Structure of the report

Part I: presentation of the TGIR Orphée/LLB

Part II: Report on the LLB research activities

Part III: Instrumental and technical groups

Annexes

PART I

PRESENTATION OF THE LABORATOIRE LÉON BRILLOUIN

A. Short Presentation

Missions and objectives

The Laboratoire Léon Brillouin (LLB)-Orphée reactor is the French neutron scattering facility. It is a research infrastructure supported jointly by the Commissariat à l'Energie Atomique et aux Energies Alternatives (CEA/DSM) and the Centre National de la Recherche Scientifique (CNRS/INP) The objectives of the LLB, as defined in the French roadmap for large scale-infrastructures (LSI), are to perform research on its own scientific programs, to promote the use of neutron diffraction and spectroscopy, to welcome and assist experimentalists. The LLB constructs and operates spectrometers that exploit the neutron beams delivered by the 14MW research reactor Orphée managed by the CEA/DEN. Over these last years, the LLB has conducted, on one hand, as a LSI, a renewal program of the instrumental suite and several international programs, and, on the other hand, as an Unité Mixte de Recherche (UMR 12), the implementation of specific scientific axis in close relation with the French academic and industrial communities. The directorate reports yearly to a Steering Committee and to an International Council for Science and Instrumentation. The LLB benefits from the exceptional scientific environment provided by the 'Plateau of Saclay', which includes the synchrotron source SOLEIL and many renowned Universities, research centers and engineering schools, while respecting its national missions and links with other universities in France. It is one of the leading hubs for neutron scattering at the international level and a part of the European network of national facilities, (MLZ in Germany, ISIS in UK, PSI in Switzerland…) in the NMI3 project under the Seventh Framework Program of the European Union Commission.

As a national facility, beyond access to neutron users, the LLB must provide –*(i)*training and education (including maintaining links with universities); *-(ii)* access for industrial partner; -*(iii)* exploratory studies and experiment preparation including development of instrumentation and methodology; *-(iv)* provision of complementary capabilities, unique instruments and increased flexibility,*(v)* ensuring the transfer of expertise and supporting the emergence of innovative research programs taking full advantages of neutron scattering. These are enduring and complementary roles alongside international centers such as the Institute Laue-Langevin and other European facilities. Recently the LLB was invited to define and coordinate the instrumental and scientific actions of the French community in the construction of new instruments at the future European Spallation Source (ESS) at Lund (Sweden).

Some key numbers

The TGIR LLB/Orphée provides a fully-supported instrument set enabling visiting national and international teams to exploit neutron scattering techniques. Every year, about 500-700 researchers visit the LLB and perform their experiments on our spectrometers. About 400 experiments selected by a scientific review committee are performed annually; 9% of our beam time is directly related to industrial issues and about 4% of our beam time is dedicated to neutron scattering training. There are two calls for experiment proposals every year. The proposals are reviewed by five peer-committees of international experts meeting twice a year.

In fall 2013, 19 instruments are distributed among three Instrumental Groups responsible for their operation and for providing expertise in data analysis, 2 are for tests and 4 under construction; all neutron scattering methods are or will be represented: **Spectroscopy (Triple–Axis, Time-of-Flight, Spin Echo), Diffraction (Powder and Liquid, Single Crystal) and Large Scale Structures (SANS and VSANS, Reflectivity, Stress and Strain, neutron Imaging).** The experimental support for running experiments and developing instruments is provided by four Technical Groups: **Instrument Development, Sample Environment, Electronics, and Information Technology. Common Platforms** have been implemented to ensure and support specific research activities (Chemistry, Biology and Modeling).

Several years ago, the LLB adopted an ambitious policy for instrumentation improvements and upgrades never undertaken previously: the first step was done via the CAP2010 program, whose financing was mainly taken without specific support. It was a modest phase before a more complete renewal with the CAP2015 program extended with new projects until 2017. With a limited financial support, in a few years, more than a half of the instruments were (or are going to be) put into service after rejuvenation or creation, insuring an average gain in flux of a factor 15 by instrument up to 27 with the current improvements until 2017.

The research activities are cross-cutting the operational groups and are organized in three different areas covering a broad scope of science addressed by neutron scattering: - **Magnetism and Superconductivity**, - **Materials and Nanosciences: Fundamental studies and Applications**, - **Soft Complex Matter**. This distinction follows the obvious specific scientific areas, but it also accounts for the different ways to work in terms of collaborations, contracts and publications, and their expertise in specific neutron scattering techniques. One should note that research at the LLB has a dual character as it encompasses both the own research programs carried out by the LLB members as well as the collaborations with visitors associated with the activities of local contacts and with instrument development.

The scientific production from 2008 to mid 2013 includes 959 publications registered so far in IsI Web Of Science with an impact factor, 59 proceedings and book chapters in IsI WoS, 52 proceedings not in IsI WoS. These articles represent 65% of the scientific production of the French Neutron Scattering Community. The 2009-2012 Annual Reports summarize the scientific highlights of the LLB users and the evolution of the facility. The average publication rate per researcher and year is about 3-4, as well as the average impact factor of the publication journals. The strong international visibility of our scientists is illustrated by the citation rate of our articles (on average over 5 years, 16% of them are in the top list of the 10% most cited in Physics, 2% in the 1%), and by the number of invitations at international conferences (178 over the period) and seminars given. Another measure of success is the number of ongoing contracts and external funding; they correspond to projects selected by different organizations at local, national or international levels. It evidences the strong involvement in national and international programs and keeps the scientific relevance of neutron scattering.

Organization of Training sessions is a part of the main assignments of the LLB in addition to its own training activity through research programs for PhD students. It consists in Summer schools and lectures, but also implies to offer beam time access to students. The LLB proposes PhD and post-doc training sessions, such as "les FANS du LLB"(**F**ormation **à** la **N**eutronique), each year in autumn; the purpose is to offer a wide spectrum of the instruments used in a specific scientific domain. They are completed by practical experience for students at bachelor or Master Levels, or from engineering schools. The LLB also organizes visit tours for everybody interested. Recently we have created a new group in charge of the Training and Education; it illustrates the strong involvement of the personnel (2/3 are involved in various teaching activities) and the recognized importance of this mission. Thanks to this group, the LLB increased its actions toward education and training by welcoming more students in master classes and by setting up a series of training for young researchers and industrial engineers.

In September 2013, the LLB comprises 107 people, permanent and non-permanent and 60 people at the Orphée reactor. The scientific production and the technical performances would not be achieved without the strong commitment and professionalism of the whole staff. Thus this staff is the major strength of the LLB. However an important threat for our facility is its demographics; we reached already for years a critical size of the teams for maintaining and developing new scientific areas and associated instrumentation; our main weakness is the non-ability or non-control of the replacement of our staff. Additionally, the perpetual uncertainties on the budget and its consequence on the operation of the reactor (even few weeks before the next year) are strong threats for users and their scientific plans, and do not allow reliable planning and developments, neither the achievement of our international commitments.

In order to keep a French community at its level of excellence, build the future generation of users for the ESS and get involved in its instrumental project, it is essential to attract young scientists, students and engineers with permanent position. Moreover regarding the time required to build an instrument, a large-scale facility and a community able to exploit it efficiently, availability and easy access to a (national) neutron source over the next decades are essential. The absence of a long term operation of a national neutron source will strongly impact critical decisions for the instrumentation strategy needed in the different scientific domains. The suggested changes of the European landscape won't sustain these activities and might become a strong threat for lack of motivation and considerably reduce these research fields. With the possible closure of the Orphée reactor in 2020, the beam time access of the French community will be reduced by 60%, only 40% will be left between ILL and other European facilities, for which European financial support might also be abandoned in 2020, while the access to ESS was advised to be minimal and will certainly be delayed, and the ILL might stop in 2030. All together this dark landscape for our community makes the establishment of a complete project management and overall instrumental strategy of our infrastructure very difficult to handle.

A detailed SWOT analysis was proposed in 2011; most of the arguments are still valid and can be found therein.

Content of this report

This report is organized as follows. We start with a general presentation of the infrastructure: governance, personnel, budget, scientific production, training and education, user service. The scientific activities and perspectives of the LLB axis are described in Part II from 2008 to 2012 (mid 2013) with a summary and highlights. Part III focuses on the operation of the facility, the instrumental and technical groups, platforms with their management, strategy and highlights. Finally, lists of documents (Scientific Production, Staff, Contracts, etc……) are provided in the annexes.

The LLB/Orphée overall activity is regularly reviewed by peer-committees of international expert as a research center and facility for users; it was reviewed already in 2011 by the AERES agency for a midterm period from 2008 to 2010. The whole document and report from the committee are in the supplementary material. Considering that, at that time, it was a model for national large scale facility evaluation, much more details of our infrastructure are provided in these documents not repeated here.

B. Governance

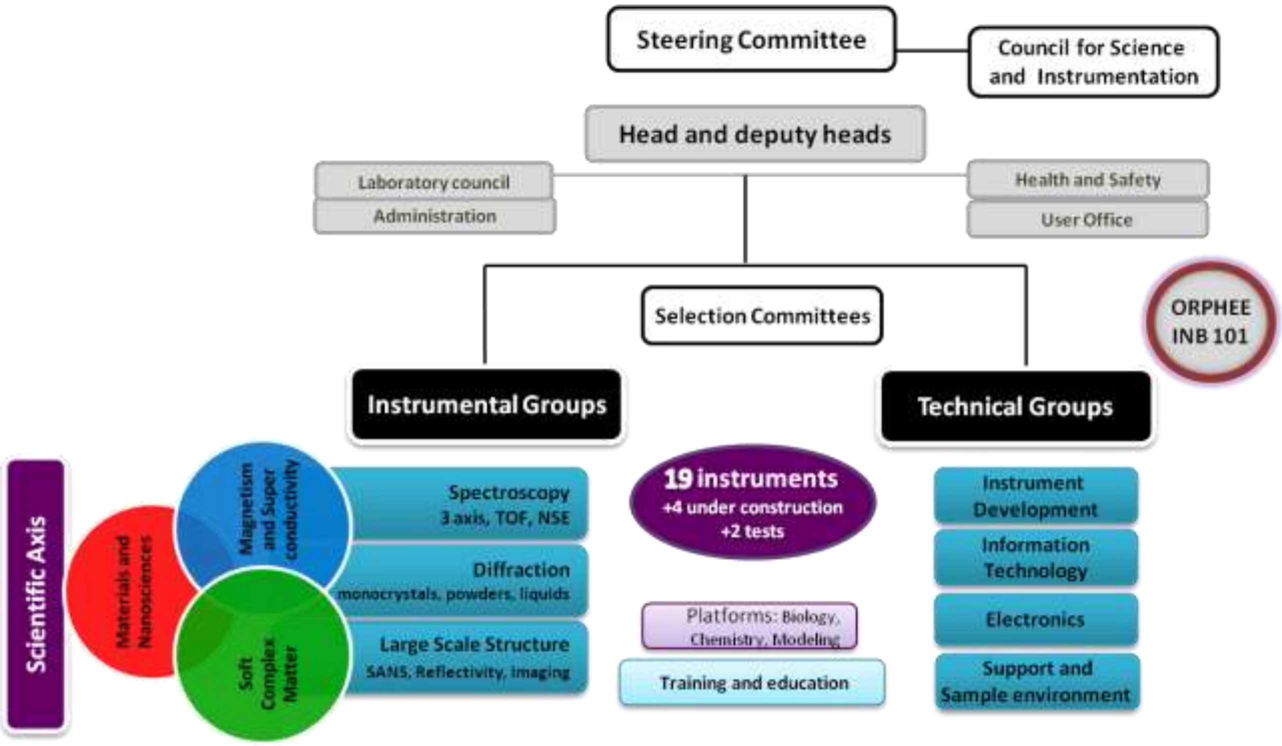
Organization chart

The governance of the LLB is assumed by a **Steering Committee** composed of three representatives of the CEA and the CNRS. The status, objectives, organization and financing is subject of a Convention between the two agencies, renewed every five years; the last version dated from January 2006 was amended in January 2012, by introducing in particular a **Council for Science and Instrumentation** (CSI).

The mission of the CSI is to provide expert advices on the development of the instrumentation, research and education missions of the LLB, and recommendations on how to reach the long-term goals within the national and international context, and with respect to the future operation of the Orphée reactor. The committee reports formally to the LLB Steering Committee.

The CSI is comprised of three members with two year terms, with Claude Berthier (CNRS), Gérard Gebel (CEA), and Ian Anderson (ORNL – chair). Three additional members, with one year terms, can be solicited each year to address the particular focus that year. In October 2012, the CSI was asked to focus its attention on the status and development of the neutron instrument suite, neutron techniques and related education activities. In October 2013, the Steering Committee asked the CSI to address the science programs at LLB, with particular emphasis on the relevance of the science programs on the instruments under development and the scientific collaborations in which LLB participates.

The **management team** consists of the Director, nominated by the Steering Committee with the mutual agreement of the CEA and the CNRS, deputy Director and an assistant head in charge of the relation with the reactor. It is supported by an **Administration Group**, a **Selection Committee** for evaluation of beam time proposals and a **Laboratory Council (LC)** dealing with interaction with the staff.



The organization chart of the LLB is governed by the use of the neutron beam provided by the Reactor Orphée and the best operation of the spectrometers. Since the last AERES visit, according to the recommendations, the internal organization of the LLB was modified to increase its consistency and optimize the use of the resources. Besides the management group, the **Instrumental and Technical groups** were reinforced in their tasks and the research endeavor of **Scientific Axis** strengthened as their interactions with the overlapping **Platforms** and **Training and Education** unit.

The lists of members of each council, committee and groups acting over the last five years are given in the chapter 'Personnel' in the annexes.

Scientific animation and Communication

Regular meetings are organized during the year, preferentially during reactor shutdowns: -The LC meet about 6 times a year; all information about the laboratory is discussed, a report is written by one of the members and distributed to the whole laboratory; -the Group leaders and the Axis coordinators meet the direction on a monthly basis; these meetings set up the execution of the budget, technical and scientific programs; - The LLB directorate meets several times a month the head of Orphée reactor, C. Blocquel, and coordinate the planning; - Meetings within the Groups and Axis are organized at their convenience and vary from one group to another depending on their size, their actions and the urgency. Each spectrometer responsible organizes meeting for the planning of visitors, the maintenance of the instrument with members of other groups.

The scientific animation is marked by several events: - Seminars are proposed on a regular basis (on Tuesday, and Friday), in addition to internal seminars within the Axis. The list of seminars is given in the appendix. However, one should mention here the difficulties to reach the centre and the consequent low participation of external researchers; - PhD and Habilitation defenses (about four PhD and two Habilitations (HDR) per year). 'Specials days' are organized to encourage the young researchers to present and discuss their results: - in June for the PhD students and the post-docs;- 'a one-day meeting 'la journée des ANRs' in order to keep informed all the LLB members on new projects that have been accepted and funded by the ANR; - A LLB outside day for inter-group communication was organized in 2013.

Web sites and annual reports

The LLB possesses two websites: one internal for communication inside the laboratory. It is used for organization and announcement of events, for experiment scheduling, for handling reports of all kinds and highlights; most of our attention is dedicated on this website and it is essential for the coordination of our activities. We have also an external website dedicated to users, which is regularly upgraded with news on instrument and reactor operations, scientific highlights, calls and workshops announcements (http://www-llb.cea.fr/fr-en/spectros_p.php). However it still needs strong improvements for handling visitor invitations and we are testing a common portal with the Synchrotron Soleil.

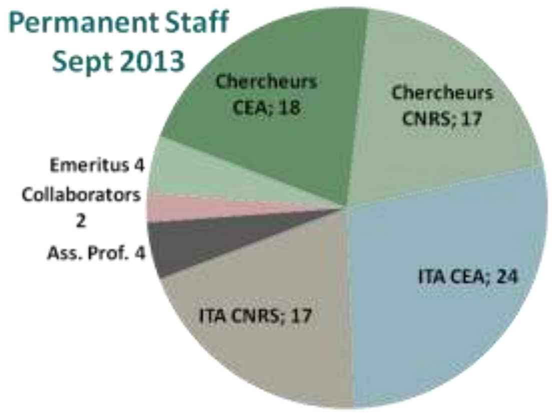
Since 2009 LLB publishes yearly an Annual Report focusing on the recent news of the infrastructure, the strategy developed for instrumentation, education and Workshops announcements and especially Scientific Highlights from user's experiments. For the latter a call is made every year and a selection is made internally.

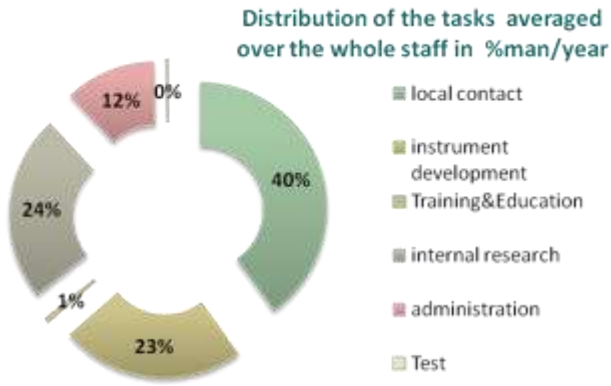
Unfortunately, the LLB does not possess a specific unit dedicated to communication and all these actions are dependent on the willingness of some researchers and engineers.

C. Human resources : permanent and non permanent staff

Staff in September 2013

The permanent staff of the LLB consists of people from the CEA/DSM and from the CNRS/ INP or INC. For the CEA, two categories are considered researchers-engineers (A1) and technicians (A2); they are researchers, associated to publications, and engineers building the instruments, not always part of the publications, and technicians. For the CNRS, the labels are slightly different: the researchers on one hand, associated to publications, and, engineers, administrative and technicians (ITA) on the other hand, the engineers being nevertheless often considered as researcher. The technicians are assigned to a specific instrument or to a technical (or administrative) group. The researchers and engineers belong to instrumental or technical groups as well, and are members of Axis, Platforms and Education unit. Noticeably, most of the staff has multiple roles in the different component of the matrix organization, and regarding the staffing level, overload is always a threat. The distribution of the tasks in terms of % of man/year is showed below.

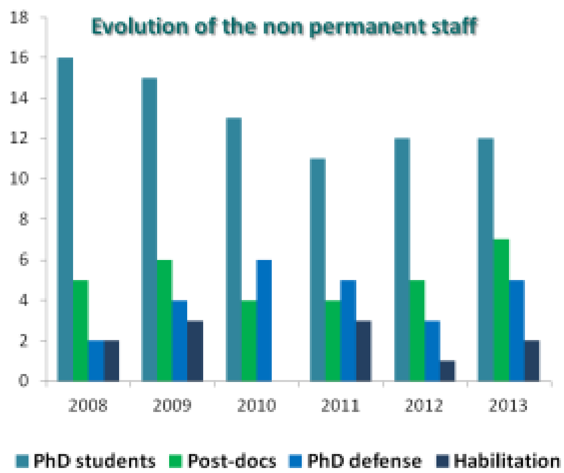


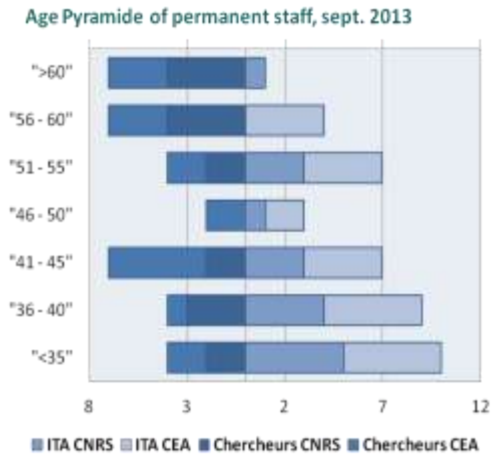


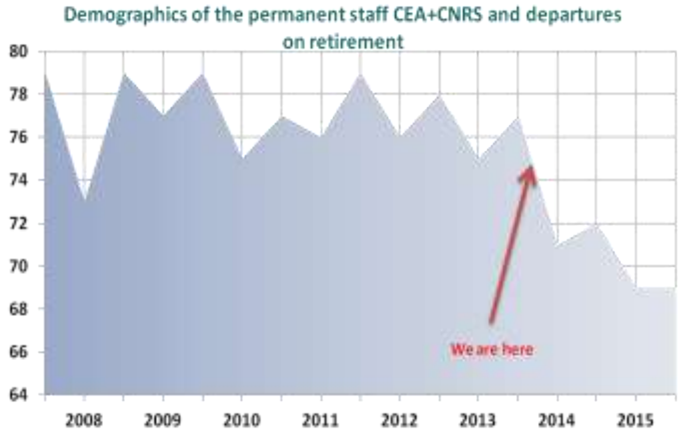
The staff comprises 76 permanent staff, 4 emeritus, 2 researchers from KIT in charge of their CRG, 4 associate professors at part time from different universities around Paris, and 5 close external collaborators from various institutions, who requested partial attachment to our unit; the non permanent staff, *i.e.*, PhD students, Post-Docs and Technicians under contracts include the last people arrived in fall 2013. The list of personnel over the period is given in the annexes, and each group lists them in their report given in Part III.

EVOLUTION

The number of non-permanent staff slightly increases thanks to new instrumental projects and the PhD program shared with Soleil. The financial supports of the PhD students arise from various origins, from the CEA, CNRS, MESR, Industry, Regions, while Post-docs salaries come from ANR or European contracts. One should also keep in mind that the real number of PhD students supervised in their experiences is quite large: depending on the available beam time, between 90-140 students and about 50 post-docs are welcomed and helped every year. More than 70% of our researchers got the 'Habilitation' (HDR) diploma and young researchers are deeply encouraged to defend it.







However our major concern is the low number of permanent people acting around the spectrometers. Moreover most of the departures until 2015 are scientists and instrument responsibles: we will lose 25% of the research staff.

Continuing Education of the Personnel

Continuing education for the personnel includes permanent and non permanent members; it is important both its career and to build a project management for the various programs. Two local correspondents are in charge for the CEA and the CNRS proposals; the needs focus on security, Methods and Instrumentation, various schools, English. Moreover the LLB might provide its own training sessions (safety, radioprotection, specific technical software or equipments….)

Memberships

The staff of the laboratory participates actively in the national and international scientific life; this is noticeable by its multiple contributions in committees for PhD and HDR vivas, in the organization of national and international conferences (local organizing or scientific advisory committees), in various boards of evaluation (AERES, ANR, RTRA, CNRS), in the selection committees of the other facilities (ILL, MLZ, HZB, ESRF..), in various working groups.

D. Financial resources

The Steering Committee, according the convention between the CEA and the CNRS, decides the way the budget evolves, and should be distributed among the tasks of the LLB. The budget received by our facility consists of *i-* the purchase of the neutron beam time frome the Orphée reactor, *ii-* the CEA and CNRS fundings for running the whole infrastructure, *iii-* the personnel from CEA/DEN for the reactor, CEA/DSM and and CNRS/ INP-INC for the LLB. A full cost analysis for 2012 is presented below.

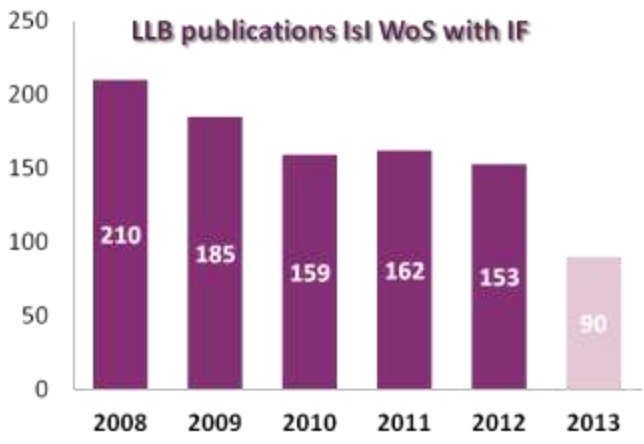
With the help of the French-Swedish contract, we are able to complete our instrument suite with 3 instruments: in 2012, this extra funding allowed the purchase of the detection system of the time-of-flight spectrometer Fa# (^{3}He detectors) and a part of the SANS machine PA20 for 2.5M€. However assessing the performance of existing instruments and ensuring a minimal renewal following the technology progresses would require in the future an increased funding from both recurrent subsidies and external contracts. Any contribution to ESS instrumentation for 2020-2025, would obviously involve new source of funding.

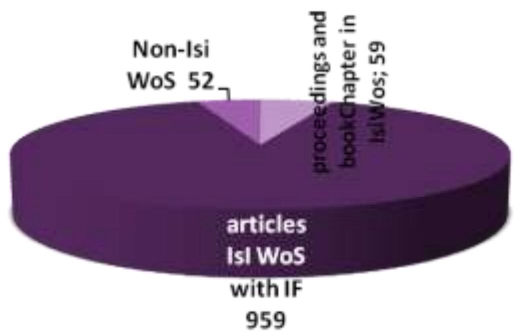
E. SCIENTIFIC PRODUCTION

PUBLICATIONS

To assess the performance of the infrastructure, we have collected all the publications in a list presented in the annexes from 2008 to mid 2013. This list takes into account *i-* the research work performed and initiated by the LLB members described in Part II, *ii-* the research with collaborating users often associated in specific national or international programs, and *iii-*finally the articles of other external users published with or without LLB authors. The latter list is the most difficult to complete: for some publications the LLB affiliation does not appear explicitly, which makes their accounting problematic. On the basis of the user's feedback that we have explicitly asked for the year 2008, we have estimated that about 10% of the publications might be missing for all the years (except for 2008). We have also added in the appendix the list of book chapters or editorial activities. The whole set is then distinguished between three categories of publications:

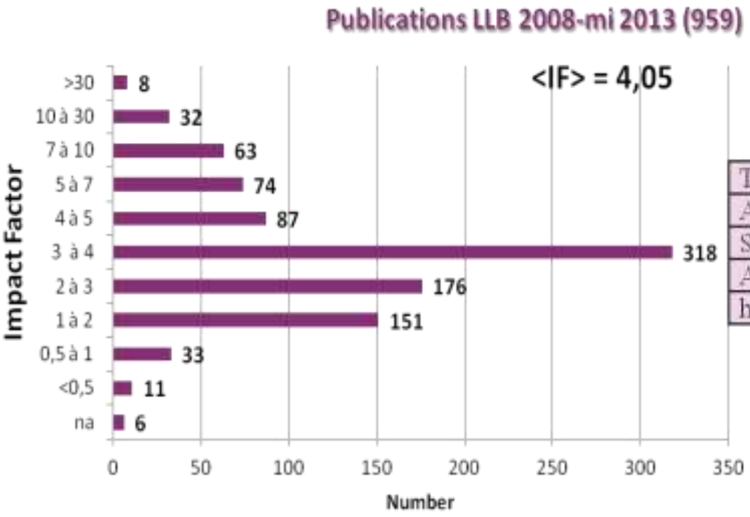
- the publications with an Impact Factor, or close to get one, found in IsI Web Of Science: total of 959, (869 over the 2008-2012 period).
- the proceedings found in Isi WoS, 59.
- the publications non listed in Isi WoS, 52 in total, 35 from 2008-2012.





Analyzing the impact factor of the journals of publications, we observed that about 10% of the articles are accepted in highly ranked journals (Nature, Nat.Phys., Science, Angewandte Chemie, Physical Review Letters …). 50% of the publications are in medium-high ranked journals 3<IF≤7; their number increases with time reducing the number of publications in journals with an IF below 3. The concerned journals are representative of key scientific topics studied by neutron scattering at the LLB especially in Material Sciences, Soft Matter and Biology, while hotly debated topics related to magnetism and superconductivity belong to the former

category. A similar analysis is provided for each scientific axis below, where the trends can be better seen.



Citation Metrics September 2013

Total Articles in Publication List:	959
Articles With Citation Data:	937
Sum of the Times Cited:	8213
Average Citations per Article:	8.77
h-index:	36

Nevertheless the real impact of a publication is measured by its number of citations. When the above publications are associated to a citation index, the result shows the quality of the work performed, one should also be aware of citation index basis, which might vary between fields and over time. On average, all the publications from 2008 till 2012 are cited 8-9 times, with 36 of them cited more than 36 times. With the help of M.-A. Leriche from the Direction of the Scientific Information of CEA, we have conducted an analysis comparing the LLB publications to the world average and the top 10% most cited articles. In this analysis, over the period 2008-2012, 140 articles pass the threshold of the 10% most cited, 14 the threshold of the 1% most cited, and only 13% remain not cited so far. Here is a selection of them from 2008 to 2012.

75- Electronic liquid crystal state in the high-temperature superconductor YBa2Cu3O6.45, Science, 319; 5863, 597-600, (2008) Hinkov, V. Haug, D. Fauque, B. Bourges, P. Sidis, Y. Ivanov, A. Bernhard, C. Lin, C. T. Keimer, B. 199 citations.

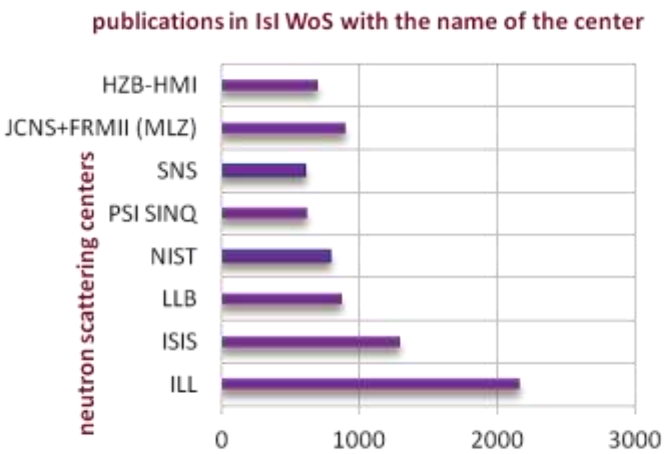
109- Electric-field-induced spin flop in BiFeO3 single crystals at room temperature, Physical Review Letters, 100; 22, 4,(2008) Lebeugle, D. Colson, D. Forget, A. Viret, M. Bataille, A. M. Goukassov, A., 186 citations.

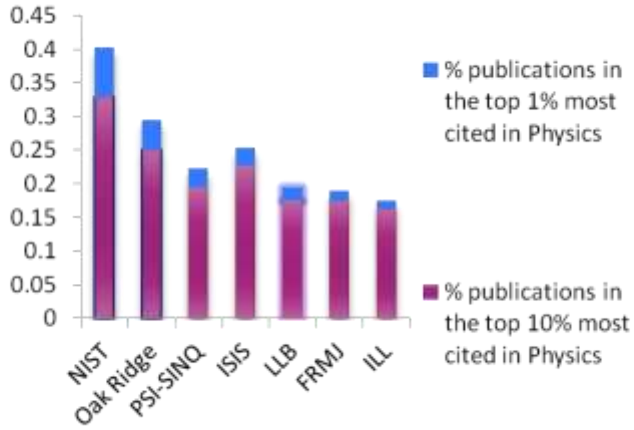
577- C. Chevigny, F. Dalmas, E. Di Cola, D. Gigmes, D. Bertin, F. Boue and J. Jestin, 'Polymer-Grafted-Nanoparticles Nanocomposites: Dispersion, Grafted Chain Conformation, and Rheological Behavior', Macromolecules 44 (2011), 122-33.40 citations.

868- X. W. Zhang, S. Boisse, C. Bui, P. A. Albouy, A. Brulet, M. H. Li, J. Rieger and B. Charleux, 'Amphiphilic liquid-crystal block copolymer nanofibers via RAFT-mediated dispersion polymerization', Soft Matter 8 (2012), 1130-41. 17 citations.

The average publication rate per researcher of the LLB and per year is about 4; it becomes 3 when only own research is considered. To evaluate these numbers one should obviously bear in mind the particular status the facility; one should also consider the distribution of tasks of the personnel, the considerable amount of time dedicated to maintenance and development, training (with low number of staff on each spectrometer as notified already in 2011) and dedicated to get extra funding.

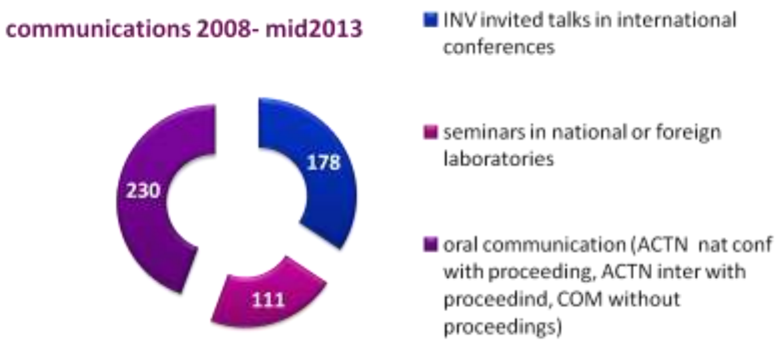
A comparative study between neutron scattering centres was also performed limiting the analysis to publications where the affiliation of the centers appears in the address of the authors. This is a rough comparison where no normalization was done; one can observe the strong European leadership in the scientific production, however the study shows that the number of citations evolves with time slowly in Europe when compared to United-states.





Communications at Conferences

A measure of success could also be how the scientists are internationally recognized and invited to international conferences or seminars. A compilation of these invitations over the years 2008-2012 is presented according to the AERES classification and a complete list is added in the appendix. The number of Invited Talks (keynote or lectures) at International Conference and Seminars given in French or foreign Universities is quite high, and the number of Oral Communications demonstrates the training through research of young scientists, PhD students and Post-Docs.



Patents

2 patents have been submitted.

-Brevet FR2963481 / WO2012013603. J.-M. Zanotti, K. Lagrené, "Mineral electrolyte membrane for electrochemical devices",. Brevet public depuis le 03/02/2012.

- Brevet d'invention avec extension PCT, CNRS: DI 05815-01 FR12: « Nouveau Procédé et Dispositif de production du froid » P. Baroni, L. Noirez et P. Bouchet. (2013).

Outreach

The LLB staff actively participates in the scientific community by taking part on national and international conferences either contributing or co-organizing, by sponsoring workshops with other large scale facilities or specific scientific communities or Societies such as the French

Neutron Scattering Society (SFN). A list of our sponsorships and contributions to special events is provided in the annexes. Moreover recently we decided to co-organizer and lead or initiate workshops dedicated to some scientific topics where neutrons are one of the experimental probes between others. One can cite: "Horizons in Hydrogen Bond Research" in 2009, PNCMI in 2012, "Water at Interfaces: New Developments in Physics, Chemistry and Biology" in April 2013; "Structure of Disordered Systems" September 2013, GDR meeting MICO « Matériaux et Interactions en Compétition » in Nov 2013. Several other events are planned for 2014, starting in January 2014 with the international year of crystallography in Paris, and continuing with an Imaging workshop and "Food and Neutrons III" in July 2014. Hosting advanced neutron scattering schools and training sessions will be presented in the next paragraph.

Besides these actions, we have proposed meetings to inform the French community about the recent ESS advances, and organized working days around specific work-packages that we could support within the instrumentation program of the ESS.

