. Quentin Debray (M2, ENSTA)

TEAM 2: ADVANCED ELECTRON MICROSCOPY (LEMMA)

1- Presentation of the LEMMA team

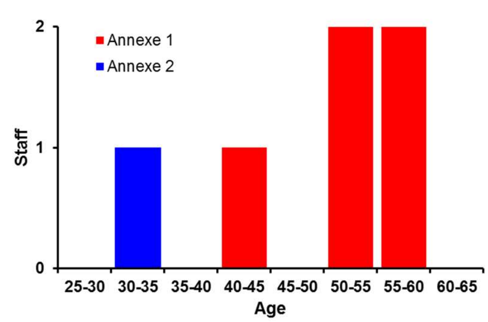
Introduction

The LEMMA group develops and uses electron and ion beam microscopy, as well as associated spectroscopies, to study inorganic and, more recently, organic materials. The main task of our team is to improve new or advanced characterization techniques using TEM, SEM, FIB and MEIS instruments, required for IRIG's materials research activities (INAC before 2018). Prior to 2016, LEMMA was a part of the SP2M (Service de Physique des Matériaux et des Microstructures), which conducted research activities primarily on the photonic and electronic properties of semiconductor and inorganic materials. The team has thus been specialized for a long time in atomic scale imaging, strain mapping and thin film chemical analysis, which are essential for the development of crystalline semiconductor materials. Today the techniques we develop are extended to other areas such as materials for energy applications and biology, in accordance with the strategy established by the IRIG institute, as well as for microelectronics in a strong collaboration with the LETI institute. Located on Minatec's Nano characterization platform (PFNC), LEMMA has access to a wide range of state-of-the-art equipment, including 2 aberration-corrected transmission electron microscopes (TEMs) and 4 dual beam FIB-SEM machines. The team possesses a large variety of skills in advanced atomic-scale microscopy techniques and quantitative imaging, chemical analysis, tomography and field mapping that contributes to local and national scientific communities.

Unit's workforce and means

Permanent staff		Non-permanent staff	
JALABERT Denis	Researcher	Thesis defenses 2014-2019 : 8	
JOUNEAU Pierre-Henri	Researcher	Post-Docs 2014-2019 : 6	
MOLLARD Nicolas	Technician	PhD : 8 (13 total supervisions)	
OKUNO Hanako, team leader	Researcher	Visitors: 3	
ROBIN Eric	Researcher	Internships (M1, M2): 5	
ROUVIERE Jean-Luc	Researcher		
		Extended collaborators (permanent staff of CEA)	
Left in 2014 to INAC direction		BOUGEROL Catherine	CEA-CNRS: PHELIQS
BAYLE-GUILLEMAUD Pascale	Researcher	GAUTIER Eric	CEA-CNRS: SPINTEC

The team is composed of six permanent staff (5 scientific researchers and 1 technician) and an average of 4 non-permanent staff (postdocs and PhD students). The team also hosts two researchers from neighbouring laboratories, PHELIQS and SPINTEC, who work mainly for the needs of their own scientific topics. All our research is carried out in the microscopy and spectroscopy facilities at the CEA-MINATEC PFNC. During the evaluation period, the permanent team has not changed. The last recruitment in the LEMMA team was in October 2012 (H. Okuno); with no additional staff since the last departure in September 2014 (P. Bayle-Guillemaud then joined INAC management). The age pyramid of the LEMMA is given below. This underscores the serious need to recruit additional young staff even though the number of experienced researchers is also a strength for the team. One of the main tasks of the team is to maintain the equipment. The lack of permanent staff becomes a crucial problem of the team. It resulted in closing an activity on Medium Energy Ion Scattering (MEIS), developed since many years. The MEIS equipment was moved to another laboratory (IN2P3 Lyon) in 2016.



The laboratory budget is of about 250 k€ per year, which is shared into running costs (approx. 60 %), non-permanent salaries (approx. 35 %) and investments (approx. 5 %). Most of the funding is coming from European and ANR projects.

Scientific policy

The scientific objective of our group is to develop and apply new or advanced characterization techniques for nanomaterials studies. As a result of the current technological improvement in instruments and the increasingly demanding characterization needs, the team aims to provide outstanding microscopy techniques using the latest generation instruments available at the PFNC. In line with the recommendation made in the previous HCERES report, our current activities are not limited to extreme microscopy as a goal, but aims to use these techniques to directly contribute to nanomaterials researches by addressing a wide range of issues. The team is strongly involved in IRIG materials research activities, and assists neighbouring laboratories in a wide variety of topics ranging from microelectronics, photonics, spintronics, and, more recently, energy materials and cell biology. This generates a dynamism that far exceeds the LEMMA team.

The team's activities are based on several industrial and institutional contracts. Industrial contracts mainly supported technical developments, e.g. in order to integrate new analytical systems in the instruments in collaboration with equipment manufacturers (FEI), or to develop analytical methods that meet the specific requirements of microelectronics companies (IBM, ALEDIA). The team members are directly involved in several national (ANR), regional (LANEF) and international (European) projects with national and international collaborations. In addition to historically developed scientific topics, such as semiconducting materials in the photonic electronics and spintronics fields, the team is opening up its expertise to new area of research through intensive involvement in several projects with wide consortiums. For example, two consecutive European projects on the advanced characterization of battery materials (BACCARA and SINTBAT) have brought opportunities to specialize in materials for energy application for 6 years. Other consecutive European projects (Gladiator and CONNECT) enabled the team to develop low-voltage microscopy for low-dimensional materials to provide the information required for the development and fundamental understanding of1D and 2D components.

2- Presentation of the LEMMA team's research ecosystem

PFNC: the nano-characterization platform of Minatec

The LEMMA team is a founding partner of the nano-characterization platform (PFNC) at Minatec initiated by the three institutes of CEA-Grenoble, IRIG, LETI and LITEN, on which the characterization tools are shared for the activities of basic and applied research activities of each division. Through the PFNC, we have access to funding for a significant number of advanced equipment, including state-of-the-art microscopes. PFNC researchers specialize in specific advanced techniques, enabling high-level research through strong and extensive collaborations. Our team plays an important role in the scientific animation of the PFNC in order to maintain the excellence and visibility of the platform at the global level. Our privileged collaboration with CEA-DRT institutes (LETI and LITEN) within the PFNC also offers us opportunities for involvement in industrial contracts.

Training, Support and Education

LEMMA is a part of a joint unit between the CEA and the UGA and is also attached to the PEM (Physics, Engineering and Materials) division of research of the University. In this context, the team is involved in research training through the supervision and co-supervision of PhD students as well as in training programs (ESONN). The team also offers its expertise and facilities to local and national scientific communities (PFNC-Grenoble and the French network of microscopy METSA). The training and support of IRIG microscope users is also an important task.

Main collaborators

Industrial partners: FEI, ALEDIA, IBM

Institutional partners: IRIG: MEM/L_Sim, PHELIQS/NPSC, SPINTEC, IBS, BIG/PCV, BIG/CBM, GIN, LETI, LITEN, IRAMIS, Institute Néel.

3- Research products and activities for the LEMMA team

Research products: Over the past five years, the LEMMA team has published approximately 130 articles, including 4 papers in the Nature publishing group, 7 in the ACS journals and 7 in Nanoletters. Some thirty contributions were presented at conferences, including 10 invited talks at international conferences on microscopy and materials science.

During this period, the technical developments have been extended in line with what was proposed in the previous HCERES report. Both high-resolution TEM and STEM techniques, at low and high voltage, have been greatly improved by the availability of a new Gatan OneView camera and the development of an in-house software for acquiring image series summations. Remarkable progress has also been made in nanodiffraction techniques for faster and more accurate analysis. The evolution of the EDX analytical method for quantitative chemical analysis initially developed in the team was demonstrated on the aberration corrected TEM (Titan Themis) equipped with the new generation Super-X detectors, and on the FlatQuad detector on the scanning electron microscope (SEM). One of the strengths of our team was to be able to develop these different techniques despite of our small size. This has been made possible through proper investments within the PFNC, and because these developments are often based on small changes in equipment and methods. By doing these developments, the LEMMA team expects to help researchers better understand the structures they have elaborated and to establish a link between atomic structure and properties. In addition to its historical scientific topics, such as semiconducting materials, over the last 5 years, low voltage aberration-corrected TEM has been developed in order to answer various questions about two-dimensional (2D) materials. Development of 3D FIB-SEM analytical methods have been strengthened, particularly for applications in the fields of biology and battery materials. This is beginning to shed light on new-scale analysis for a deeper understanding of these sensitive materials. Other activities such as EELS spectroscopy and MEIS have been continuously developed in this period. Especially the chemical analysis of battery materials has been intensively studied using STEM-EELS technique. The MEIS has been used for a precise quantitative chemical analysis of diverse interfaces associating to the strain information. However, the MEIS activity in the team was closed in 2016 due to the heavy task to maintain the equipment.

Technical developments in the team

High Resolution techniques

Permanent staff: H. Okuno, J.L. Rouvière, PhD student: B. Haas, F. Agnese, Post-doc: C. Alvarez, R. MacLeod, Visitor: Pr J Zuo

Improvements and studies in high-resolution (HR) imaging have always been an important specialty of the LEMMA group. The last 5 years have seen a series of small improvements in tools and methods that have led to interesting material results. HR-TEM experiments are now mainly used for 2D-materials studies and nanocrystals analysis (see 2D-material below, and[22]). The low-voltage Cs-corrected HR-TEM (80kV) with the use of a gun monochromator has been particularly optimized. The new Gatan OneView camera allows one to record movies and permits to understand the creation of defects under the electron beam (see Highlight 6). The quality of HR-STEM images has been impressively improved by acquiring series of image that nearly get rid of scanning errors and improve the signal to noise ratio. For instance, the average chemical width of CdTe/HgTe interfaces has been determined[23]. Initially, STEM image series were acquired with scripts, and then registered and averaged with a python software, Zorro[24]. However, the Thermofisher Velox acquisition/averaging software is currently the easier way to make these image series. To analyze these STEM images, two main homemade softwares have been used. The old GEM-GPA script written more than 10 years by Dr J-L Rouvière is regularly upgraded and improved. Thanks to Prof. J Zuo, a new template matching algorithm (TeMA) has been written[25] and used in several studies. In particular in Kim (2018)[26], TeMA was used to separate adjacent atomic columns in InAs/GaSb multilayers and provided cation and anion sub-lattice strain maps. Cation and anion vacancies were detected thanks to the induced local lattice relaxations. In addition, the quantitative method described by Dr. Lebeau and Rosenaeur has also been successfully applied to several materials issues (see semiconductor part and PhD B Haas).

Diffraction based techniques

Permanent staff: J.L. Rouvière, PhD student: B. Haas, F. Agnese, Y. Martin. Post Doc: R. MacLeod, V. Boureau

Diffraction patterns contain a lot of information and have been used and developed at LEMMA for about 15 years for two main applications: polarity determination and strain mapping. Recently, we also used N-PED patterns to measure electric and magnetic fields ([27]see highlight below). In the last 5 years, we have improved our 'old scripting' acquisition by developing either a new driver for our Hamamatsu camera (in collaboration with Dr E. Rauch), but the stability and resolution of the microscope on which it was installed was not sufficient, or, more successfully, a new precession acquisition module on our TITAN Themis. Developed within the joint CEA/FEI laboratory, this module improves the precession tuning, simplifies the selection of the map area by using STEM images and considerably increases the acquisition speed by a factor 6. As a result of these improvements

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[22] PhD Thesis, F. Agnese, Advanced transmission electron microscopy studies of semiconductor nanocrystals synthesized by colloidal methods, 2018
[23] Haas, B., T. , C., Jouneau, P.-H., Bernier, N., Meunier, T., Ballet, P., Rouvière, J.-L., 2017. Appl. Phys. Lett. 110, 263102.
[24] https://github.com/robbmcleod/zorro
[25] Zuo JM , Shah AB, Honggyu Kim H, Yifei Meng Y, Gao W, Rouviére JL., Ultramicroscopy 136 (2014) 50-60
[26] Kim, H., Meng, Y., Kwon, J.-H., Rouviére, J.-L., Zuo, J.M., 2018, IUCrJ 5, 67–72.
[27] B Haas, D Cooper and JL Rouviere Patent FR1658594 - US20180076005A1

N-PED has been widely applied and has led to a significant number of publications (see the semiconductor section below). In collaboration with D. Cooper of LETI, we started a detailed comparison of electron techniques that measure electric and magnetic fields. Due to the filing of a patent, only holography and new DPC (Differential Phase Contrast) measurements for electric field have been compared in the first published paper[28]. In a post-doc work, V. Boureau improved the calculation of the centre of mass and we have made maps of magnetic and electrical field around a magnetic nanowire by using holography and diffraction pattern series[29]. These first results are encouraging but will require further analysis.

Quantitative chemical analysis

Permanent staff: E. Robin, N. Mollard, PhD students: M.A. Luong, Post-doc: M. Lopez-Haro. Visitor: M.V. Braza-Blanco

Important work has been done to improve the detection, distribution and quantitation of chemical elements analyzed by Energy Dispersive X-ray (EDX) spectrometry in SEM and TEM. In the frame of the IZAC project, partly supported by FEI (post-doc M. Lopez-Haro) we have developed a new chemical quantification routine that can be applied over a wide range of accelerating voltage (5-200 kV) and on various materials, i.e. from thin to massive structures and from pure substances to multi-element compounds.[30] The routine was also implemented with a new model of X-ray absorption allowing absorption correction of low energy X-ray in heterogeneous specimens. The routine is today currently applied in SEM and TEM to the determination of the mass-thickness and composition of a wide variety of nanostructures with relative precision better than ±5%[31]. In the frame of the DOMINO project, supported in part by Aledia, a start-up from CEA-LETI, we have developed the potential of wide solid angle EDX detectors (FlatQuad 5060 in SEM and SuperX detectors in TEM) for characterizing low-levels of dopants (down to 10^{18} at/cm^3) at the nano-scale (10 nm) in SEM and TEM (see highlight 2). This can be achieved thanks to three major innovations that are: 1) the use of specific X-ray filters for enhancing the signal-to-noise ratio, 2) the implementation of new analytical procedures for removing the residual background of X-ray spectra, and 3) the development of a new computational code for processing and quantifying data using the IZAC code. At last, we have developed in collaboration with the Neel institute, in the frame of the ANR-ESPADON project, a fast and easy method for the quantitative 3D reconstruction of core-shell nanostructures[32].

3D imaging techniques (FIB-SEM nano-tomography & atom probe tomography (APT))

Permanent staff: P.-H Jouneau, PhD B. Bonef, L. Amichi

FIB-SEM nano-tomography has been developed in our team since the acquisition of the Zeiss Nvision 40 instrument in 2010. This technique is based on the observation of a bulk sample in a dual-beam FIB-SEM microscope: a gallium ion beam is used to progressively remove thin slices (typically 2 to 30 nm) from a sample. The exposed surface is sequentially imaged, slice after slice, with the electron beam, with a spatial resolution down to 2 nm. FIB-SEM thus provides in a few hours a complete automatic scan of a large volume (of up to several tens of microns) with an isometric resolution down to three nm. This technique is today required in diverse research fields (i.e. microelectronics, materilas for energy such as batteries, biological samples etc.), to provide robust and automated acquisitions of large volumes with a resolution comparable to that of TEM. During the last 5 years an important work has been done in the team on developing tools and methods to get better, faster and more quantitative results. An important progress has been achieved on data analysis. Besides image quality, successful 3D FIB-SEM reconstructions of biological samples rely on high quality analysis of very large data sets (automatic segmentation, statistical analysis) to obtain quantitative measurements. Thus, we have implemented in our laboratory software for (semi-)automatic segmentation (Ilastik, Fiji, etc), analysis and visualization (Paraview, Avizo, Blender...). A new instrument was installed in 2018, in collaboration with LETI and LITEN, in the frame of the PFNC. This microscope, a Zeiss Crossbeam 550, is the first dedicated specifically to 3D-imaging in our institute. During its installation, particular attention was given to improve the long-term stability (with, for example, a control room separated from the instrument in order to increase thermal stability).

In addition to the FIB-SEM imaging technique, we have also investigated these last 6 years the potential of atom probe tomography (ATP) to analyze semiconductors materials at the nanoscale. This technique should make it possible to obtain quantitative 3D chemical information, such as the distribution of dopant atoms in 2D or 1D structures grown by epitaxy techniques (MBE, MOCVD). This work was carried out mainly in the context of two PhD work in co-supervision between LEMMA and PHELIQS (Bastien Bonef, 2012-2015, and Lynda Amichi, 2015-2018), focused on the characterization of doping respectively in II-VI and GaN semiconductor structures. These studies have allowed us to understand the interests, but also the limits of this technique, which actually make it

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[28] Haas, B., Rouvière, J.-L., Boureau, V., Berthier, R., Cooper, D., 2019, Ultramicroscopy 198, 58–72.

[29] Boureau V, Rouvie□re JL, Stano M, Fruchart O and Cooper. Abstract IMC2018 Melbourne

[30] E. Robin, Patent N°EP3032244B1, 2016

[31] *B. Bonef, M. Lopez-Haro, L. Amichi, M. Beeler, A. Grenier, E. Robin, P.H. Jouneau, N. Mollard, I. Mouton, C. Bougerol, E. Monroy, Nanoscale Research Letters, 2016, 11, pp.461*

[32] *P. Rueda-Fonseca, E. Robin, E. Bellet-Amalric, M. Lopez-Haro, M. Den Hertog, Y. Genuist, R. André, A. Artioli, S. Tatarenko, D. Ferrand, J. Cibert, Nano Letters, 2016, 16 (3), pp.1637-1642*

possible to image dopants in 3D (such as very small clusters of a few tens of atoms), but which require careful precautions to obtain quantitative results.

Materials study using advanced techniques

Semiconductors

Permanent staff: J-L. Rouvière, E. Robin, PhD students: B. Haas, F. Agnese, M.A. Luong, Post Doc: R. MacLeod, M. Lopez-Haro, Visitor: Pr J Zuo, M.V. Braza-Blanco

The team has been involved in several projects (EMOUVAN, ESPADON) supported by academic partners (ANR, LANEF) or industrial partners (Aledia, IBM, FEI) for the study and characterization of semiconducting materials. For instance, improvements in high-resolution and diffraction-based techniques have allowed us to study various semiconducting multilayers and devices. This has resulted in numerous publications on microelectronics devices (coll. IBM, coll. LETI) and semiconducting 2D layers (GeSn, topological HgTe/CdTe, fluorine-doped SnO2 (FTO) thin films on (110) rutile TiO2, AlGaN/GaN layers, coll. Sofia Antipolis). Another example concerns the improvement of the EDX technique which made it possible to quantify the low levels of P dopants in Ge nanowires and the Mg, Si, Ge and In dopants in (Ga,Al)N nanostructures (see Highlight 2). The EDX technique has also been successfully applied for the quantitative reconstruction of chemical core-shell nanostructures on a wide range of semiconducting materials (ZnCdTe, AlInGaN, InGaAs, SiGe, GeSn etc...) with several applications in the field of nanoelectronics, optoelectronics and photonics. In addition, we have explored a new TEM sample preparation method we called FIB-MicroCleavage (FMC). It consists of using a FIB and a micromanipulator to make grooves and cleave locally a tiny sample under vacuum.

Lithium-ion batteries

Permanent staff: P.-H Jouneau, P. e Bayle-Guillemaud, PhD Student: M. Bonniface, Post-doc: P. Kumar

The growing need for high-performance lithium-ion batteries has led to a considerable research effort to develop new materials, as well as to understand the reaction processes of electrode and electrolyte, as well as their degradation during use. These studies involve structural and chemical characterizations at various scales, from the micrometer to the atomic scale. Our laboratory has been involved in this field for a long time with the development and the use of innovative tools, notably based on HR-(S)TEM imaging, EELS spectroscopy and FIB-SEM nano-tomography. This work has been done on a continuous basis in the frame of several major projects (e.g. European projects Baccara and Sinbat) and thanks to several PhD and postdocs. The originality of our involvement lies in the fact that it is always carried out in close collaboration with the other CEA laboratories working on these problems, either those working on complementary characterization techniques within IRIG (such as NMR spectroscopy, x-ray or neutron diffraction, in-situ analysis at ESRF), or those more focused on technological developments (mainly at the LITEN institute). This positioning within complementary teams has proved fruitful in this area where the problems are intrinsically multi-scale and of various aspects (structural, chemical, electrical…). This gives us now a real legitimacy to apply to new projects of the European research community in this field. Our works try mainly to unravel the crystal structures and chemical evolution of the electrode and solid-electrolyte materials, the fluctuations near the interfaces, and the evolution of the coexisting phases (see Highlight 4). For example, the STEM method developed in order to map the SEI and Li_xSi distribution has complemented other characterization investigations such as NMR and surface analysis (TOF-SIMS & XPS) to help the understanding of the failure mechanism of Si-based electrodes[33], and the investigation of new electrolytes to improve battery safety[34] [3]. A major issue in this field is to understand the mechanisms of fading capacity, and numerous perspectives exist to better investigate these materials, such as the use of local diffraction analysis, 3D reconstruction, and cryo-electron FIB-SEM and TEM.

1D & 2D materials: Structure and Dynamics

Permanent staff: H. Okuno, PhD Student: Y. Martin, Post-doc: C. Alvarez, D. Kalita

During 5 years, the team has been involved in several national and international collaborative projects to study 2D materials. Various techniques of microscopy, required to answer diverse questions, have been developed using low-voltage aberration-corrected TEMs (Titan ultimate and Titan Themis). The detection of structural anomalies at the atomic scale in these low-dimensional materials, including defect, dislocations and dopants, is crucial to understand and to control their growth and physical properties. In the early years, atom-by-atom TEM and STEM imaging of atomically thin layers were developed in conjunction with a sophisticated and appropriate sample preparation process. Multiscale analysis consisting of domain mapping and detailed analysis of the atomic structure on grain boundaries and defects revealed a new growth mechanism based on

[33] N. Dupré, P. Moreau, L. Quazuguel, M. Boniface, A. Bordes, C. Rudisch, P. Bayle-Guillemaud, D. Guyomard, Electrodes in Full-cell configuration Chemistry of Materials 2016, pp2557-2572

[34] N. Dupré, P. Moreau, E. de Vito, L. Quazuguel, M. Boniface, H. Kren, P. Bayle-Guillemaud, D. Guyomard, Chemistry of Materials 2017, 29(19) pp8132-8146

a peculiar recrystallization in CVD graphene, in collaboration with CEA-LITEN.[35] In multilayer 2D systems, the metastable phases that appear in epitaxially grown TMDs were determined in conjunction with synchrotron XRD experiments and DFT calculation. The result revealed the possibility of a commensurate charge density wave structure in $TiTe_2$[36] and of Weyl semimetal phase in $MoTe_2$[37] at room temperature, in the framework of LANEF Chair of Excellence with A. Dimoulas (National Centre for Scientific Research, Greece). Direct imaging of individual dopant atoms in low-dimensional materials (1D and 2D) has also been performed in the framework of European projects (FP7: Gladiator and H2020: CONNECT). A novel Pt-salt doping of graphene for high-conductive transparent electrodes were developed and an atomic scale dopant analysis has revealed evidences of the stabilization of the dopant on the surface of graphene[38]. The developed doping concept was integrated into carbon nanotube interconnect device and EELS analysis under the electron irradiation confirmed the degradation mechanism. Structural defects in 2D materials are considered as a parameter-tuning knob for material properties. In order to explore the ability to engineer atomic defects, an in-situ observation of structural modification was developed using a high-speed camera and an in-situ heating sample holder, in the framework of CEA-internal project (2D Factory) and ANR projects (MoS_2 Valley Control). The recording of temporary resolved atom displacements under electron irradiation and/or temperature, defect dynamics in 2D monolayers were studied. Recently, the impact of van der Waals substrate on the defect formation in $MoSe_2$ layer was demonstrated using such in-situ observation (see Highlight 5).

Biological materials

Permanent staff: P.-H Jouneau, PhD Student: Clarisse Uwizeye (co-supervision with BIG/PCV)

Originally used in materials science, FIB-SEM nano-tomography revolutionizes the field of 3D imaging in cell biology by providing automated acquisitions of large volumes with a resolution comparable to that of the TEM for these samples. To meet the growing demand, we have developed long-term collaborations with several teams of biologists (mainly from IRIG laboratories) both to improve the use of this technique in biology, and to apply it to some interesting topics.

A special effort was put on biological samples preparation using protocols similar to those used for TEM. In collaboration with Benoit Gallet (IBS) and D. Falconnet (BIG/PCV), we studied new protocols (harder resins, cryo-fixation, larger heavy metal labelling) to better preserve biological structures, and improve resistance to high doses of electrons.

Then, the technique has been applied in the framework of various project (DRF-impulsion FIB-BIO, several co-directed PhD theses and M2 internships) for:

- Imaging of subcellular organelles in algae and plants (collaboration with BIG/PCV). FIB-SEM helps to reveal the 3D arrangement and the interactions of plastids in various marine taxa (Highlight 3). We therefore expect a better understanding of the mechanisms of photosynthesis, as well as its structural adaptations following acclimation to environmental changes (light, temperature, nutrients, and pH).
- To understand the fate of silver nanoparticles in hepatocytes (collaboration with BIG/CBM). The health impact of Ag-NPs, which are increasingly used for their antibacterial and antifungal activity, raises questions. Between 20 and 80 µg of Ag-NP are ingested per day, which can cross biological barriers and accumulate in the liver. We investigate the fate of Ag-NP in hepatocytes on a 3D model of HepG2 cells in culture. Coupled with other imaging techniques and spectroscopies, FIB-SEM help us to describe the kinetics of the distribution of Ag-NP from cellular entry to excretion into the bile duct, and to evidence the associated structural changes, e.g. on the mitochondrial network.
- Study the configuration of synapses in animal models of brain diseases (collaboration with GIN). Astrocytes that fill the interstices of the neural network and cover the synapses play a key role in the neural network. Their alteration can contribute to the pathophysiology of diseases such as epilepsy, Alzheimer's disease or Huntington's disease. By imaging neuronal tissue with FIB-SEM, we try to identify astrocytic processes and to measure the surface of contact between astrocytes and synapses.

H1: Simultaneous strain and electric field maps

J.-L. Rouviere, B. Haas, Coll. D. Cooper (CEA/LETI)

[35] *A. V. Tyurnina, H. Okuno, P. Pochet, J.Dijon, Carbon, 2016, 102, pp.499-505*

[36] *S. Fragkos, R. Santo, C. Alvarez, A. Bosak, P. Tsipas, D. Tsoutsou, H. Okuno, G. Renaud, A. Dimoulas, Advanced Materials Interface, 2019, pp.1801850*

[37] *P. Tsipas, D. Tsoutsou, S. Fragkos, R. Sant, C. Alvarez, H. Okuno, G. Renaud, R. Alcotte, T. Baron, A. Dimoulas, ACS Nano, 2018, 12 (2), pp.1696-1703*

[38] H. Le Poche, A. Carella, J. Dijon, H. Okuno, E. Roux, Patent FR3 058 997-A1, 2017

We show that by adjusting correctly the size of the diffraction pattern and the camera length, the in-plane electric and strain field maps of a sample can be computed simultaneously from the same set of diffraction pattern (figure H1)[39] [40]. However, this simultaneous measurement may not be the easiest and optimum way to proceed. Indeed, in order to have a high precision in the electric field, the transmitted beam must be spread over a large number of pixels. In order to have a high precision in the strain field, several diffracted beams must be present. Both requirements lead to a quite large diffraction pattern size (at least 2k×2k pixels), which significantly slows down the acquisition time. As, in addition, a second series of diffraction must be acquired under vacuum for electric field measurement in order to correct for the intrinsic diffraction shift during beam shift, these leads to quite large data sets. If one is not interested to measure the electric field and the strain field exactly at the same place, performing 2 separate diffraction maps of reasonable size (512×512), one optimized for strain and the other optimized for electric field is presently more efficient for speed and data storage. After this first proof of concept that led to a patent, we plan to apply this new tool to studies on transistors and III-nitride material.

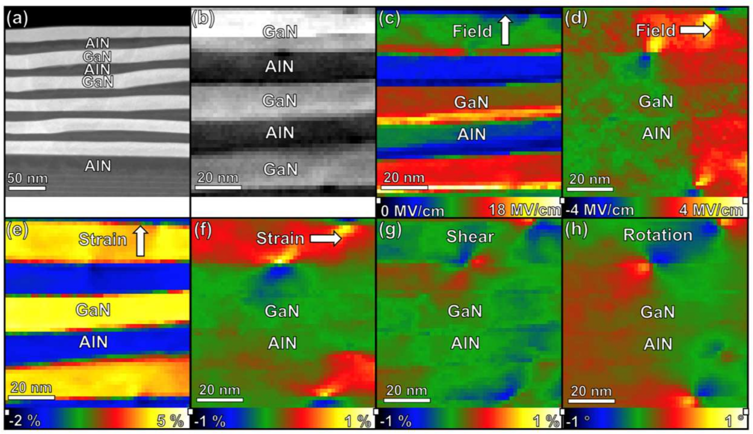


Figure H1: Simultaneous electric and strain field measurement from N_PED on an a-plane AlN/GaN multilayer observed along the c-direction. (a) A typical N-PED pattern of the 50x50 pattern series displayed in a logarithmic colour scale. (b-c-d-e-f-g-h) Different images obtained from the numerical analysis of the N-PED series. (b) Numerical HAADF. The bright region on top is an artefact from intensity fluctuation. It should be noted how this intensity variation has no influence on the other images. (c) Vertical in-plane electric field (d) horizontal in-plane electric field (e) 'Vertical' in-plane strain ε_{yy} (f) 'Horizontal' in-plane strain ε_{xx} (g) in-plane shear ε_{xy} and (h) in-plane rotation. A dislocation is seen at the centre of the top interface.

H2: Si donor incorporation in GaN nanowires

E. Robin, N. Mollard, Coll. Fang Z., Robin E., Rozas-Jimenez E., Cros A., Donatini F., Pernot J., Daudin B. (CEA-PHERIQS)

With increasing interest in GaN-based devices, the control and evaluation of doping are becoming more and more important. We have studied the structural and electrical properties of a series of Si-doped GaN nanowires (NWs) grown by molecular beam epitaxy (MBE) with typical dimensions of 2−3 μm in length and 20−200 nm in radius. In particular, high-resolution energy dispersive X-ray spectroscopy (EDX) revealed a higher incorporation of Si in the NWs than that in two-dimensional (2D) layers, and the segregation of Si at the edge of the NW with the highest doping (see figure). Moreover, direct transport measurements on single NWs have shown a controlled doping with a resistivity of 10^{2} to 10^{-3} Ω·cm, and a carrier concentration increasing from 10^{17} to 10^{20} cm^{-3}. Field effect transistor (FET) measurements combined with finite element simulation by NextNano3 software have put in evidence the high mobility of carriers in the non-intentionally doped NWs[41].

[39] B. Haas, D Cooper and JL Rouviere Patent FR1658594 - US20180076005A1

[40] B. Haas 2017 PhD Thesis. Development of quantitative diffraction and imaging-based techniques for scanning transmission electron microscopy.

[41] Fang Z etal., Nanoletters 2015, 15, 6794-6801

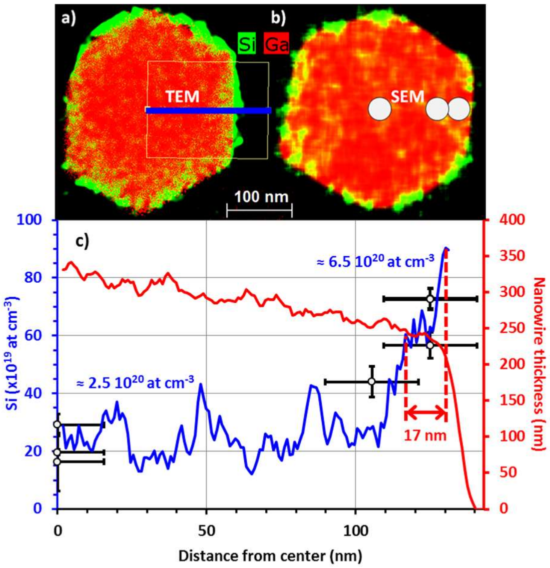
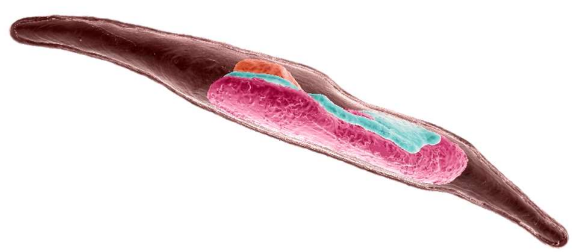


Figure H2: EDX maps of the cross-section of two NWs grown with Si cell temperature of 950 °C obtained (a) at 200 kV using a FEI Osiris TEM equipped with super X detectors and (b) at 4 kV using a ZEISS Ultra 55 SEM equipped with FlatQuad annular detector (PFNC-Minatec, CEA-Grenoble). Two elements are highlighted, with green and red corresponding to Si and Ga, respectively. Si concentrations and specimen thickness (red curve in (c)) were computed using the $\Phi(\rho z)$-corrected zeta-factor method (see IZAC project above) both for STEM EDX data (blue line profile in (c)) and SEM EDX data (white circles in (c)) showing a good agreement between both techniques. The solubility limit of Si in GaN NWs can be estimated to be around $2.5 \pm 0.5 \; 10^{20}$ at/cm^3 according to the maximum concentration in the core.

H3: Understanding the cell organization and metabolism of Phaeodactylum tricornutum

In collaboration with BIG / PCV (S. Flori, J. Lupette, D. Falconnet, G. Finazzi, et al.)

Phaeodactylum tricornutum belong to the family of diatoms, a major phylum of the phytoplankton, which has been present in oceans and freshwater ecosystems for about 100 million years. Diatoms are responsible for about 20% of photosynthesis activity on Earth. This particular diatom has been extensively analyzed by FIB-SEM tomography, and the 3D images, supplemented by the results of others techniques, have provided several novel insights on the organization of this cell and on its metabolism.



FigureH3: 3D reconstruction of a *Phaeodactylum* cell with its various organelles (chloroplast, mitochondria and nucleus).

A first result was obtained on the ultrastructure of the periplastidia compartment (PPC), which is the residual cytoplasm of a red algal symbiont[42]. This opens up new perspectives for understanding the origin of the secondary plastid formation in diatoms and its sophisticated relationship with other cell compartments. FIB-SEM images have also provided a better understanding of the mechanism of photosynthesis in this alga[43]. Photosynthesis, a mechanism for the production of chemical energy from light energy, is made possible by two small photochemical plants, called photosystems I and II. However, for photosynthesis to occur, these two photosystems must not be in contact to avoid short circuits. In plants, they are separated by structures that do not exist in phytoplankton. However, our images have permitted to evidence the existence of micro-domains separating, as in plants, the two photosystems, thus allowing an efficient photosynthesis. Finally, in the case of nitrogen stress, diatoms reallocate carbon to triacylglycerol storage inside lipid droplets (LDs), a potential precursor of bio-fuel. The architecture of the diatom LDs is unknown, and the FIB-SEM images have provided new information[44], revealing that the LD proteome highlights connections with the outermost membrane of the plastid and the endomembrane vesicular system.

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[42] Flori, S. et al. Protist 167, 254 (2016)
[43] Flori, S. et al. *Nature communications* 8, 15885 (2017)
[44] Lupette, J. et al., Algal Research 38, 101415 (2019).

These three results, obtained on the same algae, illustrate the power of the FIB-SEM technique. This helps to better understand the organization of subcellular components, and this can give new ideas on their functional process.

H4: Nanoscale chemical evolution of silicon negative electrodes characterized by low-loss STEM-EELS

M. Boniface, L. Quazuguel (CNRS/IMN), J. Danet, D. Guyomard (CNRS/IMN), P. Moreau (CNRS/IMN) and P. Bayle Guillemaud

Silicon is one of the most promising negative electrode materials for next generation lithium-ion batteries with a maximum uptake of 3.75 Li per Si atom, while graphite is limited to 1 Li atom per 6 C atoms corresponding to a gravimetric capacity about ten times lower. Its use as a commercial electrode is however limited by the high irreversible capacity caused by the colossal volume expansion (up to 270% for $Li_{3.75}Si$) upon its electrochemical alloying reaction with lithium. This swelling causes interfaces to be repeatedly shifted during cycling, thus exposing unpassivated surfaces to the electrolyte and leading to the continuous formation of solid electrolyte interface (SEI) and irreversible lithium consumption. In addition to the loss of cyclable lithium in a full cell configuration, swelling also causes silicon nanoparticles (SiNPs) to be progressively isolated from both electronic and ionic transport networks because of the morphological instability and the SEI accumulation, respectively. Continuous solid electrolyte interface (SEI) formation remains the limiting factor of the lifetime of silicon nanoparticles (SiNPs) based negative electrodes in batteries. Methods to clearly diagnose electrode degradation are of utmost necessity to streamline further developments. We have demonstrated that electron energy-loss spectroscopy (EELS) in a scanning transmission electron microscope (STEM) can be used to quickly map SEI components and quantify Li_xSi alloys from single experiments with resolutions down to 5 nm[45]. Exploiting the low-loss part of the EEL spectrum allowed us to circumvent the degradation phenomena that have so far crippled the application of this technique on such beam-sensitive compounds. Our results provide unprecedented insight into silicon aging mechanisms in full cell configuration. We observe that the SEI morphology is extremely heterogeneous at the particle scale but with clear chemical evolutions with extended cycling coming from both SEI accumulation and a transition from lithium-rich carbonate-like compounds to lithium-poor ones. Thanks to the retrieval of several results from a single dataset, we were able to correlate local discrepancies in lithiation to the initial crystallinity of silicon as well as to the local SEI chemistry and morphology. This study emphasizes how initial heterogeneities in the percolating electronic network and the porosity affect SiNPs aggregates along cycling. These findings pinpoint the crucial role of an optimized formulation in silicon based thick electrodes.

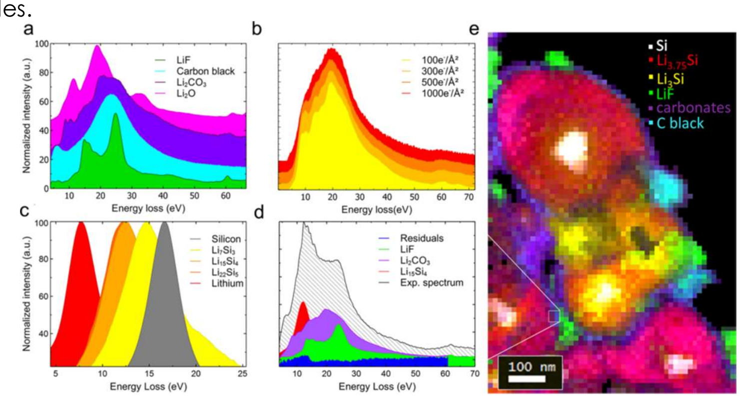

Figure H4: (a) EELS reference plasmon spectra for LiF, carbon black, Li2CO3, and Li2O. (b) Plasmon spectra of Li2CO3 acquired at different electron doses. (c) Reference plasmon spectra of LixSi alloys. (d) Example of MLLS fitting on the experimental spectrum integrated over four pixels of (e). e) Color map of LiF (green), Li2CO3 (purple), carbon black (teal), Li7Si3 (yellow), Li15Si4 (red), and Si (white), obtained through MLLS fitting of aspectrum image from an electrode charged to 3000 mAh g−1. The scale bar is 100 nm.

H5: In-situ observation of formation and migration of defects in van der Waals heterostructure

C. Alvarez, H. Okuno, Coll. P. Pochet (CEA/MEM/L_Sim), T.M. Dau, M. Jamet (CEA/Spintec), H. Le Poche (CEA/LITEN)

[45] M. Boniface, L. Quazuguel, J. Danet, D. Guyomard, P. Bayle-Guillemaud, Nanoletters 2016, 16(2), 7381-7388

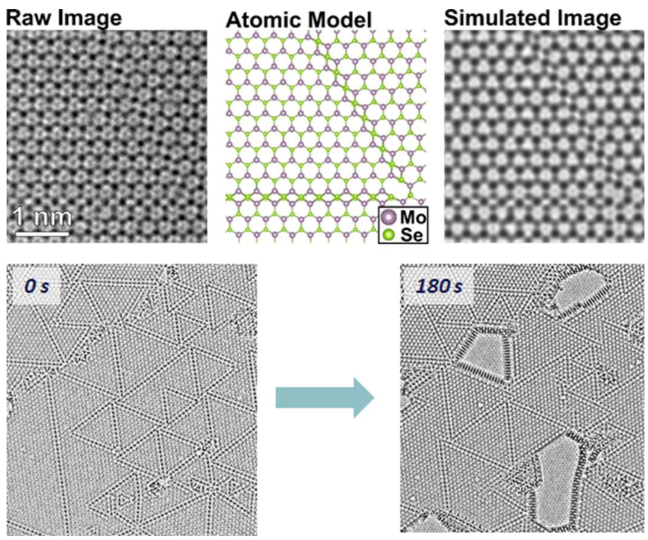

Figure H5: Atomic structure of growth related intrinsic 4/4 P line defect in $MoSe_2$ monolayer grown on graphene substrate and formation of MoSe nanowire via extrinsic defect diffusion along intrinsic line defects under the electron irradiation at high temperature.

Two dimensional (2D) layered structures, such as graphene and transition metal dichalcogenides (TMDs), have shown significant potential for future electronics and optoelectronics due to their unique electronic structures. Recently, tuning the electronic properties of these atomically thin materials has become one of the exciting challenges. The modification of atomic structures by introducing atomic scale ordered defects has been demonstrated as a powerful strategy to control their band structures. Despite the growing interest in 2D materials, the growth of defect-free 2D materials on desired substrates remains a challenge. Identification and characterization of these inevitable intrinsic growth defects are thus an essential process. Further exploitation of possible defect modifications and manipulations might shed light on potential utilization of defect structures in device processing. For this purpose, we studied intrinsic defects in MBE grown $MoSe_2$ monolayer and their role in extrinsic defect formation induced by post-growth processing using an aberration corrected transmission electron microscopy (AC-TEM) which allows to visualize atomic structures of atomically thin 2D layers and simultaneously to form defects by electron irradiation. In order to investigate the phenomena under near-device situation, we performed the irradiation experiments on $MoSe_2$ on its van der Waals graphene substrate. The formation, migration and elimination of intrinsic and extrinsic defects are studied with respect to the substrate. The results revealed the possibility to independently modify the $MoSe_2$ monolayer by the introduction of an external energy even in the presence of a strong epitaxial coupling with the graphene substrate during growth[46]. A particular intrinsic growth defects, 4/4 P type of Se deficient line defects, has been identified and this structure plays an important role as a defect diffusion canal to reorganize the beam induced defects into MoSe metallic wires. These results shed light to the defect engineering in 2D materials to design their atomic structure with new functionalities.

H6: Quasi-two-dimensional electron system at the interface between LaAlO3 and SrTiO3 band insulators

D. Jalabert, Coll. H. Zaid

An international collaboration has been set up to investigate the novel behaviour observed at the interface of $LaAlO_3/SrTiO_3$ heterostructures, such as two-dimensional metallic conductivity, magnetic scattering and superconductivity. In this system, both the origins and quantification of such behaviour are complicated due to an interplay of mechanical, chemical and electronic factors. Medium Energy Ion scattering (MEIS) was used to measure with subnanometric depth resolution the chemical and strain profiles near the interface of $LaAlO_3/SrTiO_3$ heterostructures. Depending on the thickness of the $LaAlO_3$ film deposited on a $SrTiO_3$ substrate, the interface evidences different electrical behaviour. Above a critical thickness of 4 unit-cells (1 u.c. ~ 0.38 nm) the sample exhibits a conductive interface while below this limit, the sample remains insulating. To investigate this phenomenon, two samples were grown with respectively 3 and 5 u.c. of $LaAlO_3$ deposited on $SrTiO_3$. The intermixing and structural distortions within the crystal lattice were quantitatively measured near the interface with a resolution in depth of one unit-cell size[47]. A strong link between intermixing and structural distortions at such interfaces is highlighted: intermixing is more pronounced in the hetero-couple with conductive interface, while in-plane compressive strains extended deeper within the substrate of the hetero-couple with the insulating interface. This allows a better understanding of local interface mechanisms leading to conductivity[48].

[46] C. Alvarez, M.T. Dau, A. Marty, C. Vergnaud, H. Le Poche, P. Pochet, M. Jamet and H. Okuno, Nanotechnology 2018, 29, 425706.

[47] H. Zaid, M. H. Berger, D. Jalabert, M.Walls, R.Akrobetu, I. Fongkaew, W. R. L. Lambrecht, N. J.Goble, X. P.A.Gao, P. Berger and A. Sehirlioglu, Scientific Reports 6, 28118 (2016)

[48] H. Zaid, M. H. Berger, D. Jalabert, M. Walls, R. Akrobetu, N. Goble, X. Gao, P. Berger, I. Fongkaew, W. Lambrecht, and A. Sehirlioglu, Journal of Applied Physics, 123, 155304 (2018)

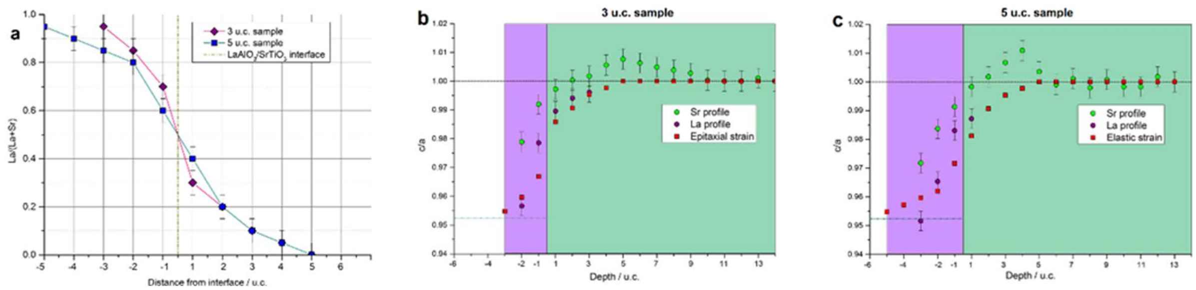

Figure H6: (a) La/(La+Sr) chemical profile throughout the first atomic layers. (b & c) Profiles of c/a for samples with film thicknesses of 3 u.c. (b) and 5 u.c (v). Purple circles represent the c/a values around La atoms. Green circles represent the c/a values around Sr atoms. Red squares represent the theoretical c/a values for intermixed heterostructure assuming a sole elastic epitaxial strain.

4- Organisation and life of the LEMMA team

The team organizes a weekly group meeting for technical and scientific exchanges. PhD students, pot-docs and permanent staff regularly present the most recent results. Several external researchers are invited to give seminars in extended group meetings. The team contributes to the organization of the nano-characterization platform (PFNC) with the microscopy groups of LETI and LITEN. Technical and scientific meetings are held once a month to discuss the status of each equipment and common short- and long- term strategies for a good operation and scientific developments within the PFNC. There is a steering committee within the PFNC, which consists of IRIG, LETI and LITEN directions. Important collective decisions concerning operational and financial matters within the PFNC are steered and validated by the steering committee. A scientific steering committee consisting of external researchers (from French and Swiss laboratories) also follows the operation and activities at the PFNC. This helps to operate the platform with a world-class excellence.

Parity: The team consists of a permanent staff with 1 woman and 5 men. The gender balance in non-permanent staff (PhD students and post-docs) has been relatively good over the evaluation period.

Safety: A safety responsibility is organized in a relationship between the team, the PFNC and the IRIG institute to assure experimental and human safety.

