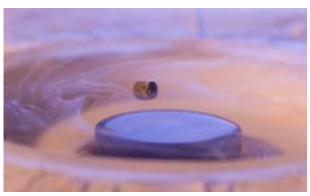
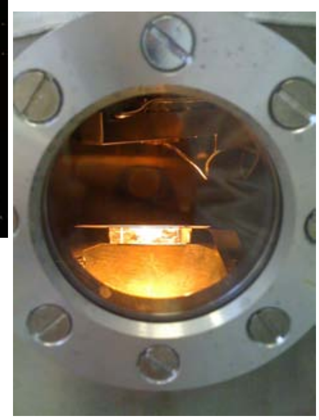
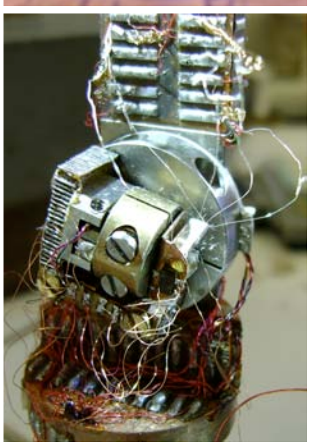
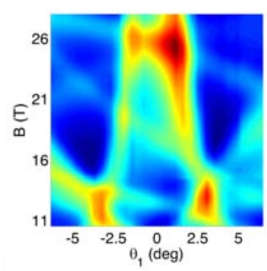
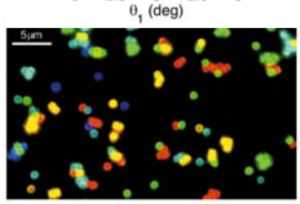
Physics and Materials Laboratory
UMR 8213











2007-2012 Scientific Report

LPEM PRESENTATION

1 Short presentation and history

"Laboratoire de Physique et d'Etude des Matériaux" (Physics and Materials Laboratory in english, LPEM is the french acronym) is installed on the "Ecole Supérieure de Physique et Chimie Industrielles Paris Tech" (ESPCI ParisTech) campus, downtown Paris. It is a common laboratory ("Unité Mixte de Recherche" UMR in administrative french language) of ESPCI ParisTech, the National Center for Scientific Research (CNRS) and "Pierre and Marie Curie University" (UPMC-Paris VI). Staff members belong to the three institutions, while PhD students are registered at UPMC. In july 2012, around 60 people were working at LPEM (28 permanent researchers and professors, 8 technicians, engineers and support, 21 PhD students and 7 Post-Docs, plus internship students and visitors).

Historical background

The present report covers the period January the 1st 2007 to July the 31st 2012. A short historical mention has to be made, since a major reorganization happened in 2008-2009. When examined by AERES in 2008, this laboratory was named "Laboratoire de Spectrométrie en Lumière Polarisée" and governed as a "Unité Propre CNRS" (UPR). Since the three aformentionned institutions (ESPCI, CNRS, UPMC) were strongly involved in the laboratory resources (both financial and human), the present (and new) direction proposed to change both the administrative status (going from UPR to UMR) and the name of the laboratory. This was approved by the AERES committee, which also ranked A^+ the laboratory at this occasion. Hence "Laboratoire Photons Et Matière" (LPEM) was about to be created as an UMR, when ESPCI direction decided unilaterally to reshuffle the cards, and to create a new laboratory, the Langevin Institute, including a sizable part of LPEM staff. In addition, LPEM was required to change its name.

After a while, a new situation emerged, starting officially in January 2009, with the creation of UMR 8213, "Laboratoire de Physique et d'Etude des Matériaux" with the same acronym LPEM. Roughly a third of permanent staff members and the associated PhD students left for Langevin Institute, while technical staff staid with LPEM.

Therefore, the following scientific report is based on the 2009-2012 period only. The publications from 2007-2008 of the staff members who left are listed separately. In the diagrams which show evolutions on a yearly basis, the two periods are indicated.

Research activity

At LPEM, research is focused on the physics and applications of materials, from basic electronic properties studies at ultra low temperature and high magnetic field, to the realization of devices and sensors with industrial partners. A strong and visible activity on synthesis and characterization of materials at the nano-scale recently emerged, and the coupling with industry has been reinforced when researchers from the former ESPCI "Laboratoire d'Electricité Générale" joined LPEM. Basic studies on quantum matter has always been a cornerstone of the laboratory in terms of high impact publications, and the recent hiring of both young talented and senior staff members boosted this activity furthermore.

The scientific topics at LPEM are organized around three main themes or axis:

- Nanophysics, nanostructures and nanomaterials

- Strongly correlated and low dimensionality electronic systems
- Instrumentation

In the first theme, research is mainly focused on electronic and optical properties of nanoparticles. In the first place, synthesis and characterization of colloidal nanoparticles of semiconductors (mainly II-VI materials) is made, whose properties are based on discrete energy levels controlled by their nanometric size according to quantum mechanics: they are called "quantum dots" (QD). Their optical properties are studied and applications in biological imaging are developed. The main recent achievements at LPEM are the synthesis of core/shell structures which strongly suppresses the optical blinking, a major drawback of the QD for applications, and the discovery of nanoplatelets (the 2D version of the QD), whose optical fluorescence spectrum is the finest among all the QDs. Metallic QD can be organized as 2D arrays, and quantum transport studied at very low temperature: co-tunneling events have been clearly identified at LPEM for the first time.

Nanostructures made on High Tc superconducting films display reproducible Josephson behavior, which are suitable for many applications which are successfully developed, such as magnetometers and THz detectors. Metallic plasmonic nanostructures and nano-heaters are studied with a new near field fluorescence microscope invented at LPEM which display unsurpassed resolution.

The second theme deals with fundamental issues in condensed matter physics, namely the quantum electronic orders that take place in materials, especially when strong correlations between fermions show up. Superconductivity, magnetism, multiferroicity and other related phenomena are studied at large scale by transport and optical measurements, but also at small scale, since quantum phase separations may occur in strongly correlated materials. Oxides have been studied with high impact results on High Tc superconductors, multiferroics or manganites. Fluctuations in this "quantum matter" have been extensively investigated. Unconventionnal superconductors such as heavy fermions or low electronic density semi-metals have also been successfully studied, with the observation of giant thermoelectric effects which has a large impact in the scientific community.

The third theme is devoted to the realization and study of new advanced tools either for the scientific community, either for industrial applications. Modeling and design of new sensors is a specialty of LPEM researchers. Most of the instrumentation developed at LPEM is related to electromagnetism, from sensors to localize charges in semi-conductors to RF antennas and circuit for telecommunications applications. New developments on NMR imaging are starting.

LPEM staff is also involved in the VIRGO project to detect gravitational waves by optical interferometry. Its role moves from the development of new optical instruments to qualify VIRGO's optics to the design and test of a dynamical system to stabilize the mirrors in the huge Fabry-Perot cavity (CALVA project).

Research at LPEM is, for now, exclusively based on experiments. The main techniques which are used to study matter and materials are electronic and optical probes, both in the transport and in the spectroscopic modes. When dealing with ground state properties of the materials, low temperature measurements down to 10 mK can be performed, while strong magnetic field (18 T) can be applied. Optical, NMR and RPE spectroscopies are routinely used, including pump-probe techniques, and near field probes like STM (Scanning Tunneling Microscope), EFM (Electrostatic Force Microscope) and SNOM (Scanning Near field Optical Microscope) are being developed. Nanoparticles and nanostructures are made, both chemically and by advanced e-beam lithography, and studied. High frequency RF devices are designed, simulated, made and measured. Powerful material characterization tools such as XRD (X Ray Diffraction), SEM

(Scanning Electron Microscope) and TEM (Transmission Electron Microscope JEOL 2010 FX) equipped with X-Ray analysis are operated within LPEM, and a new MEB-FEG (Field Effect Gun Magellan FEI) is about to be installed.

As described in more details in the scientific production section, the overall quality of the scientific output increased over the period as measured by the number of publications and their impact factors, while patents are regularly deposited. This shows the delicate but successful equilibrium reached at LPEM between fundamental issues in science and applications. A spin-off of LPEM (SOLARWELL) devoted to photovoltaic devices has been created in 2010, is an example of innovative initiatives in the laboratory.

Scientific environment

LPEM scientific activity benefits from its environment, in first place, the ESPCI campus. Strongly supported by ESPCI (human resources and budget), researchers are naturally coupled to other on-site laboratories and teams, to access specific tools or to develop new research. Foreign visitors from all over the world come and visit ESPCI including LPEM, thanks to good support (grants and lodging capabilities) from ESPCI. There is a clear willingness of the "owner" of ESPCI, namely the "Mairie de Paris" to promote high level scientific research, innovation and first class student training. LPEM benefits from this policy, with the possibility of hiring professors and associate professors, and the access to a solid budget. The difficulty however is that buildings are far from the required standard to develop XXIst century research: they are old, in very bad shape, not very safe. Moreover, the extension capabilities are very short or even null.

LPEM is located on the "Montagne Ste Geneviève", and is strongly involved in partnerships with its neighbors, mainly ENS and ENSCP-ParisTech. Collaborations with these partners are active on nanomaterials (nano-probes for SNOM systems, QD physics and applications), but also on nanophysics and nanostructures. It is worthwhile mentioning that LPEM is an active member of the Paris Cleanrooms Center ("Salle Blanche Paris Centre - SBPC": ENS, PVI, PVII, ESPCI). Nanodevices are made by LPEM members within SBPC facilities, and a few common equipment are based at LPEM, such as UHV evaporators and very soon a new MEB-FEG (Magellan FEI). Finally, an "anechoic chamber" is shared with Langevin Institute.

LPEM is also engaged in partnerships with UPMC laboratories on different projects. It is a member of the MATISSE (MATériaux, InterfaceS, Surfaces, Environnement) LABEX of UPMC. This collaboration of 18 partners aims at working on materials: LPEM members represents 7% of the researchers of the consortium. It is involved in the "axis #4" of the project:"dimensionality and confinement". The main support of MATISSE will be to provide PhD and Post-Docs grants for joined programs between two different laboratories on materials science subjects.

In a broader perspective, LPEM, through ESPCI, is a founder member of the PRES PSL (Paris Science et Lettres), which gathers partners on the "Montagne Ste Geneviève" (ENS, ESPCI-ParisTech, ENSCP-ParisTech, Observatoire de Paris, Collège de France), and of its IDEX extension PSL*, including ten others partners.

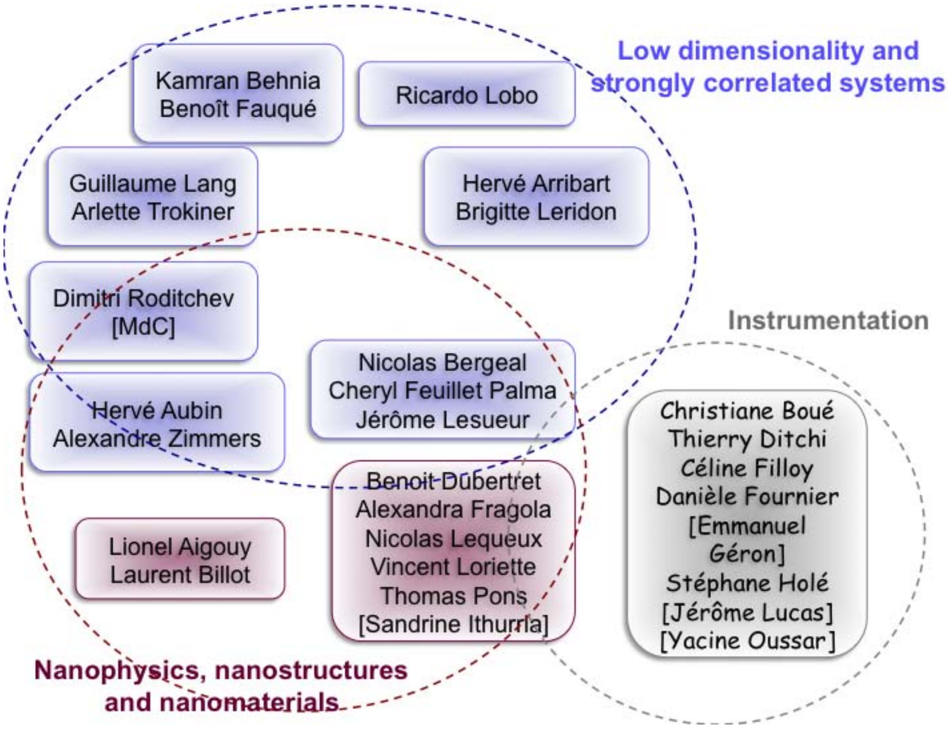
2 Organization, governance and management

Organization

The modest size of the laboratory is compatible with a light organization, which leaves room for initiatives and rapid changes required by fast moving science and the diversity of scientific domains. A given team may be more efficient with a critical mass of 15 members, whereas another one is as successful when composed of a PI and 2 students. Therefore, we do not present LPEM research organized in "teams", but structured along three mains themes as mentioned above:

- Nanophysics, nanostructures and nanomaterials
- Strongly correlated and low dimensionality electronic systems
- Instrumentation

People can work in different themes. Of course, people working together on the day to day basis form internal teams. The Parisian Quantum Dot team and the Instrumentation one have a strong internal structure ; work on basic solid state physics is made by small teams. The important feature is that the organization provides good conditions to staff members to produce the best science possible, and is sufficiently fluid to be rapidly adapted to new situations. Here is a schematic of the present situation:



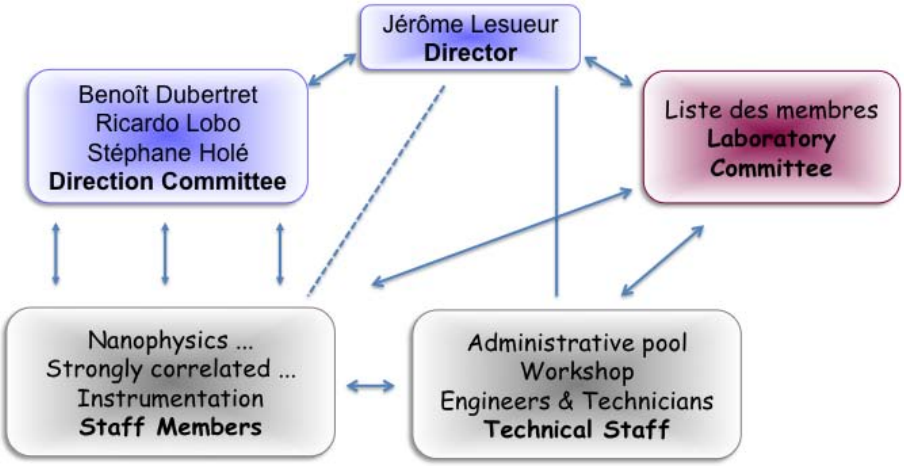
[Names in braquets are new staff members in january 2014)]

Technical staff and support

LPEM is understaff in what concerns technical personnel. In the present time, general support is made of an administrative pool (three persons), a machine-shop pool (one mechanic and a part-time projector) shared with another laboratory (UMR 7615), a computer specialist-webmaster. Engineers are directly involved in research: one in optical instrumentation, one in TEM studies.

Governance and management

The director is helped by a "direction committee" composed of three staff members (one from each scientific theme), which meet when decisions need to be prepared or made. The most important decisions, namely budgets and recruitment are discussed first with permanent staff, then in a "Laboratory Committee" (Conseil de Laboratoire) session, before a final decision is made by the director. The Laboratory Committee members join together at least three times a year to discuss the above quoted subjects, and of course, all the questions related to LPEM life. Minutes are written and published after each session. Members of the Laboratory Committee are elected or appointed by the director according to internal regulations (see appendix 4 and 8).



