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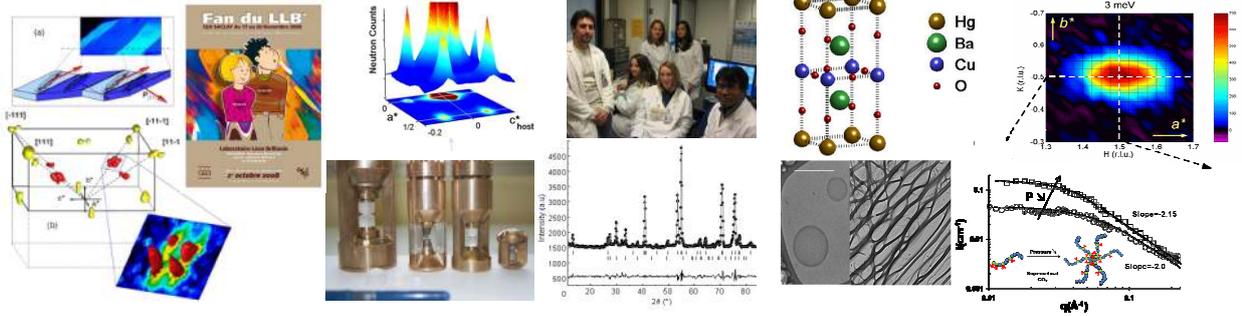


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# LABORATOIRE LEON BRILLOUIN

## ANNUAL REPORT





**The Laboratoire Léon Brillouin  
thanks all the people  
who contributed  
to the production of this report**

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# Foreword

The Laboratoire Léon Brillouin is a French research infrastructure supported jointly by *the Commissariat à l'Énergie Atomique et aux Énergies Alternatives* (CEA) and the Centre National de la Recherche Scientifique (CNRS); it constructs and operates spectrometers around Orphee, a 14MW reactor operated by the CEA since 1980. Its exceptional situation in the south west of Paris, in the scientific centre of Saclay, nearby faculties, engineers schools and other large scales facilities such the synchrotron Soleil, promotes contacts, discussions and stimulates new collaborations. As a national facility, its management of beam time is quite flexible allowing more tests, thoughts and discussions between beginners and experts, exploring new areas or experiment preparation, and access to industrial partners.

The year 2009 saw the beginning of a number of projects and new thinking which allow us to start the coming decade with enthusiasm and a constructive attitude. Each of these operations corresponds in various ways to the three goals of this large scale facility LLB-Orphée : research, service and training. If we were to sum up our activities in a few numbers, we would say that this laboratory is composed of 112 people, with about 30% of students, post-docs and visitors, carrying out more than 400 experiments a year with 500 visitors, 33% of whom are from foreign countries (mostly supported by the European Access Program of NMI3), on 22 spectrometers which will be renewed in the CAP2015 project. These experiments were performed thanks to the very great competence of the scientific and technical teams, their unity and their ambition in research. Thanks are also due to the technical teams at Orphée, under the guidance of X. Bravo, which made it possible to function with 194 days of beam time. The harmony between these teams encouraged us to choose a new logo, with which each person can identify. The scientific results obtained were printed in 190 publications in refereed scientific journals per year in 2008 and in 2009, more than 70% of national production being associated with the LLB according to an advanced publishing survey which we have undertaken.

We have brought our selection committees, now numbering five, up to date according to the scientific community's demands. New directions in research are at the center of the instrumental development program CAP2015 at LLB. This program provides for the modernization or reconstruction of nearly half the laboratory's instruments by 2015. This will give LLB an excellent quality of instrumentation at the disposal of the French and European communities in the following years.

Aside its function of large scale facility, the LLB carries out its own scientific research program summarized in three main axes: hard matter and magnetism, soft matter and biophysics, materials and nano-sciences. Internally, it should also be noted that our researchers have achieved a high level of success with their national and international projects. We have hired new engineers and technicians to replace those who retired in 2008, we have added a young researcher in the CNRS competitive exam, and teacher-researchers requesting attachment to our unit, emphasizing our involvement in training and education. Training sessions organized by LLB (such as the FAN) and those in which we participate (JDN, HERCULES) are completed by practical experience for students during their Master degrees, or from engineering schools. We plan to offer this opportunity to a larger number of students in the future, in collaboration with the synchrotron Soleil. The year 2010 will see some major events strengthening of our local, national and international collaborations and the visit of the scientific and technical advisory committees of experts nominated by the French safety authority (ASN) settling and ensuring the activity of our reactor for the next ten years.

We present here some of the technical and scientific achievements of 2009 and hope you will enjoy them.