Five-year project and strategy of the LEMMA team

LEMMA SWOT analysis

Strengths	***Weaknesses***
Diversity of competences and expertise A strong network of local, national and international collaboration Substantial scientific production (about 140 articles)	Limited opportunities to have young researcher, PhD, post-doc Lack of man power: number of personnel
Opportunities	***Threats/risks***
PFNC (inter-institutional platform) allows access to numerous equipment and a dynamic with researcher in other institutes Thanks to PFNC, access to more collaborations with important industrial companies (FEI, IBM, STMicroelectronics, ZEISS…) Grenoble scientific environment: excellent academic laboratories and industrial and applied research organisms Extension to new research field: re-organization of the institute opens collaboration with biological laboratories	No immediate opportunities for hiring new staffs Limited financial support/projects for new equipment Divergence of activities and strategies between the different institutes at PFNC

Most of strengths and opportunities of the team are related to the PFNC allowing access to a wide range of out-standing microscopes. The LEMMA team has played an important role since long years in the PFNC by developing the advanced analytical methods thanks to the rich environment. However, in these years it has been seen an imbalance between the institutes (IRIG, LETI and LITEN) due to the lack of man power and funding for new investments within the IRIG institute. This would bring a crucial problem in near future to be able to continue high level of fundamental research activities in the team.

Structure, workforce and scientific orientations

The LEMMA team wishes to continue to expand in the near future the various themes and techniques developed over the last 5 years: high-resolution (S)TEM imaging, diffraction, FIB-SEM nano-tomography and chemical analysis, to provide further capabilities for materials science. These developments will be driven by the needs of material/device improvements, where chemistry, strain, structure and charges have to be controlled at the nanoscale and atomic scales. The ambition of the team is to provide within the PFNC a full set of electron and ion imaging techniques, allowing characterizing in 3D complex nanostructures or devices.

Some of the most promising perspectives are described below:

New diffraction-based techniques towards ultimate material information

For more than 30 years, LEMMA has built knowledge on high resolution and diffraction methods. Recent advances in computing methods and techniques tends to indicate that ptychography should become one of the most complete and powerful technique of microscopy. Ptychography is a particular 4D-STEM experiment where there are overlaps of the probe during its scanning and where all the information are obtained after an iterative numerical analysis of the acquired diffraction patterns. LEMMA has been preparing these ptychography studies for many years, for instance through its strain-electrical-magnetic field studies and through Pr. Zuo’s chair of excellence on a connected topic: 3D-coherent Diffractive Imaging (3D-CDI) and through its

collaboration with Dr V. Favre-Nicolin who was part of the 3D-CDI project and goes on developing an efficient GPU ptychography software, pynx, at ESRF. By combining 4D-STEM and ptychography we hope to answer important characterization topics in piezo-electric, microelectronics, spintronics and 2D materials. Our preliminary results on 4D-STEM on electric field measurements are very encouraging. In principle, ptychography experiments should give unique information such as the details characterization of the input wave function, the wave functions and the atomic projected potentials within a limited number of slices of the observed samples. Most of the ptychography softwares assume a weak interaction between the electron beam and the sample, which is best achieved in 2D-materials. This is why we plan to have a strong connection between our 2D-materials studies and ptychography/4D-STEM developments: two PhDs working in collaboration on ptychography and 2D materials should start in 2019 and the MAGICVALLEY ANR project should combine spectroscopy/4D-STEM/Ptychography/ab-initio simulations to study charges and structures in doped 2D materials. Following our preliminary results, the deviation of the centre of mass of the direct transmitted beam in 4D-STEM diffracted patterns should permit to obtain atomic resolution charge density maps, while ptychography and spectroscopy techniques should give more structural and chemical information that could be compared to ab-initio simulations. In addition, in collaboration with Dr V. Favre-Nicolin from ESRF, we would like to develop a ptychography software that gets rid of the weak beam interaction approximation.

New developments in EDX quantitation

We will continue the development of the IZAC code towards low energies (<5 kV), and to improve databases for low X-ray emissions, especially for L and M lines. Our objective is to explore the potential of the method for quantifying soft X-ray emissions. Indeed, in the future we would also like to investigate a new type of spectrometer that allows the simultaneous detection of soft X-rays over a wide range of energies, with a nominal lower energy of 50 eV and an unprecedented spectral resolution of 0.3 eV. This should allow mapping the chemical bonding state of several elements, including Li, with huge applications in the field of Li-ion batteries. Because of its excellent peak/background ratio, this new technology has a high potential for analyzing traces of light elements at concentrations as low as a few tens of ppm, as demonstrated in the literature. In addition, soft X-ray emissions can be obtained at low voltage (below 2 kV) leading to a spatial resolution better than 10 nm for most materials. These promising features should permit many applications in materials science including nanoelectronics, optoelectronics and photonics. For example, it should be possible to investigate the elemental and chemical state 2D distribution of a wide range of dopants, including B and P in (Si, Ge); N in (Sb, Ga)As; Mg, Si, Ge in (Al,Ga)N,… Another promising application is the quantitative 3D reconstruction of chemical and biological nano- and micro-structures using the FIB-SEM "slice&view" technique.

Progresses in FIB-SEM 3D analysis

In the near future, we expect to develop the FIB-SEM nano-tomography activity around several new technological and conceptual developments, always in collaboration with various partners in our institute. Our aim is to be able to develop new and original competencies thanks to our technical expertise of the instrument. One of the main limitations of FIB-SEM nano-tomography is its relative slowness imposed by the acquisition time of SEM images, with the typical durations of a few hours to a few days. Faster acquisitions of FIB-SEM volumes through the use of compressed sensing techniques will be developed in next 5 years. We intend to investigate new and potentially faster strategies for acquiring images based on "compresed sensing" methods in collaboration with several CEA groups havig a strong expertise in image acquisition algorithm, especially at Neurospin (Philippe Ciuciu, medical imaging) and Cosmostat (J.L. Stark, data acquisition in astronomy) and with Zeiss to implement them technically on the microscope, taking into account the experimental limits (constraints related to the detectors, to the physical systems of image acquisition, to the software controlling the microscope ...). Depending on the material structure and size, the strategies could be slightly different. This is why, we plan to optimize the acquisitions for two different fields: images of cells (brain, liver tissue, algae) and images of semiconductor structures (nano-porosity in vias or implanted layers). A DRF-impulsion grant will allow us to start this work soon.

Improvement in data handling and in large volume segmentations

Big-data handling and quantitative analysis of large 3D-volumes have become important issues in many of the developed techniques and particularly for Time resolved in-situ observation, 4D-STEM data sets and FIB-SEM 3D. LEMMA will try to find efficient solutions to these issues by having helps, advices and investments from local and CEA partners, Comsmostat (J.L. Stark, data acquisition in astronomy) e.g. in compress. PFNC is aware that it has to develop efficient data handling and analysis and tests are presently performed on GPU-based systems. The collaboration with Dr. V. Favre-Nicolin who wrote the GPU-accelerated Pynx software at ESRF and L_SIM who developed GPU-accelerated ab-initio software will be particular important.

Equipment, platforms

The team is located at Nano-Characterization Platform and has access to following equipment by sharing with other institutes (mainly LETI and LITEN). Percentage of our access time is defined for each microscope.

1. Titan Ultimate Thermo Fisher (2013-): 40% of machine time

Equipped with double aberration corrector, dual-EELS, monochromator, high-speed Camera (one-view), 80-300kV

- HR-TEM and STEM
- Diffraction based techniques
- 4D-STEM
- STEM-EELS spectroscopy

Mainly used for
- Technical developments
- 2D materials study

2. Titan Themis Thermo Fisher (2017-): 40%

Equipped with probe aberration corrector, dual-EELS, Super X EDX detector, 80-200kV

- HR- STEM
- Diffraction based techniques
- STEM-EELS spectroscopy
- STEM-EDX spectroscopy

Mainly used for
- Technical developments
- Battery materials study

3. FIB-SEM cross-beam Zeiss XB550 (2019-): 33%

Dual beam (electron/ion): SEM, FIB

Mainly used for
- TEM specimen preparation
- FIB-SEM slice & view 3D imaging
- Technical development
- Battery materials study
- Biological materials study

4. FIB-SEM Zeiss NVision 40 (2010-): 25%

Dual beam (electron/ion): SEM, FIB
EDX detector

Mainly used for
- TEM specimen preparation
- FIB-SEM slice & view 3D imaging
- Technical development
- Biological materials study

5. Other TEMs: 3
6. Other FIBs: 2
7. SEMs: 3

Research products and activities of the LEMMA team

Articles

Scientific articles (total number) 137

20% most significant articles

2019

1. *Understanding the crystallization behaviour of surface-oxidized GeTe thin films for phase-change memory application, A.N.D. Kolb, N. Bernier, E. Robin, A. Benayad, J.-L. Rouvière, C. Sabbione, F. Hippert and P. Noé, ACS Appl. Electron. Mater.,2019, (10.1021/acs.aelm.9b00070)*

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The remaining 80% articles

2019

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Books

Book chapters (total number) 2

Book chapters in English or another foreign language 5

1. D. Jalabert, Ian Vickridge, and Amal Chabli. *Swift Ion Beam Analysis in Nanosciences*. John Willey & Son, (2017). ISBN 9781119005063. doi: 10.1002/9781119005063.
2. *M. Kuhn, S. Cea, J. Zhang, M. Wormington, T. Nuytten, I. De Wolf , J-M Zuo and J-L Rouviere Transistor Strain Measurement Techniques and their applications* (chapter 6) of the book : Metrology and Diagnostic Techniques for nanoelectronics edited by Z. Ma and D. G. Seiler Pan Stanford Publishing (2016) ISBN-13: 978-9814745086

Electronic tools and products

Softwares

1. **IZAC :** software in VBA for EXCEL
2. **GEM-ED :** script of digital micrograph
3. **TeMA** : a mixture of C++ and digital micrograph script
4. **Precession Mk_II** : a new module in FEI microscope software
5. **Zorro** : python software

Databases

IZAC database for K, L, M emissions lines and associated relative intensities

Instruments and methodology

Prototypes

Here is the liste of software realised by LEMMA and its close collaborators during the reporting period.

1. **IZAC** is a new EDX quantification routine wirtten by E. Robin. It is based on a patented Φ(ρz)-corrected zeta-factor method (Robin, patent N°EP3032244B1, 2016) whose principle is to correct the measured X-ray intensity from matrix effects which affect the generation and emission of the radiations resulting from the partial deceleration of the electron beam and potential absorption of the radiations in the analyzed volume [Robin, 2016]. It thus differs from commonly used quantification routines which assume either no deceleration (k- and zeta-factor methods) or total deceleration (ZAF and Φ(ρz) methods) of the electron beam in the specimen. The routine is also implemented with a new model of X-ray absorption allowing absorption correction of low energy X-ray in heterogeneous specimens. The routine can applied both in SEM and TEM over a wide range of accelerating voltage and on various materials, i.e. from thin to massive structures and from pure substances to multi-element compounds.
2. **GEM-ED :** A user friendly Digital Micrograph script to analyse series of local diffraction patterns written by J-L Rouvière. Used by close collaborators (IBM and CEA-Grenoble), the software read different format of diffraction patterns (individual files or one stack of patterns) and compute different types of image : bright field, dark field, High Annular Dark field, different 2D strain and electric/magnetic maps. During the last 5 years, the software have been greatly improved by introducing new image filters, different material phases and the measurement of electric and magnetic fields. Within the CEA-FEI common lab, the core functions of GEM-ED for strain measurements have been implemented in the **FEI epsilon software**, sold by FEI on demand.
3. **TeMA :** Template Matching algorithm package written by Pr. J. Zuo during its chair of excellence in Grenoble. It is a mixture of C++ and Digital Micrograph script. Available on demand to Pr. J. Zuo. Dr J-L Rouvière has also written a simpler, but partial version Digital Micrograph script, **GEM-TeMA**. The software starts by defining templates by averaging smartly different subareas. These templates are then used to determine accuratly the atomic column positions. Finally strain maps are constructed.
4. **Precession Mk-II :** A new precession module developped within the CEA-FEI common lab. It was written by FEI, but pushed and tested by PFNC. This module improves the precession tuning, simplifies the selection of the map area by using STEM images and importantly increases by a factor 5 the previous acquisition speed.
5. **Ptychocontrol :** python software written by Dr R. McLeod during its post-doc position. The software allows to control the different lenses of microcope and acquire series of local diffraction patterns or images obtained by varying a given parameter of the microscopy.
6. **Zorro :** https://github.com/robbmcleod/zorro. python software written initially by Dr R. McLeod during its post-doc position, but further extended by himself in Swizerland for biological applications. The code registers and eventually averages series of images.

Platforms and observatories

The team is a part of the METSA (the French microscopy network), whereas the team provides access the micrroscopes and their expertises to French research community.

Reviewing activities

Reviewing of articles

The members of the team are solicited as referees for the following scientific journals:
Advanced Materials, Carbon, Nuclear Instruments, Methods in Physics Research B, Ultramicroscopy, Nature

Academic research grants

European (ERC, H2020, etc.) and international (NSF, JSPS, NIH, World Bank, FAO, etc.) grants – coordination

1. BACCARA : Battery and supercapacitor characterization and testing (FP7, 2013-2016)

European (ERC, H2020, etc.) and international (NSF, JSPS, NIH, World Bank, FAO, etc.) grants – partnership

2. Gladiator : Graphene Layers : Production, Characterization and Integration (FP7, 2013-2017)
3. CONNECT: Carbon nanotube composite interconnects (H2020, 2016-2018)

4. SINTBAT: Silicon based materials and new processing technologies for improved lithium-ion batteries (H2020, 2016-2020)

National public grants (ANR, PHRC, FUI, INCA, etc.) – partnership

1. MoS2ValleyControl : Valley dependent optics and transport in MoS_2 monolayers and deviced (2015-2017)
2. MAGICVALLEY: Magnetism induced valley polarization in large scale 2D materials (2018-2022)
3. ESPADON : Engineering Spins and Photons from Anisotropic DOts in Nanowires (2015-2019)
4. EMOUVAN : EMissiOn de lumière UV Avec des Nanofils/UV light emission with nanowires (2015-2019)
5. MECHASPIN : atomic scale control and MECHAnical SPIN driving of a magnetic atom (2018-2021)
6. SESAME : Impact of SizE and StrAin on crystallization of chalcogenides for the ultimate scaling of phase change Memories (2016-1019)
7. COSMOS : Correlation du microscopie électronique en transmission avec des mesures optique et électrique effectués sur le même nanofils unique (2013-2015)

Local grants (collectivités territoriales) – coordination

1. CEA DRF-impulsion project FIB-Bio
2. CEA DRF-impulsion projet Fast_FIB-SEM

Local grants (collectivités territoriales) – partnership

1 CEA project 2D factory

PIA (labex, equipex etc.) grants – partnership

1. LANEF 3D-CDI: Chair of excellence of Pr. Zuo
2. LANEF: Chair of excellence of Pr. A. Dimoulas

Visiting senior scientists and post-doc

Post-docs (total number): 6

Post-docs financed by ANR, CEA funding, European projects, LANEF

1. Robert Mc Leod
2. Bastien Pellegrin
3. Andrea Kolb
4. C. Allvarez
5. Dipankar Kalita
6. Praveen Kumar

(Foreign post-docs: 5)

Visiting scientists (total number) 3

1. Prof. Jim Zuo: University of Illinois, U.S.
2. Prof. Althanasis Dimoulas: NCSR, Greece
3. Maria Veronica Branza-Blanco: Université de Cadix

(Foreign visiting scientists: 3)

Scientific recognition

Organisations of meetings and symposia (out of France)

1. J-L Rouvière : Rouvière co-organizer of the microscopy session Quantification and modelling at the Microscopy Conference in Lausanne (2017)
2. J-L Rouvière : Symposium co-organizer at MRS spring meeting, Phoenix (USA) symposium CM1 New Frontiers in Aberration Corrected Transmission Electron Microscopy (2016)
3. J-L Rouvière : Organizer of a microscopy session on Strain Map and Orientationat the French Microscopy Conference in Nice (2015)

Invitations to meetings and symposia (out of France)

1. H. Okuno, Structure control of two-dimensional layers : toward functional materials, presented at MSE 2016, Darmstdt, Germany 2016
2. H. Okuno, Structural investigation of 2D materials: From growth toward controlled properties, presented at 3rd EU-Japan Workshop on Graphene and Related 2D materials, Sendai, Japan 2018
3. D. Jalabert, MEIS analysis at the nanoscale, advantages/disadvantages versus X-Rays and TEM, presented at the 22nd International Conference on Ion Beam Analysis, Opatija, Croatia 2015
4. J-L Rouviere, AVS (American Vaccuum Society) San José (USA) oct. 18-23, 2015

5. J-L Rouviere, Frontiers of Characterization and Metrology for Nanoelectronics (FCMN) Dresde (USA) 14-16 avril 2015
6. J-L Rouviere, M&M 2015 (Microscopy and MicroAnalsis) Aug. 2-6 2015, Portland (USA)
7. J-L Rouviere, invité au workshop ACEM (Aberration Corrected Electron Microscopy) 27mars 2016
8. J-L Rouviere, M&M 2017 St Louis (USA)

Interaction of the LEMMA team with the non-academic world, impacts on economy, society, culture or health

Patents

Accepted patents 5

1. Procédé de recristallisation d'un materiau carboné, tel que du graphene ou des nanotubes de carbone, J. Dijon, H. Okuno, P. Pochet, R. Ramos, Patent WO2019037396, 2016
2. Procédé de dopage de graphène, H. Le Poche, A. Carella, J. Dijon, H. Okuno, E. Roux, Patent FR3 058 997-A1, 2017
3. Method for the determination of the mass thickness and the composition of a zone of an object using an electron beam and measurements of x-ray intensities, E. Robin, Patent N°EP3032244B1, 2016
4. Method of determining the deflection of an electron beam resulting from an electric field and/or a magnetic field, B Haas, D Cooper and JL Rouviere Patent FR1658594 - US20180076005A1
5. Méthode de traitement de clichés de diffraction permettant d'obtenir des images offrant un contraste plus homogène. brevet déposé le 5 mai 2017 par B. Haas, N. Bernier et J. L. Rouvière

Socio-economic interactions

Industrial and R&D contracts

1. Collaboration agreement CEA/FEI (one year post-doc in 2014): developing EDX quantification in TEM using the zeta-factor method
2. Collaboration agreement CEA/ALEDIA (2015): quantifying Mg dopant in GaN with wide solid angle EDX detector
3. Simulation Monte Carlo de l'interaction gammas et neutrons dans des écrans de protection de type NOVASHIELD développés par la société LEMERPAX (2016) https://www.lemerpax.com/produits/novashield/

Creation of labs with private-public partnerships

Joint laboratory with FEI

Involvement of the LEMMA team in training through research

Training

Habilitated (HDR) scientists: 3

1. Jean Luc Rouviere
2. E. Robin
3. D. Jalabert

PhD students (total number): 13 (Supervision in the team and co-supervision)

Defended PhDs in the team: 7

1. Yannick Martin (2014)
2. Bastien Bonef (2015)
3. Maxime Boniface (2017)
4. Kalim El Hajraiyu (2017)
5. Benedkt Haas (2017)
6. Fabio Agnese (2018)
7. Lynda Amichi (2018)

Mean PhD duration 39 months

Internships (M1, M2): 5

TEAM 3: MAGNETISME ET DIFFRACTION NEUTRONIQUE (MDN)

1- Presentation of the MDN team

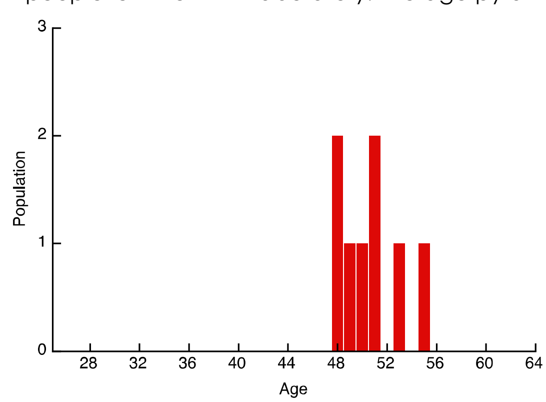
Introduction

The Magnétisme et Diffraction Neutronique (MDN) laboratory operates three neutron scattering instruments as Collaborating Research Group (CRG) at the Institut Laue Langevin (ILL), Grenoble in collaboration with the Jülich Centre for Neutron Science (JCNS). The aim of the laboratory is to develop and make available to the users community neutron scattering tools, through the running of the following single crystal neutron scattering instruments: the two-axis diffractometer D23 and two three-axis spectrometers IN22 and IN12. An own research activity of the team often performed in collaboration is grafted on this main task. The laboratory is specialized in polarized neutrons (longitudinal and sphE. al polarimetry, spin echo) and high magnetic field studies (static 15 T and pulsed 40 T). Research activities are focused on basic knowledge in condensed matter physics spanning topics from fundamental magnetism (low dimensional quantum spin systems, frustrated magnetism, multipolar and orbital physics, unconventional metals and superconductivity, …) to functional materials (multiferroic, magnetocaloric, thermoelectric materials, …).

Unit's workforce and means

Permanent staff (01/2019)	Non-permanent
F. Bourdarot, CEA researcher, responsible of IN22	**Post-doctoral researchers**
P. Fouilloux, CEA technician	K. Beauvois, co-responsible of D23 (2017-2019)
B. Grenier, Assistant professor at UGA	S. Chattopadhyay, co-responsible of D23 (2015-2016)
B. Longuet, CEA technician	K. Zhernenkov, instrumentation for IN22 (2013-2015)
F. Mantegazza, CEA engineer	
S. Raymond, CEA researcher, co-responsible of IN12, team leader	**PhD students** (affiliated at MDN)
E. Ressouche, CEA researcher, responsible of D23	Q. Faure (2015 - 2018)
B. Vettard, CEA technician	N. Biniskos (2015 - 2018)
	(ILL students not affiliated at MDN)
	S. Turner (2018 - …)
	L. Chaix (2011 - 2014)

Two researchers from JCNS (K. Schmalzl and W. Schmidt) are attached to the CRG instruments (see Annex MDN-A). A specificity of the MDN staff is the high ratio of support (technician/engineer) to research staff and a small amount of non-permanent staff, which is a commonality among large scale facilities. Over the evaluation period two CEA employees retired: the distinguished researcher and responsible of IN22, L.P. Regnault, and the IN22 technician, B. Geffray. The last recruitment in the MDN team dates back to January 2000 (S. Raymond) and since that time about ten people left the MDN laboratory. The age pyramid of MDN is given below.



The laboratory budget is of about 850 k€, which breaks into salaries (approx. 73 %), infrastructure costs (approx. 18 %), running costs (approx. 6%) and investments (approx. 3%). Most of the funding is coming from dedicated CEA budget originating from the action 13 (large scale facility) of the program 172 (interdisciplinary technological and scientific research) of the ministry of innovation research and higher education. This

allocation does not cover the entire running costs of the spectrometers and therefore since 2015, the laboratory rents beamtime to Switzerland though a collaboration agreement between MDN and Ecole Polytechnique Fédérale de Lausanne that represents SERI (state Secretariat for Education, Research and Innovation). This agreement was a Swiss-based initiative originating from a former MDN post-doctoral student now professor at EPFL and president of the Swiss Neutron Science Society. Typical financial flows are given in the table below for the year 2018. Salaries of permanent staff are not indicated, they are covered from action 13 budget at the level of 463 k€, which covers four researcher/engineer and three technicians, the missing part being taken by another CEA budget line. In 2017, the French federation for neutron scattering (2FDN) has been created to organize the French neutron community access and helps the laboratory for missions and small investments. It also provides reimbursement for French users coming on our instruments. This latter funding for users does not appear on MDN budget but is very important for the attractivity of the laboratory. Two ANR grants have been obtained during the evaluation period MAGFINS (12/2010-12/2015) for the development of high pulsed magnetic fields and DYMAGE (10/2013-03/2018) for the study of multiferroic materials. Finally, PhD grants were obtained from UGA for Q. Faure and from JCNS for N. Biniskos (1/2 grant for a PhD common between CEA and JCNS).

Financial flows for investment, running costs and non-permanent staff (except PhD) in 2018.

Funding source	Incomes		Expenses	
Large scale facility (action 13)	Running costs	122 k€	Infrastructure at ILL (2 CRG):	107 k€
			Helium:	20 k€
Swiss-CRG contract		155 k€	Post-doc D23:	55 k€
			Running costs (outside helium):	30 k€
			Investment:	15 k€
			Institute financial levy:	50 k€
2FDN		12 k€	Mission:	2 k€
			Investment:	10 k€
Total		289 k€		289 k€

Scientific policy

The main mission of the laboratory is to operate the D23, IN22 and IN12 instruments at ILL, to provide beamtime to the neutron scattering user community and to propose new neutron instrumentation and new neutron scattering methods. Our activity concerns both the European and French user communities (See section 4 for the allocation of beamtime). D23 and IN22 belong to CEA and IN12 belongs to JCNS. A contract between JCNS and CEA defines the repartition of beamtime and staff (See Annex MDN-A). In this frame, the day to day technical responsibility of the three instruments is undertaken by CEA technical staff.

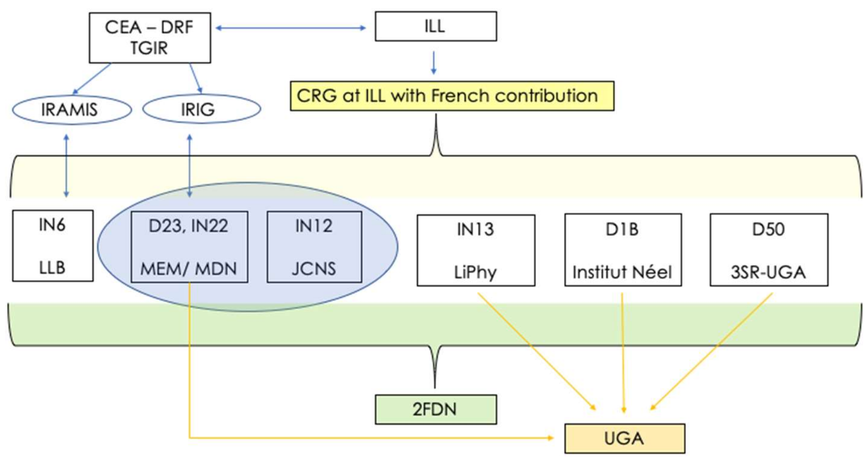
Before 2016, the MDN team was part of the SPSMS laboratory (Service de Physique Statistique, Magnétisme et Supraconductivité) which was built on the synergy between crystal growth, bulk measurements, neutron scattering and theory. In 2016, MDN was attached to the MEM laboratory which gathers several teams that are built around a technic rather than around a scientific topic and are aiming to explore matter and characterize materials. This change has reinforced the strategy for MDN to be oriented toward the user community. This evolution turns out to be in line with two elements external to MDN: the phasing out of the research axis "magnetism and superconductivity" at INAC in 2013 and the shutdown of the Orphée reactor in LLB, Saclay planned in 2019. The first point leads to a shift of the research topics in MDN and two researchers, previously involved in the study of strongly correlated f-electron systems with other SPSMS teams, have developed a research activity on energy materials (magnetocaloric and thermoelectric compounds). It is to be noted that the dichotomy between fundamental physics and functional materials is somehow artificial since concepts as well as measurement methods are intertwined. Furthermore, fundamental questions of magnetism often serve as benchmark for new instrumentation (see e.g. the development of the 40 T pulsed field magnet). The second element that comes into play in the change of strategy of MDN is the planned shutdown of the Orphée reactor. Some of the instruments of LLB have their counterpart in the MDN ones (2T versus IN22, 6T2 versus D23, 4F1 versus IN12) and shifting our activity towards French users helps to partly compensate the corresponding lack of beamtime to come.

Considering all the activities in MDN, it is estimated that 2/3 of the time is devoted to the user community and 1/3 of the time to the own research activity of the team which is not limited to scientific topics and encompasses the development of neutron instrumentation and methods.

The MDN team is involved in research training through the supervision of PhD students (MDN students and co-supervision of ILL students), in the organization of schools and courses (e.g. HERCULES) and in lectures (or practicals/tutorials) regularly given in summer schools leading often to publications of neutron scattering techniques related articles or presentations available on internet (See Annex MDN-C).

2- Presentation of the MDN team research ecosystem

The operation of the CRG instruments is governed by a contract between ILL and CEA and a subcontract between CEA and JCNS. The CEA instruments are governed by the IRIG (INAC before 2019) and the TGIR (Très Grandes Infrastructures de Recherche) unit at the DRF (Direction de la Recherche Fondamentale). The coordination between the different French institutes operating neutron scattering instruments in France (LLB and CRG at ILL) is now made by the 2FDN. The 2FDN organizes the selection panels of French experiments (See section 4). The MEM laboratory is a mixed unit (UMR) with UGA, attached to PEM (Physics, Engineering and Materials) research pole. The diagram below indicates the link between the different actors involved.



Beyond this local ecosystem, the work at MDN involves key collaborations with JCNS (Germany), Barcelona University (Spain), EPFL, ETH-Zurich and PSI (Switzerland), LLB (Saclay), LNCMI (Toulouse) and Institut Néel (Grenoble).

3- Research products and activities for the MDN team

CRG Instruments operation

Over the period 2014-2018, the ILL reactor performed 14 cycles leading to 629 days of reactor operation. This is significantly below the "normal operation" due to foreseen long maintenance shutdowns combined with unexpected technical and administrative failures (pending work authorization from French Nuclear Safety Authority). In fact, the reduction of available reactor days by approximately 33% over the evaluation period largely helped us to run our instruments given the limited staff. The first table below indicates the total number of experiments and the balance between experiments and instrumentation days. Lost days corresponds to failure of electronics, mechanics, cryogenics or sample. In total, the MDN laboratory provided about 1600 days of beamtime for users (including the JCNS time) accommodating 264 experiments. Since on average 2-3 users come for an experiment, this leads to 660 continuous-flow hosted users.

	Number of experiments	Days for experiments	Days for Instrumentation	Lost Days
IN12	95	526	83	20
IN22	83	510	82	37
D23	86	559	54	16

The requested beamtime is summarized in the Table below for the ILL selection panel (over 2014-2018) and for the 2FDN selection panels that started in 2016. The repartition and allocation of beamtime between the different parties is given in section 4. Depending on the instrument and the selection panel, the oversubscription rates lie in the range between 2.2 and 3.8. These ratios are narrowed to the range 2.5 - 3.5 when averaging the ILL and 2FDN panels. French users often apply in both panels for different or same proposals. These overall data show that our techniques are of high interest to many active scientific fields.

	ILL requested days (2014 - 2018)	ILL allocated days (2014 - 2018)	2FDN requested days (2016 - 2018)	2FDN allocated days (2016 - 2018)
IN12	576	150	152	57
IN22	495	164	228	65
D23	426	194	230	71

Instrumentation activity

The instrumentation developments performed during the evaluation period are given in the Annex MDN-C, part I.5. It consists in the:

- Start of the commissioning of the multi analyser option on IN12.
- Installation of the 40 T pulsed magnetic field instrumentation.
- Construction of an analyser on D23.
- Construction of a 15kV electric field stick.

Scientific activity

The MDN team has produced about 90 articles, including 16 papers in the Nature publishing group, one in the Science group and 9 papers in the Physical Review Letters. Almost half of the total number of papers were published in the Physical Review B. About forty contributions have been presented in conferences among which 8 invited talks in international conferences on magnetism, energy materials and neutron scattering techniques.

The scientific highlights below showcase fundamental researches ranging from basic knowledge (H1-H3) to functional materials (H4-H6). These highlights focus on the topics where researchers of the MDN team are strongly involved. H1 reports studies performed in the frame of the PhD of Q. Faure evidencing a new topological phase transition under magnetic field in an Ising-like spin chain. H2 illustrates how fundamental questions on correlated 5f electronic states can be addressed by new instrumentation, here the 40 T pulsed magnetic field cryo-magnet developed in the frame of the MAGFINS ANR. In H3, studies on the unconventional superconductor $CeCoIn_5$, both by a Swiss team and by our group, show the connection between dynamical and static magnetic properties, their interplay with superconductivity and the switching of magneto-superconducting domains. H4 presents results on multiferroics systems studied within the ANR DYMAGE and the PhD of the ILL student I. Urcelay-Olabarria. Finally, H5 and H6 deal with energy materials and associated thermal properties of solids through the study of compounds for room temperature magnetic refrigeration (joint CEA and JCNS PhD of N. Biniskos) and thermoelectricity with the determination of phonon lifetime by spin echo methods on three-axis spectrometer.

H1: Topological quantum phase transition in the Ising-like antiferromagnetic spin chain $BaCo_2V_2O_8$
Publication: Nature Physics 14 (2018) 867.

The universal concept of phase transitions, usually characterized by a symmetry breaking, has played a crucial role in the description of many phenomena in condensed matter physics. However, since the seminal ideas of Berezinskii, Kosterlitz and Thouless (Nobel Prize in Physics 2016), topological excitations are at the heart of our understanding of a novel class of phase transitions. Here, we provide evidence for a quantum phase transition separating two unconventional magnetic phases characterized by different kinds of topological excitations.

Topology has brought a new insight into our understanding of condensed matter physics. For instance, topological phase transitions exhibit no symmetry breaking and are characterized by topological defects / excitations (discontinuities that cannot be removed by a continuous deformation unless annihilated by their anti-defects). In most cases, those transitions are controlled by a single type of topological objects, for instance solitons (kinks and 360° rotation of a pendulum for instance) or vortices. However, there are richer and less studied classes of topological phase transitions where two different sets of topological excitations compete with each other.

This is what we have found in the magnetic oxide $BaCo_2V_2O_8$. In this material, which consists in chains of Co^{2+} carrying effective anisotropic spin-1/2, the spins are strongly coupled antiparallel to each other in the "Ising" **c**-direction (see Figure 1a). Due to crystallographic peculiarities, applying a transverse uniform magnetic field H along **b** produces an additional transverse and staggered magnetic field along **a**. The latter competes with the Ising anisotropy and forces the spins to bend along **a** (see Figure 1b), as evidenced by neutron diffraction on D23, driving a phase transition beyond H_c = 10 T.

The nature of this transition was further elucidated by combining inelastic neutron scattering experiments and numerical infinite Time Evolving Block Decimation (iTEBD) calculations. We could show that for H = 0, the spin dynamics is governed by an assembly of spinons carrying a spin ½. Because of inter-chain couplings, they are tied up in bound states coming in three flavors (2 transverse degenerate, polarized along **a** and **b**, and 1 longitudinal, polarized along **c**), forming a discrete excitation spectrum. Above H_c, however, the low-energy spectrum essentially consists in two well-defined branches, corresponding to spin 1 transverse modes polarized along **b** and **c**. These excitations can be understood as kinetic bound states formed by two *adjacent* spinons strongly attached by the staggered field and moving together along the chain. Figure 2 illustrates how the magnetic field operates this change. Importantly, the very good agreement between the experimental and numerical results (see Fig. 2a and 2b, respectively) fully validates our model.

The magnetic excitations characterizing the phases on both sides of the transition are thus two different types of solitonic topological objects in competition. Our experimental results can be further described in field theory by the so-called "double sine-Gordon model", that can be relevant well beyond the field of magnetism.

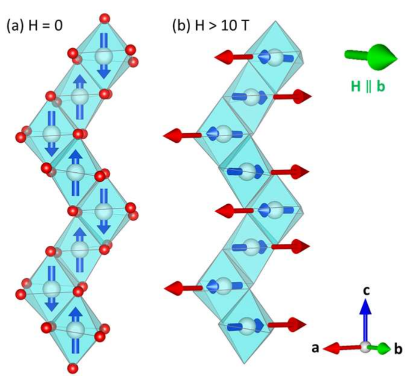


Figure 1: (a) Zero field magnetic structure (blue arrows) of a single Co^{2+} screw chain of $BaCo_2V_2O_8$ (blue and red spheres are Co and O respectively). (b) Applying a magnetic field along **b** creates an additional staggered field along **a** (red arrows), which produces a new rotated magnetic arrangement shown by the blue arrows.

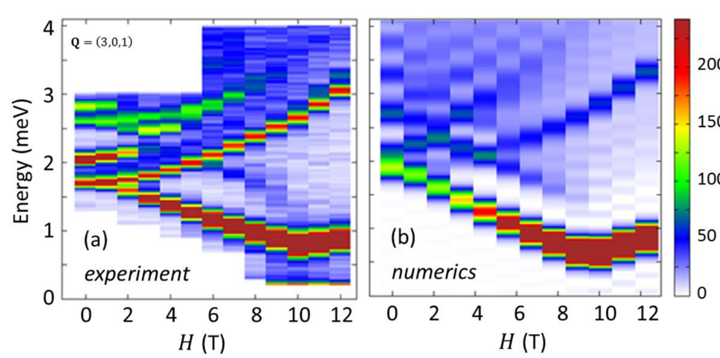


Figure 2: (a) Experimental intensity color map showing the magnetic field dependence of the magnetic excitations in $BaCo_2V_2O_8$ for a transverse field applied along the **b**-axis and for **Q** = (3, 0, 1). (b) Theoretical intensity color map to be compared to the experimental one, obtained from iTEBD calculations of the S(**Q**,E) neutron scattering dynamical structure factors. The intensity color scale shown on the right is in arbitrary units.

H2: Field-induced spin-density wave beyond hidden order in URu_2Si_2

Publication: Nature Comm. 7 (2016) 13075.

Over the last four decades, URu_2Si_2 remains one of the most enigmatic strongly-correlated-electron systems. Its physics has revealed a unique richness and offers a fertile testing ground for new concepts in condensed matter science. In spite of intense research, no consensus on the order parameter of its low-temperature hidden-order (HO) phase exists. Here, thanks to neutron diffraction under new pulsed magnetic fields up to 40 T, we identify the field-induced phases of URu_2Si_2 as a spin-density-wave state. The transition to the spin-density wave represents a unique touchstone for understanding the HO phase.

The application of extreme conditions permits to modify the ground state of a system and get new insights about it. In URu_2Si_2 under pressure, the HO state is replaced by an antiferromagnetic phase, whose order parameter is a moment aligned antiferromagnetically along **c** with the wavevector $\mathbf{k_0}$ = (0 0 1). Under a high magnetic field applied along **c**, a cascade of first-order phase transitions at the fields H_1 = 35 T, H_2 = 36-37 T (rising-falling fields), and H_3 = 39 T, leads to a polarized paramagnetic regime above H_3. Thanks to a new cryo-magnet (developed by the LNCMI-Toulouse, the CEA-Grenoble, and the ILL-Grenoble) allowing neutron diffraction up to 40 T, we have determined the magnetic structure of URu_2Si_2 in fields between 35 and 39 T.

After testing different wave-vectors, a magnetic signal has been detected at $\mathbf{k_1}$ = (0.6 0 0) and at (1.6 0 -1) (an equivalent position). The figure *a* shows the time profile of a magnetic field pulsed up to 38 T, as well as the time dependence of the diffracted neutron intensities recorded during 38-T pulses of these **Q**-vectors. Figure c emphasizes in the window 32-41 T, the field-dependence of the diffracted neutron intensities. The enhancement of the intensity at T = 2 K, absent at T = 18 K, shows that long-range ordering with wavevector $\mathbf{k_1}$ is established at high field and low temperature. At T = 2 K, the intensity with wavevector $\mathbf{k_1}$ is enhanced in fields higher than H_1 = 35 T and drops down to the background level in fields higher than H_3 = 39 T. With a normalization on structural Bragg peak, the magnetic intensity has been estimated to correspond to a Fourier component (m_k) of 0.25 µB/U.

In the HO state, the magnetic fluctuations peaked at the hot wavevectors $\mathbf{k_0}$ and $\mathbf{k_1}$ indicate the proximity of quantum phase transitions associated with long-range ordering with the same wavevectors. With pressure, the disappearance of the hidden order coincides with a transfer of weight from magnetic fluctuations with wavevector $\mathbf{k_0}$ into antiferromagnetic order with the same wavevector. With magnetic field, we have observed here that a spin-density wave is stabilized with the wavevector $\mathbf{k_1}$, which may coincide with a loss of intersite magnetic fluctuations, as indicated by the enhanced magnetization (see Figure b).

In an itinerant picture of magnetism, long-range magnetic ordering with a wavevector $\mathbf{k_m}$ can be related to a partial or complete nesting of two parts of the Fermi surface. In URu_2Si_2, 5f electrons hybridize with electrons from the conduction bands. Modifications of the Fermi surface and carrier mobility accompany the establishment of the HO phase at the temperature T_0, and Fermi surface nestings with the wavevectors $\mathbf{k_0}$ and $\mathbf{k_1}$ can be identified. However, no long-range magnetic order has been reported yet in the HO phase of URu_2Si_2. Resulting from the Fermi surface reconstruction at H_1, the nesting with $\mathbf{k_1}$ is probably reinforced, leading to the onset of the spin-density wave with the wavevector $\mathbf{k_1}$.

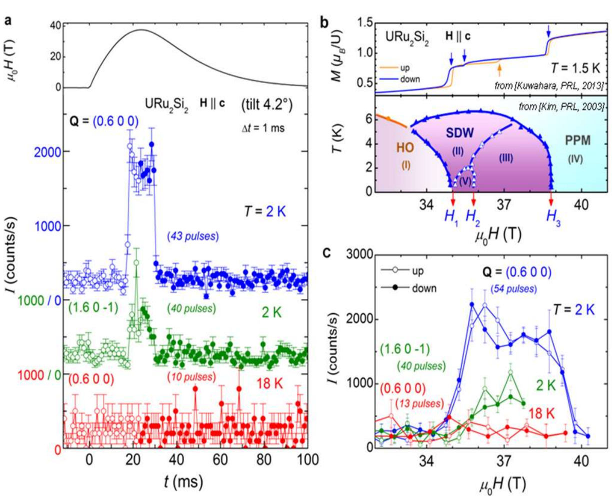


Figure a. Time profile of a magnetic field pulsed up to 38 T, and corresponding time-dependence of the neutron diffracted intensity at **Q** = (0.6 0 0) and **Q** = (1.6 0 -1). **b.** Magnetization versus magnetic field at *T* = 1.5 K and magnetic-field-temperature phase diagram of URu_2Si_2. **c.** Field-dependence of the neutron diffracted intensities in fields up to 40.5 T.

H3: Interplay between magnetism and superconductivity in $CeCoIn_5$ Switching of magnetic domains reveals spatially inhomogeneous superconductivity

Publication: Nature Physics 10 (2014) 126.

The interplay of magnetic and charge fluctuations can lead to quantum phases with exceptional electronic properties. A case in point is magnetically driven superconductivity where magnetic correlations fundamentally affect the underlying symmetry and generate new physical properties. The superconducting wavefunction in most known magnetic superconductors does not break translational symmetry. However, it has been predicted that modulated triplet p-wave superconductivity occurs in singlet d-wave superconductors with spin-density-wave (SDW) order. Here we report evidence for the presence of a spatially inhomogeneous p-wave Cooper pair-density wave in $CeCoIn_5$. We show that the SDW domains can be switched completely by a tiny change of the magnetic field direction, which is naturally explained by the presence of triplet superconductivity. Further, the Q-phase emerges in a common magneto-superconducting quantum critical point. The Q-phase of $CeCoIn_5$ thus represents an example where spatially modulated superconductivity is associated with SDW order.

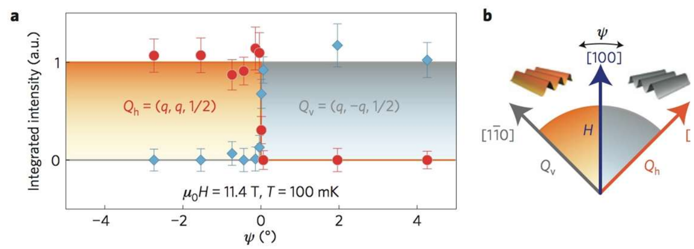


Figure: Switching of magneto-superconducting domains. **a**, Integrated intensity of the Q_h - (red circles) and the Q_v -domain (blue diamonds) as a function of the tilt angle Ψ (see sketch **b**). A mono-domain population is observed for $|\Psi| \geq 0.05°$, which can be macroscopically switched in a sharp transition. Thus, the magnetic field direction allows direct access and control of the complex quantum state in the Q-phase.

H4: Ising Incommensurate Spin Resonance of $CeCoIn_5$: A dynamical precursor of the Q-phase

Publication: Phys. Rev. Letters 115 (2015) 037001.

The concept of soft mode is central to condensed matter physics; it emphasizes the relationship between the excitation spectrum and the ground state of different phases of matter in a variety of situation ranging from lattice dynamical instability in ferroelectrics to Bose-Einstein condensation of magnons in magnetic insulators. Unconventional superconductivity modifies the magnetic excitation spectrum of a metal by a feedback effect corresponding to the apparition of a new collective mode, the spin resonance. Here it is shown by detailed inelastic neutron scattering experiments performed on the model *d*-wave unconventional superconductor $CeCoIn_5$ that the spin resonance mode has the same symmetry as the adjacent field induced magnetic ordered phase, the Q-phase. Up to now the spin resonance of $CeCoIn_5$ was believed to be peaked at the antiferromagnetic wave-vector $\mathbf{Q}_{AF}$=(0.5, 0.5, 0.5). However, a new generation of experiments, made possible by the IN12 upgrade, allow to evidence that the resonance peak position is in fact incommensurate and peaked at $\mathbf{Q}_{IC}$=(0.45, 0.45, 0.5) corresponding to the propagation vector of the Q-phase. It is also found that the resonance spin fluctuations are of Ising nature and their direction corresponds to the one of the magnetically ordered moments of the Q-phase. These facts strongly support a scenario where the static order is realized by a condensation of the superconducting spin resonance.

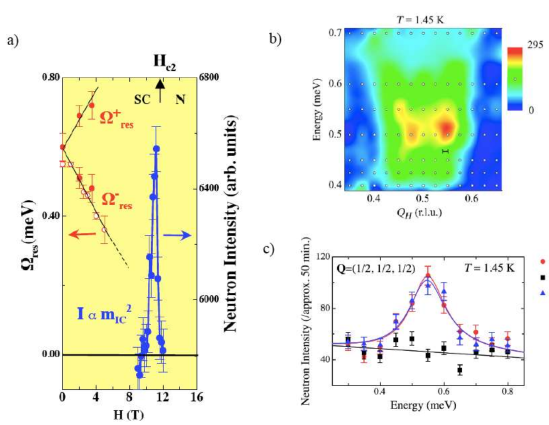


Figure: a) The spin resonance energy (red points) splits under magnetic field while the incommensurate ordering, the Q-phase, (blue points) induced at 10.5 T in the superconducting phase disappears at H_{c2} in the normal phase. b) Color-coded intensity plot of the INS spectra as a function of Q_H and E for $\mathbf{Q}$=(Q_H, Q_H, 0.5) at 1.45 K c) Polarized INS spectra taken at $\mathbf{Q}_{AF}$=(0.5, 0.5, 0.5) in the spin flip channel for the three canonical polarization directions.

H4: Multiferroics

Multiferroics are materials that combine two (or more) types of ferroic ordering, most commonly ferroelectricity and either ferro- or antiferromagnetism. Such materials in which ferroelectric and magnetic orderings are coupled have huge potential applications: indeed, a magnetic field can control the electric polarization, and an electric field can control the magnetic structure. This property is particularly interesting for devices with switchable magnetic states. How the magnetoelectricity occurs at the atomic level is therefore a hot topic.

Magnetoelectric effect and phase transitions in CuO in external magnetic fields

Publication: Nature Communication 7 (2016) 10295.

Apart from being so far the only known binary multiferroic compound, CuO has a much higher transition temperature into the multiferroic state, 230K, than any other known material in which the electric polarization is induced by spontaneous magnetic order, typically lower than 100K. Although the magnetically induced ferroelectricity of CuO is firmly established, no magnetoelectric effect has been observed so far as direct crosstalk between bulk magnetization and electric polarization counterparts. Bulk measurements show that high magnetic fields of ~ 50 T are able to suppress the helical modulation of the spins in the multiferroic phase and dramatically affect the electric polarization. Moreover, just below the spontaneous transition from commensurate (paraelectric) to incommensurate (ferroelectric) structures at 213 K, even modest magnetic fields induce a transition into the incommensurate structure and then suppress it at higher field. Thus, remarkable hidden magnetoelectric features are uncovered, establishing CuO as prototype multiferroic with abundance of competitive magnetic interactions.