Common equipment and resources

The common resources at LPEM are mainly twofold: (a) the administrative and budget management pool, which deals with all the paperwork related to the laboratory activity ; and (b) the workshop. For several years, the workshop has been shared with another UMR at ESPCI (UMR 7615). An engineer (AI) is in charge of the drawings of the mechanical parts and the planning of the work. He can also make parts by himself. A technician is devoted to the realization of most of the pieces. Given LPEM recent growth, this is not enough, and a new technician should be hired. The optimization of the machines and the building of a new and decent workshop at ESPCI has been discussed for many years, and often postponed. It becomes urgent to solve those problems, and to find a global perennial solution at the ESPCI level.

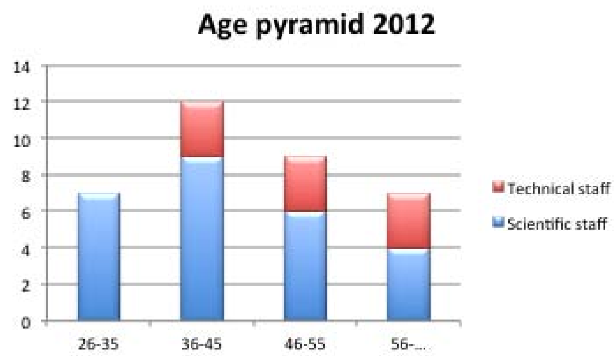
Recently, LPEM staff members lead collaborative initiatives to buy new common equipment. The first one, lead by B. Leridon and financed by Region Ile de France and ESPCI is an ensemble of low temperature - high magnetic field apparatus to study quantum matter: i) a multipurpose PPMS set-up equipped with a 14 T magnet and a rotational sample holder allows transport (and in the future, calorimetric) measurements down to 1.8K: ii) a SQUID VSM system with a 7T magnet to make ultra-sensitive magnetometry measurements down to 2K. This equipment is of common use in the laboratory and also opened for collaborations, mainly within Ile de France research teams. The second initiative, lead by H. Aubin, is an extension of the capabilities of the Paris Clean-room Center (SPBC). It consists in a MEB-FEG apparatus equipped with e-beam lithography, to observe, characterize and make nanostructures. It will be working at ESPCI as a common equipment.

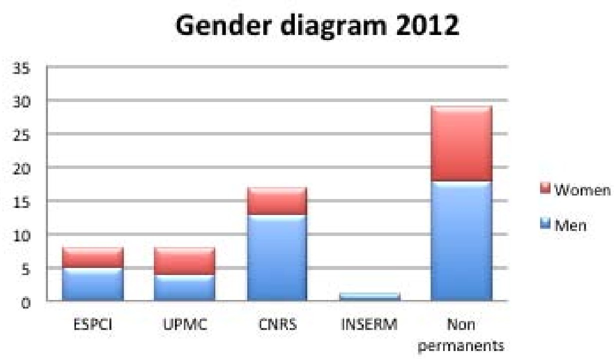
3 Human resources

General features

In July 2012, 62 people are working with LPEM (not counting 25 graduate and undergraduate students): 26 scientific staff members, 9 technical staff members, 21 PhD students and 7 Post-docs. By January 2014, at least two new staff members will join us. Moreover, three ESPCI associate professors (E. Géron, J. Lucas, Y. Oussar) from the exctinct "Laboratoire d'Electricité Générale de l'ESPCI" will join the LPEM Instrumentation team.

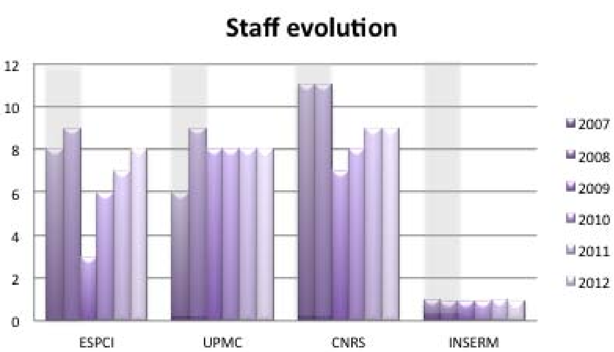
The age pyramid of permanent staff has a peak in the 35-45 years old range, with 7 colleagues younger than 35. This is the result of the vigorous hiring policy at LPEM. Females represent roughly 1/3 of the total population.

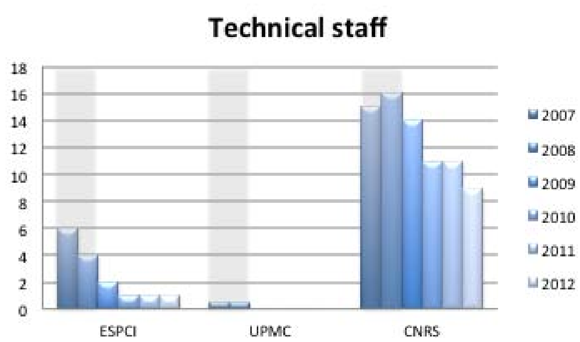




Permanent scientific staff

The graph presents the evolution of the number of staff members (researchers, associate professors and professors) on the period, displayed per institution.





[Shaded area: before the split]

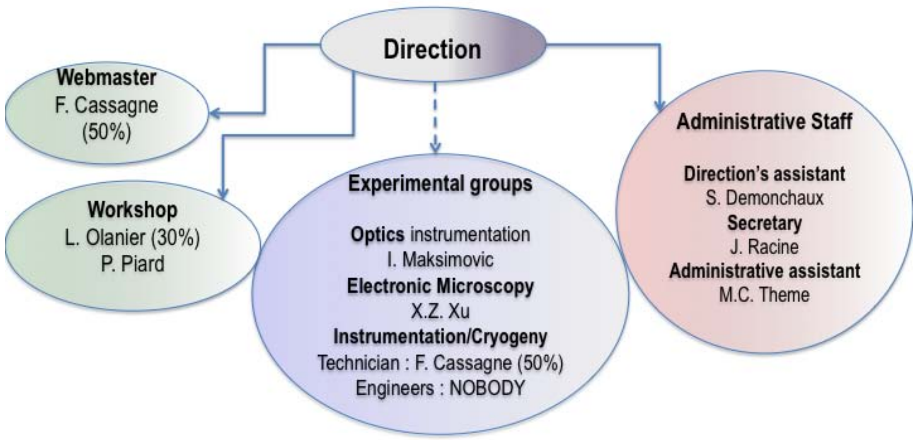
If we ignore the two first years (before the split-up), we observe that the three main institutions contribute at the same level to permanent staff members appointment. Over the last three years, ESPCI and CNRS staff has increased, while UPMC staff has remained constant. INSERM is a marginal contributor. Hiring policy has been vigorous, and highly talented young researchers joined LPEM. This contributes to the dynamic development of new subjects, and more generally to the laboratory itself. It is worthwhile mentioning that this recruitment mainly concerns the theme “Strongly correlated and low dimensionality electronic systems”.

Professors and associate professors represent more than 60% of the permanent staff members. There are only 10 full time researchers at LPEM. Given the heavy teaching duty of the formers, research lead by team fully composed on professors and associate professors may suffer of lack of competitiveness.

Permanent technical staff

If the situation is satisfactory for scientific staff, it becomes worse and worse for technical staff. The graphic shows a strong decrease of technical workforce at LPEM, which will impair its development in the very near future. While UPMC does not contribute at all, ESPCI and CNRS input is getting very low. Shortage is especially dramatic as far as high level engineers (Ingénieur de Recherche in CNRS language) are concerned. Indeed, the techniques developed at LPEM are more and more sophisticated and complex. For example, the number of dilution fridges rose from 1 to 4 over the period: high magnetic field cryostats ($\succ$ 5 T) from 2 to 8: near-field microscopes from 2 to 4: QD synthesis facilities from 2 to 5.

The diagram below summarizes the technical staff organisation. Directly linked to the Direction are the administrative pool, the workshop (shared with UMR 7516) and the IT/Webmaster technician. In the operationnal scientific teams, engineers work in close interaction with the researchers in optical instrumentation and elctron microscopy. A technician also supports a team activity in electronics.

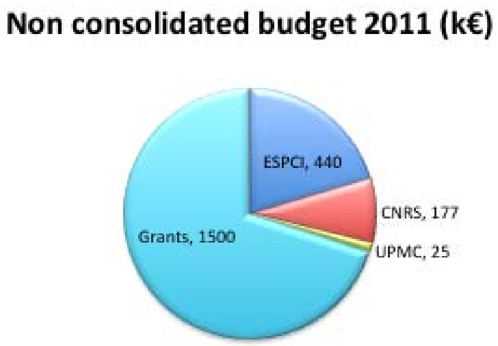


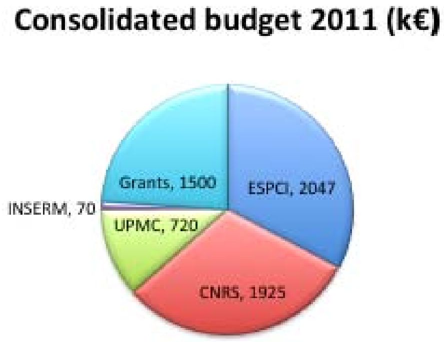
4 Financial resources and budget analysis

Budget structure

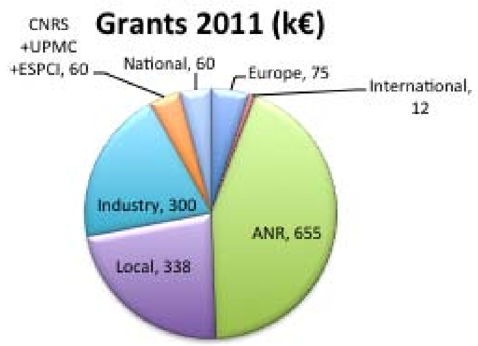
The non consolidated budget analysis shows that 70% of the resources come from grants (see graphic for 2011). They have been calculated on a yearly basis (total grant divided by the number of years devoted to the project). The second contributor is ESPCI which strongly supports LPEM activity and development. In addition to this average contribution, ESPCI provides special funds for dedicated operations. In 2012 for example, a startup budget for Dimitri Roditchev hired as a full professor (270 k€), a special budget for lowering liquid helium consumption (160 k€), an extra contribution to complete a regional budget for electronic microscopes renewing (175 k€) has been added. CNRS budget became weaker and weaker over the period. The graphic shows a decrease by a factor 2 in 4 years (see graphic below). This is a real concern since it is the most flexible part of the budget, which can be used for common expenses in the laboratory, or to launch new programs. Finally, UPMC direct contribution is constant but weak and INSERM's one null. We hope that these two institutions will support more LPEM development in the future.

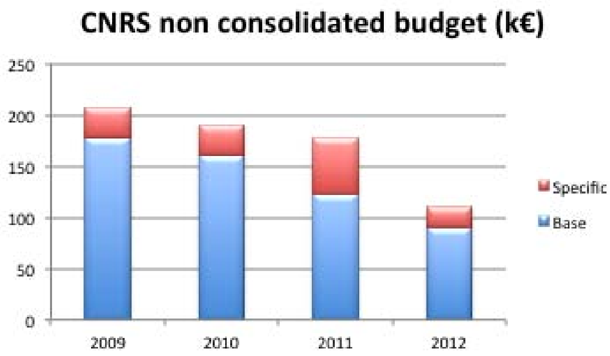
The consolidated budget of 6 262 k€ in 2011 (see graphic for 2011) reveals more balanced contributions between ESPCI and CNRS: this is because CNRS contributes to all the technical staff salaries (except one).





In 2011, funding through grants reaches 1.5 M€. Almost 45% are ANR grants (including two international ANR with Austria and China) as shown on the graphic below. Regional funds (CNano IdF , SESAME , Emergence Ville de Paris programs) represent the second contribution. Partnerships with industry (St Gobain, Bostik, GE ...) also provide substantial support to LPEM activity. Special operations from CNRS ("crédits spécifiques"), UPMC (BQR) and ESPCI represent 60 k€ in 2011. Finally, European and international collaborations represent a small fraction of the total. This is something which can be improved in the future.





Common budget and overheads

When 70% of the budget come from dedicated grants, it is not easy to fund common needs in the laboratory. Obviously, the corresponding scientific activity generates costs on the general budget of the laboratory. Since there are rather few common scientific equipment in LPEM which could justify a direct funding from grants, it is not simple to balance a fair common budget. We recently (in 2012) succeeded in finding a solution such as staff members can contribute to the common budget, in proportion of their grant.

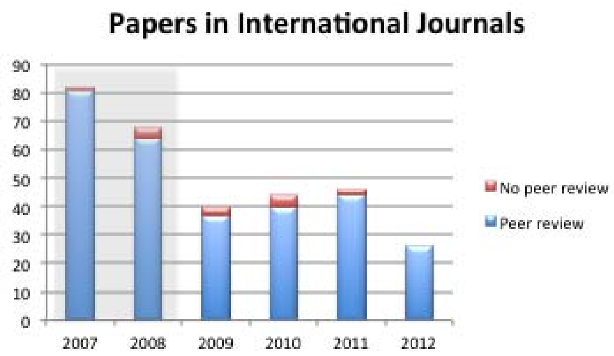
The common expenses mainly cover needs in general supplies and running costs, repairs, common computer resources, workshop tools and materials, internships salaries ... If there is budget left (depending on basic funding and grants), it is devolved to support new and/or specific projects, which has been discussed within the Laboratory Committee.

5 Scientific production and collaborations

Publications

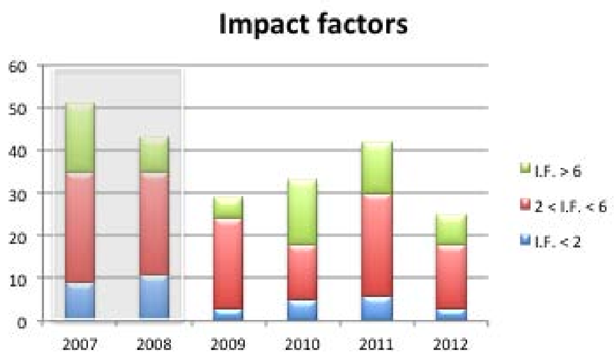
To assess the impact of research at LPEM, we provide in this report a short bibliographic analysis of the papers published by LPEM staff members over the period. The discontinuity observed between 2008 and 2009 is related to the split mentioned above, and the numbers in 2012 correspond to half a year.

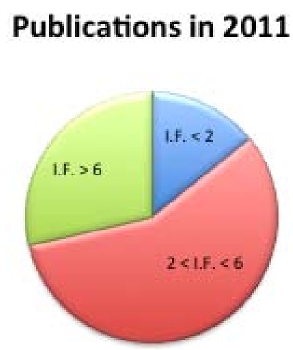
Publications are essentially published in peer-review journals. Their number increases gradually from 2009 and should reach around more than 50 papers in 2012, that is roughly two papers a year per staff member. This is a very good number for research in physics, all the more so 60% of the staff is professor or associate, and count for one half, according to AERES rule. This is a first strong indicator that research is vigorous at LPEM.



[Shaded area: before the split - 2012: Half a year]

The graphic below shows that the impact is also growing, with a net increase of the high Impact Factor (I.F.) publications over the period. As an example, almost 30% of the publications in 2011 has an I.F. higher than 6, and 85% higher than 2. These high level publications are related to almost all the research domains at LPEM, may be excepted for some research on instrumentation, where patents are taken, slowing down the publication process.





[Shaded area: before the split]

Highlights

Beyond the general Impact Factor of a journal, the number of citations of a given paper is a more specific measure of the direct impact of scientific results on the community. Absolute number of citations may be somewhat misleading, since the size of a scientific community and the total number of published papers strongly vary from a domain to another one (chemistry, physics, biology, applied physics ...). Since research at LPEM is multidisciplinary, one has to handle these numbers cautiously.