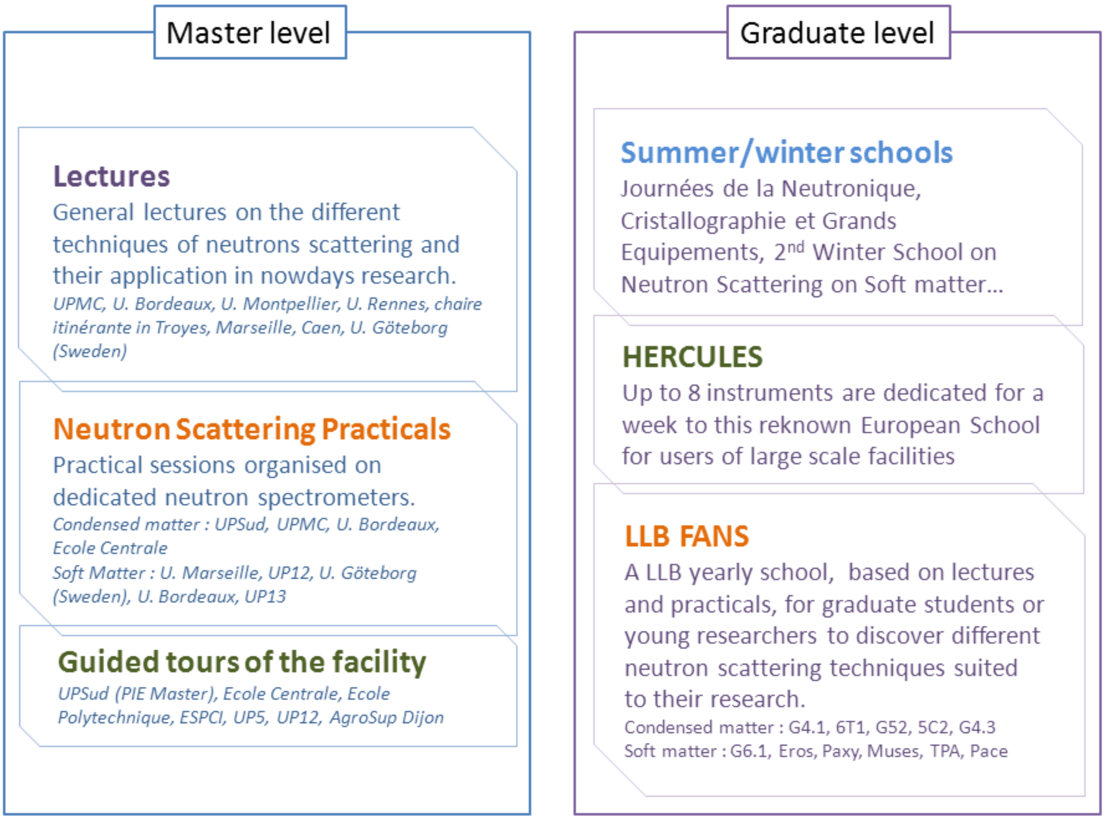
F. Training and education

Neutron scattering experiments can only be performed in large scale facilities. The training of students and scientists, along with the education of the general French public about the use and achievements of neutron techniques, are therefore important missions of the LLB as a national laboratory.

To this end, LLB researchers are very active in presenting neutron scattering techniques at scientific meetings, and in co-organizing advanced neutron scattering schools and workshops (like, for instance, the Spin Dynamics Calculations Workshop organized recently by S. Petit), often working jointly with other large scale facilities, and in close collaboration with the French Neutron Scattering Society (SFN). Several other ongoing actions include publications, exhibits and displays, Internet website, books, and the supervision of PhD and master students. In addition, lectures are regularly given to undergraduate students, and guided tours of the facility, as well as on site science practicals, are regularly organized in collaboration with different Universities.

The coordination of all these actions is ensured by the '*training and education' unit*, composed of F. Cousin, F. Damay, A. Menelle, S. Petit, F. Porcher, and Y. Sidis, and also involves most of the other members of the laboratory, including the technical staff.

The different educational programs offered by the LLB are summarized in the following chart:



Teaching and Supervision, bachelor and master degrees

Several researchers and PhD students are currently involved in M2 lectures and practical sessions on dedicated neutron instruments.

Lectures on the different neutron techniques and uses in science:

- French neutron scattering society (SFN) itinerant chair at Marseille, Caen and Troyes Universities, and at the Swedish University of Göteborg.

- L3-M1-M2 lectures: for instance:
  - Master "NANOMAT" (University Paris VI).
  - Master "Biomatériaux" (University Paris XIII)
  - Master "Mamaself" (Montpellier and Rennes Universities)
  - Master (Bordeaux I University)

M2 practicals :

Several Universities have now included in their courses a day of practicals at LLB, or a visit of the facility. For example, in 2012, 50 undergraduate students from Bordeaux-I, Paris XI, Paris XIII, Paris UPMC, have had access to a LLB instrument, to study various aspects of neutron scattering, from phonon excitations on a triple-axis spectrometer, to structure and dynamics in biological samples on a spin-echo instrument. Such practicals are a unique opportunity, as they are not available at other European facilities such as the ILL or FRM-II. Supervision of the practicals is ensured by LLB researchers and LLB PhD students, affording the latter with a useful teaching experience.

Training through research, PhD students and Post-Docs

PhD program at the LLB:

Every year about 10-15 PhD subjects are proposed, as well as Master degrees. On average, 10 to 15 'in house' PhD students are working in the different research fields developed in Part I (13 students in March 2011).

PhD students and Post -Docs visiting the LLB

The training of visiting scientists is also an important responsibility of the LLB local contacts, who take great care to provide students or new scientific visitors with the knowledge they need to perform experiments safely, and to treat their data properly. The estimated number of PhD students is about 100 – 120, and 60 Post -docs per year.

PhD and Post -Doc training sessions

The LLB traditionally participates actively in the organization and financial support of a number of Summer schools, acknowledged by the CNRS and CEA « continuing education». The aim is to encourage and familiarize new users to neutron techniques, from PhD students to Professors.

For instance, the laboratory is involved every year in the Summer schools organized by the French Neutron Scattering Society (3 to 6 scientists for about 12 hours' lectures per year since 1998): Inelastic neutron scattering studies in condensed matter (May 2008 – Albé), Neutrons and soft matter (May 2009 - La Grande Motte), Neutrons and numerical simulations (June 2010 - Rémuzat), etc…A complete *corpus* of the lectures given during these schools is available in print and online for free, at the EDP publishing site EDP (http://www.neutron-sciences.org), and can be used as a the basis for lectures and practical work on neutron scattering.

Summer Schools

The LLB is also involved, in collaboration with the Soleil Synchrotron, in the « Cristallographie et Grands Equipements » school. It consists in series of M2 lectures and practicals on crystallography, proposed to PhD students and regular scientists, mixing theoretical and experimental approaches.

The annual organization of HERCULES training session *(**H**igher **E**uropean **C**ourse for **U**sers of **L**arge **E**xperimental **S**ystems*), together with the ILL, ESRF and Soleil Synchrotron. 16 LLB's researchers participate to the supervision of the practicals (over 3 days), which are completed with a total of 6 hrs of lectures.

The laboratory proposes its own yearly training session, called « *les FANS du LLB* » (***F**ormation **à** la **N**eutronique*: http://www-llb.cea.fr/fan). The aim is to provide young French speaking researchers with a first contact to real experimental neutron scattering. Students and post-docs working in all scientific areas where neutrons can provide valuable insights are welcome. After a 4 hours introduction to neutron sources and neutron scattering, 10 different practicals on various LLB instruments are proposed to students. The usual number of students (nationals/non-nationals) is about 35 in total, 2/3 French, 1/3 non-french. This training session is validated by all the "Ecoles Doctorales" and allows gaining ECTS points.

(left)Practical session during the Spin Dynamics Calculations Workshop 4-6 Nov 2013; (right) Students from Bordeaux-I on Mibemol in Nov 2011.

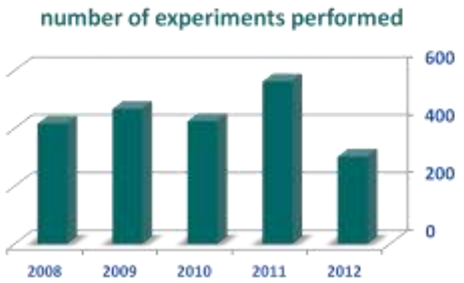
G. Access to Beam Time and User Service

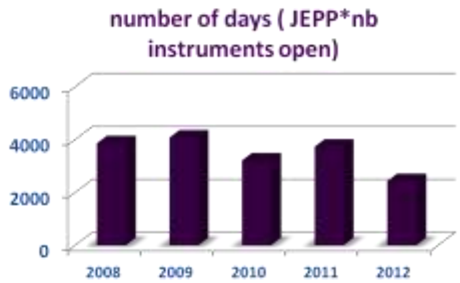
In this section, we present the overall service activities, user programs, beam time statistics and cooperation developed at the LLB.

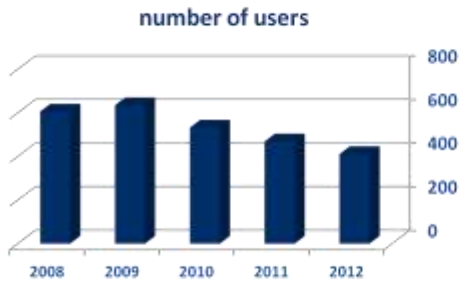
User Program evolution

We present in this section the main facts and figures related to users activity of the LLB/Orphée facility followed by beam time statistics.

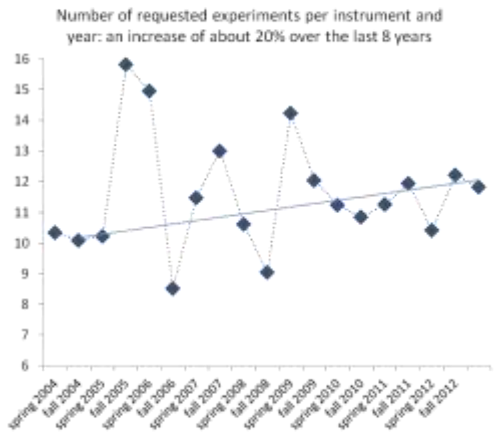
Over the 5 years, 2177 experiments have been performed, by 2653 visitors (counted only once a year) for a total of 894 FPED (JEPP) (about 17000 instruments. days) proposed. An analysis of the users will be done below as well as how the beam time is distributed among the different tasks. The number of "experiments days" employed by users is high; it is shared between about 80% of the "beam days" proposed for the type A proposals, and the remaining 20% of the time for continuing or repeating experiments that encountered problems (technical or scientific), , industrial offers, training sessions and upgrade and maintenance.







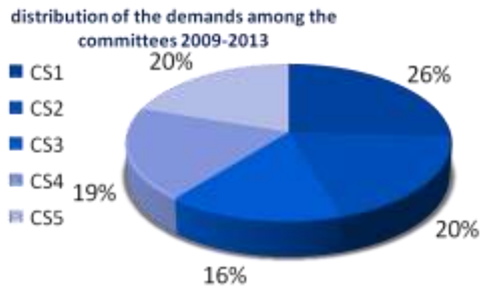
Considering the evolution of number of available days per year and instrument, we have an increase of about 20% of the number of requested experiments over the last 8 years.



Beam time access

To perform an experiment, the researcher must submit a proposal on a special form where he specifies his scientific interest and describes the proposed experiment. Deadlines for submission are April 1st and October 1st of each year.

Proposals for experiments are selected through a peer review. **Selection Committees (SC)** are composed of scientists from France and other European Countries and meet twice a year, typically a



month after the deadline for submission.

In 2009, it was found necessary to reorganize and update the structure of the Selection Committees. The aim of this reorganisation was to make the selection process more efficient because theme "Magnetism and Supraconductivity" had twice as many proposals as the other themes. The new thematic classification is the following:

Theme 1: CHEMICAL PHYSICS, BIOLOGICAL SYSTEMS

Theme 2: CRYSTALLOGRAPHIC AND MAGNETIC STRUCTURE

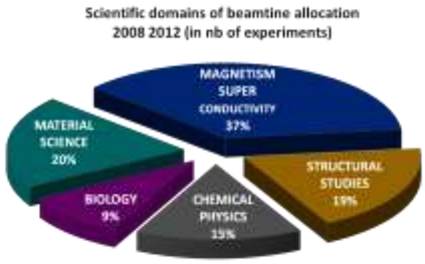
Theme 3: MAGNETISM/ SYNGLE-CRYSTAL SYSTEMS AND THIN LAYERS

Theme 4: DISORDERED SYSTEMS, NANOSTRUCTURED MATERIALS AND MATERIAL SCIENCE

Theme 5: EXCITATIONS

The list of the actual members is given in the annexes.

80% of the total number of beam days is initially allocated to experiments considered as important with a guaranty of programming.



No beam time is reserved for in-house research and LLB scientists must go through the same evaluation procedure. The figure on the left illustrates the scientific domains covered.

The LLB offers two other ways to request beam time, which nevertheless remain exceptional (Submission of a proposal for an experiment to the laboratory management and a procedure for quick access).

The average number of days for an experiment is 6; however it goes from 3-4 for SANS and powder diffraction up to 8-10 for triple axis and stress and strain. This number slightly decreases these last years thanks to instrument improvements.

INTERNATIONAL COLLABORATIONS

Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy (NMI3)

We detail in this section the most significant formalised collaborations. They are basically of two types; on one hand, LLB is active and leading member of multi-partner projects as The Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy (NMI3). On the other hand, LLB cultivate and develop various privileged bi-lateral partnerships.

NMI3 includes all major facilities in the field, opening the way for a more concerted, and thus more efficient, use of the existing infrastructure while reinforcing European competitiveness in this area. The present NMI3-FP7 program is a consortium of 22 partners from 13 countries, including 11 research infrastructures (18 in the call of November 2010). Via the Transnational Access, scientists from different European countries, counts for almost 30% of our visitors. Thanks to Joint Research Activities (JRA) focusing on specific R&D areas, we can develop techniques and methods for next generation instrumentation. LLB was involved in the following research networks: Neutron Optics, Polarized Neutrons and Sample environment, and more recently in new detector development (jointly with CEA/DSM/IRFU), in Structural and Magnetic Imaging and in leading Advanced Neutron Tools for Soft- and Bio-Materials (A. Brulet).

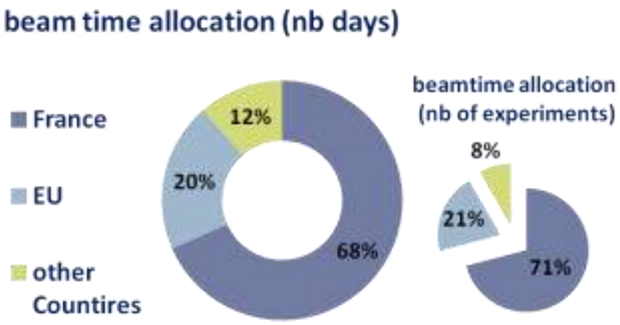
A collaboration exits since the beginning of the facility with the Forschungszentrum Karlsruhe (KIT) and a CRG triple-Axis spectrometer is shared on a thermal beam line 1T1. A two year cooperation agreement is regularly updated; 1/3 of the beam time is exclusively for KIT while 2/3 is made available for the LLB users program.

ESS instrumentation Project.

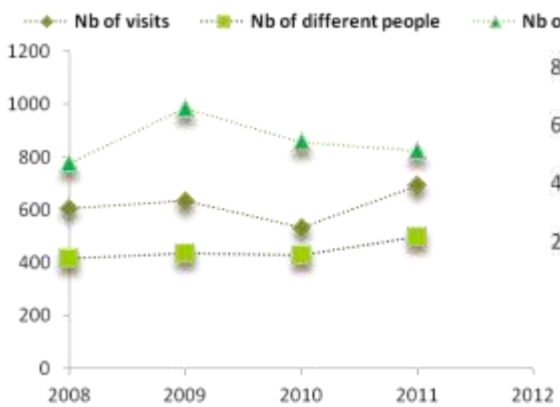
The LLB members were involved in several discussions, meetings about the ESS project; recently the LLB was invited to become a more active partner as coordinator of the French contribution in science and instrumentation and helping the French community in its preparation. Accordingly we are preparing proposals jointly with French users, German colleagues from MLZ (JCNS and TUM) and ESS teams for a SANS, reflectometry and ToF instruments for example. Other work packages are under study involving more French groups, in particular on Sample Environment and Diffraction.

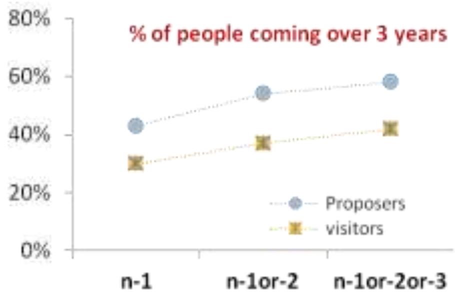
Analysis of the beam time allocation and users

Within the NMI3 access program, we welcomed foreign users and provided them access to beam time: about 20% of the beam time was allocated to EU members (mainly Germany for 8% and Italy for 2 %), while 10% was distributed for non European countries (USA, Israel...). the latter visitors in addition to the long term visitors we collaborated with, between 2008-2012, is a sign of the attractiveness of our centre.



When focusing to the French users, and extracting their address, one can try to estimate how the various organisms are associated: researchers and students from the universities are the most common visitors. The major contributing laboratories of the CNRS come from the Institute of Chemistry.



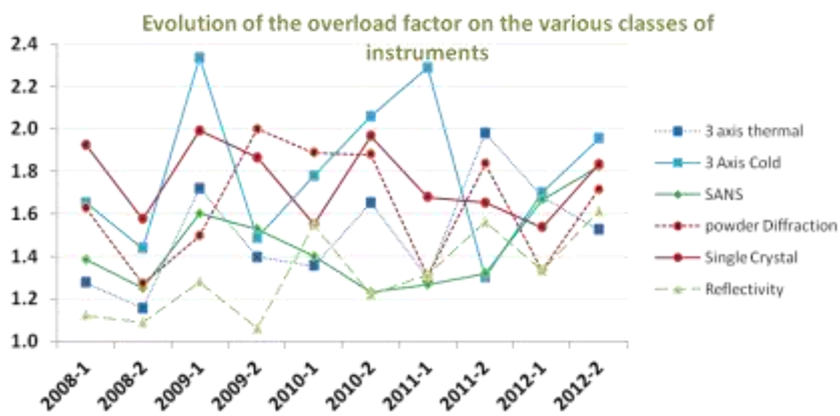


Another analysis of the welcomed users informs about the regularity and the renewal of the community. The total number of people involved in a proposal, the total number of visits over the year and the total number of different persons coming at the LLB are shown below. The sequence considers only the statistics over 3 years, an average for a study, PhD or contract.

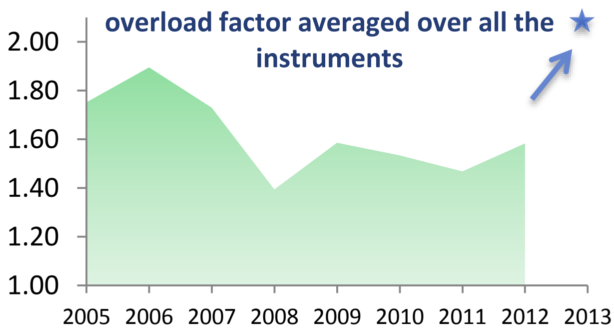
The slight increase is of course consistent with the first figure of the demands, but it also shows the loyalty of the users and the high percentage of new users (about 40%). Thus over the last ten years more than 2600 new users were welcomed for neutron scattering training.

ANALYSIS OF THE DEMAND AND BOOKING RATE

The overload factor (ratio of demand over offer) at the LLB on average over all the instruments increased over the years with an average at 1.6; the progress in their performances thanks to the instrumental program reduces the number of days per days, allows more experiments to be done and fulfills the missions to open access to neutron scattering as much as possible.



The techniques that are highly demanded are SANS, Triple Axis and Diffraction; the rejuvenation of successful quasi elastic spectrometers (Multimuses and Fa#) will certainly attract users and increase the expertise of the whole French community in these methods.



The situation of 2013 stays around 1.6 if the number of days of the reactor is the one expected. However the perpetual uncertainties on the budget and the functioning of the reactor (even few weeks before the next year) are strong threats for user, does not allow consistent planning and make the overall instrumental strategy of our infrastructure very difficult to manage. A reduction of the number of operational days of the reactor in 2014 will put the overload factor above 2 for all the instruments.

OPERATION OF THE INSTRUMENTS

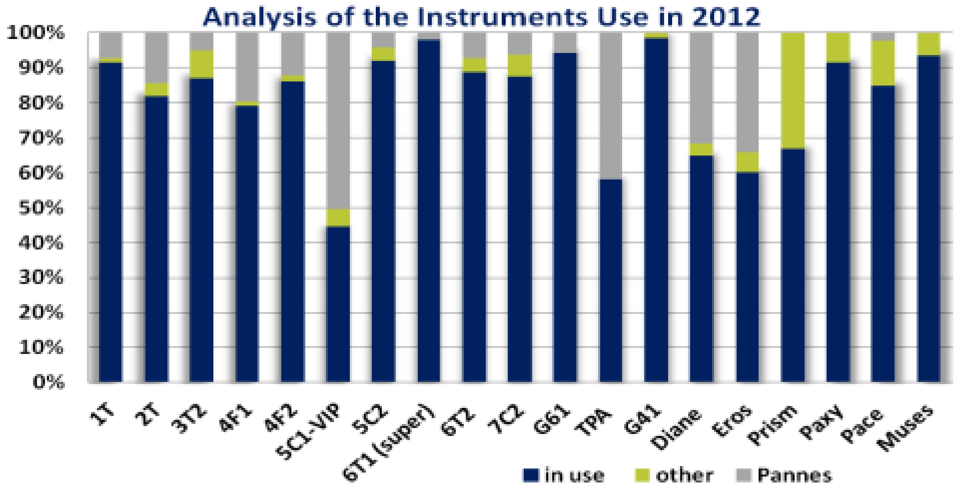
We are now considering the_rate of use of each instrument. In the previous section we showed how 80% of the total beam time was allocated to experiments the year before its use; the remaining 20% is kept for continuing or repeating experiments that encountered problems (technical or scientific), maintenance, training and industrial use. The planned upgrades and known backlog are automatically subtracted.

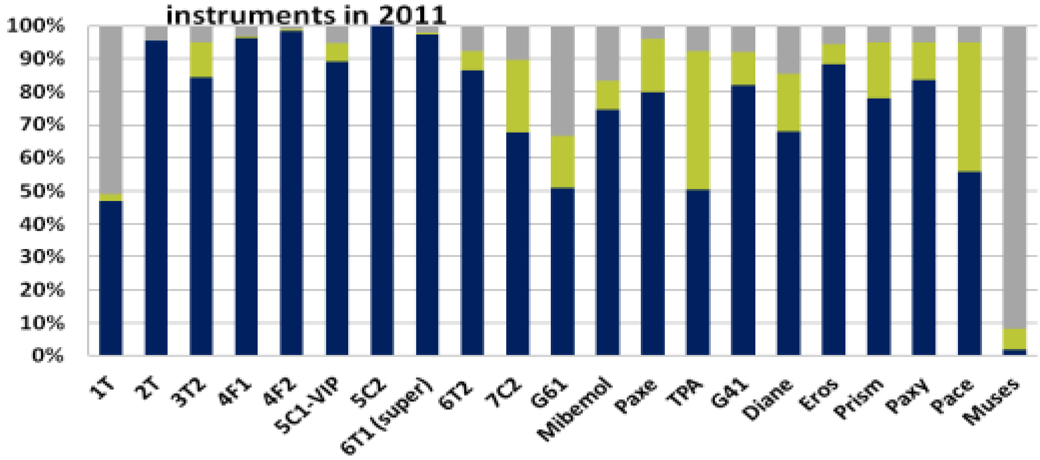
For 2011 and 2012, we have carefully investigated how the spectrometers are effectively used and where new problems happened. The full operation of the instruments was decomposed in 3 classes

- Normal use(**in Use**) : experiments selected by the committees, urgent experiments, training and education, industrial use , backlog of the previous year, repair scheduled in advance.

- Maintenance **(pannes=breakdown)**: unexpected breakdown (TPA-4F1: detector instable during a long experiment to reschedule, EROS: guide implosion, VIP: breakdown of the cryo-magnet)

- **Other**: absent users, problem with samples, HNO periods of the center and difficulties to enter, delay in the reactor planning, no local contact, time for implementation and transfer. (PRISM: no sample available, tests on PAXY)





In 2012, two instruments, PAXE-SANS and Mibemol-ToF, were stopped; the new sans PA20 will take place on PAXE, EROS-reflectometry (now called HERMES) will be installed on Mibemol, which allows doubling its flux.

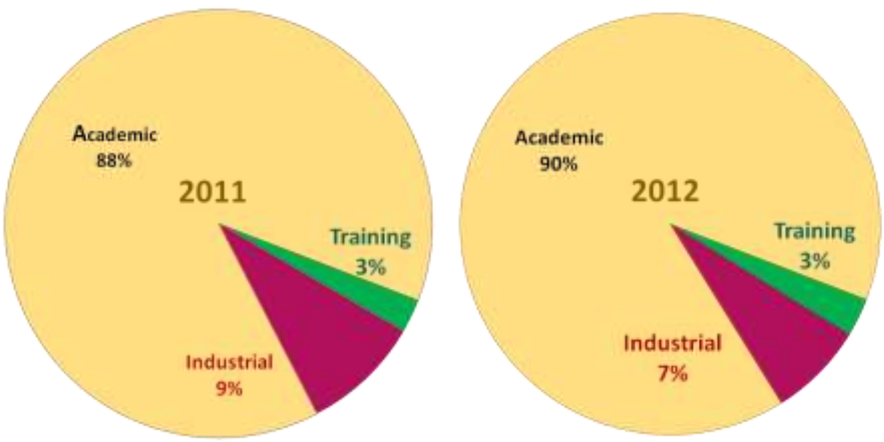
INDUSTRIAL PARTNERSHIP

A detailed study of the full cost of the infrastructure was performed in 2011 with Mazars Consulting at the initiative of the Ministry (MESR) to better evaluate the industrial fees. The followed sections are the results of this study. The goal was to help companies in increasing their competitiveness with a better use of large scale infrastructures, to increase external resources with an increased contribution of industrial users, to inform academic users about the cost of an experiment. One should mention that at the LLB no personnel is specifically dedicated to these tasks, here again it is added to the rest and depends on the willingness and commitment of the personnel.

Neutron scattering has a great potential of application in applied research and industrial research, mainly in the fields covered by the chemical industry (polymers, green chemistry....), and by the metallurgical and mechanical industry. The materials for nuclear industry constitute a wide range of research topics. Occasionally, specific studies are undertaken about the development of new advanced materials (magnetic materials, ceramics, cements....). Finally, neutron radiography and the neutron irradiation, commonly used by some industrial customers to Orphée, can also be requested through collaborations with the LLB. The LLB displays a high expertise, already exploited in a number of industrial collaborations. In the field of materials science, the laboratory has developed various recurrent collaborations. Indeed, in the metallurgy field, we have partnerships with the automotive and aerospace sectors (PSA, Renault, EADS, Dassault Aviation), generally to study residual stresses in industrial parts. In the area of nuclear energy, the LLB is working with CEA, EDF, AREVA in order to define new materials and study their performance in service. In the physical chemistry field, strong collaborations with IFP, Michelin, INRA, Arkema, have been developed over the last decade

The neutron scattering techniques most used at the LLB, in industrial collaborations or in contract research are the following: - *Diffraction Technique*: analysis of residual stress and crystallographic texture(39%), localization of light atoms and in particular, to determine the hydrogen content in a structure of materials for hydrogen storage or molecules inserted in zeolites for catalysis (11%). *–SANS* (37%) for the characterization of microstructural heterogeneities of materials at the nanoscale (1 to 50 nm typically). In the metallurgy field, the SANS technique contributes to the development of new composite materials constituted of a metallic matrix reinforced with nanoparticles (carbides, oxides, intermetallic...). and its evolution (precipitation, porosity ...) under thermomechanical processing, aging and under loading (irradiation, milling) ; In the physico-chemical field, one can cite the characterization of asphaltenes in crude oils, the study of gelation (for food industry), the development of nanocomposite materials (elastomers filled with inorganic particles); - *reflectivity (8%)*: information on the composition, thickness and roughness (or distances of interdiffusion between layers) of a surface. The main studies of industrial interest concerned the analysis of Supermirrors for neutron guides but also, the study of polymers at interfaces, grafting, and bonding; *-neutron irradiation*, study the induced damage and neutronography (5%). There are various type of contracts and partnerships:-proposals with industrial collaboration or from applied research (ANR and AII projects); -PhD Co-funding; -INRA or industrial, Michelin; - Irradiation; -Study & characterization of materials for Gen IV reactors; - Services: material characterization.

As a summary on how the beam time is exploited, we found 88-90% for academics, 3% for training and education and 7-10% in strong connection with industrial interests.



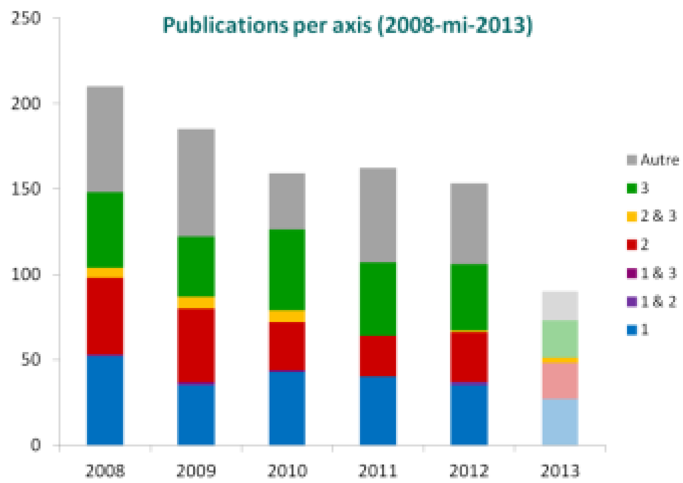
H. INTRODUCTION TO PART II AND PART III

PART II

The exceptional properties of neutrons make available information often impossible to obtain by using other techniques and provide a precise picture of where the atoms are and how the atoms move and vibrate. Not surprisingly, the research done at the LLB is multidisciplinary, applied or fundamental, and covers a broad range of domains spanning basic physics, chemistry and biology. However we have tried to summarize and link the topics through three scientific axis, - **Magnetism and Superconductivity, - Materials and Nanosciences, - Soft Complex Matter**. The three axis share not only the obvious neutron scattering techniques, but are also gradually associated to each other via the length-time scales and the dimensionality of the observed phenomena; accordingly, fundamental questions addressed in Axis 1 in bulk strongly correlated systems are complemented by the more work done on thin films in Axis 2. The coupling, ordering or disordering, of length-scales starting from few angstroms to microns in Axis 2 provides the necessarily bridges and hierarchical structures of objects involved in the Axis 3, the two latter bringing in turns some chemistry description, empiricism and concepts to the first Axis.

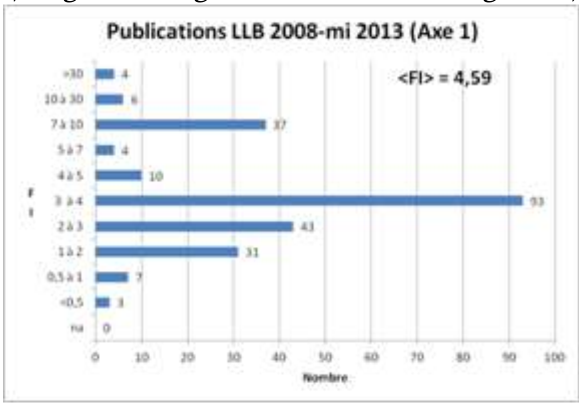
The research activities along this axis are conducted in various ways in terms of collaborations, contracts and publications. For each scientific axis, we made the choice of a short general presentation of the research activities supported by four highlights referring to the second half of the period (2008-mid 2013); the previous AERES report written in 2011 provides a whole coherent picture of these axes and some sort term perspectives.

The scientific production of each of them has been considered and selected among the general list provided; a classification per axis and year has been made in the supplementary material, plus a fourth category including works performed in collaboration but not ranging in these topics and publications where the LLB affiliation was not explicit. Here is an abstract of each of the scientific axis.



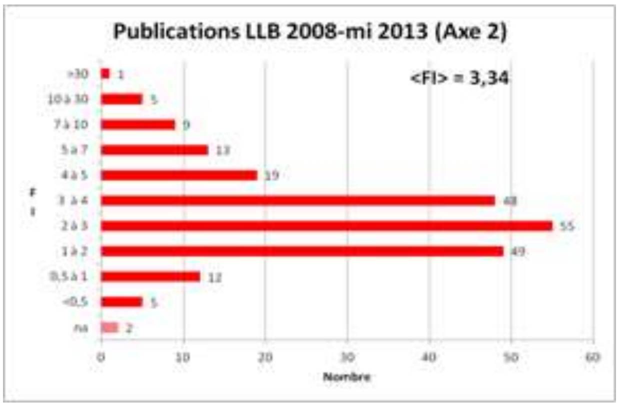
Axis 1 Magnetism & Superconductivity

Strongly correlated electron systems and complex magnetic systems are central topics of nowadays condensed matter physics and many body problems. The extensive study of static and dynamical magnetic correlations through magnetic neutron scattering, including polarization analysis, provides the inputs that are essential to describe the microscopic state and to understand its fundamental properties, a necessary step before the elaboration of new theories and concepts. One common feature in many of these materials is the coexistence of several degrees of freedom associated with the electron- (charge, spin, orbital) or lattice sub-systems, whose interplay is responsible for a large variety of ground states and excitation spectra. Well-known examples studied at LLB, both experimentally and theoretically, include cuprate or ferropnictide high-T_c (HTC) superconductors, "giant magnetoresistance" manganites, compounds with short-range magnetic interactions subject to geometrical frustration (multiferroics, spin-ices, etc.), lanthanide-based heavy-fermion systems and "Kondo insulators", as well as a number of materials in which unconventional orders occurs, such as the "magnetic blue phase" of MnSi, or the multipole-order states found in rare-earth hexaborides. Because neutrons interact with both the atom nuclei and their electron shells, neutron scattering is one of the best tools to study this type of physics involving interplay of lattice and magnetic properties.



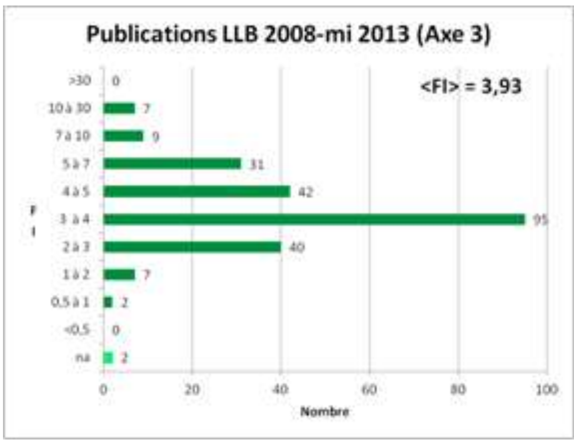
Axis 2 Materials and Nanosciences : Fundamental Studies and Applications

This scientific axis covers the activities related to the research in material sciences and more generally in hetero-systems (interfaces, alloys, composites materials, confined systems). The topics cover the study of the detailed structure of nano-objects, the interactions between nano-objects, and the role of nanostructures in composite materials. In these studies, the length-scales which characterize the properties of the systems range between 1-100nm. The neutron scattering techniques used for these studies range from diffraction to small angle scattering and reflectivity completed by other spectroscopic characterization methods. More specifically the topics which are addressed at the LLB cover the following areas: Magnetic nanostructures (nano particules – molecular magnets – thin films); Composite materials (Polymer based and metal based nano-composites); Metallurgy (Microstructure, mechanical properties, aging in various alloys or nuclear materials); Confined systems (Microporous or Mesoporous materials , guest-hosts systems for catalysis, energy or biological interest); Disordered systems (Amorphous materials, oxide glasses, liquids).