*Christiane Alba-Simionesco*

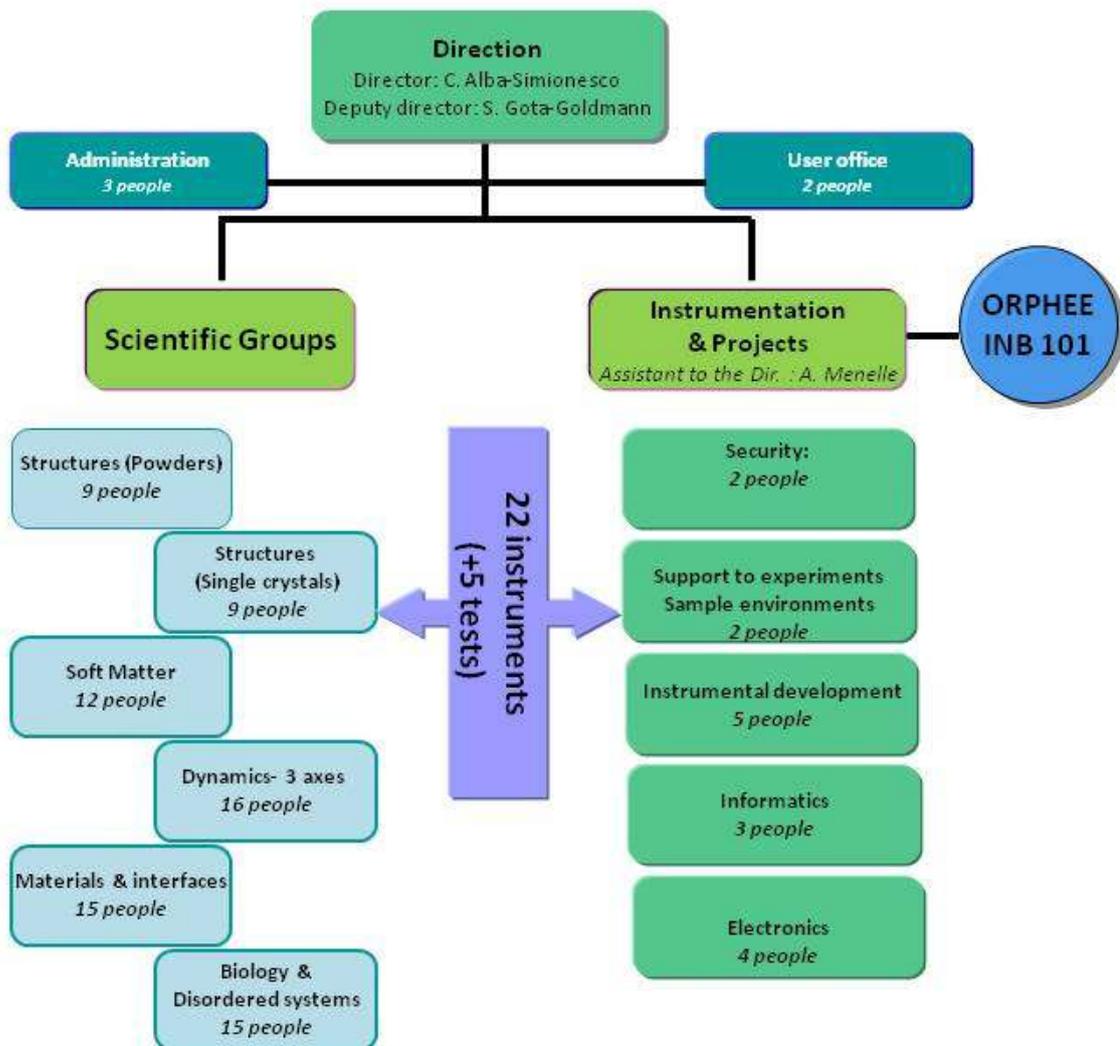
*Susana Gota-Goldmann*

*Alain Menelle*

# ORGANISATION OF LLB (June 2009)



## Flow chart of the organisation of LLB



# SCIENTIFIC HIGHLIGHTS

## Condensed Matter

- Unusual magnetic order in the pseudogap region of the superconductor  $\text{HgBa}_2\text{CuO}_{4+\delta}$
- Electronic liquid crystal state in high temperature superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_{6.45}$
- Magnetoelectric coupling in  $\text{BiFeO}_3$  single crystals and thin films
- Superconducting pairing and electronic anomalies induced by the spin collective mode in HTCSuperconductors

## Structure and Phase transitions

- Influence of the alteration layer morphology on the silicate glass corrosion mechanism : role of Ca and Zr
- Spin ladder iron oxide :  $\text{Sr}_3\text{Fe}_2\text{O}_5$
- Hidden degrees of freedom in aperiodic crystals
- Ising versus XY Anisotropy in Frustrated  $\text{R}_2\text{Ti}_2\text{O}_7$  Compounds as “Seen” by Polarized Neutrons

## Soft Matter

- Control of roughness at interfaces and the impact on charge mobility in all-polymer field-effect transistors.
- Anisotropic reinforcement of nanocomposites tuned by by magnetic orientation of the filler network.
- Slooking at micellisation of gradient copolymers in supercritical carbon dioxide.
- Self assembly of fatty acids in aqueous solutions.

## Life Sciences and Biology

- Influence of macromolecular crowding on protein stability.
- Coupling of laser excitation and inelastic neutron scattering measurement.
- Neutrons and water structure: the heavy water bridge.

## Publications



## UNUSUAL MAGNETIC ORDER IN THE PSEUDOGAP REGION OF THE SUPERCONDUCTOR $\text{HgBa}_2\text{CuO}_{4+\delta}$

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The pseudogap region of the phase diagram is an important unsolved puzzle in the field of high-transition-temperature ( $T_c$ ) superconductivity, characterized by anomalous physical properties below a certain temperature,  $T^*$ [1]. In contrast to the superconducting temperature  $T_c$  which exhibits a dome-like shape, the pseudogap phase is observed only at low doping in the underdoped region of the cuprates phase diagram. There are open questions about the number of distinct phases and the possible presence of a quantum-critical point underneath the superconducting dome. In particular, it has been proposed [1,2] that the pseudogap is characterized by an ‘hidden’ order breaking time reversal symmetry. In addition to this, a separate but perhaps more important issue remains to be settled in the cuprates: are fluctuations of a such ‘hidden’ order, associated with a quantum phase transition, capable of providing the pairing glue and explaining the anomalous normal-state properties ?

Recent pioneering polarized neutron diffraction work [3], carried out at the LLB, has established that in underdoped  $\text{YBa}_2\text{Cu}_3\text{O}_{6+d}$  (YBCO) a new form of order sets in below  $T^*$ . This “hidden” magnetic order, presumably due to charge currents circulating around the Cu-O plaquettes[2], appears to be in competition with superconductivity and is found to disappear at higher doping.

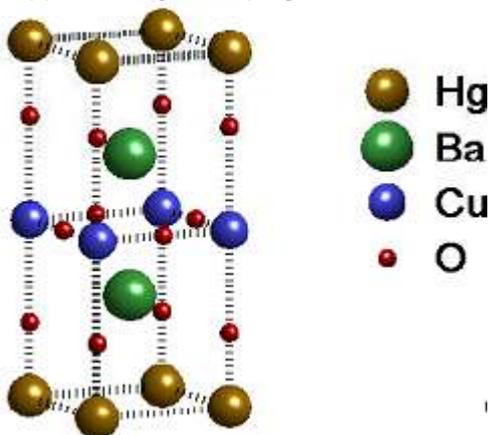


Fig. 1: Crystal structure of  $\text{HgBa}_2\text{CuO}_{4+\delta}$

While the high- $T_c$  superconductors all contain the Cu-O plane as a basic building block, they also exhibit significant structural and chemical differences. YBCO is a double Cu-O layer compound, with orthorhombic symmetry and Cu-O

chains in addition to the Cu-O planes, and a maximum  $T_c$  of about 93 K. A structurally simpler material, with higher  $T_c$ , is the Hg-based compound,  $\text{HgBa}_2\text{CuO}_{4+\delta}$  (Hg1201) (Figure 1): it contains only one Cu-O layer per unit cell, has a tetragonal crystal structure, a wide spacing between the Cu-O planes, and a very high  $T_c$  of 98 K at optimal doping. Quite generally, the Hg-based superconductors are known to have the highest values of  $T_c$  among single-layer, double-layer, etc. cuprates.

We have used polarized neutron diffraction on the triple axis spectrometer 4F1 at the ORPHÉE reactor of the Laboratoire Léon Brillouin [4] to demonstrate in a serie of underdoped crystals for the model superconductor Hg1201 that the characteristic temperature  $T^*$  marks the onset of an unusual magnetic order. This was possible by the recent success in growing gram-sized Hg1201 single crystals at Stanford University [4] and the high polarization efficiency of the spectrometer.

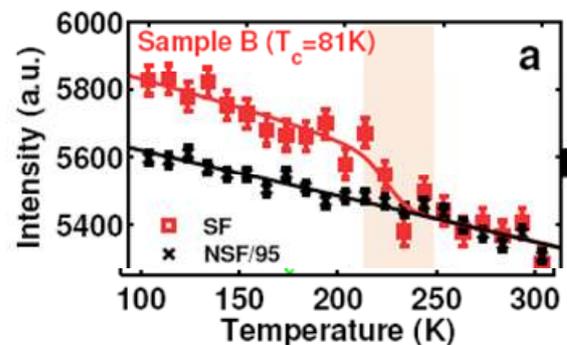


Fig. 2: Spin-flip (SF) and non-spin-flip (NSF) intensity of Bragg peak (101) in one underdoped sample of Hg1201 (from [4]). A magnetic signal is observed below 250 K.

Figure 2 demonstrates the existence of a magnetic component in the spin-flip (SF) geometry for an underdoped sample. Due to the relatively strong intensity from unavoidable nuclear Bragg peak leakage in the SF geometry, the measurement is done at a weak nuclear reflection  $\mathbf{Q}=(101)$ . The neutron polarization was parallel to the momentum transfer,  $\mathbf{P} \parallel \mathbf{Q}$ , a geometry in which all magnetic scattering occurs in the SF channel. The linear slope of the nuclear scattering observed in the non-spin-flip channel can be accounted for by the Debye-Waller factor.