Among all the papers published in high impact journals by LPEM staff members, some have been quoted more than 50 times (see list below). Clearly, researches on colloidal quantum dots and on thermoelectricity at low temperature are the most visible scientific activities in the laboratory, according to this criteria. Highly cited publications from the VIRGO collaboration have been counted separately.

- B. Mahler, P. Spinicelli, S. Bull, X. Quelin, J.P. Hermier, B. Dubertret "Towards non-blinking colloidal quantum dots", NATURE MATERIALS, 7, 659-664 (2008). Times cited: 172.
- F. Dubois, B. Mahler, B. Dubertret, E. Doris, C. Mioslowski "A versatile strategy for quantum dot ligand exchange", JOURNAL OF THE AMERICAN CHEMICAL SOCIETY, 129(3), 482-482 (2007). Times cited: 95.
- L. Aigouy, P. Lalanne, J. P. Hugonin, G. Julie, V. Mathet, and M. Mortier. Near-eld analysis of surface waves launched at nanoslit apertures. PHYSICAL REVIEW LETTERS, 98(15):153902, (2007). Times cited: 64
- Y. Kasahara, T. Iwasawa, H. Shishido, T. Shibauchi, K. Behnia, Y. Haga, T.D. Matsuda, Y. Onuki, M. Sigrist, Y. Matsuda "Exotic superconducting properties in the electron-hole-compensated heavy-fermion "Semimetal" URu_2Si_2", PHYSICAL REVIEW LETTERS, 99(11), 116402 (2007). Times cited: 58
- T. Pons, E. Pic, N. Lequeux, E. Cassette, L. Bezdetnaya, F. Guillemin, F. Marchal, B. Dubertret "Cadmium-Free CuInS2/ZnS Quantum Dots for Sentinel Lymph Node Imaging with Reduced Toxicity", ACS NANO, 4(5), 2531-2538 (2010). Times cited: 56
- K. Behnia, L. Balicas, Y. Kopelevich "Signatures of electron fractionalization in ultra-quantum bismuth", SCIENCE, 317(5845), 1729-1731 (2007). Times cited: 55.
- P. Spinicelli, S. Bull, X. Quelin, B. Mahler, B. Dubertret, J.P. Hermier "Bright and Grey States in CdSe-CdS Nanocrystals Exhibiting Strongly Reduced Blinking", PHYSICAL REVIEW LETTERS, 102(13), 136801 (2009). Times cited: 50

Prizes

The Ludwig-Genzel-Prize has been awarded to Ricardo Lobo as a "young scientist for exceptional contributions to the field of condensed-matter spectroscopy" in 2008. This award is given every two years by an international committee.

Conferences and seminars

Another indicator of research impact is the invitations in international conferences. The 49 invitations over the period show that LPEM research is present at the international level.

However, a closer look to the data shows that these invitations concern mainly one researcher (K. Behnia), and then a few others. This is partly related to the youngness of the staff, but one has to make sure that more brilliant LPEM researchers are invited in international and even national conferences.

Staff members and students do regularly participate to conferences and present their work. However, only two international conferences have been organized by LPEM ("On the Heavy Fermions Road" in 2010 by K. Behnia and LEES 2014 by R. Lobo), and one International Summer Scool ("Multiband and Multiorbital Effects in Novel Materials" in 2011 by R. Lobo). This is also a weak point for the international influence of LPEM. A national summer school "Atelier INSERM 195 Novel imaging techniques for biology: superlocalization and superresolution" has been organized by LPEM members and a national workshop on Superconducting devices (SEFIRA 2007) by J. Lesueur.

Seminars are regularly organized in the laboratory, mainly on two types of subjects: basic solid state physics, and quantum dots physics and applications (complete list in appendix 4).

LPEM staff members are involved in national and international collaborations. Some are active members of different national CNRS-GDR (complete list in appendix 2), mainly "MICO" on materials and competing electronic orders, "PHOTOMED" on photo-activated molecules for therapy, "microscopie fonctionnelle du vivant" on new microscopy techniques for bio imaging, "ONDE" on waves, from quantum to classical, and "NANOTh" on thermal behavior at a nanoscale and THERMOELECTRICITE. ANR grants and other contracts are the major source of national collaborations. With more than 49 contracts over the period, LPEM is involved in a dense network of partnerships and collaborations (details are given in the "Results" section. A lot of informal international collaborations are very active in the laboratory, and a few ones get special funding through ANR (France-China, France-Austria), CNRS PICS programs (France-Brazil) or other programs with Argentina (ECOS-Sud), India (IFPCAR) or Ukraine. All collaborations are listed in the "Results" section and in appendix 2.

LPEM staff members are referees for most of the high impact factor journals in physics and inorganic chemistry. Some of them belong to advisory boards of national collaborations (GDR) or organizations, and only a few to international committees.

Industrial partnerships and patents

This is a tradition at ESPCI, to mix basic and applied research in the same laboratories, such as a continuum of knowledge can be made, bridging the gap between these two very often separated areas. This culture is present at LPEM, especially among the researchers working on instrumentation (S. Holé, T. Ditchi, C. Boué, C. Filloy-Corbrion, D. Fournier, E. Géron, J. Lucas). Contracts with industrial partners such as Bostik, General Electric, SAGEM are regularly made directly or through specific ANR programs. A partnership with St Gobain is also very active, with H. Arribart and H. Aubin contracts on optical and electrical properties of nanomaterials. Moreover, a spin-off (SOLARWELL) of LPEM has been created by B. Dubertret in 2010, on applications of colloidal Quantum Dots. LPEM is part of an important contract (FUI TOCATA) within the "Pôle de compétitivité" ASSECH associated with 13 others partners including SNECMA, DASSAULT, AREVA ... C. Boué is the LPEM PI working on the detection of defects by IR thermography. A recent partnership with the start-up company Neelogy has been set-up by B. Leridon on applications of magnetic nanoparticles for current sensors, within the "Pôle de compétitivité" ASTECH.

Over the period, 11 patents have been deposited by LPEM staff members. Here is a list of significative ones:

- Dubertret, B.; Gardeazabal Rodriguez, P. F. & Loriette, V. Systeme d'illuminations structurée d'un échantillon, Brevet n°FR 2922658, 24/04/2009, 2009

- Mokhtari, Z.; Holé, S. & Lewiner, J. Smoke detector Demande de brevet américain 13/192,633, 28/7/2011, 2011

- Cuvigny, N.; Ditchi, T.; Géron, E.; Holé, S. & Lewiner, J. Procédé et dispositif de mesure d'humidité sur un flux de plaquettes forestières, Demande de brevet n° 1161586 à l'INPI le 13/12/2011, 2011

- Mokhtari, Z.; Holé, S. & Lewiner, J. Capteur à dérive, Demande de brevet français 1100356, 4/2/2011, 2011

- Ditchi, T.; Holé, S.; Géron, E.; Houdali, N.; Léchéa, N. & Boué, C., Route intelligente, en cours de dépôt au CNRS, 2012

- Boué, C.; Tessier, G.; Roger, J. P. & Streza, M. Procédé d'évaluation de la profondeur d'une fissure Demande de brevet n°1258940 le 24/09/2012, 2012

6 Training and education

BSc and MSc training

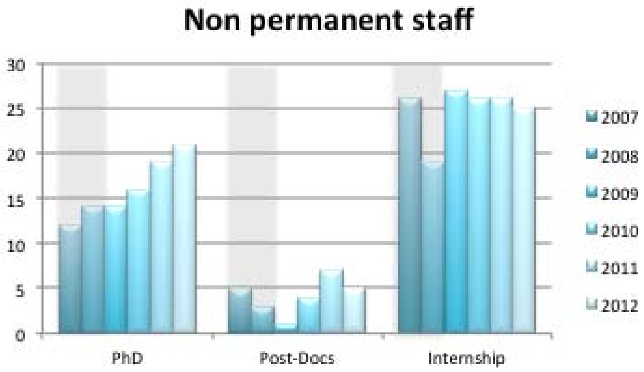
As already mentioned, a majority of staff members are professors or associate-professors at ESPCI ParisTech or at UMPC. Therefore, they are strongly involved in training MSc and BSc (see a complete list in appendix 5). They mostly teach basic physics (General physics, Electromagnetism, Quantum mechanics, Telecoms, Optics, Special relativity), material sciences (Crystallography, Solid State Physics, NMR and Optical spectroscopies) and instrumentation (Electronics, Micro-waves devices, Optics, Sensors ...). ESPCI ParisTech professors are responsible of lectures at each level of the course, including practical-works and tutorials. UPMC associate professors are in charge of two MSc (Master 2 *Capteur Mesure Instrumentation CMI, & Lasers, matériaux, milieux biologiques*) and two BSc (L1 *Capteur, mesure & Instrumentation optique et visualisation*). LPEM researchers were also involved in MSc (*M2 Concept fondamentaux de la physique, parcours "physique du solide" & M2 Science des Matériaux et Nano-objets*), but their contributions becomes weaker or even null for the next versions of those formations. This is an alarming point.

LPEM welcomes roughly 25 internship students per year, mainly from MSc (M2) courses and for ESPCI third year projects. Students usually work in the laboratory from 2 to 6 months.

PhD and post-docs

In the recent years, the number of PhD students has increased significantly (see graphic). In addition to the natural increase correlated to the permanent staff one, the success of LPEM researchers at getting grants (mainly ANR) including PhD salaries comes into play. LPEM is already an attractive place for PhD students, but with an average of one student per permanent staff, there is still possibilities to increase this number. Through international collaborative programs (mainly with China), foreign students are also educated at LPEM. It is worthwhile mentioning that industrial partnerships also provide possibilities of hiring students. Research and PhD training at LPEM goes from basic science to applications for industry. After the thesis, students follow diverse roads: roughly 40% work for private companies just after the defense, and 60% go for a post-doc (half of it abroad).

The number of post-doc is rather constant. Half of them are foreigners.



[Shaded area: before the split]

7 Outreach

For the 100th anniversary of the discovery of superconductivity in 2011, LPEM staff members working in this field (in the first place B. Leridon and J. Lesueur) were very active to propose and organize events in Paris on the sub ject. They involved in numerous exhibitions (photographs, art and science performances, scientific experiments ...) and public conferences for unspecialized audience at ESPCI. They were major actors of events in Paris, such as “Supra Day” in June 2011 at “Arts et Métiers” museum, or “Superconductivity week” at “Cité des Sciences de la Villette”. They were also present in large audience medias (France Culture, France Inter, RFI, FR3, Universcience), and even collaborated to a comics for kids made by J. Y Duhoo and published in “Journal de Spirou”.

External communication of LPEM results mainly used CNRS communication channels. Since 2007, 6 “Brèves CNRS” and 2 articles in “Journal du CNRS” from LPEM have been published (see list below).

- Les nanoplaquettes: de nouveaux nanocristaux bidimensionnels (juin 2012)
- Polarisation des vallées de Dirac du bismuth par le champ magnétique (juin 2012)
- La Supraconductivité prend son envol (avril 2011)
- Oscillations quantiques de l’effet Nernst (juillet 2010)
- Trois nano-stars des écrans (octobre 2008)

- A quelle température les paires de Cooper se forment-elles dans les supraconducteurs à haute température ? (septembre 2008)
- Le Prix Genzel attribué à Ricardo Lobo (juin 2008)
- L'électron se fractionne-t-il dans le bismuth ? (octobre 2007)

RESULTS

SCIENTIFIC RESULTS

Nanophysics, nanostructures and nanomaterials

Permanent staff: L. Aigouy, P. Bassoul, N. Bergeal, L. Billot, B. Dubertert, A. Fragola, N. Lequeux, J. Lesueur, T. Pons

Non permanent staff:

PhD: S. Bouccara , C. Bouet , E. Cassette , A. Dijksman , P.F. Gardeazabal-Rodriguez, S. Helas-Othenin, S. Ithurria, C. Javaux, B. Mahler, E. Muro, E. Saidi, B. Samson, G. Sitbon, M. Tessier, P. Vermeulen

Post-doc: L. Biadala, L. Lalouat, B. Nadal, B. Mahler, W. Pan

Key words: quantum dots, colloidal nanoparticle, fluorescence, super resolution, biological probes, nano thermics, plasmonics, THz detector, semiconductor, superconductor

Scope and Organisation

Nanosciences are emerging as a major topic in modern science, for both basic research and applications. LPEM is strongly involved in this field, from synthesis and characterization of new nano-materials to the development of advanced nano-detectors, via specific nanophysics studies. Indeed, a world wide recognized activity on colloidal nanoparticles (Quantum Dots) synthesis and characterization lead by B Dubertret has emerged, with applications ranging from biological imaging probes to photovoltaics applications. On another hand, a unique near field fluorescent probe has been invented by L Aigouy, for studying thermal and optical properties of materials and nanostructures at a nanoscale. Finally, high frequency detectors using High Tc Superconductors are made by N Bergeal using nano Josephson Junctions.

Scientific activity on QD is a strongly integrated project, with a large autonomy within LPEM, and recognized as an “ESPCI team”. The “Quantum Parisian Group” focuses on inorganic chemistry to synthesize new QD, and to spectroscopy studies to understand their fundamental optical properties and optimize their characteristics for various applications. A start-up, SOLARWELL, has been founded to develop applications of QD. Correlated projects on advanced super-resolution imaging systems are also developed in the group. Fruitful interactions with researchers in theme “Strongly correlated and low dimensionality electronic systems” have been made to produce other types of nanoparticles.

Research on plasmonics and nano-thermics is made by a very active small team (L Aigouy & L Billot), in strong interactions with theoreticians in the domain, while superconducting devices are studied by a team (N Bergeal, J. Lesueur) which shares its activity between basic and applied science.

1 Synthesis and characterization of novel semiconductor nanoparticles

(T.Pons, B. Dubertret, N. Lequeux, A. Fragola, V. Loriette)

1.1 Toward non blinking CdSe/CdS semiconductor nanoparticles

At a single-molecule level, fluorophore emission intensity fluctuates between bright and dark states. These fluctuations, known as blinking, limit the use of fluorophores in single-molecule experiments. The dark-state duration shows a universal heavy-tailed power-law distribution characterized by the occurrence of long non-emissive periods. Here we have synthesized novel CdSe–CdS core–shell quantum dots with thick crystalline shells, 68% of which do not blink when observed individually at 33 Hz for 5 min [51]. We have established a direct correlation between shell thickness and blinking occurrences. Importantly, the statistics of dark periods that appear at high acquisition rates (1 kHz) are not heavy tailed, in striking contrast with previous observations. Blinking statistics are thus not as universal as thought so far. We anticipate that our results will help to better understand the physico-chemistry of single-fluorophore emission and rationalize the design of other fluorophores that do not blink.

1.2 Synthesis of very thick shell nanocrystals

We report the synthesis of CdSe/CdS semiconductor core/shell nanocrystals with very thick (5nm) CdS shells. As in the case of core CdSe nanocrystals, we show that a thick-shell CdSe/CdS core/shell structure can be synthesized in either a pure wurtzite (W) or a zinc-blende (ZB) crystal structure. While the growth of thick-shell wurtzite CdSe/CdS is quite straightforward, we observe that the growth of a CdS shell on zinc-blende CdSe cores is more difficult and leads to wurtzite/zinc-blende polytypism when [22] primary amines are present during the shell formation. Using absorption spectra analysis to differentiate zinc blende from wurtzite CdSe, we show that primary amines can induce a nearly complete structural transformation of CdSe ZB cores into W cores. This better understanding of the CdSe ligand-dependent crystal structural evolution during shell growth is further used to grow large (10 nm)-diameter perfect zincblende CdSe core crystals emitting above 700 nm, and perfect ZB thick-shell CdSe/CdS nanocrystals. We observed that all thick-shell CdSe/CdS QDs have extremely reduced blinking events compared to thinshell QDs, without any significant influence of crystalline structure and polytypism.