Axis 3: Soft Complex Matter

The "Soft Complex Matter" axis has oriented his studies to many new systems involving different components, length and time scales that are of major interest in soft matter and biology. Its ambition is to understand both i) the behavior of the individual building blocks (molecules, nanoparticles, polymers, surfactants and phospholipids) whose characteristic sizes lay in the 0.1–10 nm range and ii) the underlying mechanisms of their self-assembly and dynamics, to, in the end, finely, control and tune the very specific properties of inert, functional or



biological matter at the nanometer scale (1-100 nm). The following topics are presently developed: polymers and hybrid systems; foams, emulsions and asphaltenes; surface and finite size effects on confined systems; protein dynamics and water; crowding in biophysics; membranes and adsorbed molecules. Keeping in mind the advantages of neutron scattering, i.e. labeling and contrast matching , we probe the structure either in bulk (diffraction, SANS) or at surfaces (reflectometry), and measure relaxations up to the several tens of nanoseconds (time-of-flight or spin-echo), completed by other spectroscopic methods and supported by the chemistry and biology platforms.

PART III

The LLB provides world class facility for neutron investigation of materials across a large range of science disciplines. It is operationally organized in instrumental and technical groups and platforms as mentioned above around the spectrometers. Each group presents its activities, instrument suite, challenges and projects, with special attention on highlights; it also considers international interactions via NMI3 (JRA), and possible ESS contributions.

The Orphée reactor is a medium power research reactor designed to produce neutrons beams for research and some industrial applications (silicon doping, radio-isotopes production and industrial neutron radiography). The 14-MW compact core is cooled by light water with a heavy water reflector. It provides a thermal flux of 3.10^{14}neutrons/cm^2/s. with a core life time of about 100 days; to ensure radiological shielding and easy handling, the core and the heavy water tank are immersed in a pool filled with demineralised light water. The reactor hosts 9 horizontal and 9 vertical channels. 27 experimental ports are currently available. The reactor went critical in December 1980, and the recent safety assessment carried out in 2010 was successfully completed allowing its operation until 2020. The results of the 'post-Fukushima' evaluation were recently given by the French safety authorities and the modifications are under way until 2015.The Orphée reactor is a reliable source providing about 180 full power equivalent days (JEPP in French); however, some unexpected shutdown might happen reducing its activity and the beam time access.

In fall 2013, 19 spectrometers are in user operation, they cover the whole range of neutron scattering techniques thanks to the three types of moderators of the reactor, cold thermal and hot. In addition there are 2 instruments for tests and 4 under full construction. The instrument suite ranges from

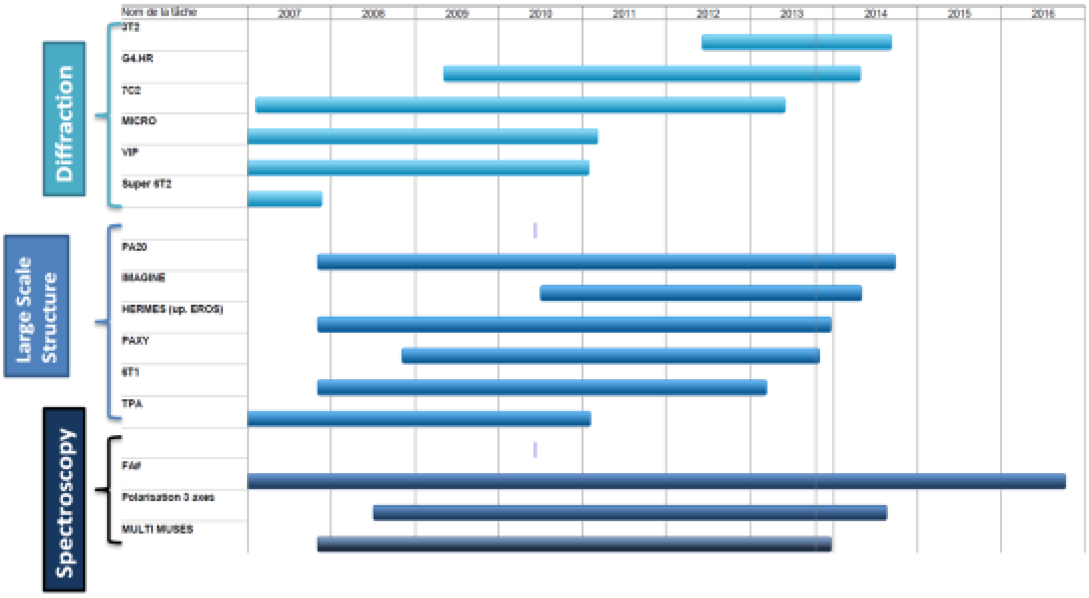
- diffractometers in the **Diffraction Group** for liquids, powders and single crystals, to

- texture and strain, Small Angle, VSANS, reflectometer, imaging via the **Large Scale Structure Group**, and

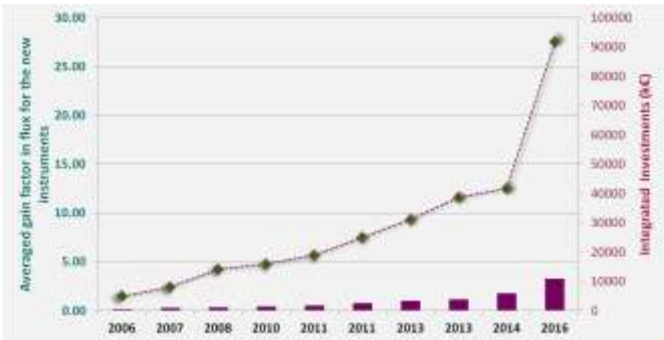
- medium to high resolution spectrometers with Triple Axes, Quasi-elastic and Spin Echo Scattering in the **Spectroscopy Group**

Programs (CAP2010-2015-17) for the renewal of the instrumentation were required to allow our facility to stay internationally competitive. An overall strategy for an infrastructure in terms of instrumentation is always a balance between a process of prioritizing new instruments and a process of assessing the performances of the existing ones. The strong connection with the French user community and the deep involvement in research and technical expertise of our staff helped in defining instruments for scientific requirements. In 2005, 7 projects were already selected for modernization (super 6T2, 3T2, Micro, 7C2, TPA, Fa#, Eros2 and VIP); unfortunately the lack of budget did not allow their developments, since the budget was squeezed continually by everyday operating needs. It is only recently that most of them are supported and completed by new ones (6T1, PA20, Hermes-Eros3, Paxy, Multimuses, Imagine, 3axis polarization, G4.4). Moreover their successes depended strongly on the technical groups created at the end of 2008: for **Instrument Development** and design, **Sample Environment**,

Electronics and **Information Technology**. In the figure below, the actual project management, including pre-project studies, design, realization and commissionning, is presented and one can see the current achievement within the three instrumental groups.

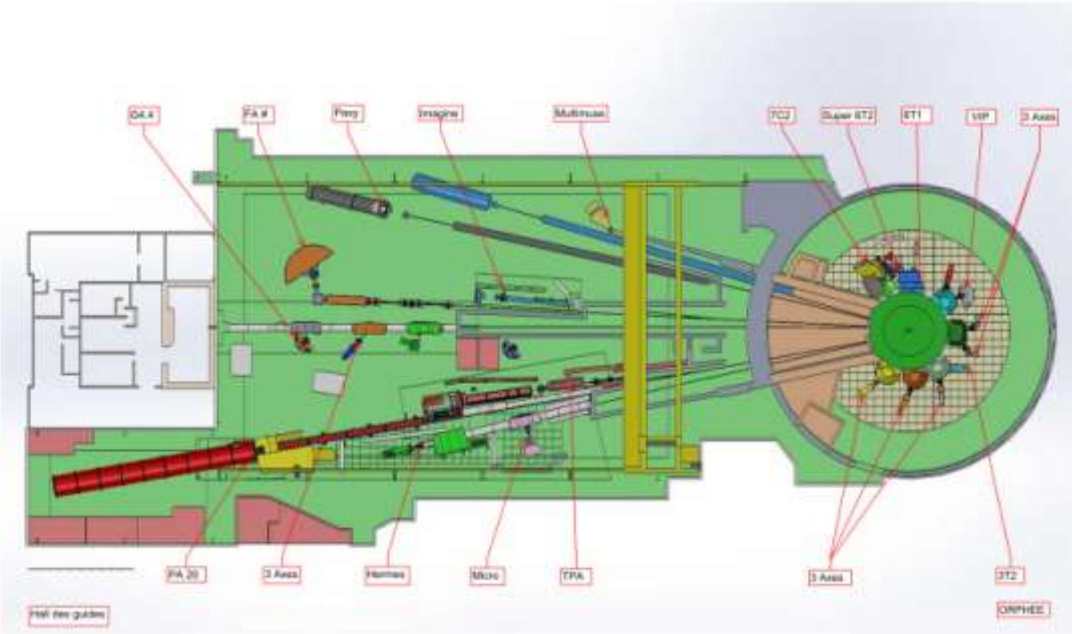


The technical groups are also accompanying the instrument technician and scientist for the maintenance and the development of required sample environments. The support to experiments is also provided by the common **Platforms** recently implemented for **Biology**, **Chemistry** and for **Theory and Modeling**; their goals and the services proposed are described as for the other groups.



By the end of 2016, the LLB facility will offer 15 new world class instruments which performances will deserve academic and industrial studies; the averaged gain factor over all these instruments is up to 27 for a minimal integrated cost of 11M€; this investment is not only used for a gain flux but also to improve the ratio signal over noise or extend the some typical environment (such as high magnetic field). The figures below illustrate their position in the infrastructure.

However, we must face now a manpower problem, manpower that is never included in project plans: many instruments are run by a single scientist and a half technician, those technical and scientific expertise will be completely lost when they will leave the LLB.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Multimuses
7C2
Super 6T2
6T1
VIP
3 Axes
PA 20
3 Axes
Hermes
Micro
TPA
3 Axes
3T2
Hall des guides
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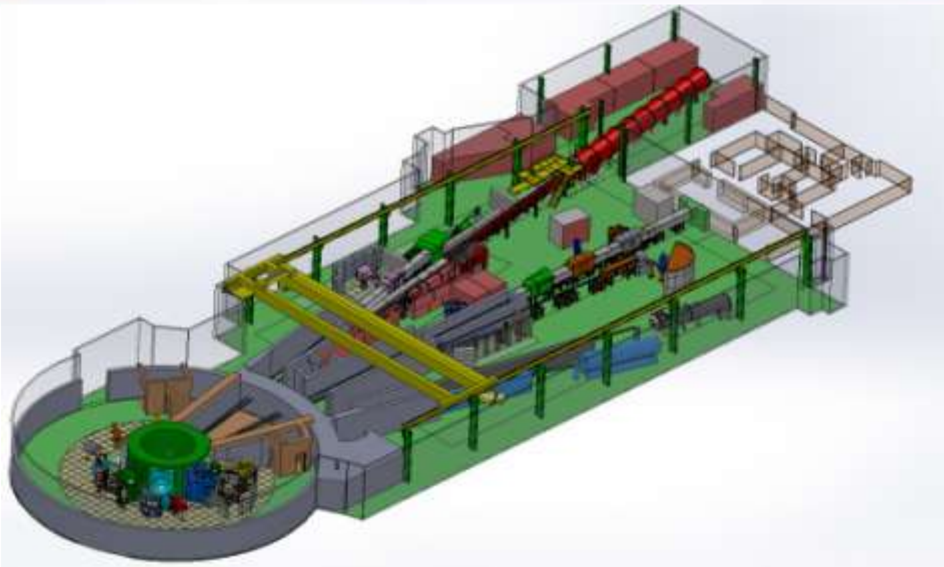

PART II

REPORT ON THE LLB RESEARCH ACTIVITIES

Axis 1: Magnetism & Superconductivity

The scientific programs developed by condensed matter physicists at LLB cover a large number of research areas, at the forefront of science and technologies. These programs, through strong partnerships with the French and international research communities, rely on the unique possibilities offered by neutron scattering techniques to explore a whole spectrum of physical phenomena occurring in condensed matter.

Strongly correlated electron systems and complex magnetic systems are central topics of nowadays condensed matter physics and many body problems. The extensive study of static and dynamical magnetic correlations through magnetic neutron scattering, including polarization analysis, provides the inputs that are essential to describe the microscopic state and to understand its fundamental properties, a necessary step before the elaboration of new theories and concepts.

In a number of modern functional magnetic materials, one is often confronted with complex spin configurations, i.e., non-collinear, or incommensurate, such as helimagnetic magnetic structures, which result from frustrated magnetic interactions. Since the macroscopic functional responses in these materials are direct consequences of their complicated magnetic structures and fine tuning of their magnetic interactions, the detailed knowledge of their crystal and magnetic structures, and of their magnetic excitation spectra, is mandatory. In the classical cases, the study of spin wave excitations in the magnetically ordered state will lead to the appropriate modelling of the relevant magnetic interactions in the system. However, it is also often required to identify and understand unconventional excitations, very different from classical spin waves: indeed, even in the absence of long range magnetic order, collective magnetic excitations can be observed, like in superconductors, Kondo-insulators, low dimensionality quantum magnetic systems, or spin liquids.

In this general framework, the recent advances in LLB, not mentioned in the previous AERES report, have focused on:

Superconductivity without phonons

Superconducting materials provide a new technological solution to the problems of energy distribution and efficiency. Nowadays, the technology based on superconducting cables exists and is fully operating at a small scale. There is no doubt that new classes of superconductors await discovery, but such breakthroughs will be favored by guided investments in materials synthesis. In this respect, LLB physicists try to shed light on the microscopic mechanisms driving unconventional superconductivity. High temperature superconductors, such as cuprates and iron-based materials, exhibit strong electronic correlations, yielding to exotic phase diagrams, in which superconductivity can coexist or compete with other states of matter (spin density wave state, nematic electronic state, loop-current order). Coupling of electrons with different kinds of magnetic bosonic modes can lead to new superconducting pairing mechanisms, without phonons and unconventional electron liquids. [*see highlight 1*].

Exotic ground states in 4f-electron systems

Fermi liquids provide a fruitful theoretical model of interacting fermions, describing the normal state of most metals at sufficiently low temperatures. However, anomalous properties observed in certain strongly correlated electronic systems challenge our current knowledge of metallic systems. Understanding the ground states of heavy-fermion, mixed-valent, and quantum-critical alloys requires a detailed investigation of magnetic fluctuations and excitations. In 4f electron systems, the competition between exchange interactions and Kondo-type magnetic fluctuations produces renormalized Fermi-liquid quasiparticle states, heavy effective masses and sometimes, heavy-fermion superconductors. Some materials do not fit into this framework, however, because other degrees of freedom (such as the multipole moments) are involved, or because the renormalization leads to a semiconducting ground state (Kondo insulators). [*see highlight 2*]

Geometrically frustrated magnets & monopoles

In chemically ordered compounds with short - range magnetic interactions, geometrical frustration appears when all interactions cannot be satisfied simultaneously, owing to the lattice geometry. A well-known example is a triangle of antiferromagnetically coupled spins. Frustration results in a strong degeneracy of the magnetic ground state, since many configurations have the same energy. It now appears as a powerful ingredient in the design of new materials and the tuning of physical properties, as frustrated magnetic states may be easily switched by small perturbations in the energy balance. Pyrochlores $R_2Ti_2O_7$ (where R^{3+} is a rare earth ion) are model systems showing exotic short-range magnetic orders called " spin liquids" and " spin ices", with unconventional excitations akin to magnetic monopoles (namely, single quasiparticles, which behave as individual sources or sinks of magnetic field). Current research at LLB, in collaboration with SPEC, focuses on the $Tb_2Ti_2O_7$ spin liquid, also called quantum spin ice. An antiferromagnetic order of static monopoles has been observed, as well as a cooperative excitation at very low energy, suggesting a strong and original coupling between crystal and magnetic lattices [*see highlight 3*]

Chiral magnetic & skyrmions

In chiral itinerant magnets such as MnSi, FeGe and the recently synthesized MnGe, long wavelength magnetic spirals (up to 1000 Å) are observed, possibly stabilized by the competition between a strong ferromagnetic exchange and spin-orbit interaction. At the borderline between ordered and paramagnetic phases, these frustrated magnetic interactions induce complex spin arrangements, such as skyrmions, created either as regular lattices or standalone particles. Skyrmions or magnetic vortices could provide building blocks for new complex magnetic textures, which could be either manipulated with electric or spin currents or, conversely, used to direct the motion of spins and charges, with potential applications for data storage. They also lead to new electric features, such as the Topological Hall Effect (THE). Researches at LLB focus on the magnetic phases of these chiral magnets, and their possible tuning by temperature, pressure and magnetic field. In MnSi[1], a magnetic "blue phase" was observed as in liquid crystals, which consists of a spin liquid phase of chiral skyrmions. In MnGe[2], neutron studies in collaboration with a Russian group showed the low angle satellite for the first time, the short period of the helix being at the origin of its giant THE. Studies of quantum critical points encompassed by applying pressure are also being performed.

Multiferroic & electromagnons

The discovery of multiferroic materials, in which ferro-electricity and magnetism are intimately coupled, allows one to design promising new electronic devices, such as high-speed memories with magnetically and electrically addressable states. In this context, major efforts are currently carried out by LLB physicists, in collaboration with various French laboratories (ICMMO, CRISMAT, Institut Néel), to understand multiferroic properties on fundamental grounds. A strong spin lattice coupling combined with magnetic frustration seems to be the basic physical ingredient for multiferroicity, giving rise to delicate and complex magnetic structures, as well as unconventional excitations, the celebrated electromagnons. Neutron scattering experiments have proven to be an unparalleled tool in this field, resolving complex magnetic structures, in complex sample environments including high-pressure[3], and allowing to determine the delicate balance of magnetic interactions which stabilize the ground state. Current studies at LLB focus on $GaFeO_3$, $LuFe_2O_4$[4], $RMnO_3$ and RMn_2O_5 compounds, where R is a rare-earth ion (R = Ho,Yb,Y,Tb), as well as on stacked triangular lattices, like $CuCrO_2$ and delafossite-related materials[5].

Molecular magnets & photo-induced magnetism

Molecular magnetism is a relatively new field of research, which has attracted growing interest among physicists since the discovery, fifteen years ago, of the first "single molecule magnet" (SMM), Mn_{12}-acetate, which behaves as a magnet at the molecular scale. Molecular magnets are materials which are currently still of fundamental interest, but which will become technologically significant in the future, with the growing demand for increasingly small and faster magnetic memories. The present challenges in this field consist in understanding how to control magnetic anisotropy in order to obtain SMM behavior at higher temperatures, and in designing new multifunctional materials possessing other functional properties in addition to magnetism, e.g. magnetic photo-switchable compounds. Several French chemistry groups are represented in these studies and actively collaborate with LLB physicists. [*see highlight 4*].

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1. Spin or orbital magnetism for high-T_c superconducting cuprates

After 20 years of intense research, the mechanism of superconductivity (SC) in the high-T_c cuprates is still unknown. The observed structure of the low energy electron spectrum is often considered as a signature of a strong electron coupling to some boson, which then is suspected to be a mediator of the SC pairing. In addition, the cuprate phase diagram exhibits a mysterious pseudo-gap (PG) state, out of which the SC state emerges. Elucidating the role and nature of this PG state and identifying the boson mediator in cuprates are more than ever the hot topics for condensed matter physics. At LLB, theorists and experimentalists explore different scenarios in parallel.

The LLB theory group has recently focused on the study of the impact of spin fluctuations (SFs) (observed by neutron spectroscopy) on electronic properties. At high carrier density, the spin fluctuation spectrum is dominated by a spin resonance mode. Considering the strong coupling of electrons with this mode, the microscopic theory[6] (without adjustable parameters but with the use of known properties of SFs and bare electrons) is able to give the most important electronic anomalies, among which: the d-wave symmetry of pairing, the high absolute value of the SC gap, its U-shape angular dependence and the anomalous form of electron density of states. At low carrier density, the spin fluctuations become critical in approaching the antiferromagnetic (AF) instability, i.e. become strong and soft. The theoretical studies[7] of their impact on electronic properties show that quite surprisingly and contrary to the case of phonon-mediated superconductivity, they rather destroy than mediate superconductivity. Indeed, the same SFs destroy the coherence of the anti-nodal electrons, and tend to promote competing spin density wave instability. This leads to a SC state exhibiting anomalous electronic properties close to those observed experimentally (ARPES, STM, ERS). Only when their characteristic energy of becomes relatively high and their intensity decreases, SFs become most effective for pairing. In this way an optimal doping appears at some distance from the AF quantum critical point. All these results, obtained within strong coupling model and using experimentally observed SFs as an input, constitute an important argument in a favor of a spin fluctuations mediated superconductivity in the high-T_c cuprates.

Besides, researches performed within the LLB spectroscopy group have addressed the issue of the PG phase intrinsic nature. Polarized neutron diffraction shows that the PG phase is an ordered state, characterized by an intra-unit-cell magnetic (IUC) order. This IUC order has been identified in four distinct superconducting cuprate families over a large range of carrier concentrations[8,9,10,11]. This order state breaks time-reversal symmetry (Figure 1), but preserves the lattice translation invariance, as theoretically proposed in the loop-current theory of the pseudo-gap. Within this model, a set of currents flowing between copper and oxygen produce staggered orbital magnetic moments within the unit cell. The rotation of the loop current pattern is predicted to produce two discrete magnetic modes. In the prototype system $HgBa_2CuO_{4+\delta}$ (collaboration LLB/Minnesota Univ.) two quasi-non dispersive modes have been indeed recently reported[12], providing further experimental support to the loop-current scenario. Coupling of electrons with loop-current fluctuations could provide a new route towards unconventional superconductivity in cuprates.

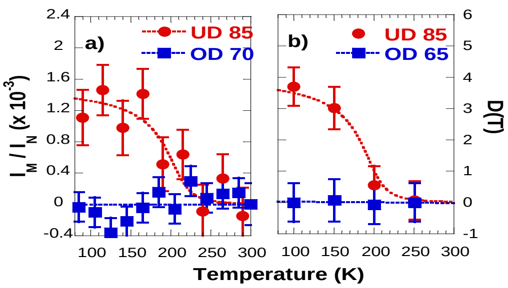

Figure 1 Two distinct techniques[11], polarized neutron scattering (a) and circularly polarized photoemission spectroscopy (b), demonstrate on a common system, $Bi_2Sr_2CaCu_2O_{8+\delta}$, that time reversal symmetry is broken in the PG state. Different symbols stand for samples where PG develops around 200 K (red) or is absent (blue): (a) full magnetic intensity normalized by the nuclear intensity on the (101) Bragg reflection and (b) dichroic effect observed at the anti-nodes. (collaboration LLB/ IUT-Blois).

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2. Exploring new avenues in *f*-electron magnetism: Unconventional order and spin dynamics in Kondo insulator $CeRu_2Al_{10}$

The low-temperature properties of heavy-fermion systems—including, in some of them, the occurrence of unconventional anisotropic superconductivity—are dominated by strongly renormalized quasiparticle states with very large effective masses. Most of these compounds exhibit metallic, Fermi- (or sometimes non-Fermi-) liquid behaviors. However, cases have been reported ($Ce_3Bi_4Pt_3$, $CeOs_4Sb_{12}$, etc.) in which a similar renormalization of the electronic states eventually leads to the opening of a very narrow gap (a few tenths of to a few milli-electronvolts) at the Fermi energy. A new "Kondo insulator" regime thereby occurs when the material is cooled below a characteristic temperature T^*, which is typically in the 10–100 K range.

The recently discovered CeM_2Al_{10} (M: Ru, Os, Fe) family of orthorhombic Ce compounds is of outstanding interest because it presents a very unique interplay of Kondo insulator properties and long-range magnetic order, which can further be tuned by means of hydrostatic pressure or chemical substitution. We have undertaken a comprehensive study of those "1-2-10" compounds in collaboration with Prof. Sera's group at Hiroshima University. In $CeRu_2Al_{10}$, the first step was to ascertain the magnetic nature of the phase transition observed at T_0 = 27.3 K, which was originally questioned on the basis of ^{27}Al NQR results, and to characterize the order parameter.[13,14] This was achieved through a series of powder (G4-1) and single-crystal (6T2) diffraction experiments, supplemented by neutron polarization analysis on 4F1 (Figure 2).

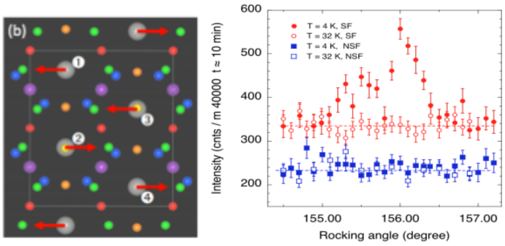

Figure 2 Left: magnetic structure of $CeRu_2Al_{10}$ in the (b, c) plane with Ce moments (μ_{Ce} = 0.34 μ_B) oriented along c. Right: temperature dependence (4 K and 32 K) and polarization analysis of the 301 magnetic Bragg peak (double-peak arises from two crystallites with slightly misaligned axes).

The antiferromagnetic (AF) order in $CeRu_2Al_{10}$ has some intriguing issues. The high value of T_0, as compared to T_N = 16.5 K for $GdRu_2Al_{10}$ (despite $\mu_{Gd} \approx 7\ \mu_B$) suggests an additional coupling channel besides normal RKKY interactions. Furthermore, the direction of the ordered AF moments $\mu_{Ce} \parallel c$ contradicts the single-ion anisotropy observed in the paramagnetic state, which strongly favors the a axis. To probe the magnitude and anisotropy of the exchange interactions, we have carried out unpolarized (2T, IN8) and polarized (IN20) inelastic scattering experiments.[15] Below T_0, a spin gap of about 4 meV is observed at the AF zone center, and dispersive magnon branches span an energy range extending up to 8.5 meV. Most interestingly, a very large anisotropy is found between the transverse components of the magnetic correlations: $\langle m_a(i)\, m_a(j)\rangle \gg \langle m_b(i)\, m_b(j)\rangle$. Magnon calculations[15] performed in the RPA approximation satisfactorily reproduce the data provided one assumes a strongly enhanced AF coupling, $J_c \approx 58$ K $\gg J_a$, $J_b \approx 2.7$ K, between the c components of nearest-neighbor Ce moments (labeled ① and ➀ in Figure 3).

This phenomenon is thought to result from a strong anisotropic c-f hybridization, already pointed out in previous magnetic[16] and optical conductivity[17] studies. Recent theoretical work further suggests that the CeM_2Al_{10} series could be located close to a localized-itinerant transition.[18]

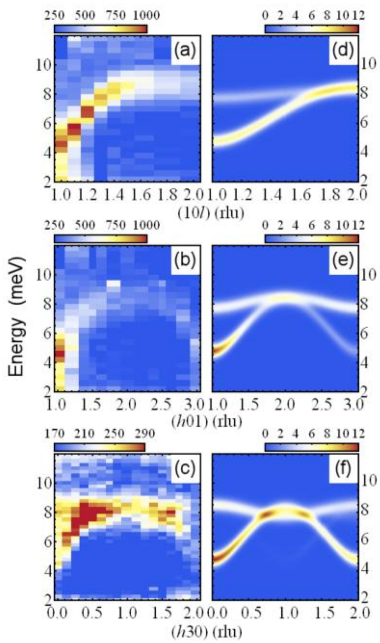

Figure 3: Measured (left) and calculated (right) magnon dispersion branches in $CeRu_2Al_{10}$.

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3. $Tb_2Ti_2O_7$ spin liquid: static "monopoles" and exotic spin excitations

Frustration has been studied extensively for the last two decades, especially because of its strong potential for novel and exotic ground states. Geometrically frustrated pyrochlore magnets, with their lattice of connected tetrahedra, are model systems in this field. For instance, the rare earth titanates $Ho_2Ti_2O_7$ and $Dy_2Ti_2O_7$, where ferromagnetically coupled spins are constrained along 111 axes, show an (almost frozen) correlated paramagnetic state called "spin ice", because its residual entropy is identical to real ice. In each tetrahedron, magnetic moments obey the "two in-two out" ice rule (Figure 4a), a constraint akin to a divergent free fictitious magnetic field. This results in an emergent gauge field structure, and leads to exotic excitations called monopoles, which behave as single magnetic charges. These excitations propagate along a Dirac string of first neighbor spins at low energy cost.

By keeping the same geometry but relaxing the spin ice constraint, even more exotic "spin liquid" or "quantum spin ice" states can be obtained. This can be done by changing the crystal field anisotropy, the exchange interactions, or by introducing quantum fluctuations. The celebrated $Tb_2Ti_2O_7$, a controversial subject for the past 10 years, is a good candidate in this context. In contrast with spin ices, spins fluctuate down to T~0 at neutron time scales. We have been studying $Tb_2Ti_2O_7$ by combining diffraction and inelastic neutron scattering on a single crystal.

a) b)

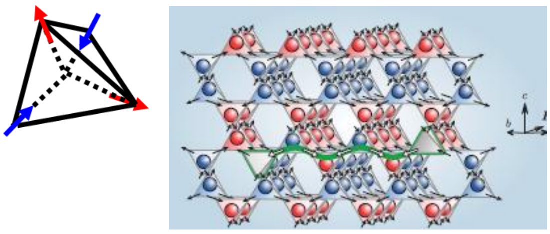

Figure 4: a) the ice rule ; b) double layer monopole order in $Tb_2Ti_2O_7$ spin liquid, deduced from single crystal neutron diffraction data. Monopoles are shown by red or blue balls and spins by black arrows.

double layers[19] (Figure 4b). Flipping a single spin in this static monopole medium would create a In $Tb_2Ti_2O_7$, the magnetic structure induced by a field H//110 can be viewed as a 3 dimensional arrangement of monopole and antimonopole monopole vacancy which can propagate, so that $Tb_2Ti_2O_7$ behaves as the exact mirror image of the $Ho_2Ti_2O_7$ genuine spin ice.

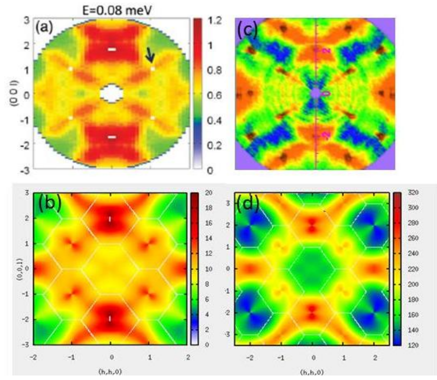

Figure 5 : Inelastic scattering map of $Tb_2Ti_2O_7$ in the (hh0,00l) plane. a) and c) experiment; c) and d) mean field calculation involving anisotropic exchange and symmetry breaking.

Up to now, such single particle-like excitations have not been evidenced in $Tb_2Ti_2O_7$ and this remains a major challenge. We have however observed low energy cooperative exotic excitations at 50 mK, shedding light on the interactions at play. The spectrum consists in a dual response with static and inelastic contributions, showing striking Q-dependencies (Figure 5a). In collaboration with SPEC, it has been modeled using mean field calculations (Figure 5b), involving anisotropic exchange and assuming a symmetry breaking, induced by a distortion, and yielding a two–singlet ground state for the Tb^{3+} ion[20,21]. This model accounts for the main features of the experiment, and for the field evolution of the magnetic order, but fails to reproduce the elastic contribution. More sophisticated approaches, involving quadrupolar interactions and the hybridization of crystal field levels with an acoustic phonon mode, as suggested by recent inelastic polarized neutron data, are being currently investigated.

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4. Probing the local magnetic anisotropy in Molecular crystals

Molecular magnetism gets increasing interest since the discovery of the first single-molecule magnet [Mn12], which behaves like a magnet at the molecular level below a certain temperature called blocking temperature[22]. One of the conditions for the future application of these nano-objects for information storage is the existence of a high energy barrier between the two spin states ±S, which requires a strong axial anisotropy. It is therefore very important to control the factors favoring this type of anisotropy, in particular the influence of molecular geometry on the magnetic anisotropy.

In highly anisotropic paramagnetic compounds, the induced magnetic moments on the atomic sites are not collinear with the applied magnetic field. Macroscopic magnetic measurements only give information on the resulting magnetic moment induced in a given field direction and therefore do not permit to characterize the magnetic anisotropy at the microscopic level.

In contrast, polarized neutron diffraction (PND) enables to probe the real distribution of the induced magnetic moments in magnitude and direction in the crystalline lattice. The recent development at LLB of a methodology based on the local magnetic susceptibility tensor for analyzing PND data [23] enables the visualization of the moments on the different atomic sites. This approach used so far only for inorganic compounds was successfully applied for the first time to the study of the magneto-structural relationships in a molecular compound, $[Co_2(sym\text{-}hmp)_2](BPh_4)_2]$ where hmp = hydroxymethylpyridine [24].

The organometallic complex $[Co_2(sym\text{-}hmp)_2]^{2+}$ is composed of two Co^{2+} ions connected by two bridging oxygen atoms. Each Co^{2+} ion is at the center of a distorted octahedron formed by two nitrogen atoms and four oxygen atoms. Magnetic susceptibility measurements on single crystal clearly show highly anisotropic behavior that can be reproduced within the model of effective spin ½, for two antiferromagnetically coupled Co^{2+} ions, with a tilt angle of 39 degrees between the local moments[24]. However, this model is purely phenomenological and it does not give the true values and orientations of the moments of Co^{2+} ions, which have a 3/2 spin.

The experiment of polarized neutron diffraction on a single crystal of this compound was carried out on the 5C1 diffractometer at LLB at low temperature under a strong magnetic field applied along the a-axis.

Data analysis by the method of local susceptibility tensor provides access to the actual value of the magnetic moments of the Co^{2+} ions (3 μB, as expected for a spin S = 3/2) and their orientation with respect to the molecular geometry: the obtained magnetic moments are symmetrical with respect to the main symmetry axis (b-axis, C2) and in the direction perpendicular to the axis of axial distortion of the octahedron centered on each Co^{2+} ion. Their directions thus are in an angle of 37(±1) degrees which agrees with the refined angle obtained in the spin ½ model.

In conclusion, polarized neutron diffraction shows that the molecular magnetic anisotropy in the Co^{2+} dimer complex is governed by the local anisotropy of each Co^{2+} ion (single-ion anisotropy). The complex behaves as a system of two ions of spin 3/2 antiferromagnetically coupled, the directions of the local magnetic moments being imposed by the local geometry.

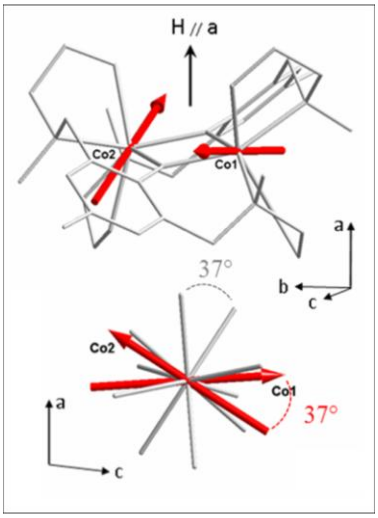

Figure 6 Top: Induced magnetic moments of the cobalt atoms by a 7-Tesla magnetic field H, applied along the a-axis. Bottom: View projected along the b axis (only first neighbors are shown for clarity). The same 37° angle is observed between the directions of the moments and between the local distortion axes

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Perspectives

Superconductivity without phonons

In superconducting cuprates, the theoretical study of the role of spin fluctuations to explain the unconventional properties of the superconducting state will be extended in the context of the t-J model which emphasizes the Mott physics present in cuprates and the role of the exchange interaction J as a direct attractive interaction between electrons. Besides, polarized neutron scattering measurements has revealed that the pseudo-gap state, out-of which superconductivity emerges, is a long range order state, characterized by an intra-unit cell magnetic order. New inelastic polarized neutron scattering experiments will be dedicated to search for the low energy local magnetic fluctuations that could further mediate superconductivity.