As expected, due to non-zero leakage in the SF geometry, the SF data exhibit a linear nuclear scattering contribution as well. However, the SF data furthermore exhibit an additional component below  $T_{\text{mag}} \sim 250$  K, which we conclude to be of magnetic origin [4]

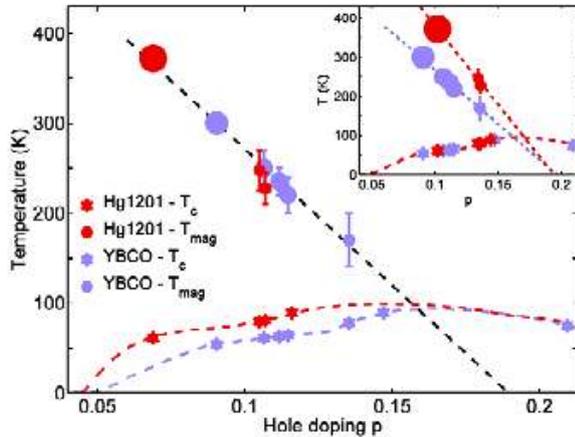


Fig.3: Universal pseudogap phase diagram of Hg1201 and YBCO (from [4]). The temperature  $T_{\text{mag}}$  extracted from the neutron data [2,4] reproduces the doping behaviour of  $T^*$ . The combined data extrapolate to  $T_{\text{mag}} = 0$  K at  $p_c = 0.19$ .

The onset of magnetic order in YBCO has been associated with the pseudogap temperature  $T^*$ , where  $T^*$  is the characteristic temperature obtained from resistivity measurements. Below  $T^*$ , the resistivity is sub linear with temperature. Interestingly, that deviation from linear resistivity scales with the magnetic intensity. Resistivity data for a separate small crystal of Hg1201 with the same  $T_c$  (81 K) follow quite strongly the same trends, suggesting that the observed magnetic and charge properties share the same physical origin.

Together with the recent results for YBCO[2], this observation constitutes a demonstration of the universal existence of such a state as it is shown in the figure 2 [4]. (i) In both cases, the order preserves the translational symmetry of the underlying lattice, unlike conventional antiferromagnetism, which occurs at  $(1/2, 1/2, 0)$  and equivalent reflections; (ii) The magnetic scattering develops below a temperature which coincides with the pseudogap temperature  $T^*$  determined from transport measurements, suggesting that the novel order involves both magnetic and charge degrees of freedom; (iii) The magnetic signal is of comparable strength for the two compounds, it is strongest in very underdoped samples, and the transition appears to be continuous.

The findings appear to rule out theories that regard  $T^*$  as a crossover temperature rather than a phase transition temperature. Instead, the experiments for Hg1201 and YBCO are qualitatively consistent with a true magnetic phase transition, suggesting a novel

state of matter with broken time-reversal symmetry having the symmetry of two counter-circulating charge current loops per  $\text{CuO}_2$  plaquette [3] (Figure 4, left). More specifically, the results suggest a variant of the previously proposed charge current-loop order that involves apical oxygen orbitals [5] (figure 4, right).

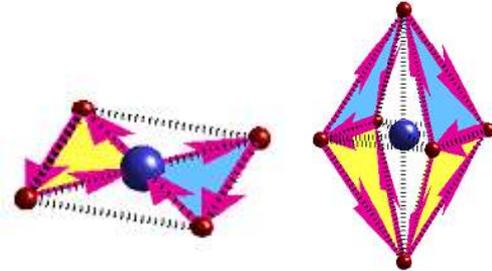


Fig. 4: Simplified schematic illustrations of two circulating current ordered states that break time-reversal symmetry, but preserve translational symmetry. Left) Planar circulating currents phase originally proposed by C.M. Varma [3]. Right) circulating currents involving apical oxygen [5].

The maximum  $T_c$  occurs close to where the experiment fails to discern a magnetic signal. It appears likely that the order competes with the superconductivity with the notion that many of the unusual properties arise from the presence of a quantum-critical point [1,3] at  $p_c = 0.19$  (figure 3). One intriguing possibility is that the fluctuations associated with an underlying quantum critical point are directly responsible for the appearance of superconductivity and the unusual normal state properties, such as the linear resistivity found up to remarkably high temperatures.

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## ELECTRONIC LIQUID CRYSTAL STATE IN HIGH TEMPERATURE SUPERCONDUCTOR $\text{YBa}_2\text{Cu}_3\text{O}_{6.45}$

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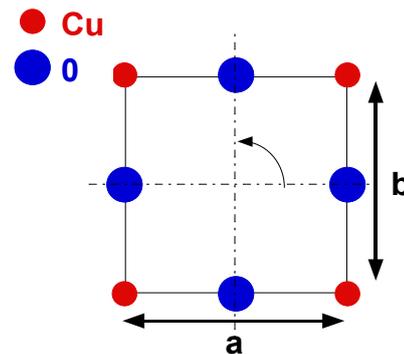
The strongly interacting conduction electrons can be accurately represented as a gas of weakly interacting electron-like excitations. This description, known as Fermi liquid theory, works for many metallic systems. However, over the past two decades, new types of metallic materials with strongly correlated electrons have been discovered that do not fit this standard description. The list includes the superconducting copper oxides and many other materials.

Fermi liquid behavior occurs where the kinetic energy of the electron fluid dominates the particle dynamics. In contrast, where the Coulomb interactions are dominant, electrons are known to form an insulating electron crystal. The electron fluids in strongly correlated metals lie in an intermediate range where neither of these energies is dominant. An analogy can be made with complex classical fluids. There is generally a gas phase at high temperatures, where entropy dominates, and a crystalline solid phase at low temperatures, where interaction energies dominate. In many cases there is an intermediate liquid phase in which interactions and entropic considerations must be treated on an equal footing. In some cases, there are additional “liquid crystalline” phases (e.g., nematic and smectic) that can flow like a liquid but exhibit patterns of broken symmetry that are somewhat like those of a solid. At the end of the 90’s, it has been proposed that strong correlations can induce electronic liquid crystal phases. These phases are quantum fluid (conducting) states with a pattern of spontaneous symmetry breaking that is intermediate between those of the simple fluid and an electron crystal.

The nematic metal is named in analogy with a classical uniaxial nematic liquid crystal [1]. One can think of an electron nematic as a partially (quantum) melted version of an anisotropic electron crystal. For instance, correlated materials can exhibit “stripe order” in which the electrons form a striped pattern that breaks translational symmetry and chooses a preferred axis in the crystal. One can imagine a nearby phase in which quantum fluctuations melt the stripe order but the preferred direction of the stripes remains as a memory of the proximate ordered phase. An alternative approach to the metallic nematic phase considers the effect of increasingly strong interactions in Fermi liquids. It has long been

known that a Fermi liquid becomes thermodynamically unstable if certain interactions are sufficiently strongly attractive. It was shown that in some cases, rotational invariance is spontaneously broken while translation symmetry is preserved.

*High temperature copper oxide superconductors are quasi-two dimensional material, made of the stacking of  $\text{CuO}_2$  planes. These planes can be described as the juxtaposition of squared  $\text{CuO}_2$  plaquettes (see below). Physicals properties should therefore be invariant under a rotation of  $90^\circ$ .*



In the high-transition-temperature superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_{6.45}$  [2], magnetic excitations, that develop around the antiferromagnetic wave vector  $(0.5, 0.5)$ , exhibit a net quasi-1D anisotropy at low energy and low temperature (Fig.A). The low magnetic excitation spectrum is characterized by a set of two incommensurate spin excitation along  $a^*$  direction at wave vectors  $(0.5 \pm \delta, 0.5)$  (Fig.B-C). The one-dimensional, incommensurate modulation of the spin system appears spontaneously upon cooling below  $\sim 150$  K (Fig. D), whereas static magnetic order is absent above 2 kelvin. The evolution of this modulation with temperature and doping parallels that of the in-plane anisotropy of the resistivity (see insert in Fig.D). Our observations highlight the existence of an electronic nematic phase that is stable over a wide temperature range. The results suggest that soft spin fluctuations are a microscopic route toward electronic liquid crystals and that nematic order can coexist with high-temperature superconductivity in underdoped copper oxides.