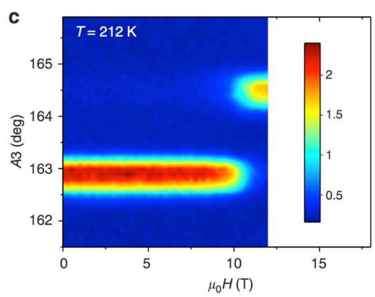


Figure: Commensurate to incommensurate field-induced phase transition in CuO at 212 K: neutron rocking curves (rotation of the crystal around the diffractometer vertical axis) as a function of H. The position A3 ~162.8° corresponds to **q** = (0.500 0.000 0.500), whereas A3 ~ 164.5° corresponds to **q** = (0.509 0.000 0.483).

X phase of $MnWO_4$

Publication: Physical Review B 90 (2014) 024408.

The natural mineral hübnerite $MnWO_4$ is regarded as one of the best examples of a magnetically induced multiferroic system with spiral magnetic structure. In zero magnetic field, a polar cycloidal magnetic structure, the so-called AF2 phase, exists between about 7.4 and 12.5 K and is sandwiched between two nonpolar phases with collinear magnetic structures known as AF1 and AF3. A long-standing issue, the magnetic structure of the so-called X phase of $MnWO_4$, has been solved by single-crystal neutron diffraction. Emerging from the AF2 ferroelectric phase which presents an electric polarization P//b, this phase is reached by application of a magnetic field along the b axis and is characterized by a flop of the electric polarization towards the a axis. We have identified this new phase as a reoriented cycloidal spin structure, AF2'. Unlike for the usual magnetic-field-induced spin-flop transitions, this high-field flopped phase does not directly develop from the low-field phase but emerges via the nonpolar collinear commensurate AF1 phase, which creeps in between. This evolution is in good agreement with the observed field-induced change in P_b and P_a electric polarization as well as with magnetic susceptibility data.

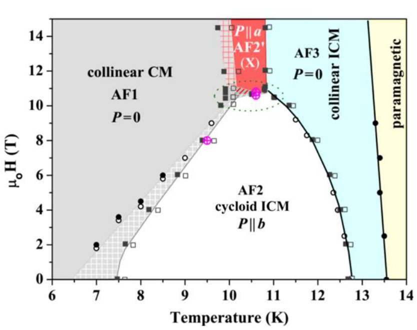


Figure 1: Magnetic phase diagram of $MnWO_4$ (H//b). The dash green ellipse indicates the part of the phase diagram on which our neutron diffraction study is focused (CM= commensurate ; ICM = incommensurate).

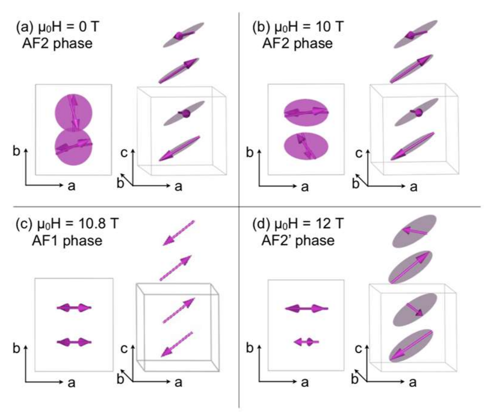


Figure 2: Magnetic structures under (a) zero field and (b) 10 T, (c) 10.8 T, and (d) 12 T field applied along the b axis at 10.6(2)

H5: Spin fluctuations drive the inverse magnetocaloric effect of Mn_5Si_3

Publication: Phys. Rev. Lett. 120 (2018) 257205.

Interest for magnetocaloric materials has been increasing in the last decades in view of applications for room temperature refrigeration. In this context the system $Mn_{5-x}Fe_xSi_3$ has recently been investigated for its moderate magnetocaloric effect (MCE) corresponding to the cooling by adiabatic demagnetization. In that respect, the compounds with a composition around x=4 present, near 300 K, an isothermal entropy change of about -2 J/kg.K for a field variation between 0 and 2 T. In contrast to the common direct MCE, the parent compound Mn_5Si_3 exhibits, below 70 K, an inverse MCE, a cooling by adiabatic magnetization, of comparable magnitude, but opposite sign. On cooling from the paramagnetic state, Mn_5Si_3 undergoes two successive antiferromagnetic phase transitions towards AFM2 ($T_{N2}\approx100$ K) and AFM1 ($T_{N1}\approx66$ K) states. AFM2 is a collinear phase while AFM1 is a non-collinear and non-coplanar magnetic moments phase. In both antiferromagnetic states, magnetic and non-magnetic states of Mn coexist among the different crystallographic sites. The inverse MCE is associated with the transition from AFM1 to AFM2 under magnetic field.

Inelastic neutron scattering (INS) studies were performed in Mn_5Si_3 in order to get microscopic information on its spin and lattice dynamics and to reveal key ingredients at play in its magneto-thermodynamics properties. By using polarized INS, we have shown that the high temperature antiferromagnetic phase (AFM2) is characterized by an unusual magnetic excitation spectrum, where both propagative spin waves and diffuse spin fluctuations coexist. Moreover, we have shown that the cooling by adiabatic magnetization is associated with field induced spin fluctuations. Indeed, when the magnetic field induces the switch from AFM1 to AFM2, the peculiar magnetic excitation spectrum of AFM2 is restored starting from a zero field AFM1 phase, where only spin-waves exist. Since spin fluctuations involve more microscopic states than the discrete spin-wave modes, their reappearance increases the magnetic entropy. This peculiar situation leads to the counterintuitive effect of an entropy increase associated with a magnetic field increase, which corresponds to the inverse MCE. These properties of functional interest are intricately related to fundamental questions of magnetism: the competition between bounding and magnetism in Mn-based systems and geometrical frustration in metallic magnets.

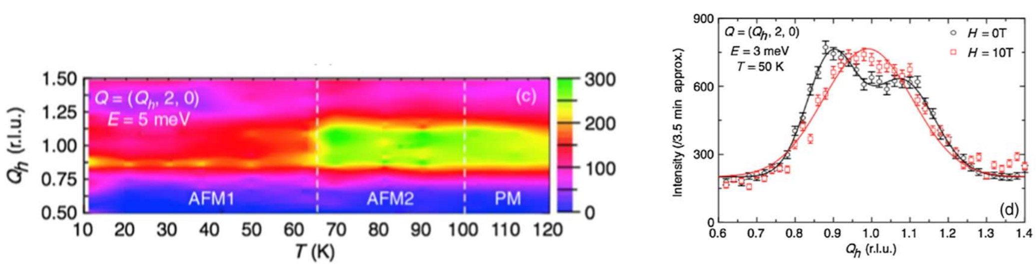


Figure: (Left) Color-coded intensity plot of the INS data collected at a constant energy transfer of 5 meV as a function of **Q**=(Q_h,2,0) and temperature. The broad diffuse signal characteristic of the PM and AFM2 states is replaced by sharp spin-waves in the AFM1 phase. (Right) Spectra obtained at 50 K around **Q**=(Q_h,2,0) under 0 and 10 T magnetic field for an energy transfer of 3meV. The sharp spin-waves at 0 T are replaced by broad signal characteristic of the AFM2 phase at 10 T.

H6: Direct measurement of individual phonon lifetimes in the clathrate compound $Ba_{7.81}Ge_{40.67}Au_{5.33}$

Publication: Nature Comm. 8 (2017) 491

The engineering of thermal conductivity in semiconducting materials is a central issue in the development of modern technologies. Low thermal conductivity is important in materials used in technology products as it provides thermal insulation and thus the reduction of heat transfer, ensuring that products do not overheat. It is also important for thermoelectrics, as it drives its efficiency. This study provides a direct quantitative measurement of phonon lifetimes in a clathrate, providing a novel picture of thermal conductivity in complex materials.

Understanding how heat propagates in a material is a key issue in many different fields. For instance, with thermoelectric materials, which can transform heat into electricity, researchers try to reduce the thermal conductivity - a key parameter in the efficiency equation of these materials - as much as possible. However, an understanding of the detailed mechanism of heat propagation in structurally complex compounds is still lacking. The heat is carried out by phonons that, while travelling in a material, collide with defects or other phonons, in turn reducing the thermal conductivity. The more collisions there are, the smaller the thermal conductivity. This is measured by the mean distance a phonon travels without collision (phonon mean free path) or by the mean time between two phonon collisions (phonon lifetime): the phonon mean free path is the product of the phonon lifetime by the phonon velocity.

We have experimentally measured at the ILL, for the first time, the phonon lifetime of a thermoelectric material clathrate renowned for its very low, 'glass-like', thermal conductivity. This material is a periodic packing of cages constructed with Ge and Au atoms enclosing heavy Ba atoms. For a long time, it was believed that the glass-like thermal conductivity of clathrates resulted from 'phonon glass' behaviour, with very short mean free paths of the order of interatomic distances (*i.e.* 0.3-0.5 nm). As it has been shown in previous experiments on clathrates, an overview of the dispersion measured on three-axis instruments at the LLB and on the time-of-flight spectrometer IN5 at the ILL demonstrates that the acoustic phonons that are effective in carrying heat are in fact confined to a very limited wavevector and energy range, typically of between 0 to 6 meV.

Measuring long lifetime with triple-axis instruments is challenging because of the energy resolution that can be achieved. Using a high-resolution set-up and detailed comparison with a Ge reference sample already gives a very long, measured phonon mean free path. However, a more conclusive experimental input is achieved using the neutron spin-echo resonance techniques installed on the IN22 three-axis spectrometer. Thanks to the extremely high quality of the single grain sample used, with an overall mosaic spread equal to 0.01 °, a phonon mean free path by one order of magnitude larger than expected by thermal conductivity can be measured.

Our results were very surprising: a phonon mean free path ranging from tens to hundreds of nm, much larger than that expected in the 'phonon glass' scenario. In particular, even close to the Brillouin zone boundary, phonon mean free paths were of the order 15 nm, *i.e* 10 unit-cells. This is shown in the figure, where the phonon lifetime is illustrated by the decay of the wave representing the phonon frequency. Such a result can in fact be reconciled with a simple theory of heat propagation, once the number of acoustic phonons carrying heat is properly considered.

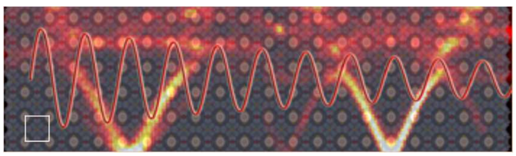


Figure: The background illustrates the periodic structure of the clathrate, with a unit cell (shown as a white square on the left with a parameter 1 nm). The Ba atoms in red are enclosed in the cages. The colored curve is the inelastic neutron scattering as measured on the neutron time-of-flight spectrometer IN5 at room temperature around the strong zone centre Γ_{600} (Q = 3.6 $Å^{-1}$). The decaying sinusoidal curve illustrates the phonon propagation and its time decay (or lifetime) for a phonon wavelength equal to 2 nm.

4- Organisation and life of the MDN team

Steering, life, organisation in the unit

Steering: Each year an annual report on each instrument is provided and is evaluated by the ILL scientific council. The steering committee of the 2FDN, which meets once a year, provides the global recommendations of CEA, CNRS and UGA authorities for the operation of French CRG instruments at ILL. The management board of 2FDN, which includes the MEM director and the MDN group leader, meets every month to organize the daily life of the 2FDN as well as to define operational and technical perspectives for neutron scattering in France.

Animation: The affectation of the staff on the instruments in given in the annex MDN-A. Technical meetings are organized on average every two months and are aiming to discuss the maintenance work to be performed on the instruments (cryogeny, mechanics, and electronics) and the technical upgrades. The scientists participate to the ILL group meetings they are attached to: ILL diffraction group for D23 and ILL spectroscopy group for IN22 and IN12. The opportunity of internal seminars (by group members including post-doctoral PhD students or users of our beamlines) are transformed into formal seminars mostly in the frame of college 4 or 5 seminars at ILL and unconventional magnetism seminars at the Institut Néel.

Beamtime allocation: The overall beamtime is distributed by three different selection panels at ILL, Maier-Leibnitz Zentrum (external German beamtime and internal JCNS) and "tables rondes" of the 2FDN. A new website (Phoenix) was created by LLB and 2FDN in 2016 in order to submit French proposals for the CRGs and the LLB instruments. The Swiss beamtime is attributed through the ILL selection panel, picking up high grade proposals rejected by national balance. The repartition of beamtime is given in the Table below. The internal time acts as a buffer due to the limited scientific staff and it serves mostly for instrumentation development and tests. In the past years, due to the erratic operation of the ILL reactor, this internal time has been in practice cut and largely given back to the external users.

%	D23	IN22	IN12
ILL	30	30	30
CRG-JCNS	11	12	35
CRG-CEA Swiss contract	11	15	8
CRG-CEA 2FDN time	15	15	13
Internal time	33	28	14

It is to be specified that the reception of users as concerns accommodation and safety formalities is provided by ILL services as stipulated in our CRG contracts and that logistics for 2FDN selection panel and users involves administrative work done by LLB staff.

Parity; scientific integrity; health and safety; sustainable development and environmental impacts; intellectual property and business intelligence.

Parity: The only woman of the team is assistant professor at the UGA. For PhD students, there is a better gender balance over the evaluation period.

Safety: A safety responsible is present in the team and works closely with ILL and CEA safety officers.

Scientific integrity: The deployment of Digital Object Identifier (DOI) has started at ILL in 2014 and such a permanent number is affected to each experiment carried out on IN22 and IN12 and will be implemented on D23 in relation with the future software change. Data DOIs promote experiments to peers and potential funding bodies, as well as to publishers and journals. DOI are cited in publications arising from a given experiment. An internet portal gives public access to the data after a non-disclosure period during which access to data is restricted to the experimental team.

Five-year project and strategy of the MDN team

MDN SWOT analysis

Strengths	***Weaknesses***
State to the art neutron instrumentation -> *attracts users, allow to perform unique experiments.* Experienced staff -> *high success rate of experiments, high impact publications*	Lack of manpower for local contact duties, aging staff -> *Psychosocial risk, accident risk* Balance between users platform and own research -> *Lack of continuity* Fragmentation of beamtime between many bodies -> *Lack of visibility* Lack of recognition by Institute of the role for users -> *Lack of support for funding and staffing* Complexity of organization of neutrons in France -> *Loss of efficiency*
Opportunities	***Threats/Risks***
Orphée reactor shutdown -> *Shift of barycentre of neutron scattering activity towards Grenoble* New CRG instruments at ILL -> *Collaborations and resourcing for the MDN group* Development of 2FDN -> *Visibility and recognition of the role of MDN*	Lack of budget for instrument operation and maintenance -> *reduction of activity* Absence of recruitment -> *reduction of activity, risks for staff* Diversion of users, of single crystal sample growth -> *reduction of activity* Lack of investment on instruments -> *fatal failure, loss of neutron cycle* Unanticipated ILL technical or administrative shutdown -> *end of activity*

Structure, workforce and scientific orientations

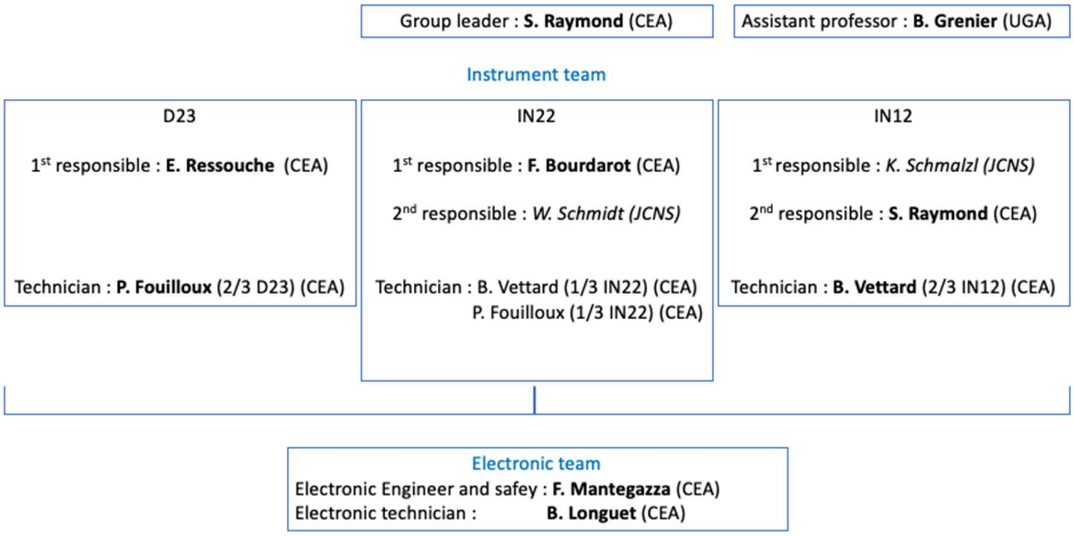
The perspectives for MDN laboratory are strongly tied to its ecosystem, the operation of the ILL reactor and the situation of French neutron scattering with the closure of the Orphée reactor. The roadmap of the 2FDN (http://2fdn.neel.cnrs.fr/IMG/pdf/Feuillederoute_2FDN.pdf) presents a frame allowing to keep an expertise in neutron scattering techniques in France. It is proposed to run a suite of CRG instruments at ILL (and at PSI, Switzerland) and a national compact accelerator driven neutron source in parallel to the rise of the European Spallation Source (ESS).

In this scheme, it is foreseen to perpetuate the three instruments in which MDN is involved and the important dates are the renewal of the IN12 contract and the subcontract with JCNS for 2021 and the renewal of D23 and IN22 contracts for 2022. It is to be noted that ILL has also to overcome its difficulties with French Nuclear Safety Authority and to succeed its renovation project (Endurance) in order to operate fully after the long shutdown foreseen for 2020 (about 12 months). The constructions of new CRG instruments by LLB at ILL will lead to increasing collaboration with this laboratory. The future cold three-axis spectrometer GAPS has obvious similarities to IN12 and together with IN22 and IN6, this will constitute a critical mass of people working on neutron spectroscopy. The future small angle scattering instrument SAM will have a polarized neutron option with spin echo techniques similar to the ones developed on IN22. Thanks to the good relationships between colleagues of MDN and LLB, a constructive working is expected. Nonetheless in order to fulfil these missions, the recruitment of a scientist and an increase of funding are needed. This will allow to stop the beamtime access contract with Switzerland and shift the corresponding beamtime for the French users.

As concern scientific orientations, basic research constitutes the "core business" of MDN given the specificity of its neutron scattering instrumentation. While there is no scrutiny right on the scientific topics proposed by the users, the trend at MDN is to develop the studies on functional materials, energy materials and materials for information storage. On another hand the excellence in fundamental problems should be kept. Among others, medium term studies will concern the effect of longitudinal magnetic field and impurities on topological phase transitions and the study of the magnetic structure and spin dynamics of the frustrated Cairo pentagon lattice system. Multiferroics will be investigated through new materials recently synthetized like non-centrosymmetric ferrites (e.g. Brownmillerite Ca_2FeAlO_5). For thermoelectricity, through the PhD of S. Turner and the spin echo option on IN22, new high entropy alloys and electron-phonon coupling in selected materials exhibiting temperature dependent Seebeck effect will be studied. On magnetocaloric compounds, the conclusion obtained on materials for room temperature refrigeration will be strengthened and materials for adiabatic demagnetization refrigeration at low temperatures (below 2 K) like $Yb_3Ga_5O_{12}$ will be investigated (space applications). The main instrumentation project is the upgrade of the spin echo option on IN22 with a conversion from a prototype to a user-friendly option. A part concerning the capacitor is already taken over by LLB engineers and technicians. Other instrumentation projects are the commissioning of UFO on IN12 by demonstrating its performance using well-studied samples. A change of D23 software in line with the choices of the ILL diffraction group is also foreseen and other perspectives for D23 include an improvement of the polarized neutrons setup.

To conclude, over the last five years, MDN changed its paradigm from primarily in-house oriented research to primarily users-oriented activity, changed from SPSMS to MEM, changed from INAC to IRIG. Despite these continuous changes and their associated extra work burden, the MDN laboratory kept providing neutron beamtime on three instruments and a high quality of research output. This was realized at the expense of working conditions for the staff who is likely not able to maintain this rythm in the near future, especially when ILL will recover its full operation. Today, the challenge for MDN is unique and consists in running instruments at large scale facilities in a multiple ecosystem (IRIG, 2FDN, LLB) without sufficient recurrent funding and without recruitment over the past twenty years.

Operational organisation chart



Equipment, platforms

The laboratory is involved in the operation of three instruments at ILL and of a sample environment fleet.
Diffractometer D23
D23 is a two-axis diffractometer with a lifting detector installed on a thermal supermirror guide and can be operated in the range 1-3 Å. D23 options are:

- A graphite PG002 or a Cu200 monochromator.
- A polarized neutrons option (heusler111 in transmission).
- An independent analyser and detector bench on the lifting arm.
- A four-circle option.

Thermal neutron three axis spectrometer IN22
IN22 is installed on a thermal supermirror guide and can be operated in the 5-120 meV incident energy range. The available options are:

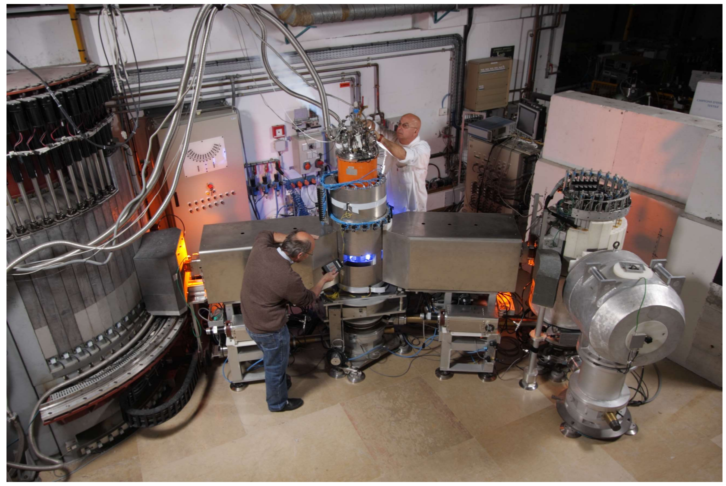
- A graphite PG002 or a Cu111 monochromator.
- A polarized neutrons option (Heusler monochromator and/or Heusler analyzer).
- A 40 T-pulsed magnetic field option.
- Zero field spin-echo option.

Cold neutron three axis spectrometer IN12
IN12 is installed on a cold supermirror guide. The velocity selector and graphite PG002 monochromator provide incident neutrons in the energy range 2.5-30 meV. IN12 options are:

- A polarized neutrons mode (transmission polarizing cavity and/or Heusler analyzer).
- A multi-analyser multi-detector option (in commissioning).

Sample environment
The MDN laboratory operates its own sample environment (and exchange it both ways with ILL instruments) :

- A 12 T vertical field magnet (asymmetric for polarized neutrons).
- A dilution fridge insert (50 mK for a cryostat or the 12 T magnet).
- An ^{3}He fridge insert (300 mK for a cryostat or the 12 T magnet).
- An electric field stick (15 kV).
- A sphE. al polarization device CRYOPAD.
- Five helium flow cryostats.

12 T vertical field magnet on D23 and spin echo option on IN22

Research products and activities of the MDN team

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Production in conferences / congresses and research seminars

Conference proceedings

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Contributions to international conferences/workshops (invited talks are listed again in another section)

2018

Polarized Neutrons for Condensed Matter Investigations 2018 (Abingdon, UK) :

K. Beauvois (oral), N. Biniskos (invited), S. Raymond (poster)

Highly Frustrated Magnetism 2018 (Davis, USA) :

K. Beauvois (poster)

International Conference on Magnetism 2018 (San Francisco, USA) :

B. Grenier (invited), Q. Faure (poster)

Institut Laue Langevin & European Spallation Source user meeting (Grenoble, France):

B. Grenier (oral)

New perspectives for low-temperature refrigeration with advanced magnetocaloric materials (Cergy, France):

S. Raymond (invited)

2017

International Conference on Neutron Scattering 2017 (Daejeon, Corea) :

N. Biniskos (poster), B. Grenier (oral)

Strongly Correlated Electron Systems 2017 (Prague, Czech republic) :

Q. Faure (oral)

EUROMAT 2017 (Thessaloniki, Greece) :

N. Biniskos (oral)

APS march meeting 2017 (New Orleans, USA):

S. Raymond (invited)

2016

Single-crystal Diffraction with Polarized Neutrons, Flipper 2016 (Tutzing, Germany):

S. Raymond (invited)

Highly Frustrated Magnetism 2016 (Taipei, Taiwan):

B. Grenier (invited)

International Conference on Magnetic Refrigeration at Room Temperature, 2016 (Turin, Italy):

N. Biniskos (poster)

Neutrons for Energy, MLZ conference 2016 (Bad Reichenhall, Germany):

N. Biniskos (invited)

Workshop From Matter to Materials and Life, 2016 (Hamburg, Germany):

N. Biniskos (poster)

2015

European Conference on Neutron Scattering 2015 (Zaragoza, Spain):

B. Grenier (oral), S. Raymond (oral)

2014

Strongly Correlated Electron Systems 2014 (Grenoble, France):

S. Raymond (poster), B. Grenier (poster), E. Ressouche (invited)

International workshop "Quantum Spin Dynamics: From Exotic Excitations to Novel Transport and Non-Equilibrium Phenomena" (Dresden, Germany):

B. Grenier (oral)

Contribution to national conferences/workshops in France

Journées de la Matière Condensée 2018 (Grenoble) :

K. Beauvois (oral), F. Bourdarot (oral), Q. Faure (oral), S. Raymond (poster)

Journées De la Neutronique 2018 (Roquebrune sur Argens):

K. Beauvois (oral)

Journées De la Neutronique 2017 (Carry le Rouet):

N. Biniskos (oral), B. Grenier (invited)

Journées De la Neutronique 2016 (Carqueiranne):

Q. Faure (oral)

Journées De la Neutronique 2015 (Evian):

S. Raymond (oral)

GDR Matériaux, Etats ElecTroniques, Interactions et Couplages non-Conventionnels, School 2018 (Banyuls-sur-mer):

Q. Faure (poster)

GDR Matériaux, Etats ElecTroniques, Interactions et Couplages non-Conventionnels 2017 (Latresne):

Q. Faure (oral)

GDR Matériaux et Interactions en Compétition 2015 (Mittelwihr):

B. Grenier (oral)

Complementarity between Optics and Neutron Spectroscopy in the THz domain, SON2017 (Grenoble):

Q. Faure (oral)

Contribution to national conferences/workshops in Germany

Deutsche Physikalishe Gesellschaft 2018 (Berlin): N. Biniskos (oral)

Deutsche Physikalishe Gesellschaft 2017 (Dresden): N. Biniskos (oral)

Deutsche Physikalishe Gesellschaft 2016 (Regensburg): N. Biniskos (oral)

Research Seminars

Paul Scherrer Institute, Switzerland : S. Raymond, Q. Faure

Forshungzentrum Jülich, Germany : S. Raymond

ETH Zurich, Switzerland : B. Grenier

Laboratoire Léon Brillouin, Saclay, France : B. Grenier, Q. Faure,

Institut Néel, Grenoble, France : Q. Faure, S. Raymond

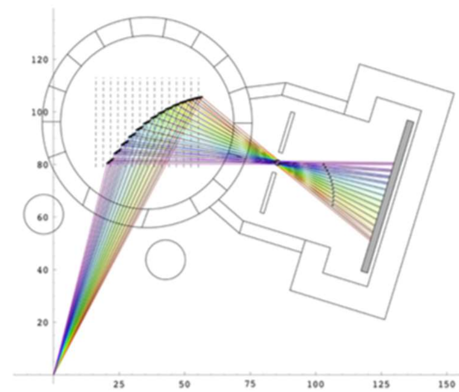
LNCMI, Toulouse, France : F. Bourdarot

Electronic tools and products

The group developped its own software for the instrument control of IN22 until 2017 and then shifted to the ILL software and participates in its upgrade.

Instruments and methodology

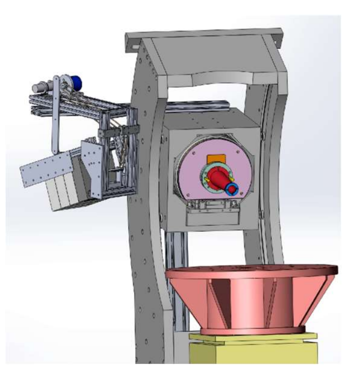
UFO on IN12 : IN12 is developing a multi analyser – multi detector option with more flexibility than existing RITA type instruments: IN12-UFO (Universal Focussing Option). This new secondary spectrometer is interchangeable with the present set-up. 15 analysers can be moved on rails and can be rotated individually, and a two-dimensional detector consisting of 15 horizontal position sensitive ^{3}He-tubes will allow to program simultaneous scans in Q-ω-space. A focal point that is realised by a movable diaphragm defines the only opening between the analysers and detector in order to prevent cross talk and to reduce the background. In the recent years several tests with neutrons have been performed. Issues concerning mechanical stability and background reduction have been identified and the necessary improvements have been successfully carried out. Currently software for scan construction and data extraction and analysis will be established for a user-friendly handling of this multiplex set-up. First promising results have been obtained on the magnetic excitation spectrum of $BaCo_2V_2O_8$ that was studied with the conventional TAS option. This option is developped by JCNS staff with technical help from CEA staff.

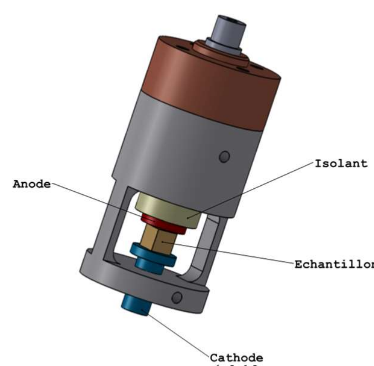


Schematic drawing of the IN12 UFO multi analyser – multi detector option.

40 T on IN22 : Within the framework of the MAGFINS ANR project (CEA/INAC/SPSMS, LNCMI/Toulouse, ILL and Institut Ne□el collaboration), we have developed a new pulsed-field set-up for the CRG thermal three-axis spectrometer IN22, optionally transformed into a powerful diffractometer (high counting rate ^{3}He detector, time-resolved acquisition system). The challenge was to design and built a high field, long pulse and high repetition rate, pulsed-field cryo-magnet, by strongly improving the nitrogen-based cooling system. Successful measurements with this new cryo-magnet were performed since 2014 with several publications. On the technical side, these first results confirmed the main characteristics of the device (nominal field up to 40 T (30 T previously), large pulse duration ~100 ms (10 times larger than before), pulse repetition rate of 1 minute at 20 T and 6 minutes at 40 T (the same than before but with a larger maximum field), liquid nitrogen consumption of about 600 l/day, but a consumption of He 5 times lowers than before. A new high-counting rate ^{3}He multi-wire detector (collaboration with Joint Institute of Nuclear Physics, Dubna) is also available. It is used when the diffracted beam has a rate larger than 15000c/s as its saturation is above 1000000c/s. Because of a lower efficiency (70%) it is not used when the rate is lower than 15000c/s (efficiency of usual ^{3}He detector ≈100%).

Analyzer for D23 : in order to improve the signal to noise ratio, a PG analyzer has been installed on the lifting arm of D23 (and therefore is not limited to the equatorial plane). This device was built as a full option with its own detector allowing for a quick change between the two configurations during an experiment if needed. It has a fixed diffraction angle, that is a fixed k_f : the incoming wavelength should be adapted to this value (and corresponds to k_f =2.662 Å^{-1}). The necessary **Q**-space trajectory corrections for the scattering angles were implemented in the software. This development was fully made by the MDN team from the conception to the realization. It was funded by the ANR DYMAGE.





Layout of the analyzer option on D23. Vacuum chamber with electrodes.

Electric field stick : For studying phenomena in solid-state physics, temperature, magnetic field, and pressure are the most common parameters that one can vary during an experiment. With the resurgent interest in magneto-electric multiferroics materials, electric field emerges as a necessary supplementary control

parameter. However, since most of the experiments are performed at low temperature, in the presence of an He exchange gas to assure good thermal contact between the sample and the cryogenic device, a strong limitation in the applied voltage on the sample is due to the break down voltage of the gas. To overcome this dificulty, we have designed a stick with a sample containing vacuum chamber that fits into all cryomagnets and standard cryostats at our disposal. The stick allows for voltages up to 15 kV on typically 5x5x5 mm^3 samples, and can be adapted for different scattering geometries. This project was funded by the ANR DYMAGE and fully developped by the MDN team.

Other products

The delivery of the instrument beamtime is addressed in the main text (section 3).

Reviewing activities

Papers

32 papers were rewieved among which 94% for the American Physical Society (Phys. Rev. B, Phys. Rev. Lett.).

1 research project was evaluated for the 2017 program « Canada 150 research chairs ».

Experiments

Members of the MDN team participated in the following experiments selection panel at large scale facilities :

FRM-II, Munich : Spectroscopy panel (S. Raymond).

Laboratoire Léon Brillouin, Saclay : Diffraction panel (E. Ressouche), Inelastic panel (F. Bourdarot).

CZ beamtime on THALES selection panel at ILL (L.-P. Regnault).

Expertise

L.P. Regnault is member of the Instrument Development Team of the HYSPEC spectrometer operated by Brookhaven National Laboratory at Oak Ridge National Laboratory.

Academic research grants

ANR MAGFINS 2010-2015

Coordinator : LNCMI Toulouse ILL

Other partners outside MDN : ILL and Institut Néel

Scope : Intrumentation and physics with pulsed field magnet.

Summary of outcome : realization of 40 T pulsed magnetic field for neutron scattering, 5 publications.

ANR DYMAGE 2013-2018

Coordinator : Institut Néel

Other partners outside MDN : Laboratoire Léon Brillouin, synchrotron SOLEIL

Scope : Identify, understand and manipulate magnetoelectric objects (structures, domains, excitations) in multiferroic compounds and associated neutron instrumentation.

Summary of outcome : Realization of electrical field stick and D23 analysers, 8 publications.

Swiss-CRG contract 2015-2016, 2017-2018, 2019

Partner : EPFL representing the Swiss state Secretariat for Education, Reseach and Innovation

Scope : beamtime access

Summary of outcome : 11 publications, contributes to the formation of Swiss PhD students (Perren, Mazzone, Bettler, Gurtinder, Simulus…)

Visiting senior scientists and post-doc

Post-doc

Ketty Beauvois, second responsible of D23 diffractometer

Funded by the Swiss-CRG contract

Publication in progress, 4 oral and poster presentations

Sumanta Chattopadhyay, second responsible of D23 diffractometer

Funded by the Swiss-CRG contract

2 publications

K. Zhernenkov (2013 - 2015), instrumentation for IN22

Funded by MAGFINS ANR

1 publication

Scientific visitors

Yuji Inagaki, Kyushu University (Japan) : grant from the Japan Society for Promotion of Science (summer 2018).

Quantum magnetism, sample growth and bulk measurements

Igor Zalyzniak, Brookhaven National Laboratory (USA) : invited professor grant from UGA (summer 2017).

Quantum magnetism, neutron instrumentation and methods

Temur Enik, Joint Institute for Nuclear Research, Dubna (Russia) (Sept.-Oct. 2014)

Development of high counting rate detector for the pulsed magnetic field on IN22

Scientific recognition

Invited talk in international conferences and workshops

Polarized Neutrons for Condensed Matter Investigations 2018 : N. Biniskos.

International Conference on Magnetism 2018 : B. Grenier

New perspectives for LT refrigeration with advanced magnetocaloric materials 2018 : S. Raymond

March meeting, APS 2017 : S. Raymond

Single-crystal Diffraction with Polarized Neutrons, Flipper 2016 : S. Raymond

Neutrons for Energy, MLZ conference 2016 : N. Biniskos

Highly Frustrated Magnetism 2016 : B. Grenier

Strongly Correlated Electron Systems 2014 : E. Ressouche

Interaction of the unit and of each team / theme with the non-academic world, impacts on economy, society, culture or health

Socio-economic interactions / Patents

None.

Socio-economic interactions

Indirect economic output (transport, accomodation) from the 660 continuous-flow users coming for experiments on our CRG instruments.

Expertise

Consulting was made by L.-P. Regnault for the AZ-system company specialized in mechanics for neutron scattering instrumentation and that has equipped D23, IN22 and IN12 since the construction of the CRG.

Public outreach

Scientific highlights for institutional websites (CEA, UGA):

The apparent inner calm of quantum materials

https://www.univ-grenoble-alpes.fr/news/headlines/the-apparent-inner-calm-of-quantum-materials-345513.kjsp

A phase transition between two phases ... that are nearly identical!

http://www.cea.fr/drf/english/Pages/News/Scientific-results/2017/a-phase-transition-between-two-phases-that-are-nearly-identical.aspx

Article in the japanese newspaper « The science News » (31/01/204) on the interplay between magnetism and superconductivity in $CeCoIn_5$.

Involvement of the MDN team in training through research

Educational outputs

A lecture by B. Grenier « Structures cristallographiques et magnétiques par diffraction des neutrons : la puissance des symétries » was video recorded in the 2018 school of the GDR « Materiaux Etats ElecTroniques, Interactions et Couplages non-Conventionnels ». It is avaialbe on the website of the school and usefull for people wishing to deepen their understanding of this topic.

Three chapters (Reminder: Magnetic structures description and determination by neutron diffraction, polarized neutron diffraction and magnetic excitations) were published in the *École thématique de la Société Française de la Neutronique*, Neutrons et Magnétisme , V. Simonet, B. Canals, J. Robert, S. Petit and H. Mutka eds, EDP Science 2014, Vol.13, pp.02001. They are accessible on internet and free of charge.

Scientific productions (articles, books, etc.) from theses

13 articles arising from MDN PhD students have been published in international journals. This includes articles from students of the present (Biniskos, Faure, Chaix) and from the former evaluation period (Canevet, Urcelay-Olabarria). In general all articles related to PhD works are indeed not published during the time of the PhD. Beyond the usual publication time, this is also due to the long process between proposal submission, beamtime allocation, experiment time and data analysis.

Training

Habilitation

S. Raymond (2006)

F. Bourdarot (2013)

E. Ressouche (2015)

PhD students affiliated at MDN laboratory

Quentin Faure

Quantum phase transition in the quasi-1D Ising-like antiferromagnet $BaCo_2V_2O_8$

UGA grant between Institut Néel and MDN

10 / 2015 – 11 / 2018

Directors / Supervisors by : V. Simonet (Institut Néel), B. Grenier (MDN)

Nikolaos Biniskos

Inelastic neutron scattering on magnetocaloric compounds.

1/2 JCNS and 1/2 CEA grants

06 / 2015 – 09 / 2018

Directors / Supervisors : T. Brückel (JCNS), S. Raymond (MDN), K. Schmalzl (JCNS).

PhD students outside MDN laboratory and supervised by MDN researchers

Laura Chaix

Couplage magnéto-électrique dynamique dans les composés multiferroïques : langasites de fer et manganites hexagonaux

ILL grant

04/2011 - 09/2014

Directors / Supervisors : V. Simonet (Institut Néel), S. de Brion(Institut Néel), E. Ressouche (MDN)

Shelby Turner

Comprendre le transport thermique: le défi de résoudre les durées de vie des phonons acoustiques

1/2 ILL grant +1/2 IDEX UGA

10 / 2018-…

Directors / Supervisors : H. Schober (ILL), S. Pailhes (ILM, Lyon), V. Giordanni (ILM, Lyon), M de Boissieu (SINAPS, Grenoble), F. Bourdarot (MDN)

Organisation of schools

HERCULES School 2016 – 2018

Since Autumn 2016, B. Grenier is director of studies of the European school HERCULES on Neutrons and Synchrotron Radiation for Science (previously, she was a member of the organising committee). This annual one-month school takes place in Grenoble with one week outside of Grenoble in a partner large scale facility. It provides training for students, postdoctoral and senior scientists from European and non-European universities and laboratories, in the field of **Neutron and Synchrotron Radiation** for condensed matter studies (Biology, Chemistry, Physics, Materials Science, Geosciences, Industrial applications). The school receives about 80 students of more than 20 different countries per year. The course includes more than 50 lectures, hands-on practicals and tutorials in small groups, visits of partner facilities, and a poster session. Each student follows a dedicated 'practicals and tutorials' programme, distributed over about 15 half-days, hosted in partner institutions (ILL, ESRF, CNRS, CEA, IBS in Grenoble, SOLEIL/LLB, Elettra/FERMI, PSI, or DESY/European XFEL outside Grenoble).

UGA Science Summer School 2014 – 2016

B. Grenier has been coordinating the scientific course "Introduction to large scale facilities (LSF): probing matter with neutron and synchrotron radiation" of the UGA Science Summer School in Summers 2014, 2015, and 2016 (this school no longer exists). This 6 weeks school was dedicated to high level bachelor students in physics, chemistry, or biology, from all over the world, willing to become researchers. It welcomed about 12 students per year in the LSF course, and about the same number for each of the two other scientific modules ("Introduction to Physical Computing" and "Introduction to Biochemistry"). The LSF module included lectures, tutorials, and lab work on crystallography, neutron scattering and synchrotron radiation, as well as a visit of ILL and ESRF.

HERCULES Specialised Course 2015 (HSC18)

B. Grenier co-organised a HERCULES Specialised Course in September 2015 on "Neutrons and Synchrotron Radiation for Magnetism". This one-week school welcomed 24 participants (mostly PhD students) in Grenoble and consisted in 3 days of lectures and 2 days of hands-on practicals at ILL and ESRF.

Interventions in schools

Lecture in the SISN Advanced School 2018 the "Magnetic structures from neutron diffraction", B. Grenier

Lecture in the 2018 School of the GDR Materiaux Etats ElecTroniques, Interactions et Couplages non-Conventionnels (MEETICC) "Structures cristallographiques et magnétiques par diffraction des neutrons : la puissance des symétries", 2018, B. Grenier

Lecture in the School ANF Microdiffraction Laue "Cristallographie", 2017, B. Grenier

Three-Axis Spectrometer practical in Hercules specialized course "Neutrons and Synchrotron for Magnetism", 2015, S. Raymond

Lecture in the annual HERCULES school "Refresher lecture on crystallography", 2014 – 2018, B. Grenier

Lectures, practicals, and tutorials in the annual UGA Science Summer School "Crystallography" and "Neutron scattering", 2014 – 2016, B. Grenier

Lecture in the School 2014 of the GDR Matériaux et Interactions en Compétition (MICO) "Matériaux "Cristallographie et techniques expérimentales associées", B. Grenier

TEAM 4: NANOSTRUCTURES AND SYNCHROTRON RADIATION (NRS)

1- Presentation of the NRS team

Introduction

The missions of the NRS laboratory are focused on the study of nanostructures with synchrotron radiation. First called *Interface and Synchrotron Radiation Laboratory*, it has been created at the early stages of the European Synchrotron Radiation Facility (ESRF). It contributes to the scientific and technological objectives defined by CEA and by the French community within the framework of the French X-ray synchrotron large scale facilities. These tasks require very specific synchrotron equipments and scientific skills to carry out high-level experiments on a broad range of subjects supporting both academic research and societally relevant applications (labelled A1-7 in the next chapters). NRS laboratory is mainly using ESRF beamlines located in Grenoble to perform its research, but also the French synchrotron Soleil, and other European and worldwide synchrotrons when their uses are technically justified. We operate three instruments on the BM32-IF beamline of the ESRF whose construction and development rely on the laboratory members. The laboratory is located on two sites at ESRF and at CEA.

Unit's workforce and means

The team is composed of 6.5 researchers (equiv. full time) and 1 engineer. Over the evaluation period, *Vincent Favre-Nicolin, an assistant professor at UGA belonged to the permanent staff of the laboratory, he has been on leave at ESRF since 2016. François Rieutord became the head of MEM, after being the head of NRS. He continues to contribute scientifically to several themes of the laboratory (A1, A4, A5, A6). Joël Eymery coming from IRIG/PHELIQS/NPSC took the head of the laboratory at the end of 2016 and Samuel Tardif was the last permanent person hired by the laboratory in Dec. 2016.* Marie-Ingrid Richard, coming from Aix-Marseille University will be hired by CEA to work at the NRS laboratory before the end of 2019 (the exact date is not known at this time).

Permanent staff (01/2019) & activity label (see below)		Non-permanent (01/2019)	
X. Biquard, CEA researcher, HDR	A5, A6	**2 Post-doctoral researchers**	
J. Eymery, CEA researcher, HDR, team leader	A1, A2, A6	Loïc Renversade	A6
P. Hecquet, CEA researcher	A7	Maciej Jankowwski	A3
G. Renaud, CEA researcher, HDR	A3, A6	**2 PhD students** (affiliated at NRS)	
C. Revenant (50 %), CEA researcher, HDR	A4	Gaetan Girard (located at ESRF)	A2
F. Rieutord (20%) CEA researcher, HDR	*A1, A2, A4-6*	Roberto Sant (located at ESRF)	A3
O. Robach (80%), CEA researcher	A6, A2, A5		
S. Tardif, CEA researcher	A1, A2, A4	**9 PhD students**	
O. Ulrich, CEA engineer	A6, A1-5	(NRS staff direction)	A1-A-6

Jean-Sébastien Micha, a CNRS engineer belongs to the permanent staff of the IRIG/DIESE/SYMMES laboratory with the UMR status with CNRS. He is involved in the management of the BM32-IF beamline and also in the operation of the µLaue instrument and code development. He is not a permanent staff of NRS, but he participates directly in our objectives (A4-A6).

2 PhD students have been included to the laboratory budget, and 11 PhDs have been managed by the NRS researchers during the period (8 are under way). Most of these PhDs are registered at the University Grenoble Alpes and principally at the *Ecole Doctorale de Physique* and *Ecole Doctorale d'Electronique, Automatique, Traitement du Signal*, when CEA/Leti is involved. One of the comments of the last HCERES evaluation of the laboratory was "*Attracting PhDs and post docs may be difficult on such instrumental teams, which may explain the small number of PhDs (2)*." This is still true but that is compensated by strong involvement in many PhDs of CEA laboratories. This has the advantage (i) to participate to our research axes (referred to A1-7 in the annex list), (ii) to establish strong scientific collaborations, (iii) to promote the use of synchrotron in other laboratories and for CEA missions, and (iv) to have external funding.

The laboratory budget is about 500 k€ per year, which breaks into non-permanent salaries, running costs, and investments (See details in the ANNEX 4). The funding, quite diverse as shown later, is coming from French government, ANR, CEA, Europe and industry.

Scientific policy

The previous recommendations of the HCERES committee pointed out the "*question to find topics where this complex characterization can bring an impact which should be as large as the tools are complex and expensive*". This point has really been a major concern and we developed indeed a *structured* scientific policy based on the following *two main axes*:

- ***E1 - Involvement at ESRF with** the operating of the French-CRG beamlines of the ESRF*, and more specifically the Interface BM32-IF beamline ("maille: Très Grands Instruments de Recherche"). The ***CNRS*** and the ***CEA*** have jointly constructed and operate the CRG-IF beamline at ESRF to propose powerful X-ray beams, state-of-the art techniques and analysis tools to French surface/interface researchers. One third of the beamtime is allocated in counterpart to the European scientific community. This beamline opened at the beginning of the ESRF, was the first CRG Beamline operational. BM32 is now composed of technical and scientific staff coming from ***CEA*** (NRS) and ***CNRS*** (INP/SYMMES UMR 5819 and Institut Néel). CRG-IF beamline is member of the 5 French beamlines located at the ESRF forming the F-CRG facility, which is coordinated by CNRS and CEA with operating structures: "*Structure d'exploitation*", "*conseil d'administration*", "*conseil scientifique*". Detailed reports on the beamtime allocation, users, domains… can be found in the following link: http://f-crg.fr. It allows us to optimize the financial resources and state our strategy with respect to other beamlines at ESRF and SOLEIL by specifying the skills to promote or to share... This coordination leads to a strong complementarity between the instruments and topics. Indeed, we develop mainly experiments using the high X-ray energy range provided by ESRF that is much above what is accessible at Soleil. A major task started in Dec. 2018 with the Extremely Brilliant Source project of the ESRF (see the five-year project).
- ***E2 - In-house thematic studies** with the fulfilment of the CEA missions in fundamental and applied research*, mainly driven by three programs of CEA (called "mailles"): (i) Fundamental Research in Physics and associated instruments, (ii) *Information and Communication Technologies* (ICT) and (iii) *New Technologies for Energy*. In this context, we participate also to the Nano Characterization Platform PFNC of CEA in the X-ray field.