Exotic ground states in 4f-electron systems

The so-called 1-2-10 Ce compounds (CeT_2Al_{10}) provide the unique opportunity to study the competition between the Kondo Insulator (KI) formation (T=Fe) and the onset of antiferromagnetic order (T=Ru, Os). This effect touches on the question of local vs. nonlocal quantum criticality, a highly controversial issue in heavy-fermion systems, which is scarcely documented for KI. Our measurements on $CeFe_2Al_{10}$ will keep focusing on the study of “resonant” magnetic modes and the tunability of the KI character of the material by substituting Ru for Fe and/or by applying pressure.

Geometrically frustrated magnets and monopoles

One of the consequences of magnetic frustration is the appearance of new states of matter, like spin ice or spin liquids. We will explore in more detail the ground state and excitations of rare earth based pyrochlores ($Tb_2Ti_2O_7$ and $Pr_2Zr_2O_7$) with uniaxial anisotropy, for which the lack of long range ordering is probably due to a strong coupling with the lattice. The propagation of hybrid spin-lattice excitations is expected to reconfigure both spins and strains over long distances, frustrating long-ranged magnetic order. In parallel, other phenomena, such as the order by disorder mechanism will be explored, on the particular case of Erbium pyrochlores with planar anisotropy.

Chiral magnetism and skyrmions

Chiral magnets of the B20 family attract increasing attention due to their complex and highly tunable spin structures, potentially usable for spintronic applications. Their helical spin structure results from the competition of three interactions. Skyrmions, topologically protected spin textures built on these helixes, can be stabilized in bulk or thin layers. Applying a stress or hydrostatic pressure may induce or reveal a skyrmion lattice. Our current prospects deal with the study of these pressure induced states in the recently synthesized MnGe, where the existence of a skyrmion lattice and the origin of the Giant Topological Hall effect are strongly debated.

Multiferroics and electromagnons

One of the consequences of a strong magneto-dielectric coupling in multiferroic materials is the existence of a new kind of spin excitations, that can be triggered by *ac* electric fields, the electro-magnon. Efforts will be focused in the near future on a better identification of the magnetic and polar components of this electromagnon, using a combination of polarized neutrons scattering and optical spectroscopy techniques (IR, Raman), on known multiferroic compounds, such as hexagonal manganites. In parallel, new candidates for multiferroic properties will be explored, based on crystal topologies favouring competing magnetic interactions, such as magnetic triangles, and playing with new components affecting both the polar and magnetic orderings, such as the isotope effect.

Molecular magnets & photo-induced magnetism

Single Molecule Magnets (SMM) could be used for future applications in information storage. The current challenge is to understand the parameters that govern their magnetic anisotropy. The precise relationships between structure and local magnetic anisotropy provided by polarized neutron diffraction are crucial information for testing the theoretical predictions of the magnetic behavior of such molecules. The early experimental study of highly anisotropic molecular complexes with 3d transition metal ions will be extended to one-dimensional systems and to rare earth complexes.

AXIS 1 Research:
Magnetism & Superconductivity

Keywords

Unconventional superconductivity, multipole orders, Kondo insulators, geometrical frustration, chiral magnets, multiferroics, molecular magnets.

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Scientific collaborations *(in France and abroad are listed below)*

FRANCE	EU	Non - EU
CRISMAT- CAEN ILL - GRENOBLE ESRF - GRENOBLE INT. NEEL-GRENOBLE UNIV BORDEAUX UNIV NANTES UNIV PARIS 11 UNIV PARIS 6 UNIV RENNES UNIV STRASBOURG SPEC - CEA	CZECH REPUBLIC- INST ACAD PRAGUE GERMANY - JULICH GERMANY - KIT- KARLSRUHE GERMANY - MPI – STUTTGART GERMANY - UNIV COLOGNE GERMANY - UNIV DRESDEN GERMANY - UNIV GOTTINGEN GERMANY - UNIV MUNICH UK - RUTHERFORD APPLETON LAB UK - UNIV ST ANDREWS BELGIQUE UC-LOUVAIN	JAPAN - TOHOKU UNIV SENDAI JAPAN - ADSM HIROSHIMA UNIV JAPAN - JAPAN ATOMIC ENERGY AGENCY, TOKAI RUSSIA - NRC KURCHATOV INST. MOSCOW SWITZERLAND- PSI SWITZERLAND- EPFL USA – ARGONNE USA-UNIV MINNESOTA

Scientific contracts

ANR	CEDA, MAGD0, NEWTOM,
	CEDA: Convergence of electron spin, charge and momentum densities analysis MAGD0: magnet ism induced by nonmagnetic impurities NEWTOM: new transition metal oxides
RTRA	EXTREME, GLACEDESPIN, CRISPY, MAGCORPNIC, SUPRA2010
	EXTREME: high-pressure instrumentation. GLACEDESPIN: financial support of post -doc, H. Cao. CRISPY: crystal growth of rare-earth pyrochlores. MAGCORPNIC: magnetism and electronic correlations in superconducting pnictides. SUPRA2010: workshop on superconductivity
JRA	NMI3-7th Framework Program of the European Commission
PALM	NEUTROPRESS, INSPHOTO
	NEUTROPRESS : visit of scientific researcher INSPHOTO:instrumentation for photo-induced magnetism

Axis 2: Materials and Nanosciences - Fundamental Studies and Applications

This scientific axis covers the activities related to the research in materials science and more generally in hetero-systems (i.e., interfaces, alloys, composite materials, and confined systems). The topics cover the study of the detailed structure of nano-objects, the interactions between nano-objects, the role of nanostructures in composite materials and the behavior of amorphous and glassy materials.

LLB nanoscience research benefits from a variety of available neutron scattering techniques: **SANS** (Small Angle Neutron Scattering) - for studying the structure and organization of nanoparticles, **Reflectivity** - to study thin film structures (polymers, magnetic films, liquid surfaces), **Diffraction** - to study the crystalline or the magnetic order of nanostructures. The 4-circles spectrometer (6T2) was recently upgraded to be capable of performing diffraction studies on epitaxial thin films. The different powder diffractometers are also used to characterize the structural and magnetic properties of nanoparticles. Two instruments (6T1 and DIANE) are specifically dedicated to the study of textures and strains in metallurgical materials. The liquid diffractometer 7C2 has been refurbished for studies on amorphous and glassy materials. Other characterization techniques are also readily available for LLB scientists, including light scattering, rheology, magnetometry (VSM-SQUID), I.R. spectroscopy, X-ray reflectivity.

The studies in the fields of materials and nanosciences range from the detailed study of nanoparticles (structural and magnetic properties) to the role of these nanoparticles in composites systems (either metallic or polymer). The scales which characterize the properties of these systems range between 1-100 nm.

The following topics are presently developed at the LLB:

Nanosystems / Heterosystems

Polymer based and metal based nano-composites, magnetic nano-objects (nano particules – molecular magnets – thin films), microporous and nanoporous materials (zeolites, MOFs and hybrids (guest@host)), confined molecules and liquids, structures and nanostructures of biological and pharmaceutical interest.

Physical metallurgy

Microstructure, precipitation, crystallographic textures mechanical properties, aging.

Disordered systems

Liquids (simple, polymeric, ionic), liquids in special conditions (high pressure, confined), oxide glasses and chalcogenides, amorphous solids, glass transition and crystallization, amorphous alloys, glass formation.

Nanocomposites

In the field of nano-composites several PhD thesis have been supported (~8). PhD thesis have ranged from the study of the grafting of polymer chains on oxide particles, to the study of the dynamic and conformation of these polymer chains around these particles, the fabrication of reinforced polymer materials (isotropic[25] and anisotropic[26]) to the fabrication of bonded rare-earth free permanent magnets[27]. This last topic is supported by a FP7 European contract. These studies often combine SANS and SAXS characterizations which are performed at SOLEIL.

NanoMagnetism

In the field of nanomagnetism, a number of studies have focused on magnetic thin films [*see highlight 5*]. We should underline that the 4-circles diffractometer 6T2 is now especially suited for such studies thanks to the use of a Position Sensitive Detector[28-29]. These studies are also performed on the polarized neutron reflectometer PRISM. Among the different recent studies a large effort has been dedicated to Cr epitaxial thin films and tunnel junctions (1 PhD thesis) in which the local order at Cr/MgO interfaces has been studied by neutron diffraction, ARPES, X-ray diffraction and polarized neutron reflectivity. Other studies on the coupling in oxide layers have been performed using polarized neutron reflectometry in collaboration with IRAMIS laboratories (interfacial coupling between $SrRuO_3$/$LaSrMnO_4$ thin films[30], anti-ferromagnetic exchange coupling via an organic layer[31]). In the field of nanomagnetism there is also currently a renewed effort to study photo-magnetic materials (1 PhD). 4-circles diffraction, powder diffraction, inelastic neutron diffraction, SANS and

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[26] A. S. Robbes et al, Macromolecules **44,** 8858-65 (2011).
[27] T. Maurer et al, J. Appl. Phys. **110** (2011).
[28] A. M. Bataille et al, J. of Appl. Phys. **105** (2009).
[29] A. M. Bataille et al, Journal of Applied Crystallography **46,** 726-35 (2013).
[30] A. Solignac et al, Phys. Rev. Lett. **109** (2012).
[31] C. Blouzon et al, Appl. Phys. Lett. **103**, 042417 (2013).

reflectivity are used to study these systems at various length-scales.

Metallurgy

In the area of metallurgy, a wide research program is conducted in collaboration with CEA/DEN, to develop ODS (Oxide Dispersion Strengthened) steels [*see highlight 6*]. The objective, as part of a thesis completed at the LLB, is to control the formation of nano-oxides and their influence on the mechanical properties. The characterizations of the microstructures are performed by SANS and by deformation measurements in situ under load[32]. Moreover, in the field of crystallographic textures and deformations, LLB is partner of the METAFORES ANR launched in November 2012. This project is focusing on high mechanical strength and high electrical conductivity Cu/Nb metallic nano-composites wires, whose primary application is the development of high field pulsed magnets. These materials are composed of a Cu matrix reinforced by Nb nano-filaments. The aim of the project is to understand the thermo-elasto-(visco)-plastic behavior of these Cu/Nb architectured nano-composites by coupling microstructural and mechanical analyses at different scales to a micromechanical modeling. Neutron diffraction being a unique non-destructive tool for the measurement of internal strains in individual phases, the 6T1 diffractometer will allow the determination of strain pole figures which is an essential input to the characterization of the Nb and Cu mechanical responses at various scales.

Confining Materials and Guest-Host systems

Other fields of study cover (guest@host) systems [see highlight 7]. A PhD thesis has been started on this topic in collaboration with the SOLEIL synchrotron and Versailles-St Quentin University. The thesis focuses on the archetype MOF MIL-53 and relies on the collaboration between the three teams of the Plateau de Saclay taking advantage of their specific equipments and savoir-faire (Multiprobes solid-state NMR at UVSQ, High flux XRPD at SOLEIL, NPD at LLB). Concerning nanoporous silicates, we proposed a novel and unusual method for loading an aqueous solution of Cu II into hydrophobic MCM-41 materials under high pressure up to 400 MPa. The aquated Cu II species are retained in the pores creating a hydrophilic environment within a hydrophobic one. Combination of IR, TEM, adsorption isotherm measurements indicate that the structure within the hydrophobic pore is modified by the presence of the Cu II aquated species. This unusual method demonstrates how an aqueous environment surrounded by a hydrophobic one can be created using pressure and a metal salt on the nanoscale.

At LLB, the study of such materials will benefit of the new high resolution powder diffractometer G4.4, dedicated to the structural studies of complex systems in complement to the high flux, large scale-structure diffractometer G6.1.

Liquids and Glasses

Short-wavelength neutrons from the hot source of the ORPHEE reactor offer a decisive tool for the characterization of short-range order in non-crystalline materials (i.e., liquids and amorphous materials). The 7C2 diffractometer allows to measure structure factors over a widely scattered vector range so that pair distribution functions can be determined.

There is considerable interest in phase transition in liquids, and some very accurate studies performed at the LLB have concerned the melting of some simple elements. However, the emerging fields of interest for the structure of liquids and amorphous materials are often related to engineering materials, such as the covalent glasses [*see highlight 8*] or the new bulk metallic glasses with their high level mechanical properties [33]. In the metallic or metalloid elements of the 14-16 group, some very accurate measurements of the thermal properties and the anomalous thermal behavior of sound velocity were carried out[34] . Studies of confined molecules[35] and liquids[36] are also being performed the spectrometer 7C2.

Finally, recent dynamic experiments at the LLB show that it is possible to measure shear elasticity in liquids at low frequencies[37] whereas it was known at very high frequencies only. The long timescales involved point out solid-like properties so far neglected in various liquids. Further investigations are under way (1 PhD student).

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5. Dual anti-ferromagnetic coupling at $La_{0.67}Sr_{0.33}MnO_3/SrRuO_3$ interfaces

Magnetic tunnel junctions (MTJ) are composed of a pinning layer which is insensitive to external magnetic fields and of a soft magnetic layer which senses the external applied field. Metallic MTJs exhibit magnetoresistances up to several hundred percent with MgO barriers. $La_{0.67}Sr_{0.33}MnO_3$ (LSMO) based tunnel junctions are much less investigated despite magnetoresistances of thousand percent [3]. This is due to the major temperature dependence of LSMO magnetic properties and to the requirement of a stable pinning layer compatible with LSMO growth. For this pinning layer, a very promising one is the LSMO/SrRuO3 (SRO) bilayer which can be seen as a prototypal full-oxide heterostructure and which shows an antiferromagnetic order whereas both materials are ferromagnetic. In this work we have resolved the complex ferromagnetic behavior of the LSMO/SRO coupled bilayer through a combination of magnetometry measurements, X-Ray diffraction (XRD) and Polarized Neutron Reflectometry (PNR) as experimental tools while using an extended Stoner-Wohlfarth model to analyze our data.

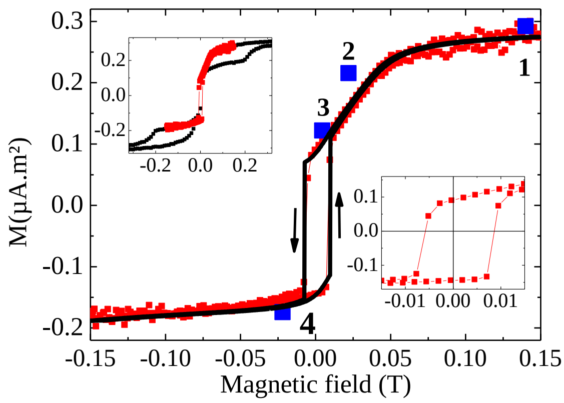

Figure 7 Minor magnetic hysteresis cycle of a bilayer LSMO/SRO, corresponding to the LSMO switching, while the SRO magnetization is blocked at 45° with respect to the positive direction of the applied field. The simulation using two coupling strengths is presented in black. The moment calculated from the PNR data are shown in blue. The inset at the top is a superposition of the minor and major loop. The inset at the bottom is a zoom of the central part of the hysteresis.

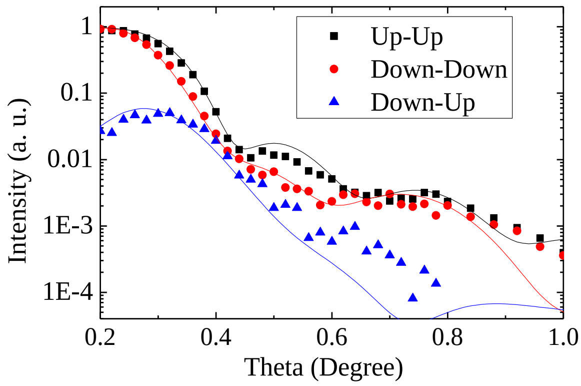

Figure 8 Typical polarized neutron reflectivity measurement. (at point 2 in the previous figure; at 80K).

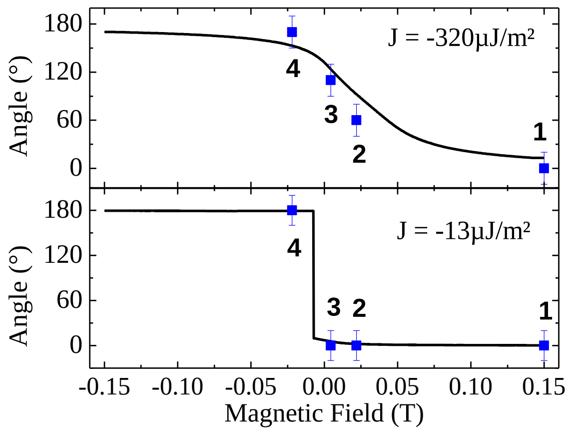

Figure 9 LSMO magnetization angles simulated (line) and measured from PNR (dots) in both domains: high exchange coupling, J=-320µJ/m² (a) and low exchange, J=-13µJ/m². The SRO layer is blocked around 45° in all the cases.

By introducing an extended Stoner-Wohlfarth model, we have been able to describe both qualitatively and quantitatively the experimental results. We found that in epitaxial LSMO/SRO bilayers two well-defined areas with different exchange coupling appear: one with a strong exchange energy of -320µJ/m2 and the other with a weak exchange energy of -13µJ/m2. The experimental evidence of two magnetic coupling strengths in this oxide magnetic heterostructure calls for further investigations in order to elucidate the origin of this dual coupling.[38]

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6. Effect of chemical composition on the coarsening kinetics of the oxides in ODS Fe14%Cr steel

The development of nuclear reactors for future generations requires the availability of structural materials with very high mechanical creep properties and excellent resistance to irradiation. In this context, Fe 9-14% Cr steels strengthened by a fine dispersion of oxides $Y_2Ti_2O_7$ are booming. These materials are obtained by powder metallurgy. One fabrication route is to produce alloyed powders by mechanical alloying and consolidate the material by hot extrusion or HIP (Hot Isostatic Pressing). The precipitation occurs during the hot consolidation step and evolves during subsequent annealing at high temperature. However, the tensile or creep behavior of ODS steels varies with the oxide distribution. With a view to understand the mechanisms of precipitation and the nanostructure evolution, the oxides precipitation coarsening was studied as a function of nominal content of Y, Ti and O of the alloys using small-angle neutron scattering technique (SANS) and Transmission Electronic Microscopy (performed at CEA/SRMA)[39,40].

The effect of the fluctuation of the Y, Ti and O contents around reference values, usually used, was analyzed with the dual objective of specifying the precipitation kinetics and the recrystallization conditions between 850 ° and 1450 ° C. The reference material is a Fe-14Cr-1W-0, 0-3Ti, $3Y_2O_3$ ferritic steel, and 4 nuances were elaborated with different additions (1% FeO_3, 1% TiH_2, 1% and 0.3% Y).

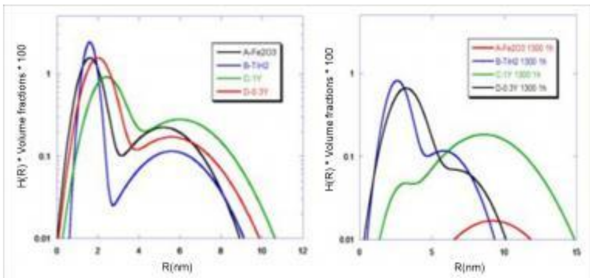

Figure 10 : Oxides size distribution determined by SANS in the different alloys at the consolidated state then after annealing of 1h at 1300°C.

Dispersions of nano-oxides formed after consolidation in the various alloys are close enough (bimodal centered on 2 and 5 nm).Their crystallographic structure is consistent with the expected Pyrochlore phase $Y_2Ti_2O_7$. On the other hand, after annealing at high temperature (between 1300 and 1450°C), we note a very wide disparity in the nanostructure evolution.

It was highlighted that the shape, size, and orientation relationship between the particles and the matrix are clearly dependent on the material. For alloys with a concentration ratio [Ti]/[Y] near or greater than 1, nano-oxides are cubic with coherent orientation relationship with the matrix (cube on cube ([100] oxides//[100] matrix).) The coalescence is so slow. In the yttrium rich alloy ([Ti]/[Y]<1), the oxides are spherical, large and the orientation relationships are not or partially coherent with the matrix.

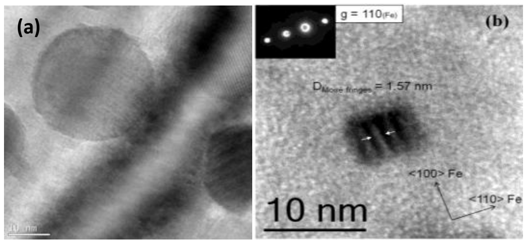

Figure 11 : TEM observations after an annealing of 1h at 1300°C on: (a) Y rich alloy; (b) Ti rich alloy.

The difference of orientation relationship between precipitates and the matrix can explain the differences in behavior during annealing. The energy of oxide/matrix interface depends on the orientation relationships and increases with the disorientation. This parameter governing in part the coarsening kinetics, it explains the pseudo stability of oxides for alloys with [Ti] / [Y] ≥ 1 even at high temperature (1450°C). Contrary, in the O or Y rich alloys, the high energy of interface associated with the disorientation oxide/matrix is responsible of the fast coarsening of the particles. The recrystallization process is then affected. It was observed only in alloys exhibiting large oxides and is blocked on alloys keeping small size oxides.

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[39] *S.Y. Zhong et al.* J. Nucl. Mat., **428,** 154 (2012)
[40] *J. Ribis et al.* J. Nucl. Mat. In Press, *dx.doi.org/10.1016/j.jnucmat.2012.10.051*

7. Studying adsorption in porous materials at LLB

Adsorption rates and selectivity of porous materials depend crucially on the hydrophobicity of their inner surface, the accessibility of non-framework cations (if any) or the flexibility of their skeleton. As structural models with the required accuracy are missing, modelling adsorption at the atomic scale is still challenging. For systems like zeolites, MOF, or MCM-41, NPD, SANS or INS bring unique information on the structure and dynamics of microporous host and confined guest phases, in peculiar through H/D contributions.

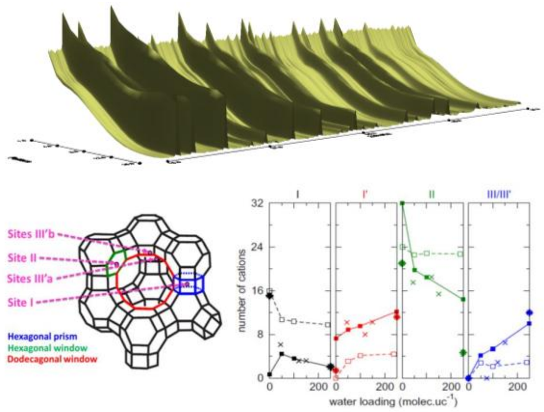

Figure 12: a) *in situ* dehydration of Na,M-X zeolite followed on G4.1 diffractometer. b) Observed and predicted cation redistribution upon water absorption with 2 different framework models.

In basic zeolites, the location of extraframework cations in the cavities controls the adsorption of small molecules but these cations are themselves relocated during ion-exchange or adsorption of polar molecules such as water; on the same time, the framework itself distorts, which would eventually lead to an amorphisation of the zeolite. This, in addition to pristine structural defects (hydroxyl nests, twinning, epitaxy) limits the sorption capacity. This behaviour has been studied[42] by X-Ray and neutron diffraction and Monte-Carlo simulations on bicationic Na,M-X Faujasite zeolites (M = transition metal). The work enlightens the role of cation relocation mechanism on the maximal ion exchange rate and the progressive disruption of the framework. It also proved the inadequacy of usual rigid-framework models to reproduce the adsorption isotherm in these zeolites.

In the emblematic MIL-53 Metallo-Organic-Framework, the breathing phenomenon is extreme with a volume variation of nearly 300% between the « open » and « close » phases[41].

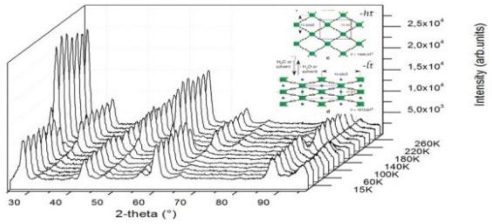

Figure 13: Variation of the pore volume in MIL-53-type materials *Pnma* to *I*2/*a* phase transition in dehydrated MIL-53(Al) followed by NPD (G6.1 diffractometer, LLB).

Despite such a tremendous change, the structure retains its crystallinity and evolves from space group P*nma* to *I*2/*a*. Although, there is still a controversy about the mechanism of this phase transformation (layer-like[42], or cell-by-cell, …) where NMR, diffuse scattering, diffraction and INS could bring useful information. This problematic is the topic of a joint LLB-Soleil-UVSQ thesis (D. Foucher, started in 2012).

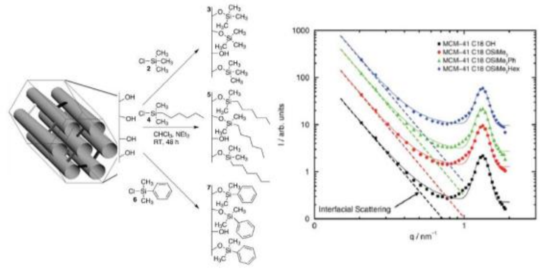

Figure 14: a) $Si(CH_3)_2R$ functionalised MCM-41 and b) their SANS diagrams (PAXE SANS machine, LLB).

Concerning mesoporous silicas, SANS is used, in compliment to SAXS and gas adsorption isotherms, to assess the pore structure of functionalized materials, as for example MCM-41 with post-synthetic grafting of $-Si(CH_3)_2R$ groups (R = methyl, phenyl, hexyl). Study[43] on this system gave access to the raw pore size (SiO_2 walls), the effective layer width, its coverage and the conformation of the hydrophobic layer (tilt angle of the R-groups. The influence of channels size and hydrophobicity on confined guest phases under pressure is also investigated.

41 C. Serre *et al*. J. Am. Chem. Soc. **124** , 13519 (2002)
42 M. Jeffroy *et al*. Microporous and Mesoporous Mat. **138**, 45-50 (2011) and references therein.
43 M. Schoeffel *et al*. J. Mater. Chem. **22** (2), 557-567 (2012).

8. Local Order in Te-rich Te-Ge-X (X=Ga, I, Se) for Infra-Red optical glasses

Due to their excellent glass forming ability, selenium based glasses are extensively used as optical fibers in the mid infrared region. However the cut off of these glasses is near 14 µm, making them inappropriate for novel far infrared applications. Replacing selenium by a heavier element e.g. tellurium shifts the cut off to longer wavelengths, but tellurides are poor glass formers. An extremely fast quenching of 10^6 K/s is necessary to obtain small chips of vitreous Te. In order to shape lenses or fibers for optical devices, one must improve the glass forming ability of such glasses.

Glasses with a good thermal stability can be obtained in the binary Ge_xTe_{100-x} system at x≈15-25[44], but the quenching rate around 10^3 K/s restricts the thickness to about 0.05 mm. The addition of gallium, iodine or selenium is used to improve the glass forming ability and thermal stability of binary Ge-Te glasses.

Alloy	N_{TeTe}	N_{TeGe}	N_{TeX}	N_{Te}
$Te_{85}Ge_{15}$	1.41	0.70	–	2.11
TGI	1.00	1.02	0.03	2.08
TGS	1.07	0.93	0.09	2.09
TGG	1.36	0.57	0.43	2.36

Table 1. Coordination numbers of Te obtained in various Te-Ge-X glasses (X=I, Se, Ga)[45].

The aim of our study was to gain insight into the structure of bulk glass formers $Te_{78}Ge_{11}Ga_{11}$, $Te_{70}Ge_{20}Se_{10}$ and $Te_{73}Ge_{20}I_7$ (denoted with TGG, TGS and TGI, respectively) and understand how the third component builds in the host network.

For this purpose, we have carried out neutron and X-ray diffraction measurements as well as extended X-ray absorption spectroscopy (EXAFS) experiments at Ga, Ge, Se, Te and I K-absorption edges. The experimental datasets were fitted by the RMC simulation technique[46]-[47] [5,6]. In this method, large scale atomic models compatible with experiments and physical constraints (e.g. density, coordination constraints) are generated.

The obtained coordination numbers of Te are listed in Table 1, with the corresponding values for $Te_{85}Ge_{15}$ glass. N_{Te}, the average coordination number is close to 2 in TGI, TGS and in the binary glass while it is significantly higher in TGG. And in TGG $N_{TeGe}+N_{TeTe}$ is very close to 2 suggesting that Ga participates in the 'third bond' of Te atoms. Thus, unlike Se or I, Ga does not build into the Ge-Te covalent network. Instead, it forms a covalent bond with the non-bonding p electrons of Te, which results in an increase of the average Te coordination number. This is consistent with the expected role of Ga in the initial composition: catching the Te lone electron pairs to prevent tellurium from crystallizing. Figure 15 shows a schematic model of TGG based upon the above results.

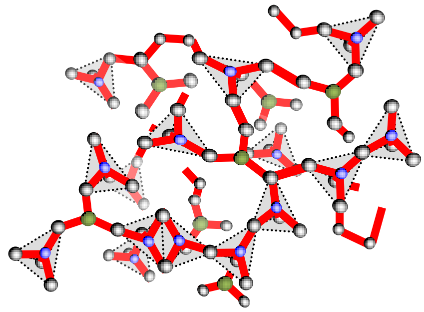

Figure 15. A model structure of TGG with threefold coordinated Te atoms (Ge: blue, Ga: green, Te: gray).

Bond lengths are summarized in Table 2. Our results clearly show that the third component has strong influence on the average Te-Te distance. While the Ge-Te distance is essentially the same in all alloys investigated (2.60±0.02 Å), the Te-Te bond is significantly longer in TGG (2.80±0.02 Å) than either in TGS (2.73±0.02 Å) or in TGI (2.70±0.02 Å). The Te-Te distance in $Te_{85}Ge_{15}$ (2.75±0.02 Å) is just half way between the corresponding values of TGI and TGG.

Alloy	r_{TeTe}	r_{GeTe}	r_{XGe}	r_{XTe}
$Te_{85}Ge_{15}$	2.75	2.60	–	–
TGI	2.70	2.60	2.58	–
TGS	2.73	2.60	2.35	2.60
TGG	2.80	2.60	–	2.60

Table 2. Comparison of the r_{ij} bond lengths (in Å) found in glassy $Te_{85}Ge_{15}$, TGI, TGS and TGG.

According to these observations, the strength of $GeTe_4$ (respectively $GeTe_3I$, $GeTe_3Se$) 'units' is very similar in $Te_{85}Ge_{15}$, TGG, TGI and TGS, but the connection between these units is different. Shorter Te-Te distances in TGS and TGI suggest that Te-Te bonding is stronger in these alloys than in TGG.

By combining different experimental techniques it was possible to determine short range order parameters in Te-Ge-based glasses. It was shown that the improvement of glass forming ability is achieved by entirely different strategies. While I and Se build in the covalent network making Te-Te bonding stronger, Ga increases the average coordination number of Te (and also network connectivity) but decreases Te-Te bond strength.

[44] I. Kaban *et al*, J. Alloys and Comp. **379** 166 (2004)
[45] P. Jóvári *et al* J. Phys.: Cond. Matt. **22** 404207 (2010)
[46] R. L. McGreevy *et al* , Mol. Simul. **1** 359 (1998)
[47] O. Gereben *et al*, Adv. Mater. **9** 3021 (2007)

Perspectives

A large number of instrumental developments on the LLB spectrometers will provide increased capabilities in the materials science research.

Nanosystems / Heterosystems

The main new instrumental project which will impact this field is the new SANS instrument PA20. In particular the PA20 spectrometer has been designed from the very beginning to be optimized for metallurgy (short wavelengths) and magnetic studies (polarized neutrons). In the field of polarized SANS we are presently hindered by the closure of the PAPYRUS spectrometer so that all the studies in this field are being performed in other facilities (FRMII – ILL). Nevertheless we are developing a research program around the topic of magnetic nano-objects covering the field of photo-sensitive organic magnetic nanoparticles, magnetic nanoparticles for permanent magnet applications (EU program REfreePerMag), arrays of ordered magnetic nano-objects for recording applications in collaboration with Institut Néel Grenoble and INSA Toulouse. An ANR program will support a new post-doc to work in this field. Besides these experiments on various systems, the NMI3 Joint Research Program "Neutron Imaging" is supporting a methodology effort to develop new tools to process and exploit the Polarized SANS data in a comprehensive and extensive way. We are also trying to develop a research program around the field of magneto-transport in anti-ferromagnetic materials. An extensive work has been performed on Cr thin film systems (1 PhD). Another work is presently supported by the funding of a PhD student with a SOLEIL-LLB grant. The contribution of the LLB in to try to probe the structure of anti-ferromagnetic domain walls by means of SANS and reflectometer as well as diffraction experiments. These experiments are complemented by resonant X-ray reflectivity, magneto-optic and transport measurements.

In the field of mesoscale systems, especially (guest@host) systems, the research program will benefit from the new high resolution powder diffractometer G4.4, dedicated to the structural studies of complex systems and to the high flux, large scale-structure diffractometer G6.1.

Physical metallurgy

This field will primarily benefit from the on-going upgrade of the 6T1 spectrometer which will be equipped with a position sensitive detector and a new monochromator. The scientific project is centered on the ANR project METAFORES which will require the use of various neutron characterizations. The methodology developed to analyze peak shapes will be applied to the Cu/Nb systems. This project also involves a coupling between multi-scales numerical simulations and macroscopic properties.
Besides this line of research, the start of the new neutron imaging station IMAGINE should open possibilities for new types of studies in the field of metallurgy. We are still in a prospective phase since this field is not an area in which the LLB has an historical experience.

Disordered systems

The renovation of the 7C2 spectrometer has been completed in 2012 and a gain in counting rate of a factor 25 has been achieved, which has already allowed some new kinds of measurements: crystallization kinetics, very small samples, installation of a laser heating device with sample levitation in collaboration with the Universities of Orleans and Paris 6&7, for very high temperature or supercooling experiments.

Besides, a new protocol has been elaborated at the Lab. Léon Brillouin, to determine the low frequency response of liquids. A recent observation is the cooling regime has been revealed for the first time in liquids (collaboration with IRFU). We plan to confirm its generic character and to point out the role of the inter-rmolecular interactions. For that, bridges between different disciplines (micromechanics, micro-tribology, rheology, micro-rheology and surface science) will be required.