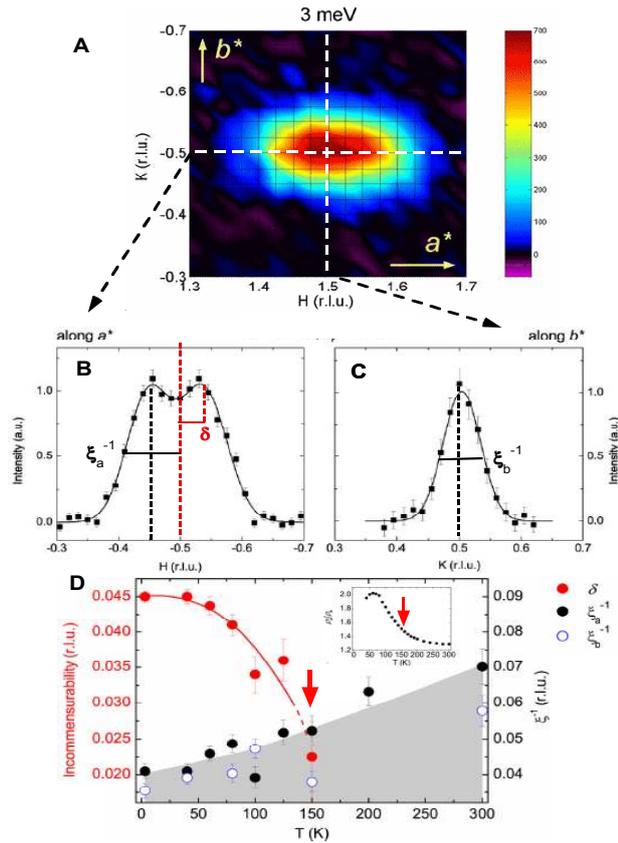


Fig.: A) Intensity map of the quasi-1D incommensurate at 3 meV and 5 K. B-C) Constant energy scans at 3 meV along directions  $a^*$  and  $b^*$ . D) Temperature dependencies of the incommensurability parameter  $\delta$  and of the magnetic peak widths along  $a^*$  and  $b^*$ . The insert shows the temperature dependence of the  $a$ - $b$  anisotropy of resistivity  $\rho_a/\rho_b$

The energy and momentum dependence of the spin excitations we have observed helps to develop a microscopic description of the nematic state, and to discriminate between different theoretical descriptions of the coupling between spin and charge excitations in the cuprates. Incommensurate peaks in the magnetic neutron scattering pattern can arise either from a longitudinal modulation, where the magnetic moments are collinear but their amplitude is spatially modulated, or from a transverse modulation, where the moment direction varies but the amplitude remains constant. Our data are compatible with slow fluctuations characteristic of either type of modulation. Spin-amplitude modulated states naturally go along with a modulation of the charge carrier density, and the carrier mobilities along and perpendicular to the modulation axis are generally expected to be different [3]. It has also been shown that a transverse modulation with spiral spin correlations can lead to anisotropic hopping transport in weakly doped cuprates with diverging low-temperature resistivity [4]. Further work is required to assess whether this mechanism can be generalized to metallic electron systems such as the one in  $\text{YBa}_2\text{Cu}_3\text{O}_{6.45}$ .

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## MAGNETOELECTRIC COUPLING IN BIFEO<sub>3</sub> SINGLE CRYSTALS AND THIN FILMS.

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BiFeO<sub>3</sub> is a multiferroic materials in which ferroelectric and anti-ferromagnetic orders coexist well above room temperature ( $T_N=643$  K,  $T_C=1093$  K), with a high polarization (over  $100 \mu\text{C}/\text{cm}^2$  [1]). We have shown at the LLB by neutron diffraction that these two order parameters interact and that the magnetization of the material can be modified by the application of an electric field. This opens the way towards the implementation of this material in spintronic devices in which magnetization could be controlled by a small electrical voltage rather than by currents or magnetic fields.

Although magnetoelectric materials have been known for decades, no *direct* proof of the coupling between the two co-existing physical effects had been reported in the literature. We have demonstrated this magneto-electric coupling by neutron diffraction on BiFeO<sub>3</sub> single crystals. One key to success was the exceptional quality of the high purity single crystals which are magnetically and electrically single domain.

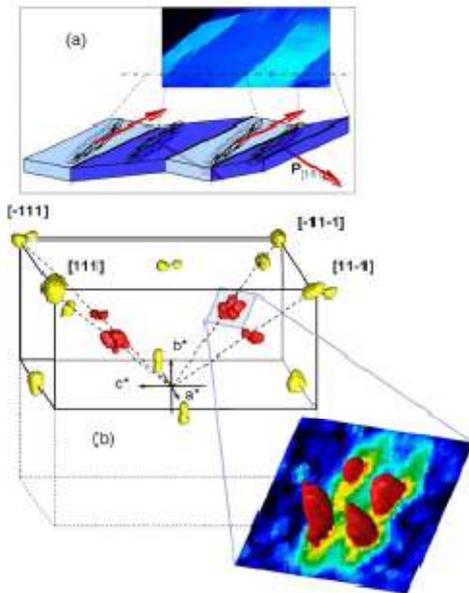


Fig. 1: Effect of an electric field on a BiFeO<sub>3</sub> single crystal. (a) electric domains observed with polarized optical microscopy. (b) mapping of the neutron intensity in the reciprocal space. Two sets of splitting appear for the nuclear intensity (yellow spots): one because of the presence of two rhombohedral distortions along  $[111]$  and  $[-1-1]$ , the other because of the buckling of the crystal induced by twinning.

The magnetic peaks (red spots) are further split because of the cycloids.

The first step of this work consisted in a verification of the magnetic structure of BiFeO<sub>3</sub> (several structures were consistent with powder diffraction data). Our single crystal results confirmed [2] that the spins of the Fe<sup>3+</sup> ions lie in the  $(111)$  planes (G-type antiferromagnet) forming a circular cycloid with a long period of 62 nm.

In a second step, an electric field was applied on the crystal which was in a single ferroelectric domain state with the polarisation along  $[111]$ . The electric field was applied along the  $(001)$  and switched the sample into a multidomain ferroelectric state (Figure 1 (a)). Using neutron diffraction we showed that the propagation vector was the same as in the virgin state, but that the spins were then lying in two different planes. These can be accounted for by considering that 55% of the crystal volume has switched its polarization by  $71^\circ$ , and induced a tilt of the rotation plane of the Fe moments, thus inducing a spin flop of the antiferromagnetic sublattice (see Figure 2). These experiments evidence that although a macroscopic linear magnetoelectric effect is forbidden by symmetry, a rather strong coupling between M and P still exists at the atomic level via the Dzyaloshinskii-Moriya interaction.

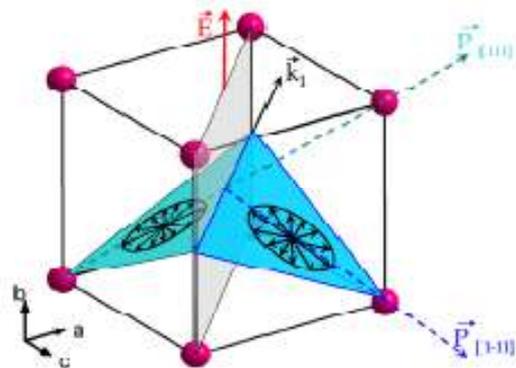


Fig. 2: Schematics of the planes of spin rotations and cycloids propagation vector for the two polarization domains separated by a domain wall (in light gray).