1.3 Atomically flat semiconductor nanoplatelets

The syntheses of strongly anisotropic nanocrystals with one dimension much smaller than the two others, such as nanoplatelets, are still greatly underdeveloped. Here, we demonstrate the formation of atomically flat quasi-twodimensional colloidal CdSe, CdS and CdTe nanoplatelets with well-defined thicknesses ranging from 4 to 11 monolayers [50, 9]. These nanoplatelets have the electronic properties of twodimensional quantum wells formed by molecular beam epitaxy, and their thickness-dependent absorption and emission spectra are described very well within an eight-band Pidgeon–Brown model. They present an extremely narrow emission spectrum with full-width at half-maximum less than 40meV at room temperature. The radiative fluorescent lifetime measured in CdSe nanoplatelets decreases with temperature, reaching 1 ns at 6 K, two orders of magnitude less than for spherical CdSe nanoparticles. This makes the nanoplatelets the fastest colloidal fluorescent emitters and strongly suggests that they show a giant oscillator strength transition.

1.4 Anisotropic core/shell structure with 2D polarized emission

We report the synthesis and properties of a novel class of nanocrystals with mixed dimensionality: a dot-in-plate core/shell nanostructure [19]. This system was synthesized by growing a flat, disk shaped, CdS shell on spherical CdSe cores. The anisotropic pressure induced by the shell drastically splits the first exciton fine structure in two: the "heavy hole" and "light hole" states become separated by up to 65 meV. As a result, these nanocrystals exhibit an emission strongly polarized in two dimensions, in the plane perpendicular to the wurtzite crystal c axis. We use polarization measurements on single nanocrystals and ensemble anisotropy studies to confirm the nature and position of the excitonic energy levels. These nanocrystals orient spontaneously when evaporated on a substrate, enabling a precise control of the orientation of their emission dipole.

2 Development of fluorescent quantum dots as biological probes

(T.Pons, B. Dubertret, N. Lequeux, A. Fragola, V. Loriette)

Semiconductor quantum dots present several advantages over conventional organic dyes for biological imaging and detection. They offer narrow emission spectra, tunable from the UV to the near infrared, broad excitation spectra, and excellent fluorescent quantum yields, together with high extinction coefficients and prolonged photo-stability. These unique properties have triggered the development of quantum dots (QDs) for many biological applications, ranging from single molecule tracking to ultrasensitive biochemical detection and in vivo imaging.

2.1 QD functionalization

QD surface chemistry has a crucial role in the fate of QDs in biological environments. Since QD are synthesized in organic solvents, ligand exchange or encapsulation using amphiphilic polymers is necessary to solubilize these nanoprobes into water. We have first developed novel hydrophilic zwitterionic (sulfobetaine) ligands. These ligands provide the QDs with superior stability in aqueous media and minimal non specific adsorption on biological membranes and biomolecules, while being much more compact than commonly used poly-ethylene glycol based ligands [23]. This smaller size presents many advantages in biological experiments, including better access to confined spaces, reduced steric hindrance and more efficient FRET efficiencies for reporter probes. We have further shown that standard mono- or bi-dentate ligands tend to quickly detach from the QD surface, resulting in a rapid loss of solubility and functionality. We have thus developed a copolymer ligand comprising several zwitterions for solubility, dithiol groups for enhanced QD affinity and amine groups for further bioconjugation/functionalization. These polymeric ligands offer a considerably enhanced stability compared to standard ligands due to a multidentate effect, opening the way for long term live cell imaging.

2.2 Intracellular QD delivery and targeting

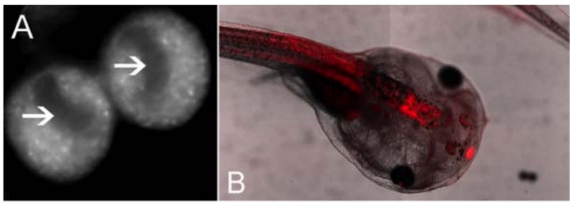


Figure 1: (A) Cells during mitosis 24h after electroporation with QDs, which are still free and bright into the cytoplasm, but are not associated with DNA (white arrows); (B) Merge of QDs fluorescence (red) and transmission (grey) images of a QDs microinjected Xanopus Laevis tadpole (2,5 weeks old).

Intracellular live cell imaging with QDs require crossing the plasma membrane. Most QD intracellular labeling methods rely on endocytosis, resulting in endosomes-trapped QDs that cannot access the cytoplasm. We have thus developed a variety of methods to deliver freely diffusing QDs into the cell cytoplasm, based on électroporation, pinocytosis and micro-injection [1]. We have demonstrated that these methods applied to different biological scenario, including live cell or embryo imaging. We have compared different QD surface chemistries and shown that our zwitterionic copolymeric ligands offer better stability in the cytoplasm of live cells compared to commercially available QDs or standard solubilization strategies. We have further shown that intracellular molecules may thus be efficiently targeted using functionalized QDs in live cells (Figure 1)

2.3 Near infrared quantum dots for in vivo imaging

The optimal spectral range for thick tissue or in vivo imaging is the near infrared NIR region, between 700 and 1000 nm, where light absorption and diffusion by tissues is minimal. Applications of QDs in medical imaging applications have long been hindered by the low fluorescence quantum yield of NIR QD and by their toxic heavy metal components. We have thus developed novel bright, less toxic, NIR QDs based on $CuIn_2\,(S, Se)$ materials [19, 25]. We have demonstrated the low in vitro and in vivo toxicity of these nanoprobes, opening the way for possible future medical applications. In particular, we have demonstrated efficient visualization of the sentinel lymph node. Resection of this lymph node during breast cancer surgery provides an important prognostic for the metastatic status. NIR fluorescence imaging could advantageously replace current protocols based on injection of radioactive tracers and a blue dye.

3 Widefield fluorescence microscopy: super-resolution and wavefront correction for dynamic 3D imaging of living samples

(T.Pons, B. Dubertret, N. Lequeux, A. Fragola, V. Loriette)

3.1 Single shot wide field optical sectioning

Optical sectioning by pattern projection is now a well established technique which main drawback is the number of partial images (3) that one has to acquire sequentially in order to obtain a single wide field optical section. We have built a microscope based on Jerome Mertz's HiLo technique, combined with a dual illumination / dual detection system and the development of specific two-color markers to realize optical sections with a single image acquisition [12]. Not only this instrument allows faster acquisition rates, but by eliminating the sequential aspect of the image acquisition step, is much less sensitive to sample movement and artifacts generated by it.

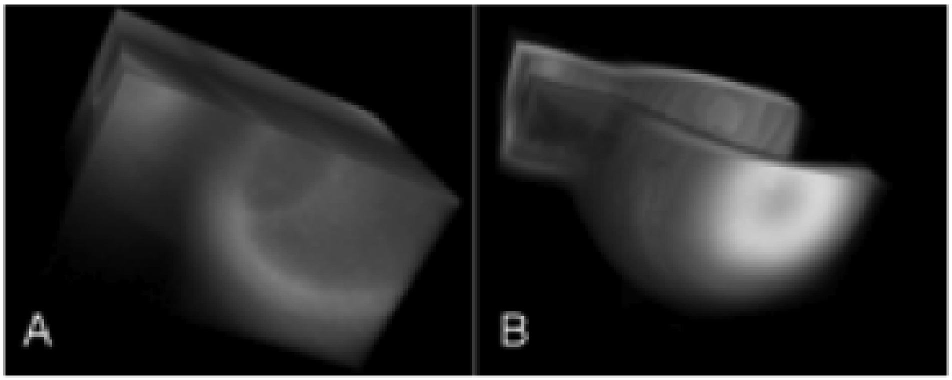


Figure 2: 3D view of two surface coated fluorescent polystyrene beads of different radii. Right image is a 3D composition from standard wide field 2D images, left image is a composition of the same sample in the same experimental con-figuration from single shot optical sections.

3.2 Dynamic super resolution imaging

Since 6 years we have developed an extended resolution system based on fringe projection microscopy. During the last three years we have aimed our efforts at writing an efficient image processing algorithm to reconstruct a super resolved image. Contrarily to the other few groups working in the field, we have developed an original method based on a bayesian approach. This way we are able to build super resolved optical sections of thick samples with a limited number of images (4 instead of 7 or 9) and acquire image stacks at video rate (see figure 3). Part of this work was financed by ANR (ANR PCV, XXL resolution) and by UPMC (BQR).

3.3 Wide field wavefront correction

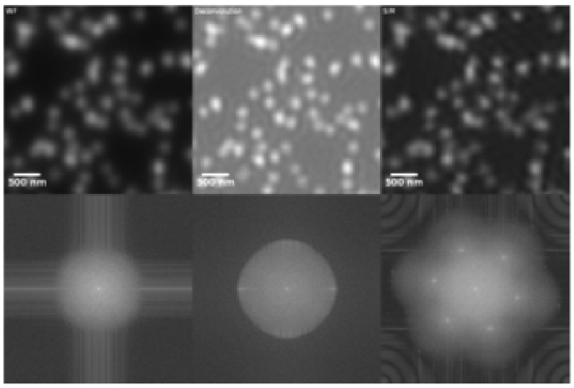


Figure 3: Comparison of widefield, deconvolved and super resolved images of fluorescent beads obtained with 4 partial images. The Fourier transforms clearly show the resolution enhancement obtained with the 4-image algorithm. The gain is equivalent to the one obtained with seven or nine images.

Adaptive optics (AO) is new in the field of optical microscopy. The first implementations of AO in microscopy were used in two-photon microscopy to improve the focusing properties of the light sources. Only recently AO has been used to improve the quality of optical images. We have implemented an AO loop on a commercial microscope body and demonstrated its ability to recover aberration-free images when imaging through fixed aberrant samples [18]. Thanks to the use of quantum dot filled beads we obtain very bright and stable point-like emitters that act as guide stars. The broad absorption band of quantum dots allows exciting the stars at a wavelength chosen not to be absorbed by the sample markers. This way we can perform wavefront correction before imaging the sample and without photobleaching it. In order to improve the setup efficiency a star atlas is recorded before applying the corrections and taking the data. The stars are then illuminated sequentially by a focused laser which position is taken from the atlas. Each star allows correcting the wavefront within a small patch. The wide field image (see figure 4)is synthesized from the many images by selecting the different corrected patches. This instrument has been presented at FOM 2012 and is the main development of Pierre Vermeulen's thesis. Comparison of images of 200 nm diameter fluores-

Figure 4: Comparison of images of 200 nm diameter fluorescent beads: left image without correction showing the deformable mirror induced aberrations; center image showing residual setup aberrations; right image showing nearly perfect aberration correction.

cent beads: left image without correction showing the deformable mirror induced aberrations; center image showing residual setup aberrations; right image showing nearly perfect aberration correction.

4 Nano-optics and plasmonics

(L. Aigouy, L. Billot)

A near-field optical technique that uses a small fluorescent particle glued at the end of a sharp W tip has been developed (see Figure 5). The advantage of this technique comes from its passivity. The measured signals and images are easy to interpret and allow to study local optical phenomena in a quantitative way, in 3 dimensions above the nanostructures.

4.1 Propagating surface plasmon polaritons

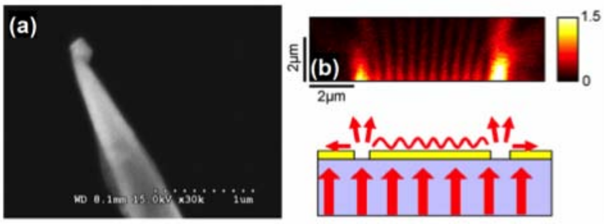


Figure 5: (a) SEM image of a fluorescent particle glued at the end of a tip; (b) experimental 3D near-field optical image showing the interference between surface plasmon polaritons launched at two nanoslit apertures

We have studied the generation of surface plasmon polaritons by nanoapertures on a thin gold film. In the case of two parallel nanoslits illuminated in a transmission mode, we have experimentally observed that the surface waves that are generated by the slits are composed of a plasmonic component and a quasi-cylindrical wave [57]. The near-field observations (see an example in Figure 5) are in excellent agreement with numerical simulations performed by P. Lalanne at Institut d'Optique Graduate School in Palaiseau [57].

Similarly, with the aim of well understanding the nature and the properties of the surface waves, we also imaged and study their generation by other types of nanostructures like nanoparticles, linear and curved ridges, and their propagation on various substrates (Au/Si, Si alone) at oblique incidence [40, 7, 58, 21].

4.2 Localized optical fields

We have studied the distribution of the electromagnetic field on gold nanodisks arrays excited near their plasmon resonance wavelength [5]. In agreement with numerical simulations performed at Instituto de Microelectronica de Madrid, we observed that the light is localized between the disks, in the direction of the incident polarization direction. Together with far-field transmission spectra, these results were helpful to design plasmonic biochemical sensors.

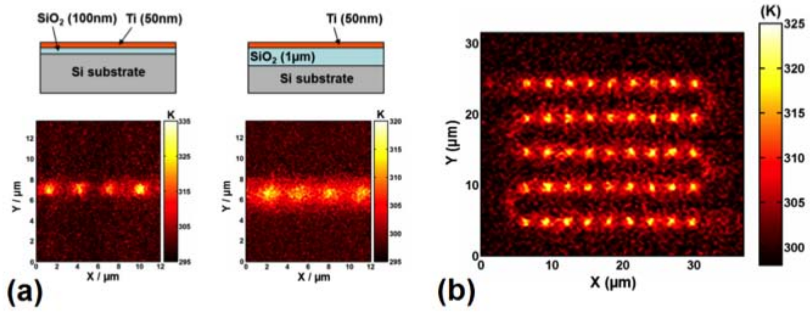


Figure 6: (a) Temperature distribution on nanostructured microwires as a function of the oxide thickness; (b) Temperature distribution on a serpentine-like nanostructured nanoheater [15].

5 Nanothermics

(L. Aigouy, L. Billot)

Thanks to the strong influence of temperature on the fluorescence emission, we have used the fluorescent particle-based tip as a scanning thermal probe. This enabled us to image the temperature distribution near electrically excited micro and nanowires with a lateral resolution better than 100nm. We are able to determine the absolute temperature of the nanostructures with a very good accuracy [55].

5.1 Hot spots in nanostructured microstripes

We have studied the influence of the substrate thermal conductivity on the temperature distribution near nanostructured microwires (see Figure 6) and designed serpentine-like nanostructured microwires (Fig. 6) for making nanoheaters with localized hot spots. We have also studied the degradation of sub 100nm-wide nanowires induced by a long electrical stress and observed the progressive appearance of hot spots [15].