AXIS 2 Research:
Materials and Nanosciences - Fundamental Studies and Applications

Keywords

Nanocomposites, Nanomagnetism, Nanoparticles, Metallurgy, Mechanical properties, Amorphous materials, Microporous materials, Mesoporous materials, Nuclear materials, Irradiation defects, Liquid and amorphous alloys, Glass, Small Angle Scattering, Reflectivity, Texture measurements, Strain measurements, Single crystal diffraction, Powder diffraction

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Post-docs(6)
F. Zighem (2010-2012), I. Panagiotopoulos (2013), C. Pantalei

Scientific collaborations *(in France and abroad are listed below)*

FRANCE	EU	Non – EU
INSTITUT NEEL UNIV. VERSAILLES IRAMIS/ SPEC IRAMIS/ SPCSI DEN / SRMA DEN / SRMP AGRO SUP DIJON ECOLE CENTRALE UMR CNRS / THALES INSTITUT JEAN LAMOUR INSA TOULOUSE CEA LE RIPAULT LPS UNIV PARIS SUD UNIV. CERGY PARIS XI ENSAM PARIS ECOLE DES MINES D'ALBI UNIV. DE TOULOUSE CEA CADARACHE CEA SACLAY DEN UNIV. DE REIMS	JCNS (GERMANY) HZ BERLIN (GERMANY) DEMOKRITOS (ATHENS) UNIV. IOANNINA (GREECE) UNIVERSITY OF SCIENCE AND TECHNOLOGY DE CRACOVIE UNIVERSITÉ DE BUCHAREST ROUMANIE	JINR (DUBNA, RUSSIA) UCONN, UWF, NU (USA) Université de Lima (Perou) UNIVERSITE DE BISKRA – UNIVERSITE D'ALGER ALGERIE SHANGHAI JIAO TONG UNIVERSITY UNIV. OF SCIENCE AND TECHNOLOGY BEIJING

UNIV. DE POITIERS UNIV. DE NANTES		

Scientific contracts

ANR	AMARAGE Aciers MARtensitiques Alliés de nouvelle Genération (2006-2008) MAGAFIL : Elaboration d'aimants permanents haute température à base de nanofils organisés par une approche bottom-up (2008-2010) ELECTRA Pilotage électrique de l'ordre antiferromagnetique dans les nanostructures-ELECtrical Tuning of the antifeRromagnetic orderingof nAnostructures (2011-2014) PROMETFOR Réalisation d'outillage rapide en forge par projection métallique (2007-2010) AXTREM Aciers ferritiques/martensitiques renforcés par nano-particules pour application à haute température en conditions extrêmes (2008-2011) METAFORES : Materials with Elementary Tailored Architecture for Functional Optimized Response from Experiments to Simulations (2012-2016) StruDynaL : Structure et Dynamique de liquides à liaison hydrogène (2001-2014) BIOSELF AUTO-ASSEMBLAGES DE NANOGELS ET NANOCOMPOSITES BIOINSPIRES (2009-2012)
RTRA	RELAXAN, PA 20 building, IMAMINE OCTUCOMETE électro-aimant OCTUpolaire pour l'étude du COuplage MagnETo-Electrique.
C' nano	PA 20 building FilaSpin Filtres de spin à base de couches d'oxydes MAGELLAN Couplage MAGnétoELectrique dans les fiLms et nANostructures multiferroïques (2008-2011)
FP7	NMI3-7th Framework Program of the European Commission : Neutron Imaging MAGMANet (Molecular Approach to Nanomagnets and Multifunctional Materials) (2009) REFREEPERMAG Rare Earth Free Permanent Magnets (2012-2015)
Industrial contracts	CIFRE for PhD in partnership with MICHELIN
CEA/DEN	SANS measurements on steel samples
Bilateral	Partenariat Hubert-Curien UTIQUE France-Tunisie (2008-2010) CNRS-MTA Hungary Cooperation (2008-2009) CNRS-USA Programme (2008-2010)
Egide	Collaboration avec Dubna/ Russie (chercheur visiteur)

Axis 3: Soft Complex Matter

Soft Matter and Biology share a common ground: the delicate balance of interactions of the order of k_BT can lead to the formation of large complex architectures showing specific dynamics, kinetics or lifetime. The ambition of the "Soft Complex Matter" axis of the LLB is to understand both *i)* the behavior of the individual building blocks (molecules, nanoparticles, polymers, surfactants and phospholipids) whose characteristic sizes lay in the 0.1–10 nm range and *ii)* the underlying mechanisms of their self-assembly and dynamics, to, in the end, finely, control and tune the very specific properties of inert, functional or biological matter at the nanometer scale (1-100 nm). Within a neutron facility like the LLB, our specificity is the in-house definition, design and production of the samples perfectly adapted to our scientific targets. The availability of the LLB Chemistry and Biology platforms are key assets to ensure the success of this sample preparation stage. As a general trend, our experimental strategy is built over the advantages offered by the full palette of the neutron scattering techniques combined with H/D isotopic labeling. We probe the structure either in bulk (diffraction, SANS) or at surfaces (reflectometry), and measure relaxations up to the several tens of nanoseconds (time-of-flight or spin-echo).

Polymers

Following the long-term tradition of LLB since the pioneering works of the 70's, polymer physics continues to be a very active field of our Axis. Among the different topics covered, specific effort has been devoted toward the understanding of the origin of the remarkable mechanical properties of polymer-based nanocomposites (highlight 9), and behavior of polyelectrolytes, either in pure solutions or complexed with objects of opposite charges (highlight 10).

Thermosensitive polymers (PNIPAM) were also used in association with gold nanoparticles to control their plasmonic properties. In an original approach, modeling of the plasmon peak positions in terms of the dielectric functions of the polymer shell and of the solvent have been carried out for a series of gold nanoparticles of different core sizes grafted with different polymer chain lengths, dispersed in different solvents; this has allowed us to establish a *quantitative* and *universal* structure-property relationship[48]. For glassy polymer, the molecular weight dependence of the glass transition temperature has been considered[49].

[48] C. Said-Mohamed *et al.* J. Phys. Chem. C **116**, 12669 (2012).

[49] C. Dalle-Ferrier *et al.* Macromol. **43**, 8977 (2010).

In collaboration with several leading French research groups, intense research has also been performed on polymersomes. These systems are formed by the self-assembly of amphiphilic block copolymers. As they are of flexible chemical design, stable, robust, show both physical and chemical stimuli-responsiveness, they appear as excellent candidates for drug delivery or imaging agent carriers. We have explored the properties of several polymersomes under various stimuli (temperature, pH changes or osmotic shock…) and have focused on photo-responsive liquid crystalline polymersomes, intrinsically sensitive to external stimuli, magnetic field application or UV irradiation. The bursting, the change of permeability or other important structural modifications of the system have been followed by SANS[50]. The localization of the magnetic nanoparticles incorporated within the polymersomes to ensure the drug delivery control by hyperthermia was proved by contrast matching[51].

Biopolymers are also under scrutiny. In this field our goal is the design of new materials mimicking Nature (ANR *Bioself*, collaboration CERMAV Grenoble). We have for example shown in the case of a neutral polysaccharide (Xyloglucans) with a backbone decorated with several dangling groups of various hydrophobicity, that structural dependent interactions can be tuned[52] by small changes in composition to pass from good solvent to theta solvent. We have evidenced remaining intrinsic physical associations, a key for associations with other species like cellulose nanocrystals that we are also currently considering.

Foams, emulsions and asphaltenes

Soft Matter research was however not limited to polymers. In collaboration with INRA, we have studied the self-assembly of new green tensioactives based on long chains hydroxylated fatty acids. We have shown that they form "onion"-like lamellar phases of cylindrical shape. The diameter (of the order of micron) can be tuned by temperature both in bulk and at air/water interface. We have, in particular, demonstrated that foams made out of these structures have outstanding properties: they show an exceptional lifetime at ambient temperature (~several months). Moreover, since their stability can be triggered by temperature, they are the first thermo-stimulable foams ever designed[53].

We have also worked on the design of *colloid-ISA-somes*. These systems are aqueous dispersion of submicronic lyotropic liquid crystalline domains

[50] S. Hocine *et al.* Soft Matter **7** (6), 2613 (2013).

[51] C. Sanson *et al.* ACS Nano **5** (2), 1122 (2011).

[52] F. Muller *et al.* Biomacromolecules ***12***, 3330 (2011).

[53] A.-L. Fameau *et al.* Ang. Chem. Int. Ed. **50**, 8264 (2011).

surrounded by solid nanocolloids of different geometries (disks[54], spheres). These fillers form a dense protective *armor* and control the stabilization of the system. Both the internal phase and the armor can be further functionalized by soft chemistry so that these hierarchical architectures are promising for industries requesting safe transport in water or/and controlled delivery of drugs.

Intense research was also carried on asphaltenes, one of the main components of crudes oils, aiming at describing their complex behavior both in bulk and at surfaces (collaboration LLB/IFPEN). In particular, for the first time, the structure of asphaltenes aggregates was accurately described by an elegant study that coupled SAXS and SANS with contrast variation[55].

Confined systems

Under nanometric confinement, the complex interplay of the confinement topology, dimensionality (3D to 1D) and surface/volume ratio significantly affects the physical properties of the confined material. The highlight 11 illustrates, in the field of the new technologies for energy, how to take advantage of confinement to drastically change the behavior of electrolytes. Polymers at planar interfaces have also been considered. The issue was to identify the origin of the difference of the glass transition temperature between bulk and ultra-thin polymer films. This phenomenon may originate from different effects: *i)* the possible presence at the surface of a polymer layer with high mobility, *ii)* the reduction of the diffusion coefficient of the polymer chains due to chain adsorption on the substrate or *iii)* a change in the characteristic distance between entanglements. We have addressed the question of the chain conformation in polystyrene (PS) films of h=15nm thickness. When increasing the confinement ratio h/R_{gbulk}, we do observe, at the local scale, a modification of the conformation compared to the bulk. As this effect is independent of the molecular weight, it is ascribed to an interfacial effect rather than to the confinement of the chain.

Water and protein local dynamics

The physics of water is still a hot topic and has driven an impressive amount of activity over the last decade. Our most recent contribution has focused on water properties under nanometric confinement with a special emphasis on the nature of the interface (hydrophili/hydrophobic[56]) in connection to protein dynamics[57], and the extension of the HBond networks in binary mixtures[58] (International ANR *Strudynal*).

Crowding in Biophysics

In biological systems, the interior of cells is a very congested place. This congestion may either arise from the presence of macromolecules inert to the reaction of interest (macromolecular crowding) or arise from the physical sequestration of quasi-stationary elements such as fiber lattices and membranes (confinement). By respect to the *in vitro* situation, the congestion *in vivo* may significantly affect the behavior of proteins and nucleic acids (NA) (conformation, stability, kinetics…). Our activity covers the influence of macromolecular crowding on: *i)* the proteins conformations, stability, reduction of mobility and the related physiological implications (highlight 12), *ii)* helix to coil transition of polypeptide chains, *iii)* the nature of nanoassemblies made by NA compaction and folding induced by interaction with binding and non-binding ligands, including proteins[59].

Membranes

Interaction of living cell membrane with adsorbed molecules and the way their uptake happens are at the heart of a number of biological issues. Among these molecules, antimicrobial peptides attract special attention as being the keystone of the innate immune system of multicellular organisms. Antimicrobial peptides basically cause lipid-bilayer permeation by producing pores. Their universal presence in animal and plant kingdoms, their non-specific broad-spectrum and their elementary structure let us expect their action also obeys to widespread and universal physical mechanisms. We have addressed this problem using voltage-clamp and neutron reflectivity measurements. We have shown that pore opening displays a slow and glass-like dynamics, which is consistent with fluctuations of peptide concentration at the crowded surface of the bilayer and is compatible with the "Random Surface Addition" (RSA) model[60]. More recently, we have focused on the thermodynamics of pore opening and on its voltage-temperature dependence. Our results guide us to revise the only current theoretical model. We have introduced a common and general contribution of both peptide adsorption and electric field as being responsible for an unbalanced tension of the two bilayer leaflets. We have also shown that for a held membrane the main entropy cost of one pore opening comes from the corresponding excluded area for the translational entropy of lipids[61].

[54] F. Muller *et al.* Soft Matter **40**, 10502 (2012).
[55] J. Eyssautier *et al.* J. Phys. Chem. B **115**, 6827 (2011).
[56] J. Deschamps *et al.* PCCP. **12,** 1440 (2010).
[57] D. Russo *et al.* JACS **133**, 4882 (2011).
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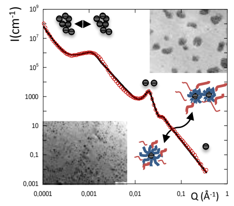
[59] R. Lease *et al.* Front Life Sci. **6**, 19-32 (2012).
[60] G. Fadda *et al.* Phys. Rev. Lett. **103**: 180601 (2009).
[61] G. C. Fadda *et al.* Phys. Rev. Lett. **111**: 028102 (2013).

9. Polymer-particles Nanocomposites: From Simplified to Industrial System to elucidate the Reinforcement

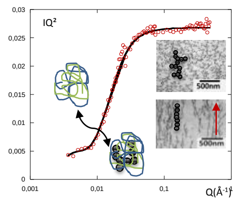
The composite materials present a strong innovative potential for many industrial fields like tire, food packaging or cosmetics. Reducing the size of the particle close to the one of the polymer using nanoparticles opens the way of improving the performance of the materials with reduced costs. This also leads to new fundamental questions related to the correlation between the macroscopic behaviors of the material (the reinforcement) and the local organization of the particles and of the polymer chain matrix. Nanoparticles can indeed adopt multi-scale structures from individual dispersion to complex 3D network with intermediate fractal aggregates of various morphology, density and size while the polymer chain conformation and dynamic can be affected by the particles due to confinement or entropic effects. Updating the classical hydrodynamic model with implementation of quantitative parameters describing these contributions is currently the challenge to overcome a new step in the understanding of the reinforcement mechanisms. We have developed since a few years a series of simplified systems of nanocomposite in which we can control the particle dispersion with different strategies: grafting, orientation with an external magnetic field, control of the processing condition or impact of additive agents. The complexity of the systems is increasing from well-defined polystyrene matrix and colloidal particles to industrial rubber (SBR) and fractal filler (Michelin industry).

We used different SANS determination of the polymer chain conformation in correlation with particle dispersion (SAXS, TEM) and mechanical reinforcement. We first control with grafting[62] the particle (silica or maghémite) dispersion in polystyrene matrix (Figure 16a), individual or aggregates, with the grafted to free chain ratio[63]. Using a specific SANS contrast method, we demonstrate that these dispersions can be related to a collapsed or stretched conformation of the grafted brushes6. On non-grafted silica/model (PS) and industrial (SBR) systems[64], we show using the Zero Average Contrast (ZAC) method (Figure 16b) that the polymer chain is not modified by the filler at rest[65]9, under deformation[66] and orientation with an external magnetic field[67] illustrating that chain deformation does not contributed significantly to reinforcement. We can reproduce the mechanical behavior with a percolation model (Figure 16c) including aggregates parameters (size, fractal dimension and connectivity) deduced from scattering modelling[68]; reinforcement becomes very high with the formation of connected network via inter-particle interactions. To understand the latter at low and large deformation, we currently tackle the important question of the dynamic chain contribution.

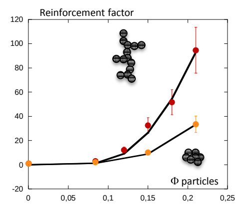
At LLB was also studied the dispersion structure of filler of different shape, nanoclay platelets inside natural latex, under deformation in correlation with reinforcement (with Uni-Campinas, Brasil)[69].



(a) SAXS and TEM on grafted particles systems as a function of the grafted (blue) to free chain (red) ratio that govern the dispersion



(b) SANS on non grafted systems with mixture of H and D chain to extract the single chain form factor. The chain conformation is unperturbed by the filler whatever the filler structure



(c) Reinforcement factor as a function of the aggregate morphology.

Figure 16

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[62] Chevigny *et al.* Soft Matter **5**, 3741 (2009**);** Chevigny *et al.* Polymer Chem. **2**, 567 (2011); Robbes *et al.*, Soft Matter **8**, 3407 (2012).
[63] Chevigny *et al.* Macromol. **44**, 122 (2011)**;** Robbes *et al.* Macromol. **45**, 9220 (2012).
[64] Jouault *et al.* Macromol. **42**, 2031 (2009)**;** Robbes *et al.* Macromol. **43**, 5785 (2010)**.**
[65] Jouault *et al.* Phys. Rev. E **82**, 031801 (2010).
[66] Jouault *et al.* Macromol. **43**, 9881 (2010).
[67] Jestin *et al.* Adv. Mat. **20**, 2533 (2008)**;** Robbes *et al.* Macromol. **44**, 8858 (2011).
[68] Jouault *et al.* Polymer **53**, 761 (2012**);** Chevigny *et al.* J. Polymer Sciences Part B: Polymer Phys. **49**, 781 (2011)**;** Bouty *et al.* Macromol. to be submitted (2013).
[69] L.T.Lee *et al.* Polymer **51**(16), 3644-365 (2010).

10. Polyelectrolytes in bulk, at surfaces and within complexes

Polyelectrolytes (PEL) have a huge potential for applications, either in 3d (bulk water) or 2d (here adsorbed at the air/ water interface), because their conformation and/or degree of association with host molecules or nanoparticles can be finely tuned by the intricate combination of interactions that take place in water: electrostatic, hydrophobic and hydrogen bonding. In turn, this requires more understanding of their structure. Due to the unique power of neutron scattering for the study of polymer-containing systems (stemming from contrast matching techniques), the in-house research in this area is an example of long-term tradition in LLB with 2008-2013 being another highly productive period, both for pure PEL systems and complexes of PEL with proteins or nanoparticles.

Concerning bulk aqueous solutions, we focused on "hydrophobic" PEL that are soluble thanks to charged moieties, despite having hydrophobic parts (backbone in our case). Partially sulfonated polystyrene is considered a reference: a sequence of pearls (radius R_{pearl}) and strings ("pearl necklace") arises from the balance between electrostatic repulsion and hydrophobic contraction as predicted (Dobrynin-Rubinstein). When improving solvent quality by adding to water a good solvent of the backbone (THF), or increasing temperature T, R_{pearl} decreases consistently, until we get a 100% string conformation[70]. The $T-\Theta$ dependence of the "polyelectrolyte peak" abscissa q* (Θ neutral solvent temperature) is as predicted, with a very nice correspondence with macroscopic viscosity. We also explored ionenes[71] (cationic PEs), exhibiting a striking counterion effect in their chain-chain interactions in water, stemming from the varying spatial extent of the counterion atmosphere. Hydrophobicity of their alkyl backbone is masked by electrostatics up to a backbone charge separation of 13Å. This contrasts with the widely used idea of hydrophobicity defining the aqueous behavior of small tetra-alkyl ammonium (TAA) cations (building blocks of ionenes). Our study of TAA halide solutions highlights the penetrable nature of small TAA ions (with short rigid alkyl chains) as an influence on the surrounding water and counterions.

At the air/water interface, were anchored PS-PAA copolymers, by the hydrophobic PS blocks. We demonstrated that electrostatics tunes the structure of the PAA branches: the morphology of self-assembled surface aggregates is driven by a competition between the release of spreading solvent and electrostatic repulsions between the PAA charged branches[72]. Such aggregates were further used as 'nanomolds' for designing controlled silver nano-objects by reducing in situ silver ions condensed onto the PAA branches.

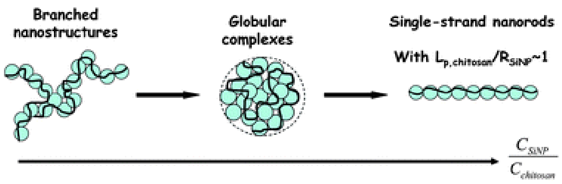

Figure 17: Electrostatic complex fractal dimension vs silica [NP(-)]/[chitosan PEL(+)] concentration ratio

Complexes between PEL and species of opposite charges, are also governed by electrostatics. For globular proteins (monodisperse in size but heterogeneously charged); former studies with PSS (flexible, small persistence length Lp) were extended to various pathways of complexation and to more rigid (higher Lp) pectin (heterogeneous charge), and hyaluronan (homogeneous charge). Interaction potential profiles seem at the core of different evolutions, e.g. aggregation versus coacervation[73]. For nanoparticles (NPs) (homogeneously charged), extensive studies performed either on silica NPs or in-lab synthesized gold NPs were carried out with same PELs (PSS, HA). This gave a wide range of Lp/R, enabling to conclude that it controls the fractal dimension of the complexes from rod (1) to globule (3) (see Figure 17) via Diffusion Controlled Aggregation (1.8…2.5). Finally, astonishing mixed "metacrystals" of hyaluronan/gold NPs were also obtained[74].

[70] Collaboration LLB/INRAP Tunisia. W. Essafi *et al.* Macromol. **42**, 9568-80 (2009); J. Phys. Chem. B **115**, 8951-60 (2011); J. Phys. Chem. B **116**, 13525-13537 (2012).

[71] Collaboration LLB/PECSA Paris 6. N. Malikova *et al.* PCCP **14**, 12898-12904 (2012); D. Bhowmik *et al.* Eur. Phys. J. Special Topics **213**, 303-313 (2012); PhD of D. Bhowmik (2011).

[72] Collaboration LLB/INSP Paris 6. Z. Gennouni *et al.* Soft Matter submitted (2013), PhD Of Z. Guennouni (2013).

[73] F. Cousin *et al.* Langmuir **26**, 7078–7085 (2010); F. Cousin *et al.* Adv. Col. Int. Sci. **167**, 71-84 (2011); I. Morfin *et al.* Biomacromol. **12**, 859–870 (2011); Collaboration LLB/INRA I. Schmidt *et al*, Biomacromol. **10**, 1346-1357 (2009).

[74] Collaboration LLB/MSC Paris 7 : PhD of L. Shi (2013). L. Shi *et al.* ACS Macro Lett. **1**, 857–861 (2012); Soft Matter **9**, 5004-5015 (2013).

11. Ionic liquids under confinement: high mobility in tight spaces?

Fueled by concerns about climate changes, intensive activity is currently spent on the quest of advanced materials with high ionic conductivity to be used in electrochemical devices. Recently, we have suggested that confinement of polymer electrolytes in nano-channeled materials such as Anodized Aluminum Oxide (AAO) would increase significantly ionic conductivity through the pore[75,76,77]. We are now extending this concept to the case of a promising class of electrolyte: Ionic Liquids (IL).

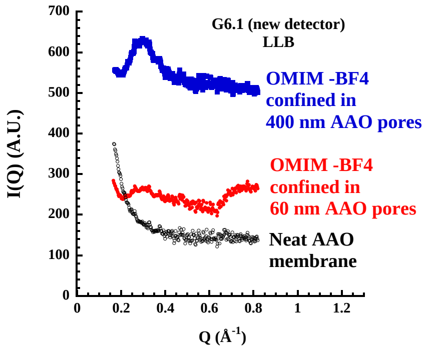

Figure 18: S(Q) of ^{h}OMIM-BF_4 (fully hydrogenated form) at RT. As in bulk (not shown), under confinement in large (400 nm diam.) AAO pores the prepeak induced by the nano-structuration of the IL is clearly detected. This nano-structuration is frustrated under confinement in 60 nm pores (the prepeak has vanished).

● **IL: a surprising propensity to self-organize.** IL show a surprising propensity to self-organize in transient nanoscopic domains. While MD simulations suggest a nano-structuration with a characteristic size around 10 nm, because of the extremely weak contrast between moieties of the pure bulk liquid, the transient local organization and the underlying density fluctuations, do not induce any detectable SAXS or SANS signal. The IL nano-structuration has so far only been detected by the presence of a prepeak (Figure 18) measured by diffraction in the 0.1-0.5 Å^{-1} region. We identify this self-organization in transient nanoscopic domains as possibly responsible for the IL low ionic conductivity at long time (ms) and large scale (µm)[78].

● **IL confinement in a single pore**. We have reported[79] voltage-clamp measurements through single conical nanopore obtained by chemical etching of a single ion-track in polyimide film. Special attention has been paid on the *pink noise* of the ionic current (i.e. 1/f noise) measured with different filling liquids. The relative pink noise amplitude is almost independent of concentration and pH for KCl solutions, but varies strongly using ionic liquids. In particular depending on the ionic liquid, the transport of charge carriers is strongly facilitated (low noise and higher conductivity than in the bulk) or jammed. These results show that the origin of the pink noise can be ascribed neither to fluctuations of the pore geometry nor to the pore wall charges but rather to a cooperative effect on ions motion in confined geometry.

● **Improving the conductivity by frustrating the formation of the IL nanometric domains.** As a follow-up of this important finding, we propose to lift the IL poor conductivity by frustrating the formation of the IL nanometric domains using confinement of the IL within the controlled porous structure of AAO membranes. On decreasing the pore diameter, we detect a shallow transition where the prepeak vanishes (Figure 18) and therefore expect *i)* to be able to estimate the characteristic size of the nanometric domains so as *ii)* to undertake a full characterization of the confined "de-segregated" IL transport properties: diffusion coefficient by QENS[80], PFG-NMR[81] [7] and ionic conductivity measurements.

Next to the potential applied research interest, our primary goal is to develop a general multiscale approach to bridge the broad time and spatial scales relevant to the physics of fluids under nanometric confinement. We ambition to develop the alliance of these *classical* "ensemble average" techniques (a multitude of pores are simultaneously probed at once) with a single pore approach: on a multitude of non-connected pores, mobility fluctuations are not in phase and only the averaged mobility is measured. Only the single pore technique can reveal anomalies of the electrolyte mobility fluctuations (e.g. concerted zones of mobility) and thus give a key insight into the underlying physical mechanism of the ionic conductivity.

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[75] K. Lagrené *et al.* Phys. Rev. E. **81**, 060801 (2010).
[76] J.-M. Zanotti, K. Lagrené, patent FR2963481 & WO2012013603 (2012).
[77] J. M. Zanotti *et al.* Eur. Phys. J. Special Topics **213**, 129-148 (2012).
[78] C. Tasserit *et al.* Phys. Rev. Lett. **105**: 260602 (2010).
[79] Phung Le *et al.* J. Phys. Chem. B **114**, 894-903 (2010).
[80] K. S. Panesar *et al.* J. Phys. Soc. Jpn. (in press)
[81] K. S. Panesar *et al.* Microporous Mesoporous Matter, **178**, 79-83 (2013).

12. Proteins in crowded environments: diffusion and conformation

In vivo, the cytoplasm and many extracellular compartments are filled with very high quantities of macromolecules that occupy a total volume fraction in the range of 30-40%. As a consequence, interactions between components are significantly enhanced due to their close proximities, which are of the order of 1 nm. The term "macromolecular crowding" (MC) is generally used to describe these environments, when only the effect of excluded volume is considered (but not the specific interactions). It is now well established that MC affects the thermodynamic equilibriums and dynamic properties of proteins with respect to those in dilute solutions. Neutron scattering techniques are invaluable methods for studying these effects, first because they probe the characteristic length- and time-scales for proteins within the typical ranges of size and intermolecular distance. Secondly, thanks to the scattering length density difference between hydrogen isotopes and contrast variation methods, it is possible to observe the signal of macromolecules at low concentration in the presence of very high concentrations of other components.

Diffusion

Hemoglobin (Hb) and myoglobin (Mb) are oxygen storage molecules present in blood and muscles, respectively. Their diffusion is since a long time suspected to facilitate oxygen transport. We have investigated by neutron spin echo their diffusion at intermolecular scale, both *in vitro* and *in vivo*[82]. These experiments have shown that theories developed for colloid diffusion can be applied to understand long and short protein diffusion times if one includes the water shell in the hydrodynamic volume of the molecules, usually assumed to be ~0.35 g water/g protein. Using the concentration dependence of transport diffusion coefficient in Hb, we are able to show that Hb concentration inside the red blood cells corresponds to an optimum in oxygen transport for individuals sustaining strong physical activity.

Conformation

Excluded volume due to the presence of cosolutes diminishes the accessible volume of a protein. The more extended conformations are more affected than the more compact ones. The unfolded state ensemble is thus destabilized with respect to the compact native state (Figure 19). The physical observation is the compression of the unfolded state ensemble.

We first observe by small angle neutron scattering (SANS) the compaction of a gaussian chain due to the presence of inert cosolutes[83]. We show that when the free volume is reduced by high concentration of macromolecules (300 g/L ~physiological concentration), a gaussian polymer chain is compressed and its radius of gyration is reduced by more than 30%. To compare our results with the predictions of excluded volume theory, we studied the scaling effects, *ie.* by varying the size ratio between the random coil and the crowder.

We then studied the temperature dependence of Mb protein unfolding in the presence of macromolecular crowder[84]. We observe the compression of the protein unfolded state at high MC fraction but the relation towards protein stabilization still requires some experiments that are under progress. The unfolded state compression occurs indeed in association with a second effect, also predicted by MC theory: the aggregation of the unfolded chains. This phenomenon is also observed without crowder but is strongly enhanced by excluded volume.

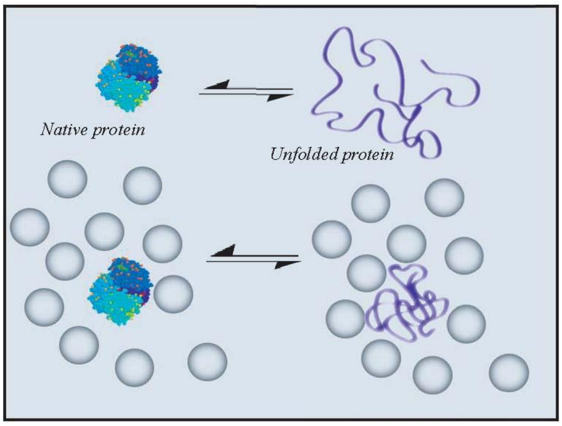

Figure 19: Native/unfolded state equilibrium without and with macromolecular crowding. This effect shifts the native ↔ unfolded state equilibrium towards the native state.

We also report SANS and circular dichroism measurements on the helix-coil transition of poly(L-glutamic acid) polypeptide in solution with polyethylene glycol (PEG) as crowder[85]. Unexpectedly, we notice that the increase of PEG concentration reduces the mean helix extent at the transition. Our results strongly suggest two regimes: helices shorter or longer than the mesh size.

[82] W. Doster and S. Longeville Biophys. J. **93**, 1360 (2007), C. Le Cœur *et al*. Chem. Phys. **345**, 298 (2008).

[83] C. Le Cœur *et al.* Phys Rev E **79**, 031910 (2009); C. Le Cœur *et al*. Phys Rev E **81**,061914 (2010).

[84] S. Combet *et al.* in preparation.

[85] A. Koutsioubas *et al.* J Chem Phys, **136**, 215101 (2012).

Perspectives

In all the fields developed by the Soft Complex Matter axis, the work accomplished so far, will serve as a settlement for the years to come.

Nano-composites & Smart materials:

The polymer know-how of the axis will make it possible to orient this activity towards smart responsive polymer based nano-architectures: copolymers, bear and grafted nano-capsules/nano-particles.

We have now expertise in designing polymer-based nanocomposites models systems with various and controlled dispersions. This means a perfect knowledge of both the conformation of the polymer chains and the organization of the fillers. To go further in understanding the origin of the outstanding mechanical properties of these systems, we will now address by QENS and NSE the underlying dynamical behavior of the polymer chains.

We have now established a pertinent framework to finely bridge the optical response of polymer gold nanoparticles with the conformation of the chemically grafted polymer chain. This universal structure-property relationship provides a promising route to finely tune their optical signature. Future studies will now concentrate on interfacial layers and thin films. Our goal is to improve the monolayers stability so as to be able to generate ordered multilayers. This work will also extend to gold nanoparticles functionalized with macrocyclic host molecules. Here, the idea is to induce the spontaneous formation of networks through specific host-guest interactions. The reversible expansion or collapse of the network could be for example controlled by thermosensitive guest polymers.

Biopolymers & Food Science:

We have recently reached an accurate description of the aqueous behavior of Xyloglucans, an hemicellulose extracted from the cell walls. This progress opens the way to a controlled association of Xyloglucans with cellulose nanocrystals. This pumps-up a new ambition in the field of biopolymers: to design new green hydrogels showing controlled mechanical properties. In collaboration with ECE Paris we will continue the design and characterization of colloidosomes-based emulsion.

We will capitalize on our recent successful use of SANS for the fine characterization of foams and emulsions to develop our blooming food science activity. Food products are multi-components systems showing numerous and competing structural, dynamical and thermodynamical interactions. By applying the concepts developed in the field of soft matter physics (self-assembly, gelation, glass transition…) to these complex matrices, we will tackle, on very specific examples, a nanoscale description of the different steps of food structuring. This field will directly benefit from the in-house availability of a new imaging station: this specific neutron technique will be a key asset for the micronic description of food matrices. This activity is currently developed in a close collaboration with Agrosup Dijon.

Membranes:

Following our recent success in this field, we will extend our study on the physics of phospholipid membranes. A target is to reach a fine understanding of peptide insertion within biological membranes.

We will extend our studies of 1D confinement of electrolytes towards narrower pores with no interaction of the confined material with the pore internal surface. This will come through the design and synthesis of carbon nanotube (CNT) membranes. The common use of the voltage clamp technique will be a point of convergence in this field of membrane systems.

Biophysics:

We ambition to probe crowding effects in situation of direct biological relevance: biological macromolecules (proteins and nucleic acids) surrounded by partially deuterated cellular material or artificial membrane environments created by surface modified silica nanoparticles or nanoporous materials. We intend to use mesoporous structures to mimic the constraint due to the cellular environment. These structures are particularly adapted as they present an hydrophilic environment within an hydrophobic one, they are biostable, biocompatible and surface modification or deuteration are possible. Our analysis will focus on the effect of confinement on self-assembling proteins (amyloid fibers, actin like...) and proteins showing specific interactions with nucleic acids.

Last but not least, we expect that all these topics will directly benefit from the strong forthcoming rejuvenation of our experimental park, in particular, in a close future from *i) Imagine,* the neutron imaging station, *ii) PA20*, the new SANS spectrometer, *iii)* the new versions of the *EROS* reflectometer. Meanwhile, we develop the *Multi-MUSES* spin-echo and Xtal ToF machine *Fa#*, in the optic of undertaking time-resolved inelastic experiments.

AXIS 3 Research:
Soft complex Matter

Keywords

Structure, dynamics, complex fluids, polymers, surfactants, colloids, self-assembly, nanoparticles, grafting, nanocomposites, nanopores, multicomponents systems, gels, networks, room temperature ionic liquid, saline solutions, ionic conductivity, protonic conductivity, HBond, nucleic acids folding and structure, protein folding-unfolding, protein crowding, protein dynamics, proteins/polyelectrolyte, biocompatible polymers, biopolymers, membranes, hydrophobicity, water properties.