Integration of multiferroics into spintronic devices requires growing thin films. The group of Agnès Barthélémy at the UMR CNRS/Thales managed to grow high quality single phase BiFeO<sub>3</sub> epitaxial films by pulsed laser deposition. The first issue was to

determine if the magnetic structure of thin films. Neutron diffraction has shown that the cycloidal order is lost in thin films [3] (see Figure 3a). This change in the magnetic order affects the magneto-electric coupling since it allows a linear effect

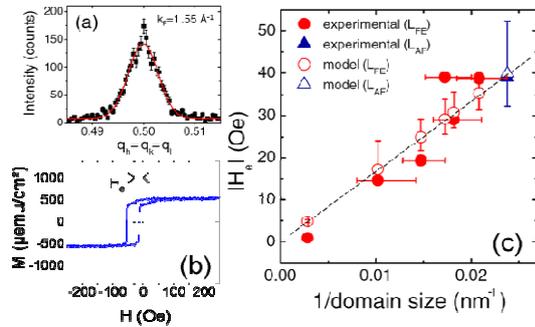


Fig. 3: (a)  $(1/2\ 1/2\ 1/2)$  magnetic Bragg peak observed with neutron diffraction on a 70 nm thick BiFeO<sub>3</sub> (001) thin film. (b) Hysteresis loop of a CoFeB/BiFeO<sub>3</sub>(70nm) bilayer. The exchange field is noted  $H_e$ . (c) Exchange field as a function of the inverse of domain size. The data are in quantitative agreement with the Malozemoff random field model.

The next step of the study has consisted in investigating the interaction of BiFeO<sub>3</sub> thin film coupled to a ferromagnetic layer (CoFeB in our case). In these heterostructures, both layers are coupled via the exchange bias mechanism [4] which was demonstrated by the shift of the hysteresis loops (Figure 3b). The Malozemoff model of the exchange bias states that the atomic scale disorder at the interface between ferro- and antiferromagnet results in domains within the antiferromagnetic layer. In this framework, the exchange field  $H_e$  is expected to be inversely proportional to the antiferromagnetic domain size (see Figure 3c). The anti-ferromagnetic domain size  $L_{AF}$  was extracted from neutron diffraction (measured on 4F1) and compared to the ferroelectric domains size  $L_{FE}$  measured by piezoresponse force microscopy. Both sizes have been shown to be identical which suggests that the magneto-electric coupling still exists in these thin film structures. The exchange field was also shown to be inversely proportional to the domain size as predicted.

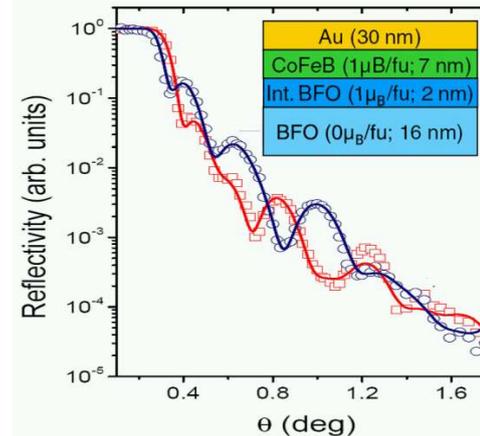


Fig.4: Polarized neutron reflectivity on a CoFeB/BiFeO<sub>3</sub> heterostructure showing the presence of an induced magnetization in the interfacial BFO layer.

Furthermore, the analysis of the data with Malozemoff's model suggests that a net magnetization should be observed in BiFeO<sub>3</sub> near the interface with CoFeB. To test this prediction, polarized neutron reflectometry was performed on CoFeB/BiFeO<sub>3</sub> bilayers on the PRISM spectrometer. The data (see Figure 4) are well reproduced by taking into account a 2 nm thick layer carrying a 1 μ<sub>B</sub> per formula unit magnetic moment. This is far larger than the surface magnetic moment predicted by Malozemoff's model, and suggests that unpinned spins exist in BiFeO<sub>3</sub> near the interface along with the pinned moments predicted by the model. As the ferroelectric domain structure can be easily controlled by an electric field, these results open the route to the electrical manipulation of magnetization at room temperature, in BiFeO<sub>3</sub>-based exchange-bias heterostructures.

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# SUPERCONDUCTING PAIRING AND ELECTRONIC ANOMALIES INDUCED BY THE SPIN COLLECTIVE MODE IN HTC SUPERCONDUCTORS

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This work is part of a theoretical project which aims to unravel the mechanism at work in the high temperature cuprate superconductors. In the recent article [1] we have studied the role of spin fluctuations, and namely of the collective spin mode, for the superconducting pairing and numerous electronic anomalies observed in cuprates. This mode strongly coupled to the electrons (which develops in the vicinity of the antiferromagnetic wave vector and has an anomalous downward dispersion) was first predicted theoretically [2] and then observed by neutrons [3], Fig.1.

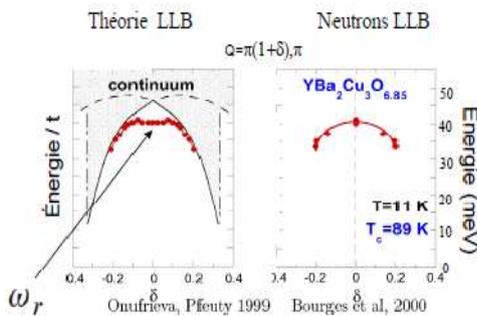


Fig.1 Spectre de fluctuations de spin avec le mode collectif (rouge)

The microscopic theory [1] is based on a dynamic strong coupling approach. The final equations are integral equations for the electronic correlation functions. They allow to describe all electronic properties. In [1] we analysed the superconducting properties together with the properties of the so called “normal” electrons for which important anomalies have been observed by photoemission and tunneling spectroscopy.

We found that the properties of the superconducting state induced by the spin mode are very close to those observed in the cuprates: The superconducting order parameter changes sign in the Brillouin zone while the superconducting gap

angular dependence presents an anomalous shape very close to that observed by photoemission, Fig.2 (effect unexplained until now). The value of the maximal gap is high (high Tc). The theoretical electronic spectrum is very close to that observed by photoemission, namely the nodal spectrum (spectrum in the part of the Brillouin zone where the gap vanishes) exhibits a kink. [The problem of the nodal kink is a hot problem in the field since its energy  $\Omega_{\text{kink}}$  represents the lowest energy scale in the electronic properties]. Not only the form of the theoretical spectrum is very close to that in the experiment (Fig.2), but the relation between  $\Omega_{\text{kink}}$  and the spin mode energy  $\omega_r$ ,  $\Omega_{\text{kink}} = -2\omega_r$ , obtained in [1], corresponds very well to the two independent experiment data (photoemission and neutrons),  $\Omega_{\text{kink}}=66\text{meV}$ ,  $\omega_r=34\text{meV}$  (for YBCO with  $T_c=61$  K),  $\Omega_{\text{kink}}=78\text{meV}$ ,  $\omega_r=40\text{meV}$  (for YBCO with  $T_c=90$  K). Finally, for the first time the anomalous form of the conductance (proportional to the electronic density of states) observed by tunneling spectroscopy has been explained (Fig.2) and the relations between the energies if its characteristic points and  $\omega_r$  have been obtained. Again the energy  $\omega_r$  extracted is in a good agreement with that seen by neutron.

All these results obtained within unified theory constitute an important argument in favour of the spin fluctuation mediated superconductivity in the high Tc cuprates.