6 High-TC Josephson nano-junction devices

(N. Bergeal, J. Lesueur)

In collaboration with the LPN-Marcoussis, our team has developed a powerful technique to structure High-Temperature Superconducting YBa2Cu3O7 thin films at the nanoscale combining advanced electronic lithography technique with ion irradiation . In particular we have been able to produce reliable and reproducible superconducting Josephson nano-junctions, which can be used as building blocks for more complex devices such as electronic circuits or photon detectors. After the first demonstration of the fabrication method on single junctions and SQUIDs, our work focused on the optimization of the characteristic parameters of the junctions in anticipation of their implementation in devices [242, 264, 263, 262, 209]. The main advantages of these junctions lie in the possibility to control easily the parameters of the junction (critical current and normal resistance) by changing the dose and the energy of the ions and in the high-density integration of the junctions on a chip. The current research focuses on the realization of Josephson mixer detectors for the terahertz range of frequency. The operation principle is based on the heterodyne mixing of the signal (frequency f_S) with a reference signal produced by a local oscillator and whose frequency f_{LO} can be tuned continuously over the range of interest. The signal at intermediate frequency $f_{IF} = f_S - f_{LO}$ can be read out directly by

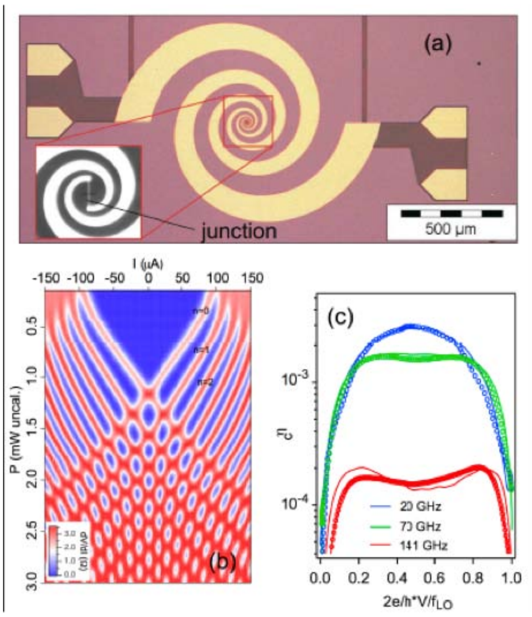


Figure 7: a) Picture of a Josephson junction embedded in a spiral THz antenna. b) Oscillations of the Shapiro steps as a function of the 20 GHz radiation power. c) Conversion efficiency of the Josephson mixer for different frequencies of the local oscillator.

suitable microwave instruments (band 4-8GHz). We have recently demonstrated the operation of a Josephson nano-junction made by irradiation in the range 20-140GHz in good agreement with theoretical prediction (see Figure 7) [156]. Ongoing measurements in the range 140-420 GHz should confirm these encouraging results. In a short term, mixing up to 1THz seems to be entirely within reach. At the same time, we also study the emission properties of single junctions and array of synchronized junctions in order to build a Josephson local oscillator whose frequency could be tuned by dc biasing.

Scientific input and output

International Collaborations:

Fakultaet Physik Experimentelle Physik 2, TU Dortmund, Germany (Manfred Bayer, Dimitry Yakovlev) - LAL, LMA (UCBL1) - VIRGO - Bariloche, Argentine (M. Sirena) - Instituto de Microelectronica de Madrid (IMM-CSIC) (A. Garcia-Martin, M.U. Gonzalez)

National Collaborations:

Service de Marquage Moléculaire et de Chimie Bio organique CEA/Saclay (E. Doris) - CAV, Nancy (L. Bolotine, F. Marchal) - CIC-IT, CHU Nancy (M. Beaumont) - ENS Cachan (G. Peyroche), UPS-IEF (N. Hildebrandt) - DSV/CEA, Saclay (E. Doris, E. Gravel) - NRL, USA (I. Medintz) - Université Paris Descartes (P. Dalko) - Institut Optique Graduate School (P. Lalanne) - ENSCP (M. Mortier) - LAAS (Christian Bergaud) - Ecole Centrale de Paris (S. Volz) - Groupe d'étude de la Matière Condensée, CNRS UMR8635, Université de Versailles Saint Quentin (Jean-Pierre Hermier) - UMR CNRS 7653, Laboratoire Hétéroéléments et Coordination (N. Mézailles) - URA CNRS 2582, Unité d'Analyse d'Images Quantitative (J.-C. Olivo-Marin) - UMR CNRS 8502, Laboratoire de Physique des Solides, Université Paris-Sud XI, 91405 Orsay (Mathieu Kociak) - LPN, Marcoussis (G. Faini, C. Ulysse) - UMR Thales- CNRS (J. Briatico, R. Bernard, D. Creté, Y. Lemaitre, B. Marcillac) - IMEP-LAHC, Chamberry (P. Febvre) -

Grants and contracts:

ANR CORE/SHELL - ANR DELIGHT - ANR EVALON - ANR NANOTHERMOFLUO - ANR QUANTICON - ANR SENOQI - ANR Resolution XXL - CNANO IDF NANOFLEG - CNANO IDF NanoPlasmAA - CNANO IDF NBNC - Projet NanoCTC - Projet Européen Nanomagma - VIRGO - SOLARWELL - St Gobain -

Highly Qualified people training:

PhD:

S. Bouccara "Détection résolue en temps de cellules tumorales circulantes in vivo",

C. Bouet "Développement de dispositifs photovoltaïques nanostructurés par voie chimique en solution organique",

E. Cassette "Synthèse et caractérisation de nanocristaux semi-conducteurs fluorescents dans le proche IR et à toxicité réduite pour l'image in vivo",

A. Dijksman "Spectroscopie de boîtes quantiques colloïdales de forme contrôlée",

P.F. Gardeazabal-Rodriguez "Développement d'une technique d'imagerie 3D haute résolution",

S. Helas-Othenin "La chimie des quantum dots pour une nouvelle génération de cellules photovoltaïques à haut rendement",

S. Ithurria "Synthèses et caractérisations de nanoparticules de semi-conducteurs II-VI de géométries contrôlées",

C. Javaux "Synthèse de nanoparticules de semiconducteurs colloïdales et étude de leurs propriétés de fluorescence",

B. Mahler "Synthèse et caractérisation de nanocristaux colloïdaux de semiconducteurs II-VI à structure coeur/coque. Contrôle de la cristallinité et des propriétés d'émission",

M. Malnou "Détecteurs THz en supraconducteur à haute Tc",

E. Muro "Quantum dots pour le ciblage en cellules vivantes et la microscopie Hilo Bicouleur",

E. Saidi "Etude de l'échauffement de micro et nanofils par microscopie thermique à particule fluorescente",

B. Samson "Imagerie thermique par microscopie en champ proche à sonde fluorescente",

G. Sitbon "Synthèse de sondes pour l'imagerie multimodale",

M. Tessier "Puits quantiques colloïdaux: synthèse et spectroscopie",

P. Vermeulen "Microscopie à illumination structurée et optique adaptative pour l'imagerie de fluorescence 3D",

T. Wolf "Etude de nanojonctions Josephson et nanofils supraconducteurs à haute Tc en vue d'applications THz",

Post-doct:

L. Biadala
L. Lalouat
B. Mahler
B. Nadal
W. Pan
M. Sirena

Publications: 73 ACL, 4 patents, 16 invited talkS

Strongly correlated and low dimensionality electronic systems

Permanent staff: H. Arribart, H. Aubin, K. Behnia, N. Bergeal, B. Fauqué, B. Leridon, J. Lesueur, R. Lobo, A. Trokiner, A. Zimmers

Non permanent staff:

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Key words: Strongly correlated materials, superconductivity, optical spectroscopy, NMR, transport properties, oxides, multiferroic, cuprates, manganites, semi-metals

Scope and Organisation

For decades, the Fermi liquid description of electrons in solids has been a powerful paradigm to explore the properties of most of the metals and semi-conductors: it lead to a comprehensive description of numerous properties of the materials, and a efficient way to use them for applications. In the recent years, new materials appear, among them High Tc superconductors, manganites, multiferroic systems, which display strong electronic correlations, and therefore, whose behavior strongly departs from the Landau description of solids. Moreover, they present very interesting features for applications. In addition to strong correlations, low dimensionality often plays a significant role. Researchers at LPEM use advanced experimental tools to address the exciting fundamental questions raised by these new materials. Their research is mainly oriented towards ground state properties and their fluctuations, and therefore requires low temperature measurements. Electronic and thermo-electric probes, but also magnetometry, NMR and optical spectroscopy, near field detectors are used to explore those materials. Research is organized as small teams with two to technique and focus on the most debated subjects in solid state physics. They are very often at the frontier of science, and their results are published in high impact factor journals. They share some equipment and techniques, and sometimes gather to apply to grants, as for example the Sesame program of Ile de France to buy PPMS and SQUID VSM equipment.

1 Fluctuating superconductivity

After a quarter century of research, the pairing mechanism in high-T_c cuprate superconductors (HTSc) is still unknown. The nature of the normal state pseudogap has been a subject of intense debate. In this context, understanding the superconducting fluctuations above T_c is crucial. If the pseudogap is a precursor feature of the superconducting phase, associated with preformed pairs, then it should be accompanied by vortex-like phase fluctuations and not conventional gaussian fluctuations of both amplitude and phase. Several experiments in our laboratory have been performed to address this issue. They all converge suggesting that superconducting fluctuations above T_c in cuprates are not fundamentally different from other superconductors.

1.1 Search for preformed pairs in high T_c cuprates

(N. Bergeal, J. Lesueur)

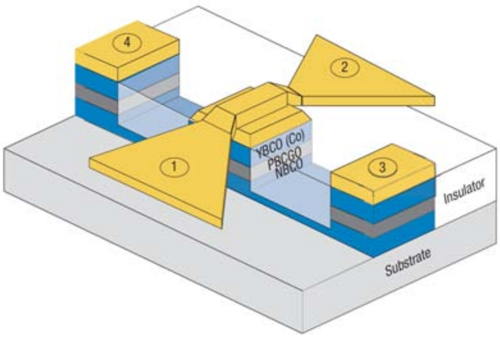

Figure 8: Schematic view of a c axis YBa2Cu2.8Co0.2O7 (100 nm)/ PrBa2Cu2.8Ga0.2O7 (30 or 50 nm)/NdBa2Cu3O7 (200 nm) junction. For the sake of clarity, the insulating part in front of the junction is not represented. Each junction is protected by an in situ gold layer and is connected with four electrodes (1 and 2 on the top, 3 and 4 at the bottom).

In underdoped cuprates, i.e. those with a doping level lower than the one providing the maximum T_c, a loss in the single particle excitations spectrum has been evidenced by numerous experimental probes, both in the charge and in the spin channels, below a characteristic temperature T^* higher than T_c. This "pseudo-gap" regime has been often proposed as a proof that pairing in cuprates occurs at high temperature (T^*), whereas long range phase coherence, and therefore true superconductivity, takes place at T_c, as opposed to the BCS scenario where pairing and condensation are simultaneous. The loss in the density of states would be due to the presence of pre-formed pairs, but no direct probe of these fluctuating pairs was available. We built-up a pseudo-Josephson experiment to directly measure these pairs in the pseudogap regime of the $YBa_2Cu_3O_7$ superconductor (see figure 8). A junction between an optimally doped HTSc and an underdoped one has been measured between the two corresponding T_c, and a specific signature of fluctuating pairs in the current-voltage characteristic recorded. Our conclusion is that this signal vanishes well below T^* of the underdoped material, and follows the temperature and voltage dependence expected from regular thermodynamic fluctuations in a BCS superconductor [217]. We therefore propose that the pseudo-gap cannot be related to preformed pairs, and may originate from a competing order.

1.2 Paraconductivity in high T$_c$ superconductors

(B. Leridon)

We have performed pulsed high magnetic field experiments on underdoped$La_{2-x}Sr_xCuO_4$ thin films in order to obtain the normal state resistivity (under 49T) and we have extracted the paraconductivity (conductivity due to fluctuations). The obtained paraconductivity is remarkably close the the Aslamazov-Larkin predictions for 2D gaussian fluctuations therefore pointing toward the absence of sizable phase fluctuations [253]. Observing the exact prefactor with $\epsilon = Ln(T/T_C)$ is an indication of coupling between relatively free fermions [198]. This work was done in collaboration with Moshchalkov group in Leuven and Grilli group in Rome.

1.3 The Nernst signal produced by gaussian superconducting fluctuations

(H. Aubin, K. Behnia, J. Lesueur)

The observation of a sizeable Nernst signal was believed to be as evidence for preformed Cooper pairs in underdoped cuprates. But what about conventional superconductors ? Between 2006 and 2009, we explored superconducting fluctuations in disordered thin films, which host a dirty conventional BCS superconductor in the 2D limit. This exploration began with the discovery of the superconductor-to-insulator transition in Nb_xSi_{1-x}[1].

A Nernst signal generated by gaussian fluctuations of the superconducting order parameter could be detected in a temperature range 30 times above the superconducting critical temperature[2,] [259, 206]. The magnitude of this signal was in excellent agreement with the theoretical predictions for gaussian fluctuations. Moreover in both Nb_xSi_{1-x} and the low-density superconductor InO_x [235], Nernst effect can directly detect a field scale in the normal-state associated with superconducting fluctuations known as the ghost critical field.

2 Iron-based superconductors

In the pre-high-T_c era, "common sense" said that superconductivity and magnetism were mutually exclusive. Little did we know that Hosono's group[3] would find superconductivity in another non-copper oxide material based, precisely, on iron and arsenic. These so-called pnictides have already reached critical temperatures over 50 K and have a phase diagram which seems to be strongly driven by magnetism. A global picture of these materials is still not achieved. They are definitely more complex than conventional superconductors, but many successful theoretical predictions were made based on electronic structure calculations. Two experiments, one probing superconducting states and one focusing on the normal state, have been performed in our laboratory.

2.1 The gap symmetry in iron-pnictide superconductors

(R. Lobo)

One of the key questions in superconductors concerns the gap symmetry. Iron pnictides are a fascinating system in this sense as they are multiband superconductors and the main stream theoretical proposition is that these materials would have a $s_\pm$ symmetry. This means that each band would have a convention gap but that this gap would change sign (or rather have a phase shift of π) between bands. The $s_\pm$ symmetry allows for an interesting property. Non magnetic impurity scattering can create nodes in the gap. The idea is that if carriers are scattered from a band having a s_+ gap to another having a s_- gap, they will annihilate each other.

That is precisely what we observe when comparing the infrared spectra of $BaFe_2As_2$ doped with either K in Ba sites or $Co\ in\ Fe$ sites. Figure 9 shows the optical conductivity of Co and K doped $BaFe_2As_2$ [186]. In the former, Co atoms go into the $FeAs$ planes and act as impurity centers inducing pair breaking if the gap symmetry is $s_\pm$. This leads to a residual sub-gap optical conductivity, indicated by the shaded area. Conversely, K atoms go out of $FeAs$ planes and hence no extra unpaired quasiparticles are expected. This is shown by the negligible low energy optical conductivity in this compound. This result is a very strong experimental support for the $s_\pm$ symmetry in iron pnictides.

2.2 Thermoelectricity of an iron-based superconductor

(K. Behnia)

[1]H. Aubin et al., Phys. Rev. B, **73**, 094521 (2006)
[2]A. Pourret et al., Nature Physics, **2**, 683 (2006)
[3]Y. Kamihara et al., JACS, **130**, 3296 (2008)

We have measured the Nernst and Seebeck effect in the $FeSe_{1-x}Te_x$ family of iron-based superconductors [172]. The Seebeck coefficient allows to extract a Fermi temperature. By systematic consideration of various bulk properties (thermopower and specific heat, upper critical field and penetration depth), we deduce that the carrier density is low (0.05 carrier per formula unit) and the electrons have a sizeable effective mass (about 20 m_e). The results suggest that the distance between Cooper pairs in this system is barely longer than the inter-electron distance. Thus, this superconductor lies close to the BCS-BEC cross-over. The critical temperature is almost one-tenth of the Fermi temperature, one of the highest ratios known in any superconductor.

3 Superconductivity at oxides interfaces

(N. Bergeal, J. Lesueur)

In 2004, Ohtomo et Hwang[4] showed that the interface between two insulators can be highly conductive. At the interface between $SrTiO_3$ and $LaAlO_3$, a two-dimensional electron gas (2-DEG) takes place within a few nanometers, whose mobility can exceed $10^4 cm^2/Vs$. Soon after, it has been shown that this 2-DEG is a superconductor at 250 mK, whose carrier density can be tuned by a gate voltage. These discoveries open a new and fruitful domain for both basic and applied research, sometimes called "oxitronics". Other $SrTiO_3$ based heterostructures display similar behavior. We discovered that the interface between the band insulator $SrTiO_3$ and the Mott insulator $LaTiO_3$ is superconducting [184]and explored its phase diagram as a function of temperature and gate voltage (see figure 10). By measuring the transport properties at very high magnetic field (45 T), we evidenced that in addition to low mobility carriers always present in the 2-DEG, highly mobile carriers develop for positive gate voltage, that is, when superconductivity shows-up [151]. We built a self-consistent model of the 2-DEG which accounts for these experimental facts, and proposed that superconductivity is triggered by the occurrence of highly mobile carriers which set at the boarder of the quantum well. In addition, we explored the dynamics of the 2-DEG upon voltage biasing, and showed that carriers thermally escape from the well formed by the conduction band-bending of $SrTiO_3$ [152]. The magneto-transport in the 2-DEG has been measured at low temperature, where a weak localization behavior can be seen. A detailed analysis shows that the spin-orbit coupling can be tuned by the gate voltage, and could be due to a Rashba type of coupling.