  The laboratory carries out cutting edge research on focused domains based on our equipment and on the staff specific expertise. And in addition to these focused in-house research (detailed hereafter), the lab members have also a three-fold involvement with our collaborators: (*i*) to help in proposal writing, in identifying the *ad hoc* techniques to solve a scientific or technical question and in designing the most relevant experiment, (*ii*) to act as an interface between the CEA laboratory users and synchrotron facilities, (*iii*) to process the raw data and extract useful information. To perform these tasks, the laboratory has proactive roles to improve some instruments, to manage experiments and imagine new ones, and to participate to data analysis, but also to find resources and contracts that are mandatory to keep a leader position.

We got a second remark from the committee about our 5-year strategy saying "This is ambitious. However, if this allows opening the thematic and establish more collaboration in domains with real challenges and large impact, then it is worth doing this technological effort." This report will show in the following that the promised technical development of the beamline has been performed and also that it led to the definition of real scientific challenges with the establishment of French and international collaborations.

2- Presentation of the NRS team research ecosystem

E1 - Involvement at ESRF

The BM32 beamline is dedicated to study the physics and chemistry of surfaces, interfaces, thin films and nanostructures and the mechanical properties at the micron scale. It holds three experimental stations that operate either with polychromatic or monochromatic beam of ~100 µm to sub-µm sizes, 6 days/week except annual shutdowns (see Fig. NRS1). It is granted by the Large-Scale Facility program of CEA and CNRS.

Fig. NRS1: View of the three instruments of the BM32 beamline at ESRF.

The **GMT** station uses penetrating hard X-ray (30 keV) to investigate interfaces between dense materials (liquid/liquid or liquid/solid) by well-established techniques, among others: X-ray Reflectivity (XRR), Wide Angle (XRD/WAXS) and Small Angle Scattering (SAXS). Because of its very versatile design, the GMT instrument fits the needs of a wide community for "soft » and « hard » condensed matter with both fundamental and technology-related aspects, *e.g.* nanowires, wafer bonding, *in operando* Si nanoparticles swelling during Li-ion battery cycling charge (electrochemical cell developed by CEA.

The **INS2** station is based on the combination of an MBE/CVD/UHV reactor chamber and of an X-ray diffractometer allowing for the preparation of surfaces/interfaces and the growth of nano-films/particles and for their simultaneous studies, *in situ* and/or *operando*, using techniques such as Surface X-Ray Diffraction (SXRD) and Grazing Incidence X-ray Scattering/Diffraction at wide (GIXS/D) and Small (GISAXS) angles. A recent EQUIPEX funding (CRG/F) allowed to design and realize a completely new state of the art experimental station providing unprecedented capabilities for *in situ* experiments with a highly-equipped reactor chamber (large panel of CVD/MBE/gas sources) combined with ultra-high vacuum surface tools. It allows for the characterization/growth/modifications of a large variety of materials, with specific developments insuring the lowest possible background; hence the highest achievable signal-over-noise ratio and fast data acquisition. The addition of a gas injector allowed for the development of recent CEA research subjects using this station, especially the *in-situ* growth of 1D semiconductor nanowires and 2D materials such as graphene.

The **µLaue station**, is unique in Europe. Using a white (5-30 keV) X-ray nanobeam (~500 nm) with appropriate 2D detectors provides a 2D microstructural mapping using Laue microdiffraction. The recent addition of techniques providing structural information with high resolution in strain and depth resulted in an X-ray 3D microscope suited, *e.g.* for accurate metrology in microelectronic devices and to stress level control in heterogeneous materials for energy and metallurgy.

E2 - In-house thematic studies

Several long-term kinds of research are conducted by the laboratory personnel on a few selected topics (detailed in the next chapter):

- *A1: Physics of SOI materials:* for the past ten years, the laboratory has been conducting in-depth studies with local partners of the CEA DRT/Leti, particularly with groups involved in the development of these materials as part of a common CEA-SOITEC laboratory. These studies are related to the understanding of the physics underlying the technology processes (*i.e.* implantation, bonding, fracture) and make use of several laboratory experimental setups: mass spectrometer to quantify implanted species, infra-red and confocal microscopes to study bubbles and cavities, laser interferometry to estimate wafer separation during the fracture. The gap and sealing conditions between bonded wafers have been extensively measured by high-energy X-ray reflectivity on the BM32 beamline, fracture nucleation by full-field X-ray microscopy at ID01@ESRF and propagation by time-resolved topography at ID19@ESRF. This action is presently funded by the SOITEC company through the CEA-SOITEC laboratory and by the ANR Fraindy (Fracture Initiation and Dynamics, 2019-2022).
- *A2: Electronics & optics materials* strain and composition in Si transistors and nitride materials for light emission studied by nanobeams and new approaches (coherent and full-field diffraction). We have a strong scientific collaboration with the ESRF X-Ray NanoProbe (XNP) group (ID01, ID13, ID16 beamlines) and the development of analysis softwares. We participated to a Long-Term Project (LTP) with Aix-Marseille University, CNRS/SiMAP and University Paris Sud called *Quantitative diffraction on single nano-structures for mechanical, photonic and electronics applications* (2012-2015). This work benefits from strong relationships with CEA IRIG/PheliQs/NPSC laboratory for nanowire crystal growth, optics and structures, the CNRS/C2N for nanodevice realization and CEA IRIG/PheliQs/SiNaPS and DRT/Leti for state-of-the-art samples of transistors, emitters, detectors and technology access. We participated to the Lanef and GaNEX ANR Labex (recently extended). We get funding from CEA Bottom-Up programs LUMIX (2017-2020) and ANR funding: *Mechanics of Nano-Objects studied by in-situ X-ray diffraction*, MECANIX (2011-2015), *Nanowire based integrated platforms*, Platofils (2014-2018), Exploratory Carnot, *PiezoFlex* (2015-2017).

- *A3: 1D & 2D materials grown under the X-ray beam: structure and kinetics*. For a long time, the laboratory has been conducting *in situ* synchrotron X-ray scattering studies of nano-materials (3D, 2D and 1D) during their growth, using physical (MBE) or chemical (CVD) evaporation processes on BM32. The most recent studies were the growth of graphene on Iridium and the organization of metallic particles on top of the resulting moiré superstructure; the growth of silicon-germanium nanowires according to the VLS process, and, more recently, the growth of different 2D materials, mostly based on Te and S as chalcogen; this later being provided thanks to H_2S gas. In parallel to investigating the growth laws and processes, the team developed the related techniques (GIXD, SXRD, GISAXS) and their analyses, as well as the corresponding diffractometer. A completely new diffractometer and associated UHV-CVD setup (called INS2) has been developed thanks to an EquipEx funding (CRG/F 11-EQPX-0010, 2012-2019) and has shown its broad versatility with many experiments accepted by the committees. A fruitful collaboration with A. Dimoulas who has got a Chair of excellence in 2016 from the Lanef Labex and with other IRIG partners (SPINTEC, MEM/L_Sim, 2D factory program) and CNRS Institut Néel partners, allowed many successful studies of important Te-based dichalcogenides. The lab is also involved in a H2020 FET-Horizon project (Liquid Metal Catalyst, LMCAT, 2017-end 2020) aiming to study the ongoing chemical reactions and growth mechanisms on molten catalysts (*e.g.* graphene on liquid copper in a dedicated reactor designed by the consortium) with many European partners.
- *A4: Crystallographic phases and kinetics in battery & sol-gel materials* with IRIG partners (*i.e.* MEM/SGX and SYMMES/STEP), and with DRT/Liten. Granted by the project H2020-685716 (Silicon based materials and new processing technologies for improved lithium-ion batteries, Sintbat, 2016-2020).
- *A5: II-VI materials for high-performance IR detectors*. Micro-Laue and EXAFS studies granted by CEA internal transfers (between DRT/DOPT and MEM/NRS). Also, in collaboration with IRIG/PheliQs/NPSC and the Technical University of Lisbon.
- *A6: Instrumental developments*. The laboratory must continue its constant efforts to keep our equipments at the best level, for the beamline optics in order to accompany the ESRF upgrade, as well as detectors and instruments. We have also to facilitate the users work to perform some data analysis that become always heavier due to the generalization of fast 2D detectors with many pixels. This task is done in coordination with ESRF scientific policy and European synchrotron procedures. One important project corresponds to the Micro-Laue methodology developments. Granted by a French-German ANR-16-CE92-0024 project (X-ray Laue Microscopy to Understand Fatigue Damage, XMicroFatigue, 2017-end 2019) with the Max-Planck-Institut für Eisenforshung, Düsseldorf. This work contributes directly to all the users of this instrument. The CRG/F INS2 EquipeX project mentioned just above (A3) has also benefited from the high-level expertise and experience of the CRG/IF beamline staff as well as that of the SERAS laboratory of the CNRS-Grenoble for the 3D computer assisted conception.
- *A7: Simulation of model vicinal surfaces*. An activity is focused of the quantification and relationships between surface energies and stresses by using empirical potentials for silicon and metallic vicinal surfaces.

Main collaborators external to MEM

Industrial partners: SOITEC, SOFRADIR

Institutional partners: IRIG : PHELIQS/NPSC ; CNRS : Institut Néel, C2N ; Leti ; Liten ; Leiden University Netherlands ; NCSR Democritos Greece; Technical Univ of Lisbon Portugal

3- Research products and activities for the NRS team

Scientific track record

During this 5-year period, the NRS team has produced about 112 articles, with 24 papers in high-impact factor journals (IF>7) including 1 (IF>20). About 29 proceeding papers have been written. The laboratory staff participated also to the writing of 5 book chapters and to the editorial management of 3 journals (lists are given at the end of this part.

NRS Scientific topics:

A1. Physics of SOI materials

Permanent staff: F. Rieutord, S. Tardif, J. Eymery, PhD student: D. Massy

Solid/solid interfaces are ubiquitous in micro- or opto-electronics devices but are difficult to study using standard surface probes. Hard X-rays used under grazing incidences combine the benefits of a large penetration depth and interface sensitivity. These techniques have been developed on the BM32 beamline at ESRF taking full advantage of the ESRF source features. Wafer bonding interface studies were

performed in collaboration with DRT-LETI and the SOITEC company (see highlight H1). Fundamental issues associated to the contact of solid surfaces at nanometre scale were addressed, showing the influence of van der Waals attraction, surface roughness and material elasticity. Specific adhesion behaviours could also be evidenced[49]. In the important case of hydrophilic surface bonding, the role and reactions of interfacial water was studied. The transport kinetics of this confined fluid along the interface could also be studied thanks to the good time and size resolution achievable with these synchrotron X-ray beams[50]. The nanometre gaps obtained by bonding and their study using X-rays have been used subsequently by colleagues from CEA DEN (Marcoule) to investigate the effect of confinement on the reactivity of ionic solutions. Finally, opposite to bonding interfaces, crack interfaces and dynamic crack propagation in crystals was also investigated. Important results were obtained on the role of acoustic waves in the field of dynamic fracture of brittle materials.

A2. Electronics & optics materials: strain and composition

Permanent staff: J. Eymery, V. Favre-Nicolin, F. Rieutord, S. Tardif, O. Robach, O. Ulrich, J.-S. Micha (CNRS/SYMMES), PhD student: G. Girard

Nanobeams and coherent diffraction for (opto-)electronic nanostructures

Optimizing the performances of semi-conductor nanostructures for electronic and optoelectronic devices relies on a precise control of the strain, shape, defects with an accurate characterization of individual objects. Two main assets of the last generation X-ray synchrotron beams, *i.e.* nanofocusing and coherence have been used extensively to perform (i) *coherent diffraction imaging* (the sample is illuminated at a single position and is rotated either to gather a tomography dataset or around a Bragg diffraction peak) and (ii) *ptychography* (the sample is illuminated at a series of overlapping positions by the incident beam with diffraction recorded on a 2D detector either in forward scattering or at Bragg condition). Bragg diffraction analysis with the PyNX package, allows recovering both the strain in the object and the X-ray probe features. A ptychographic reconstruction of a 1.7 nm insertion of InAs in a GaAs nanowire is given as an example[51] in Fig. NRS2.

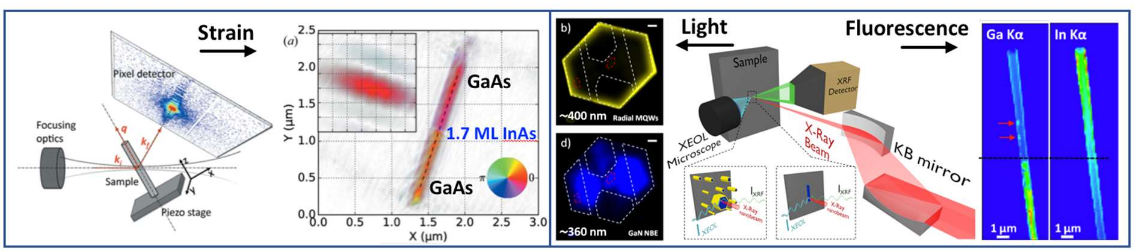


Fig. NRS2: (Left) Ptychographic reconstruction of the (113) reflection of a GaAs nanowire with an insertion of 1.7 ML of InAs. Brightness corresponds to the amplitude and colour to the phase (related to strain). The probe shape is shown in the inset.[51] (Right) Illustration of the XEOL technique (with energy images corresponding to the emission of a core-shell InGaN/GaN wire) combined with X-ray fluorescence mapping.[54]

High-energy focused beams (around 50 nm in diameter) are also used to investigate the relationships between light emission and composition by combining X-ray Excited Optical luminescence and X-ray fluorescence. We performed mappings recording at each points the light emission and the fluorescence spectra. In,Ga-composition of core-shell InGaN/GaN wire heterostructures and photovoltaic devices have been analysed on ID16B@ESRF to get light and composition variations in nanostructures as well as the influence of polarity inversion on doping concentration and emission features[52]. Such multispectral analyses are also extended to working devices (LEDs, transistors and µ-displays) taking advantages of high-energy X-ray. Two mainstream activities of CEA corresponding to state-of-the-art 10 nm thick Si transistor channels and 10 nm AlGaN layers for high power HEMT transistors have been measured on the ID01 at ESRF beamline with the partial illumination of a single object or by (coherent-) diffraction mapping [53]. The aim of these measurements - that can be also performed under electrical excitation - is to go

[49] F. Rieutord, C. Rauer, H. Moriceau, *Interfacial closure of contacting surfaces*, EPL, 107, 34003, 2014.
[50] M. Tedjini, F. Fournel, H. Moriceau, V. Larrey, D. Landru, O. Kononchuk, S. Tardif, F. Rieutord, *Interface water diffusion in silicon direct bonding*, Appl. Phys. Lett., 109, 111603, 2016.
[51] O. Mandula, M. Elzo Aizarna, J. Eymery, M. Burghammer, V. Favre-Nicolin, *PyNX.Ptycho: a computing library for X-ray coherent diffraction imaging of nanostructures*, J. Appl. Cryst., 49, 1842, 2016.
[52] D. Salomon, A. Messanvi, J. Eymery, G. Martínez-Criado, *Silane-Induced N-Polarity in Wires Probed by a Synchrotron Nanobeam*, Nano Letters, 17, 946, 2018.
[53] F. Mastropietro, J. Eymery, G. Carbone, S. Baudot, F. Andrieu, V. Favre-Nicolin, *Time-Dependent Relaxation of Strained Silicon-on-Insulator Lines Using a Partially Coherent X-Ray Nanobeam*, Phys. Rev. Lett., 111, 215502, 2013.

beyond the determination of average strain value, to get inhomogeneities inside patterns and statistics in-between single objects (see Fig. NRS3a). It generally involves new experimental and computational developments. X-ray diffraction methods operated on BM32 and BM02 have been applied to Light Emission Diodes based on multiple quantum wells and quantum dots. It participated significantly to the optimization of new MOVPE and MEB nitride-wire heterostructures by using X-ray diffraction[54] and have been complemented by XAFS techniques to refine the interplay between strain and short-range order in InGaN alloys. Nitride piezoelectric materials have been also studied by coherent diffraction imaging on ID01@ ESRF to revisit the atomic structure of inversion domain boundaries in GaN[55] that is one of the most important defects in nitride materials (see Fig. NRS3b).

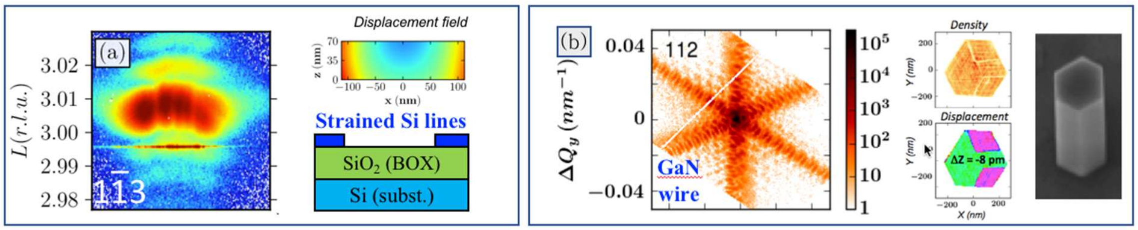


Fig. NRS3: (a) (11$\bar{3}$) diffraction from a strain Silicon on Insulator line (width 225 nm, height 70 nm) with a partially coherent x-ray nanobeam and related displacement field along depth (u_z).[53] (b) Coherent X-ray Bragg imaging (112 reflection) of a phase object corresponding to an Inversion Domain Boundary in GaN wires[55] with extracted density and displacement fields (ID01@ESRF beamline).

Laue microdiffraction mapping of orientation and deformation

The Laue microdiffraction (µLaue) instrument addresses the needs of the CEA to map crystal orientations and deformations at the sub-micron scale, *e.g.* in integrated circuits, micro structured components (but also nuclear fuel, piping steels or fuel cells). Suitable for any mono- or polycrystalline (possibly defective) material with grains larger than 100 nm, the µLaue provides a local measurement of the orientation of the crystal (Euler angles) and of the symmetries (Laue class) without any need to rotate the sample, as opposed to other diffraction imaging techniques (*e.g.* 3DXRD or CDI). Bending and twisting within the crystal can be obtained from the spatial derivatives of the orientation angles. Measured strain values can also be converted in terms of stress assuming the mechanical parameters of the material (see the example shown in Fig. NRS4). Large sample space and relatively quick acquisitions allow *in-situ* measurements (applied fields, temperature, etc.). Compared to high angular resolution electron back-scattering diffraction, µLaue offers a larger analysis depth and a better absolute precision on the strain, at the cost of a lower spatial resolution.

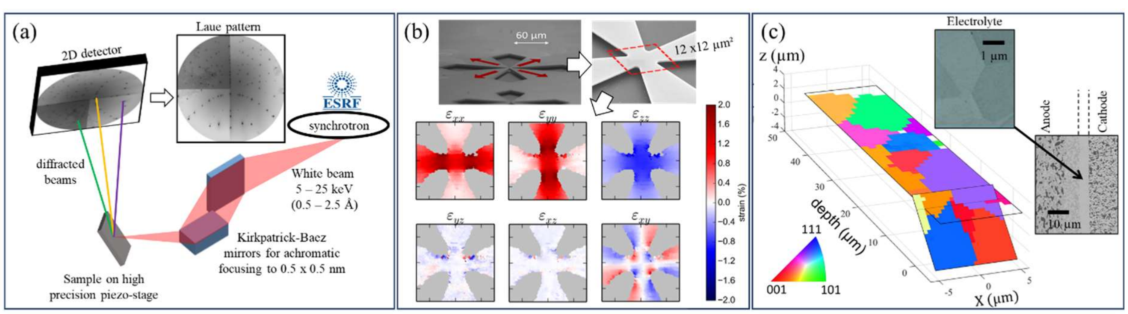


Fig. NRS4: (a) µLaue experimental setup, using the LaueTools software. (b) Full strain tensor components measured in microstructured, suspended monocrystalline Ge membranes (DRF/IRIG). (c) Differential aperture depth resolved grains orientation in Z.

A limitation of the µLaue is the insensitivity to hydrostatic changes since the energy of the diffracting photons is usually unknown. We have developed several technical improvements to circumvent this issue,

[54] C. Durand, JF. Carlin, C. Bougerol, B. Gayral, D. Salomon, JP. Barnes, J. Eymery, R. Butté, N. Grandjean, *Thin-Wall GaN/InAlN Multiple Quantum Well Tubes*, Nano Lett., 17, 3347, 2017.
[55] S. Labat *et al.*, [11 authors – J. Eymery], *Inversion Domain Boundaries in GaN Wires Revealed by Coherent Bragg Imaging*, ACS Nano, 9, 9210, 2015.

i.e. using the "rainbow filter" method or energy sensitive detectors.[56] For example, we were able to measure the full strain tensor in Ge micro-structures under tensile stress[57] and to evidence the non-linear relation between longitudinal strain and Raman spectral shift. Additionally, recent efforts pushed towards depth-resolution using the differential aperture technique (*i.e.* by scanning an absorbing wire over the surface to triangulate the origin of the Bragg reflections) and using tomography (see next paragraph).

3D strain mapping using µLaue topography

The need to resolve the strain distribution across interfaces and in actual devices has driven the development of depth-resolved µLaue, *e.g.* for the failure analysis in embedded interconnects (see Fig. NRS5). Using the tomography approach, *i.e.* by rotating the sample in the beam and recording the corresponding Laue patterns, a detailed 3D description of the grains (strain and orientation) in the probed volume can be obtained. It was successfully applied to evidence the strain fields in and around through-silicon via (TSV) made of polycrystalline cylinders of copper in silicon [58].

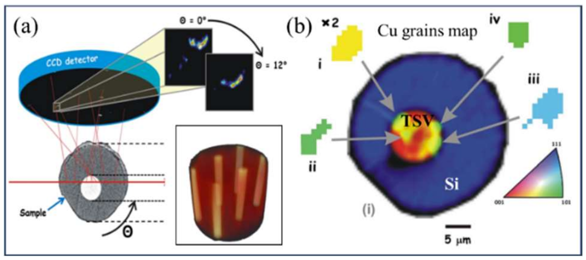


Fig. NRS5: (a) Tomo-Laue experiment, a Cu TSV sample is rotated in the beam while recording the Laue pattern, which allows the grain map reconstruction.
(b) Absorption nano-tomography reconstruction of six Cu TSV in Si to illustrate a typical sample (TSV diameter).

A3. 1D & 2D materials grown under the X-ray beam: structure and kinetics

Permanent staff: G. Renaud, O. Ulrich, PhD students: T. Zhou, B. Jean, R. Sant, Post-Docs: V. Cantelli, M. Jankowski, associated CNRS members: especially O. Geaymond

The capability to monitor structures during their growth under suitable clean atmosphere is one of the assets of large facilities. Optimizing the growth to achieve 1D & 2D materials of the highest structural quality and over large areas is a major challenge that has been tackled by the INS2 instrument along two main directions: (1) the *in-situ* growth of SiGe nanowires (NWs) with the dedicated instrumental development[59] of UHV-CVD using digermane and disilane and (2) the *in-situ* growth and structural studies of novel 2D materials: graphene and Transition Metal Dichalcogenides (TMDCs).

(1) 1D SiGe NWs have been the object of intense studies because of their high potential for many applications *e.g.* in micro-nano-optoelectronics. Their properties are directly linked to their shape, strains and composition, which in turn are determined by the growth. MBE and CVD were combined to grow SiGe NWs by the Vapor-Liquid-Solid (VLS) process. The growth of state-of-the-art pure Ge, Si as well as core-shell and axial GeSi heterostructures was achieved and they were analysed during their growth *in situ*, combining small and wide-angle X-ray scattering. The incubation time between the onsets of gas-exposure and NW growth has been studied as a function of size, temperature and flux and qualitatively interpreted. The strain and intermixing were analysed in core-shell NWs grown under different conditions by use of anomalous diffraction. Oriented MBE deposition of Ge and Au on one side of Si NWs was also performed to bend them *in situ*. A comparison of experimental and with theoretical bending profiles allowed deducing the surface and interface stresses during growth.

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[56] O. Robach, JS. Micha, O. Ulrich, O. Geaymond, O. Sicardy, J. Hartwig, F. Rieutord, *A tunable multicolour 'rainbow' filter for improved stress and dislocation density field mapping in polycrystals using X-ray Laue microdiffraction*, Acta Cryst., A69, 164, 2013.
[57] S. Tardif *et al.*, [17 authors – 8 CEA authors], *Lattice strain and tilt mapping in stressed Ge microstructures using X-ray Laue micro-diffraction and rainbow filtering*, J. Appl. Cryst., 49, 1402, 2016.
[58] D. Sanchez *et al.*, [11 authors – O. Ulrich, J.-S. Micha, O. Robach], *X-ray µ-Laue diffraction analysis of Cu through-silicon vias: A two-dimensional and three-dimensional study*, J. Appl. Phys., 116, 163509, 2014.
[59] V. Cantelli, O. Geaymond, O. Ulrich, T. Zhou, N. Blanc, G. Renaud, *The In-situ growth of Nanostructures on Surfaces (INS) endstation of the ESRF BM32 beamline: a combined UHV–CVD and MBE reactor for in situ X-ray scattering investigations of growing nanoparticles and semiconductor nanowires*, J. Sync. Rad., 22, 688, 2015.

(2) The discovery of stable 2D materials such as graphene (Gr), having exceptional mechanical, electronic and catalytic properties led us to develop their synthesis under ultra-clean conditions, with the objective to study and optimize their structural quality. Strains, that are known to strongly affect the properties of low-dimensional materials, were measured during the C_2H_2-based CVD growth of Gr on Ir(111) and during temperature variations. Large strains, above 2%, are present during growth,[60] as well as during annealing because of the substrate expansion that forces positive rather than the predicted negative Gr thermal expansion.[61] The atomic structure of the 10% misfit-induced moiré was also quantitatively determined by Surface X-Ray Diffraction.[62] Nanoparticles, either preformed or grown by atomic evaporation, were found to organize on this moiré.[63]

Topological Dirac and Weyl semimetals are a new state of matter that shows linear dispersions (Dirac cones) in all three dimensions in the reciprocal space. Weyl fermions predicted by quantum field theory have never been observed in free space, so their low energy « incarnations » in semimetals offers a unique opportunity to merge high energy elementary particle physics with condensed matter. Discovering and engineering topological semimetals from the family of 2D TMDC materials could open the way for exploitation of their unique topological properties by fabricating thin epitaxial films and devices on suitable crystalline substrates.

The growth and structure of different TMDCs on different substrates (AlN, InAs, graphene, Au, Pt) was explored, starting with $MoSe_2$, an indirect semiconductor in the bulk that transforms to a direct one when monolayer-thick[64]. Similar studies were done on $PtSe_2$, which transforms from semi metallic in the bulk to semiconductor for a monolayer. The complete structure was solved, as well as the relation with the substrate. An H_2S gas source was added to investigate the CVD growth of the TMDC prototype, MoS_2, on Au(111). Intercalation using Cs atoms allowed a complete decoupling of the MoS_2 layer from its substrate, paving the way for the realization of large, defect-free MoS_2 flakes. We also collaborated with NCSR DEMOKRITOS in Greece to investigate the growth and structure of 2D $HfTe_2$,[65] $ZrTe_2$,[66] $TiTe_2$ and $MoTe_2$[67] thin films by MBE. These materials are rotationally aligned with the InAs substrates, having the lowest in-plane mosaicity observed so far, and have a clear quasi van der Waals (vdW) gap with the substrate, indicating high quality vdW epitaxy. Imaging of electronic band structure by *in-situ* provides for the first-time compelling evidence that 1T-$HfTe_2$ and 1T-$ZrTe_2$ are Dirac semimetals. The 2D Dirac cones persist down to the ultimate 2D limit of a single layer indicating that $ZrTe_2$ could be regarded as an electronic analogue to graphene. Moreover, we made the first direct observation at room temperature of the orthorhombic (T_d), type-II Weyl semimetal phase in epitaxial $MoTe_2$. Its stability is a result of tensile strain from the substrate which stabilizes an elongated interlayer antibonding state characteristic of T_d-$MoTe_2$. In 1 ML 1T-$TiTe_2$ we obtained evidence for a charge density wave instability at room temperature.

The current state-of-the-art synthesis method of two-dimensional materials (2DMs) involves the dissociative adsorption of gas-phase precursors (CVD) on a solid catalyst. This process is slow by nature, inefficient, and environmentally unfriendly. Using liquid metal catalysts (LMCats) instead of solid ones bears the

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[60] N. Blanc, F. Jean, A.V. Krasheninnikov, G. Renaud, J. Coraux, *Strains Induced by Point Defects in Graphene on a Metal*, Phys. Rev. Lett. 111, 085501, 2013.

[61] F. Jean, T. Zhou, N. Blanc, R. Felici, J. Coraux, G. Renaud, *Effect of preparation on the commensurabilities and thermal expansion of graphene on Ir(111) between 10 and 1300 K*, Phys. Rev. B, 88, 165406, 2013 ; F. Jean, T. Zhou, N. Blanc, R. Felici, J. Coraux, G. Renaud, *Topography of the graphene/Ir(111) moiré studied by surface x-ray diffraction*, Phys. Rev. B, 91, 245424, 2015.

[62] D. Franz *et al.*, [11 authors - G. Renaud], *Atomic structure of Pt nanoclusters supported by graphene/Ir(111) and reversible transformation under CO exposure*, Phys. Rev. B, 93, 045426, 2016.

[63] L. Capiod, A. Bardotti, A. Tamion, O. Boisron, C. Albin, V. Dupuis, G. Renaud, P. Ohresser and F. Tournus, *Elaboration of Nanomagnet Arrays: Organization and Magnetic Properties of Mass-Selected FePt Nanoparticles Deposited on Epitaxially Grown Graphene on Ir(111)*, Phys. Rev. Lett. 122, 106802, 2019.

[64] M.T. Dau *et al.*, [20 authors - G. Renaud], *Beyond van der Waals Interaction: The Case of $MoSe_2$ Epitaxially Grown on Few-Layer Graphene*, ACS Nano, 12, 2319, 2018.

[65] S. Aminalragia-Giamini, J. Marquez-Velasco, P. Tsipas, D. Tsoutsou, G. Renaud, A Dimoulas, *Molecular beam epitaxy of thin $HfTe_2$ semimetal films*, 2D Mater., 4, 015001, 2017.

[66] P. Tsipas, D. Tsoutsou, S. Fragkos, R. Sant, C. Alvarez, H. Okuno, G. Renaud, R. Alcotte, T. Baron, A. Dimoulas, *Massless Dirac Fermions in $ZrTe_2$ Semimetal Grown on InAs(111) by van der Waals Epitaxy*, ACS Nano, 12, 1696, 2018.

[67] P. Tsipas, S. Fragkos, D. Tsoutsou, C. Alvarez, R. Sant, G. Renaud, H. Okuno, A. Dimoulas, *Direct Observation at Room Temperature of the Orthorhombic Weyl Semimetal Phase in Thin Epitaxial $MoTe_2$*, Adv. Funct. Mater., 1802084, 2018.

prospect of a continuous production of 2DMs with unprecedented quality and production speed. LMCat is a multinational collaborative project in the frame of FET-Open H2020 European calls; with the aim of in situ investigation of graphene growth on the surface of molten copper using synchrotron X-ray scattering, Raman spectroscopy, and optical microscopy techniques. The first results obtained by in situ optical microscopy using our newly developed LMCat reactor are very exciting. We show that the shape of the nucleated graphene flakes is a strong function of the CVD conditions, and the flakes tend to self-assemble into a hexagonal mosaic pattern on molten copper. Analysis of the real-time movies taken by in situ optical microscopy has provided us with unpreceded details about the dynamics of the flake growth and self-assembly. Combined with theoretical simulations, a theoretical model has been developed to explain the observed long-range interactions between the flakes, based on the capillary and electrostatic forces between them.

A4. Crystallographic phases and kinetics in battery & sol-gel materials

Permanent staff: S. Tardif, F. Rieutord, C. Revenant, O. Ulrich, J.-S. Micha (CNRS/SYMMES)

Recent years have seen numerous developments of experimental *in situ* and *operando* techniques (*i.e.* while the device is running) to probe electrochemical systems such as batteries. This is due to the strong, worldwide push in battery research, as well as by the necessity to probe metastable states during the out-of-equilibrium operation of such devices. For example, the exact lithiation and delithiation mechanism of graphite at all scales in the ubiquitous Li-ion battery are still debated. Another example is the question of sodiation mechanisms in novel layered oxide material in which self-discharge prevents the *ex situ* identification of the phases involved. To perform *operando* measurements, the experimental technique must be sensitive enough to access the relevant timescales (*i.e.* signal/noise ratio must be optimized). It must also be able to probe the region of interest in the battery. As a result, each technique requires a bespoke, optimized electrochemical cell, behaving as close as possible to a standard cell, yet allowing the measurements to be performed. Therefore, a series of *ad hoc* cells have been designed to probe (i) electrode interfaces using reflectivity, (ii) nanostructured electrode morphology using small-angle (x-rays and neutron) scattering, (iii) electrode crystal structure using wide-angle scattering, (iv) 3D distributions thereof using tomography, (v) electrode internal strain using diffraction and Raman spectroscopy.

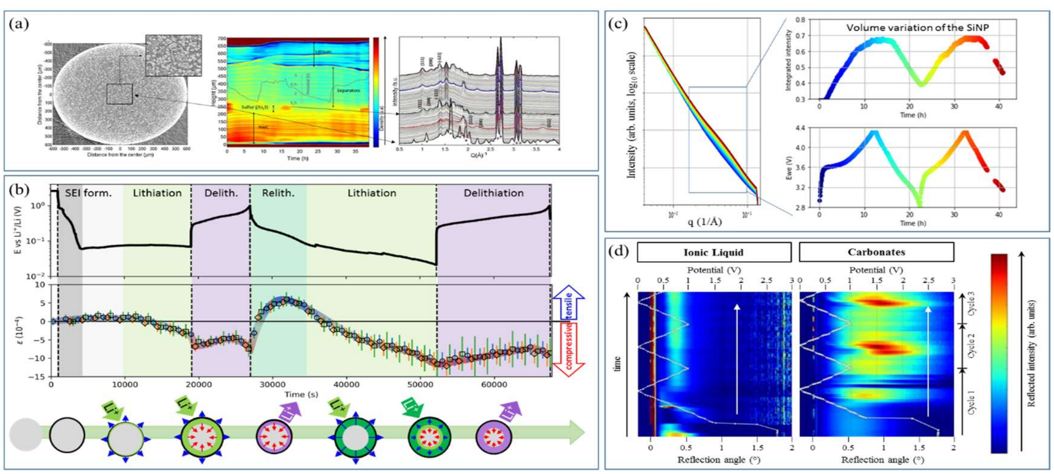


Fig. NRS6: Examples of operando measurements: (a) coupled measurement of X-Ray Diffraction and Tomography imaging of a Li/S system, (b) measurement of the internal strain in a Si nanoparticle electrode using high-resolution diffraction, (c) morphological changes in similar particles using SAXS, (d) SEI formation and evolution during cycling in different electrolytes using reflectivity.

Sol-gel and polymer structures

A wide range of experimental results were obtained using these electrochemical cells, some of which are illustrated in Fig. NRS6. The internal strain in the core of crystalline Si nanoparticles could be measured using both X-ray diffraction and Raman spectroscopy. For the first time, the effect of cumulative strain upon successive partial lithiation cycles were evidenced.[68] Furthermore, the combination of experimental techniques has proven very useful to achieve a better understanding of the mechanisms at play. An

[68] S. Tardif *et al.*, [12 authors – 12 CEA authors], *Operando Raman Spectroscopy and Synchrotron X-ray Diffraction of Lithiation/Delithiation in Silicon Nanoparticle Anode*, ACS Nano, 11, 11306, 2017.

interesting example is the simultaneous measurement of the wide- and small-angle scattering to measure the crystal structure and grain morphology, respectively, at the same time.

Nowadays, there is a tremendous development of solution-processed thin films due to the possibility of deposition on large surfaces, even flexible and at low cost. Sol-gel and molecular thin films are examples of solution-processed thin films. Sol-gel processed thin films were studied with the example of In-Ga-Zn-O. Such transparent conducting oxide plays a critical role in the fabrication of many current and emerging optoelectronic devices. Despite a massive use of sol-gel thin films, there has been relatively little effort directed toward understanding the fundamental physics of sol-gel thin films. In sol–gel processing, annealing at high temperatures is required to turn the gel film into an oxide compound through chemical reactions. Annealed IGZO films exhibit pores produced by the evaporation of residual organics. We have shown direct evidence of the nanocluster organization using STEM/EDX and GISAXS on CRG-F BM02@ESRF.[69] Nanoclusters are not arranged at random, but undergo self-organization closely related to the pore arrangement in films. In addition, the local structure of the nanoclusters was studied by X-ray absorption spectroscopy CRG-F BM30 and was linked to the electronic properties.[70] A concentration-driven instability model well describes the roughness and the porosity of the film and the structural data were used to derive fundamental quantities, like the diffusion coefficient of the solution and the oxide surface energy. The drying-shrinkage model illustrated for IGZO thin films is applicable to other porous thin films and solvents. With the knowledge of fundamental parameters, it is possible to rationalize the morphology of sol-gel thin films and ultimately to control the properties. We also study thin films elaborated from molecular solutions. These films offer numerous applications, such as innovative electrolytes for batteries or proton-exchange membrane fuel cells. The morphology of such deposits was studied by GISAXS to determine the organization of the molecules with respect to the substrate as a function of film thickness and substrate nature.

A5. II-VI materials for high-performance IR detectors

Permanent staff: X. Biquard, O. Ulrich, J.S. Micha (CNRS/SYMMES)

High-performance IR detector materials are developed at CEA/Leti/DOPT since more than 30 years. For military and space applications, they are made of a micrometer thick $Hg_{1-x}Cd_xTe$ detection layer deposited onto $Cd_{1-y}Zn_yTe$, with x being chosen as a function of the targeted IR domain and y for lattice match. HgCdTe is a brittle material with a low elastic limit and the resulting dislocations are detrimental for carrier collection. The final device performances are therefore directly linked to both HgCdTe crystalline quality and strain, mostly coming from the heteroepitaxial lattice mismatch. To assess any progress in the field of low residual strain on a micrometric sized pixel, we need a high-performance measurement tool combining sub-micrometric spatial resolution with extreme strain resolution, namely µLaue. Dedicated experimental and software developments made us increase strain resolution by an order of magnitude (see top right Fig. NRS7). The strain is found null inside substrate while HgCdTe displays a constant compressive strain (defect-free zone, coherent with a bi-axial hypothesis) that reaches zero again (dislocation zone) when arrived at the free surface of the layer. Once the HgCdTe layer has been epitaxially grown onto the CdZnTe, three major technological processes are sequentially applied to full-plate sample to obtain IR photodiodes (see bottom of Fig. NRS7): dry-etching to make pixel, capping layer deposition for protection and finally annealing to ensure electrical activation. We used the same full-sized HgCdTe/CdZnTe plate to process our three samples so that we may individually assess the influence of these technological processes[71]. This example demonstrated that the µLaue technique is perfectly suited to study high-performance IR photodiodes since it simultaneously provides the local rotation at excellent resolution and the local strain with an extreme spatial resolution the sub-micrometric scale.

[69] C. Revenant, M. Benwadih, Morphology of sol-gel porous In-Ga-Zn-O thin films as a function of annealing temperatures. *Thin Solid Films*, 616, 643, 2016.
[70] C. Revenant, M. Benwadih, O. Proux, Local structure around Zn and Ga in solution-processed In-Ga-Zn-O and implications for electronic properties. *Physica Status Solidi-Rapid Research Letters*, 9, 652, 2015.
[71] A. Tuaz, P. Ballet, X. Biquard, F. Rieutord, Micro-diffraction Investigation of Localized Strain in Mesa-etched HgCdTe Photodiodes, *Journal of Electronic Materials*,46, 5442, 2017.

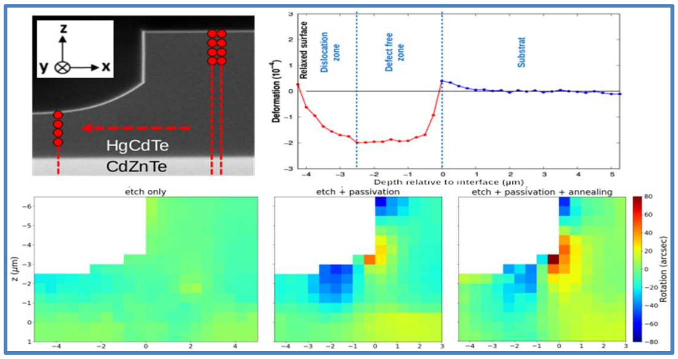


Fig. NRS7: *Top right: strain evolution inside layer and substrate with depth demonstrating a 10^{-5} resolution. Top Left: TEM image of an IR detector pixel with µlaue 2D raster scan shown as red dots (lower corner is rounded because the rotating 35° ion milling is partially shadowed by the deposited protection resin). Bottom: Comparison of the 2D repartition of the local (xz) orientation using the same ±80 arcsec rotation scale to assess effect of the three main technological processes of etching, passivation and annealing.*

A6. Instrumental developments

Permanent staff: O. Ulrich, X. Biquard, J. Eymery, G. Renaud, F. Rieutord, O. Robach, S. Tardif, J.-S. Micha (CNRS/SYMMES)

LaueTools - 3D analysis - energy scans: A Python open-source software package (LaueTools: http://sourceforge.net/projects/lauetools/) has been developed and is still under improvement to analyse the 2D Laue patterns. LaueTools is a multi-developers modular and documented package allowing digital image processing, Laue pattern recognition and structure refinement over a massive set of data (parallel computing). Graph theory algorithms, template matching and suited projective transforms are used to handle complex cases such as superimposition of many Laue pattern or peaks overlaps when standard analysis fails. Thanks to its flexibility, LaueTools supports the integration of two major extensions of the standard Laue techniques allowing the high-resolved decomposition of the X-ray scattered intensities with photons energy (by means of promising 2D energy dispersive detector) and depth inside the specimen. Strong efforts are made towards a better ergonomic software for users to fully handle and exploit their collected data.

Next-generation monochromators for X-rays: The unique combination of CEA expertise in synchrotron instrumentation (DRF/IRIG) and in direct bonding techniques (DRT/LETI) allows the exploration of a novel architecture for X-ray monochromators. The current monochromators design serves a double purpose: selecting the energy and focusing the beam in the sagittal plane by curving the second crystal. Energy selection dictates the crystal orientation, which is usually not mechanically optimal for focusing. The new architecture is proposed on F-CRG beamlines to decouple and simultaneously optimize these two requirements by using the direct bonding of a diffracting thin layer on a structured substrate. The former can be selected based on bandwidth and transmittance and the latter can be selected based on its mechanical properties. Initial finite elements modelling has shown promising results that will be tested on a prototype in 2020 (CEA PTC project BondMono, 2018-2019, WO2006077329A1 patent).

Other tools/instruments developed by CEA at synchrotrons: Numerous custom *operando* electrochemical cells have been designed for different types of experiments, *e.g.* observing interface effects using reflectivity or small angle scattering, or bulk effects using (micro-) diffraction (ID13) or tomography (ID31). Actual micro- and nano-devices also require careful preparation to be measurable, and smaller probes to study properties at the relevant nano-scale (ID01, ID16). New techniques are also explored, such as X-ray Raman Spectroscopy to probe chemical states in battery electrode materials (ID20). The development of dedicated analysis software is also often required to obtain meaningful data from complex and possibly indirect measurements (*e.g.* for coherent diffraction techniques).

X-ray excited optical luminescence (XEOL) is an efficient technique to measure the optical properties of materials. XEOL have been performed at the ID16B ESRF beamline, to study e.g. the exciton confinement and radiative time decays for blue light emission in InGaN/GaN multiple quantum core-shell wells deposited around GaN wires, and the effect of crystalline polarity on Si-dopant incorporation and wire emission[72,73]. XEOL has been also combined with XRF to correlate optical properties and In-composition, one of the most important parameters driving the emitted light colour. Experiments with nano-beams (~60 nm) provide high spatial resolution on nanostructures, but mappings are time consuming and probe

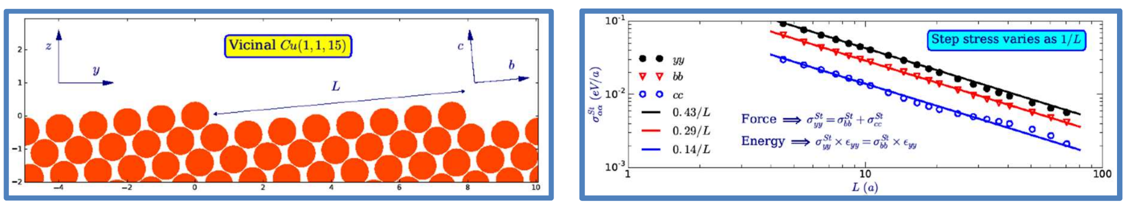
[72] G. Martinez-Criado, J. Segura-Ruiz, B. Alén, J. Eymery, A. Rogalev and A. Homs, *Exploring Single Semiconductor Nanowires with a Multimodal Hard X-ray Nanoprobe*, Advanced Materials, 26, 7873, 2014.
[73] D. Salomon, A. Messanvi, J. Eymery and G. Martinez-Criado, *Silane-Induced N-Polarity in Wires Probed by a Synchrotron Nanobeam*, Nano Letters, 17, 946, 2017.

a limited area. The CEA Transverse Nanoscience project LUMIX (2018-2019), coupled larger XEOL scanned areas by collecting the light with parabolic mirrors and structural information by using the µLaue setup of BM32. It provides strain and rotation information without sample alignment, and the sub-µm source fits well with scanning measurements with typical second counting time. This unique combination of technique has been successfully demonstrated on core-shell wires and phosphor materials to correlate strain and grain distribution to visible-light emission features. It is presently extended to µ-displays and detectors to perform statistical analysis of the device pixels.

A7. Simulation of model vicinal surfaces with empirical potentials

Permanent staff: P. Hecquet

This work is devoted to the understanding of the inter-step interactions on vicinal surfaces to go beyond the usual Marchenko-Parshin model. The step stresses σ_{ij} are calculated both in the *vicinal system* (x,y,z) where z is normal to the vicinal and in the *projected system* (x,b,c) where b is normal to the nominal terrace (see the left of the figure below), and also with two methods: the first, called the 'Force' method, consists to calculate directly the pressure forces that exist on surface, while the second, called the 'ΔE' method, gives the step stresses by deforming the vicinal in the direction parallel to the vicinal surface (ε_{yy} strain) and by calculating the energy excess proportional to this deformation.



In the 'Force' method, the step stress is $\sigma_{yy}=\sigma_{bb}+\sigma_{cc}$, all components vary as 1/L (see the right part of the figure) indicating a repulsion by the step stresses. The normal component zz of the step stress always vanishes over the period L. In the 'ΔE' method, $\sigma_{yy} = \sigma_{bb}$ and the first order deformation energy is $\sigma_{bb}.\varepsilon_{yy}$. Along the bb direction, the energy corresponds to a step stress located at the step multiplied by an interaction term due to neighbouring steps which varies as 1/L. The cc component is excluded in the calculation of the first order deformation energy because the corresponding step stress located at the step vanishes in the cc direction for large L, as for the normal zz direction. This has been confirmed by the calculations on two different vicinals Cu(11n)[74] and Cu(01M)[75]. This original result is also important to study the faceting phenomenon, as observed *e.g.* in Au(111) vicinals.