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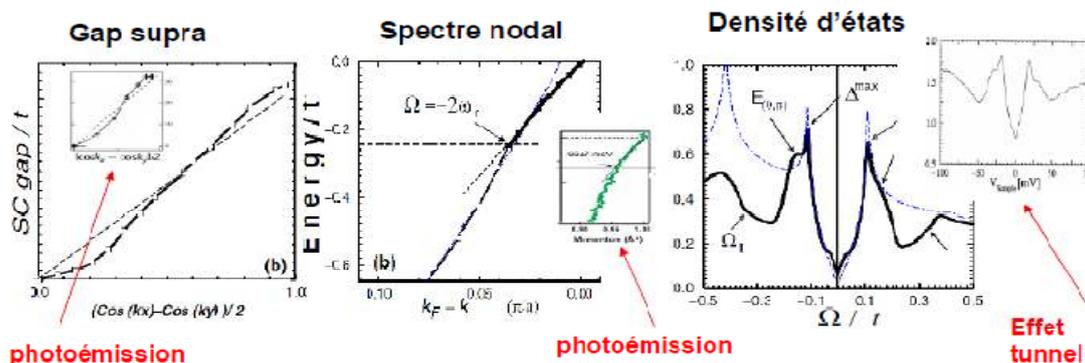


Fig. 2: Intensity map of the quasi-1D incommensurate at 3 meV and 5 K

## INFLUENCE OF THE ALTERATION LAYER MORPHOLOGY ON THE SILICATE GLASS CORROSION MECHANISMS: ROLE OF CA AND ZR

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This study investigates the long term behavior of glasses used for confinement of nuclear wastes. The results have been obtained from a fruitful collaboration between different CEA laboratories (LLB, LIONS, CEA Marcoule) and the Ecole Polytechnique. The corrosion process of the glasses by water creates at the glass surfaces, an alteration porous layer, hydrated and amorphous, called "gel". This gel, which is the result of the release of soluble elements, of hydrolyses and of silica network recondensation can in specific conditions strongly limit the exchange process between the glass and the solution. Our hypothesis to describe this phenomenon is the closure of the gel porosity.

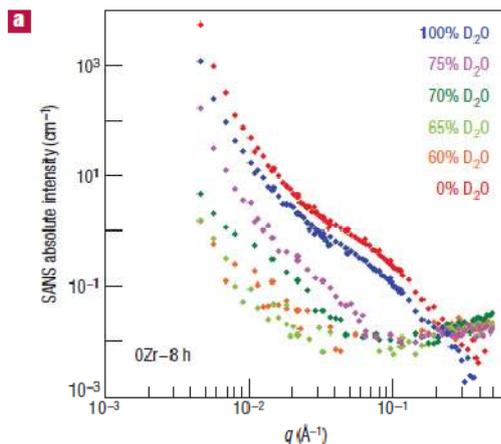


Fig. 1: Scattered intensity (SANS) for different isotopic mixtures for the sample without Zircon (0Zr) after 8 hours of alteration (a), after two months;

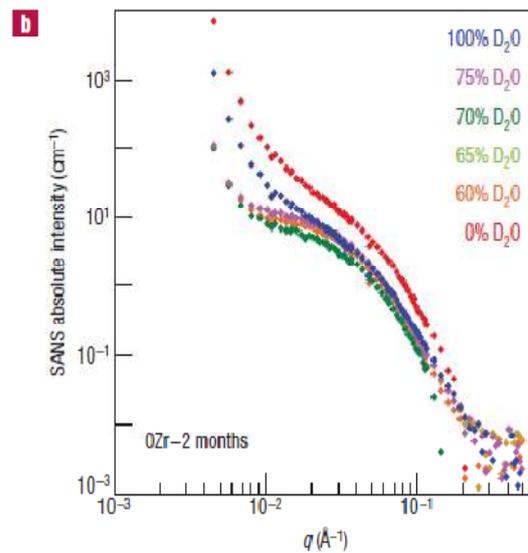


Fig. 1: Scattered intensity (SANS) for different isotopic mixtures for the sample without Zircon (0Zr) after 8 hours of alteration for the sample containing 4% of Zircon (4Zr) after leaching 2 months;

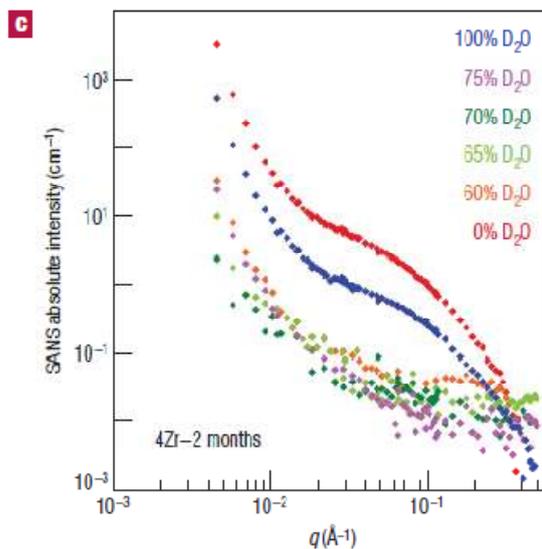


Fig. 1: Scattered intensity (SANS) for different isotopic mixtures for the sample without Zircon (0Zr) after 8 hours of alteration (a), after two months (b), for the sample containing 4% of Zircon (4Zr) after leaching 2 months (c). The residual signal observed on figure (b) for the « matching » composition (70%D2O) illustrates the existence of the closed porosity.

We present an original approach of the corrosion of silicate glasses, usually describe in term of chemical thermodynamic, and based on the influence of the morphological modifications of the gel on the slowing down of the kinetic of dissolution. The studied glasses are of composition of  $\text{SiO}_2\text{-B}_2\text{O}_3\text{-Na}_2\text{O-CaO-ZrO}_2$ . These elements constitute the different species present in nuclear glasses which can be classed according to their reactivity with water: soluble (B, Na), partially soluble (Si, Ca) and less soluble (Zr). We have been interested in the specific effects of Calcium and of Zirconium on the kinetic of alteration. We have observed that substituted the Calcium by the sodium improves the reticulation of the glass network to finally gives a decrease of the alteration rate. Regarding Zirconium, the increase of Zr-content decreases strongly the initial dissolution rate, which was expected, but increases paradoxically the degree of glass corrosion. Conversely, the increase of pH increases the initial dissolution rate but decrease the corrosion. To explain this surprising effect, the morphology of gels has been probed by Small Angles X-ray Scattering (SAXS).

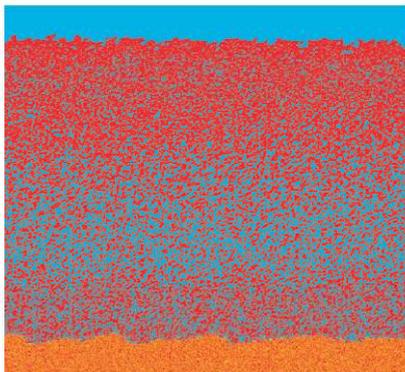


Fig. 2: Monte Carlo simulation of the altered gel without Zirconium (0Zr). The silicon atoms are shown in red, other element in yellow, water in blue.

These experiments have highlighted the reorganization of the porous network of the gel during the alteration and show that this reorganization is limited by the increase of the Zr-content. This suggests that this is the restructuring of the gel which is at the origin of the blockage of the alteration observed with the glass containing low Zr-content. This hypothesis has been successfully confirmed by Small Angle Neutron Scattering (SANS) according to an original application of the contrast matching method. Measurements for various solvent compositions ( $\text{H}_2\text{O-D}_2\text{O}$ ) have showed the porosity is closed for the glasses without Zircon (0Zr) and stay open for the glasses containing Zircon (figure 1).

These experiments of Neutrons Scattering, coupled with Monte Carlo1 (figure 2) simulations, permit to clearly establish a link between the gel morphology and the kinetic of alteration for a series of simplified glasses mimicking composition of nuclear glasses.