4 Other superconducting phenomena

4.1 Anomalous Josephson effect

(B. Leridon)

We have proposed to measure the Josephson current between a conventional superconductor and a superconductor in which time and inversion symmetry are simultaneously broken. In such superconductors, the distinction between odd and even parity is blurred and the order parameter is an admixture of s or d-wave and i times p-wave. We have calculated the current and shown that in this case, the symmetry in field is lost around H=0 and the current shows oscillations with no decrease in 1/H [252]. This effect has been experimentally observed in a heavy fermion compound $CePt_3Si$ by another group[5] and might apply to high T_c superconductors.

[4] Ohtomo and H.Y. Hwang, Nature, **427**, 423 (2009)
[5] A. Sumiyama et al., J. Phys. Soc. Jpn., **74**, 3041 (2005)

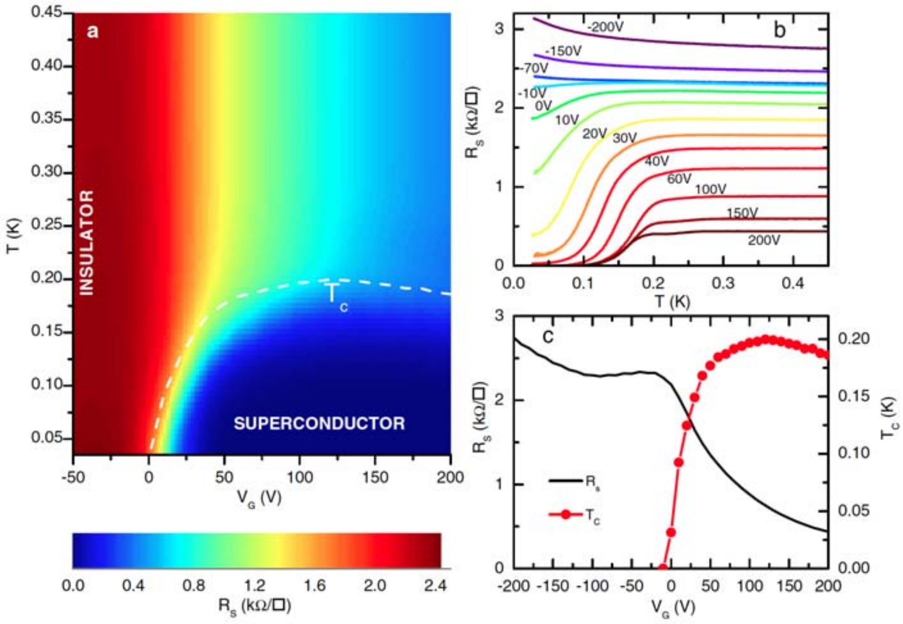

Figure 10: Superconducting transition as a function of gate voltage (a) Sheet resistance in color scale as a function of temperature and gate voltage from -50 V to +200 V. (b) Sheet resistance as a function of temperature for selected gate voltages. (c) Sheet resistance at 450 mK (left scale) and superconducting transition temperature (right scale) as a function of gate voltage.

4.2 Phase transition, satellite phase and oxygen ordering in $YBa_2Cu_3O_{7-\delta}$

(B. Leridon)

We have studied the dc magnetization of $YBa_2Cu_3O_{7-\delta}$ polycrystalline samples of various doping. A change of slope in the susceptibility versus temperature has been evidenced at a temperature close to the temperature of observation of a symmetry breaking from polarized neutron scattering experiments done by another group. These observations point toward the existence of a phase transition in this part of the phase diagram [203].

Furthermore, our study evidenced a new magnetic transition at high temperature (336K) in a satellite phase of $YBa_2Cu_3O_{7-\delta}$, namely $BaCu_3O_4$. This transition is probably due to AF ordering of the Cu spins in diamond chains of Cu and O atoms. Remarkably enough, these chains bear a net magnetic moment and are weakly coupled to one another [158].

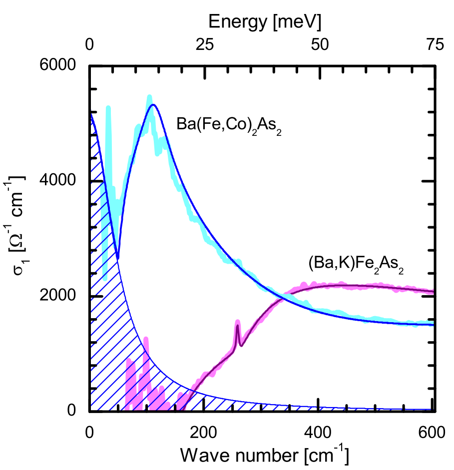

Figure 9: Optical conductivity of Co and K doped $BaFe_2As_2$

We have also quantitatively studied the oxygen order in the chains in $YBa_2Cu_3O_{7-\delta}$ through the variation of paramagnetic Curie term, in relation to the pseudogap susceptibility and the superconducting critical temperature. The order has been varied by changing the annealing conditions without changing the overall stoechiometry in oxygen [153].

4.3 Interplay of Anisotropic scattering and superconductivity in URu_2Si_2

(K. Behnia)

In contrast to almost all anisotropic superconductors, the upper critical field of URu_2Si_2 is larger when the field is oriented along the less conducting direction. We have performed a study of resistivity and Seebeck coefficient of the normal state extended down to sub-Kelvin temperature range by destroying superconductivity with a magnetic field. The study uncovers a singular case of anisotropy. When the current is injected along the c-axis URu_2Si_2 behaves as a low-density Fermi liquid. In contrast, when the current flows along the a axis, even in presence of a large field, neither the resistivity nor the Seebeck coefficient do not display canonical Fermi-liquid behavior even at the lowest measured temperatures. Thus, the characteristic energy scale is anisotropic and vanishingly small in the basal plane [213].

5 Nernst effect in semi-metals

Because they host an extremely dilute gas of highly mobile electrons, semi-metals such as bismuth and graphite dwarf any other metal with their Nernst response [197]. During the last five years, the study of the Nernst response in a strong magnetic field has led to a number of interesting results.

5.1 Nernst quantum oscillations: a new window to the Landau spectrum

(K. Behnia, B. Fauqué)

In 2007, we found that the Nernst response in bismuth presents giant quantum oscillations in the vicinity of the quantum limit [241]. This limit is attained when the magnetic field is strong enough to confine electrons to their lowest Landau level. For ordinary bulk metals, the size of the required magnetic field is well beyond the limits of current technology. But in a low-density metal, this limit can be attained with an accessible magnetic field.

In 2009, our measurements of the Nernst effect in graphite [196] resolved a signal sharply peaking each time a Landau tube exits the Fermi surface. The profile of these quantum oscillations is qualitatively different from what was seen in the case of graphene by other groups. While in graphite each oscillation consists of an asymmetric peak, in graphene, two peaks of opposite signs sandwich a vanishing signal. A theoretical treatment of this qualitative difference between 2D and 3D cases has been recently proposed.

5.2 The three-dimensional electron gas beyond the quantum limit

(K. Behnia, B. Fauqué)

The extreme sensitivity of the Nernst effect makes it an ideal tool to explore the high-field Landau spectrum [239]. We have designed a set-up to measure the Nernst effect for a magnetic field oriented along an arbitrary orientation up to 28T. Several months of exploration allowed to us to map the angle-resolved Landau spectrum of bismuth up to 28 T. This spectrum becomes complex in the vicinity of the quantum limit for several reasons. To preserve charge neutrality, emptying a Landau level of one type of carriers, should be compensated with an equal change in the density of the carriers of the other sign. The chemical potential shifts as the magnetic field is scanned and quantum oscillations are no longer strictly periodic in B^{-1}. Moreover, the Zeeman

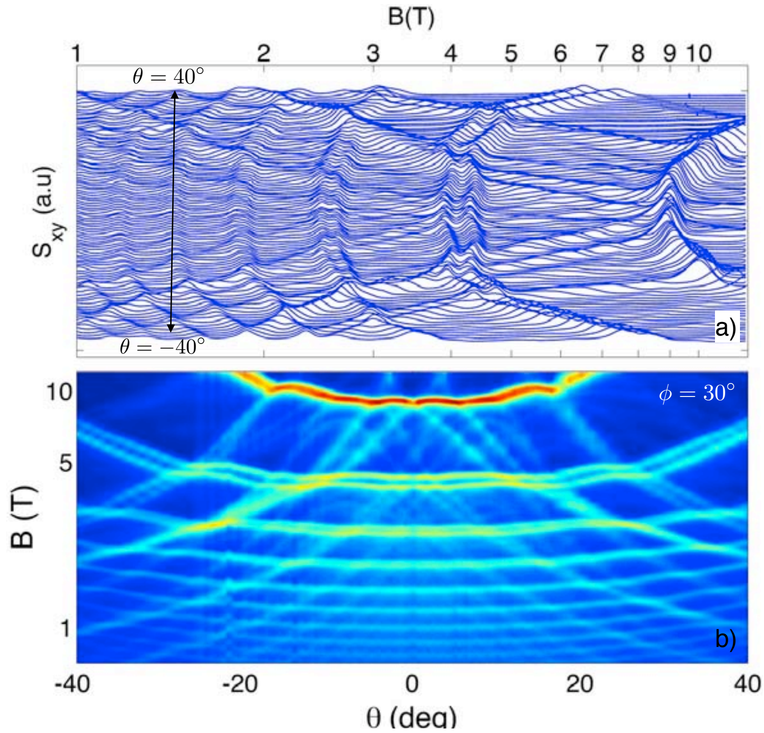

Figure 11: Top: Nernst quantum oscillations in bismuth; Bottom: the color plot of the Nernst data reveals the angle-resolved Landau spectrum.

energy is drastically angle dependent. For both electrons and holes, the anisotropy of the "spin mass" is different from the anisotropy of the cyclotron mass. By comparing experiment and theory the optimal band parameters can be pinned down [178].

Our high-field measurements on bismuth and graphite document a divergence between the fate of the three-dimensional electron gas pushed beyond the quantum limit. In the case of graphite a thermodynamic phase transition destroying metallicity occurs at a temperature-dependent magnetic field. This leads to a sudden drop in the Nernst response [166], which may be attributed to the destruction of Landau tubes by nesting. On the other hand, in bismuth, the complex phase diagram upto 28 T can be explained by the one-particle theory.

5.3 Dirac valleys in bismuth and angle-resolved magnetoresistance

(K. Behnia, B. Fauqué)

In 2011, we discovered that the presence of Dirac valleys in bismuth leads to a particularly robust angle-dependent magnetoresistance [162]. The magnetoconductivity can be explained as a sum of contributions by three extremely anisotropic electron pockets and the isotropic contribution of the hole pocket. The anisotropy of the electron pockets is such that orbital magnetoresistance can play the role of a valley valve. In absence of magnetic field, the three electron valleys are degenerate, but the degeneracy is lifted with the application of an in-plane magnetic field. We also found that in all samples studied, the threefold symmetry of the lattice is spontaneously lost as the temperature is lowered or the magnetic field increased. This observation may be the first experimental manifestation of the recently proposed valley-nematic Fermi liquid state.

6 Multiferroics

(R. Lobo)

Lattice dynamics and magneto electric coupling in multiferroic compounds Magneto-electric multiferroic materials show the coexistence of ferroelectricity with a magnetic ordering, either ferromagnetism or, more commonly, antiferromagnetism. The interplay between these two order parameters open a myriad of applications in information storage and spintronics. Of particular interest are, so-called, class II systems where ferroelectricity is a consequence of an incommensurate magnetic order. However, the underlying mechanism of the physical interactions leading to the magneto-electric coupling in multiferroics is not yet understood.

Utilizing optical spectroscopy, we address several open questions about these materials: (i) is the link between the magnetic and the ferroelectric order parameters a direct coupling of electronic charge and spin degrees of freedom or is there a mingling third party, such as ferroelasticity and magnetostriction that connect these quantities ? (ii) Recently, a new excitation specific to multiferroics, called electromagnon, was found in the far infrared. Is this particle responsible for the magnetoelectric coupling or is it rather a consequence of it ? Is it restricted to multiferroic materials? (iii) Is a class II system ferroelectricity conventional in the sense that it is provided by moving ions away from their equilibrium position ?

The optical conductivity addresses these issues by looking at the phonon and electromagnon spectral response and analyzing the renormalization of their spectral response as well as spectral weight transfer between different excitations. Amongst our studies, we can cite two particularly interesting results.

In $BiFeO_3$, a class I multiferroic, we followed the infrared temperature dependence of phonons [256] to show that the ferroelectric transition is soft mode driven. These results were further confirmed by Raman spectrocopy [208].

In class II multiferroic $TbMnO_3$ [187], we showed that the electromagnon is actually build upon two particular phonons. When crossing the antiferromagnetic transition a new excitation, electromagnon, appears in the far infrared. Through a spectral weight transfer analysis, we showed that this electromagnon is built from two specific phonons. This observation leads to the conclusion that the electromagnon is a magnon-phonon hybrid mode instead of a new stand alone particle.

7 Manganites

(A. Trokiner)

The mixed valancy of Mn in manganites leads to a rich phase diagram where metallic, insulating, orbital ordered states and colossal magnetoresistance can be found. NMR spectroscopy is a powerful tool to explore these states and understand the microscopic origin of the phase transitions.

Among our results, the study of magnetic polarons in $CaMnO_3$ is detailed below.

In the AF state [210]**:** In $CaMnO_{3.00}$ only$Mn^{4+}i$ons should exist. Its ground state is the AF, G-type state. Up to now, it has been difficult to synthesize a sample without oxygen vacancies. These O vacancies create Mn^{3+}ions with additional d-electrons (e_g) in the compound, raising the question on the effect of doped electrons in an AF spin lattice of compounds with mixed valences. A homogeneous canted AF phase was first predicted but it was shown later that in doped manganites an electron phase separation was more favourable. In particular, at low doping, the AF lattice of localized spins should coexist with micro size areas of a modified phase, referred to as magnetic polarons. The magnetic polaron contains one electron "dressed" with a ferromagnetic (FM) polarized cloud of neighbouring magnetic ions.

Our $CaMnO_{3-x}$ polycrystalline sample has the smallest vacancies amount, $x<0.01$, with a high Néel temperature (T_N=123 K). ^{17}O NMR spectroscopy evidences the existence of FM domains embedded in the AF spin lattice. Furthermore, the almost temperature independence of the magnetic moment in the FM entities together with the thermally activated spin dynamics agree with the model of small size self trapped magnetic polarons. Finally, we find that the magnetic polarons start to move above 40 K in a slow diffusion regime, keeping their static properties almost unchanged.

In the paramagnetic state **[192]:** $CaMnO_{3-x}$was studied by ^{17}O NMR and by magnetic susceptibility up to $T = 670K$. Above 160 K, the spin density of the itinerant electrons is equally shared between the Mn^{+4} ions. Below 160 K, the spin density of the doped electrons (e_g) becomes inhomogeneously distributed since some of them start to be involved in strong correlations with the localized Mn spins. Thus, a separation of the doped electrons into slow carriers, forming the magnetic polarons, and fast carriers develops few tens of degrees above T_N.