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Post-docs(6)
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Scientific collaborations *(in France and abroad are listed below)*

FRANCE	EU	Non – EU
AGROSUP DIJON CEA CADARACHE CEA/DRT/LITEN CERMAV GRENOBLE ENSTA PALAISEAU ESPCI PARIS ICS STRASBOURG ILL GRENOBLE INSTITUT CURIE, PARIS CEA/INAC, GRENOBLE INRA NANTES INST FRANCAIS PETR LIONS CEA SACLAY UNIV AIX MARSEILLE UNIV BORDEAUX UNIV CLERMONT-FERRAND UNIV LYON UNIV MONTPELLIER UNIV ORLEANS UNIV PARIS 6 UNIV PARIS 7	CNR, ITALY JCNS, GERMANY ROSKILDE UNIV DK, H. HEINE UNIV, GERMANY UNIV BRUSSELS - BELGIUM UNIV PATRAS - GREECE UNIV HELSINKI - FINLAND UNIV OF GDANSK, POLAND UNIV OF MADRID (UAM), SPAIN UNIV BRATISLAVA, SLOVAKIA	ARIZONA STATE UNIV, USA ANSTO – AUTRALIA BHABHA INSTITUTE, INDIA CUNY, NEW YORK, USA IMA, UFRJ, RIO, BRAZIL UNIV FUKUOKA , JAPAN UNIV,SHINSHU JAPAN UNIV MONASTIR, TUNISIA UNIV OF TENNESSEE, USA UNIV OF WISCONSIN, USA UNIVNIV OF TEXAS, USA UNIV OF CONNECTICUT, USA UNIV OF SINGAPORE UNIV OF SEOUL, S. KOREA UNIV OF LA PLATA,

UNIV PARIS 11 UNIV PARIS 12 UNIV PARIS 13 UNIV PAU UNIV RENNES UNIV TOULOUSE SOLEIL, ST-AUBIN		ARGENTINA

Scientific contracts

ANR	BIOSELF, LISSIL, MULTICLICK, BIOSTAB, TEMPLDISCO, TRANSFOLDPRO, DYPOLYPO, ANR DYNHET, ANR STRUDYNAL
	BIOSELF :Auto-assemblages de nanogels et nanocomposites bioinspirés LISSIL Développement d'une nouvelle famille d' electrolytes solides nanohybrides. Dynamique moléculaire en milieu liquide ionique nanoconfiné MULTICLICK : Application of click chemistry for the elaboration of hybrid multilayer architectures BIOSTAB : Optimisation de la stabilité de matériaux biologiques pour de nouvelles stratégies thérapeutiques TEMPLDISCO : Template confinement effects on discotic liquid-crystals TRANSFOLDPROT : Dynamics and mechanics of protein t ransport , interact ion and folding through different protein channels at the single molecule level DYPOLYPO, Modelling the dynamic properties of polyelectrolytes in charged porous media
RTRA	RELAXAN, PA 20 building
C' nano	PA 20 building
JRA	NMI3-7th Framework Program of the European Commission : Sample environment Coordinator : A Brûlet
Industrial contracts	- CIFRE for PhD in partnership with MICHELIN - Contract with IFPEN
OSEO-programme GENESIS	Nanomateriaux" , coordonne par ARKEMA.
Programme bilateral Ecos Sud	A09B02 2010-2013: " Etude interdisciplinaire et multi-échelle de l' interaction entre la couche-S des bactéries lact iques et les protéines de la membrane externe des bactéries Gram(-)"
CEA/DSM	DSM-Energie : Projet CONFLUENT
CEA/DRT	Programme transverse DRT/DSM : PILPOIL
RTRA	**2013-0116T – WATSURF**
Egide	PHC Polonium (27701VG)

PART III

INSTRUMENTAL AND TECHNICAL GROUPS

Spectroscopy group

The *Spectroscopy* group is a merger of the former *Dynamics–triple-axis* group and the quasielastic scattering activity previously within the *Biology and disordered systems* group. It uses the power of inelastic neutron scattering to study the dynamics of atoms or magnetic moments in a wide range of systems. Its activity touches on topics of high current interest, such as magnetism and superconductivity in strongly correlated electron systems, molecular liquids under confinement, or proteins in crowded environments. A huge dynamic range (6 orders of magnitude) can be covered using an instrumental suite composed of 4 thermal- and cold-neutron triple-axis spectrometers (TAS), 1 time-of-flight (TOF) spectrometer,[86] and 1 neutron-resonance spin-echo spectrometer. The group is also in charge of the test diffractometer 3T1 and of the decommissioned TAS G4-3, now devoted to student training courses (see *Training and Education* section), which also features a small-angle-scattering mode based on the large area position sensitive neutron detector (PSD) "*Barotron*" (LLB patent).

Triple-axis spectrometers

The four TAS located inside the reactor hall are world-class instruments, whose performance has been continuously improved over the years by implementing state-of-the-art neutron optics (focusing monochromators and analyzers on all of them, supermirror guides on the incident beam of 4F1/2 and, more recently, 1T) and/or optional neutron polarization analysis (2T, 4F1). However, the aging of various elements, some of them over 20 years old, put the experiments increasingly at risk for technical failures. A major upgrade was thus undertaken, including the monochromators (underway) and parts the primary protection (completed) on the German CRG 1T,[87] the electropneumatic system (completed) on 2T, or the entire electronic rack cabinets (performed during the 2013 summer shutdown) on 4F1 and 4F2. The latter twin spectrometers, sharing the same neutron channel thanks to a double–PG-monochromator system, have been optimized for neutron polarization (supermirror bender + Heusler analyzer) and high magnetic fields (replacement of existing modules by nonmagnetic parts), respectively. The next important step will be the development of a homemade, *muPAD*-type sample chamber for spherical polarization analysis on 4F1.

With improved reliability, enhanced performance and versatility, and a comprehensive fleet of ancillary equipments, the LLB TAS can address a broad spectrum of problems in magnetism and lattice dynamics, and cope with challenging experimental conditions (small samples, extreme temperature, pressure, or magnetic field).

Sample environments

Over the last 5 years, special emphasis was placed on improving the performance, dependability, and user friendliness of sample environments. Besides conventional CCRs and He-flow "orange" cryostats, the pool of equipments, available interchangeably on all TAS,[88] includes top- and bottom-loading, two-stage, 3-kelvin CCRs, one "dry" (helium-free) dilution refrigerator and one insert for measurements in high fields, as well as a 1400°C furnace, a 10 T cryomagnet, and a 8-kbar hydrostatic He gas pressure system. The upgrade of the latter system was part of the LLB contribution to the "Sample Environments" JRA of the NMI3 Integrated Infrastructure Initiative (EU Framework Programme FP7)

Numerical simulation

Calculations of non-linear spin dynamics, in connection with inelastic neutron scattering studies, now benefit from ongoing code development and new computing facilities on a 48-node fast processing computer (see *Theory and Modeling* platform). Members of the Spectroscopy group took an active part in the development of this new platform.

[86] The Mibémol TOF spectrometer was decommissioned and dismantled during the reporting period. A new instrument is currently under design (see "Fa#" below).

[87] Operated by the KIT Karlsruhe.

[88] Except the 10 T magnet, compatible only with 4F2 and (in a reduced field range) 2T.

MUSES

MUSES is a mixed conventional / resonance neutron spin echo (NSE) spectrometer installed on the guide G1bis. The aim of this instrument is to study medium- to high-resolution quasi-elastic neutron scattering in the intermediate wavevector range (0.05 $Å^{-1} < Q < 4$ $Å^{-1}$), taking over from TOF spectroscopy[1] at longer time scales.

The spectrometer consists of two distinct components, a conventional NSE spectrometer for measurements at short times (typically $t <$ 100 ps for $\lambda \sim 5$ Å), and a neutron-resonance spin-echo (NRSE) option for measurements at longer times (to 40 ns).

In resonance spin-echo spectrometry, the two high-magnetic-field precession coils are replaced by four radio-frequency (RF) coils: two in the first "arm" and two in the second "arm" of the instrument. The spectrometer is compact, with a distance of 1.8 m between the resonance coils in each arm. The polarized neutron flux at the sample position is ~10^7 n.cm^{-2}s^{-1}, with high homogeneity over the beam area of 4×4 cm^2. Very short Fourier times (0.2 ps) can be measured because beam depolarization due to the earth's magnetic field, or any environmental field, is suppressed by means of mu-metal shielding.

Figure 20 The new RF coils used for neutron-resonance spin-echo spectrometry.

New resonance coils have been under construction and testing in the last few years. They have been installed on the spectrometer in the spring of 2011. The new design improves RF field stability and permits higher currents (100 A) to be applied. This should allow us to reach Fourier times of the order of 40 ns. Tuning of the coils was performed from late 2011 to the beginning of 2013, and the spectrometer is now fully operational.

The installation of these new RF coils was a prerequisite for the multi-Muses project—a multi-detector upgrade of the current spectrometer (see below).

The Fa# project

After almost 30 years of good and faithful service, the performance of Mibémol, the LLB-Orphée TOF spectrometer, was challenged by a new generation of instruments, already in operation or scheduled to come on-line in the next few years. In order to maintain a first-rate scientific program in this field, LLB has undertaken the design of a new TOF machine: the so-called Fa# project. In the long run, Fa# will remain the only TOF spectrometer at LLB. This machine must therefore offer competitive performance in both quasi-elastic and inelastic measurements, for disordered systems (liquids, glasses, biology, chemistry, soft matter) as well as physics).

The design of Fa#, based on the FOCUS[89] machine machine in operation at the Paul Scherrer Institute in Switzerland, has been fully directed toward maximizing the neutron counting rate. This has been achieved by combining the most advanced neutron optics and detection equipments:

- a large section elliptic neutron guide with m = 2 to m = 5 supermirrors, delivering a significant thermal flux (10^{10} neutron/s at λ = 2 Å),
- a set of doubly-focusing monochromators (with both vertical and horizontal curvature),
- a short flight path from sample to detector to maximize the detection solid angle (1.7 sr),
- an extended array of 240 high-pressure (5 bars) ^{3}He PSDs.

[89] J. Mesot, S. Janssen, L. Holitzner and R. Hempelmann, J. Neutron Res., **3** (1993) 293. http://spectroscopy.web.psi.ch/focus/

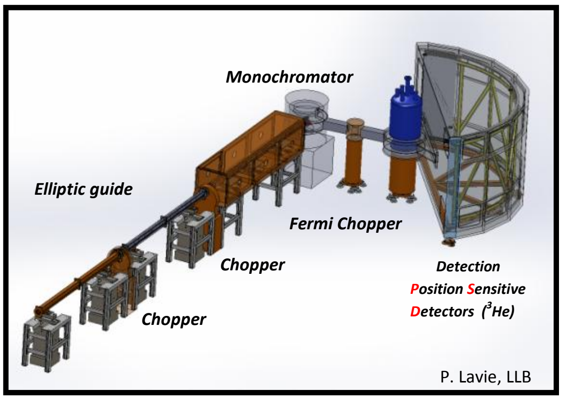

Figure 21: The Fa# spectrometer. Key elements of the spectrometer are shown. In order to accommodate high magnetic fields up to 15T.

The ample area (150 m^2) available in the LLB neutron guide hall will allow for an extended range of geometrical configurations (takeoff angles), making it possible to reliably map energy resolution in the 15 to 500 μeV range over an extended Q domain from 0.05 to 5 $Å^{-1}$. With variable guide-to-monochromator and monochromator-to-sample distances, Fa# will stand out as a hybrid instrument capable of switching from time focusing to monochromatic focusing.[90] Having both modes available on one and the same machine will be a key feature to optimize the flux *vs* resolution balance in the different scientific fields targeted by the instrument.

Taken together, the technical choices outlined above will entail a gain in counting rate by more than two orders of magnitude with respect to Mibémol (10^5 neutron.cm^{-2}.s^{-1} at sample position for 100 μeV resolution) so as to challenge the best TOF spectrometers currently on line, and bring Fa# to the forefront of international competition.

[90] In connection with the high thermal flux delivered by the guide and the excellent resolution it provides for measuring on the neutron energy loss side, the latter mode will be particularly relevant to magnetic single-crystal studies down to the millikelvin range.

The TOF-TOF project at ESS

In collaboration with the Technische Universität München, we are designing a new TOF-TOF spectrometer to be proposed for construction at the European Spallation Source (ESS) in Lund. This spectrometer should be a versatile cold-neutron the IN5-type chopper spectrometer. It should address range of medium-energy, slow-motion, excitations in condensed matter, chemistry, material science, biology and related topics (*i.e.* pico- to nanosecond characteristic times on length scales ranging from angstroms to nanometers). Due to the specificity of the ESS source (long pulse, 2.86 ms, and low frequency, 14 Hz) the source to sample distance should be of the order of 120 m to achieve $\Delta E/E \sim 1\%$, corresponding to a wavelength band of $\Delta\lambda \sim 2.5$ Å. This will allow measurements to be performed simultaneously at different wavelengths, thus covering a wide range of energy resolutions within one data acquisition, while increasing the counting rate with respect to reactor spectrometers.

Members: *P. Baroni (CNRS), P. Bourges (CEA), P. Boutrouille (CEA), B. Homatter (CEA), D. Lamago (KIT Karlsruhe), J.-P. Castellan (KIT Karlsruhe), F. Legendre (CNRS), S. Longeville (CEA – deputy head), F. Maignen (CNRS), N. Malikova (CNRS), C. Meunier (CEA), J.-M. Mignot (CNRS – head), H. Moudden (CNRS), S. Petit (CEA), D. Petitgrand (CNRS), J. Robert (CNRS), Y. Sidis (CNRS), J.-M. Zanotti (CEA).*

1. Efficiency of a thermal neutron focusing guide on a triple-axis spectrometer

The triple-axis spectrometer is one of the most powerful instruments for investigating structural and dynamical phenomena in condensed matter physics. Unfortunately, the use of rather large samples (of the order of a cubic centimeter) is normally required because of the relatively low neutron flux available at current neutron sources. Therefore, neutron guides focusing the beam onto a small spot appear attractive for cases where only small samples are available. It is only very recently that the development of supermirrors allows one to fabricate such guides, which are also suitable for thermal triple-axis spectrometers. We have tested the efficiency of a focusing guide on the 1T instrument.

The guide was manufactured by Swiss Neutronics and has a coating m = 6, to improve the reflectivity of the guide for higher neutron energies. The total length of the guide is only 500 mm. This is certainly too short for obtaining large gains but was chosen for reasons of costs and space: the test guide can be inserted very easily on the incident beam. The dimensions of the incoming and outgoing openings are 25×25 mm and 11×11 mm, respectively. The distance to the focal point is 150 mm, which allows one to use standard closed cycle refrigerators (CCR). A position sensitive neutron scintillation camera *Babyrotron* (Arinax, brevet LLB) placed at the sample position was used to align the neutron guide accurately and to analyze the shape and intensity of the beam.

The wavelength distribution of the integrated intensity in the focal spot (5×5 mm) with and without guide is shown in Figure 22. The comparison reveals a maximum intensity gain of 1.6, which corresponds approximately to the result of the simulations (not shown) performed by Swiss Neutronics. We note that the gain remains approximately constant down to neutron wavelengths of 1.5 Å but decreases significantly for smaller wavelengths, as expected from the simulations.

We used this setup to measure phonons in a small CoO single-crystal of approximately 50 mm^3. A comparison of the phonon intensities observed at $\boldsymbol{Q}$ = (-0.4, 2, 2) with and without the focusing guide revealed a moderate gain in intensity by about 1.2 (Figure 23). Further tests are necessary to find out why the gain was smaller than expected from the flux measurements. Although the data were taken with only very low statistics due the limited beam time, it was observed that using the guide results in a back-ground reduction by approximately a factor of 2, while the energy resolution is slightly improved.

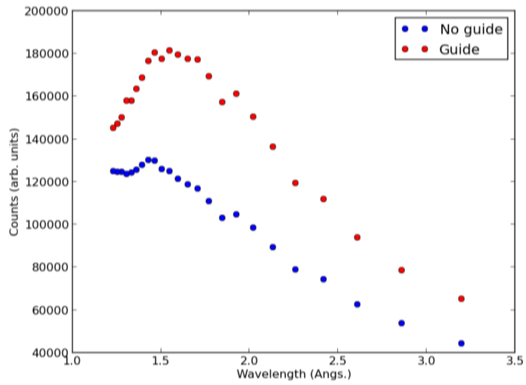

Figure 22: Comparison of the integrated intensity at the focal spot as a function of incident neutron wavelength.

In conclusion, we found that, with the advent of multi-layer coatings, focusing guides can be built with rather high efficiencies for neutron energies up to about 35 meV, i.e. the energy range of many experiments performed on triple-axis spectrometers. In order to fully exploit the potential of such focusing systems, guides of much larger dimensions have to be used. In principle, there is sufficient space available on 1T for such a guide, i.e. up to 1.5 m in length. Purchasing such a guide is now under consideration.

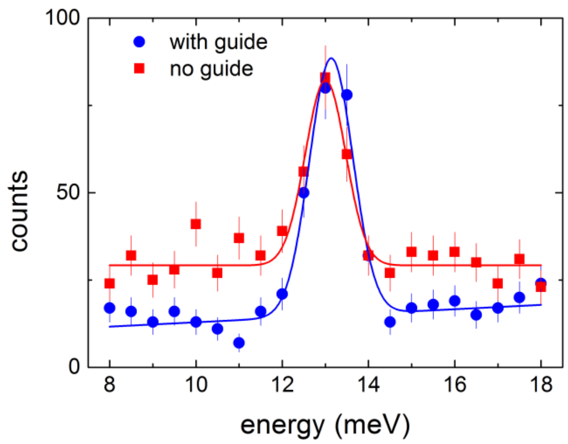

Figure 23: Measurements of a transverse phonon mode in CuO at $\boldsymbol{Q}$ = (-0.4, 2, 2). Full circles (full squares) are data obtained with (without) the focusing neutron guide. Solid lines are Gaussian fits to the data.

2. Improving polarized neutron setup on triple-axis spectrometers

Whereas the power of polarization analysis is highlighted in most neutron scattering textbooks, the number of experiments taking full advantage of this unique technique remains limited. Limitations are actually more stringent on the supply side, *i.e.* the performance of instruments available at existing facilities, than on the demand side. Nowadays, polarizing and analyzing a neutron beam can be achieved using three types of devices: monochromators and analyzers made of polarizing Heusler single crystals, polarizing super-mirrors and polarized ^{3}He neutron spin filters. Polarization analysis further requires controlling the direction of neutron polarization by means of applied magnetic fields. This is achieved using either *XYZ* Helmholtz coils for basic "longitudinal" polarization analysis, or more sophisticated devices for full spherical polarization analysis. In the last decade, effective polarized neutron optics and polarization analysis devices have become commercially available. At LLB, a program was recently initiated to upgrade and improve polarized neutron equipments on triple-axis spectrometers (TAS).

Of the four world-class triple-axis spectrometers now available to LLB uses, two can be operated in the polarized-neutron mode. The thermal-neutron TAS 2T uses a focusing monochromator and a focusing analyzer made of a mosaic of selected Heusler alloy (111) single crystals.[91] On the cold-neutron TAS 4F1, the incident beam polarization is performed by a curved super-mirror guide (bender), and the polarization analysis again by a focusing Heusler analyzer.

The Heusler blades of the 2T monochromator (11 × 14 cm^2) and analyzer (10 × 12 cm^2) will be replaced by new single crystals with improved reflectivity and mosaic spread, as well as a more accurate cut. This should result in a gain in flux by a factor of ~4. Fast neutrons, causing high background level in polarized thermal-beam neutron measurements with long counting times will be suppressed by inserting a sapphire filter on the primary neutron beam.

On the cold-neutron TAS 4F1 (Figure 24), the existing Heusler analyzer has been replaced by the one previously used on 2T, which has a much higher efficiency (larger-surface, high-quality Heusler single crystals). To this end, the analyzer module on 4F1 has been entirely re-designed to accommodate a Heusler analyzer with larger size (Figure 24). The incoming neutron beam will be equipped with a new, more compact, polarizing super-mirror, which will be effective over an extended range of neutron wavelengths.

Both spectrometers will further benefit from new high-precision motors and encoders to improve the accuracy in the positioning of the sample and analyzer rotation tables, which is prerequisite for high-quality polarized neutron measurements.

The outcome of a polarized neutron experiment relies on the ability to collect separate spin-flip and non-spin flip cross sections, and on a perfect control of neutron spin direction before and after the scattering process within the sample. Existing polarized neutron setups on 2T and 4F1 include standard *XYZ* Helmholtz coils for field generation, and Mezei flippers. A cryo-flipper, consisting of two magnetic coils and a zero-field superconducting Nb spacer, will also be reassembled. This type of flipper is less compact that Mezei flippers, but has the major advantage of being effective at arbitrary neutron wavelength.

The study of complex magnetic structures, magnetic chirality, and magneto-elastic phenomena require going beyond standard *XYZ* polarization analysis and carrying out so-called "spherical" neutron polarimetry measurements. This is possible using either the cryogenic polarization analysis device (CRYOPAD), developed at the ILL and now implemented on the D3/ILL diffractometer, the IN22/CEA and TAS-1/JAERI TAS,[92,93] or the mu-metal polarization analysis device (MuPAD)[94]. Adding MuPAD to our triple-axis spectrometers will be a significant improvement.

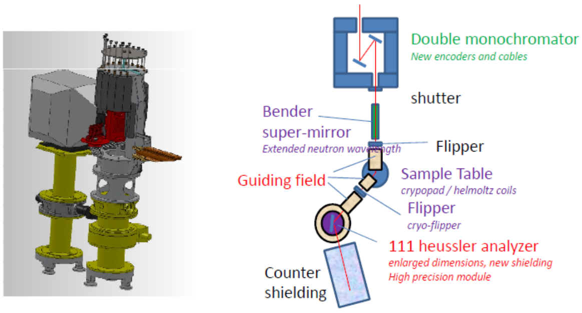

Figure 24: Schematic view of cold neutron TAS 4F1 with the new analyzer protection redesigned to accommodate a larger Heusler analyzer

[91] P. Courtois, Physica B **267-268**, 365 (2003).

[92] L.P. Regnault *et al.*, Physica B **335**, 255 (2003).

[93] E. Lelièvre-Berna *et al.*, Physica B **356**,131 (2005).

[94] M. Janoscheck *et al.*, Physica B **397,** 125 (2007).

3. MULTI-MUSES: Toward a two order of magnitude increase in detection rate on MUSES

The aim of the MULTI-MUSES project[95] is the implementation of a multi detector system on the Neutron Resonance Spin-Echo[96] (NRSE) instrument MUSES and thereby to gain two orders of magnitude in solid-angle detection. The curved large-solid-angle (LSA) resonance coils are the critical difficulty we have to overcome in order to make the project come through. Two such coils, with different radii of curvature (0.4 and 2.2 m), have to be constructed for the "second arm" of the spectrometer (Figure 25a). Each of these coils consists of a vertical winding producing a static magnetic field ranging from 1.5 to 35 mT. A pair of radiofrequency (RF) coils is inserted inside this static coil, is used for the generation of an oscillating field in the frequency range from 50 kHz to 1 MHz (Figure 25b).

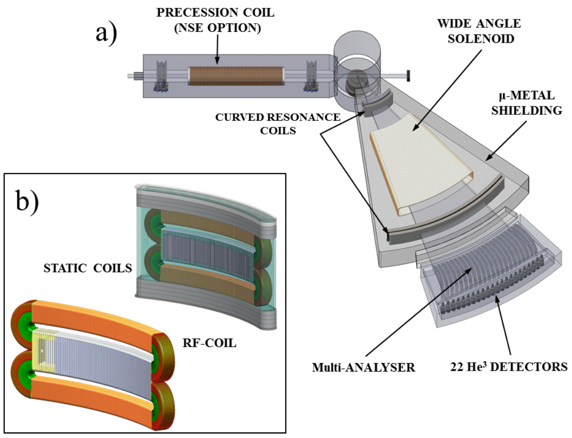

Figure 25: Layout of the Multi-Muses spectrometer (a) and design of the first curved radiofrequency and static coils situated at a distance of 40 cm from the sample (b).

The homogeneity ($\delta B/B$) of the static and RF fields should be of ~10^{-3} and 10^{-2} respectively over the whole range of use. One of the key points consists in minimizing the stray fields created outside the coils, which normally compensate on each side of a flat coil, but no longer do when the plane of symmetry is lost. In order to reach 35 mT, the required current is of the order of 100 A, which, owing to the need to use aluminum wires on the trajectory of the neutron beam, generates a thermal power of 2 kW per coil. Therefore, a highly efficient cooling system, including water and air circuits, must be designed.

We started with the design and construction of the first pair of RF curved coils, which correspond to the smaller radius of curvature and thus are more difficult to build, in terms of good field homogeneity. Magnetic field calculations for this geometry showed that the homogeneity of the static magnetic field is approximately three orders of magnitude higher when using a mu-metal yoke for each coil, rather than mu-metal plates for the interconnection between the two coils of the pair. The residual magnetic field is also two orders of magnitude lower in case of using mu-metal yokes. The RF coils are presented in Figure 26, while being tested in the beam of the spectrometer. The field homogeneity currently achieved perfectly fulfills the requirements for high-resolution measurements.

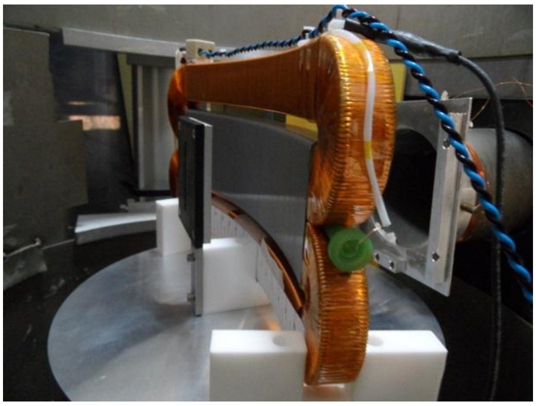

Figure 26: The new RF coils used for neutron resonance spin-echo spectrometry during the test of its field homogeneity in the neutron beam on Muses. The effective beam area covered is 22×5 cm^2.

[95] J.-M. Zanotti, S. Combet, S. Klimko, S. Longeville and F. Coneggo, Neutron News **22** (2011) 24.
Present and future of the quasi-elastic neutron spectroscopy at LLB. More than simply samples: devices.
[96] R Golub and R Gähler, Phys. Lett. A **123** (1987) 43

Diffraction group

The Diffraction group operates three single crystal instruments **5C2**, **Super-6T2**, **VIP**, four powder and liquid diffractometers, **3T2**, **G4.1**, **G6.1**, **7C2** in operation and **G4.4** under construction.

5C2, hot neutron four-circles diffractometer, focuses largely on the high resolution crystallographic studies of complex structures, in particular in hydrogen containing molecular compounds.

Super-6T2, highly polyvalent brand new diffractometer completed in 2007, offers rapid data collection, opening up the field of in situ photo-excitation experiments, high fields (< 7.5 T) and very low (50 mK) temperatures. Its high flux in combination with highly efficient PSD Detector allows routine measurements of sub millimetric crystals and epitaxial single crystals and extensive exploration of the crystal reciprocal space (see Figure below).

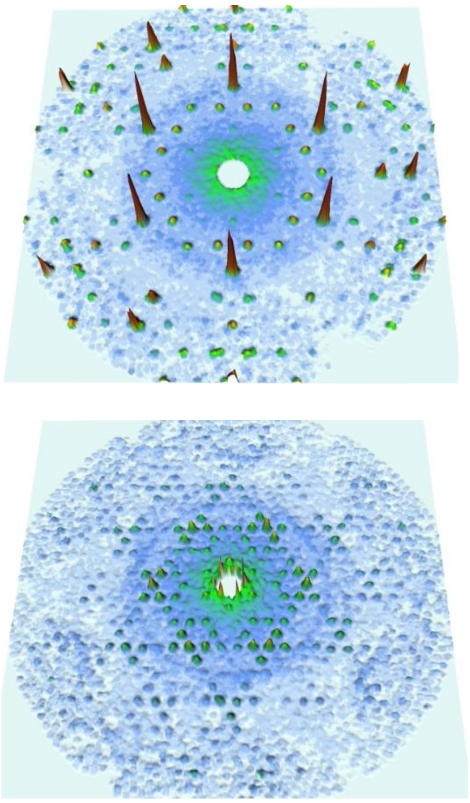

Figure 27: Nuclear (up) and magnetic (bottom) scattering from “distorted kagome” type compound $Dy_3Ru_4Al_{12}$ measured on Super-6T2.

VIP (see highlight) the most recent instrument delivered in October 2010 is a new type of polarized single crystal diffractometer equipped with a large PSD covering 1 steradian on the diffraction sphere.

3T2 upgraded in 2007 with a new 50 collimators/detectors block, is a generalist, high resolution, thermal diffractometer more particularly adapted to precise crystallographic determinations of compounds with cell volume < 1200 Å^3.

G4.1 is a polyvalent, medium resolution and high flux instrument equipped with an 800 cells multidetector, optimized for magnetic structure determination and study of phase transitions in function of temperature.

G6.1 (see highlight) is a high luminosity cold neutrons (4.7 Å) instrument with 2D detector for long period structures, liquids and studies under pressure.

7C2 (see highlight) located on the hot source of the reactor, performs structure factor measurements over a wide scattering vector range, necessary for the determination of the atomic correlations and the molecular interactions in glasses, amorphous materials, liquids and solutions. An in-depth transformation of the instrument has beenachieved in 2011 by using an assembly of 256 position sensitive tubes increasing the counting rate by a factor 25.

G4.4 project: high resolution banana-type diffractometer for the refinement of complex structures such as zeolites and pharmaceuticals, under construction. The diffractometer will offer 3 wavelengths (λ=1.8Å : Ge 115 / λ= 2.3Å : Ge 004 / λ =2.8Å : Ge 113) and a large 2θ range (~4° < 2θ < 170°) and will be naturally complimentary to G4.1 (similar wavelength, better maximum resolution Q_{max}, but lower flux) and 3T2 (better resolution $\Delta q/q$, but lower Q_{max}). It will be equipped with an Orange cryostat and will share a furnace (T_{max} ~1200°C) with 3T2. Typical data acquisition time is expected to be ~15h/scan.

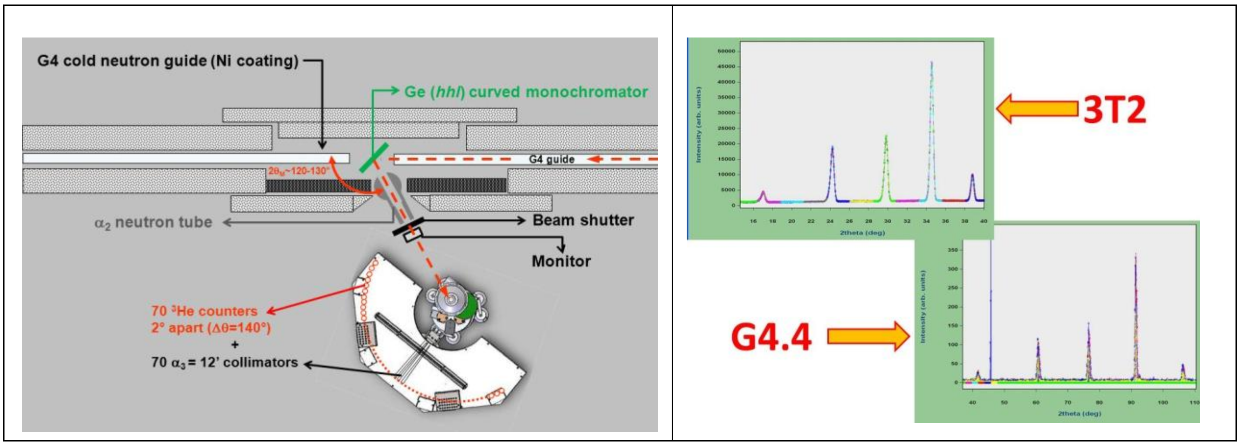

Figure 28 Schematic layout of G4.4 high-resolution diffractometer installed on cold G4 guide. The first diffractogram measured on G4.4 on a perovskite test sample

Sample Environments Developments

High pressures neutron diffraction

The G6-1 diffractomer can be used for high pressure studies. In the high pressure version, focusing devices are combined with sapphire anvil cells (Kurchatov-LLB type), inserted in an orange type cryostat. In this version, experiments on small samples (about 50 mg or a few mm3) have been recently performed up to 4 GPa and down to 1.5 K in collaboration with a Russian team. The pressure is quasi-hydrostatic with an uniaxial component or stress of about 0.2 GPa. The new flat two -dimensional detector enabled the study of the spin textures induced by this stress. In the low pressure version, the spectrometer is used as in the ambient pressure configuration, with a piston-cylinder Cu-Be cell, inserted in the orange cryostat down to 1.5K. The sample volume is higher (typical volume of 0.3 cm3) and the maximum pressure about 1.2 GPa.

Wide Angle VEctor magnet (WAVE) project

The aim of the WAVE project is to develop and build a wide aperture vector magnet suitable for neutron scattering experiments, to overcome the limitations of the unidirectional magnets now used in single crystal diffraction and inelastic scattering measurements. Such a device would open new opportunities for studies on multiferroics, molecular magnets and spin ice compounds. We propose a very innovative design (French patent #1262070), able to fulfill all our requirements: in collaboration with the IRFU/SACM division of the CEA, we have work on a design in which the stray field coming from quaternions of superconducting magnets is used to obtain the horizontal components of the magnetic field (see figure). With this design, the diffraction plane (and a ± 10° vertical aperture) is completely free so that wide angle neutron scattering is feasible. The sample bore is very large (diameter is 100 mm) and the field homogeneity is excellent (much better than 0.1 % on a 1 cm diameter sphere).

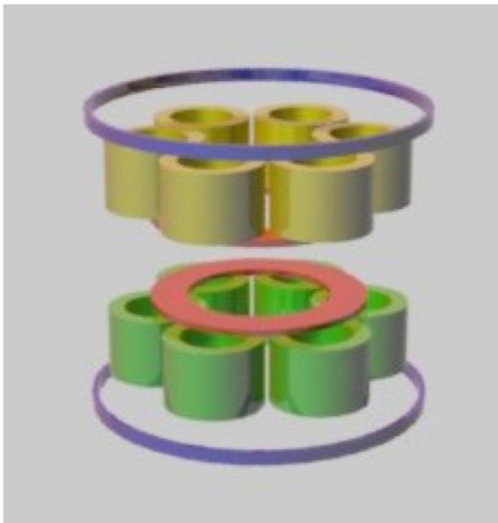

Figure 29: schematic representation of the coils of the superconducting magnet. The horizontal components of the field are obtained through 3 sets of 4 magnets (yellow and green), and the vertical component thanks to a standard Helmholtz coil (in orange). A blue coil is used for active magnetic shielding

Levitation

An aerodynamic levitation technique has been tested on the 7C2 spectrometer last May. This experiment has been developed and fine-tuned by the group of Louis Hennet (CEMHTI- CNRS, Orléans), with the collaboration of Laurent Cormier (IMPMC, UPMC-CNRS, Paris). The experiment setup consists of a nozzle connected with an Argon bottle: a small sample (2-3mm size) is put on the top of the nozzle and a mass flow controller (delivering between 0.5l/min and 2.5l/min) allows the sample to levitate. Once the sample is levitating, it is heated by means of a CO_2 laser unit of a maximum output power of 170W. The temperature is monitored by means of an optical pyrometer which is calibrated from the melting point of the studied sample.

The first measurement on 7C2 has been performed in September and beam time has been already requested for next year.

NMI-3

Within the NMI3 JRA "Polarized Neutron Technique" (2010-2012), the group was engaged in the development of large solid angle magnetic environment friendly spin-handling devices. LLB have developed a system of neutron guide fields which can be mounted on the 4-circles diffractometer and allows to orient the incident (and final) polarization in any desirable direction. This will allow to measure longitudinal polarization terms for any crystal orientation, while the existing devices CRYOPAD or MUPAD allow doing that only for one preliminarily selected crystal plane.

ESS Outlook

In the perspective of the future European neutron Spallation Source Diffraction group is involved in the development of news concepts to prepare and design the future neutron scattering instruments. In particular a Project of Magnetism Diffractometer at ESS is conducted by the group members in view of its submission as a French in-kind contribution to the ESS.

Scientific target for MSCD (Magnetism single crystal diffractometer at ESS) are complex magnetic systems having a large number of degrees of freedom (typically charge, magnetic and orbital moments on several inequivalent atoms).These include functional materials of the high Tc superconductor family, multiferroics, materials for hydrogen storage applications and molecular crystals for high-density, three-dimensional storage materials, known as single molecule magnets (SMMs). First Monte Carlo simulations have demonstrated that the exceptional qualities of the long-pulse spallation source translate into strongly enhanced performance of single crystal diffractometer. This is due to the fact that the ESS duty cycle matches perfectly well the wavelength resolution required for single crystal diffraction. This opens new opportunities to study microscopic samples under extreme conditions of pressure, temperature, magnetic field and enables in-situ photo excitation measurements.