Key events

- It is particularly important for NRS to participate in training activities. V. Favre-Nicolin is the present Director of the European School HERCULES (http://hercules-school.eu/) established in 1991, which provides training for students, postdoctoral and senior scientists in the field of Neutron and Synchrotron Radiation for condensed matter studies. Several persons of the laboratory participated actively to lectures, seminars and practical on the beamline. Our formation effort is also extended to the promotion of the µLaue technique by organizing sessions to explain the data treatments with the LaueTools software at the USRF Users meeting and during specialized CNRS training for data treatment.

- The laboratory participated in the organization of some conferences. One of them is important in our field is *The Biennal conference on High-Resolution X-ray Diffraction and Imaging*, XTOP 12th that we organized in Villard de Lans, France (14-19th Sept. 2019). V. Favre-Nicolin was the chairman and several persons were involved in the organizing committee. Another illustration of our investment in the materials research field was the organization of the 17th European Workshop on Metal-Organic Vapour Phase Epitaxy in Grenoble, France (18-21st June 2017).

- We selected the three scientific highlights:

[74] P. Hecquet, *Surface energy and surface stress on vicinals Cu(1 1 n)*, *Surface Science*, 683, 7, 2019.
[75] P. Hecquet, *Surface energy and surface stress on vicinals by revisiting the Shuttleworth relation*. *Surface Science*, 670, 58, 2018.

*1: Bonding and fracture: study of solid/solid interfaces, t*o show our commitment with the SOITEC Company
*2: UHV-CVD growth of SiGe nanowires studied by X-ray scattering: growth, composition, strain & bending t*o demonstrate the interest to perform in situ measurement during a crystal growth
3: Synchrotron X-rays follow invisible moving crystalline defect to illustrate the performance of the µLaue technique during in situ fatigue mechanical cycles

H1: Bonding and fracture: study of solid/solid interfaces

For a number of years, we have been studying different key steps of the SmartCut™ technology, in collaboration with applied research (CEA-LETI) and industry (SOITEC). These studies lead to fundamental physics questions, related e.g. to the dynamics of confined fluid or of crack propagation in fragile rupture.

SmartCut™ technology

The SmartCut™ technology is the leading technology for Silicon on Insulator (SOI) substrate manufacturing. The SOI substrates offer key benefits compared to standard silicon substrates when increased performances are sought with a reduced energy consumption (e.g. mobile electronic devices). In the SmartCut™ process, a thin film of crystalline silicon is transferred to a substrate using successive bonding and fracture steps (see figure below).

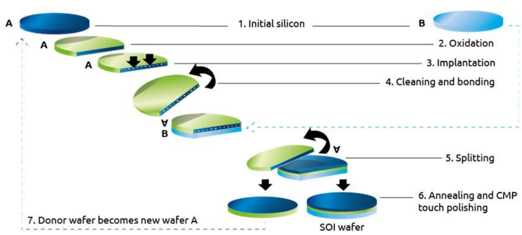


Fig. 1: Sketch of the SmartCut™ process (SOITEC)

The high level of optimization of these steps requires a fine control and deep knowledge of the processes.

Flow of water in nanometer gaps

When bonding two solids together (step 4 in fig), an interface is formed between them which exhibits some voids, due to the roughness of the two solids. When bonding two flat and clean silicon wafers, the gap has sub-nanometric width and is partially filled with water. This can be evidenced using hard X-ray reflection, as the beams are able to cross one full wafer thickness to reach the interface. Thanks to the high flux of the BM32 beamline, this interface structure can be measured at different points, as a function of time. As a consequence, we could study the dynamics of the water front propagation along the interface. The experiments show that despite the high confinement (<1nm), the water is still able to flow.[76]

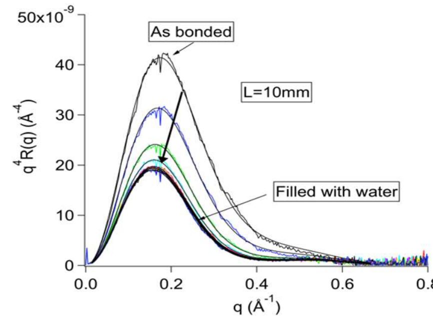


Fig. 2: Evolution with time of the interfacial reflectivity, at 10mm from the wafer edge due to water intake. Time lag between curves is 1.5h.

The dynamics of interfacial water could be modelled from standard wetting parameters in a peculiar porous medium equation.[77]

Lamb waves in crack dynamics

The splitting (step 5 on fig) is the other key step of SmartCut™. As observed experimentally, the crack propagation is not straight and suffers from a variety of instabilities that translate into some roughness after splitting. The high level of control in the process has allowed a detailed study of the mechanics of crack propagation. Using mostly optical and piezo electric observations (and occasionally fast X-ray imaging and reflectometry) we could model the acoustic wave emission by a propagating crack front. We could demonstrate that the instabilities of the fracture front originate in Lamb waves (mostly A0 waves) self-interacting with the crack front[78]. We could demonstrate some wave selection mechanisms. Finally, the whole distribution of crack patterns on the wafer could be directly modelled.

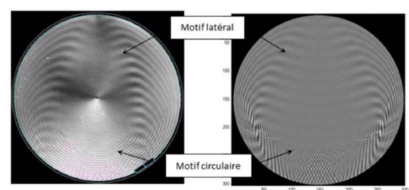


Fig. 3: Post-split pattern visible (by diffuse light scattering) on a 300 mm wafer (left: experiments, right: calculations).

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[76] Tedjini M. et al., Appl. Phys. Lett. 109 (2016) 111603.
[77] Rieutord F. et al., ECS Transactions 86 (2018) 39.
[78] Massy D. et al., Phys. Rev. Lett. 121 (2018)195501 – see also https://physicsworld.com/a/cracks-interact-with-sound-in-silicon-to-create-patterns-resembling-kelvin-wakes/ and https://physics.aps.org/articles/v11/113

H2: UHV-CVD growth of SiGe nanowires studied by X-ray scattering: growth, composition, strain & bending

MBE and CVD were combined in the INS chamber of the ESRF CRG BM32 beamline to grow Ge and Si nanowires (NWs). State-of-the-art pure Ge, Si as well as core-shell and axial GeSi nanowires were grown and analyzed *in situ*, during their growth, with X-rays scattering at small and wide angles. The incubation time between the exposure to gas and the onset of NW precipitation has been studied as a function of temperature, flux and size, and qualitatively interpreted. The strain and intermixing were analyzed in core-shell NWs grown under different conditions by use of Multiple Anomalous Diffraction under Grazing Incidence (GI-MAD). Oriented MBE deposition of Ge and Au on one side of Si NWs was used to bend them *in situ*, due to surface and interfacial (misfit) stresses. The bending profile was recovered by diffraction for different deposits. A comparison with an *ad hoc* mechanical theory allows deducing the surface and interface stress during growth.

In situ studies of Si and Ge nanowire growth

The observations performed during the growth of Si NWs are summarized in Fig. 1.

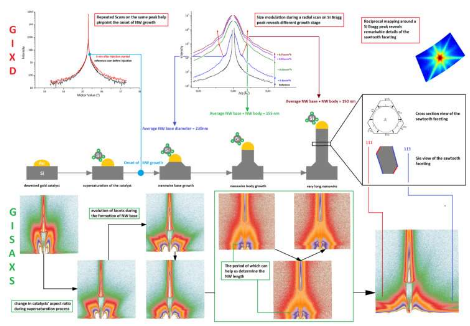

Fig. 1: GIXD & GISAXS measurements during the VLS growth of Si NWs on Si(111) substrate.

With GISAXS, we first observed a sudden change in the contact angle of the liquid catalyst as soon as the precursor gas was introduced. This is related to the increase of Si content in the liquid alloy during the supersaturation process, responsible for the so-called incubation time. NW size modulation was measured during growth using GIXD. The size decreased from 230 to 150 nm, revealing the initial growth of a larger NW base followed by the smaller NW body. The NW length was deduced from modulations in GISAXS measurements. The well-known sawtooth faceting was observed by both GISAXS and GIXD.

Growth of GeSi heterostructured NWs

We next moved to growing nanowire heterostructures *(Fig. 2)*.

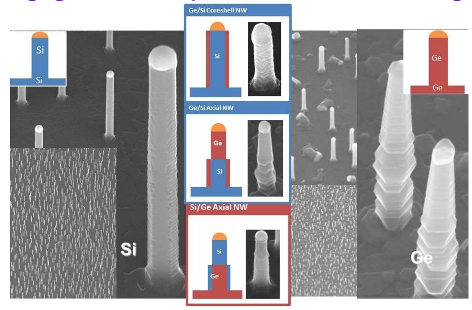

Fig. 2: SEM images of typical Si, Ge and SiGe heterostructured NWs grown in the INS setup.

GI-MAD were performed on growing Ge/Si core-shell NW, and the resulting strain/composition profiles deduced at different growth stages.

In situ bending of NWs

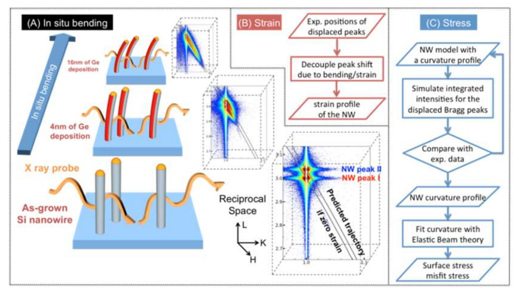

Fig. 3: (A) Schematics of the bending experiment and process flow chart of the analysis routine used for retrieving (B) strain and (C) stress information from the diffraction data.

The *in situ* bending of Si NWs was induced by Ge deposition on one side of them. The strain profile along the longitudinal direction of the NWs was obtained by analyzing the position shift of the displaced Bragg peaks whereas the total stress applied on the NWs was deduced by fitting the NW curvature, retrieved from integrated intensities of the displaced Bragg peaks, with a formula based on classic beam theory.

We were able to reconstruct the bended shape of the NWs (Fig. 4A), and to retrieve the related strain and stress (Fig. 4B) information. The reconstructed shape agrees well with *ex situ* Electron Microscopy observations. Our findings indicate that the bending induced by Ge deposition on Si NWs sidewall at 220°C is mainly driven by the misfit stress, which scales almost linearly with Ge film thickness. The result is distinctively different when conducted at RT: the bending is found to be driven by the surface stress, which evolves from tensile eventually to compressive in the later stage.

H3: Synchrotron X-rays follow invisible moving crytalline defect

A considerable part of current works on materials and applied science focusses on the development of setups and analysis tools for extracting the structural properties at the relevant length scale, which are often not available. Among attractive techniques, the 3D mapping by Laue microdiffraction allows to extend the vision capability of researchers on the key structural parameters and fundamental mechanisms that govern device reliability and efficiency.

Intense micrometre scale X-ray probe
The Laue microdiffraction setup still unique in Europe attracts a dozen of program committees selected experiments per year in addition to in-house research beamtime dedicated to instrumental developments. With a several hundreds of nanometers spatial resolution, it provides 2D orientation and strain mapping routinely. Due to its high strain sensitivity (10-4) the technique has become a reference and metrological tool for microelectronics materials (e.g. Ge for bandgap engineering[57], through-Si vias[79] or energy-related polycrystals in particular UO_2 [PhD M. Ibrahim 2015]). The great advantage of µLaue is to perform non- invasive/non-destructively direct measurements (without any assumptions) of microstructure or internal single objects structure in situ or operando. Valuable information on structural parameters can then be provided (/supplied) to understand and control the physical and chemical phenomena under investigation.

Non-destructive and accurate tomographic mode
Contrary to scanning electronic microscopy, X-ray µLaue microscope probe more in depth specimen leading to a more representative and relevant description of the structural properties including crystalline defects. However, in case of heterogeneous materials – such as single crystal with inner high strain or misorientation gradients – depth decomposition of X-ray scattered signal is required. Moreover, complete 3D resolved microstructure leads to better reliable and accurate data on real materials to be confronted to models. To fill these needs a 3D X-ray microscope was developed thanks to ANR-DFG XMicroFatigue project funding. As an extension of the standard µLaue technique, the Differential Aperture X-ray Microscopy scheme is applied allowing to obtain all X-ray scattering contributions originating from small slices along the beam beneath the surface. By 3D mapping of a bent reference thin Si wafer crystal, it has been demonstrated that accuracy on strain and orientation was conserved even in this tomographic mode.

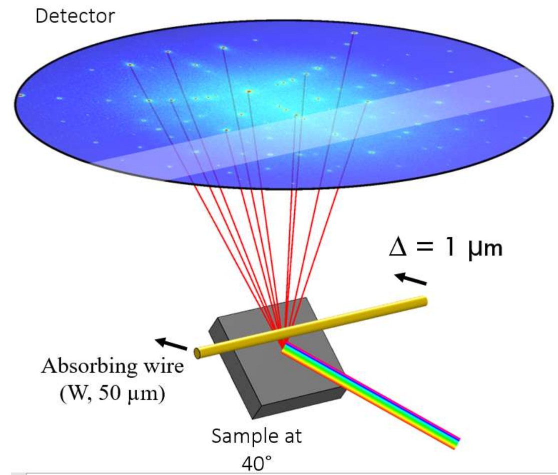

Fig. 1: Principles of Differential Aperture Microscopy. A 50µm in diameter absorbing wire is scanned close to the sample surface while acquiring X-ray Laue peaks on 2D detector. The localization of the X-ray scattering in sample is obtained by triangulation using digital difference of images taken at two calibrated contiguous wire positions intercepting the Laue peak direction (red line).

Understanding fundamentals of fatigue damage initiation
Both in structural and functional materials key-questions concerning the mechanical properties remain unanswered. Within the XMicroFatigue project, the combination of 3D µLaue and in situ low cycle fatigue loading is applied onto a fundamental and life limiting problem at the micron scale: the cyclic interaction of few dislocations with a grain boundary. Unprecedented insights into the structural evolution of damage in materials under deformation were recently obtained on micro pillar made of two grains. At each loading step, from 3D Maps complete microstructure – namely dislocation locations and orientations and stress-induced strain field- could be quantitatively measured. These data will be subsequently used for the calibration of Discrete Dislocation Dynamics simulations for modelling crystal plasticity onset.

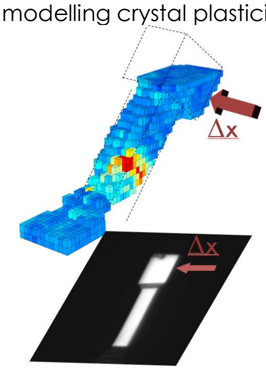

Fig. 2: (bottom) view of a bicrystal pillar after the first deformation step of a fatigue cycle. (top) Corresponding 3D map of Laue peak elongation related to the number of stored dislocations at the grain boundary (vertical plane perpendicular to applied deformation Δx at the middle of the pillar). Voxel size: 1x1x1µm³.

[79] M. Deluca M. et al., *Journal of Applied Physics*, 120 (2016)195104.

4- Organisation and life of the NRS team

Steering, life, organisation in the unit

We have few team meetings (3 per years), but small groups discuss every day at the two sites (ESRF and CEA). Technical meetings are structured with respect to the different actions of the laboratory (SOITEC, CRGs, Europe, ANR …).

Parity; scientific integrity; health and safety; sustainable development and environmental impacts; intellectual property and business intelligence.

The permanent staff is composed of 7 males and 2 females (soon 3) and we apply the general rule at CEA to promote parity and professional equality between men and women. We apply also all the safety, IP and business intelligence rules of the CEA, IRIG institute.

Five-year project and stragey of the NRS team

NRS SWOT analysis

Strengths	***Weaknesses***
Technical and scientific skills. State-of-the-art instruments (high energy X-ray, growth in UHV, µLaue…). Clear scientific objectives with many collaborations. Strong request for our beamtime. ESRF project dynamic. Strong organizational structures (F-CRG, CEA) and common objectives with CNRS. Constant evaluation for improvement.	Missing engineer-researcher position for the INS2 experiment and growth facility. Scientific field diversity may lead to dispersion and visibility decrease. Small ratio person/subject. Two sites (ESRF, CEA) leads to difficulties to share information and develop a laboratory life. People often involved in synchrotron experiments.
Opportunities	***Threats/Risks***
ESRF-EBS upgrade opens new opportunity for science and new scientific X-ray developments. Broad applications of our techniques serving several fields. Appropriate positions of our activities in the CEA, French & EU context for funding.	No open funding call for F-CRG to accompany the ESRF-EBS upgrade. Too many financing windows, difficulty to manage a scientific policy under these circumstances. Necessity to get contracts for all kind of research, even exploratory. Losing the balance between fundamental/applied research funding. Policy of France & CEA about very large-scale facilities.

With the ongoing upgrade of the ESRF machine, the ESRF-EBS (Extremely Brilliant Source) is scheduled to be completed in the middle of 2020 with the planned start of new and refurbished beamlines and the exploitation of novel X-ray detectors in 2022-2023. ESRF will become the first high-energy synchrotron worldwide to offer an increase of a factor 40 in the brilliance and ~100 in coherence of the X-rays delivered to insertion device users. It will open new fields of research for condensed matter and life sciences.

The laboratory must be an actor and take profit of this major improvement to accomplish our scientific policy.

E1-Involvement at ESRF

Due to the bending magnet source used by all the CRGs, the potential gain is much lower than the two order of magnitudes of the insertion devices. Our improvement is directly depending on the upgrade of the optical elements of the beamline (mirrors and monochromators) to take advantage of the high brilliance. Note that this parameter is fundamental for grazing incidence experiments and nanobeams measurements, *i.e.* most of the experiments carried on by the laboratory. To divide the counting time (for the same experiment) by a factor 3-4 seems to be an achievable and significant objective for the users. Another significant increase of the BM32 throughput can be also obtained by transferring the µLaue setup to a dedicated room (up to now, GMT and µLaue are sharing the same experimental hutch). A new installation should simplify all the setup alignment and considerably decrease the time and effort of the beamline personnel in-between different types of experiments. This option has been precisely studied and the costs have been evaluated by the operating structure of the F-CRG. It has been already proposed to an ANR commission, which evaluated very positively the project (ranked 2nd), but an ANR PIA3 funding call has not been open yet. This delay is already very penalizing with respect to the ESRF-EBS project and a lack of funding is a major threat for our laboratory to maintain our competitivity and attractivity with respect to other synchrotrons (*e.g.* DESY in Germany, Lund in Sweden). Therefore, we must continue efforts in this direction regardless of the acceptance of this request for funding to continue our strong and fruitful collaboration with the ESRF. The first actions correspond to hire a software specialist to participate to the implementation of the new BLISS command software (the older was SPEC) on the F-CRG beamlines (on a shared-cost basis), to refurbish part of our optics (polishing of mirrors) and to participate to new detector acquisition.

Another priority corresponds to the increase of the workforce dedicated to INS2. With only one CEA researcher contributing to the local contact and to our research program, the situation is quite delicate, and a CEA research engineer is needed to prepare the experiments, to make full use of the technical upgrade of INS2 and to continue to improve it. Note that this instrument combines the difficulties to reproduce good growth conditions in a non-dedicated setup, to find suited kinetics and to perform the X-ray measurements.

Another important task to tackle for the laboratory is also to improve data management and analysis. Indeed, with the use of new 2D detector with many pixels and sometimes with operando measurements, a large volume of data is generated, *e.g.* more than 10 To of diffraction pattern data for a week-long µLaue experiment. This is the famous *data deluge* that is also observed in many other fields of science. We will have to participate to the development of metadata treatment on large scale facilities (*e.g.* the Datahub/ICAT+ of the ESRF) and to follow the PaNdata rules (consortium of all photon and neutron sources in Europe dedicated to sharing good practices of data management). Apart from these data management evolution, we will have to provide some fast and efficient tools to users to perform in-line pre-treatment and first data analysis. As an example, some extensions of the Laue technique must be implemented in LaueTools towards energy resolution and in-depth sample analysis. Graph theory algorithms, template matching and suited projective transforms, are already used to handle complex cases such as superimposition of many Laue pattern or peaks overlaps when standard analysis fails. A collaboration with L_Sim and other partners could help us to take advantage of powerful advanced machine learning or compressed sensing methods, as well as to improve our efficiency in data treatment by using CEA computing infrastructures.

A second point in terms of data treatment is related to the opportunity to use the outstanding coherence quality provided by the ESRF source. As already illustrated in A2, coherence is especially important for the study of strain and defects in nanostructures, and implies a strong methodology development. We will have to continue to collaborate, and expand our collaborations with ESRF partners. The role of UGA could be strengthened in this field by opening a professor position in the Grenoble Large Scale Facilities (there is none at this time), to participate in X-ray and Neutron teaching, to coordinate the Hercules European School, and also to drive international collaborations.

E2-Evolution of in-house studies

Within the next five years, we can anticipate the development of new pixel energy-detectors that will add a multispectral analysis to the µLaue technique. It will be important to collaborate to the development of this new

type of equipment that will led to new opportunities in materials science, especially for the fast determination of the full strain tensor, spot analysis and composition mappings. This track is linked to the data deluge treatment and scientific cases will be chosen accordingly.

As already said in the unit's workforce description, Marie-Ingrid Richard, coming from Aix-Marseille University will be hired by CEA to work at the NRS laboratory before the end of 2019. She got a consolidator ERC grant (called *Carine*). One of our objectives will be to accompany her project, which fits well in the axes A2 and A3 of the laboratory. Marie-Ingrid Richard will develop new state-of-the-art *in situ* techniques based on coherent x-ray scattering and complementary chemical characterization to study in situ and *operando* the structural evolution of catalytic nanoparticles in realistic conditions during a chemical reaction. This work will use the unique capabilities of coherent diffraction Bragg imaging (CDI). Recent proof-of-concept experiments using Pt nanocrystals demonstrated the sensitivity and spatial resolution of CDI under liquid conditions. New dedicated instruments for CDI have just reached user operation on the ID01 beamline and will take profit from the ESRF-EBS upgrade. It is only now that this new imaging technique can be applied during a catalysis reaction and can probe the structural changes of individual nanocrystals. This project might shed light into most relevant unsolved issues (durability, activity…) that limit the efficiency of today's industrial processes and will open new horizons in catalysis research.

Equipment, platforms

The three equipments (INS2, GMT, µLaue) operated by the laboratory and the French-CRG at the BM32 beamline of the ESRF are already described in the main text.

Research products and activities of the NRS team

Articles

Scientific articles (112) – 23rd May 2019

The most 20% significant (22 articles, chronological order):

1. Elaboration of Nanomagnet Arrays: Organization and Magnetic Properties of Mass-Selected FePt Nanoparticles Deposited on Epitaxially Grown Graphene on Ir(111). P. Capiod, L. Bardotti, A. Tamion, O. Boisron, C. Albin, V. Dupuis, G. Renaud, P. Ohresser, F. Tournus. *Physical Review Letters*, 2019, 122 (10), ⟨10.1103/PhysRevLett.122.106802⟩
2. Room Temperature Commensurate Charge Density Wave in Epitaxial Strained TiTe 2 Multilayer Films. S. Fragkos, R. Sant, C. Alvarez, A. Bosak, P. Tsipas, D. Tsoutsou, H. Okuno, G. Renaud, A. Dimoulas. *Advanced Materials Interfaces*, 2019, pp.1801850. ⟨10.1002/admi.201801850⟩
3. Beyond van der Waals Interaction: The Case of MoSe$ 2$ Epitaxially Grown on Few-Layer Graphene. M. Tuan Dau, M. Gay, D. Di Felice, C. Vergnaud, A. Marty, C. Beigne, G. Renaud, O. Renault, P. Mallet, T. Le Quang, J.-Y. Veuillen, L. Huder, V. Renard, C. Chapelier, G. Zamborlini, M. Jugovac, V. Feyer, Y. Dappe, P. Pochet, M. Jamet. *ACS Nano*, 2018, 12, pp.2319 - 2331. ⟨10.1021/acsnano.7b07446⟩
4. Massless Dirac Fermions in ZrTe 2 Semimetal Grown on InAs(111) by van der Waals Epitaxy. P. Tsipas, D. Tsoutsou, S. Fragkos, R. Sant, C. Alvarez, H. Okuno, G. Renaud, R. Alcotte, T. Baron, A. Dimoulas. *ACS Nano*, 2018, 12 (2), pp.1696-1703. ⟨10.1021/acsnano.7b08350⟩
5. Crack Front Interaction with Self-Emitted Acoustic Waves. D. Massy, F. Mazen, D. Landru, N. Ben Mohamed, S. Tardif, A. Reinhardt, F. Madeira, O. Kononchuk, F. Rieutord. *Physical Review Letters*, 2018, 121 (19), pp.195501. ⟨10.1103/PhysRevLett.121.195501⟩
6. Direct Observation at Room Temperature of the Orthorhombic Weyl Semimetal Phase in Thin Epitaxial MoTe 2. P. Tsipas, S. Fragkos, D. Tsoutsou, C. Alvarez, R. Sant, H. Okuno, G. Renaud, A. Dimoulas. *Advanced Functional Materials*, 2018, 28 (33), pp.1802084. ⟨10.1002/adfm.201802084⟩
7. Crystallographic orientation of facets and planar defects in functional nanostructures elucidated by nano-focused coherent diffractive X-ray imaging. M.-I. Richard, S. Fernandez, J. Eymery, J. P. Hofmann, L. Gao, J. Carnis, S. Labat, V. Favre-Nicolin, E. Hensen, O. T. , T. Schülli, S. Leake. *Nanoscale*, 2018, 10 (10), pp.4833-4840. ⟨10.1039/C7NR07990G⟩
8. Operando Raman Spectroscopy and Synchrotron X-ray Diffraction of Lithiation/Delithiation in Silicon Nanoparticle Anodes. S. Tardif, E. Pavlenko, L. Quazuguel, M. Boniface, M. Maréchal, J.-S. Micha, L. Gonon, V. Mareau, G. Gebel, P. Bayle-Guillemaud, F. Rieutord, S. Lyonnard. *ACS Nano*, 2017, 11 (11), pp.11306-11316. ⟨10.1021/acsnano.7b05796⟩
9. Micro-diffraction Investigation of Localized Strain in Mesa-etched HgCdTe Photodiodes. A. Tuaz, P. Ballet, X. Biquard, F. Rieutord. *Journal of Electronic Materials*, 2017, 46 (9), pp.5442-5447. ⟨10.1007/s11664-017-5691-6⟩

10. Thin-Wall GaN/InAlN Multiple Quantum Well Tubes. C. Durand, J.-F. Carlin, C. Bougerol, B. Gayral, D. Salomon, J.-P. Barnes, J. Eymery, R. Butté, N. Grandjean. *Nano Letters*, 2017, 17 (6), pp.3347 - 3355. ⟨10.1021/acs.nanolett.6b04852⟩

11. Silane-Induced N-Polarity in Wires Probed by a Synchrotron Nanobeam. D. Salomon, A. Messanvi, J. Eymery, G. Martinez-Criado. *Nano Letters*, 2017, 17 (2), pp.946 - 952. ⟨10.1021/acs.nanolett.6b04291⟩

12. Flexible White Light Emitting Diodes Based on Nitride Nanowires and Nanophosphors. N. Guan, X. Dai, A. Messanvi, H. Zhang, J. Yan, E. Gautier, C. Bougerol, C. Durand, J. Eymery, M. Tchernycheva. *ACS photonics*, 2016, 3 (4), pp.597-603. ⟨10.1021/acsphotonics.5b00696⟩

13. KB scanning of X-ray beam for Laue microdiffraction on accelero-phobic samples: application to in situ mechanically loaded nanowires. C. Leclere, T. Cornelius, Z. Ren, O. Robach, J.-S. Micha, A. Davydok, O. Ulrich, G. Richter, O. T. . *Journal of Synchrotron Radiation*, 2016, 23 (6), pp.1395-1400

14. Interface water diffusion in silicon direct bonding. M. Tedjini, F. Fournel, H. Moriceau, V. Larrey, D. Landru, O. Kononchuk, S. Tardif, F. Rieutord. *Applied Physics Letters*, 2016, 109 (11), pp.111603. ⟨10.1063/1.4962464⟩

15. PyNX.Ptycho: a computing library for X-ray coherent diffraction imaging of nanostructures. O. Mandula, M. Elzo Aizarna, J. Eymery, M. Burghammer, V. Favre-Nicolin. *Journal of Applied Crystallography*, 2016, 49 (5), pp.1842-1848. ⟨10.1107/s1600576716012279⟩

16. Lattice strain and tilt mapping in stressed Ge microstructures using X-ray Laue micro-diffraction and rainbow filtering. S. Tardif, A. Gassenq, K. Guilloy, N. Pauc, G. Osvaldo Dias, J.-. Hartmann, J. Widiez, T. Zabel, E. Marin, H. Sigg, J. Faist, A. Chelnokov, V. Reboud, V. Calvo, J.-S. Micha, O. Robach, F. Rieutord. *Journal of Applied Crystallography*, 2016, 49 (5), pp.1402-1411. ⟨10.1107/s1600576716010347⟩

17. Inversion Domain Boundaries in GaN Wires Revealed by Coherent Bragg Imaging. S. Labat, M.-I. Richard, M. Dupraz, M. Gailhanou, G. Beutier, M. Verdier, F. Mastropietro, T. Cornelius, T. Schülli, J. Eymery, O. T. . *ACS Nano*, 2015, 9 (9), pp.9210 - 9216. ⟨10.1021/acsnano.5b03857⟩

18. On the reversibility of dislocation slip during small scale low cycle fatigue. C. Kirchlechner, P. J. Imrich, W. Liegl, J. Poernbacher, J.-S. Micha, O. Ulrich, C. Motz. *Acta Materialia*, 2015, 94, pp.69-77. ⟨10.1016/j.actamat.2015.04.029⟩

19. Tensile Strained Germanium Nanowires Measured by Photocurrent Spectroscopy and X-ray Microdiffraction. K. Guilloy, N. Pauc, A. Gassenq, P. Gentile, S. Tardif, F. Rieutord, V. Calvo. *Nano Letters*, 2015, 15 (4), pp.2429-2433. ⟨10.1021/nl5048219⟩

20. Self-organized nanoclusters in solution-processed mesoporous In–Ga–Zn–O thin films. C. Revenant, M. Benwadih, M. Maret. *Chemical Communications*, 2015, 51 (7), pp.1218-1221. ⟨10.1039/C4CC08521C⟩

21. Exploring Single Semiconductor Nanowires with a Multimodal Hard X-ray Nanoprobe. G. Martinez-Criado, J. Segura-Ruiz, B. Alen, A. Rogalev, J. Eymery, R. Tucoulou, A. Homs. *Advanced Materials*, 2014, 26 (46), pp.7873 - 7879. ⟨10.1002/adma.201304345⟩

22. Integrated Photonic Platform Based on InGaN/GaN Nanowire Emitters and Detectors. M. Tchernycheva, A. Messanvi, A. de Luna Bugallo, G. Jacopin, P. Lavenus, L. Rigutti, H. Zhang, Y. Halioua, H. Julien, J. Eymery, C. Durand. *Nano Letters*, 2014, 14 (6), pp.3515-3520. ⟨10.1021/nl501124s⟩

Review articles (1)

23. Germanium based photonic components toward a full silicon/germanium photonic platform. V. Reboud, A. Gassenq, J.M. Hartmann, J. Widiez, L. Virot, J. Aubin, K. Guilloy, S. Tardif, J.M. Fédéli, N. Pauc, A. Chelnokov, V. Calvo. *Progress in Crystal Growth and Characterization of Materials*, 2017, 63 (2), pp.1-24. ⟨10.1016/j.pcrysgrow.2017.04.004⟩

Other articles (professional journals, etc.) (89)

2019

24. Incorporation of Europium into GaN Nanowires by Ion Implantation. D. Nd. Faye, X. Biquard, E. Nogales, M. Felizardo, M. Peres, A. Redondo-Cubero, T. Auzelle, B. Daudin, L. Tizei, M. Kociak, P. Ruterana, W. Möller, B. Mendez, E. Alves, K. Lorentz. *Journal of Physical Chemistry C*, 2019, ⟨10.1021/acs.jpcc.8b12014⟩

25. Carbon Monoxide Oxidation Promoted by a Highly Active Strained PdO Layer at the Surface of Au 30 Pd 70 (110). M.-C. Saint-Lager, M.-A. Languille, F. Aires, A. Bailly, S. Garaudée, E. Ehret, O. Robach. *ACS Catalysis*, 2019, 9, pp.4448-4461. ⟨10.1021/acscatal.8b04190⟩

26. Epitaxial growth and structure of cobalt ferrite thin films with large inversion parameter on Ag(001). M. de Santis, A. Bailly, I. Coates, S. Grenier, O. Heckmann, K. Hricovini, Y. Joly, V. Langlais, A. Ramos, C. Richter, X. Torrelles, S. Garaudée, O. Geaymond, O. Ulrich. *Acta Crystallographica Section B, Structural Science, Crystal Engineering and Materials*, 2019, 75 (1), pp.8-17. ⟨10.1107/s2052520618016177⟩

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111. M -Plane GaN/InAlN Multiple Quantum Wells in Core–Shell Wire Structure for UV Emission. C. Durand, C. Bougerol, J.-F. Carlin, G. Rossbach, F. Godel, J. Eymery, P.-H. Jouneau, A. Mukhtarova, R. Butté, N. Grandjean. *ACS photonics*, 2014, 1 (1), pp.38-46. ⟨10.1021/ph400031x⟩

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Books

Management and coordination of scientific books / scientific book edition in English or another foreign language (1)

1. XTOP: high-resolution X-ray diffraction and imaging. V. Favre-Nicolin, J. Baruchel, H. Renevier, J. Eymery, A. Borbély. *Journal of Applied Crystallography*, 48 (3), pp.620-620, 2015

Book chapters (5)

2. Nitride Nanowires for Light Emitting Diodes. N. Guan, X. Dai, F. Julien, J. Eymery, C. Durand, M. Tchernycheva. *Light Emitting Diodes: Materials, Processes, Devices and Applications*, pp.425-484, 2019, 978-3319992105. ⟨10.1007/978319-99211-2_12⟩

3. Chapitre 4 : Analyse avancée des gradients d'orientation et des contraintes par microdiffraction Laue des rayons X. O. Robach, J.-S. Micha, O. Ulrich, B. Devincre, T. Hoc, G. Daveau, V. Consonni, J. Petit. *Rayons X et Matière 5, RX2013*, 2017

4. MOVPE growth and characterization of core-shell nanorod heterostructures for optoelectronics. J. Eymery, D. Le Si Dang. *Modeling, Characterization and Production of Nanomaterials*, pp.323-335, 2015, 9781782422280. ⟨10.1016/B978-1-78242-228-0.00012-0⟩

5. Metal-Organic Vapor Phase Epitaxy Growth of GaN Nanorods. J. Eymery. *Wide Band Gap Semiconductor Nanowires 1: Low-Dimensionality Effects and Growth*, Wiley-ISTE, pp.245, 2014, 978-1-848-21597-9

6. Laue Microdiffraction at the ESRF. O. Robach, C. Kirchlechner, J.-S. Micha, *O. Ulrich*, X. Biquard, O. Geaymond, O. Castelnau, M. Bornert, J. Petit, S. Berveiller, O. Sicardy, J. Villanova, F. Rieutord. *Strain and Dislocation Gradients from Diffraction*, Imperial College Press, pp.156-204, 2014

Edited theses (2014-2018)

2 PhDs have been directly managed by the laboratory at the Univ. Grenoble Alpes:

- Damien Massy, Étude dynamique de la fracture dans la technologie Smart-Cut, 11 déc. 2015, F. Rieutord director. A1.
- Tao Zhou, In Situ Synchrotron X-ray Scattering of SiGe NWs : Growth, Strain and Bending, 3. Nov. 2014, G. Renaud director. A3.

11 PhDs have been supervised by NRS researchers (8 are presently under way), see the list below:

- Markus Baum, Le rôle des propriétés de l'eau et des effets spécifiques des ions sur l'évolution du nanoconfinement de la silice, 9 Nov. 2018, F. Rieutord supervisor. Grant CEA/ICSM Marcoule. A6.
- Aurélien Lardeau, Dopage de couches de GaN sur substrat silicium par implantation ionique, 13 July 2018, J. Eymery director. Grant CEA/Leti/DTSI. A2.
- AymE. Tuaz, Investigations structurales haute-résolution de photodiodes infrarouges de nouvelle génération, 21 Dec. 2017. X. Biquard supervisor, F. Rieutord director. Grant CEA/Leti/DOPT. A5.
- Marwan Tedjini, Etude du collage direct Silicium Silicium, Gestion de l'eau à l'interface, 24 Oct. 2017, F. Rieutord director, Grant CEA/Leti/DTSI, A1.
- Amine El Kacimi, *Capteurs piézoélectriques souples à base de microfils de GaN en structure capacitive* 10 Nov. 2017, J. Eymery director. Grant CEA/leti/DSYS. A2.
- Elodie Bêche, Etude des collages directs hydrophiles mettant en jeu des couches diélectriques, 6 Oct. 2017, F. Rieutord director. Grant CEA/Leti/DTSI. A1.

- Jean-Baptiste Marijon, Caractérisation 3D de la microstructure et des déformations élastiques des polycristaux par microdiffractiodiffraction Laue, 11 Jul. 2017, O. Robach co-director. Grant Art Métier Paris Tech. A6.
- Agnès Messanvi, Composants photoniques à base de nanofils de nitrures d'éléments III : du fil unique aux assemblées,16 Dec. 2015, J. Eymery director. Grant GANEX shared with U. Paris Sud. A2.
- Anna Mukhtarova, InGaN/GaN Multiple Quantum Wells for photovoltaics, 6 March 2015, J. Eymery director. Grant CEA/IRIG/PHELIQS. A2.
- Fabien Jean, Growth and structure of graphene on metal and growth of organized nanostructures on top, 16 Jul 2015, G. Renaud director. Grant Ecole Doctorale de Physique Grenoble. A3.
- Caroline Rauer, Collage de silicium et d'oxyde de silicium : mécanismes mis en jeu, 9 Jul. 2014, F. Rieutord director, Grant CEA/Leti/DTSI, A1.

Production in conferences / congresses and research seminars

Articles published in conference proceedings / congress (29)

20% most significant (5)

1. (Invited) Water Transport Along Si/Si Direct Wafer Bonding Interfaces. F. Rieutord, S. Tardif, I. Nikitskiy, F. Fournel, M. Tedjini, V. Larrey, C. Bridoux, C. Morales, D. Landru, O. Kononchuk. *AIMES 2018*, Electrochemical Society ECS, Sep 2018, CANCUN, Mexico. pp.39-47, ⟨10.1149/08605.0039ecst⟩
2. InGaN/GaN nanowire flexible light emitting diodes and photodetectors. N. Guan, X. Dai, H. Zhang, L. Mancini, A. Kapoor, C. Bougerol, F. Julien, N. Cavassilas, M. Foldyna, C. Durand, J. Eymery, M. Tchernycheva. *2017 19th International Conference on Transparent Optical Networks (ICTON)*, Jul 2017, Girona, Spain. ⟨10.1109/ICTON.2017.8024808⟩
3. Cavity mode analysis of highly strained direct bandgap germanium micro-bridge cavities. F. Armand-Pilon, T. Zabel, E. Marin, C. Bonzon, S. Tardif, A. Gassenq, N. Pauc, V. Reboud, V. Calvo, J. Hartmann, J. Widiez, Alexei Chelnokov, J. Faist, H. Sigg. *2017 IEEE 14th International Conference on Group IV Photonics (GFP)*, Aug 2017, Berlin, France. pp.17-18, ⟨10.1109/GROUP4.2017.8082174⟩
4. Highly strained direct bandgap Germanium cavities for a monolithic laser on Si. T. Zabel, E. Marin, R. Geiger, C. Bozon, S. Tardif, K. Guilloy, A. Gassenq, J. Escalante, Y. Niquet, I. Duchemin, J. Rothman, N. Pauc, F. Rieutord, V. Reboud, V. Calvo, J. Hartmann, J. Widiez, A. Tchelnokov, J. Faist, H. Sigg. *2016 IEEE 13th International Conference on Group IV Photonics (GFP)*, Aug 2016, Shanghai, China. pp.40-41, ⟨10.1109/GROUP4.2016.7739082⟩
5. (Invited) Water Stress Corrosion in Bonded Structures. F. Fournel, C. Martin-Cocher, D. Radisson, V. Larrey, E. Beche, C. Morales, P.-A. Delean, F. Rieutord, H. Moriceau. *13th International Symposium on Semiconductor Wafer Bonding - Science, Technology and Applications as part of the 226th Meeting of the Electrochem Soc*, Oct 2014, Cancun, Mexico. pp.121-132

Other conferences (24)

6. Nanogenerators based on piezoelectric GaN nanowires grown by PA-MBE and MOCVD. L. Lu, N. Jamond, J. Eymery, E. Lefeuvre, L. Mancini, L. Larzeau, A. Madouri, O. Saket, N. Gogneau, F.H. Julien, M. Tchernycheva
2018 IEEE 18th International Conference on Nanotechnology (IEEE-NANO), Jul 2018, Cork, Ireland. pp.1-2, ⟨10.1109/NANO.2018.8626416⟩
7. Cavity mode analysis of highly strained direct bandgap germanium micro-bridge cavities. F. Armand-Pilon, T. Zabel, E. Marin, C. Bonzon, S. Tardif, A. Gassenq, N. Pauc, V. Reboud, V. Calvo, J. Hartmann, J. Widiez, Alexei Chelnokov, J. Faist, H. Sigg. *2017 IEEE 14th International Conference on Group IV Photonics (GFP)*, Aug 2017, Berlin, France. pp.17-18, ⟨10.1109/GROUP4.2017.8082174⟩
8. Flexible Light Emitting Diodes Based on Nitride Nanowires. N. Guan, X. Dai, A. Messanvi, H. Zhang, J. Yan, E. Gautier, C. Bougerol, M. Vallo, F. Julien, C. Durand, J. Eymery, M. Tchernycheva. *CLEO: Science and Innovations*, May 2017, San Jose, United States. pp.STh3I.1
9. High-quality and homogeneous 200-mm GeOI wafers processed for high strain induction in Ge. A. Gassenq, S. Tardif, K. Guilloy, N. Pauc, M. Bertrand, D. Rouchon, M. Hartmann, J. Widiez, J. Rothman, I. Duchemin, J. Faist, T. Zabel, H. Sigg, F. Rieutord, A. Chelnokov, V. Reboud, V. Calvo, Y.-M. Niquet. *SILICON PHOTONICS XII*, Jan 2017, SAN FRANCISCO, United States. ⟨10.1117/12.2251790⟩
10. Inductively coupled plasma etching of germanium tin for the fabrication of photonic components. L. Milord, J. Aubin, A. Gassenq, S. Tardif, K. Guilloy, N. Pauc, J. Rothman, A. Chelnokov, J. Hartmann, V. Calvo, V. Reboud. *SPIE OPTO*, Jan 2017, San Francisco, United States. pp.101080C, ⟨10.1117/12.2252280⟩
11. Adhesion Energy and Bonding Wave Velocity Measurements. V. Larrey, G. Mauguen, F. Fournel, D. Radisson, F. Rieutord, C. Morales, C. Bridoux, H. Moriceau. *PRIME 2016*, Oct 2016, Honolulu, United States. pp.145-152, ⟨10.1149/07509.0145ecst⟩

12. Impact of Water Edge Absorption on Silicon Oxide Direct Bonding Energy. F. Fournel, M. Tedjini, V. Larrey, F. Rieutord, C. Morales, C. Bridoux, H. Moriceau. *PRiME 2016*, Oct 2016, Honolulu, United States. pp.129-134, ⟨10.1149/07509.0129ecst⟩

13. Edge Water Penetration in Direct Bonding Interface. F. Rieutord, S. Tardif, D. Landru, O. Kononchuk, V. Larrey, H. Moriceau, M. Tedjini, F. Fournel. *PRiME 2016/230th ECS Meeting*, Electrochemical Society, Oct 2016, Honolulu, United States. pp.163-167, ⟨10.1149/07509.0163ecst⟩

14. Transfer of Ultra-Thin Semi-Conductor Films onto Flexible Substrates. P. Montméat, I. de Nigris Brandolisi, S. Tardif, T. Enot, G. Enyedi, R. Kachtouli, P. Besson, F. Rieutord, F. Fournel. *PRiME 2016/230th ECS Meeting*, Electrochemical Society, Oct 2016, Honolulu, United States. pp.247-252, ⟨10.1149/07509.0247ecst⟩

15. Fabrication of 200 mm Germanium-On-Insulator (GeOI): A step toward a Germanium photonic platform. V. Reboud, J. Widiez, J. Hartmann, E. Gomez, A. Gassenq, K. Guilloy, M. Bertrand, S. Tardif, J. Escalante, N. Pauc, D. Fowler, E. Bellet Amalric, D. Rouchon, I. Duchemin, M. Niquet, F. Rieutord, R. Geiger, T. Zabel, E. Marin, H. Sigg, J. Faist, A. Chelnokov, V. Calvo. *2016 IEEE 13th International Conference on Group IV Photonics (GFP)*, Aug 2016, Shanghai, China. pp.140-142, ⟨10.1109/GROUP4.2016.7739074⟩

16. Non-linear bandgap strain dependence in highly strained germanium using strain redistribution in 200 mm GeOI wafers for laser applications. A. Gassenq, K. Guilloy, S. Tardif, J. Escalante, Y. Niquet, I. Duchemin, J. Hartmann, D. Rouchon, J. Widiez, J. Rothman, T. Zabel, E. Marin, H. Sigg, J. Faist, N. Pauc, F. Rieutord, A. Chelnokov, V. Reboud, V. Calvo. *2016 IEEE 13th International Conference on Group IV Photonics (GFP)*, Aug 2016, Shanghai, China. pp.104-105, ⟨10.1109/GROUP4.2016.7739054⟩

17. Nonlinear direct band gap and Raman shift strain dependence in germanium under high uniaxial stress. K. Guilloy, A. Gassenq, N. Pauc, S. Tardif, F. Rieutord, J.-M. Escalante, I. Duchemin, Y.-M. Niquet, P. Gentile, G. Osvaldo Dias, D. Rouchon, J. Widiez, J.-M. Hartmann, A. Chelnokov, R. Geiger, T. Zabel, H. Sigg, J. Faist, V. Reboud, V. Calvo. *E-MRS spring meeting*, May 2016, Lille, France

18. Nonlinear strain dependences in highly strained germanium micromembranes for on-chip light source applications. K. Guilloy, A. Gassenq, N. Pauc, J.-M. Escalante, I. Duchemin, Y.-M. Niquet, S. Tardif, F. Rieutord, P. Gentile, G. Osvaldo Dias, D. Rouchon, J. Widiez, J.-M. Hartmann, D. Fowler, Al. Chelnokov, R. Geiger, T. Zabel, H. Sigg, J. Faist, V. Reboud, V. Calvo. *SPIE Photonics Europe*, Apr 2016, Bruxelles, Belgium. ⟨10.1117/12.2227497⟩

19. Ultra-high amplified strain on 200 mm optical Germanium-On-Insulator (GeOI) substrates: towards CMOS compatible Ge lasers. V. Reboud, A. Gassenq, K. Guilloy, G. Osvaldo Dias, J. Escalante, S. Tardif, N. Pauc, J. Hartmann, J. Widiez, E. Gomez, E. Bellet Amalric, D. Fowler, D. Rouchon, I. Duchemin, Y. Niquet, F. Rieutord, J. Faist, R. Geiger, T. Zabel, E. Marin, H. Sigg, A. Chelnokov, V. Calvo. *SPIE OPTO*, Feb 2016, San Francisco, United States. pp.97520F, ⟨10.1117/12.2212597⟩

20. Simulation par irradiation aux ions des dégâts d'irradiation dans le combustible nucléaire usé en situation de stockage géologique : Apport des études par DRX. H. Palancher, É. Castelier, M. Ibrahim, A. Boulle, A. Richard, P. Goudeau, M. Bornert, S. Caré, R. Belin, F. Rieutord, J.-S. Micha, N. Blanc, N. Boudet, A. Ambard. *Colloque Rayons X et Matière*, Dec 2015, Grenoble, France

21. Optical spectroscopy and X-ray microdiffraction study of highly strained Ge nanostructures. K. Guilloy, N. Pauc, A. Gassenq, P. Gentile, S. Tardif, F. Rieutord, V. Calvo, J.-M. Escalante, I. Duchemin, Y.-M. Niquet, G. Osvaldo Dias, D. Rouchon, J. Widiez, J.-M. Hartmann, A. Chelnokov, V. Reboud, R. Geiger, T. Zabel, H. Sigg, J. Faist. *12th International Conference on Group IV Photonics*, Aug 2015, Vancouver, Canada

22. Photocurrent spectroscopy and X-ray microdiffraction study of highly strained germanium nanostructures. K. Guilloy, N. Pauc, A. Gassenq, P. Gentile, S. Tardif, F. Rieutord, J. Escalante, I. Duchemin, Y. Niquet, V. Calvo, G. Osvaldo Dias, D. Rouchon, J. Widiez, J. Hartmann, D. Fowler, A. Chelnokov, V. Reboud, R. Geiger, T. Zabel, H. Sigg, J. Faist. *2015 IEEE 12th International Conference on Group IV Photonics (GFP)*, Aug 2015, Vancouver, Canada. pp.173-174, ⟨10.1109/Group4.2015.7305919⟩

23. Distributed Bragg reflectors integration in highly strained Ge micro-bridges on 200 mm GeOI substrates for laser applications. A. Gassenq, S. Tardif, K. Guilloy, N. Pauc, J. Escalante, I. Duchemin, Y-M. Niquet, F. Rieutord, V. Calvo, G. Osvaldo Dias, D. Rouchon, J. Widiez, J. Hartmann, D. Fowler, A. Chelnokov, V. Reboud, R. Geiger, T. Zabel, H. Sigg, J. Faist. *2015 IEEE 12th International Conference on Group IV Photonics (GFP)*, Aug 2015, Vancouver, Canada. pp.51-52, ⟨10.1109/Group4.2015.7305999⟩

24. Design rules to control the tensile strain in Ge μ-membranes fabricated from GeOI substrates for photonics applications. G. Dias, D. Rouchon, J. Widiez, J. Hartmann, D. Fowler, A. Chelnokov, V. Reboud, A. Gassenq, S. Tardif, K. Guiloy, N. Pauc, J. Escalante, I. Duchemin, Y-M. Niquet, F. Rieutord, V. Calvo, J. Faist, R. Geiger, T. Zabel, H. Sigg. *2015 IEEE 12th International Conference on Group IV Photonics (GFP)*, Aug 2015, Vancouver, Canada. pp.133-134, ⟨10.1109/Group4.2015.7305988⟩

25. Non-linear model of electronic band structure to highly tensile-strained Germanium. J. Escalante, A. Gassenq, S. Tardif, K. Guiloy, N. Pauc, F. Rieutord, V. Calvo, G. Osvaldo Dias, D. Rouchon, J. Widiez, J. Hartmann, D. Fowler, A. Chelnokov, V. Reboud, I. Duchemin, Y.-M. Niquet. *2015 IEEE 12th International Conference on Group IV Photonics (GFP)*, Aug 2015, Vancouver, Canada. pp.77-78, ⟨10.1109/Group4.2015.7305955⟩

26. Tensile strained germanium nanowires measured by photocurrent spectroscopy and X-ray microdiffraction. K. Guilloy, N. Pauc, A. Gassenq, P. Gentile, S. Tardif, F. Rieutord, V. Calvo. *E-MRS spring meeting*, May 2015, Lille, France

27. Fracture Dynamics during the Silicon Layer Transfer of the Smart Cut Process. D. Massy, F. Mazen, J. Ragani, F. Madeira, D. Landru, F. Rieutord. *13th International Symposium on Semiconductor Wafer Bonding - Science, Technology and Applications as part of the 226th Meeting of the Electrochem Soc*, ECS, Oct 2014, Cancun, Mexico. pp.13-20, ⟨10.1149/06405.0013ecst⟩

28. Direct Bonding Mechanism of ALD-Al2O3 Thin Films. E. Beche, F. Fournel, V. Larrey, F. Rieutord, C. Morales, A.-M. Charvet, F. Madeira, G. Audoit, J.-M. Fabbri. *13th International Symposium on Semiconductor Wafer Bonding - Science, Technology and Applications as part of the 226th Meeting of the Electrochem Soc*, ECS, Oct 2014, Cancun, Mexico. pp.57-65, ⟨10.1149/06405.0057ecst⟩

29. Trapping of implanted hydrogen for ultrathin layer transfer. F. Mazen, F. Gonzatti, F. Madeira, S. Reboh, C. Deguet, F. Lallement, D. Landru, F. Rieutord, A. Royal. *20TH International Conference On Ion Implantation Technology (IIT 2014)*, Jun 2014, Portland, United States. pp.1-4, ⟨10.1109/iit.2014.6940054⟩

Instruments and methodology

Prototypes

The EquipEx funding (CRG/F 11-EQPX-0010, 2012-2019) leads to the installation of the new INS2 instrument on the BM32@ESRF beamline. The installation phase is completed (2012-2016), and the operating phase is under way.