8 Electrons in nanoparticles

8.1 Metallic and superconducting nanoparticles

(H. Aubin)

The study of quantum phenomena in disordered systems was also pursued by developing new model systems based on chemically synthesized nanoparticles. These works on nanoparticles led to the development of a new original synthesis of superconducting lead(Pb) particles [207] (See Figure 12), the development of a method for preparing Langmuir films of gold nanoparticles, where the tunnel barrier transparency between gold nanoparticles is controlled by organic ligands of different lengths [170], and the development of a method for embedding metallic nanocrystals in a semiconducting matrix [157]. Measurements of electronic transport properties of gold nanoparticles arrays at low temperature allowed showing that electron co-tunneling led to effective long range hopping and, consequently, to variable range hopping laws (Efros-Shklowskii) . Because quantum electronic transport in disordered materials is intimately related to the local electronic spectrum, recent works in the laboratory focused on the developments of methods to realize the tunnel spectroscopy of nanoparticles[6].

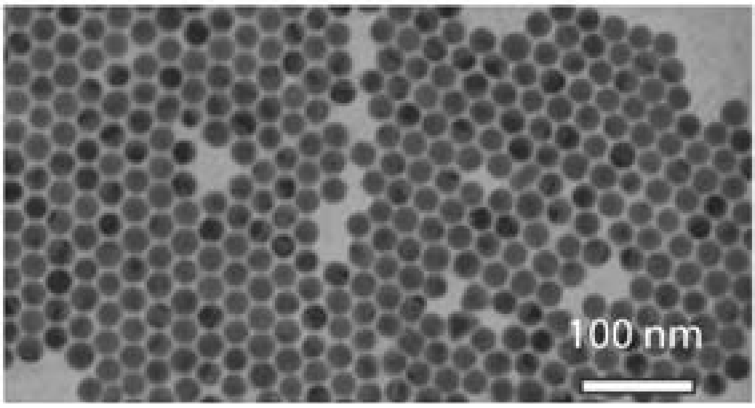

Figure 12: Superconducting Lead Nanoparticles synthesized in the laboratory.

[6]Q. Yu, C. Ulysse, A. Zimmers and H. Aubin, Submitted (2012)

8.2 Correlated electrons out of equilibrium

(H. Aubin, A. Zimmers)

A second theme of research is the study of out-of-equilibrium transport properties of correlated materials. The Zener breakdown of band insulators is a well-known phenomenon where a large electric field can promote "horizontal" inter-band tunneling, allowing electronic transport in otherwise insulating materials. In correlated insulators such as Mott insulators or charge ordered insulators, a large electric field induces a breakdown of the collective electronic order, whose theoretical description requires a many-body version of the Zener breakdown. Several observations of switching in current-voltage characteristics of magnetite (Fe_3O_4) and VO_2 have been reported and interpreted as a possible electric field induced breakdown of electronic orders in those materials. Using a laboratory-made Electrostatic Force Microscope (EFM), we have shown that electric-field induced insulator to metal transitions could be observed in the EFM signal. This observation, made in the absence of electronic current flowing between the tip and the sample, shows that electronic orders induced by Coulomb interaction can be broken down by a large electric field [177]. Using this EFM, we also developed recently a method to extract the quantum capacitance of nanoparticles, which we applied to magnetite (Fe_3O_4) nanoparticles[7].

Scientific input and output

International collaborations:

ECOS-Sud Argentine, University of Bariloche, Argentine, (M. Sirena) - CEFIPRA France-Inde (2012), NPL, Dehli, Inde, (R.C. Budhani) - Institute of metal Physics, Ekaterinburg, russie (S. Verkhovskii, A. Gerachshenko) - Kurchatov Institute of Atomic Energy, Moscou, Russie (A. Yakubovskii) - Department of Physics and Astronomy, State University of New Jersey, Rutgers, USA (S-W.Cheong) - Physikalisches Institut, Universität zu Köln, Köln, Germany (D. Khomskii) - Department of Physics, Northern Illinois University, DeKalb, USA (B. Dabrowski) - Institute of Solid State Chemistry, Ekaterinburg, Russia (V. Kozhevnikov, I. Leonidov) - La Sapienza, Rome, Italie (M Grilli, S Caprara, L Benfatto, C di Castro, C Castellani) - Osaka Univeristy (Japon) (Yuki Fuseya) - Ewha University, Corée du Sud (Pr. Woun Kang) - University of California, Riverside, USA (C. M. Varma) - UK Leuven, Belgique (V.V. Moshchalkov, J. Vanacken) - McMaster University, Canada (T. Timusk, J.P. Carbotte) - Brokhaven National Laboratory, NY, USA (C.C. Hommes) - Chinese Academy of Sciences, Beijing, China (X.G. Qiu) - UFMG, Brazil (R.L. Moreira)

National collaborations:

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[7] A. Mottaghizadeh et al., Submitted (2012)

Grants and contracts:

ANR CAMELEON - ANR COCOTRANS - ANR DELICE - ANR GAPSUPRA - ANR QUANTHERM - ANR QUANTICON - CNANO IDF Super-2DEG/EMQ - CNANO IdF JN2 : G Faini (LPN-CNRS), J Briatico (Thales-CNRS), S. Djordjevic (LNE) - Contrat EMERGENCE Ville de Paris - Contrat SESAME

Highly Qualified People training:

PhD:

J. Biscaras "Supraconductivité bidimensionnelle à l'interface de deux oxides isolants",

A. Collaudin "Etude des électrons de Dirac dans Bismuth sous fort champ magnétique,

Y. Dai "Supraconducteurs à hautes températures critiques",

S. Hurand "Nanostructures supraconductrices dans des gaz d'électrons bidimensionnels à l'interface d'oxydes",

X. Lin ,

H. Moreira "Confinement quantique dans les nanocristaux supraconducteurs et transport électronique dans les réseaux de nanocristaux métalliques",

A. Mottaghizadeh "AFM/STM spectroscopie des matériaux à forte corrélation électronique",

R. Schleck "Etude du rôle joué par le réseau cristallin dans le couplage magnéto-électrique des matériaux multiferroïques",

T. Wolf "Etude de nanojonctions Josephson et nanofils supraconducteurs à haute Tc en vue d'applications THz",

Q. Yu "Spectroscopie de nanocristaux à basse température",

Post-doc:

P.L. Lang

Z. Zhu

Publications: 109 ACL, 60 invited talks

Instrumentation

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Post-doc: Y. Felada, C. Margo, F. Sepulveda , M. Streza

Key words: Instrumentation, electrostatic, charge localization, RF devices, meta-materials, sensors, gravitational waves

Scope and Organisation

Sophisticated instrumentation for scientific applications and sensors for industrial applications are developed within the instrumentation thematics. All the research activity is built around the electromagnetism, from the static (electrostatics, magnetostatics) to optics through microwaves and infrared. The strength of this activity is the equilibrium between academic research on advanced instrumentation and strong industrial partnerships. This leads to publications in regular scientific journals on one side, and numerous patents on the other side.

Researchers involved in the instrumentation thematic come from different ESPCI laboratories, particularly from the former UPR5 (LSLP, led by D. Fournier) and from the former Electrical Engineering Laboratory (LEG, led by J. Lewiner). Their activity progressively merges with the main part of LPEM, and internal collaborative pieces of work are at play. They use to work as a strongly correlated team, sharing expertise and equipment on the different projects, and with a collective strong implication in PhD student education.

In addition to this all-integrated research on instrumentation, LPEM is also involved in the VIRGO project through V Loriette and I Maksimovic. The unique expertise in optical instrumentation developed for decades in the laboratory is required to build the new Advanced VIRGO instruments, through the CALVA project.

1 Electrostatic instrumentation and sensors

(S. Holé, J. Lucas)

Under high voltage, charges can be injected from electrodes and accumulate inside an insulator. Some parts of the insulator are then submitted to an electric field higher than the nominal electric field, a situation that can lead to breakdown and even to failures in the system in which the insulator is included. Understanding charge phenomena requires sophisticated non-destructive measurement systems and precise signal analyzes. We are particularly involved in this two points by proposing precise measurement models for the three main direct measurement methods and simple ways to analyze the different contributions to the signal in order to avoid any misinterpretations, specially in non-uniform materials. In that latter case we had shown that the signal produced by any of the three main direct measurement methods is drastically impacted by non-uniformity in the tested material [406]. As an illustration a typical signal with a laminate material under voltage is shown in Figure 13a in which spurious oscillations are mixed with charge signal. The main difficulty with non-uniform materials is the assumption of their internal structure which may induces errors in the charge estimation. Nevertheless we have shown that a judicious calibration measurement can be used as an assumption of the internal structure of the non-uniform material [479, 478, 387]. The estimation of the charge distribution is then much less erratic and the signal can be treated as if it were obtained with a uniform material. The treated signal in Figure 13b shows indeed a rapid charge buildup at the anode and a slower at the anode.

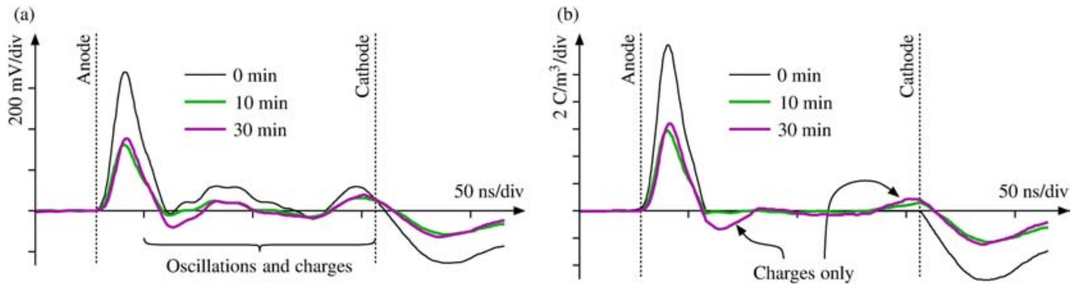

Figure 13: (a) Typical signal in a non-uniform material. Oscillations are not due to charges but to non uniformity. (c) Charge distribution estimation in a non-uniform material after corrections.

The measurement of charge distribution has attracted numerous industrial collaborations (SAGEM, GE Healthcare) for studying specific materials as well as academic collaborations (LAPLACE, IES) to work on measurement techniques in order to improve spatial resolution [434, 481, 494] by a factor 10 at least and to reach the nanometer scale.

The numerous modelings of the space charge distribution measurement methods have shed new light on various sensor physics, specially for capacitive sensors [447, 482, 483]. We have then derived analytic tools for estimating the sensitivity map of capacitive sensors and used them in the context of industrial collaborations (BOSTIK) and within the framework of the OMICAGE ANR program for estimating the water content in wood-chips, a promising sustainable energy fuel [487, 495].

Another recent achievement in the case of electrostatic sensors is the development of efficient ionic smoke sensors. It has to be said that too kinds of smoke sensor exists, optical smoke sensors and ionic smoke sensors. Conventional ionic smoke sensors are far more efficient, in terms of sensitivity and response time, than optical smoke sensors. However they usually use a minute quantity of americium 241, a radioelement that releases alpha particles to ionize surrounding air. That radioelement is no longer desirable, mainly because of recycling problems, then ionic smoke sensors are progressively forbidden in a lot of countries. In order to continue to benefit of the efficiency of these kinds of sensors we have proposed two kinds of ionic smoke sensors based on a corona discharge [474, 475]. Figure 14 shows the response to smoke of these two kinds of ionic smoke sensors which can respond more than one minute prior to optical sensors [477, 480, 496].

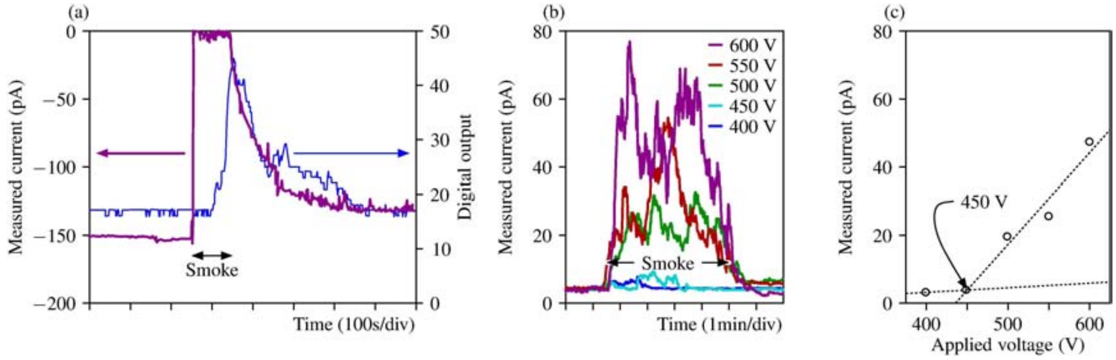

Figure 14: (a) Smoke detection by the reduction of the current in a drift chamber as soon as smoke enters the sensor. Comparison with an industrial optical sensor which responds more than one minute after our ionic smoke sensor. (b) Smoke detection by a corona discharge triggering. A discharge is triggered as soon as smoke enters the sensor. (c) The detection threshold is 450 V.

2 Low Field Nuclear Magnetic Resonance

(S. Holé, J. Lucas, C. Filloy-Corbrion)

It has been recently shown that NMR at very low field may have an important development both for spectroscopy and for imaging. Low cost but very efficient solutions can emerge, provided ultra-sensitive magnetic sensors are developed. Since HTSc SQUIDS have been recently mat at LPEM, we started developing a NMR spectrometer working in the earth field. That is a very challenging project since NMR signals are very small at earth field which requires high sensitive magnetic sensors, and noise is very important in open environment requiring particularly well suited instrumentation. We started from scratch with a commercial magnetic sensor (SQUID) working in liquid helium at about 4 K keeping in mind that it is a first step before using sensors developed at LPEM. We have designed a dedicated controllable superconducting switch to protect the SQUID during the magnetic polarization phase. That switch is currently under patent application consideration. We have also studied particular gradiometer in order to optimize the signal capture while reducing the noise [485] . Figure 15 presents our developed instrumentation and the first NMR signal obtained few month ago in the environment shown.

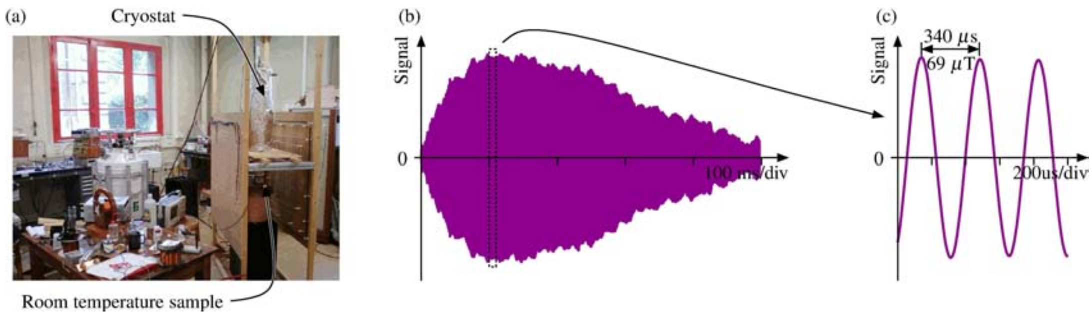

Figure 15: (a) Our low field Nuclear Magnetic Resonance instrumentation. (b) First observed signal in a completely open environment. (c) Part of the signal on a larger scale. The relaxation field is about 69 μT.

3 High frequency devices and sensors

(T. Ditchi, J. Lucas, E. Géron, S. Holé)

3.1 Adaptive antennas and meta-materials

In the microwave range, our first result during the period was the development of an adaptive antennae for DECT telecommunications (see Figure 16) [401, 492, 498]. The advantage of using adaptive antennas is to deliver energy only where necessary then increasing the range or to avoid perturbation sources then improving the transmission efficiency. The system novelty consists in the algorithm used to calculate gains and phases required at each antennae from a goal obtained by an automatic estimation of the user position and needs through the DECT traffic.