The MSCD diffractometer can be considered as a general purpose crystal and magnetic structure instrument optimized for crystals with unit cell from 15x15x15 Å to 25x25x25 Å. It will use the time-of-flight (TOF) Laue technique to access large 3-D volumes of reciprocal space in a single measurement.

In March 2014 an ESS Science Symposium on Single Crystal Diffraction will be organized by LLB in Paris, were we plan to join neutron experts and potential users of the ESS, in order to uncover the most important scientific community needs in neutron diffraction. This should help us to fit the MSCD characteristics to the community expectations.

Diffraction Group is also intended to participate in the ESS powder diffractometer project POWHOW at the construction stage and the project of High Magnetic Field environment in collaboration with LNCMI in Toulouse.

***Members**: G. André (CEA), A. Bataille (CEA), B. Beuneu (CEA), A. Cousson (CNRS), F. Damay (CNRS), J. Darpentigny (CNRS), J. Dupont (CNRS), B. Gillon (CNRS), A. Goukassov(CEA), X. Guillou (CNRS), J-L. Meuriot (CEA), I. Mirebeau (CNRS), L. Noirez (CNRS), R. Papoular (CEA), F. Porcher (CEA), B. Rieu† (CEA), T. Robillard (CEA)*

4. The new 7C2 for liquids and amorphous materials: a counting rate multiplied by more than 25!

In the framework of the CAP 2015 program, the diffractometer 7C2 has been strongly upgraded by means of a new detection device that improves the counting rate by a factor around 27, without loss in the signal to background ratio.

The instrument is dedicated to structure factor measurements (mainly liquids and amorphous materials), and it uses the high energy neutrons of the hot source of the ORPHEE reactor. 3 wavelengths are available, 1.1, 0.72 and 0.58 Å. We have kept the main asset of this instrument, which is the acquisition of the all q domain in one run. This is required for unstable systems, and for kinetics, and concerns a lot of high temperature experiments carried out with 7C2.

The new detection device consists in 256 vertical position sensitive tube detectors, of diameter 1/2'' (Figure 31). The gain in counting rate is nearly equally distributed between detector efficiency and detection solid angle:

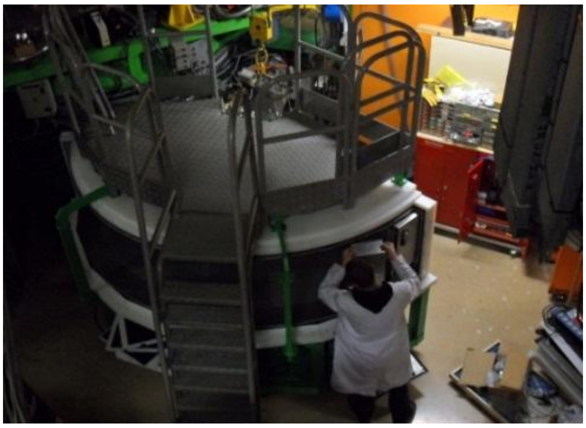

Figure 31: assembling the detector of the new 7C2 (the scattering angle width is around 135°).

- the tubes are filled with 30b of ^{3}He, which multiplies the efficiency by about 4.5 times
- the detection height around 47 cm increases the detection angle by a factor about 5.5.

The tubes are paired at bottom and grouped in independent modules of 8 pairs with their own electronics (Figure 31). Pairing the tubes reduces the number of electronics and cables without any effect on the instrument resolution (the vertical detection resolution is 0.8cm, less than the sample height).

The primary instrument remains unchanged, as well as the sample environments.

Figure 30: a detection module of 7C2: 16 tubes (paired at bottom) plus their electronics.

After a pre-mounting at the end of 2011, the new detector has replaced the previous banana in the beginning of 2012. Between the reactor start and the summer break (March 2012-June 2012), we have adjusted the electronics, and carried out stability tests and calibrations, and some first test experiments.

The new 7C2 has been opened to users after the reactor summer break in 2012.

It gives now access to structure factor measurements at 0.58 Å (the low flux -ten times less than at 0.72 Å-, was crippling before). And it has already opened to new kinds of measurements (kinetics, very small samples), requesting new sample environments. A sample changer with 40 positions is developed. A gas levitation device with laser heating is under installation for high temperatures or super cooling (Figure 32).

This upgrade puts 7C2 at the best international level of reactor diffractometer for disordered structures with D4 at ILL.

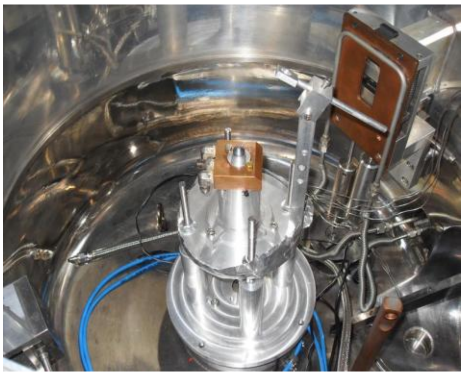

Figure 32: the gas levitation nozzle in the 7C2 vacuum chamber.

5. Very intense polarized (VIP) neutron diffractometer at the ORPHEE reactor

VIP - Very Intense Polarized neutron diffractometer is an upgrade of the former 5C1 diffractometer. It is the second instrument delivered in the framework of the LLB CAP2010 instrumentation program. The aim of the project was to decrease the measurement time of spin densities by a factor of 5 to 20, depending on the crystal lattice parameters. This has been achieved by using large Position Sensitive Detector (PSD) which allows measuring the flipping ratios on several reflections simultaneously.

VIP is installed on the beam tube located on the hot source of the ORPHEE reactor in replacement of the 5C1 diffractometer (Figure 33). Heussler polarizing monochomator is used to select the neutrons of wavelength 0.84 Å. Neutrons scattered by the crystal in the directions satisfying Bragg condition are collected by 64 position sensitive detectors (15 bar of 3He) of 19 mm in diameter covering scattering angles of 25° in vertical and 80° in horizontal directions .

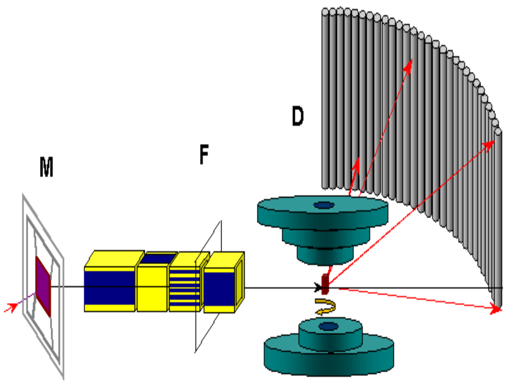

Figure 33: Schematic layout of VIP diffractometer. (M) is a polarizer, (F) is a flipper, (D) PSD.

Rotating of sample and collecting of 2D diffraction patterns allows us to record both integrated intensities and flipping ratios up to sin ϴ/ λ ~ 0.8 Å-1 in the horizontal plane and sin ϴ/ ~ 0.2 Å-1 in the vertical one. Then flipping ratios are re-measured with a longer exposition time for the frames having low statistical accuracy to reach the required one.

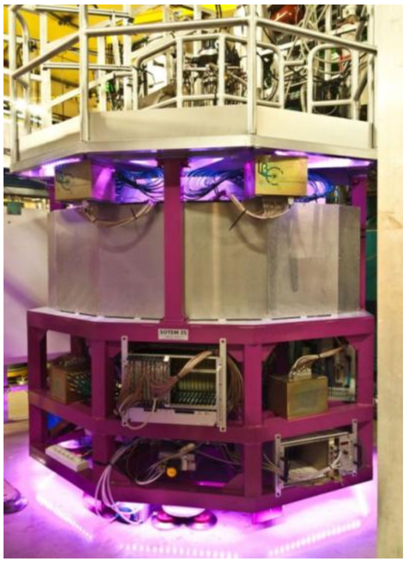

Figure 34: Picture of VIP.

To reduce the scattering from the sample environment a radial collimator is installed in the detector shielding. It consists of 64 foils coated with Gd oxide (Eurocollimators Ltd.) and covers full scattering range.

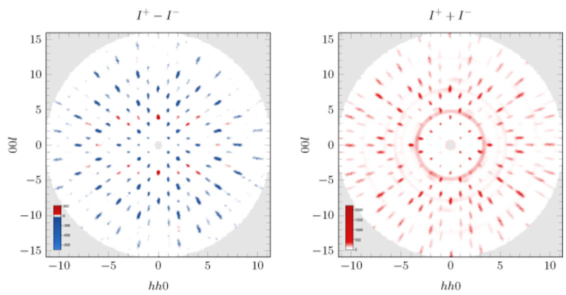

Figure 35: Two dimensional intensity cuts [hhl] in the reciprocal space measured on VIP from Yb2Ti2O7 (about 100 mm3) during 5 hours. Left panel: The difference I+ - I-. Right panel: The sum I+ + I-.

Another important gain in efficiency of PND instruments with PSD is due to a simplification of the measurement procedure, since some of time consuming processes like crystal orientation, lattice parameters refinement can be done during collection procedure or even later. Ensemble of these measures allows do decrease the data acquisition rate of magnetization densities by a factor of 5-20 depending on the crystal cell parameters.

6. Upgrade of high-flux and long wavelength G6.1 diffractometer for small samples and high pressure studies on long period systems

The G6.1 diffractometer (Micro) is dedicated to the study of weakly diffracting samples or samples under high pressure. The renewal of its detector was planned in the framework of the LLB CAP2010 instrumentation program and achieved in 2012. An upgrade of the upstream part of the instrument is planned in 2013.

The original design of the new flat Position Sensitive Detector (PSD) replacing the old 400 cells "banana-type" detector was set in order to optimize the signal-to-noise ratio of measurements on small samples in pressure cells. Here, the vertical array of sixteen, one meter-long, PSD tubes, with 6mm horizontal resolution collects the scattering intensity up to 2□ ~80° in one detector position.

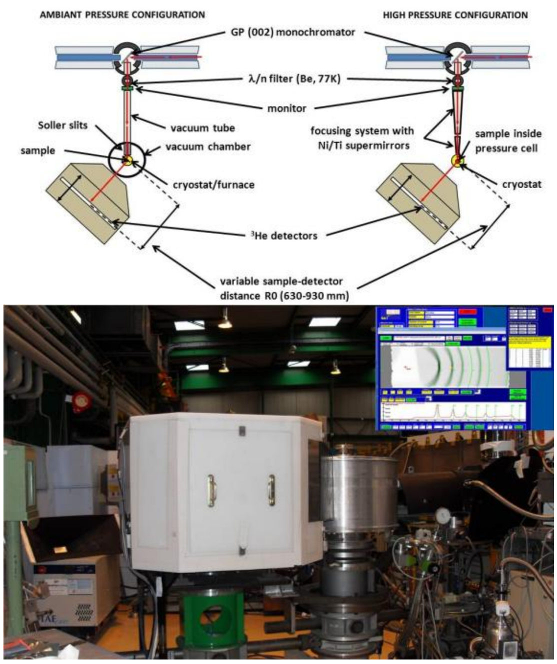

Figure 36(up) Schematic layout of G6.1 diffractometer (down) G6.1 diffractometer measuring at ambient pressure and temperature and the Debye rings from a zeolite sample, their indexation and integrated 2□-I diffractogram.

The adjustable detector-to-sample distance and versatile integration of Debye rings out of scattering azimuthal plane allows modulating the resolution of the 2θ-I diffractograms, at the expense of the intensity. Typical acquisition times range from ¼h-1h for samples at ambient pressure to 8h for measurements in sapphire-cell.

The λ=4.74 Å wavelength, coming out the PG monochromator, is filtered out by polycrystalline Be cooled at liquid N_2 temperature. The initial 15x50 mm beam can be concentrated in a ~5mm-diameter spot by focusing mirrors when high pressure cells are used. With this long wavelength the Q-range spanned is $0.13Å^{-1} < Q < 2.50Å^{-1}$, and is intermediate between SANS spectrometers and a cold diffractometer like G4.1. For that reason, the high efficiency and flat 2D detector of G6.1 is also adapted to the study of medium-size systems, such as liquids under confinement.

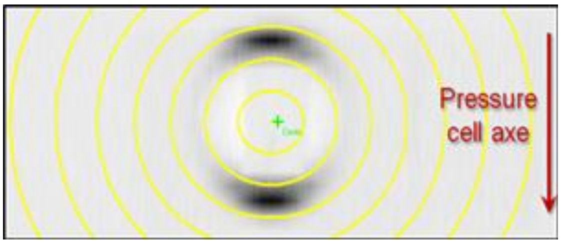

Figure 37: The pressure induced magnetic texture of the chiral magnet MnGe imaged on the zero satellite using the 2D detector. The enhancement of the intensity is along the axis of the pressure cell.

Concerning crystallized systems, the G6.1 resolution is well suited for long-period magnetic systems such as chiral magnet MnGe (Figure 37) or structurally complex systems such as zeolites or MOFs (Figure 38). The fast newly installed closed-cycle cryostat compatible with various sample cells for *in situ* pressure or dielectric measurement is a supplementary asset for G6.1.

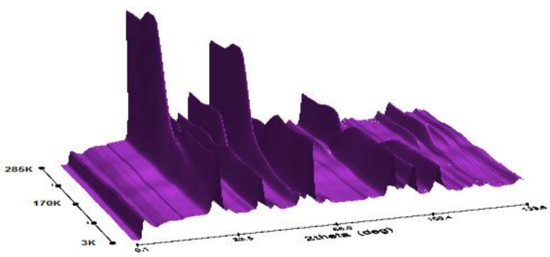

Figure 38: Following hysteretic phase transition between open- and closed- forms in MIL-53(Al) archetype MOF

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Large Scale Structure Group

The Large Scale Structure group has in charge the operating of

- **two Small Angle Neutron Scattering** (SANS) spectrometers, PACE and PAXY, for the determination of structures of all kinds in advanced engineering materials in the range 1nm - 50nm and **one new Very Small Angle Neutron Scattering** (VSANS) spectrometer, TPA, which allows to access larger scale range, 100nm - 1µm;
- **two diffractometers**, DIANE dedicated to the analysis of residual strains and 6T1 for the determination of crystallographic textures;
- **two reflectometers**, EROS the horizontal time flight reflectometer dedicated to the study of soft matter and biology (polymers, liquids) and PRISM with polarization analysis for the studies of magnetic systems.

Several important events occurred in the group recently. In 2010, the **VSANS instrument TPA** has been opened to users (highlight 1). This new kind of instrument, a Very Small Angle Neutron Scattering, aimed at filling the gap between classical Q range of SANS and the Q range of light scattering, 10^{-3} 10^{-1} nm^{-1}. On TPA, we routinely reach a minimum Q value of 3 $10^{-3}nm^{-1}$@1.2nm wavelength. Besides, the polarized SANS instrument, PAPYRUS, has been dismantled: its fixed wavelength and tedious change of sample to detector distance were two handicaps to a wide use of the instrument. Instead, we worked on the new **SANS project PA20**, the SANS/ GISANS project for soft matter, materials & magnetism (highlight 2). In July 2012, the 3rd SANS spectrometer, PAXE, has been closed in order to start on the free guide end G5, the installation of new PA20 components. The diffractometer Diane was prior moved and reinstalled on the G4 guide. This change was operated together with the development of a new instrument **Super 6T1** with a high flux, which will combine both texture and strain measurements on a thermal neutron beam (highlight 3). After the shut-down of the Time of Flight spectrometer of LLB, MIBEMOL, the guide end G6 is now prepared in order to welcome by the end of 2013 the renewed **EROS** spectrometer (highlight 4); the straight and taller guide G6 will lead a gain of a factor 4.

Developments of Instrumental Options and Spectrometers Upgrades

Aside major instrumentation works and spectrometers projects previously listed, other technical improvements have been achieved on the existing instruments during the recent past years.

Focusing MgF_2 lenses for SANS

Use of refractive lenses implemented in SANS could make possible to measure scattering patterns down to smaller Q values than usual ones and increase the number of neutrons scattered by a larger sample without decreasing the Q resolution. Such option is now available on PAXY, our 7 m long detection SANS spectrometer. A specific device made of 24 biconcave MgF_2 lenses with an active area of 22 mm diameter focusing at 6.8m distance from the sample allows a gain of minimum a factor 3 at a wavelength of 1.5nm. A similar option is foreseen for the 20m long PA20 spectrometer: a new issue could be to reduce significantly the phonon scattering by lowering the temperature of the whole device, thus reducing the absorption by the lenses packing as recently shown by a JCNS scientist group.

Time of Flight SANS

Time of flight SANS on a reactor like Orphée is obtained by removing the velocity selector which usually select of band of wavelength and introducing a chopper in order to get a short pulse of a white neutron beam. This method has been sometimes used at LLB since it allows accessing a wide Q range in a single measurement with a very high Q resolution. The latter reason has motivated recent TOF experiments on the vortex lattice structure of a single crystal of Niobium, a low T_c supraconducting material on the multidetector of PAXY. Further developments of both data acquisition and data treatment on large multidetector are required in order to gain experience in TOF SANS technique, which will be used in the future European Spallation Source.

Multitube detector for PAXY and PA20

The old BF_3 multidetector of PAXY, a 128*128* 5*5mm^2 multidetector (built in 1970), is being replaced by a new He^3 multitube detector made by ILL. The high pressure of He^3 gaz in 5mm width tubes will ensure high detection efficiency, together with a high pixel definition (5mm). We are expecting gains of factor 3 at 0.5nm and 1.7 at 1.5nm neutron wavelengths. Similar multitube detector will equip the new spectrometer PA20.

Data Acquisition and Treatments

The IT group of LLB is spreading its data acquisition program, PINGOUIN, on the LLB instruments: 6T1 is now operating with this program and EROS has started the transition. A more friendly data acquisition program has been nevertheless installed on PACE, PASiNet running with Matlab functions and a user interface developed with Visual.NET. Data treatments and fitting procedures of SANS and reflectivity measurements are also developed in the new version of PASiNET, a multiplatform software available on the Net.

Sample Environments Developments

Permanent works are achieved in the group to provide new or improved sample environments for users on the spectrometers.

Electric field cell for SANS

Application of an electric field can modify the structure and organization of soft and colloïdal materials. One problem often encountered in water solution is water electrolysis. So, in order to overcome this problem, we have designed and built an electric field cell for SANS, in which the two electrodes are located outside on both sides of the sample. The electric field is created between the two disc shaped electrodes using a high voltage power supply: as example, 0.5kV/mm with 10kV power supply. A prototype was first developed in the framework of the ANR BIOSELF (2009-2011). It is now improved in the NMI3 JRA "Advanced Neutron Tools for Soft and Biomaterials" (2012-2016) in order to provide thermalization, AC field power supply, choice of parallel or perpendicular geometry.

Stretching deformation machine

A traction machine has been designed in order to stretch in-situ rather viscous materials like polymer composites or gels. The maximum torque is 1.17Nm. The stretching velocity can be varied in the range 77µm/s - 3.85mm/s. The device is working inside a temperature isolated container regulated with an external thermostat bath: the temperature range can be varied from 12 to 80°C +/- 0.2°C. The traction machine can also be mounted on Swing, the SAXS line at SOLEIL.

EROS Sample environments

New sample environments were developed all along the rejuvenation of the EROS II spectrometer.

- A new Langmuir trough enabling in-situ measurements with either constant surface pressure or constant surface area is now routinely used. It was purchased with a funding from the "RTRA Triangle de la Physique". The same trough has been purchased and installed on the new liquid surface beamline SIRIUS at SOLEIL.
- In-situ experiments under shear were achieved using commercial controlled stress rheometer Carri-med CSL 100 with a cone/plate geometry enabling to apply shear rate up to 2600 s^{-1}. A specific cell was designed to measure reflectivity at the silicon/toluene interface while mounted on the rheometer.
- A home-made cell enabling appliance of electric field up to 5V at the silicon/water interface for in situ measurements has been developed using doped silicon.

Within the NMI3 JRA "Neutron Optics" (2009-2012), several innovative optical systems have been evaluated. A multilayer diffractive optics device (built at HZB) has been regularly tested on EROS in order to perform energy analysis so as to be able to use a full white beam in reflectivity experiments. Different concepts of multibeam focusing systems for reflectometers have been Monte Carlo simulated. A new concept of focusing SANS using reflective optics has been built (scale ¼) and tested at PSI.

PRISM Sample environments

The sample environment on PRISM offer now renewed and extended capabilities for magnetism surface studies.

- An electromagnet coupled with a closed cycle fridge allows measurements at temperatures ranging from 10K to 300K in fields ranging from --.4T to +0.4T. This sample environment is very versatile and fulfills the needs of most users: it allows to work from very low fields (10G) up to rather large fields (0.4T). The magnetic field polarization can also be reversed.
- A set of 3 Helmholtz coils allows applying small magnetic fields (10mT) in the 3 directions in space. These coils can be coupled with a displex or a furnace (240°C).
- Spectromag cryomagnets (7T or 10T) can be used when very low temperatures (down to 1.6K) or high magnetic fields (>2T) are required. Polarized mode cannot be achieved at maximum field (10T).

Within the NMI3 JRA "Neutron Imaging" (2012-2014), the LLB is engaged in the development of methodology and numerical tools for the processing of Polarized SANS data on magnetic nanostructures.

TPA Sample environments

Various equipments are progressively installed on the versatile sample stage of TPA:

- A three stages thermalized sample changer allows VSANS measurements on 24 samples at temperatures in between 12°C to 80°C (±0.5°C to ±2°C as function of the different samples respectively).
- Spectromag cryomagnets (7T, 10T) can be operated on TPA.

A Neutron Imaging at LLB

A new imaging station is being built on the cold guide G3bis for high resolution (down to 50 µm) neutron imaging. A high resolution camera with different scintillators has been successfully tested on the "white" neutron beam of the EROS spectrometer. The construction of the station could start by the end of 2013, once EROS II will be moved on the guide G6. Its operation could occur in autumn 2014. The scientific area of this imaging station will be first oriented towards foods and soils studies. Archeology and material science are also concerned.

European Spallation Source Outlook

In the perspective of the future ESS, the LLB and more specifically the LLS group is involved in the development of news concepts to prepare and design the future neutron scattering instruments. One project concerns the proposition of construction of a high intensity long SANS instrument. This project, named SKADI, driven in close collaboration with the Jülich Centre for Neutron Science (JCNS) in Germany, will be proposed to the ESS instrument committee on October 2013. The concept is a multi-purposed instrument with polarized neutrons dedicated to soft matter, biology, material science and magnetism, whose high flux performances will enable to reduce the sample sizes and perform time-resolved experiments. The LLB team will developed a VSANS multi-beam option to cover a Q range from 10^{-3} to $1nm^{-1}$. The performances of the setup are expected to be 30 times more efficient than D22 at ILL Grenoble. A second project with JCNS, the construction of a vertical reflectometer to study both magnetic or soft matter surfaces and interfaces is also on the way.

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7. Access to very small angle neutron scattering at LLB: "Très Petits Angles" (TPA) spectrometer

The construction of a Very Small Angle Neutron Scattering instrument was decided in the framework of the CAP 2010 program of LLB. We have thus built TPA (Très Petits Angles), a Very Small Angle Neutron Scattering instrument, based on the converging multi-beam principle. The aim was to extend by one order of magnitude towards the low q values, i.e. down to about 2 $10^{-3}nm^{-1}$, in order to be able to study objects from 100nm to 1000nm size.

The original design of TPA first includes a high definition (pixels size of $0.15*0.15mm^2$) Image Plate detector. Since image plate suffers from an overwhelming gamma background, cautions have been taken against γ radiation emission: heavy concrete shielding around the detector tank, ^6Li instead of ^{10}B and Cd at any other strategic places of the instrument, a double super-mirrors (m=3) monochromator instead of a velocity selector to select the wavelength, from 0.5 to 1.5 nm (FWHM=14%). In order increase the "useful" neutrons, a 3.5 m long trumpet guide (m=2) (reducing the incident divergence of the incoming neutron beam) has been installed upstream the instrument. The most innovative element of the instrument is its multi-beam collimator, consisting of a set of 13 masks made out of ^{6}Lithium (neutrons absorber) mixed in epoxy resin. Actually, each mask offers two types of collimation: the first is a pinhole collimation including approximately 600 circular holes with a hexagonal order from 1.28 mm (1st mask) to 0.9 mm (13th mask) in diameter and the second collimation is 15 vertical slits of 1.28 mm to 0.9 mm width. This set of masks accurately positioned along the beam trajectory is delivering a beam converging on to the detector located about 6.2m far from the sample. Vertical masks adjustments as function of the wavelength allow the correction of gravity effects inside the collimator, while preventing neutrons loss and cross-talks. The beam area at the sample position is about 25 mm height and 7 mm width. The overall size of the instrument remains small, 10 m, reducing the Q resolution enhancement in the vertical direction due to the broad wavelength distribution of the incident beam.

With this new instrument opened to users since 2010, we have performed VSANS experiments down to 2.8 $10^{-3}nm^{-1}$@1.2nm with very high resolution (~8 $10^{-4}nm^{-1}$) in both Q_x and Q_y directions. The slit collimation option still remains to be tested and the inherent geometry deconvolution must be validated.

TPA allows to perform VSANS measurements on soft matter systems (polymersomes solutions[97], pickering emulsions[98] or nanohybrid mixtures (emulsions stabilized with gold nanoparticles)), but also in natural materials (as for the enzymatic degradation of cellulose), in the field of material science, as on sintered graphite under pressure or on geological rock[99]. One recent example on superconducting material, as low T_c Niobium, is reported on Figure 39.

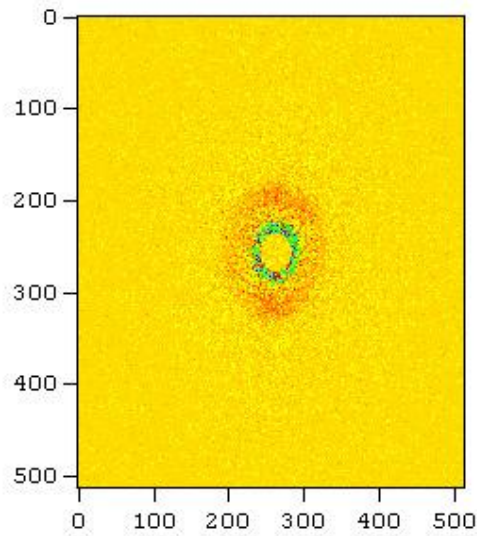

Figure 39: Scattering pattern from superconducting polycrystalline sample of Niobium obtained on TPA after 1.5hours measurement. A scattering ring centered at q=.00137 $Å^{-1}$ corresponding to a disordered vortex lattice is obtained at 8.5K after a field cooling at 75G, (unpublished data of A. Pautrat, CRISMAT Caen).

[97] A. Brûlet, V. Thévenot, D. Lairez, S. Lecommandoux, W. Agut, S. Armes, W. Du, S. Désert, J. Appl. Cryst. (2008), **41**, 1, 161-166.
[98] F. Muller, J. Degrouard, J. Jestin, A. Brûlet and A. Salonen, Soft Matter, **8**(40) (2012) 10502.
[99] G. Bicocchi, R. Magli, A. Brûlet & M.-H. Mathon Highlight LLB 2011

8. A versatile SANS/GISANS instrument for nanoscale science at LLB: PA20

SANS is well-known to be an efficient technique to determine the structure in the fields of soft matter, materials and nanoscale objects. It is particularly the case for complex or heterogeneous systems, like polymers, micelles, composite materials, and long-range magnetic orders. Prompted by growing demands from the SANS user base and by the necessity to stay competitive, LLB has included in its instrumental upgrade program CAP 2015 the construction of a new SANS instrument, PA20.[100]

PA20 will replace an older SANS instrument at the end of the G5 guide and will considerably extend our capabilities in terms of SANS for magnetism with a polarized neutron and Grazing Incidence SANS (GISANS) options. An improved dynamical Q-range can also be achieved thanks to two detectors in front and rear positions of the detector tank, and / or by using a Time-of-Flight option.

The total length of PA20 will be 40 m, including a 19 m collimation length, a 20 m detector tank containing high-resolution/high-efficiency XY detectors, and a casemate containing a monochromator (velocity selector λ= 0.3-2 nm), a chopper system for Time-of-Flight (TOF) mode, a polarizer and an RF spin flipper. The collimator will offer the possibility to use either square or slit shape apertures for GISANS or specific SANS measurements, two sets of MgF_2 neutron lenses, and also leave room for future implementation of modern techniques in neutron optics such as new guide shapes or multi-beam devices. Regarding "user-friendliness", PA20 will allow faster time-resolved measurements, with "single shot" access to a wider range of scattering vectors Q (dynamic Q-range of $Q_{max}/Q_{min} \approx 50$), on smaller samples (few mm in size). In addition, polarized neutrons will enable magnetic studies in both SANS and GISANS configurations. Studies of nanostructured surfaces and interfaces (deposited or embedded nano-objects), magnetic domain formation, multilayered materials or magnetic thin films through specular and off-specular signals will be possible thanks to the GISANS setup. The versatility of PA20 should contribute to both enlarge the neutron user community, especially in expanding areas like Nanosciences and Biology and offer improved services for users in the years to come.

The construction phase of PA20 has started in 2012 once the older SANS spectrometer PAXE has been dismantled. It will continue throughout 2013 with the commissioning phase by the end of 2014. As in May 2013, several pieces have already been delivered (casemate and its equipment, spin flipper, polarizer, collimator), the remaining parts (sample area, detector tanks and the ^{3}He detectors) are due in the course of 2013 and beginning of 2014.

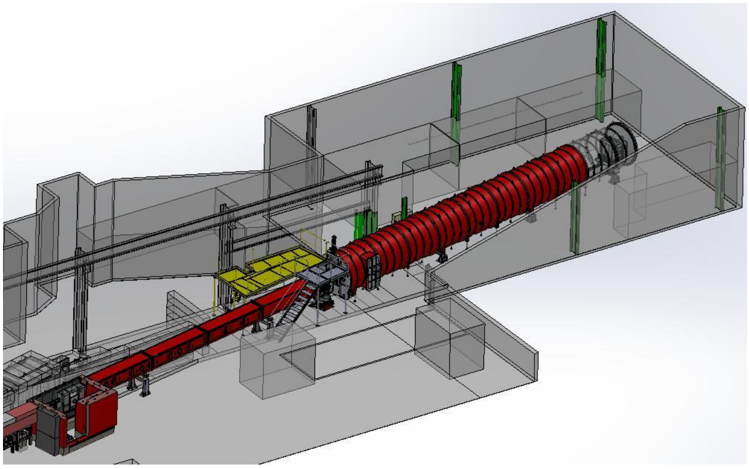

Figure 40: Future layout of the Small Angle Neutron Scattering spectrometer at LLB, PA20 in the Orphée Guide Hall. From left to right : 1) the heavy concrete casemate housing the monochromator, the polarizer and the spin flipper located at the end of the G5 guide, 2) the 16m long collimator, 3) the sample area with an upper platform, 4) the 20m long cylindrical tank housing the detectors.

[100] G. Chaboussant, S. Désert, P. Lavie, A. Brûlet, Journal of Physics: Conference Series **340**, 012002 (2012).
G. Chaboussant, S. Désert, A. Brûlet, European Physics Journal, Special Topics **213**, 313-325, (2012)

9. Super 6T1: the new diffractometer for texture and stresses analysis

In the framework of the CAP 2015 program, the texture spectrometer 6T1 is currently refurbished to increase its capabilities and then offer the possibility to measure strains and crystallographic texture using thermal neutrons. This instrumental development has been motivated by the necessity to combine the two distinct diffractometers dedicated to strain and texture studies, as well as by the growing interest of the communities of engineers and metallurgists for the characterization of microstrains and of mechanical behavior heterogeneities. This kind of research requires coupling the textures analysis and the strain measurements (including *in situ* experiments under load). The deformations will be measured with several Bragg peaks, and studies coupling stresses/textures will be achievable with shorter acquisition time.

The development of 6T1 device was designed in three stages over several years in order to spread the financing plan while keeping for users a quasi-continuous access to the spectrometer.

The first step operated in 2012 was to install a new sample - detector support which contains an innovative Euler cradle including x, y, z translations allowing to perform maps of internal strains and / or texture. In parallel, an original sample changer for measuring pole figures of twelve samples sequentially was developed. Concerning the sample environment, the new cradle is compatible with our *in situ* measurement devices under load or temperature (tensile machine associated with an image correlation system, and furnace).

The second step scheduled for autumn 2013 is the installation of the new 2D detector (from DENEX company) with a working area of 200x200 mm^2 and a set of four radial collimators that will cover a wide resolution range.

Finally in a future final step, the diffractometer will be modified in order to provide six wavelengths thanks to focusing monochromators (Cu (111) and PG (002)) and three different take-off angles. Therefore, the protection of the output beam must be modified to ensure a quick and easy change of wavelength. This development is scheduled for 2014.

Currently, the new 6T1 is already a very competitive instrument for texture analysis and microstrain, covering a large part of scientific applications in the field of mechanical engineering and metallurgy.

By the end of 2014, the variable wavelength will give access to a measurement geometry near $2\theta = 90°$ to achieve mapping of residual stresses. For industrial purposes, parts weighing up to 25 kg can be studied.

Figure 41: 6T1 sample support with translations x, y, z incorporated into Euler cradle.

Figure 42 Automatic sample changer with 12 positions for texture measurements on super 6T1 at LLB.

10. A new era for reflectometry at LLB with the boosted EROS II

EROS is a versatile horizontal time-of-flight reflectometer enabling measurements in various geometries (air/solid, air/liquid, solid/liquid) that was originally built in the early 90's. In the framework of the CAP 2010 program, it was largely upgraded as a new home-made designed multi-discs chopper with tunable resolution was installed, as well as a new collimator, to become the rejuvenated EROS II reflectometer. The gain in flux of about 50 was reached compared to its nominal version.

The benefits from such rejuvenation were noticeable over the last period 2008-2013, where the mature EROS II makes reflectometry enter a new era at LLB. First, the reduced acquisition time enabled to welcome more experiments. This resulted in a *high publication rate* in peer-reviewed journals[101] from both external users and in-house research, as well as dedicated actions towards industrial partners (Swiss neutronics, Essilor). Second, *new limits for possible minimal reflectometry* have been reached, down to a few 10^7 in a few hours[102]. The actual remaining limitations are now usually due to intrinsic physical limitations from samples, i.e. the incoherent scattering. Third, *the lower limit of sample* surface was considerably reduced, down to 0.15 cm*cm [103], a hundredth of a conventional sample (~ 10 cm^2). Fourth, *high resolution* experiments allowed by the new chopper were achieved[104]. Fifth, the strong reducing of the acquisition time, down to a few minutes, has opened the way for *kinetics studies* in various samples environments, such as humidity chamber or measurements under shear[105]. Figure 43 show the time-resolved measurements of asphaltene adsorption under shear.

Finally, *off–specular measurements*, which are usually very long, are now possible in a reasonable time with a 2D detector, that can be used for dedicated requests instead of the conventional PSD detector. Figure 44 presents the Q_x-Q_z map of multilayered tubes of fatty acids adsorbed at the air-water interface[106] for an acquisition time of 30 minutes.

By the end of 2013, the EROS II will be installed on the free G6 guide, a much taller guide with more neutron flux at short wavelength than on its present position G3bis, along with a new tank for the 2D detector to become the HERMES reflectometer.