Instruments

The three equipments (INS2, GMT, µLaue) operated by the laboratory and the French-CRG at the BM32 beamline of the ESRF are already described in the main text.

Editorial activities

Participation in editorial committees (3)

1. G. Renaud, co-editor *Journal of Applied Crystallography* (IUCr), 2010-2019.
2. V. Favre-Nicolin, co-editor *Journal of Synchrotron Radiation* (IUCr), 2015-present.
3. J. Eymery, associate editor *Progress in Crystal Growth and Characterization of Materials* (Elsevier), 2016-present.

Reviewing activities

The laboratory staff is involved in many reviewing procedures and evaluation committees.
Reviewing of articles (~80) in many Journals.
Grant evaluation (~29) in many institutions ANR, Europe…

Reviewing of research institutes (7):

J. Eymery: SOLEIL, member PCR3 committee, 2016-2018
ESRF, president Applied Physics and Engineering committee, 2014
ESRF, Peak overload member, Applied Physics and Engineering committee, 2015
European Science Foundation, 2013-present
F. Rieutord: ESRF, Peak overload member, Applied Physics and Engineering committee, 2018
O. Robach: ESRF, CRG Spline, member of the committee, 2014-2017

Participation in institutional committees and juries (1):

F. Rieutord HCERES committee member, Laboratoire de Physique des Solides, Orsay (LPS) evaluation.

Academic research grants

European (ERC, H2020, etc.) and international (NSF, JSPS, NIH, World Bank, FAO, etc.) grants – partnership

PRC7 FET-OPEN LMCat (2017-2020),

National public grants (ANR, PHRC, FUI, INCA, etc.) – coordination

ANR Fraindy 2019-2022

National public grants (ANR, PHRC, FUI, INCA, etc.) – partnership

ANR Platofil (2014-2018), ANR MECANIX (2011-2015), ANR MicroStress (2012-2015), ANR NMGEN (2011-2015)

PIA (labex, equipex etc.) grants – coordination

Equipex CRG/F, 2012-2019

Grants from foundations and charities (ARC, FMR, FRM, etc.) – coordination

Chair of excellence at LABEX LANEF 2016 for Athanasios Dimoulas, Research Director at NCSR DEMOKRITOS in Athens

Visiting senior scientists and post-doc

Post-docs (6) including foreign post-docs (3)

Samuel Tardif (2015-2016) Eurotalents, Ivan Nikitskiy (2018-2019) Eurotalents and CEA PTC, Martin Vallo (2016-2017) AND Platofil at CEA/IRIG/NPSC, Frédéric Boudaa (2014-2016) Equipex CRG/F, Maciej Jankowski (2017-2019) Eu. LMCAT, Loïc Renversade (2018-2019) ANR int. XMicroFatigue.

Visiting scientists (1)

Athanasios Dimoulas, Chair of excellence at LABEX LANEF 2016

Scientific recognition

Prizes and/or distinctions

1. 2016 EARTO INNOVATION awards for CEA (DRT/DOPT and DRF/INAC) in the Impact Expected category for the development of the wireLED™ technology, a new generation of 3D LEDs with drastically reduced production costs and higher performance. http://www.earto.eu/earto-events/earto-innovation-awards/earto-innovation-awards-2016.html

2. 2016 Physics Price of the science magazine « La Recherche » in collaboration with CNRS for the article : *Flexible Light Emitting Diodes Based on Vertical Nitride Nanowires.* X. Dai, A. Messanvi, H. Zhang, C. Durand, J. Eymery, C. Bougerol, F. H. Julien and M. Tchernycheva. *Nano Letters* ***15*** *(2015) 6958-6964*. DOI: 10.1021/acs.nanolett.5b02900

3. Best presentation award at the E-MRS Fall meeting 2015, Damien Massy. The paper was about Damien's PhD work, on Acoustic Emission from Crack Propagation in the Smart Cut™ Technology.

IUF members

V. Favre-Nicolin, junior member (2012-2016)

Organisations of meetings and symposia (participation to programme committee are not cited)

- Biennal conference on High-Resolution X-ray Diffraction and Imaging, XTOP 12th (V. Favre-Nicolin chair, J. Eymery local committee), 14-19th Sept. 2019, Villard de Lans, France.

- European Workshop on Metal Organic Vapour Phase Epitaxy, EWMOVPE 17 (J. Eymery, chair), 18-21 June 2017, Grenoble, France.

- European Conference on Surface Science, ECOSS-32 (G. Renaud, local committee) 28 Aug. - 2 Sept. 2016, Grenoble, France.

- *Journées de la Matière Condensée* SFP (J. Eymery, local committee) 27-31 Aug. 2018 Grenoble, France.

- Journées Nationales des Nanofils Semiconducteurs (J. Eymery, local committee) 13-15 Nov. 2019, Grenoble, France.

- Symposium on Surface Science 2015, 3S'15 (G. Renaud, local committee)

22-28 mars 2015, Les Arcs 1800, France.

- Joint ESRF-ILL-CEA workshop on batteries (S. Tardif, local committee) 6-7 Nov. 2017, Grenoble, France.

Invitations to meetings and symposia

- 11th International Conference on Nitride Semiconductors ICNS11 (J. Eymery, Beijing, China, Sept., 2015)

- International Reliability and Stress-Related Phenomena in Novel and Emerging Electronics Systems (S. Tardif, Singapour, Jan. 30, 2018)

- MRS Spring Meeting (S. Tardif, Phoenix, USA, April 5th)

- International workshop GISAXS 2016 (C. Brizard, Hamburg, Germany, Nov. 2016)

- XI colloque Rayons-X et Matière (J. Eymery, Grenoble, France, Dec. 2016)

- ECS Wafer Bonding (F. Rieutord, Hawaïï, USA, Nov. 2016)

- ECS High Purity Silicon (F. Rieutord, Hawaïï, USA, Nov. 2016)

- ECS Wafer Bonding (F. Rieutord, Cancun, Mexico, Nov. 2018)

- Materials for Advanced Metallization (F. Rieutord, Bruxelle, Belgium, 2016)

- Gettering and Defect Engineering in Semiconductor Technology (F. Rieutord, Berlin, Germany, Oct. 2019).

- Cristallographie, une clé de la connaissance – Crystallography, a key to knowledge

(V. Favre-Nicolin, UNESCO Paris, France, Janv. 22 2018)

Interaction of the NRS team with the non-academic world, impacts on economy, society, culture or health

Patents

Invention disclosures (6) – filed in the 2014-2018 years

Accepted patents (6)

- *Method for direct bonding of substrate including thinning of the edges of at least one of the two substrates,* F. Fourne, S. Morales, H. Moriceau, F. Rieutord (US 2018/0158719)

- *Dispositif optoélectronique à éléments semiconducteurs tridimensionnels et son procédé de fabrication*. *A. Dussaigne, J. Eymery, D. Salomon, C. Durand, P. Gilet (FR 15 56744, WO/2017/00958)*

- *Procédé d'orientation d'objets allongés disposés en surface d'un substrat*. *J. Eymery, A. El Kacimi, O. Dellea, E. Pauliac-Vaujour (FR* 15 58717, US 2017/0080457)

- *Procédé d'homogénéisation de la hauteur d'une pluralité de fils et dispositif utilisant de tels fils*. *J. Eymery, A. El Kacimi (FR* 17178042.2)

- Method of fabricating a transistor channel structure with uniaxial strain. S. Reboh, L. Grenouillet, F. Milesi, Y. Morand, F. Rieutord (US 2016/9935019)

- Method for transferring a useful layer D. Landru, O. Kononchuk, N. Ben Mohamed, D. Massy, F. Mazen, F. Rieutord (US 2015/9589830).

Involvement of the NRS team in training through research

Scientific productions (articles, books, etc.) from theses

Scientific productions (articles, books, etc.) from theses

About 3 articles per PhD in average.

Training

Habilitated (HDR) scientists (5)

X. Biquard, J. Eymery, G. Renaud, C. Revenant, F. Rieutord

HDR obtained during the period

X. Biquard, *Recherche Fondamentale via Rayons X synchrotron sur matériaux technologiques clefs,* Oct. 23rd 2018, Univ. Grenoble Alpes.

PhD students (2+11)

Defended PhDs

2+11 (see list above)

Mean PhD duration

38 months in average (on 13 PhDs)

TEAM 5: MAGNETIC RESONANCE (RM)

1- Presentation of the RM Team

Introduction

Since the last report, the major organizational change in January 2016 was the split of the RM team into two parts, on the one hand the nuclear magnetic resonance (NMR) group and on the other hand the electron paramagnetic resonance (EPR) group complemented by a senior expert in the DFT computation of spectroscopic observables. The EPR spectroscopists and the DFT expert remained in an expanded chemistry laboratory (SyMMES) while the NMR spectroscopists and several characterization and modeling physics laboratories formed the newly created MEM service dedicated to the exploration and modeling of materials. That important reorganization of the INAC institute was decided by Dr Yves Samson, the previous Head of the INAC, and not triggered by any specific recommendations of the previous HCERES visit. Concerning the NMR group, the previous synergy with the EPR spectroscopists and the DFT expert was partly compensated by the arrival of two physical chemists, Armel Guillermo (CNRS) and P. H. Fries (CEA) from the SyMMES laboratory. A. Guillermo helped us to maintain the PFG NMR activity during the period 2017- March 2019 as a volunteer scientific adviser though he retired in 2016. P. H. Fries is an expert in theoretical methods, liquid solutions and relaxometry techniques. To sum it up, since 2015, three colleagues have left the team and one has joined it. The number of permanent researchers has decreased from 9 to 7. The team is localized on two sites of the CEA campus which are of about 1.5 km apart and poorly served by motor vehicles. The DNP group (G. De Paëpe, S. Hediger, D. Lee) has been installed on MINATEC campus while the practitioners of general and other specialized NMR techniques (M. Bardet, P. A. Bayle, P.H. Fries and M. Gromova) do their research in the C5 building where are also localized the majority of the users of the NMR facility. The liquid/solid NMR facility is composed of one 400 MHz for liquid NMR, on 500 MHz (liquid, solid and HRMAS NMR) and a 200 MHz solid state NMR dedicated to operando and high-power PFG NMR. P.A. Bayle is full time in charge of the NMR facility. The two relaxometers managed by P. H. Fries are also in the C5 building. Concerning the equipment, the DNP group acquired a new DNP system and the second ultra-low-field relaxometer is under construction. Both instruments were acquired thanks to Europeans funds. Currently, we are trying to obtain funds to renew part of the NMR facility.

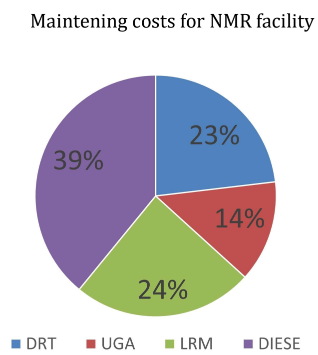
Unit's workforce and means

Permanent staff (01/2019)	Non-permanent
Michel Bardet* , CEA researcher, team leader Pierre Alain Bayle, CEA technician Gaël De Paëpe, CEA researcher P. Henry Fries**, CEA researcher Sabine Hediger, CNRS researcher Marina Gromova, Maître de Conférence at UGA Daniel Lee, CEA researcher	Post-doctoral researchers (DNP goup) C. Fernandez de Alba (2013-2015), S. Paul (2014-2015), F. Mentink-Vigier (2014-2016), S. Chaudhari (2016-2017), I. Marin-Montésinos (2015-2017), A. Smith (2017-), D. Gauto (2018-) PhD students (DNP goup) N. Duong (2012-2015), K. Märker (2014-2017), A. Kumar (2017-), S. Badoni (2017-), N. Olejnik-Fehér (2017-), R. Harrabi (2018-)

**Academic Guest at EPF from 15/10/2017 to 31/09/2018 ** Staff representative on the works council and on the health, safety and working conditions committee of the Grenoble research center, Member of the CEA scientific council (> 50 % of his time over the period))*

The main sources of funding are the following

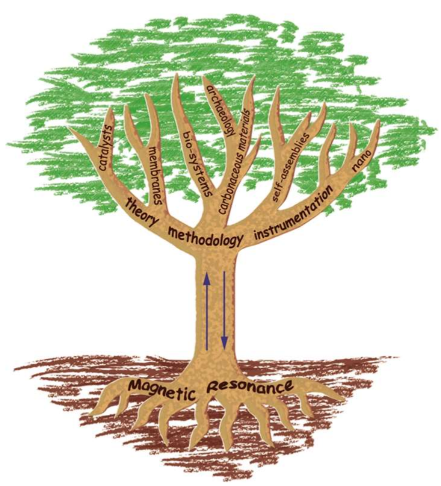
ANR – 2012 – Coord. – "CRYOMAS4DNP" – 417 k€ (consortium); ANR – 2016 – PRC – "TransPepNMR" – 168 k€



H2020-ERC – 2016 – Coord – "ULTMASDNP" – 2 M€; €; H2020 Research and Innovation Action – "IDentIFY" – 6.6 M€ (consortium); H2020-IEF – 2018 – Fellowship – "BOLDNMR" – 178 k€ , H2020-COFUND – 2017 – Partner – NaMeS – SINTBAT-TEESMAT, 1 ANR (2012-2016), 1 thèse phare CEA (2014-2017), 1 postdoc ARCANE (2012-2014), 1 postdoc ARCANE (2015-2017), 1 AGIR-PEPS (2015-2017), Eurotalent (2017-2018), 1 ANR (2017-2021), 1 ERC consolidator (2016-2021), 1 Marie Curie Fellowship (2018-2020), 1 thèse IDEX CDP Glyco@Alps (2018-2021); 1 thèse phare CEA (2017-2020), 1 CEA DRF Impulsion (2016-2017); industrial patnerships : Bruker, STELAR,

Note that for their access to the NMR facility all the users even from RM contribute to the maintenance costs. In 2018 the total contributions was 61 K€ for the three spectrometers, with the distribution given in the graph opposite.

Scientific policy



The goals of RM is to develop and apply a large range of NMR approaches to answer fundamental or applied problematics addressed by colleagues either in its near scientific environment or through international and international networks. The strategy of the group is to handle scientific projects whose durations range from a few months to several years. The choice of these projects is either motivated by the skills of the laboratory members, supported by the ANR and European projects, or induced by internal demands and CEA missions. RM has also in charge to maintain a NMR facility (spectrometer servicing, user training, analyses or advanced study achievement) dedicated to chemists and physico-chemists working mainly on CEA campus. The NMR equipment of RM is composed of two DNP solid-state NMR spectrometers dedicated to the NMR DNP group, two relaxometers for the relaxometry activity and three liquid/HRMAS/solid spectrometers dedicated to the NMR facility. One technician is in charge of the NMR facility which welcomes each year about 150 users from IRIG/DIESE and DRT/LITEN. The users are trained to be autonomous as far as possible. In some sparse cases exterior analyses are realized by RM staff. There is no recurrent budget dedicated to maintain the RM spectrometers. DNP and relaxometers spectrometers are mainly maintained through their own projects and those of the NMR facility by the users themselves including RM group.

Note that RM is localized on two geographic places on CEA campus: DNP group (3 permanents) and its DNP spectrometers on the recent MINATEC campus and the remaining part of RM (4 permanents) in the C5 building in which are also localized the majority of the users of the NMR facility. Note that G. De Paëpe is the group leader of DNP team, P. H. Fries is group leader of the relaxometry activity, since the retirement of A. Guillermo the PFG NMR activity has been partitially taken into account and further developped by M. Gromova, M. Bardet is in charge of NMR for energy and P. A Bayle is responsible for the NMR facility. In the context of CEA-Grenoble, RM team is one of the "competence centres" of Minatec Nanochaacterization Centre (PFNC) created in 2006.

2- Presentation of the RM team research ecosystem

Due to RM integration in the local, national and international contexts the following question can be addressed

What has the RM done over the last five years?

The following achievements can be answered:

- Methodology and instrument developments for DNP solid-state NMR, *operando* NMR and PFG NMR
- Designing new polarizing agents for DNP
- Developing sustainable helium spinning DNP at ultra-low temperature
- Methodology and instrument developments of Fast Field Cycling -NMR relaxometry with biomedical applications

- Understanding surface chemistry of nanocrystals: impact of ligand on stability, shape and size of nanocrystals
- Focusing on interfaces of hybrid materials: from functional polymers to biomaterials
- Structural studies and aging behaviour of materials for electrical/ hydrogen storages (batteries and fuel cells
- Track the changes and their dynamics of electrolytes and SEI during battery cycling and aging
- Structure determination of organic molecular assemblies without isotopic labeling: from small molecules to protein amyloid aggregates
- Locating and solving the structure of protein-ligand binding site: from in vitro developments to in cellulo applications towards the conception of new inhibitors
- Studies of archaeological and cultural materials using high-resolution solid-state NMR.

These results were obtained thanks to different collaborations, partnerships and networking:

CERMAV (UGA-CNRS), DCM (UGA-CNRS), Irig/SyMMES (UGA-CEA-CNRS), Irig/LCBM (UGA-CEA-CNRS), Irig/SBT (CEA-UGA), Irig/IBS (UGA-CEA-CNRS), Irig/PCV (UGA-CEA-CNRS), Institut Néel (UGA-CNRS), UPMC (LMCP), ICMG Montpellier, Warsaw University of Technology / Polish Academy of Sciences, Massachusetts Institute of Technology, Weizmann Institute of Technology, LNCMI (CNRS-UGA), NHMFL Tallahassee, PAGORA (INPG-UGA, LETI (CEA-DRT), LITEN (CEA-DRT), ARC Nucleart (CEA-G) Carnegie Mellon University, Warsaw Polytechnic, Ecole Polythechnique Fédérale de Lausanne,

3- Research products and activities for the RM team

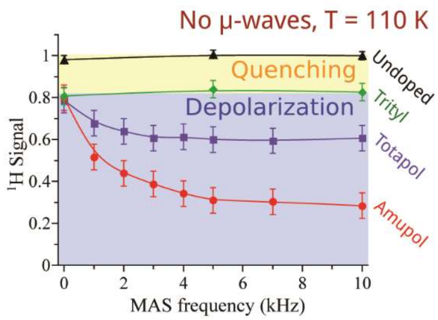
Developing new methods and instrumentation in DNP-enhanced NMR

The backbone of the DNP research carried out in the RM lab consists in advancing the field of high field magic angle spinning dynamic nuclear polarization and solid-state NMR. To achieve this goal, we actively develop advanced instrumentation and innovative methodology: from the design of improved pulse sequences to the introduction of new polarizing agents (that can be used as paramagnetic dopants in DNP experiments). Over the last 5 years, this research direction has led to the publications of 9 papers and has directly involved 5 non-permanent staff. Several collaborations have been successfully conducted with leading groups in the field (R.G. Griffin at Massachusetts Institute of Technology and S. Vega at Weizmann Institute of Science) and a long-standing and fruitful collaboration with Prof. S Sigurdsson (Iceland University) on the synthesis of polarizing agent for DNP has been initiated and will be pursued in the coming years. Finally, our collaboration with Bruker Biospin was strengthened over the last years leading to the co-development of a second generation of helium spinning DNP probe.

Sustainable helium spinning MAS-DNP: fast MAS and ultra-low temperature. Over the last 5 years, we have made significant progress in the field of sustainable helium spinning for DNP. This work implies a strong long-standing collaboration with Irig/SBT (E. Bouleau and coll.). In 2015, we were one of the first groups in the world (together with Osaka University) to demonstrate helium spinning using a closed-loop circuit combined with DNP at temperature << 100 K (which corresponds to what can be reached with the commercial setup). With this proof-of-principle, we were able to prove faster sample spinning with helium than with nitrogen and to demonstrate about one order of magnitude of sensitivity increase by performing DNP at T << 100 K. [80]Over the last two years, we have invested significant efforts in designing and assembling a second generation of helium spinning DNP probes – in close collaboration with Bruker. This new probe is currently being assembled and will be soon tested together with an improved version of the cryogenic power supply (dubbed SACRYPAN). Hopefully, this should allow us conducting sustainable helium spinning experiments on a daily basis in the coming years.

Insight into MAS-DNP mechanism using spin-dynamics simulations. The most efficient polarizing agents typically belong to the nitroxide biradicals class. Their efficiency is usually assessed by measuring the ratio between the NMR signal intensity in the presence and the absence of microwave irradiation $\varepsilon_{on/off}$. Such a comparison is valid

[80] Bouleau, E.; Saint-Bonnet, P.; Mentink-Vigier, F.; Takahashi, H.; Jacquot, J. F.; Bardet, M.; Aussenac, F.; Purea, A.; Engelke, F.; Hediger, S.; Lee, D.; De Paepe, G. *Chemical Science* 2015, 6, 6806-6812.



as long as the signal measured in the absence of microwaves is merely the Boltzmann polarization and is not affected by the spinning of the sample. In our work we demonstrate that TOTAPOL and AMUPOL both lead to observable depolarization at ~110 K, and that the magnitude of this depolarization is radical dependent. This experimental work is analyzed using a theoretical model that explains how the depolarization process works under MAS and gives new insights into the DNP mechanism and into the spin parameters, which are relevant for the efficiency of a biradical.[81] A deeper understanding of parameters affecting MAS–DNP is thus crucial for the development of new polarizing agents and the successful implementation of the technique at higher magnetic fields (>10 T). Hence, we developed an alternative approach to existing cross-effect and solid-effect MAS–DNP codes that yields fast and accurate simulations of large spin ensemble.

Coupling DFT and high-field EPR measurements. Relating Polarizing Agent (PA) chemical structure to DNP efficiency has been, and is still, a long-standing problem. Over the last years, we have investigated the relevance of advanced MAS-DNP simulations combined with DFT calculations and high-field EPR to qualitatively and quantitatively predict hyperpolarization efficiency of particular PAs is analyzed. We demonstrated this approach on AMUPol and TEKPol, two widely-used bis-nitroxide PAs. The results notably highlight how the PA structure and EPR characteristics affect the detailed shape of the DNP field profile. We also show that refined simulations of this profile using the orientation dependency of the electron spin-lattice relaxation times can be used to estimate the microwave B1 field experienced by the sample. Finally, we show how modelling the nuclear spin-lattice relaxation times of close and bulk nuclei while accounting for PA concentration allows for a prediction of DNP enhancement factors and hyperpolarization build-up times.[82]

Understanding and developing new Polarizing Agents for DNP. Thanks to our experimental and theoretical development in the field of DNP, we were able to conduct *in depth* studies comparing bis-nitroxides and Trityl-Tempo biradicals. Notably we were able to fully understand TEMTriPol-1 (a recently introduced mixed biradical) and to design and propose a new family of simple and asymmetrical bis-nitroxides called AsymPol with improved efficiency. See Highlight below.[83]

NMR Pulse Sequence development. In the field of pulse sequence development, we made two contributions: the first one extends the applicability of supercycled sequences and the second enables the removal of solvent signals in DNP-enhanced NMR spectra. More precisely, we have shown that 2D double-quantum single-quantum correlation spectra with arbitrary spectral widths can be recorded with SR26 and related supercycled recoupling sequences when applying Supercycle-Timing-Compensation (STiC) phase shifts. This concept widely extends the applicability of supercycled sequences, most importantly for obtaining long-range distance constraints for structure determination with solid-state NMR.[84] In the second work, we introduce two methods to suppress DNP solvent signals: a Forced Echo Dephasing experiment (FEDex) and TRAnsfer of Populations in DOuble Resonance Echo Dephasing (TRAPDORED) NMR. These methods reintroduce a heteronuclear dipolar interaction that is specific to the solvent, thereby forcing a dephasing of recoupled solvent spins and leaving acquired NMR spectra free of associated resonance overlap with the analyte.[85]

Main funds: ANR / H2020 / ERC / industrial contract

[81] Mentink-Vigier, F.; Paul, S.; Lee, D.; Feintuch, A.; Hediger, S.; Vega, S.; De Paepe, G. *Physical Chemistry Chemical Physics* 2015, *17*, 21824-21836.
[82] Mentink-Vigier, F.; Barra, A. L.; van Tol, J.; Hediger, S.; Lee, D.; De Paepe, G. *Physical Chemistry Chemical Physics* 2019, *21*, 2166-2176.
[83] Mentink-Vigier, F.; Vega, S.; De Paepe, G. *Physical Chemistry Chemical Physics* 2017, *19*, 3506-3522.
[84] Marker, K.; Paul, S.; Fernandez-de-Alba, C.; Lee, D.; Mouesca, J. M.; Hediger, S.; De Paepe, G. *Chemical Science* 2017, *8*, 974-987.
[85] Lee, D.; Chaudhari, S. R.; De Paepe, G. *Journal of Magnetic Resonance* 2017, *278*, 60-66

Highlight 1: Pushing NMR sensitivity limits using dynamic nuclear polarization with closed-loop cryogenic helium sample spinning

We have reported on a strategy to push the limits of solid-state NMR sensitivity far beyond its current state-of-the-art.[86] The approach relies on the use of dynamic nuclear polarization and demonstrates unprecedented DNP enhancement factors for experiments performed at sample temperatures much lower than 100 K, and can translate into 6 orders of magnitude of experimental time-savings. This leap-forward was made possible thanks to the employment of cryogenic helium as the gas to power magic angle sample spinning (MAS) for dynamic nuclear polarization (DNP) enhanced NMR experiments. These experimental conditions far exceed what is currently possible and allows currently reaching sample temperatures down to 30 K while conducting experiments with improved resolution (thanks to faster spinning frequencies, up to 25 kHz) and highly polarized nuclear spins. The impressive associated gains were used to hyperpolarize the surface of an industrial catalyst as well as to hyperpolarize organic nano-assemblies (self-assembling peptides in our case), for whom structures cannot be solved using diffraction techniques. Sustainable cryogenic helium sample spinning significantly enlarges the realm and possibilities of the MAS-DNP technique and is the route to transform NMR into a versatile but also sensitive atomic-level characterization tool.

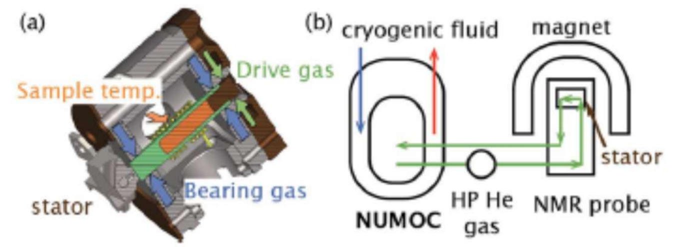
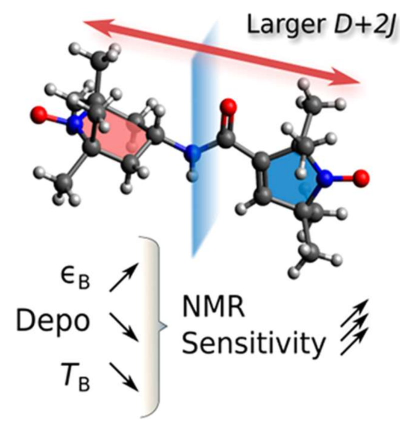


Fig. 1 Schematics of (a) an NMR stator and (b) the ultra-low-temperature MAS-DNP setup called NUMOC.

Highlight 2: Understanding and developing new radicals for DNP

Magic Angle Spinning Dynamic Nuclear Polarization allows enhancing NMR sensitivity in the solid-state by several orders of magnitude and has thus revolutionized atomic-scale structure determination. Currently, the most popular polarizing agents are composed of two nitroxides moieties tethered by an organic linker. However, for magnetic fields higher than 5 T and MAS frequencies larger than 15 kHz, the best known biradicals are still far from optimal. By investing time and efforts into understanding the spin physics behind the polarization transfer mechanism, we were able to deepen significantly our understanding and to relate the biradical structure and electronic properties to the DNP efficiency. Thanks to this progress we were able to propose a new family of polarizing agents, composed of asymmetric bis-nitroxides, in which a piperidine-based radical and a pyrrolinoxyl or a proxyl radical are linked together. [87]The design of the AsymPol family was guided by the use of advanced simulations that allow computation of the impact of the radical structure on DNP efficiency. These simulations suggested the use of a relatively short linker with the intention to generate a sizable intramolecular electron dipolar coupling/J-exchange interaction, while avoiding parallel nitroxide orientations. The characteristics of AsymPol were further tuned, for instance with the addition of a conjugated carbon−carbon double bond in the 5-membered ring to improve the rigidity and provide a favorable relative orientation, the replacement of methyls by spirocyclohexanolyl groups to slow the electron spin relaxation, and the introduction of phosphate groups to yield highly water-soluble dopants. An in-depth experimental and theoretical study for two members of the family, AsymPol and AsymPolPOK, is presented here. We report substantial sensitivity gains at both 9.4 and 18.8 T. The robust efficiency of this new family is further demonstrated through high-resolution surface characterization of an important industrial catalyst using fast sample spinning at 18.8 T. This work highlights a new direction for polarizing agent design and the critical importance of computations in this process.



Probing and developing advanced materials using (DNP-enhanced) solid-state NMR

The DNP group is also developing advanced applications in the field of materials science, with 7 publications in this domain, 4 non-permanent staff involved directly from this research direction, continued collaborations, 6 new academic collaborations, 5 new industrial collaborations. Overall, the research has targeted a variety of key areas including **biomaterials, catalysis, energy generation and storage, and nanocrystal semiconductors**. The industry collaborations have involved batteries, catalysis, phosphors, and polymers (simple organic as well as hybrid organic/inorganic). Much work has been put into developing (DNP-enhanced) solid-state NMR

[86] Bouleau, E.; Saint-Bonnet, P.; Mentink-Vigier, F.; Takahashi, H.; Jacquot, J. F.; Bardet, M.; Aussenac, F.; Purea, A.; Engelke, F.; Hediger, S.; Lee, D.; De Paepe, G. *Chemical Science* 2015, 6, 6806-6812.

[87] Mentink-Vigier, F.; Marin-Montesinos, I.; Jagtap, A. P.; Halbritter, T.; van Tol, J.; Hediger, S.; Lee, D.; Sigurdsson, S. T.; De Paepe, G. *Journal of the American Chemical Society* 2018, *140*, 11013-11019.

spectroscopy so as to gain the greatest possible atomic-level understanding of these materials, often resulting in improved understanding of the material chemistry and sometimes into optimized materials. This research is conducted in strong collaborations with leading local, national and international chemists.

Advanced methods have been developed to probe structures and chemical modifications of nano-systems important to **energy research**. When the bulk of a material is devoid of ^{1}H nuclei, as is the case with many inorganic systems, then adding exogenous polarizing agents in protonated solvents and performing ^{1}H DNP and then cross-polarization (CP) to nuclei from the analyte provides a sensitive *and* surface-selective analysis tool. We have shown that this methodology can be exploited to observe only the first surface layer of γ-alumina (Al_2O_3), a widely-employed catalyst and catalyst support, evidencing the presence of penta-coordinated alumina sites.[88] These sites, thought to be responsible for the catalytic activity, were not observed in a separate experiment designed to only detect bulk (not surface) nuclei. In addition, this methodology for sensitive *and* surface-selective NMR spectroscopy was developed to study the grafting of organic functional groups to the surface of inorganic *silica* (SiO_2) nanoparticles [see FM below].[89] The grafting of organic groups to inorganic *silicon* nanoparticles (Si NPs) was also studied with solid-state NMR spectroscopy, and was consolidated with DFT calculations.[90] The surfaces of Si NPs are highly reactive so organic functionalization was performed to passivate these surfaces, and the detailed DFT/NMR characterization determined the presence of various grafting modes, to different surface facets, as well as the presence of remaining silicon hydride moieties and the beginnings of amorphous surface oxide (thus SiO_x) layers. This result was crucial to the chemist looking to provide air-stable silicon nanocrystals.

Hybrid organic-inorganic materials have been of large interest to the RM team. In addition to the aforementioned examples, research directions have involved understanding interfacial interactions in industrial materials, as well as in **synthetic and natural biomaterials** such as bones and teeth. For the latter research direction, the RM team was able to show that with the sensitivity increases available from DNP, ^{43}Ca at natural isotopic abundance (0.1%) in carbonated hydroxyapatite (a synthetic mimic of bone mineral) was now amenable to fast analysis through NMR spectroscopy.[91] Notably, the obtainable sensitivity permitted the acquisition of 2D correlation experiments, which enabled the differentiation of core and surface calcium environments. This exciting research direction has resulted in a consortium of national actors (ICGM, LCMCP, and INSERM), led by the RM team, to submit a proposal to the ANR to continue to develop this further and the project is currently under review at the second stage. Being able to target surface calcium environments will allow for the study of interactions of biominerals with organic molecules, which is crucial to gain a better understanding of important bone diseases such as osteoporosis as well as in the design of bio-compatible bone grafts and bone replacements.

A further exciting research direction, in collaboration with a strong international partner (Warsaw Univ. of Tech. and the Polish Academy of Sciences), involves gaining insights into the dynamic surface chemistry of (predominantly ligand-capped) **hybrid semiconductor nanocrystals**, important in solar cells, for example. Depending on the ligand, as well as the synthesis route, a large variety of nanocrystal shapes, sizes, and stabilities can be produced. These differences play a large role in the resulting properties. Although there are many techniques available to study the inorganic core of these nanocrystals (TEM, PXRD etc.), being able to gain insights into ligand-surface interactions remains particularly challenging. However, the RM team have shown that a detailed understanding of these interactions, including inter-ligand spacing, ligand coordination mode etc., can be obtained using advanced NMR methodology and signals gains from DNP. This work elucidated why nanocrystals prepared via sol-gel or organometallic approaches led to materials with drastically different properties. Owing to the success of this collaboration, a joint H2020-COFUND PhD student has been hired between the teams, and a CEA sujet phare PhD student spends 50 % of her time working in this direction.

Continuing with the theme of energy-related materials, another ANR project has been submitted, with the RM team as partners (along with LISE and CIRIMAT), and involves improving and understanding the performance of next generation **graphene-based supercapacitors**. The project further expands on our developed methodology and expertise.[92] The RM team not only works to detail the atomic-level structure of these materials, but also modes of charge storage and material aging/degradation, which are critical for full device applications.

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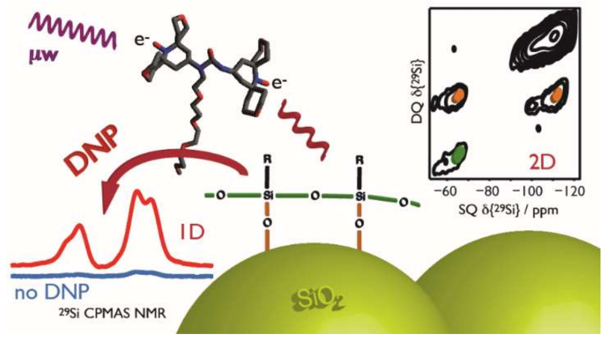
[88] Lee, D.; Duong, N. T.; Lafon, O.; De Paepe, G. *Journal of Physical Chemistry C* 2014, *118*, 25065-25076.
[89] Lee, D.; Monin, G.; Duong, N. T.; Lopez, I. Z.; Bardet, M.; Mareau, V.; Gonon, L.; De Paepe, G. *Journal of the American Chemical Society* 2014, *136*, 13781-13788.
[90] Lee, D.; Kaushik, M.; Coustel, R.; Chenavier, Y.; Chanal, M.; Bardet, M.; Dubois, L.; Okuno, H.; Rochat, N.; Duclairoir, F.; Mouesca, J. M.; De Paepe, G. *Chemistry-a European Journal* 2015, *21*, 16047-16058.
[91] Lee, D.; Leroy, C.; Crevant, C.; Bonhomme-Coury, L.; Babonneau, F.; Laurencin, D.; Bonhomme, C.; De Paepe, G. *Nature Communications* 2017, *8*.
[92] Banda, H.; Perie, S.; Daffos, B.; Taberna, P. L.; Dubois, L.; Crosnier, O.; Simon, P.; Lee, D.; De Paepe, G.; Duclairoir, F. *Acs Nano* 2019, *13*, 1443-1453.

Main funds: ANR / H2020 / ERC / industrial contract

Highlight 3: Elucidating the atomic structures at the interfaces of organic-inorganic hybrids enables a detailed understanding of their properties.[10]



Functionalization of silica nanoparticles with organosiloxanes, particularly useful in proton exchange membrane fuel cells, can produce vertically or laterally polymerized functions, or a combination of both. The determination of which type of polymerization has occurred is an old problem, with no significant solution. Two-dimensional solid-state ^{29}Si-^{29}Si correlation NMR spectroscopy, whereby proximities between various silicon sites are probed, presents itself as a technique that could answer this. Unfortunately, the low natural isotopic abundance of ^{29}Si (< 5%), the NMR-active isotope of silicon, and the expense of isotopic labeling with this nucleus prevents this kind of experiment since the resulting low sensitivity renders any experiment impracticable. However, the RM team showed that DNP could be successfully employed to substantially increase the NMR sensitivity so that these previously infeasible experiments could be performed almost routinely. An experimental time-saving factor of 625 compared to conventional NMR was achieved, allowing 2D data to be recorded in easily accessible timeframes (< 6 h). Analysis of the acquired NMR data showed that the required condensation reaction conditions produced the desired lateral polymerization.

Studying challenging biomolecules using DNP-enhanced solid-state NMR

The DNP group is also developing new applications in the field of small molecules and biomolecular NMR, with 6 publications in this domain, 4 non-permanent staff involved directly from this research direction. Overall, the research has targeted a variety of key areas where we believe DNP-enhanced solid-state NMR can make a difference compared to other atomic resolution techniques. In addition, we also kept in mind while designing this research direction that performing 10-20 T DNP experiments at cryogenic temperature can also have some strong disadvantages compared to other emerging NMR approaches, like for instance, performing ^{1}H detection at very high magnetic field (>20 T) and very fast MAS (> 100 kHz). Beyond the fact that freezing the sample does not allow studying dynamics of functional protein at RT, it also induces a (severe) broadening of the NMR resonances (that depends on the flexibility of the system). This fact, combined with current MAS-DNP probe technology limitation (<40 kHz MAS frequency) have so far prevented the successful implementation of ^{1}H detection under DNP conditions. In this context, we have focused our efforts towards methodological developments in three research directions. The first one focuses on structural investigation of organic/biomolecular assemblies at natural isotopic abundance (NA), from small molecules to protein aggregates. Such developments are especially relevant for the study of samples that can not be isotopically labeled (e.g. from plants, animals or patients). The second direction aims at developing a DNP methodology that enables addressing binding sites or interfaces in (large) biomolecular systems. The goal is not only to locate the ligand-binding site but also to extract structural information from the binding site at the atomic level. The third axis investigates the use of DNP in the context of cellular environment, with a strong focus the cell envelope itself.

Structure determination at natural isotopic abundance. In 2012, our group pioneered the development of 2D ^{13}C-^{13}C natural abundance spectroscopy by reporting the first 2D correlation experiment acquired on a sample at NA in only 20 minutes thanks to the sensitivity enhancement of MAS-DNP. We pursued our efforts to develop all methodological aspects required to establish the technique in the field of NMR crystallography and expand it to biomolecular applications. Thus, we demonstrated for the first time the feasibility of ^{13}C-^{15}N correlation spectroscopy at NA on a guanosine derivative forming supramolecular structures. Together with *J*- and dipolar recoupled carbon-carbon 2D experiments, this experiment is key to address complete and non-ambiguous assignment of molecules at NA.[93] In particular, we put into evidence the clear advantage of being at NA for measuring long-range constraints compared to enriched samples, thanks to the simplified spin dynamics, which can be considered as restricted within isolated two-spin systems. This greatly simplifies the interpretation of polarization transfer curves, as demonstrated on self-assemblies of cyclic diphenylalanine peptide, for which we

93 Marker, K.; Pingret, M.; Mouesca, J. M.; Gasparutto, D.; Hediger, S.; De Paepe, G. *Journal of the American Chemical Society* 2015, *137*, 13796-13799.

were able to probe π-stacking contacts responsible for the supramolecular arrangement.[94] We then applied this methodology specifically based on samples at NA to peptide and protein aggregates responsible for the neurodegenerative Huntington's disease.[95] Structural fingerprints and constraints obtained on this biological highly relevant system are promising for the future use of MAS-DNP in the investigation of material directly removed from patients.

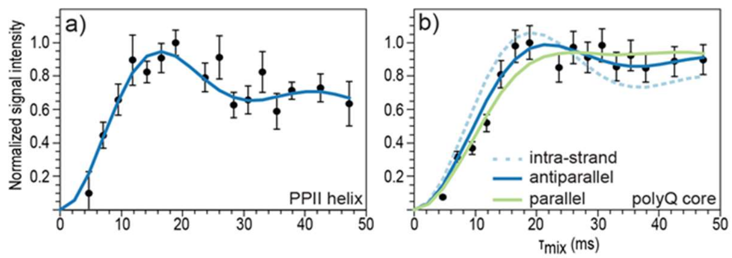


Figure. Experimental and simulated 13Ca – 13Ca buildup curves for the a) polyproline PPII-helix, and b) polyQ core of the first exon of mutant huntingtin protein (Q44-HttEx1) at NA.

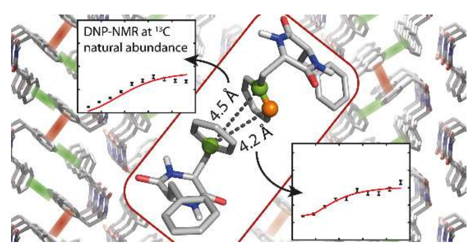
Selective DNP-enhanced NMR (Sel-DNP). The idea of fixing the polarizing agent directly on the system of interest has emerged since several years in different groups, including ours, to address the optimization of the sample preparation or to target the molecule of interest in a complex environment. This last idea suffers however from the bleaching of radical close-by resonances due to paramagnetic relaxation. We turned this effect in our favor in an innovative approach dubbed Sel-DNP, by combining ligand-functionalized paramagnetic construct with difference spectroscopy.[96] Sel-DNP allows thus selectively highlighting and identifying residues present in the ligand binding site of a biomolecular system. Demonstrated on the galactophilic lectin LecA (121 residues), Sel-DNP provided highly-resolved spectra of the binding region, which enabled the de novo assignment of the binding interface residues. We expect Sel-DNP to become an important method for the study of protein binding sites, e.g. membrane proteins, possibly in their cellular environment.

Addressing cell envelopes by DNP. Interactions of proteins with the cell envelope are central to understand many biological functions, and investigating them in a context close to their native environment is therefore of the utmost importance. Considering the sensitivity problem resulting from the dilution of the system of interest, DNP should play a crucial role in such applications, provided the complex structure of the cell membrane can be enhanced. For this purpose, we developed a specific polarizing agent able to insert inside bilayers and to hyperpolarize the cell membrane and membrane-anchored proteins. Similarly, we pursued our investigation of the bacterial cell wall to address the more complex and impermeable cell envelope of mycobacteria.