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SPIN-LADDER IRON OXIDE:  $\text{Sr}_3\text{Fe}_2\text{O}_5$ 

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The concept of “spin ladder” originally appeared to study the theoretically still controversial antiferromagnet (AF) 2D square lattice in high-temperature superconducting cuprates starting from a well understood 1D AF chain [1-2]. They can be schematized as an array of finite number of coupled chains. The motivation of theoretical investigations has been to test how the one-dimensional (1D)  $S=1/2$  AF chain system ( $n=1$ ), that is rigorously solved even when doped with carriers, could be connected to the 2D square lattice ( $n=\infty$ ) that in turn encounters various theoretical difficulties and is far from being understood. Experimentally,  $\text{Sr}_{n-1}\text{Cu}_n\text{O}_{2n-1}$  has been the only example representing a generalized spin-ladder system [3-5].

The utmost structural characteristic of cupric ( $\text{Cu}^{2+}$ ;  $d^9$ ) oxides is the favored formation of the square-planar  $\text{CuO}_4$  units, which are stabilized due to the Jahn-Teller effect [6]. This 2D coordination geometry provides strong exchange interactions within a ladder along the leg and the rung, while the interaction normal to the square plane is negligibly small due to the lack of superexchange pathways involving oxide ions. Unlike in the case of cuprates, the coordination geometries in iron oxides have been almost exclusively restricted to 3D polyhedra such as octahedra and tetrahedra. However, this barrier has recently been overcome [7], using electropositive calcium hydride at low temperatures as a reductant, the low-temperature reaction of a cubic perovskite  $\text{SrFe}^{4+}\text{O}_3$  with  $\text{CaH}_2$  stabilized  $\text{SrFe}^{2+}\text{O}_2$  with a square-planar oxygen coordination around the high-spin  $\text{Fe}^{2+}$  ion. The structure is isostructural with the “infinite” layer cupric oxides.

For the first time we show the synthesis of a novel spin-ladder iron oxide  $\text{Sr}_3\text{Fe}^{2+}_2\text{O}_5$  through the reaction of the double-layered perovskite  $\text{Sr}_3\text{Fe}^{4+}_2\text{O}_7$  with  $\text{CaH}_2$  [8]. By using neutron diffraction, synchrotron X ray diffraction and Mössbauer, we provide a complete characterization of this first spin ladder iron oxide.

A slightly oxygen deficient phase  $\text{Sr}_3\text{Fe}_2\text{O}_{7-y}$  ( $y\sim 0.4$ ) could be easily prepared by a conventional, high-temperature solid-state reaction and was used as a precursor. For  $\text{Sr}_3\text{Fe}_2\text{O}_{7-y}$  all oxygen vacancies are located at the apical O(1) sites shared by the double  $\text{FeO}_2$  sheets (Figure 1 a and b) and the structure keeps the  $I4/mmm$  space group over the entire range from  $\text{Sr}_3\text{Fe}_2\text{O}_7$  ( $y=0$ ) to  $\text{Sr}_3\text{Fe}_2\text{O}_6$  ( $y=1$ ) [9].

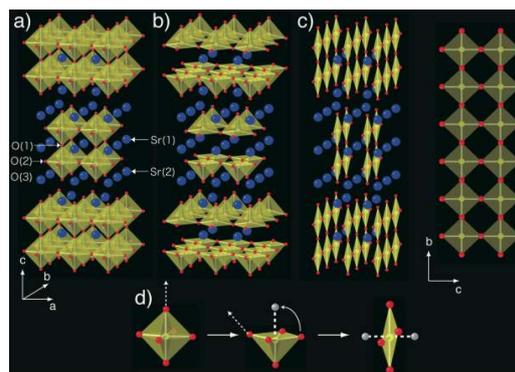


Fig. 1. Structural transformation from  $\text{Sr}_3\text{Fe}_2\text{O}_7$  to  $\text{Sr}_3\text{Fe}_2\text{O}_5$  via  $\text{Sr}_3\text{Fe}_2\text{O}_6$ , where the blue, red, and yellow spheres represent Sr, O, and Fe atoms, respectively. a) Crystal structure of a stoichiometric (fully oxidized) phase  $\text{Sr}_3\text{Fe}_2\text{O}_7$  ( $y=0$ ). b) Crystal structure of reduced phase  $\text{Sr}_3\text{Fe}_2\text{O}_6$  ( $y=1$ ), once thought to be the lower limit of the oxygen content. c) Left: Crystal structure of the new phase  $\text{Sr}_3\text{Fe}_2\text{O}_5$  ( $y=2$ ), described herein. Right: A spin ladder in  $\text{Sr}_3\text{Fe}_2\text{O}_5$  viewed along the  $a$  axis. d) Transformation from the octahedron (left) in  $\text{Sr}_3\text{Fe}_2\text{O}_7$  to the pyramid (middle) in  $\text{Sr}_3\text{Fe}_2\text{O}_6$  and to the square plane (right) in  $\text{Sr}_3\text{Fe}_2\text{O}_5$ , where the white spheres represent oxygen vacancies.

The powder X-ray diffraction (XRD) pattern of the present precursor also indicated a tetragonal  $I4/mmm$  cell with  $a = 3.872$  Å and  $c = 20.157$  Å. To prepare  $\text{Sr}_3\text{Fe}_2\text{O}_5$ ,  $\text{Sr}_3\text{Fe}_2\text{O}_{6.6}$  (0.43 g) and a two-molar excess of  $\text{CaH}_2$  were finely ground and pelletized in an argon-filled drybox, sealed in an evacuated glass tube, and brought to reaction at 623 K for 3 days. The final product was washed to remove undesirable residues,  $\text{CaH}_2$  and  $\text{CaO}$ , with a dried 0.1 M  $\text{NH}_4\text{Cl}$ /methanol solution. The Fe-oxide thus obtained is quite air-sensitive so that all the chemical and physical characterizations were conducted with efforts to minimize exposure to air. Synchrotron powder XRD pattern of the final product taken at the SPring-8 (Japan) BL02B2 beam line ( $\lambda = 0.777$  Å) showed that  $\text{Sr}_3\text{Fe}_2\text{O}_5$  adopts the  $Immm$  space group (No. 71), with  $a = 3.51685(5)$  Å,  $b = 3.95405(7)$  Å,  $c = 20.91727(36)$  Å. Neutron powder diffraction (DIA at ILL and G4.1 at LLB) analysis at 293 K confirmed the above structure and it also excluded a possibility that hydrogen atoms are incorporated into the lattice. Furthermore, Mössbauer measurements (Figure 2a) indicated not only that all the Fe atoms are electronically and

crystallographically equivalent, but also that they are most likely square-planar coordinated in high-spin state ( $S=2$ ), since the obtained isomer shift (IS) of  $0.46 \text{ mms}^{-1}$ , quadrupole splitting  $\Delta E$  of  $1.28 \text{ mms}^{-1}$  at 300 K, and the magnetic hyperfine field of  $H_{\text{hf}}=43.7 \text{ T}$  at 4 K are close to those of  $\text{SrFeO}_2$  [9]. These observations are consistent with the structural analysis. Neutron powder diffraction data down to 1.4 K on G41 (LLB,  $\lambda=2.42 \text{ \AA}$ ) (Figure 2b) and D1A (ILL,  $\lambda=1.91 \text{ \AA}$ ) revealed that the long-range AF order is characterized by a magnetic propagation vector  $q=(1/2, 1/2, 0)$  and that the iron moments of  $2.76(5) \mu_B$  are aligned parallel to the  $c$  axis.