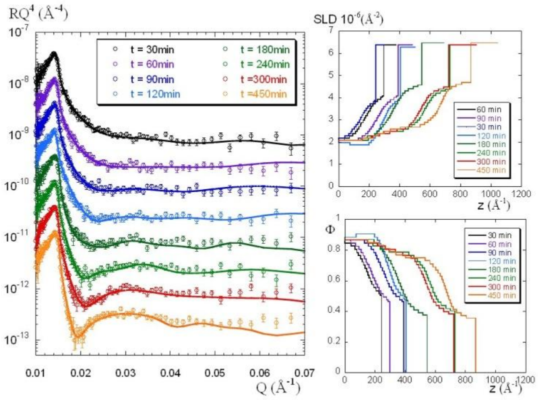

Figure 43: Kinetic measurements of asphaltene adsorption under shear[5] obtained on EROS II at LLB

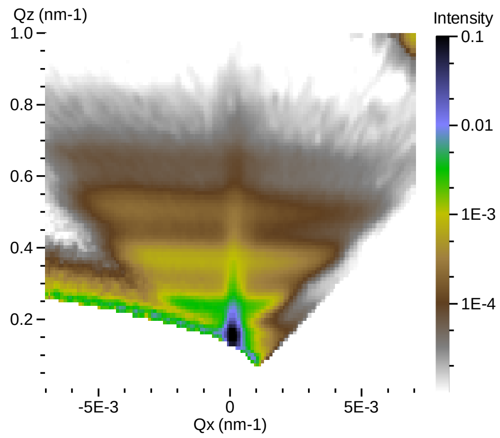

Figure 44: Q_x-Q_z map of multilayered tubes of fatty acids adsorbed at the air-water interface[6] for an acquisition time of 30 minutes obtained on EROS II at LLB

[101] 45 published papers in the period 2008 – 2012 (source ISI web of knowledge), 9 in 2013 and 2 actually submitted.

[102] F. Cousin, F. Ott, F. Gibert, A. Menelle, Eur. Phys. J. Plus, **2011**, 126, 109.

[103] F. Lechenault, C.L. Rountree, F. Cousin, J.-P. Bouchaud, L. Ponson, E. Bouchaud, Phys. Rev. Lett., **2011**, 106, 165504.

[104] O. Félix, Z. Zheng, F. Cousin, G. Decher, C. R. Chimie, **2009**, 12, 225-234.

[105] Y. Corvis, L. Barré, J. Jestin, J. Gummel, F. Cousin, Eur. Phys. J. Special Topics, **2012**, 213, 295–302.

[106] A.-L. Fameau, J-P. Douliez, F. Boué, F. Ott, F. Cousin, J. Colloid Interface Sci., **2011**, 362, 397-405.

Instrument Development Group

Technical Expertise

The Instrument Development Group (IDG) was created in the middle of the year 2008 in order to bring together the engineers in charge of projects and the engineering and design department with the aim of improving the effectiveness and the visibility of the instrumental projects of the laboratory.

To fulfill its tasks, the IDG exhibits complementary capabilities among which S. Désert covers the management of project, neutron optics and simulations; S. Klimko is an expert in magnetism – from design and simulations to fabrication, polarization of neutron and particularly the neutron spin-echo technique; P. Permingeat is an expert in mechanics and computer-aided design, resistance of materials and an engineer with a wide knowledge; S. Rodrigues concentrates on the management of projects and contracts reporting; and finally P. Lavie is the technician specialist of computer-aided design with expertise in vacuum.

Instruments

The IDG is involved at every instrumental level: it has the technical responsibility and the follow-up of the large instrumental projects within the framework of CAP2015, the contribution or assumption of responsibility in projects known as average (update of an instrument, specific equipment), the implementation of new techniques such as laser tracker alignments and new concrete floor air-pad compatible, the work "in emergency" to repair or adjust experiments, the assistance to the technicians and engineers for drafting and dimensioning, writing specification reports and scheduling, and the reporting of the contracts.

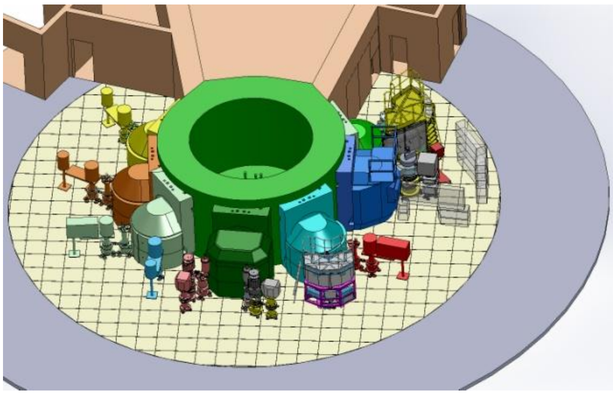

Figure 45: view of the instruments in the reactor hall.

The IDG members are involved in seven instrument projects as technical responsibles: S. Désert for **TPA** (Very Small Angle), **PA20** (Small Angle 2x20 m) and **IMAGINE** (Neutronography), S. Klimko for **Multi-muses** (Spin echo with multi detectors), P. Permingeat for the new **EROS** detector tank and S. Rodrigues for **VIP** (Crystal diffractometer) and **FA#** (Quasielastic) whereas P. Lavie is a common denominator of all these projects.

Members: *S. Désert (head, CEA), S. Klimko (CEA), P. Lavie (CEA), P. Permingeat (CNRS), S. Rodrigues (CEA)*

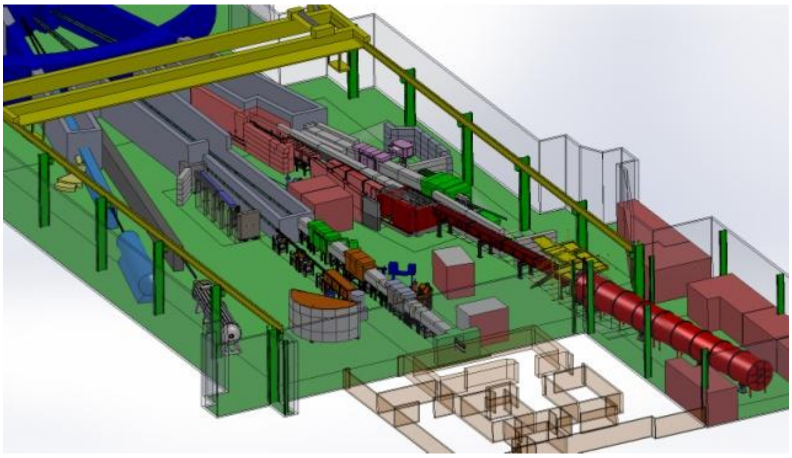

Figure 46: implantation of the new instruments in the guide hall.

Support and Sample Environment Group

Technical Expertise

The Support and Sample Environments Group was created in 2008 as a result of the restructuring of the Technical Support Group and the Office of General Studies. The aim of this group is to provide a clearly identified point of contact for external collaborators to ensure a smooth running of experiments. Historically, the support for experiments was always provided by the scientific group to which the device was attached. For the smooth running of the experiments, the Support Group includes the activities of the mechanical workshop, the service of fluids, gases and other consumables.

Mr. B. Annighöfer is responsible of the development of various devices for sample environment in collaboration with the instrument's responsible or technicians as well as the development and design of high-pressure chambers up to 3GPa. Furthermore he is supervising the production by sub-contracting.

Figure 47: High pressure chambers

Mr. K. Jiguet is responsible of the supplying of expendable goods as cryogenic fluids, gases or others. He is monitoring the recovery of helium as well as the vacuum of the neutron guides. During the remodeling or renovation work, he participates to the complete installation and alignment of the neutron guides, as well as the installation of all the other necessary elements.

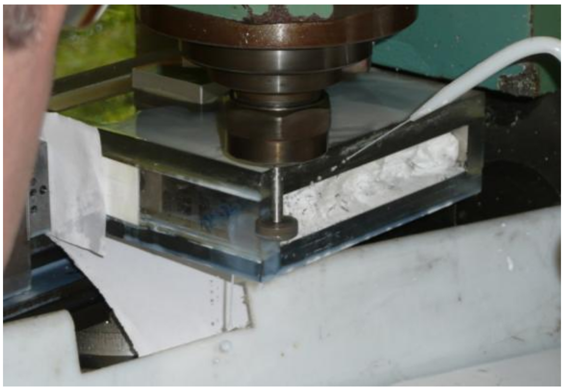

Figure 48: In-house modification of a neutron guide

Mr. O. Tessier is full-time mechanic and responsible for the workshop and the realization of devices for the experiments. He is also training the other technicians on different machine tools.

Collaborations

The Support and Sample Environments Group has participated to the NMI3 FP7 "Sample Environment" Joint Research Activity. The task 21.2 was to develop new high-pressure gas cells for neutron scattering as well as to provide a user-friendly 10kbar gas handling system. These objectives were fully achieved. The project ended in December 2012.

The Support and Sample Environments Group has developed high-pressure vessels which were intended for the use of the French ANR program "BIOSTAB".

A new NMI3-II Joint Research Activity started in 2012 with the title "Tools for Soft and Bio-Materials". The group is working for Task 2 "Kinetics and Dynamics". The aim of this sub-task is to develop improved SANS pressure cells.

Members: *B. Annighöfer (head), CEA), K. Jiguet (CEA), O. Tessier (CEA)*

Electronic Group

Activities

Electronic Group (EG) priority is to ensure operational availability and reliability of the instrument electronics.

The EG designs real time electronic systems providing the interface between driving computers and the different parts of the spectrometers such as mechanics, detectors or sample environment.

Its field is very large since it comprises motion control, detector equipment, data acquisition, up to the design, programming and microprogramming of real time controllers.

EG engineers and technicians are present in all the instrumental projects of the laboratory.

Development strategy

Given the size of the EG against the importance of the facilities, it is compulsory to conceive systems as versatile as possible and able to cope with the diversity of all the spectrometers and their evolution. Therefore EG focuses its efforts on common spectrometer features such as positioning, counting and data acquisition. To control them, the EG designed intelligent and autonomous peripherals connected to the computer thru IEEE488, USB2 and Ethernet.

One of the worthy successes of the team is to have created this instrumentation system ("Daffodil") well accepted at the lab and also sold abroad. Within this frame, the EG can improve peripherals independently from each other and designes cost-effective modules where state of art of technology is applied wisely at its best.

Leading projects

The EG with F.Coneggo, P.Lambert is emphasizing on control/command, mainly axis motion and master controllers thanks to 32 bits microcontrollers within the framework of a distributed architecture. New experiments mechanics can have more than 60 axes: see Figure 49.

Figure 49: opposite part of TPA axis control system.

PSD's systems have undergone a major renewal; VIP and G4.4 spectrometers have been equipped with a new detection equipment designed by the ILL but implemented by F.Prunes. The EG is also involved in the prototyping and testing of new ILL detectors, using wire erosion process, and eventually installed on **PAXY** and **PA20**. The EG will take the full responsibility of reproducing the PSD electronics for **Fa#** and the large angle detectors of **PA20**.

Figure 50: PAXY new detector during its assembly.

Members: *G. Koskas (head, CEA), M. Antoniades (CEA), F. Coneggo (CNRS), W. Josse (CNRS), P.Lambert (CNRS), F.Prunes (CEA)*

Information Technology Group

The Group provides computer support for the LLB neutron spectrometers.

This support includes maintenance and evolution of software developed by the service, the development of new command software for the future spectrometers LLB and a technical expertise on computer hardware, installation and configuration.

Technical Activities

R. Lautie manages the network administration and system of the LLB, A. Laverdunt is in charge of the development of the new command software for the spectrometers and G. Exil oversees the deployment of the new platform software control of LLB spectrometers and the maintenance of software developed in Visual Basic.

Technical Expertise in neutron scattering (instruments)

Developments in Visual Basic

The command software written in Visual Basic6 is deployed on PAXY (G23), PAXE (G54), 6T1, 6T2 and 5C2.

This program allows the control of all instruments present in the experimental area of these spectrometers like engines, furnace, coil magnetic field or wavelength selector. It also offers an acquisition and a 2D visualization for the multi-detectors on PAXY, PAXE and 6T2.

Developments in POOPS

The program POOPS is command software written in VisualC++ which has been developed by previous IT Team in LLB. This program is no longer maintained but the group still offers a technical support of this. It's deployed on EROS (G3B), G41, G61, G62, G52, and 7C2.

No acquisition module for 2D multi-detector has been developed for this program.

Developments in .NET/IronPython

The new command software, called Pingouin, actually developed by the group is written on .NET Microsoft Platform with IronPython for the scripts. This program has a capacity to control all existing LLB spectrometers, and should become the basis of all future instruments under development. Actually, the spectrometers equipped by Pingouin are VIP (5C1), TPA (G5B) and BAROTRON (G56).

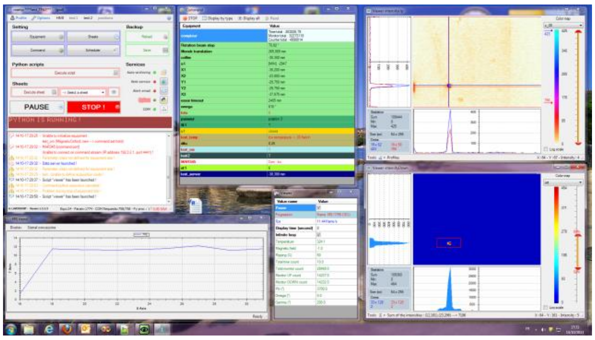

Figure 51: PINGOUIN screenshot

Members: *G. Exil (head, CNRS), R. Lautie (CEA), A. Laverdunt (CEA)*

Theory and Modeling Platform

Scientific Expertise: Theoretical developments and numerical simulations are essential in a large scale facility, as they provide the necessary basis for a successful interpretation of experimental results. While theory and analytical modeling allow unveiling the fundamental concepts and ideas behind experimental observations, they also bring a more direct and quantitative comparison with experiments, when combined with the modern tools of numerical simulation. Predicting and understanding the results of neutron scattering experiments is thus one of the main purposes of this transversal interdisciplinary group.

Neutron scattering covers a wide spectrum of applications in solid state physics, which is reflected in the various activities of the group members: S. Aubry, F. Onufrieva, and P. Pfeuty develop a full-time theoretical research, mostly in non linear physics (wave packet diffusion in a non linear medium and Anderson localization) and in the field of strongly correlated electron systems (superconductivity and other collective effects in high Tc cuprates). More information on these subjects can be found in the "scientific research" section. The other group members are experimental physicists developing and/or using various codes and numerical methods, applied to the various neutron scattering techniques available at LLB. Some examples are given hereafter[107]:

- crystal and magnetic structures in condensed matter (neutron diffraction): Reverse Monte Carlo algorithms for diffuse scattering (chemical disorder), Rietveld refinements programs;
- exotic ground-states and spin dynamics of frustrated and/or low dimensional magnets, role of spin-lattice coupling in multiferroic materials (inelastic neutron scattering, neutron diffraction): linear spin wave theory, random phase approximations, Monte Carlo methods and non-linear classical spin dynamics;
- role of the electron-phonon coupling in iron-based superconductors (inelastic neutron scattering): ab-initio calculations;
- magnetic assembly of interacting nanowires (small angle neutron scattering): numerical investigation of micromagnetic structures;
- dynamical behavior of charged ions in solutions, like electrolytes, either in bulk or in confined geometry (neutron quasi-elastic scattering): Monte Carlo algorithms, molecular dynamics.

In parallel, numerical simulations are intensively used at LLB to optimize spectrometers' performances and to design new instruments (magnetic field maps, neutron guides, etc.).

Technical Equipments: Calculations are mainly performed on a dedicated workstation with 48 cores and 128 Go RAM which was bought recently. The RAM will be extended to 256 Go in the coming months, and another similar workstation will probably be purchased in 2014-2015. Moreover, the capacity to react rapidly and the flexibility offered by such a simple workstation allow computing time to be available to external LLB users requiring numerical support, which is not possible using supercomputers.

[107] G. Kopidakis, S. Komineas, S. Flach, and S. Aubry, *et al.*, PRL **100**, 084103 (2008);
J. Robert, B. Canals, V. Simonet, R. Ballou, PRL **101**, 117207 (2008);
B. Rotenberg, V. Marry, N. Malikova, P. Turq, J. Phys. Cond. Matt. **22**, 284114 (2010);
F. Zighem, T. Maurer, F. Ott, and G. Chaboussant, J. Appl. Physics **109**, 013910 (2011);
T. Maurer, F. Zighem, W. Fang, F. Ott, G. Chaboussant, Y. Soumare, K. Ait Atmane, J.-Y. Piquemal, and G. Viau, J. Appl. Phys. **110**, 123924 (2011);
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S. Petit, P. Bonville, J. Robert, C. Decorse, and I. Mirebeau, PRB **86**, 174403 (2012);
N. Malikova, S. Cebasek, V. Glenisson, D. Bhowmik, G. Carrot, and V. Vlachy, Phys. Chem. Chem. Phys. **14**, 12898 (2012).

Chemistry Platform

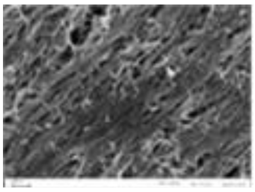
Scientific Expertise: Syntheses and characterizations constitute an integral part of research at LLB. This aspect is especially important for complex soft and hard matter projects where high-quality pure samples are synthesized partly or completely in our own laboratories. An established theme includes polymer-functionalized nanoparticles: synthesis and formulation of nanocomposites where endgroup-functionalized polymers (hydrogenated and deuterated) are synthesized, then grafted onto oxide nanoparticles for compatibility and controlled dispersion in polymeric matrices for optimal mechanical reinforcement; synthesis of thermosensitive polymers and gold nanoparticles by one-pot grafting-to process to control optical properties. Other examples include (hydrogenated and deuterated) liquid crystals and ionic liquids, and inorganic porous media for catalysis. The Chemistry Platform oversees the laboratory environment for the elaboration of these home-tailored samples. Its facilities and functions include:

- Synthesis in automated safety-equipped chemical hoods
- Analytical equipment for physical and chemical characterizations
- Maintenance of good laboratory practices for the health and safety of employees
- Separate inventoried ventilated stockroom for all chemicals

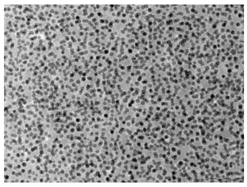
Technical Equipments: The major equipments include: differential scanning calorimeter, thermogravimetric analyzer, shear rheometer, uniaxial stretch machine, Langmuir trough, nitrogen volumetric absorption apparatus, instrument for dielectric constant and conductivity measurements, cryostatic FTIR spectrometer. Access to these facilities by external users may be granted only under supervision of a permanent member of LLB.

Nanocomposites

γ-Fe_2O_3 nanoparticles aligned in polystyrene matrix[1]



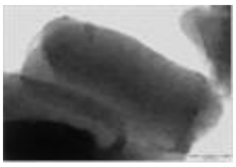
Cobalt nanowires aligned under magnetic field[2]



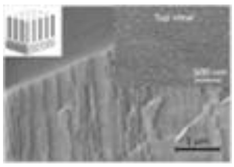
Polystyrene-graft SiO_2 in polystyrene matrix[3]

Silica-reinforced rubber matrix[4]

Porous Media



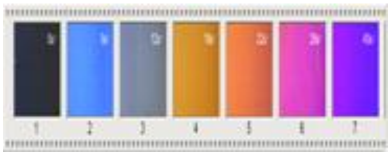
SiO_2 catalytic mini-reactors[5]



Oriented anodic aluminum oxide[6]

Optical Materials

Thermosensitive plasmonic nanoparticles[7]



Shear-induced birefringent liquid crystals[8]

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Biology Platform

Scientific Expertise: The platform expertise is mainly devoted to the production of proteins, nucleic acids (NA, *i.e.* DNA and RNA), membranes, and whole cells for analyses of bio-macromolecular structures and assemblies. Isotopic labeled samples are also prepared for neutron experiments (deuterated proteins or NA, H/D exchange, lyophilization, powder hydration). Indeed, one key-element to study structure and dynamics of biomacromolecules by neutron scattering consists in the use of contrast matching by deuterated labeling. In particular, we study how macromolecular crowding can stabilize the conformational structure of proteins and NA. Up to now, we have used deuterated crowding agents (*e.g.* D-PEG) to mimic and match the cellular environment of hydrogenated proteins[109]. We now focus on the use of deuterated bacterial compounds for this goal. Therefore, we grow bacterial strains in deuterated growth media to produce both deuterated bacterial extracts and to purify deuterated biomacromolecules (Figure 52).

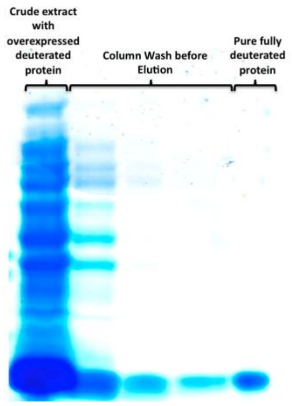

Figure 52. Overexpression and purification of a fully deuterated protein, the bacterial nucleoid protein HU. 50 mg of deuterated HU have been prepared on the platform in 2013.

We also use deuterated proteins to study by neutron scattering the fundamental role of hydration water, now widely recognized to have a role in protein flexibility, especially in the existence of the protein ‘‘dynamical transition’’ at ~220 K. We successfully produced in collaboration with another lab in the CEA a deuterated protein from cyanobacteria (C-phycocyanin) and purified at the LLB a large amount of it (~120 mg). We used powders of hydrated hydrogenated or deuterated form of this protein to probe, by elastic incoherent neutron scattering, protein dynamics and hydration water dynamics at different time- and length-scales[110].

Technical Equipments: The biology platform provides also, to both LLB and external scientists, facilities for their researches: molecular cloning, production of mutants by directed mutagenesis, chromatography (FPLC), electrophoresis and usual lab equipments (-20 and -80°C freezers, cold room, centrifuges, etc.) are routinely used to prepare biological samples; more than 300 mg of purified proteins have for instance been prepared on the platform in 2013. Lipid membranes (supported bilayers or monolayers, liposomes) are also produced for voltage-clamp and neutron reflectivity measurements especially[111]. Moreover, samples can be characterized before neutron scattering experiments by electronic absorption (UV/visible), fluorescence and FTIR spectroscopies (Figure 53), light scattering (SLS, DLS), *gel permeation chromatography* (GPC, FPLC), and laser scanning microscopy (FRAP, FCS).

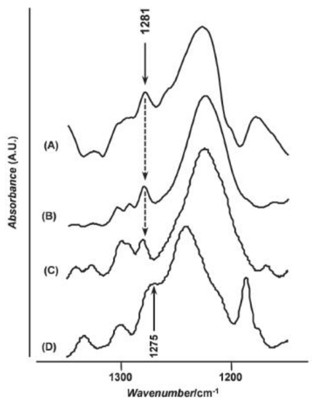

Figure 53: FTIR spectra of a complex between the bacterial protein Hfq and DNA[112].

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Administration Group & health and safety

Administration

The administration group takes care of the management of the unit. It manages the budget, the contracts, human resources... These tasks are performed in close relationship with the corresponding services of the CEA (IRAMIS institute) and the CNRS (regional delegation DR4). It has also in charge communications actions (web site, reports, publications…).

The general secretariat is assumed by A. Verdier. Human resources administration, records relating to permanent staff and non-permanent (Ph.D., Postdocs, trainees) are centralized at the secretariat and are treated in relation with relevant departments at IRAMIS or regional delegation CNRS, depending on the employer. Two correspondents are in charge of the professional training, C. Doira for CEA staff and A. Brulet for CNRS staff.

Order requests are validated at the management level and prepared by the secretariat. O. Sineau is in charge of following mission requests, all the orders taken on CNRS budget and the administrative management of the European contract for access to large facilities (NMI3). C. Doira is in charge of editing the orders taken on the CEA budget. A few specifics records are monitored at the administrative level: contracts by J.-P. Visticot, in relation with CEA and CNRS services; fluid consumption by K. Jiguet-Covex; purchase orders by A. Menelle…

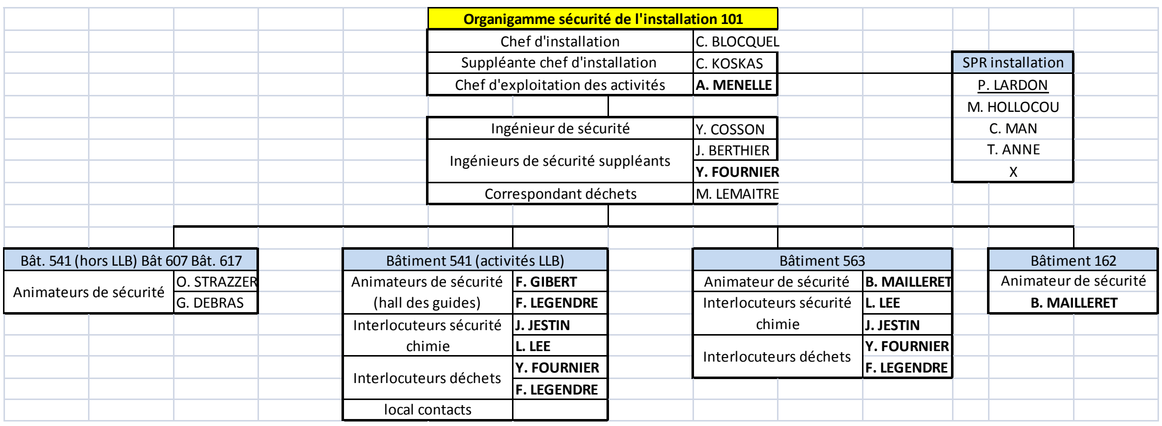
For the organization of the access to the facility, A. Touze is responsible for providing access badges, dosimeters, some logistic, housing and reimbursement to visitors. M.H. Mathon is in charge of the organization of the selection committees that distribute beam time to users.

Finally, the group is also in charge of some communication tasks. C. Doira is in charge of the edition of communication materials (brochures, posters, reports). F. Porcher is responsible for the annual report and A. Menelle of the web site. Organizations of visits are also managed by the administrative group as well as some schools, meetings and training sessions (i.e. Fan, NMI3 …).

Members : *C. Alba-Simionesco (CNRS), J-P Visticot (CEA), A. Menelle (CEA), C. Doira (CEA), A. Touze (CNRS), O. Sineau (CNRS), A. Verdier(CEA).*

Health and Safety

Every equipment and building of the LLB and of the Orphée reactor are grouped within a single entity called the INB101 (Installation Nucléaire de Base #101). The director of the Orphée reactor is the safety responsible within the whole entity, and the rules of security/safety follow the regulation of CEA which is the host of the installation. LLB members are directly involved in the organization of safety and work in close relationship with the reactor staff. There is a single security organization with people of LLB and ORPHEE.



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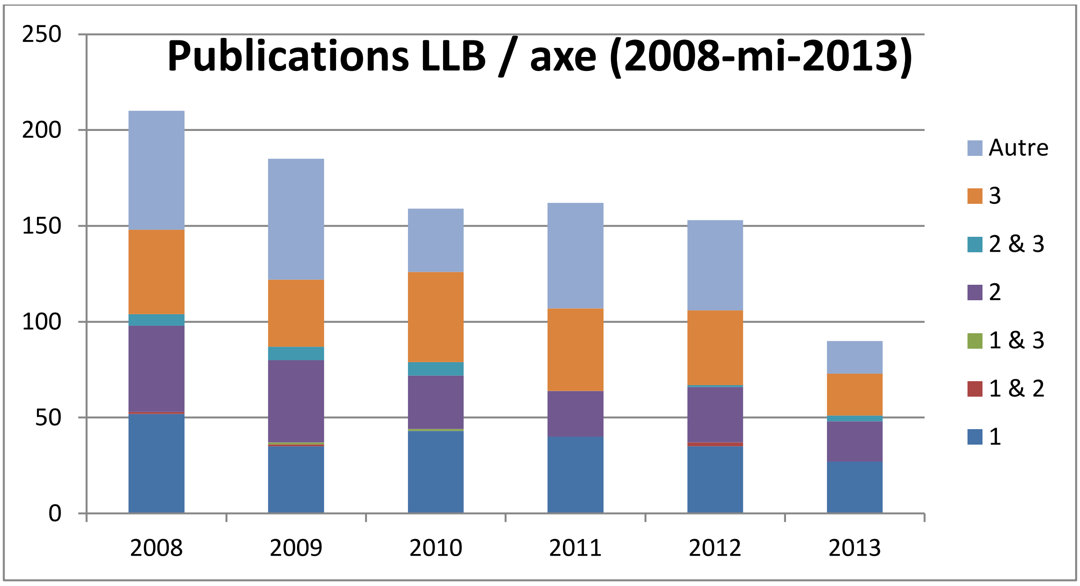

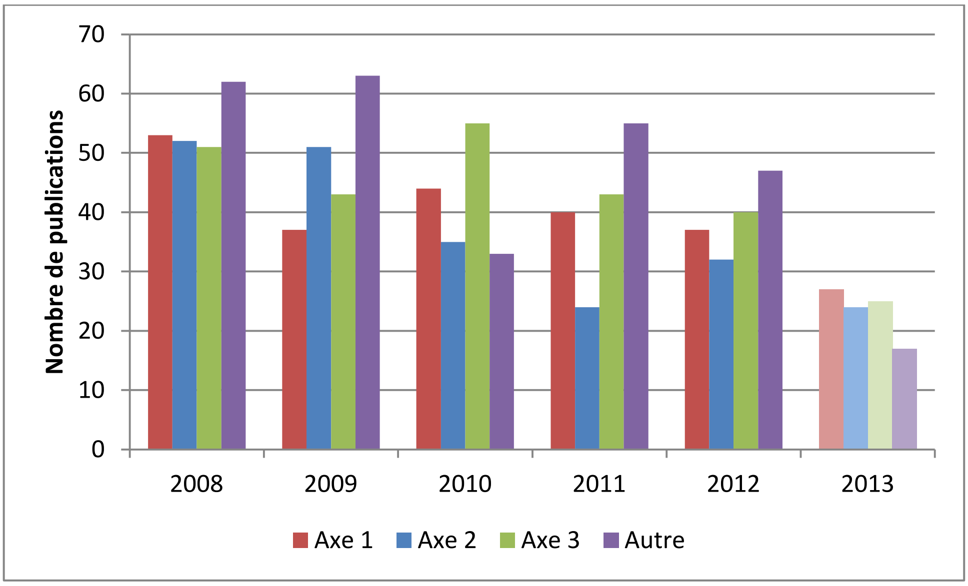

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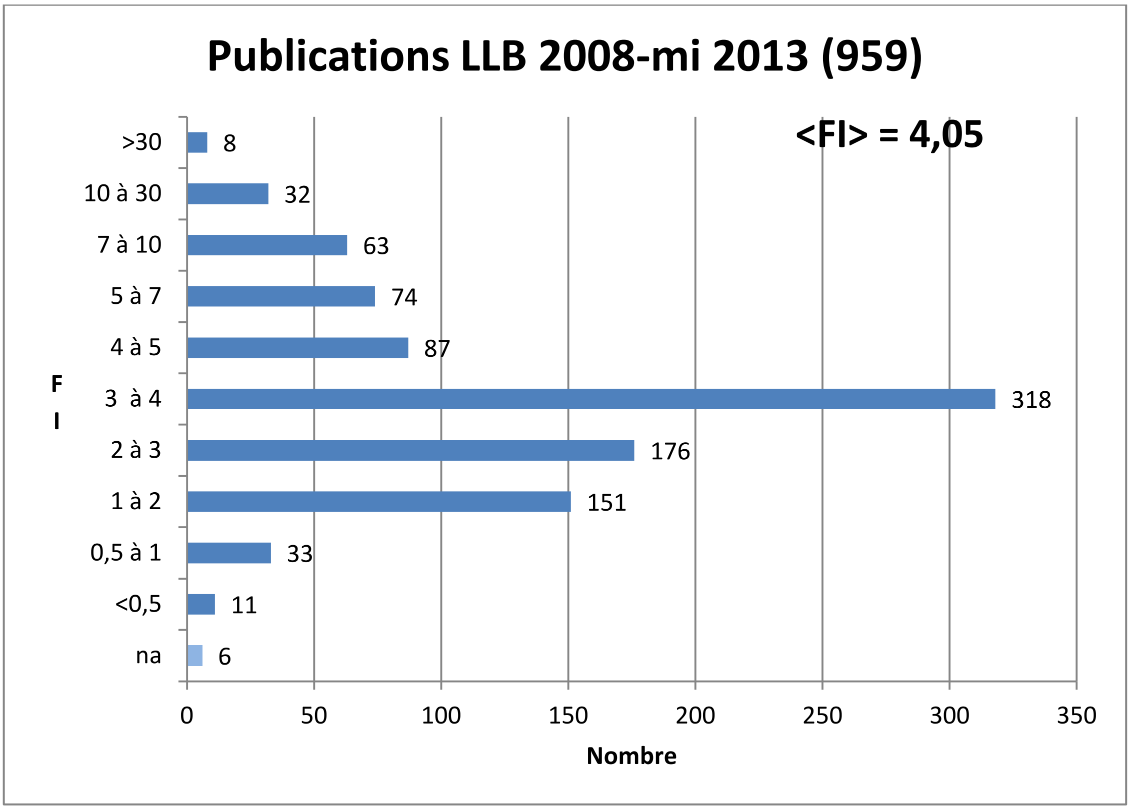

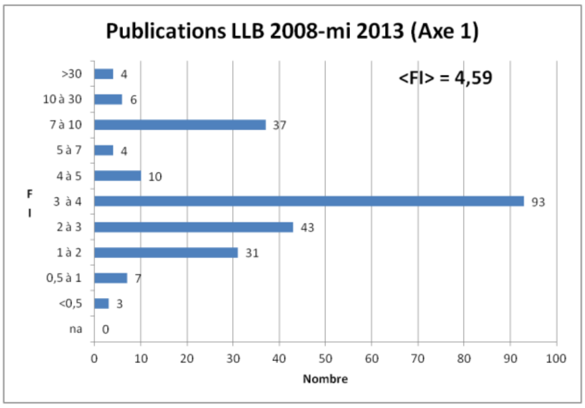

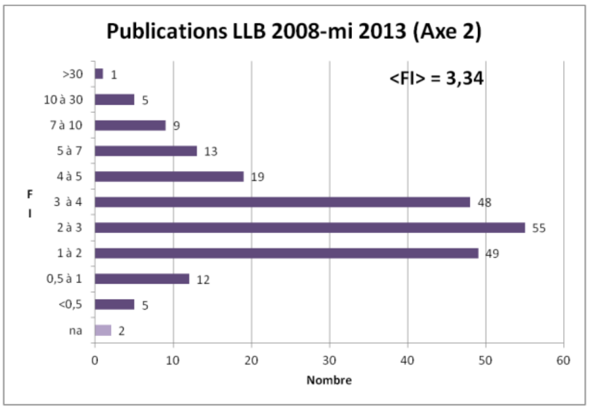

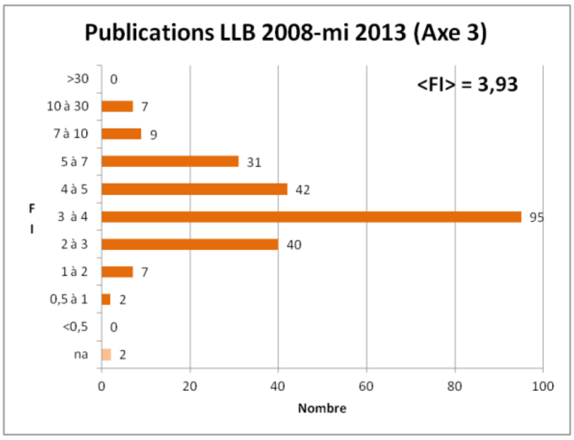

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