Main funds: ANR / H2020 / ERC / Labex ARCANE

Highlight 4: Welcoming natural isotopic abundance in solid-state NMR: from the crystallographic study of organic nanoassemblies to the structural fingerprinting of protein aggregates using DNP

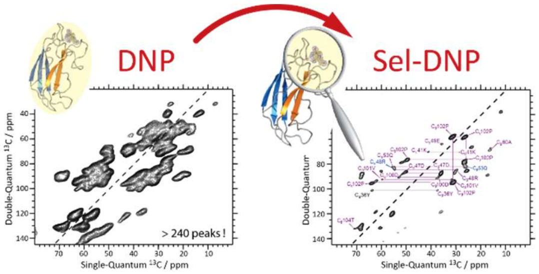
The self-assembly of small organic molecules finds applications in numerous fields. Their rational design requires a detailed knowledge of their supramolecular structure. In this context, we proved the feasibility of a novel concept of NMR-based 3D structure determination, based on the deliberate use of samples at their natural isotopic abundance (NA, 1.1% for ^{13}C), while exploiting MAS-DNP to compensate for the reduced sensitivity. Thanks to the natural dilution of NMR active spins, unique and highly informative spectra can be recorded which are impossible to obtain on an isotopically labeled system. On the self-assembled cyclic diphenylalanine peptide, we detected long-range internuclear distances up to 7 Å, allowing us to observe π-stacking, one of the most important non-covalent interactions. Beyond nanoassemblies, this approach has been also successfully applied to probe the atomic structure at NA of protein deposits responsible for the neurodegenerative Huntington's disease, enabling the extraction of valuable new long-range $^{13}C-^{13}C$ distance constraints. This work opens up a new avenue to de novo crystal structure determination by NMR and to the structural study of unlabeled protein aggregates derived from cells, patients, and other hard-to-label sources.



[94] Marker, K.; Paul, S.; Fernandez-de-Alba, C.; Lee, D.; Mouesca, J. M.; Hediger, S.; De Paepe, G. *Chemical Science* 2017, *8*, 974-987.
[95] Smith, A. N.; Marker, K.; Piretra, T.; Boatz, J. C.; Matlahov, I.; Kodali, R.; Hediger, S.; van der Wel, P. C. A.; De Paepe, G. *Journal of the American Chemical Society* 2018, *140*, 14576-14580.
[96] Marin-Montesinos, I.; Goyard, D.; Gillon, E.; Renaudet, O.; Imberty, A.; Hediger, S.; De Paepe, G. *Chemical Science* 2019, *10*, 3366-3374.

Highlight 5: Selective high-resolution DNP-enhanced NMR of biomolecular binding sites[97]

Locating binding sites in biomolecular assemblies and solving their structures is of the utmost importance to unravel functional aspects of the system and provide experimental data that can be used for structure-based drug design. This often still remains a challenge, both in terms of selectivity and sensitivity for X-ray crystallography, cryo-electron microscopy and NMR. In this context, we developed a novel method called Selective Dynamic Nuclear Polarization (Sel-DNP) that allows selectively highlighting and identifying residues present in the binding site. This powerful site-directed approach relies on the use of localized paramagnetic relaxation enhancement induced by a ligand-functionalized paramagnetic construct combined with difference spectroscopy. The methodology has been demonstrated on the galactophilic lectin LecA, for which we reported well-resolved DNP-enhanced spectra, which enabled the de novo assignment of the binding interface residues. Since this approach produces clean and resolved difference spectra containing a limited number of residues, resonance assignment can be performed without any limitation with respect to the size of the biomolecular system, and only requires the production of one protein sample (e.g. uniformly ^{13}C, ^{15}N labeled).

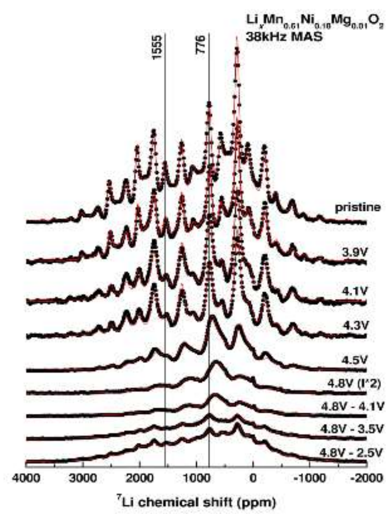


Liquid and solid-state NMR for energy

In the field of materials for energy over the last 10 years the demands for NMR, either as a classical control tool or at an advanced level, leading to publications in which NMR palys a central role, has considerably increased. The main targeted domains are the following:

a. Biomass conversion for fuels and biogas using torrefaction or pyrolyse[98],
b. Fuel-cell membranes: optimization, ageing and dynamics,
c. Materials for hydrogen storage (new axe…),
d. Materials for batteries:
   - Pristine material characterization; inorganic ($LiFePO_4$, LiMO…) and organic (PTCI, $PTCLi_6$)[99],
   - Electrolyte behaviour and dynamics (PFG-NMR),
   - Black carbon from biomass.

All these investigated fields have been conducted in strong collaboration with LITEN and about 10 publications over the period were published. The main interest of solid-state NMR is undoubtedly the possibility to follow the structural changes: To illustrate this feature the figure opposite gives ex-situ high-resolution solid-state ^7Li NMR of cathode active material (LiNiNnO) that were cycled at different voltages. In this example one could not only trace lithiation and delithiation process but also followed the structural consequences on the remaining matrice going from an ordered to a disordered structure. For this precise study a computational approach was specifically dedicated to rationalize the results. For that, Li chemical shifts, dipolar and hyperfine interactions were calculated for all the possible configurations expected from the known X-ray structure and evaluated against the experimental solid-state Li NMR data (coll. with J. M. Mouesca, Symmes). In order to minimize the effect due to hyperfine coupling, Li spectra were recorded at rather low field (4.7 T) and at high spinning rate 60 KHz.



[97] Marin-Montesinos, I.; Goyard, D.; Gillon, E.; Renaudet, O.; Imberty, A.; Hediger, S.; De Paepe, G. *Chemical Science* 2019, *10*, 3366-3374.
[98] Melkior, T.; Barthomeuf, C.; Bardet, M. *Fuel* 2017, *187*, 250-260.
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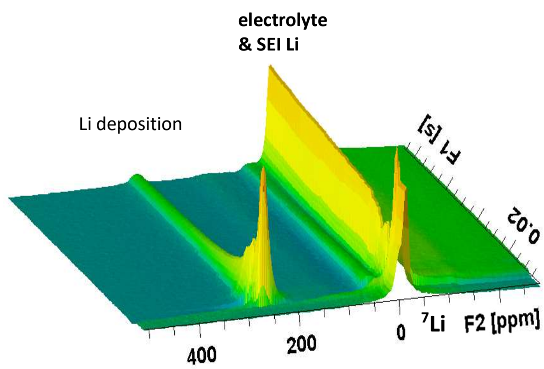
Over the last ten years a large a set of chemical shifts for Lithium in different chemical environments (graphite, silicon, LiMO) have been collected which currently makes ex situ analyses of materials from pouch cells prepared under very different types of conditions (electrochemistry, ageing, storage conditions and formulations and processes themselves) very efficient.[100] All those studies were conducted in tight collaborations with colleagues from LITEN.



Moreover, to better understand the behaviour of the studied materials operando NMR, as fruitfully developed in Grey's Laboratory, have appeared as a complementary tool of the other NMR approaches that are carried out in the laboratory. A probe was specifically modified and NMR coils designed to match the flat volume of the pouch cells that are produced at LITEN. Using the adequate filters and shielding operando NMR can be carried out to follow with ^{7}Li NMR electrochemical cycling of pouch cells.

Typical experiment is shown in the figure above carried out on our 4.7 T spectrometer showing for instance the appearance of lithium metal and then its insertion in graphite depending on the electrochemical cycling.

Main funds: Sintbat, TEESMAT (Horizon 2020)

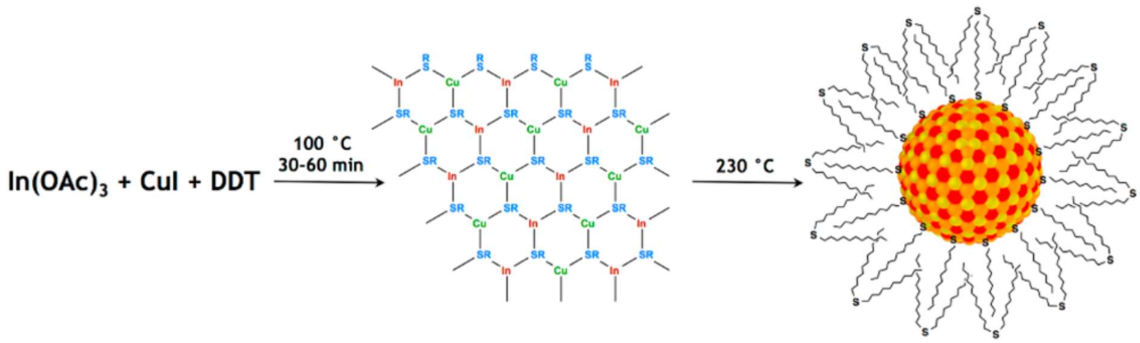
Liquid-state and PFG NMR for studies of ligands on the surface of nanocrystals.

Liquid state NMR – represent a powerful tool for the investigation of ligand shell on the surface of colloidal nanocrystals (NC). [101] Most colloidal nanocrystals, come with a capping of organic ligands, and the chemistry of this organic/inorganic interface affects their physical and chemical properties. For almost all the applications, it is necessary to replace the original ligands, by other molecules. So, it is very important to identify the surface ligands from the synthesis, but also after the step of functionalization of NCs. Different NMR experiments can be carried out in order to identify these organic molecules. In the simplest cases (well resolved spectra of ligands tightly bounded to NC), standard 1D NMR experiments can help to identify the bounded ligands on the basis of observed peak broadening, chemical shifts modifications. As NMR represents a quantitative method, the density of ligand on the surface of NC can be established. In more complex cases, two dimensional NMR and Diffusing NMR made possible the studies of the affinity of the ligands for the NC surface and the exchange dynamics between NC-bounded and free ligands in solution. In this context, we contributed to the studies of ternary metal chalcogenide nanocrystals $CuInS_2$ (CIS). These NCs possess the optoelectronic properties tunable with size and composition and they can be easily prepared with good size control and in high yield. Concerning the surface state, many studies showed that it was very challenging to replace quantitatively the surface ligands of the as-prepared NCs. In this context, we are looking to understand the formation mechanism and surface state of CIS NCs synthesized using dodecanethiol (DDT) as solvent, surface ligand, and sulfur source. By means of complementary 1D and 2D NMR techniques, we were able to draw a complete picture of the surface chemistry: (1). The ligand shell is composed of a double layer consisting of dodecanethiolate and didodecyl sulfide molecules in 1:1 ratio, assembled in a head-to-tail manner. This ligand double layer is in full accordance with the observed mass loss in thermogravimetric analysis. Furthermore, a high surface ligand density of 3.6 nm^{-2} has been determined, which has important consequences for the functionalization of the CIS NCs. They have been shown to withstand standard ligand exchange and aqueous phase transfer procedures. Nonetheless, the binding of dodecanethiolate occurs in a dynamic way, yet with a fast exchange rate ($\gg$50 s^{-1}). Its desorption and adsorption processes are accompanied by protonation and deprotonation reactions. Therefore, the use of appropriate protonating agents is a promising strategy for the surface functionalization of CIS NCs obtained with the DDT method. [102]

[100] Matadi, B. P.; Genies, S.; Delaille, A.; Waldmann, T.; Kasper, M.; Wohlfahrt-Mehrens, M.; Aguesse, F.; Bekaert, E.; Jimenez-Gordon, I.; Daniel, L.; Fleury, X.; Bardet, M.; Martin, J. F.; Bultel, Y. *Journal of the Electrochemical Society* **2017**, *164*, A1089-A1097.

[101] Lefrancois, A.; Luszczynska, B.; Pepin-Donat, B.; Lombard, C.; Bouthinon, B.; Verilhac, J. M.; Gromova, M.; Faure-Vincent, J.; Pouget, S.; Chandezon, F.; Sadki, S.; Reiss, P. *Scientific Reports* **2015**, *5*.

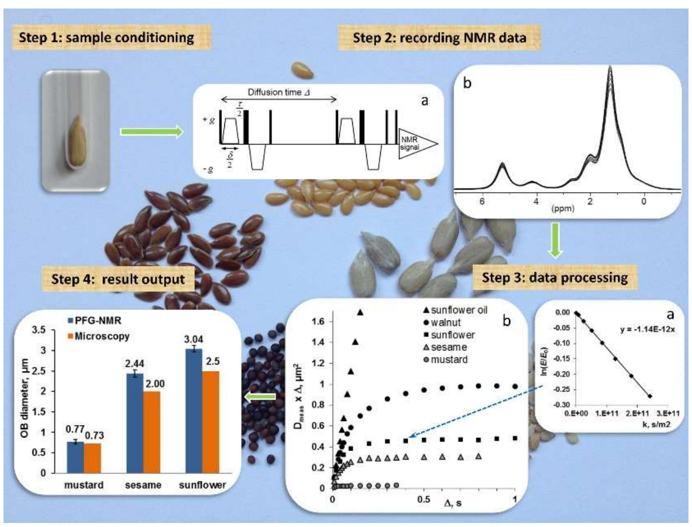
[102] Gromova, M.; Lefrancois, A.; Vaure, L.; Agnese, F.; Aldakov, D.; Maurice, A.; Djurado, D.; Lebrun, C.; de Geyer, A.; Schulli,T. U.; Pouget, S.; Reiss, P. *Journal of the American Chemical Society* 2017, *139*, 15748-15759.



Main funds: supported by SyMMES Laboratory

In situ NMR of seeds and microalgae to study lipid droplets

The neutral lipids (triacylglycerols (TAG)), for instance in oleaginous crops, are accumulated inside seeds in small sphE. al organelles – Lipid Droplets (LD). The molecules of oil - triacylglycerol (TAG) - represent a mobile part of seeds, consequently they can be studied by liquid state NMR. In particular, diffusional or pulsed field gradient nuclear magnetic resonance (PFGNMR) has been demonstrated to be a powerful method to measure the self-diffusion coefficient and to characterize the restricted diffusion in mesoporous media and confined systems. [103]



The successive steps of PFG NMR measurement of LD size in vegetable seeds.

For biological applications, the NMR approach is very interesting because it is a non-invasive method and it allows the analysis of hundreds of thousands of cells at once. In particular, we extended the use of this method to weak gradients NMR probes which represent the standard NMR equipment. The obtained results allowed the determination of the mean LD size in different seeds and also to correlate the LD size ant neutral lipid seed contents. Moreover, it was possible to demonstrate the LD-to-LD TAG displacements which could represent an interesting parameter in seed state monitoring during storage. Currently, this approach is successfully extended to the studies of oil storage in microalgae used for 3th generation biofuels developments. [104]

Main funds: CEA program" DRF impulsion"

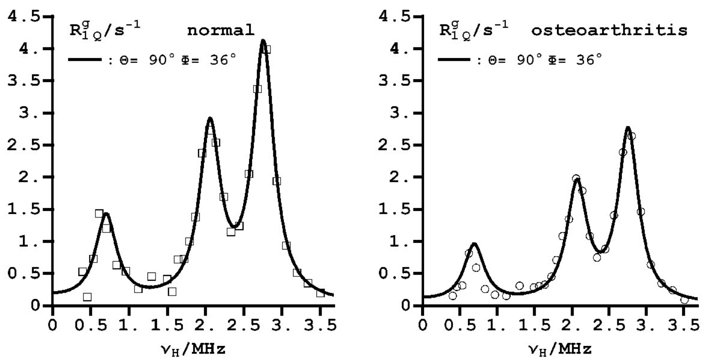
Relaxometry: new instrumentation, methods, and theories with developments to medical applications

In a fixed external magnetic field B_0, NMR relaxometry, i.e., the measurement of the relaxation rates of systems of nuclear spins, provides a Fourier analysis of time correlation functions of the positions of these spins at their Larmor resonance frequencies.[105] Fast field-cycling (FFC) NMR relaxometry allows one to perform that Fourier analysis over a frequency range covering six decades, thus providing a unique method to study, characterize and authenticate any type of condensed non-metallic material by probing its molecular dynamics properties.

[103] Gromova, M.; Guillermo, A.; Bayle, P. A.; Bardet, M. *European Biophysics Journal with Biophysics Letters* 2015, *44*, 121-129.
[104] Boulard, C.; Bardet, M.; Chardot, T.; Dubreucq, B.; Gromova, M.; Guillermo, A.; Miquel, M.; Nesi, N.; Yen-Nicolay, S.; Jolivet, P. *Planta* **2015**, *242*, 53-68.
[105] Fries, P. H.; Belorizky, E. *Journal of Chemical Physics* **2015**, *143*.

[106] Long confined to a few specialized laboratories, this technique is developing mainly in Europe. In that context, a new FFC-NMR relaxometer, capable to explore the frequency domain down to 100 Hz or less (B_0 < 2 µT), is under development at RM within the framework of the European H2020 IDentIFY project. Note that FFC-NMR instruments, where field-cycling is produced by a fast current variation in a large coil, can be transformed into FFC-MRI scanners when they are equipped with appropriate pulsed field gradients.

This milestone for exploring slow molecular motion is accompanied, first, by a reinforced cooperation with INSERM (Dr Hana Lahrech) with the goal of discovering biomarkers of pathology at low-field magnetic resonance imaging (MRI) and, second, by theoretical and methodological developments[107].[108] Thus, we derived very simple analytical expressions of the quadrupole relaxation enhancement (QRE) of the longitudinal relaxation rate of nuclear spins due to their intramolecular magnetic dipolar coupling with quadrupole nuclei of arbitrary spins.[105] These expressions were obtained by using the adiabatic approximation for evaluating the time evolution operator of the quantum states of the quadrupole nuclei. The relaxation rate profiles vs Larmor frequency of the hydrogen nuclei of water can display QRE peaks in living tissues when those hydrogen atoms temporarily bind to protein nitrogen atoms which have quadrupole nuclei. As shown in the figure, such peaks can reveal tissue modifications due to osteoarthritis which are invisible to standard high field MRI.



Besides, in FFC-NMR experiments, the magnetic field takes several values in turns. We proposed a formalism to properly account for the finite duration of the step between two field values. This allows a correct interpretation of the relaxation in systems with chemical exchange, such as living tissues where the water molecules with their observed hydrogen nuclei move back and forth between intra and extra-cellular compartments. [109]

To be complete, note that despite its versatile application to any possible materials containing magnetic nuclei in motion, the samples that can be explored by NMR relaxometry should contain a sufficient number of magnetic nuclei, for instance, at least about 1 mmol of hydrogen nuclei, which requires 10 mg of biological tissues, water, organic solvents, or polymers.

Main funds: H2020 & COST EU programs: IDentIFY & EURELAX

4- Organisation and life of the RM team

Animation:

The scientific animation in the RM team is organized through regular workgroup meetings or seminars, either inside a subgroup or at the team level. The RM team is also attending seminars from Irig when topics are matching their scientific interest. All RM team members are fully involved in the scientific animation of Irig/MEM.

RM is also part of several local networks (Pole CBS/PEM, Labex Arcane, Idex CDP Glyco@Alps, etc.) that organize regular meetings in the greater Grenoble area. The lab is often solicitated to give presentations in these networks which is a very efficient way to stimulate internal research but also to reach out to colleagues from Grenoble

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[106] Karimdjy, M. M.; Tallec, G.; Fries, P. H.; Imbert, D.; Mazzanti, M. *Chemical Communications* **2015**, *51*, 6836-6838.
[107] Fries, P. H.; Belorizky, E. Molecular Physics 2019, 117, 849-860
[108] Gimenez, U.; Lajous, H.; El Atifi, M.; Bidart, M.; Auboiroux, V.; Fries, P. H.; Berger, F.; Lahrech, H. Contrast Media & Molecular *Imaging* **2016**, *11*, 535-543.

[109] Gimenez, U.; Lajous, H.; El Atifi, M.; Bidart, M.; Auboiroux, V.; Fries, P. H.; Berger, F.; Lahrech, H. *Contrast Media & Molecular Imaging* **2016**, *11*, 535-543.

(from Large Scale Instrument facilities, CNRS/CEA/UGA campuses). Most of the students enrolled in the RM belong to structured student groups (e.g. glyco-club from the CDP glyco@Alps) which enable a better integration into their local scientific environment. This is a very effective way to strengthen local collaborations and, in the end, to support the Idex project from the ComUE (communauté d'universités et établissements) Université Grenoble Alpes.

For the NMR service M. Bardet and P.-A. Bayle have regular meetings. For the administration and general topics the permanent staff have meeting every three weeks. For the NMR service; meetings can be scheduled to discuss spectrometer planning and repair/maintenance problems with the users.

Parity: ~30 % of the permanent team are female; S. Hediger is a CNRS researcher and M. Gromova is Maître de conférence at the UGA. For PhD students, 67 % over the evaluation period are female.

Safety: Safety contacts are present at Minatec and C5 places but main safety tasks are assumed by works by CEA safety officers (L. Miquet). D. Lee assumes local safety training responsibilities for RM at Minatec. S. Hediger and P. A Bayle are first responders in terms of alarms (*équipe locale de premier secours*) for buildings 51C (Minatec campus) and C5 respectively.

Scientific integrity: Each permanent of RM has to assure and to guarantee this mission. For all the research topics the NMR data have been kept without restriction since the creation of RM. Laboratory books and data files are kept by the tutors of all the non-permanents when they left the laboratory.

Five-year project and strategy of the RM team

RM SWOT analysis

Strengths	***Weaknesses***
Project coordination Complementary skills in RM Adaptability to emerging projects NMR facility management RM is embedded in a polyvalent characterization environment International recognition of DNP group Collaborations with the Chemistry department (DIESE)[110] at IRIG, IBS and DRT	Lack of technical support Staff renewal Equipment renewal Localization on two sites RM is not part of a Chemistry Department The lab is not affiliated to CNRS Lack of EPR & DFT skills No access to local wet lab
Opportunities	***Threats/Risks***
Grenoble scientific environnement (Large Scale infrastructure + CNRS + UGA+IDEX) CEA intramural program and dynamics Chemistry/biology & material science local projects SBT partnership	Losses of expertise Critical mass of the lab

[110] DIESE, Département Interfaces pour l'Energie, la Santé, l'Environnement . IBS, Institut de Biologie Structurale. DRT, Direction de la Recherche Technologique

PIA3 « équipements structurants pour la recherche »	No recurrent funding for the maintenance and the repair of the NMR equipment No recurrent funding for new investment Outdated NMR equipment

Structure, workforce and scientific orientations

Global perspectives for RM

The RM activities are naturally embedded in the Grenoble environment (DRF-Irig and DRT-Leti/Liten) but also rely on strong national and international collaborations with leading research groups or industrial partners. The RM will continue to be very active and apply for local/institutional but also national and European funding calls. Nevertheless, the RM lab will have to face several challenges in the coming years:

First the RM lab needs to make sure that its current positioning outside the newly created chemistry department (DIESE) of Irig is sustainable and is not bringing strong practical limitations. For instance, we can question the sustainability of running chemistry activities in a research team that is not affiliated to a chemistry unit.

Second, the RM lab needs to make sure that its positioning as an advanced NMR lab for chemisty/biology and material science is sufficiently clear at the local but also at the national level. This is important, especially since the RM needs to be able to maintain/renew expensive NMR equipment (useful to the RM team but also to the multiple users of the facitlity) and also upgrade to higher magnetic fields for the DNP activity. In 2011, the RM was positioned as the 3rd lab in the world to setup DNP at 9.4 T. Today there are about 45 DNP spectrometers installed worldwide, among which 30 operate at 14 to 21 T.

The RM hopes that there will be opportunities to regroup both the team and the NMR equipment in the future. This could be the case with the renovation of the C5 building (currently envisioned but not confirmed yet) that aims at relocationg all chemists from Irig (SyMMES and LCBM) in the same building. Having all the NMR spectrometers in a common place will undoubtedly benefit local scientists and facilitate maintenance and routine operation (such as magnet filling), as well as improve future synergy within the team.

Finally, it is important to mention that the size of the RM lab has been continuously decreasing over the last years and this is likely to continue in the coming years. In addition, it is very important to resolve several problems: the lack of expertise in computational NMR, MD/DFT simulations and the severe workload and lack of technical support. Also, the RM needs to be able to attract younger talented colleagues and the fact that the team is not affiliated to CNRS (while hosting one CNRS researcher) is a clear limitation. Several young researchers have expressed interest in joining the RM team but due to the limited number of CEA/UGA openings, none of these applications could be envisioned.

NMR service and high-resolution NMR

In terms of scientific orientations for the next term, the NMR service mission assigned to RM will remain a priority. As a matter of fact, an increase in synthesis activities at DIESE is expected and the demands in NMR both as a control tool or for advanced studies should be honored. Moreover, a similar increase in the demands has be already recorded at the DRT. In this context, both Pierre Alain Bayle (CEA technician, responsible of the platform) and M. Gromova (MCF, UGA) will play a central role. M. Gromova will go on developing NMR strategy based on pulsed field gradients (PFG) techniques. PFG NMR will be used to study ligand dynamics at the surface of nanoparticles as well to measure the size of vesicles or droplets, both in synthetic or living systems (seeds and algae), using in that case restrained diffusion approach. The role of Pierre-Alain Bayle will be central to further develop NMR techniques in liquid state to allow the chemists to reach their objectives in their specific NMR projects requiring appropriate sequences, heteroatoms, temperature, basic CPMAS analyses and HRMAS studies). Over the last ten years RM has developed NMR for material for electrical collecting and storage. This goal is mainly driven by M. Bardet in the solid state and Pierre Alain in liquid. As M. Bardet will doubtless be retired within three years, the durability of the skills acquired around solid-state NMR for material for electrical collecting and storage has to be considered. As the chance of hiring permanent people is very small, colleagues at DRT will appoint, in their own laboratory, an apprentice engineer for three years who will be

involved in this thematic. A permanent colleague from DRT has also expressed the wish to be involved part of his time in NMR. Therefore PFG NMR, high resolution NMR and operando NMR in this field will be pursued and developed. Currently, operando NMR of pouch cell is fully accessible but only at room temperature, the current probe will be modified to allow variable temperature studies.

The DNP group: Dynamic Nuclear Polariztion and (low temperature) solid-state MAS NMR

Over the last 5 years, the activity of the DNP group focused on the development of Dynamic Nuclear Polarization (DNP) and its application for the study of challenging systems ranging from advanced materials to complex biomolecules. This will continue in the 5 coming years.

Notably, unique instrumental developments have allowed for preliminary tests of DNP under magic angle rotation at temperatures (30 K) lower than what can be reached with a commercial device (~ 105-115K). This proof of concept allowed us to validate the relevance of our approach (improvement of the NMR sensitivity by several orders of magnitude) and motivates the continuation of our effort in the 5 coming years (Coll. Irig/SBT). We are currently working on the development of a completely independent cryostat and a second-generation DNP probe (in collaboration with the world-leader manufacturer BRUKER). Beyond assembling a unique instrument that will strenghten the leadership of the group worldwide, it is also envisioned to take the helium spinning technology to market. This would ensure both a very high visibility and an efficient way to generate incoming funding. Several labs (~10 in the world) have shown a very strong interest to acquire the technology. The next step, to be successful, is conditioned to the hiring of a RM research engineer to interface the instrumental development between the RM group and the SBT lab, and also to enforce the collaboration with Bruker Biospin through a transfer of technology.

Our group has made significant progress in DNP methodology, with the development and use of advanced simulations (spin dynamics) that can be used to relate the properties of the polarizing agents (geometry, EPR parameters, etc.) to the DNP hyper-polarization performances. With this approach, we are in a position to design new polarizing agents in strong collaboration with synthetic chemists from Prof. S. Sigurdson's group (Iceland University). One patent application has been filed in 2018, together with a joint publication.[111] Many more bi-radical structures are currently under development and will undoubtely lead to new polarizing agents tailored to specific conditions (solvent, affinity to a given substrate, temperature, magnetic field, low or high gamma nuclei, etc.). One of the questions that needs be addressed in the coming years relates to bringing these radicals to market, in order to ensure a maximum impact of our work. Several directions are currently under consideration.

With respect to Material Science applications, we will continue to develop projects in tight collaboration with chemistry groups either through local (e.g. Irig/ SyMMES, P. Reiss / D. Aldakov / L. Dubois / F. Duclairoir) or international (e.g. Warsaw University of Technology, Prof. Lewinski) collaborations since we believe that this is the best way to ensure steady progress and to perform state-of-the-art DNP-NMR on advanced materials. The main fields of application of our work will target ZnO NCs, photovoltaic perovskites, natural and synthetic biomaterials (bone/teeth), and graphene-based supercapacitors, among others. Notably, we hope that the access to ultra-low sample spinning temperatures (using cryogenic helium) and faster MAS will enable the continuous and improved study of insensitive nuclei (^{15}N, ^{43}Ca, ^{17}O, ^{67}Zn, etc.) using smaller amount of materials (particularly useful for thin film studies). At this point it is worth pointing out that we are currently trying to secure funding to carry on in this direction. Two ANR projects have been submitted that are currently under evaluation at the second stage. Finally, in the context of a transverse CEA program, we are also starting a collaboration with T. Charpentier (CEA/DRF) and F. Angeli (CEA/DEN) on the detailed study of the surface layer of nuclear waste glasses.

Regarding Biomolecular applications, we plan to pursue the development of the Sel-DNP approach, that we recently introduced in the context of carbohydrate-binding proteins (Coll. CERMAV and DCM, A. Imberty, O. Renaudet), and extend it for the study of challenging protein-ligand or protein-metal interactions (Coll. Irig/LCBM, V. Artero, C. Cavazza, O. Sénèque), possibly under cellular environments (Coll. Irig/IBS, J.-P. Simorre).

111 Mentink-Vigier F., Marin-Montesinos I., Jagtap A. P., Halbritter T., van Tol J., Hediger S., Lee D., Sigurdsson S. T., and De Paepe G. *Journal of the American Chemical Society* 2018 140, 11013-11019

This direction of research will be supported by an ANR project (18 months postdoc secured) and possibly by an ARCANE PhD fellowship (under evaluation). In addition, we also plan to continue in the direction of natural abundance DNP NMR spectroscopy for the study of protein aggregates, possibly derived from animals or patients. Access to ultra-low temperatures and ultra-fast MAS are clearly mandatory to pursue this direction since this would allow sub mg of sample analysis. Several collaborations are envisioned with groups from the Institut François Jacob and/or the Grenoble Institute of Neuroscience.

The RM is also planning to strengthen its collaborations with industrial partners either with Bruker Biospin or with chemical companies (Michelin, BASF, Wacker, etc.). A first contract with Bruker, dealing with the collaborative development of a second generation of MAS-DNP probe, is currently under progress. A second contract is envisioned in the field of frequency-agile microwave source and ultra-fast MAS. Note that the DNP group has also applied for internal support (transverse CEA program Bottom-Up) to help accessing fast MAS technology. A R&D contract with Michelin is almost finalized and should start in 2020. Beyond this type of R&D work, the RM team is also seeking to open the DNP/NMR facility to industrial partners to help secure funding to contribute to the important running and maintenance cost of the facility. Investing further in this strategy is nonetheless very difficult because of the limited human resources of the DNP group (only 3 researchers).

Finally, it is also important to note that the RM team is one of the two main partners of an ambitious regional project that involves most of the NMR players in Auvergne - Rhône-Alpes, "l'Institut des Sciences Analytiques de Lyon" (UMR 5280 CNRS / ENS de Lyon / UCBL), "l'Institut de Biologie des Protéines de Lyon (UMR 5086 CNRS / UCBL)", as well as two laboratories from the Irig Institute, IBS (UMR 5075 CNRS / CEA / UGA) and MEM respectively. The project aims at securing the funding for a liquid/solid SB NMR spectrometer operating at 1.2 GHz/28.2 T (installed in ISA, Lyon), combined with a 800 MHz/527GHz WB DNP NMR system (installed in MEM/Grenoble). These equipments will permit to tackle new types of applications in Structural Biology as well as in Material Science, with a strong impact in medicine, catalysis, energy and environment. About 7 M€ have been secured through regional support and about 15 M€ will be requested to the PIA3 (program "équipements structurants pour la recherche") as soon as the program is launched. The project has the support of CEA and the CNRS.

The hiring of a CEA research engineer is thus strategically important: first to ensure the transfer of technology of the helium DNP technology, to help maintain the NMR instruments of the RM team, and to help secure the access of the NMR and DNP plateforms to a larger number of users (academic and industrial). At the moment the DNP group is organized as follows: GDP leads the group ensuring balanced advances in the three research axes (1. method & instrumentation, 2. material science and 3. biomolecules). DL is in charge of the DNP NMR spectrometers (maintenance and training) with the assistance of SH/GDP and is strongly involved in axes 1 and 2. SH is in charge of the safety and the finances of the group with assistance from DL/GDP, and is strongly involved in axes 1 and 3. The group has a concerted strategy regarding funding applications for which all group members are involved.

Relaxometry

The local ecosystem in medecine as well as the recognition of its usefulness for CEA missions will be decisive for the development of the relaxometry techniques for which the Grenoble site is unique because of its complementary NMR facilities allowing one to explore molecular dynamics over a very broad range of frequencies from a few tens of Hz to about 1.4 GHz.

Operational organisation chart

DNP team: G. de Paëpe, S. Hediger and D. Lee
NMR service: P. A Bayle
NMR team: M. Bardet, M. Gromova, & P.A Bayle
Relaxometry team: P. H. Fries

Equipment, platforms

NMR equipment at RM:

DNP equipment:
- 400 MHz solid-state NMR with DNP (cryo, DNP & console 2011, Minatec campus)
  Ultra-low temperature instrumentation
- 400 MHz solid-state NMR with DNP (magnet 2005, console & DNP 2017, Minatec campus)
  New console, new DNP ERC GDP

NMR Service:
- 500 MHz liquid-/solid-state NMR (2013, C-5 building)
  HRMAS & CPMAS probes
- 400 MHz liquid-state NMR (2013, C-5 building)
- 200 MHz solid-state NMR (magnet 1979, console 1999, C-5 building)
  High power PFG NMR and operando NMR

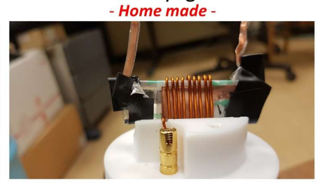
Relaxometry equipement:
- Relaxometer (2004, C-5 building)
- New relaxometer (2018 C-5 building)
  Home made prototype


DNP equipment


ULTMAS with cryogenic helium
- Home made -


NMR facility


FFC-NNR relaxometer
- Home made -


Pouch cell & coil for operando NMR
- Home made -

Research products and activities of the RM team

Articles

Scientific articles (total 53)

20% most significant articles

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Books

Book chapters (5)

54. MAS-DNP Enhancements: Hyperpolarization, Depolarization and Absolute Sensitivity. Hediger S., Lee D., Mentink-Vigier F., De Paëpe G. eMagRes, 2018 - Volume 7, Issue 4

55. NMR Studies of Fossilized Wood. Bardet M., and Pournou A.. in: G.A. Webb, (Ed.), Annual Reports on Nmr Spectroscopy, Vol 90, 2017, pp. 41-83.

56. High-Field Solid-State NMR with Dynamic Nuclear Polarization. Lee D. , Hediger S., De Paëpe G. In: Webb G. (eds) Modern Magnetic Resonance. Springer,Modern Magnetic Resonance, 2017, 1-17

57. In Situ Studies of Plant Seeds Using 13C or 1H MAS NMR and 1H PFG NMR Approaches. Gromova M., Guillermo A., Bayle PA., Bardet M.. In: Webb G. (eds) Modern Magnetic Resonance. Springer, 2017, 1519-1534 DOI: 10.1007/978-3-319-28275-6_18-1

58. Third Spin-Assisted Recoupling in SSNMR: Theoretical Insights and Practicable Application to Biomolecular Structure Determination Paul S., Takahashi H., Hediger S., and de Paepe G. in: G.A. Webb, (Ed.), Annual Reports on Nmr Spectroscopy, Vol 85, 2015, pp. 93-142. DOI:10.1016/bs.arnmr.2014.12.003

Book chapters in English or another foreign language (5)

Edited theses (2)

1- Atomic-level structure determination of organic assemblies by dynamic nuclear polarization enhanced solid-state NMR, Thesis presented Decembre 19, 2017 by Katharina Märker

2- Atomic-level characterization of nano- and micro-structured porous materials by NMR: Pushing the frontiers of sensitivity, Thesis presented November 25, 2015 by Tuan Nghia Duong.

Production in conferences / congresses and research seminars

Meeting abstracts

For SH/DL/GDP (DNP group)

- about 36 talks (with abstract) were given in international conference
- about 15 invited seminars (with abstract) were given
- participation through lectures and laboratory hand on practice to 4 winter/summer schools

In addition, we estimate the number of abstract (talk or poster) from the PhDs/postdocs of the group to be around 5/years.

Instruments and methodology

Prototypes

1. *Helium spinning MAS DNP instrumentation (probe + Cryostat) (see ERC project, G. De Paëpe)*
2. *Ultra low field relaxometer (see EU project, P. H. Fries)*

Platforms and observatories

(DNP-enhanced) solid-state NMR, solution NMR and relaxometer platform/facilities: Access are mainly through collaborative projects or as autonomous users, depending on the types of research/analysis demands and the required equipments.

Editorial activities

Participation in editorial committees (books, collections, etc.)

Archaeology Section Editor of Modern Magnetic Resonance Springer 2017 Graham A. Webb Editor

Reviewing activities

Reviewing of articles

Grant evaluation (public or charities)

For the RM team, we have estimated our contribution to peer reviewing process to:
- 72 for scientific articles
- 12 for research projects

Member of the Scientific Advisory Board of the Labex ARCANE (2012-now)

Participation in institutional committees and juries (CNRS, Inserm, etc.)

Jury of the CNRS concours for research engineer position in 2018 (S. Hediger)
UGA selection committee for MCF recruitments since 2014 (M. Gromova)

Academic research grants

European (ERC, H2020, etc.) and international (NSF, JSPS, NIH, World Bank, FAO, etc.) grants - coordination

1 ERC Cog ULTMASDNP
1 IEF BOLDNMR

Other European grants – partnership

Cofund NaMeS

National public grants (ANR, PHRC, FUI, INCA, etc.) – coordination

1 ANR CryoMAS4DNP
2 PhD Phare CEA

National public grants (ANR, PHRC, FUI, INCA, etc.) – partnership

1 ANR TransPepNMR

PIA (labex, equipex etc.) grants – coordination

3 Postdoc grants (Fernandez-de-Alba / Paul / Montesinos)
1/2 PhD grant

Visiting senior scientists and post-doc

Foreign visiting scientists

Anton Buzlukov from the Institute of Metal Physics Ural Branch of the Russian Academy of Sciences, 620990 Ekaterinburg Russia about 12 months over the period.

Scientific recognition

Chair of learned and scientific societies

2017- Member of the ISMAR council (International Society of Magnetic Resonance)
2016 - Admitted to the « Young Academia of Europe » http://yacadeuro.org/#sthash.zjMUJAW1.dpbs

Organisations of meetings and symposia (international)

1) Alpine Conference on Solid-state NMR (Chamonix) 2015 and 2017
2) Euromar (Nantes) 2018
3) Workshop UCSB – Grenoble 2019, « Functional materials and advanced spectroscopies »
4) International workshop " NMR Relaxometry for Interdisciplinary and Industrial Applications " (European COST Action EURELAX), CEA-Grenoble 19-22 mars 2019

Invitations to meetings and symposia (out of France)

G. De Paëpe:
- ATUMS Symposium on Functional hybrid material and Meeting program on occasion of 150th Anniversary of TUM, TUM (November 2018)
- Nuclear Magnetic Resonance in Chemistry, Physics and Biological Sciences, Warsaw (September 2018)
- HYP18, an international conference on Nuclear hyperpolarization, Southampton (September 2018)
- The 101st Canadian Chemistry Conference and Exhibition (CSC2018), Edmonton (May 2018)
- DFG-DNP workshop, Bonn (April 2018)
- ACS National Meeting, New Orleans (March 18-22, 2018)
- ICRIS-NMR '17: DNP-NMR Workshop (Kyoto, October 2017)
- FGMR (French-German Magnetic Resonance) annual discussion meeting (Bayreuth, September 2017)
- Telluride Workshop on DNP and paramagnetic NMR (Telluride, August 2017)
- BRUKER Pre-EUROMAR symposium (Warsaw, July 2017)
- EUROMAR (A European Magnetic Resonance Meeting) (Warsaw, July 2017)
- Developments and Applications of Solid-State NMR to Materials Science, Chemistry and Engineering Conference (Zing conference, Varna, Bulgaria, May 14-17, 2016)
- The 58th Rocky Mountain Conference on Magnetic Resonance (RMCMR) (Breckenridge, July 17-21 2016)
- The 57th Experimental Nuclear Magnetic Conference (ENC) (Pittsburgh, April 10 - 15, 2016)
- The 9th Alpine Conference on Solid-State NMR, Chamonix, France, Sept., 2015
- International Society of Magnetic Resonance (ISMAR), Shanghai, China, August, 2015
- ICRIS-NMR '14: Technological Frontiers in Solid-State NMR, Kyoto, Japon, October 2-4, 2014
- The 55th Experimental Nuclear Magnetic Conference (ENC), Boston, March 23 - 28, 2014
- BRUKER Pre-ENC symposium, Boston, March 23, 2014

S. Hediger:
- Structure and Interaction of the Bacterial Cell Wall by Solid-State NMR and DNP, Invited Seminar at Department of Chemistry, Umea University, Sweden, February 4, 2014

Members' long-term visits abroad

M. Bardet joined Emsley 's laboratory at EPFL from15/10/2017 to 30/09/2018 (Academic Guest: outgoing CEA fellowship from the CEA-Enhanced Eurotalents program, co-funded by FP7 Marie-Skłodowska-Curie COFUND program, Grant Agreement 600382).

Interaction of the RM team with the non-academic world, impacts on economy, society, culture or health

1- Socio-economic interactions / Patents

Filed patents

1 patent was filed on polarizing agents for DNP

2- Socio-economic interactions

Industrial and R&D contracts

One R&D contract with Bruker Biospin (3 years)

Several short contracts with industrials (Michelin/BASF/Wacker), one week each, 5 weeks in total

Involvement of the RM team in training through research

Scientific productions (articles, books, etc.) from theses

Scientific productions (articles, books, etc.) from theses

For the DNP group, we have estimated the number of articles related to PhD projects to 6, with 3 articles per PhD in average.

Training

Habilitated (HDR) scientists

HDR obtained during the period

G. de Paepe

PhD students (6)

DNP group: 6

Defended PhDs

DNP group: 2 (N. Duong, K. Märker)

Mean PhD duration

37 months

Internships (M1, M2)

TEAM 6: SERVICE GENERAL DE RAYONS X (SGX)

Presentation of the unit

Introduction

The SGX laboratory is in charge of the IRIG X-ray characterization tools. The main task of the laboratory is to make available to the IRIG teams up-to-date X-ray scattering instruments, providing expertise in data collection and analysis. It runs 4 diffractometers (single-crystal, powder, standard thin film, high-resolution and in-plane thin film) and one small angle scattering instrument. One Laue camera is also part of the instrumental park but is installed in a different building to meet the specific needs of the PHELIQS-IMAPEC team that operates it. The own research activity of the team, performed in collaboration, is grafted on this main task. Research activities are mainly focused on functional materials for applications in the field of new energy technologies (organo-halide perovskites for photovoltaic applications) and epitaxial nanostructures for nanoelectronics and spintronics.

Unit's workforce and means

Permanent staff (01/2019)	Non-permanent
E. Bellet-Amalric, CEA researcher (1/2 MEM-SGX / 1/2 PHELIQS-NPSC) A. de Geyer, CEA engineer A. Farchi, CEA engineer S. Lequien, CEA researcher B. Mongellaz, CEA technician S.Pouget, CEA researcher	**Co-supervised PhD students** (affiliated at SGX) D. Zapata Dominguez (2017 -) (SYMMES/STEP) **Co-supervised PhD students** (not affiliated at SGX) A. Medjahed (2017 -) (SYMMES/STEP) [P. Dally (2016 -) (*LITEN/DTS/LMPO)- effective co-supervision*]

The laboratory operating budget is of about 40 k€. The part of a recurrent budget coming from the CEA in the funding of the research activities being zero today, the mode of financing of a common service laboratory is not self-evident. Creating a pool of resources from individual and hard to obtain financing is not easy and generates tensions. We finally opted for the following mode of operation: contributions are requested from the teams at the end of the year, in proportion to their use; they are strongly encouraged to include an X-ray participation in their projects funding. The question of financing investments, which is essential to maintain a relevant and up-to-date instrumental park, is also complex. During the period 2016-2019 two instruments have been replaced. The purchase of the SmartLab thin film diffractometer for high-resolution and in-plane measurements benefited from tri-partite funding from Labex LANEF, INAC and Institut Néel. The acquisition of the D8 powder diffractometer was made as part of the DRF's investments related to the creation of IRIG.

Scientific policy

As already mentioned, the main mission of the laboratory is to make available to the IRIG teams up-to-date X-ray scattering instruments, providing expertise in data collection and analysis.

However in the last years the part of the research activity has increased, on the one hand resulting of ongoing collaborations that have strengthened over time, and on the other hand in connection with the strategy of the INAC institute and MEM service to put forward advanced characterization in the field of batteries.

It is important to emphasize that research, instrumental and / or methodological developments feed one another.

Student training has always represented an important part of the team's activity, in various forms: supervision of PhD students, Master courses given at the university, trainings in X-ray techniques regularly organized in the laboratory and recognized by the physics and chemistry doctoral schools, tutorials and practicals in the frame of the HERCULES school.

Presentation of the unit's research ecosystem

In January 2019, SGX became the X-ray service laboratory of IRIG, grouping of INAC, IBS and BIG. IBS has significant needs in X-ray characterization, covered by regular access to several beamlines at ESRF. The situation of the laboratories that were attached to BIG is different and very variable according to the groups. The institute did not have laboratory X-ray characterization facilities; but links with the SGX pre-existed the creation of the IRIG around the single crystal diffractometer, which has been co-managed by the LCBM and our laboratory

since 2017. Moreover, several teams are interested in the possibility of carrying out small angle scattering experiments using our laboratory instrument. If this need is confirmed, the instrumental park of the laboratory will have to evolve accordingly. Indeed the current "small angle" instrument is over 20 years old; it has been the subject of several instrumental developments but failures are becoming more frequent and the instrument will be obsolete in the short term. The question of human resources will also be acute.

The SGX has developed links with the CNRS/Institut Néel X-ray laboratory, particularly in the context of the Labex LANEF PHENIX project for the acquisition of the high-resolution/In Plane thin film diffractometer. It is a will of the laboratory to continue and amplify this collaboration with the perspective of a complementary evolution of our respective instrumental parks.

An X-ray characterization laboratory in Grenoble can hardly be considered without interaction with large scale facilities. Several team members have worked at one point or another in their career at the ILL , and are now regular users of neutron and synchrotron sources for their research activities.

Research products and activities for the unit

Scientific activity: The main mission of the laboratory is the service activity. With this respect, an activity report is published and distributed in the institute every two years. As concern the team research activities the SGX laboratory has for a long time worked closely with the institute's laboratories specialized in epitaxial synthesis of metals and semiconductors in various geometries (2D, 1D, 0D), in the fields of nanoelectronics and spintronics PHELIQS and SPINTEC. Moreover, for the last three years, the involvement of the laboratory in the field of functional materials for energy has been reinforced, particularly on hybrid perovskite materials for photovoltaic applications, and on silicon-based composite materials for Li-ion battery electrodes, both subject in collaboration with the SYMMES/STEP laboratory.

Scientific highlights

H1: Ferroelastic behavior of strained $CH_3NH_3PbI_3$ films synthesized by ion-exchange conversion from $CH_3NH_3PbCl_3$

In the last decade organo-lead halide perovskites (OLHP) have attracted extraordinary attention on account of the rapid rise in their solar-to-electricity conversion efficiency and more generally of their potential optoelectronic applications due to their outstanding properties including direct and tunable bandgaps, high electron/hole mobilities, strong light absorption coefficients and high defect tolerance coupled with low non-radiative recombination rates. They also appear to be easy to fabricate, even by room-temperature deposition processes for thin films, displaying a combination of soft chemical/mechanical character (low formation energy and stability, low mechanical stiffness and large thermal expansion coefficients). The soft nature of these materials such as low formation energy, low Young's modulus and high thermal coefficients make them easy to fabricate and also gives opportunities for manipulating film formation, tailoring film growth. $CH_3NH_3PbI_3$ (denoted MAPbI3 in the following) can be considered as the reference material in perovskite solar cells, due to excellent optical absorption properties with a band gap of 1.55 eV, remarkable carrier lifetime and balanced carrier diffusion length. However, mastering the synthesis and the crystalline quality of MAPbI3 thin films has proved to be a real challenge due to the weak stability of the phase and to the high sensitivity to the different synthesis parameters.