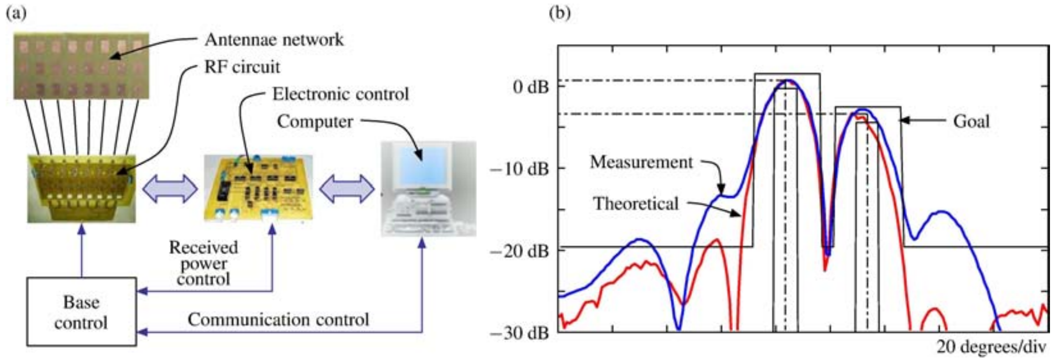

Figure 16: (a) Complete adaptive antennae system. (b) Obtained results for two users, one at 85° and the other at 115°.

With the progressive insertion of LEG researchers in LPEM during the period, we have gradually focused on the study of meta-materials for radio-communication applications. Meta-material are microstructure materials that have negative permeability and/or permittivity then exhibiting interesting characteristics such as forbidden propagation bands or phase propagation opposite to energy propagation. In the case of high frequency couplers for instance, conventional systems are made of two parallel lines of given length, the coupling factor directly depending on the gap between the lines and the operating bandwidth on the line length. We have shown that a structure made of one right-handed material line near a line made of consecutive right-handed and left-handed material cells, yields to a better coupling for a given gap which also depends on the number of cells as shown in Figure 17a. It has thus brought us to imagine and realize an electronically controlled coupler by PIN diodes which behavior is presented in Figure 17b [484, 376].

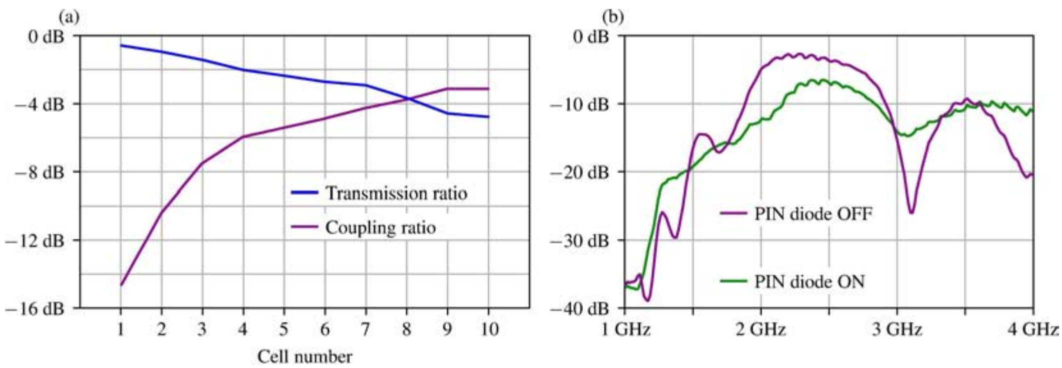

Figure 17: (a) Measured coupling factor and transmission factor as a function of the number of cells at 2.45 GHz. (b) Coupling factor as a function of frequency when the PIN diode connected between cells 2 and 3 is commutated. The coupler factor goes from -4 dB to −8 dB in the bandwidth, around 2.45 GHz.

For obtaining such results, it has been necessary to well understand the propagation of electromagnetic waves in complex linear meta-materials. For that purpose we have developed mathematical tools for recovering the signal phase when it is unreachable by conventional instruments, especially in forbidden bands. Though Kramers-Kronig relations connects real and imaginary parts of a causal signal, we have preferred to work on an adaptation to amplitude and phase of such relations since it is more appropriate for propagation phenomena [486]. As a result, Figure 18a presents a comparison between calculated and measured phases of a meta-material line. In the forbidden band the measurement shows erratic results whereas in the allowed band both calculation and measurement are in agreement. That also makes it possible to recover phase when only power measurements are possible as illustrated in Figure 18b.

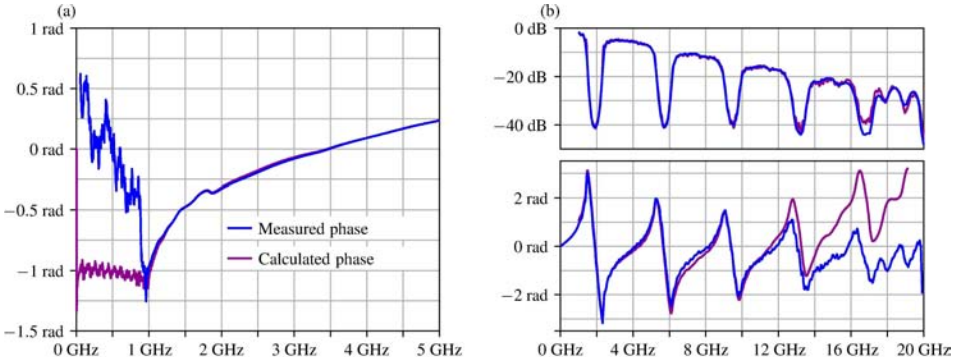

Figure 18: (a) Comparison between measurement and calculation. The measurement apparatus delivers erratic results below 1 GHz corresponding to a forbidden band. (b) Amplitude and phase comparison between a network and spectrum analyzers. The phase of the spectrum analyzer has been reconstructed from calculations.

3.2 Sensors

We have also worked on various sensors using microwaves through OMICAGE ANR project and a topic that has began with the ARCOS project.

The aim of the first project was to measure the water content in wood-chips during the shredding. It has to be said that wood-chips are one of the best renewable energy source since they require only 3% of energy input and the potential production in France can be as high as 10% of all the energy needs. But water content has to be measured for the development of the wood-chip industry. Figure 19a presents our results for the OMICAGE ANR project, one of the first realization of that kind [473].

During the ARCOS project, we had developed an efficient cooperative system for determining a vehicle position on the road in order to limit the number of run-off-road accidents which leads to almost 30% of fatalities in France [448]. However the system had some problems for practical industrialization, then we have continued to work on the subject to work around those problems. Figure 19b shows some preliminary results. A patent application is currently under consideration [470].

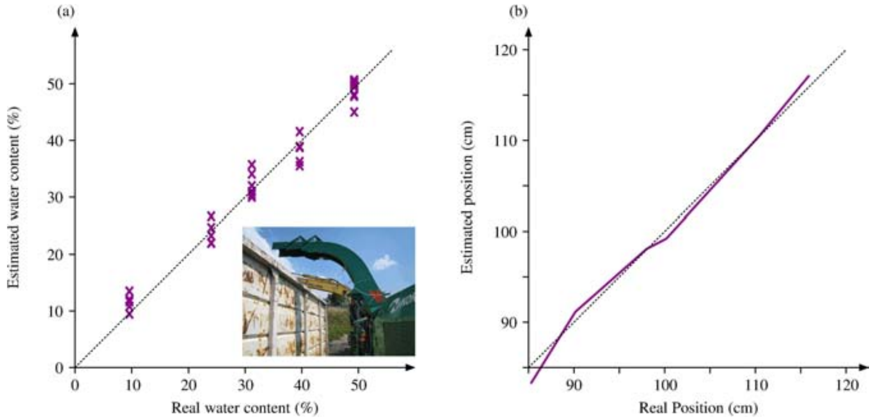

Figure 19: (a) Estimation of the water content in wood-chips during the shredding. The estimation error is only 5% for 4-s measurement. In the inset, a photography of our system into action in real conditions. (b) Estimation of a vehicle position by a cooperative approach to prevent run-off-road accidents. The estimation error is about 2 cm, a sufficient accuracy for real time position control and correction.

4 Infrared and optical instrumentation

4.1 Infrared imaging of defets

(C. Boué, S. Holé)

Instruments have been developed for measuring in continuous or harmonic regime the temperature map induced by a punctual source (for instance a laser beam focused on the sample surface) or larger sources such as flash lamps. The measurements are made without contact by a rapid infrared camera coupled to a computer. When samples are periodically heated, one part of the temperature evolution is synchronized with the excitation but another part, a slower one, is due to dissipation. We use a lock-in module for estimating the amplitude and phase of the temperature at the frequency of the heating. Automatic detection of surface cracks is one of the major result of the period, and the detection process is currently under consideration for a patent application. It is particularly useful in aircraft and nuclear domains as a workaround for bleeding. In the framework of TOCATA project (FUI number 7), active thermography concept and lock-in technique are used for detection cracks [427, 451, 491]. Thermal excitations have been judiciously adjusted and image processing can directly show cracks from thermal images. A result is presented in Figure 20.

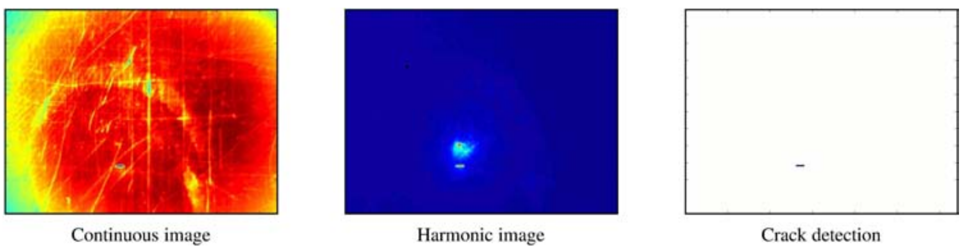

Figure 20: Automatic crack detection. Left is a continuous infrared image, surface irregularities prevent the crack detection. Middle is the infrared image locked synchronized with the heating source. Thermal defects are clearly visible, among them a crack. Right is the result of the automatic detection algorithm which detect only the crack.

Infrared imaging have also been used for determining the thermal properties of materials. Generally thermal diffusivity, thermal conductivity and thermal effusivity are important parameters for materials. But though it is relatively simple to measure thermal diffusivity, it remains quite complex to estimate thermal conductivity. We have proposed a simple and efficient way to estimate that parameter by enforcing convective or diffusive measurement conditions using a Peltier module while measuring the surface temperature with an infrared camera [381, 419, 488]. Data analysis considering measurement conditions allows thermal diffusivity and conductivity to be estimated without contact with a good accuracy.

4.2 Thermal properties of materials probed by photoreflectance

(D. Fournier, S. Holé, C. Filloy-Corbrion)

Photo-reflectance is a non contact optical method using the local reflectivity variations induced by heating to infer temperature mappings [452, 466, 467, 468]. Owing to the wide range of usable wavelengths, various types of information can be retrieved about the temperature of integrated circuits. In the visible range, the technique is now well established. It can probe temperatures through several micrometers of transparent encapsulation layers, with sub-micron spatial resolution and 100 mK temperature resolution. In the ultraviolet range, dielectric passivation layers are usually opaque and the temperature on the surface of that layer can be obtained independently of the encapsulation thickness and of the underlying materials. Near infrared light allows the measurement of the temperature at the interface between the substrate and the active layers with a spatial resolution from 1.7 μm down to 440 nm when using a silicon lens. The combination of the visible, ultraviolet and near infrared illuminations provides a clear view of the thermal processes in complex three dimensional structures of operating integrated circuits. As an illustration, Figure 21a shows measurement in the near infrared range through a 500-μm thick silicon substrate.

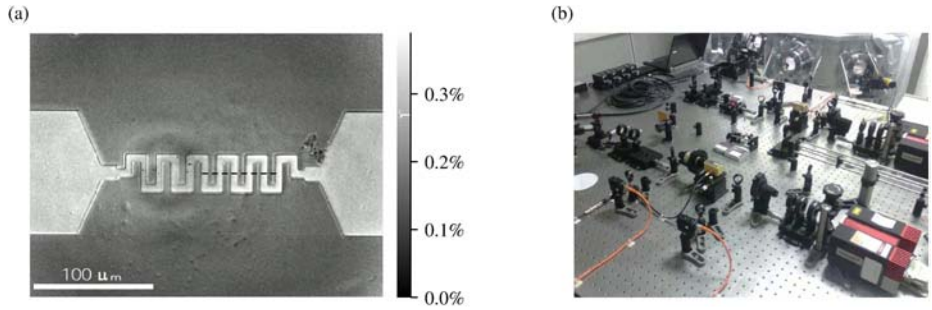

Figure 21: (a) Thermal image of a gold resistor dissipating 609 mW, obtained through the 500 μm silicon substrate with a 50-magnification 0.6-numeric-aperture objective. The resolution measured along the dotted line is 1.7 μm, which is the diffraction limit. Near the resistor, the silicon temperature is 27 K. (b) Virgo experiment.

4.3 Gravitational wave detection by VIRGO

We are currently involved in collaborative R&D activities with the LAL (UMR8607) Virgo team [407]. We are participating to the CALVA experiment which aims at defining strategies to lock the various Virgo optical cavities at resonance [393]. CALVA is made up of a double Fabry-Perot cavity: a 5 meter recycling cavity coupled to a 50 meter long cavity that mimics one Virgo arm and the power recycling cavity. The main Nd-YAG laser enters through the recycling cavity while a second auxiliary laser (1319 nm) enters through the opposite port, 55 meters away. The second laser is used to cool down the cavities in order to bring them to resonance with the main laser in a controlled way. The main task of LPEM was to design the laser injection systems, the local control systems that measure the positions and orientations of the main optical components, and to provide a solution for the locking. Today we are mainly involved at solving unforeseen technical problems, in particular the frequency jitter of the lasers prevents the lock of the long cavity with the main Nd-YAG laser. We are building a two-stage frequency stabilization system by controlling the crystal temperature of the lasers at low frequency and their cavity lengths at higher frequency. A Pound-Drever-Hall scheme is used with an external reference cavity (Figure 21b).

Scientific input and output

National Collaborations:

IES, UMR 5214 (P. Notingher, S. Agnel) - LAPLACE, UMR5213 (L. Berquez, G. Teyssedre)

Grants and contracts:

ANR OMICAGE, ANR MOQAPRO, FUI TOCATA, SAGEM , GE Healthcare, Bostik, Géo-Instrumentation

Highly Qualified People training:

PhD:

N. Cuvigny "Étude et réalisation d'un instrument pour la mesure de la teneur en eau dans les plaquettes forrestières lors du déchiquetage"

G. Dagher "Mesure des charges d'espace à l'échelle nanométrique: application à la micro-électronique"

N. Houdali "Étude et réalisation d'un système de positionnement latéral par une approche active et coopérative"

N. Lechéa "Imagerie par résonance magnétique nucléaire bas champ à SQUID à haute température critique"

Z. Mokhtari "Étude et réalisation de détecteurs ioniques de fumée sans source radioactive"

B. Salamé "Caractérisation des structures à semi-conducteurs par couplage élasto-électrique"

G. Tramoy "Détection et caractérisation de toucher sur coques complexes par ondes de Lamb"

Post-doc:

Y. Felada "Évaluation de la profondeur d'une fissure débouchante normale par thermographie infrarouge"

C. Margo "Détection de chute par une approche capacitive"

F. Sepulveda "Détection de fissures par thermographie infrarouge temporelle"

M. Streza "Détection de fissures par thermographie infrarouge synchrone"

Publications: 94 ACL, 7 patents, 4 invited talks

Appendix 1: Bibliography

Nanophysics, Nanostructures and Nanomaterials

Articles dans des revues internationales ou nationales avec comité de lecture

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Guillaume Lang

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