As part of a research activity conducted in collaboration with the SyMMES / STEP and LITEN / DTS / LMPO laboratories, we undertook a detailed study of the crystallization process and microstructure of MAPbI3 films obtained by spin coating a solution of MAI and $PbCl_2$ (3:1) in DMF.

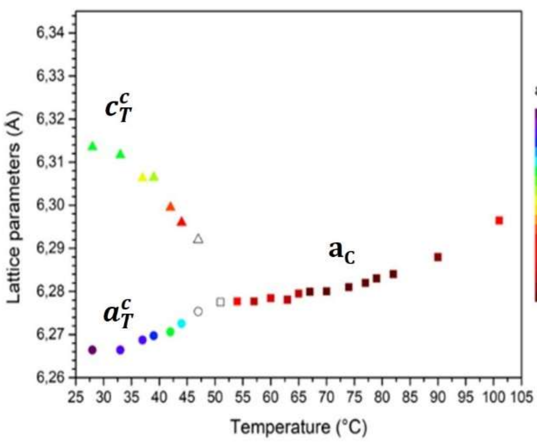

Fig1: Thermal evolution of the MAPbI3 lattice parameters. Below 55°C which corresponds to the tetragonal-cubic phase transition, the triangles and the circles represent the perpendicular pseudo-cubic lattice parameters of the [00l] and [hh0] oriented twins respectively. Above 55°C the squares correspond to the perpendicular cubic lattice parameters. The degree of strain is indicated by the color. It is calculated taking a MAPbI3 powder sample as a bulk reference (transparent patterns correspond to temperatures for which powder MAPbI3 lattice parameter values were not available).

Similar to different perovskite compounds MAPbI3 exhibits multiple crystalline phases: cubic with a Pm-3m space group above 327 K, tetragonal elongated along the c axis and alternating in-plane rotations of the PbI6 octahedra between 162 K and 327 K, and orthorhombic with the Pnma space group below 162 K. The diffractograms obtained with the perovskite films are characteristic of the presence of a tetragonal MAPbI3 phase with a double texture [hh0] / [00h]. This apparent double texture is related to the observation, by Transmission Electron Microscopy, of twins in $MAPbI_3$ films, associated with ferroelectric properties. The diffraction data also indicate coexistence with a [h00] textured cubic MAPbCl3 phase. Temperature-dependent measurements have been performed to determine the thermal behaviour of the lattice parameters. The tetragonal cell is defined such that its a edge is the diagonal of the (a, b) face of the cubic cell and it is doubled along the [001] direction. Consequently, to facilitate comparison between cubic and tetragonal phases, pseudo-cubic lattice parameters a_T^c and c_T^c were defined from the tetragonal a_T, c_T lattice parameters as follows: $a_T^c = a_T/\sqrt{2}$ and $c_T^C = c_T/2$. Fig1 presents the thermal variation of the perpendicular lattice parameters.

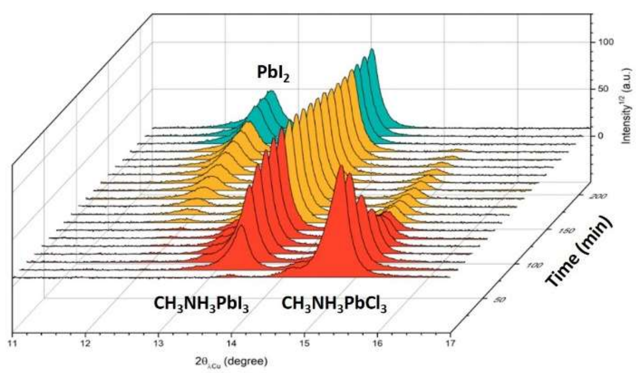

Fig2: Evolution of the XRD pattern during the annealing at 100°C. The different colors represent the different stages of the MAPbI3 formation process (red: coexistence of MAPbI3 and $MAPbCl_3$; orange: coexistence of the two perovskite phases with PbI_2; green: coexistence of MAPbI3 and PbI_2.

Considering the data obtained with a sample of powder that can be considered as "bulk reference", the MAPbI3 layer appears to be highly strained, and this for the two twin populations. In order to understand the origin of this strained state, we carried out an in situ study, recording the diffraction signal during the annealing and thus along the crystallization process of the MAPbI3 phase (Fig2). It appears that after the solution spin coating at room temperature only the $MAPbCl_3$ phase is present. At 100 ° C, the iodide perovskite is formed by ionic substitution in a strained state that amplifies until the appearance of PbI_2 which is known to result from the decomposition of MAPbI3. The MAPbI3 layer reaches a completely relaxed state at the complete disappearance of the chlorine phase, as shown in Fig3.

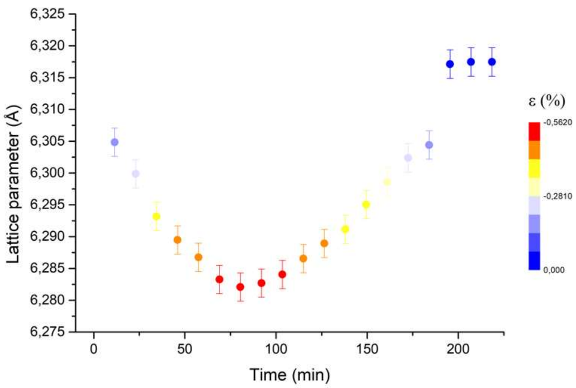

Fig3: Evolution of the $MAPbI_3$ cubic lattice parameter at 100°C along the annealing process. The colors indicate the degree of strain which is maximum before PbI_2 appears. The layer is fully relaxed when the chlorine perovskite phase has disappeared.

The comparison with less strained layers obtained by different synthesis protocols allowed us to correlate the presence of the twins with the degree of strain of the layer.

Two publications are in preparation.

H2: Silicon based anodes for Li-ion batteries

In the search for high performance electrode material for energy storage, silicon has attracted considerable attention due to its large capacity (3579 mAh/g) almost ten times higher than for the commercially employed graphite (372 mAh/g). However, the alloying process between lithium and silicon yields important volume changes on cycling (typically 280% for bulk silicon) and subsequent growth of an unstable Solid Electrolyte Interphase (SEI) upon cycling, leading to a rapid loss of performance. Different strategies can be considered to improve the stability. One way of limiting volume expansion is using silicon nanoparticles, where the small size enables Li transportation and strain relaxation. The nanoparticles can also be embedded in a matrix, thus forming a composite material. This type of material displays better behavior with respect to the aging effect: less capacity loss due to reduced SEI formation. Another possible solution is the insertion of a different element in the silicon structure. Germanium, for instance, can improve the electronic and ionic conductivity, allowing better lithium diffusion and improving the performance.

SiGe alloys for lithium-ion batteries

Si_xGe_{1-x} alloys have shown to improve the performance during cycling compared with Si-rich electrodes. In the frame of a collaboration with CEA Paris Saclay/IRAMIS/NIMBE, 3 different $Si_xGe_{(1-x)}$ nanoparticles (NPS) alloys were synthesized: $Si_{0.3}Ge_{0.7}$, $Si_{0.7}Ge_{0.3}$, and $Si_{0.44}Ge_{0.56}$ by laser pyrolysis. This technique consists in using a laser beam for decomposing silane and/or germane to obtain the NPs in the range of 20 – 100 nm (Fig4a). For the three compounds, the X-ray diffraction data display diffraction peaks characteristic of the expected diamond structure but the peak shapes indicate the presence of inhomogeneities in $Si_xGe_{(1-x)}$ composition.

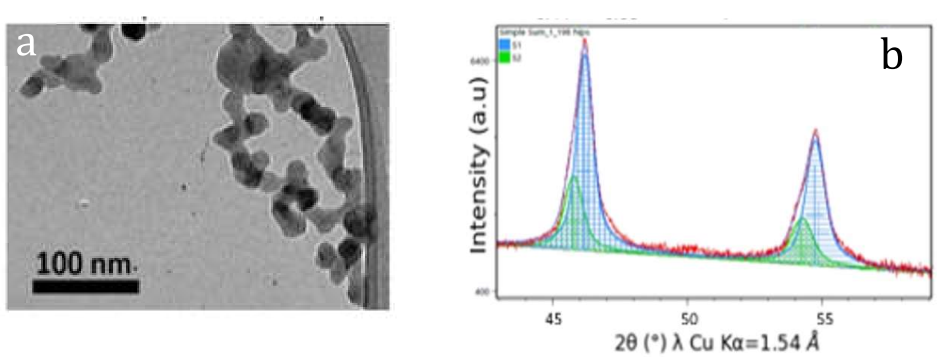

Fig4: (a) STEM picture of Si Nps ; (b) LeBail profile refinement of θ/2θ X-ray diffraction data for the $Si_{0,44}Ge_{0,56}$ NPs compound.

Data were analyzed by LeBail profile refinement, assuming the presence of diamond type crystalline phases with different lattice parameters to account for the chemical composition dispersity. In the case of the $Si_{0.44}Ge_{0.56}$ compound, two different phases were considered (Fig4b). The fitted values of the lattice parameter corresponds to silicon contents x=0.23 and x=0.44. Considering the differences in peak integrated intensities corrected from the different electronic densities, the relative amounts of the two crystalline phases (x=0.23 and x=0.44) can be estimated and are of the order of 1:3. The germanium rich $Si_{0.3}Ge_{0.7}$ compound reveals stronger composition heterogeneities, while the silicon rich one displays a weaker crystallinity.

In order to investigate the (de)alloying properties of this compound, operando synchrotron WAXS measurements were performed on $Si_{53}Ge_{47}$ based electrodes in coin cells vs lithium. The cell was cycled at a rate of C/20 (I =-80 µA) during 8 h. Then the current was increased up to 240 µA to accelerate the lithiation process. The diffraction patterns composed of Bragg refflections from the battery components (Cu and Al current collectors and Si-Ge particles) were measured every two minutes. Fig5a shows the decrease of the Si-Ge (111) peak intensity upon time during the first lithiation, reflecting the continuous amorphization of crystalline particles due to the alloying process. Along the lithiation process the diffraction peak position displays a shift towards larger Q values. This might, at least partly, result from the sequential lithiation of the different $Si_{100-x}Ge_x$ phases depending on the Ge content. Indeed considering the values of the lithiation potential E vs Li+/Li0 for Ge and Si (resp. 380 mV and 250 mV), Ge rich phases are expected to lithiate first.

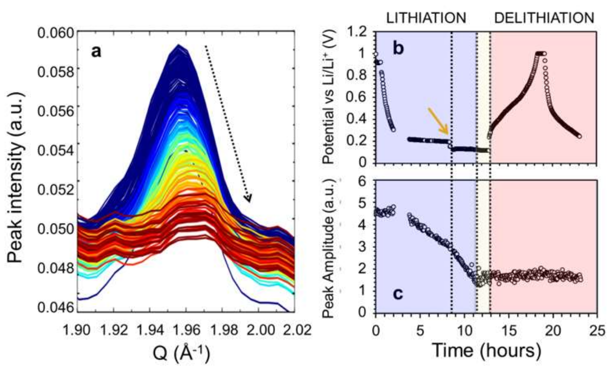

Fig5: a) Si-Ge [111] peak intensity variations measured by operando synchrotron XRD during the first lithiation. The peak decreases in intensity (from blue to red) indicating the continuous amorphization of the active particles. b) Voltage against time, with a change in current at ca. 8 hours, indicated by the arrow, and a floating period at the end of the lithiation, materialized by the yellow shadowed area. c) Peak amplitude obtained by a Gaussian fit to the XRD data, as a function of time.

Delithiation kinetics in an industrial composite for lithium-ion batteries

The aim is to understand the lithiation mechanism in a complex industrial composite graphite/ active amorphous Si + inactive $FeSi_2$ (hereafter denoted Sintbat). With this purpose, its behavior is characterized and compared to the one of pure graphite on the one hand and also to the one of a model composite made of crystalline Si Nps and graphite on the other hand. Coin cells were prepared using the three different electrode compounds. Subsequently, one complete cycle and one lithiation between 1 V – 10 mV were performed (Fig6). The cells were then opened in the glovebox and the X-ray diffraction samples were prepared using the sample holder shown in Fig6b sealed with two Kapton foils.

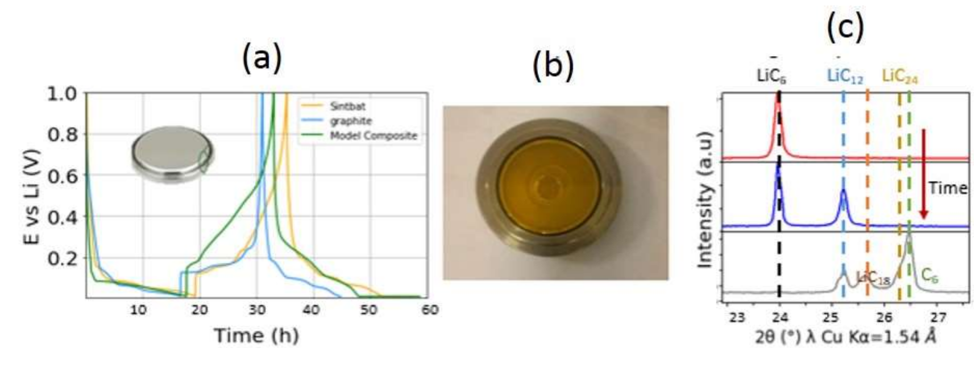

Fig6 – (a) Cycling electrochemical data – (b) Sample holder used for XRD measurements – (c) Evolution of the LiCx diffraction peaks along the graphite delithiation process.

The measurements were made using the laboratory powder diffractometer. Scattering angles 22 – 30° were collected in 0.02° steps with count time of 40 s per step to follow the kinetics of the LiC_X phases' formation until the graphite peak appears at 26.5° (Fig6c). Fig7 displays the delithiation kinetics determined from the evolution of the area of the LiC_6 and LiC_{12} (002) diffraction peaks, for the three types of electrodes. Clearly the pure graphite delithiation is much slower than the one observed in the presence of silicon reflecting interactions between the silicon and graphite lithiation processes. Nevertheless, the spontaneous pure graphite delithiation indicates residual influence of the cell which is not fully airtight. These investigations will benefit from the acquisition of a LE. he's battery cell.

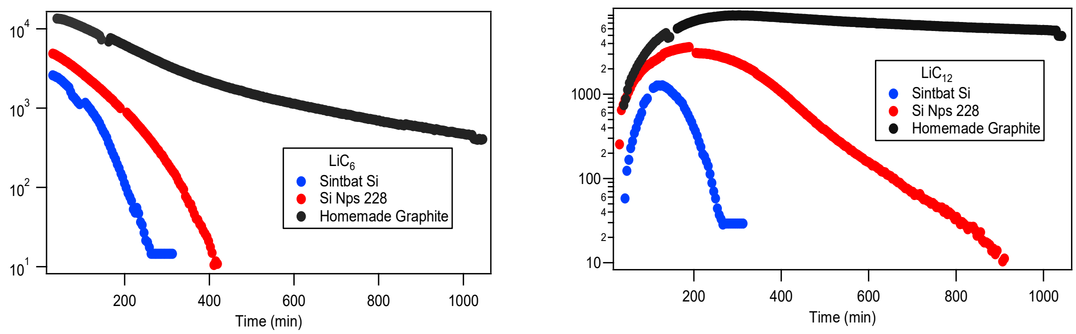

Fig7- Graphite delithiation kinetics: evolution of the LiC_6 and LiC_{12} (002) diffraction peaks areas as a function of time (t=0 corresponds to the exit of the cell from the glove box).

This activity is driven in collaboration with the MEM/NRS and SyMMES/STEP laboratories. Several publications are in preparation.

H3: Near- and mid-infrared intersubband absorption Near- and mid-infrared intersubband absorption

Engineering intersubband transitions between confined states in the conduction band have evolved into a promising alternative for the realization of infrared (IR) optoelectronic devices. Initially, intersubband technologies developed in the mid-IR using GaAs-based materials. Group-III nitrides (GaN/AlN) offer the possibility to extend the operating range of these devices into the near-IR spectral range, including the 1.3–1.55 µm wavelength window of fiber-optic telecommunications. Furthermore, it opens prospects for room-temperature operation of devices (photodetectors, quantum cascade lasers) designed for THz frequencies. For these devices the intersuband active media consists of several tens of periods of GaN/AlGaN (of a few nm thick) quantum wells. The control of the structural quality of the samples is a key parameter for the performance of the devices.

In the near-IR, the use of nanowires (bottom-up) present interesting advantages to reduce the amount of active material, and therefore the electrical capacitance of the device, without modification of the optical cross section of the device. However, structural characterization of the nanowire ensemble is challenging due to the dispersion of geometries, tilt and twist (Fig 8)

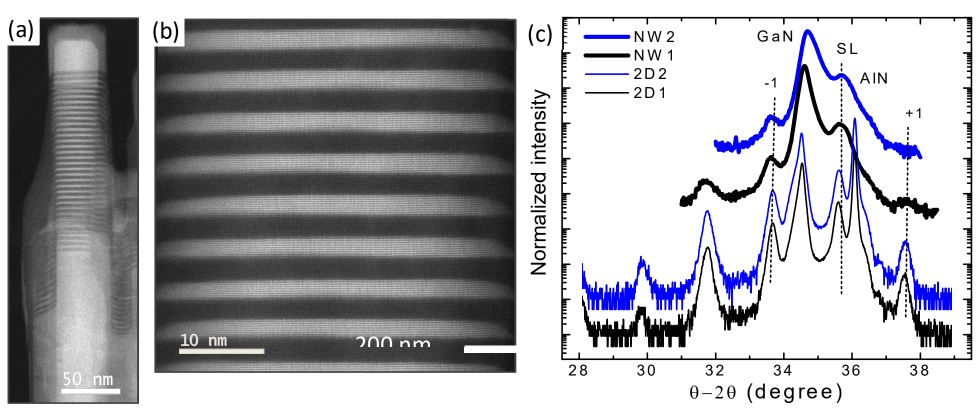

Fig8: a) and b) HAADF-STEM images of the active region of a NW for ISB device, c) HRXRD reflection and simulation of the (0002) of a planar and a NW sample.

On the other hand, operation at longer wavelengths (THz) requires reducing the internal electric field induced by spontaneous polarization in III-nitrides. With this purpose, we considered the use of nonpolar crystallographic orientations (*a*-plane and *m*-plane) grown on free-standing nonpolar GaN substrates. Here, advanced device design requires precise knowledge of the strain state of the layers, which is a challenge in these highly anisotropic crystals (Fig9).

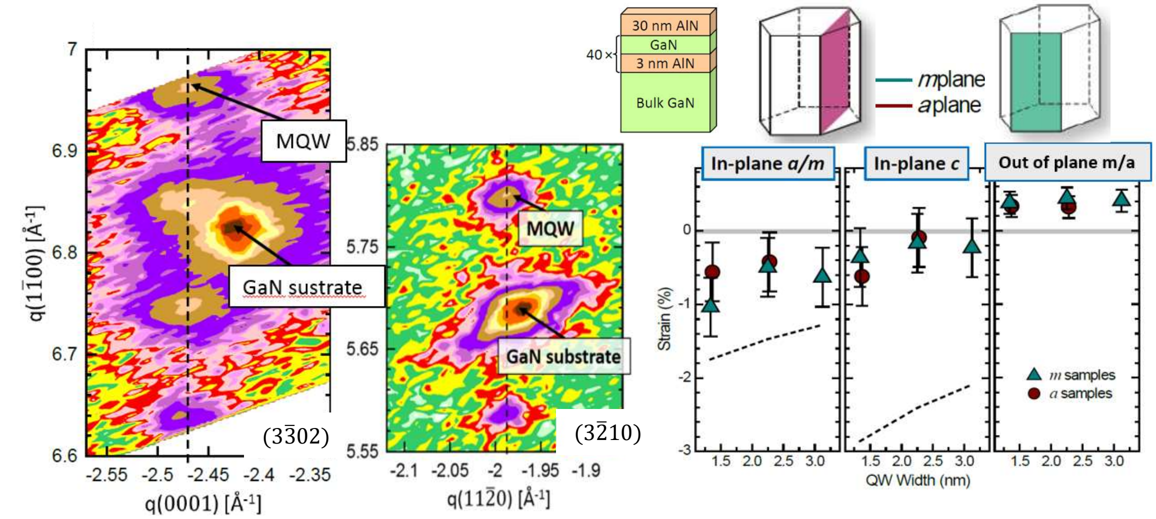

Fig9: Measurement of the stain state of *m*-plane and *a*-plane samples: (left) reciprocal space maps and (right): strain state extracted from the XRD measurements

The Rigaku SmartLab X-Ray diffractometer in a high-resolution (HR) configuration with a 4 bounce Ge(220) monochromator and a 0.114° collimator in front of the detector is a key tool for the developement of this research field. The determination of the strain state of the superlattice (in the 2 in plane directions and for the resulting out of plane direction) is obtained with the reciprocal space maps around asymmetrical reflections, while the layer thickness and concentration are given by the simulations of the superlattice satellites in the symmetrical diffractogram.

This subject, and more generally the improvement of group-III nitrides, is a field conducted for many years in close collaboration with the PHELIQS/NPSC team. For the non-polar structures [Lim et al. ***J. Appl. Phys***. *118, 014309 (2015) and* **Nanotechnology** 27 (2016) 145201] we have demonstrated that the intersubband transitions in the *m*-plane structures can cover the near, mid- and far-IR regions.

Instrumental highlights

H4: In-Plane/Out-of-Plane diffractometer (Smartlab Rigaku)

Late 2015, we welcomed a Smartlab X-ray diffractometer, by Rigaku, hosting a powerful x-ray source and offering a high measurement versatility adapted to the diversity of samples and problems specific to crystalline layers of high structural quality and even nanocrystalline objects. This project is jointly supported by CEA-INACand CNRS-Institut Néel with a co-financing of LABEX LANEF, and jointly managed by the 2 institutes.

The main charactE. s of this equipment are: i) a 9kW rotating Cu anode with high flux, ii) a versatil diffraction geometry adaptable to sample morphologies, in particular for thin fims, with precise alignments of the diffraction vector either normal to the surface of the sample or in the plane of the sample (Fig. 10), iii) the ability to completely explore the reciprocal space, the detector having two independent movements, all angles guided by a powerful simulation and all-automated control software, Iv) a modular design which enables powder diffraction, medium or high resolution diffraction, v) an oven reaching 1100°C (by Anton Paar).

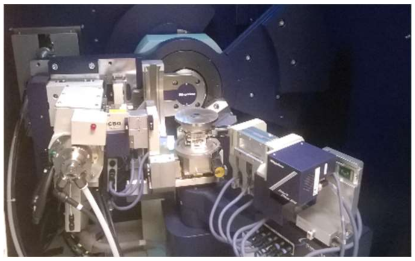

Fig10: The equipment in the in-plane configuration mode

Thanks to its large versatility, the diffractometer can be used for a broad variety of materials such as:

- Nanostructures of nitrides semiconductors (c and m-plane growth, planar or nanowire geometry) for inter-band (UV) and intra-band (THz) emission: structural quality, strain and composition, using the high-resolution mode [112].

- Organic nanometer-thick layers with very high conductivity for thermoelectric applications: in-plane and out ofplane orientation of the PEDOT chains according to the type o fdoping molecules [113].

- Hybrid perovskite films for photovoltaic applications: crystal structure and microstructure (orientation, in-plane and out-of plane domain size).

- Transition metal dichalcogenides, a new class of semiconducting two-dimensional materials, one to a few monolayers in thickness: crystal orientation, strain and grain size thanks to the in-plane measurements (Fig. 11).114

- VO2, dependence of the electric properties of thin films on substrates, growth conditions, microstructure orientation and quality: reciprocal space mapping, in-plane measurements, dependence with temperature.

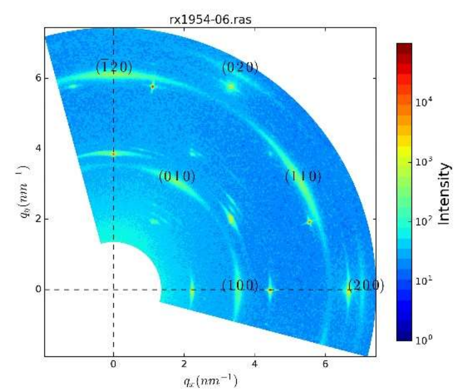

Fig 11: In plane reciprocal space map of 1.5 monolayer thick WSe_2 (indexed) epitaxially grown on mica

H5: New powder diffractometer (Bruker D8 Discover)

In December 2018 a new Bruker D8 Discover powder diffractometer was installed in the laboratory (Fig12a). It replaces the former Panalytical X'Pert, which was over 20 years old. Like its predecessor the instrument is equipped with a copper tube, programmable divergence slots and a sample changer with 15 positions. New configurations are available: i) a focusing mirror makes it possible to measure under optimal conditions in transmission mode, for example for the study of membranes, or in capillaries ii)the removal of the Kβ contribution is **efficiently** performed ($I_{K\beta}/I_{K\alpha1}$ < 0.5%) by the energy discrimination of the Lynxeye XE-T detector, thus avoiding

112 Effect of Ge-doping on the short-wave, mid- and far-infrared intersubband transitions in GaN/AlGaN heterostructures", Semicond. Sci. Technol. 32, 125002 (2017).

113 Structure and Dopant Engineering in PEDOT Thin Films: Practical Tools for a Dramatic Conductivity Enhancement, Chem. Mater. 28, 3462 (2016).

114 Millimeter-scale layered MoSe2 grown on sapphire and evidence for negative magnetoresistance, Appl. Phys. Lett. 110, 01 (2017).

the threshold effect on the shape of the diffraction peaks. iii) a motorized anti-scattering screen minimizes background at small angles.iv) a TTK600 Anton Paar temperature stage covering a temperature range from 85 K to 600 ° C (Fig12b), v) a LE. he'S battery cell for operando measurements.

This state-of-the-art instrument makes it possible to study a wide variety of materials (semiconductor nanocrystals for energy and photonics, hybrid Perovskite materials for photovoltaics, protonic conductive membranes, electrode materials for batteries, etc.).

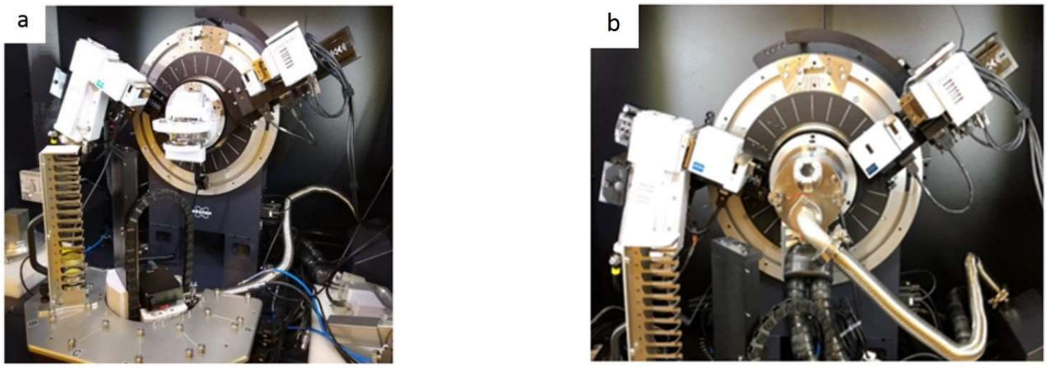

Fig12: Bruker D8 Discover diffractometer equipped with a 15-position sample changer (a) and a TTK600 Anton Paar temperature stage (b).

H6: SAXS/GISAXS laboratory setup

A long collimation SAXS laboratory setup is available for all IRIG teams. This home-built SAXS line is equipped with a large 2D gas detector (14 cm x 14 cm) and the sample-to-detector distance can be tuned from 20cm up to 4 m. Its momentum transfer range (2 A^{-1}<Q <10^{-3} A^{-1}) enables characterization of nanostructures from some nanometers to some hundreds of nanometers. The large sample chamber can accommodate various sample environments such as temperature and/or humidity stages. This makes it a precious tool in complementarity to large scale facilities measurements (synchrotron SAXS, SANS) allowing for instance long in-situ studies.

This lab-line has been widely used for investigating the structural properties of conductive (protonic or ionic) polymers, the organization in block copolymer and self-assembled structures of ionic liquid crystals with the aim of correlating it with the conduction properties. Its wide accessible Q range makes it well suited for the investigation of liquid-phase synthesis crystallization processes. A developing field is the study of electrode materials for energy storage, the SAXS technique giving access, among other structural parameters, to the porosity which is a key parameter.

In 2018 and early 2019 a GISAXS configuration was developed and installed on the instrument, for the study of thin films and interfaces. The GISAXS technique consists in measuring both the specular and off-specular reflectivities with a 2D detector. The incident angle of the X-ray beam on the film is kept constant at a value close to the critical angle. The equivalent of the SAXS signal is then obtained for the thin layer, giving access to the distribution of electron density inhomogeneities parallel and perpendicular to the plane of the film.

This GiSAXS setup has recently been used to characterize self-assembled ionic liquid crystal thin films (materials synthesized by DRT/LITEN and SyMMES). The ionic liquid crystals are designed to encode single-ion conduction properties into a variety of mesomorphous phases (for instance, lamellar and columnar geometries). They are considered as model systems for lithium-conducting and proton-conducting solid electrolytes of interest in energy conversion and storage applications. The investigation of the film self-organization is essential, to correlate the structure with the conducting properties (Fig13).

Polymer films of a thickness of the order of a few tens of nanometers can also be studied by GiSAXS on this lab-line, benefiting from the large accessible Q-range. To take full advantage of this new configuration, a sample temperature stage (-196°C < T < 400°C) with controlled atmosphere is under development for *in situ* GiSAXS measurements.

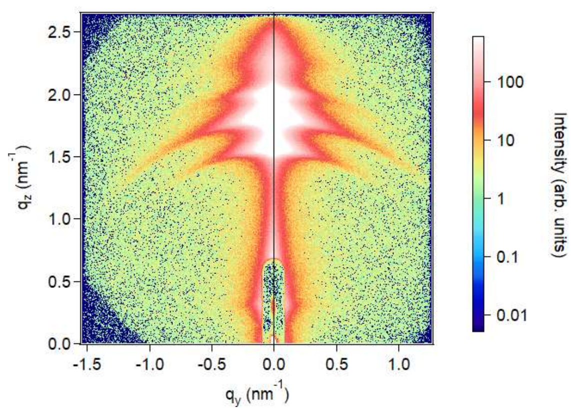

Fig.13: GiSAXS pattern from ionic liquid crystal thin film (thickness ~ 500 nm) measured on the lab-line. The sample-to-detector distance is 2 m. The diffraction peaks observed along q_z reflect the vertical organization of the ionic liquid crystal. The GiSAXS measurement indicates that the self-organization occurs perpendicularly to the surface with three characteristic peaks at 0.155 Å^{-1} (d = 40.5 Å), 0.180 Å^{-1} (d = 34.9 Å) and 0.200 Å^{-1} (d = 31.4 Å).

Organisation and life of the unit

Steering, life, organisation in the unit

Animation: The animation in the SGX team is organized through regular workgroup meetings or seminars, on a monthly basis. But the size of the team and the location of laboratories and offices favor regular exchanges.

Parity: ~40 % of the permanent team and 100% of the SGX or co-supervised PhD students are female.

Safety: The SGX safety report is updated every year. Bernard Mongelaz is "correspondant securité" for the laboratory.

Scientific integrity: The experimental data have been kept without restriction since the creation of SGX. Laboratory books and data files of non-permanent staff are kept by the tutors.

Five-year project and strategy of the SGX team

SWOT analysis

Strength

Strong collaborations with different CEA-Grenoble (more particularly ex-INAC) labs **-> better efficiency when combining skills in characterization and involvement in research issues**

Diversified and renewing Instrumental Park **-> Adaptability to emerging projects**

Strong connections with CNRS, ILL and ESRF **-> the lab is well anchored in Grenoble's scientific ecosystem (despite a limited visibility)**

Weakness

Aging staff **-> demotivation if no perspective of transmission of skills and knowledge**

Balance difficult to reach between service tasks for the community and own research **-> lack of visibility**

Complicated career paths for some team members **-> group dynamics hard to build**

Opportunities

Grenoble's scientific environment favoring opportunities for synergies **-> opportunities of collaborations**

Creation of IRIG **-> new perspectives (f. i. concerning the development of lab SAXS tools)**

Threat

Absence of recruitment prospects in the short / medium term **-> loss of expertise**

Absence of recurrent funding (operation, maintenance and investment) **–> very precarious equilibrium unfavorable to the construction of a laboratory development project**

Lack of valorization of the service function to the community **-> demotivation**

Structure, workforce and scientific orientations

At short term, the positioning of SGX as the IRIG X-ray characterization laboratory is not expected to change much.

Powder diffraction studies as well as thin film studies are carried out by Stéphane Lequien, E. Bellet-Amalric (straddling SGX and PHELIQS / NPSC) and Stéphanie Pouget, who have very complementary profiles to cover a wide variety of material studies for applications in the fields of spintronics, nanoelectronics and energy. The three associated diffractometers are up-to-date (D8 powders 2019, SmartLab thin films - high resolution / In Plane 2015, Empyrean thin film - standard resolution 2012), equipped with various sample environments (TTK600 and DHS1100 Anton Paar temperature stages, operando battery cell, ...), which positions the laboratory favorably in the short and medium term to meet the needs of new projects and emerging issues.

The single crystal diffractometer was purchased in 2008. It is co-managed by the MEM and DIESE/CBM services, which share maintenance costs, and the researcher who has the actual responsibility is Jacques Pécaut from the SYMMES/CIBEST laboratory. The main research topics concerned by this instrument are catalysis and bio-inspired chemistry as well as materials for energy. In the next four years, reflections on the renewal of the diffractometer will have to be undertaken.

The small angle scattering instrument is an important asset especially for the study of functional materials for energy such as proton conducting membranes, as well as graphene-like electrode materials. It is managed by Arnaud de Geyer and Alain Farchi. The instrument is over 20 years old. It has benefited from several upgrades, the last of which, in 2018, consisted in the development of a small angle grazing incidence (GISAXS) scattering configuration, from old device recovery elements (goniometric plates, electronics...). This new option makes it possible to address developing topics such as solid electrolyte materials. Collaborations are envisaged with teams from the DIESE / CBM / AFFOND and DBSCI / PCV / LIPMB laboratories, favored by the creation of IRIG, in the fields of amyloid fibers and membrane lipids. Unfortunately, instrumental breakdowns are more and more frequent and the instrument could become obsolete in the short term. Reflections on its renewal are undertaken, within the framework of the new institute, but also considering the positioning of such a laboratory characterization tool with respect to the large-scale facilities. Such a project, very relevant and potentially initiator of promising synergies, could however be jeopardized by lack of human resources. Indeed, Arnaud de Geyer will be retired within three years and there will be a lack of expertise in SAXS science in the group.

As concerns SGX own research, activities in the field of perovskite materials are expected to continue and develop as part of a close and solid collaboration with the laboratory DIESE / SYMMES / STEP. Interactions with the laboratory *LITEN*/*DTS*/*LMPO* at INES set up during Pia Dally's thesis (defense planned for autumn 2019) proved to be relevant and fruitful.

Research on electrode materials for energy storage remains a key topic for the institute. The laboratory, which has been equipped with a cell for operando measurements, will continue its activities in this field in connection with the NRS laboratory, and the large instruments ILL and ESRF.

The SGX laboratory has for a long time worked closely with the institute's laboratories specialized in epitaxial synthesis of metals and semiconductors, in the fields of nanoelectronics and spintronics. The geometry of the systems (2D, 1D, 0D) and the nature of the materials have evolved a lot over time. In the last years our diffractometers have been renewed for state-of-the-art instruments well adapted to address the actual issues. As a result, these collaborations will continue in the short and medium term, this field of research remaining a priority within the institute.

Operational organisation chart

E. Bellet-Amalric (1/2 SGX): thin film diffraction and reflectivity (specialist in high resolution diffraction)

A. de Geyer: SAXS

A. Farchi: SAXS

S. Lequien: thin film diffraction and reflectivity / computer control

B. Mongelaz: lab technician

S. Pouget: powder diffraction / thin film diffraction and reflectivity (group leader)

Equipment, platforms

Thin film diffractometer (Panalytical Empyrean 2012)

High resolution/In-Plane thin film diffractometer (Rigaku SmartLab 2015)

Powder diffractometer (Bruker D8 Advance 2019)

Single crystal diffractometer (Oxford Diffraction Xcalibur 2008)

SAXS/GISAXS instrument (home assembled – 1998)

Research products and activities of the SGX team

Scientific articles (total number) 43

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Books

Book chapters (total number)

Daniel Bellet and E. Bellet-Amalric In "Solar Cell Materials; Developing Technologies";Ed. G.J. Conibeer, A. Whilloughby, Wiley Series in Materials for Electronic and Optoelectronic Applications, John Wiley & Son, 2014, p35

Production in conferences / congresses and research seminars

10 talks (with abstract) were given in international conference

2 invited seminars (with abstract) were given participation through lectures and laboratory hand on practice to 8 winter/summer schools

Instruments and methodology

Platforms and observatories

X-ray characterization instrumental pool: five diffractometers and one small angle scattering instrument. See Annex 3

Academic research grants

European (ERC, H2020, etc.) and international (NSF, JSPS, NIH, World Bank, FAO, etc.) grants - partnership

H2020-NMP SINTBAT (2016 - 2020)

H2020-TWINN ORZEL (2015 - 2019) – staff exchanges with the silesian university of technology - 3×1 week in Gliwice

National public grants (ANR, PHRC, FUI, INCA, etc.) – partnership

UVLASE (2018 -2022) Electron-Pumped UV Laser ANR-18-CE24-0014

MECHASPIN (2018 - 2022) Atomic scale control and mechanical spin driving of a magnetic atom ANR-17-CE24-0024

ESPADON (2016 –2020) Engineering Spins and Photons from Anisotropic DOts in Nanowires ANR-15-CE24-0029

FLUO (2019-2022) Continuous-flow synthesis of InP based quantum dots. *ANR*-18-CE09-0039-01

PERSIL (2017-2021) Perovskite based solar cells for integration in tandem cells with silicium ANR-16-CE05-0019-02

NEUTRINOS (2017-2021) Ternary quantum dots synthesis emitting in the visible and near infrared for biologic detectione via Förster Resonance Energy Transfer. *ANR*-16-CE09-0015-03

HARVESTERS (2017 – 2020) Havesting energy with polymer based ThermoElectric generatoR to power autonomous Sensors *ANR-16-CE05-0029*

Local grants (collectivités territoriales) – partnership

PEAPLE (2018-2022) Pompage électronique de nanostructures d'AlGaN pour le développement d'émetteurs UV Projet Région ARA 2018

Scientific recognition

Invitations to meetings and symposia (out of France)

Organisations of meetings and symposia (international)

SYNOHE workshop (Synchrotron and Neutron techniques for Organic and Hybrid Electronics) - 2017

Members' long-term visits abroad

S.POUGET 3W Gliwize Poland

Involvement of the SGX team in training through research

Training sessions on X-Ray Scattering Techniques for PhDs, Researchers. (2/y)

(Recognized by Ecole Doctorale de Physique and Ecole doctorale Chimie Biologie Santé)

PhD students 2

Internships (M1, M2) 2

People in charge for a mention or a master's degree course (total number)

S. Pouget: X-Ray/neutrons scattering course in M2 “polymers for advanced technologie

ANNEX 1: LETTER OF COMMITMENT

Grenoble, May 31st 2019

I, the undersigned RIEUTORD François Director of the research unit “Modélisation et Exploration des Matériaux (MEM) ” certify, by the present letter, the exactitude of all data presented in this self-evaluation file, containing the self-evaluation document, and two Excel files named « Current contract data » and « Next contract date ».

ANNEX 2: OPERATIONAL ORGANISATION CHART

Direction MEM
Dir: F.Rieutord
Adj: T.Deutsch
Ass: C.Picot (1/2)
M. Sabate (1/2)

L_Sim team
T.Deutsch(C)

D.Caliste(C)
I. Duchemin(C)
L. Genovese(C)
F. Lancon(C)
YM Niquet(C)
P.Pochet(C)

LEMMA team
H.Okuno(C)

D Jalabert(C)
PH Jouneau(C)
E Robin(C)
JL Rouvière(C)
N. Mollard (Tech)

MDN team
S. Raymond (C)

B. Grenier(EC)
F.Bourdarot(C)
E. Ressouche(C)

B. Fouilloux(Tech)
B. Longuet(Tech)
B. Vettard(Tech)
F. Mantegazza(Ing)

NRS team
J. Eymery(C)

G. Renaud(C)
O.Robach(C 4/5)
V. Favre-Nicolin (EC, detach.)
S.Tardif(C)
X.Biquard (C)
C.Revenant(C 1/2)
P. Hecquet (C)
O. Ulrich (Ing.)

RM team
M.Bardet(C)

PH Fries(C)
M. Gromova (EC)
PA Bayle (ITA)

G de Paepe(C)
S.Hediger(DR CNRS)
D.Lee(C)

SGX team
S.Pouget(C)

E. Bellet-Amalric (C, 1/2NPSC)
S.Lequien(C)
A. Farchi(Ing.)
A de Geyer(Ing)
B. Mongellaz (Tech)

ANNEX 3: EQUIPMENT, PLATFORMS

LEMMA

Titan Ultimate Thermo Fisher (2013-): 40% of machine time

Titan Themis Thermo Fisher (2017-): 40%

FIB-SEM cross-beam Zeiss XB550 (2019-): 33%

FIB-SEM Zeiss NVision 40 (2010-): 25%

Other TEMs: 3

Other FIBs: 2

SEMs: 3

MDN

D23 Single crystal Diffractometer D23 for magnetic scattering

IN22 Thermal neutron three axis spectrometer IN22

IN12 Cold neutron three axis spectrometer IN12

Sample environment

A 12 T vertical field magnet (asymmetric for polarized neutrons).

A dilution fridge insert (50 mK for a cryostat or the 12 T magnet).

An ^{3}He fridge insert (300 mK for a cryostat or the 12 T magnet).

An electric field stick (15 kV).

A sphE. al polarization device CRYOPAD.

Five helium flow cryostats.

12 T vertical field magnet on D23 and spin echo option on IN22

NRS

BM32 Equipment @ ESRF:

MicroLaue station with focusing optics, scanning stages, and 2D detector (2006)

GMT 4-circle z-axis surface goniometer (1993)

INS2 In-situ 4-circle high speed UHV Surface and GISAXS diffractometer (2015)

RM

DNP equipment:

400 MHz solid-state NMR with DNP (cryo, DNP & console 2011, Minatec campus)

Ultra-low temperature instrumentation

400 MHz solid-state NMR with DNP (magnet 2005, console & DNP 2017, Minatec campus)

New console, new DNP ERC GDP

NMR Service:

500 MHz liquid-/solid-state NMR (2013, C-5 building)

HRMAS & CPMAS probes

400 MHz liquid-state NMR (2013, C-5 building)

200 MHz solid-state NMR (magnet 1979, console 1999, C-5 building)

High power PFG NMR and *operando* NMR

Relaxometry equipment:

Relaxometer (2004, C-5 building)

New relaxometer (2018 C-5 building) *home made (Stelar) prototype*

SGX

High resolution diffractometer (SmartLab) (shared with Néel/CNRS) (2015)

Powder diffractometer with temperature stage (100-1000K) (2019)

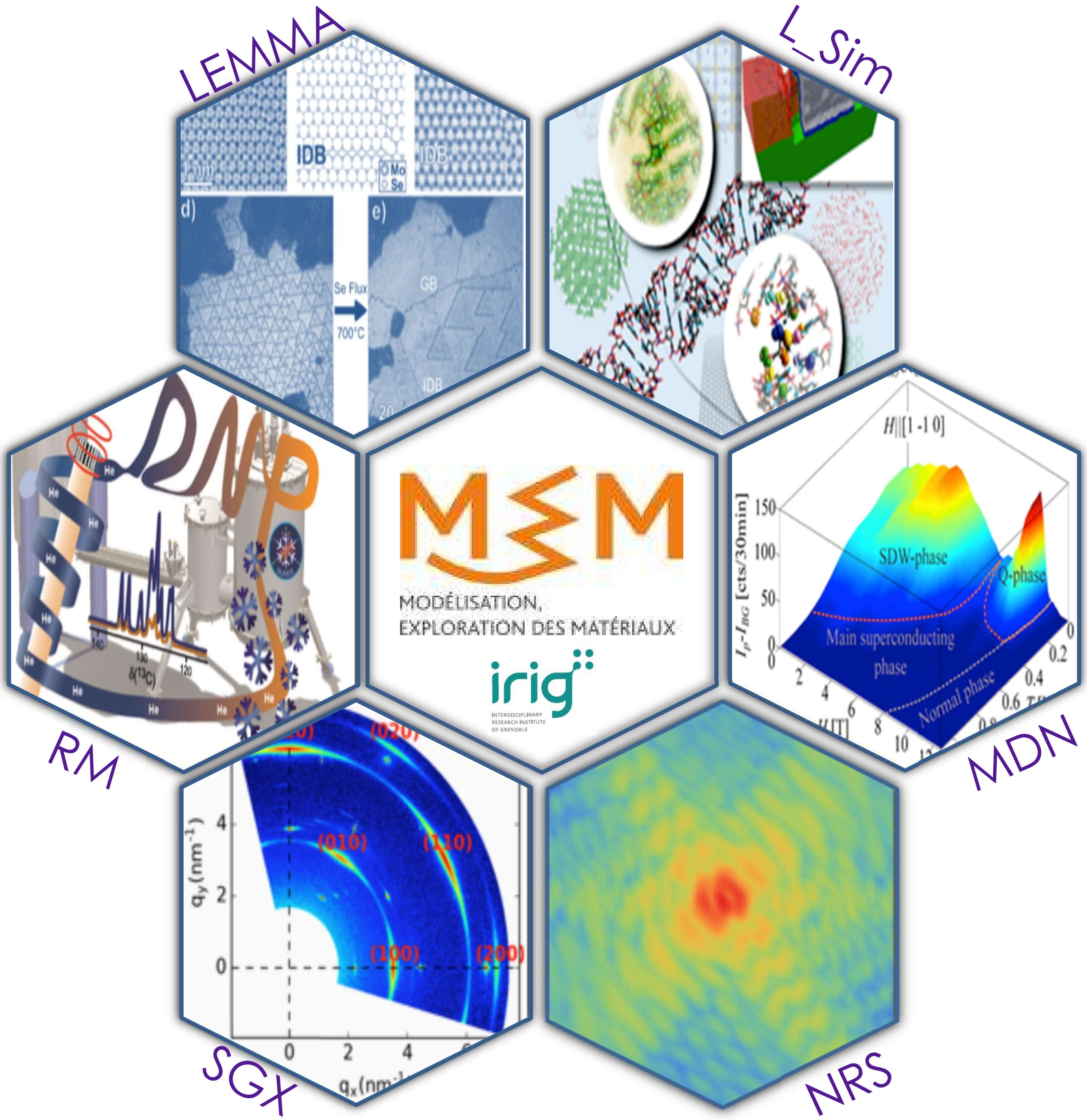
Thin film diffractometer/Reflectometer (2012)

SAXS diffractometer (home made, 1998)

4-circle diffractometer (Oxford Diffraction Xcalibur 2008) (shared with LCBM)

Laue bench with backscattering 2D detector (D6)

Surface diffractometer with rotating anode (1993)



Research Unit: *MEM – UMR_E9001*

Modeling and Exploration of Materials laboratory