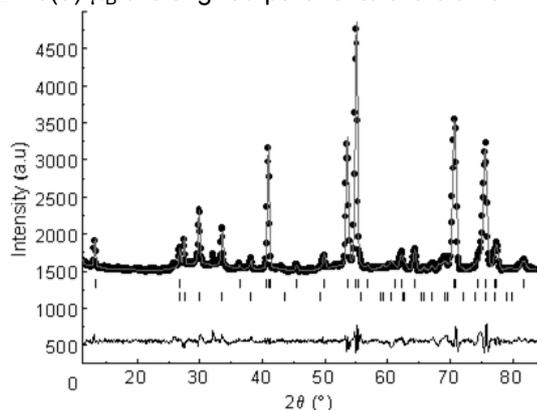
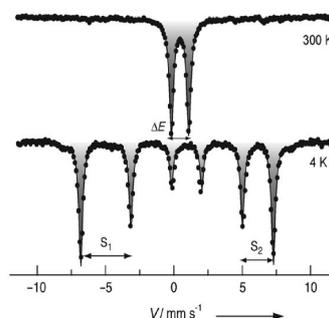


Fig 2b) Rietveld refinement of NPD data at 1.4 K measured at  $2.42 \text{ \AA}$  on G4.1 (LLB). The solid lines and the overlying crosses and bars indicate the calculated intensities, the observed intensities, and the positions of the calculated nuclear (top) and magnetic (bottom) Bragg reflections. The difference between the observed and calculated profiles is plotted at the bottom.

It is noteworthy that there are neither intermediate phases nor nonstoichiometry between  $\text{Sr}_3\text{Fe}_2\text{O}_6$  and  $\text{Sr}_3\text{Fe}_2\text{O}_5$ , as well as between  $\text{SrFeO}_{2.5}$  and  $\text{SrFeO}_2$ . This is remarkable given the presence of two intermediates,  $\text{SrFeO}_{2.875}$  and  $\text{SrFeO}_{2.75}$ , between  $\text{SrFeO}_3$  and  $\text{SrFeO}_{2.5}$ . It is also known that electrochemical formation of  $\text{SrCoO}_3$  from  $\text{SrCoO}_{2.5}$ , which are isomorphous with  $\text{SrFeO}_3$  and  $\text{SrFeO}_{2.5}$ , respectively, proceeds topotactically at room-temperature via the complex vacancy-ordered intermediate phase  $\text{SrCoO}_{2.82\pm 0.07}$  [10]. The processes occurring in the formation of  $\text{SrFeO}_2$  from  $\text{SrFeO}_{2.5}$ , whereby the  $\text{FeO}_4$  tetrahedra and the  $\text{FeO}_6$  octahedra linked to each other are both converted to  $\text{FeO}_4$  squares [11] must be more complicated than the case discussed above. We now recognize that the underlying perovskite and related lattices are flexible enough to tolerate such drastic compositional and structural events. We thus believe that oxygen transport materials working at low temperatures that feature formation of intermediate ordered structures should be promising.

Finally, we must assume that the present synthetic strategy involving  $\text{CaH}_2$  reduction can be further generalized for a more rational design of new magnetic lattices comprising extended arrays of  $\text{FeO}_4$  square planes, for which formerly only copper(II) oxides were candidates.



(b)

Fig. 2a) Magnetic order in  $\text{Sr}_3\text{Fe}_2\text{O}_5$ . a) Mössbauer spectra at 300 (top) and 4 K (bottom). The circles indicate the experimental data, while the lines denote the fits.

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## HIDDEN DEGREES OF FREEDOM IN APERIODIC CRISTALS.

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A fundamental concept in the description of the states of matter and the associated physical properties is that of order. For the solid state this has long been synonymous with periodicity, which is the basis of the notion of a crystal lattice, and a paradigm underpinning numerous fields of condensed matter. One way to probe the structure of matter on the atomic scale is to use scattering of radiation of a suitable wavelength, for example X-rays, neutrons. The existence of long-range order is then characterised by the appearance of diffraction spots which form a pattern with the symmetry of the crystal lattice. It is thus that non-periodic, yet still ordered, structures were discovered during recent decades. These *aperiodic* structures can be described in a periodic manner in a superspace with dimensions  $3+d$ , where 3 represents the usual *real* 3D space, and  $d$  the dimension of an *internal* space.

We are interested in aperiodic structures where a host framework forms subnanometric channels, which can contain and confine a network of guest molecules. We have now observed that there exists phase transitions where the structural changes correspond just to degrees of freedom *hidden* in the internal superspace. Experiments were performed on the cold-neutron triple-axis spectrometer 4F1 at LLB. In the studied nonadecane-urea inclusion compound, linear alkane molecules pack end-to-end in the channels of a honeycomb-like framework of urea molecules (figure 1). Because the ratio of repeat lengths along the channel axis of the urea host (the  $c$  axis) and the alkane guest is irrational the crystal as a whole is not periodic. The diffraction pattern requires four indices  $h, k, l, m$  with  $l$  and  $m$  the coefficients of the parallel reciprocal vectors  $c_{host}^*$  and  $c_{guest}^*$  respectively.

The average structure of the urea is described by the  $hk0$  reflections, the average structure of the alkane by the  $hk0m$  reflections, and the reflections  $hklm$  with  $l, m \neq 0$  are due exclusively to the mutual interaction between the lattices (figure 1). The high symmetry phase is described by an hexagonal four dimensional superspace group.

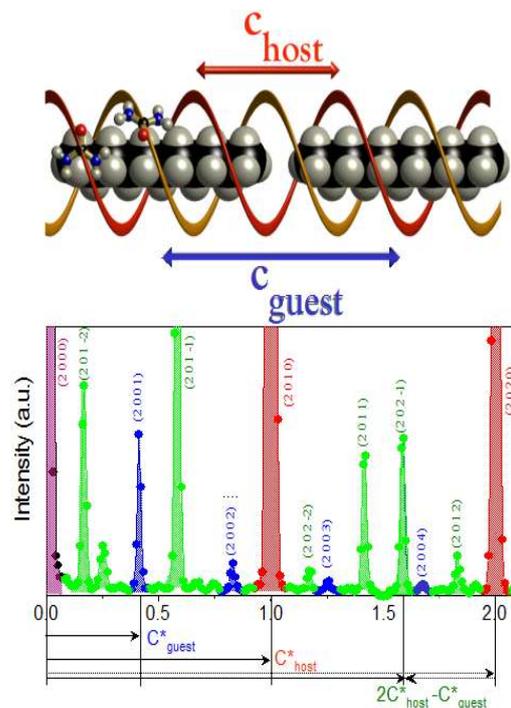


Fig 1: Real and reciprocal images of a self-assembled aperiodic material. Alkane/urea crystals are prototypical examples of nanotubular intergrowth compounds. Along the channel, the periodicity of the host urea subsystem is  $c_{host} = 1.102$  nm at room temperature, whereas the guest periodicity,  $c_{guest}$ , is roughly a linear function of the number of carbon atoms in the alkane molecule. The irrational ratio,  $c_{host}/c_{guest}$ , defines a misfit parameter  $\alpha$ . The reciprocal image is characterized by four different types of diffraction peaks. The common Bragg peaks in purple are located at  $(h, k, 0, 0)$ . The scale refers to the host periodicity, so host Bragg peaks  $(h, k, l, 0)$  in red are at integer positions along  $c^*$ , and guest Bragg peaks  $(h, k, 0, m)$  in blue are at multiple values of  $\alpha$  (which is also equal to  $c_{guest}^*/c_{host}^*$ ). Intermodulation satellite Bragg peaks, in green, are along  $c^*$  at the summation positions  $(h, k, l, m)$  with  $l$  and  $m$  different from zero. The presence of these satellites shows that each sublattice is modulated by the periodic potential of the other one [1].

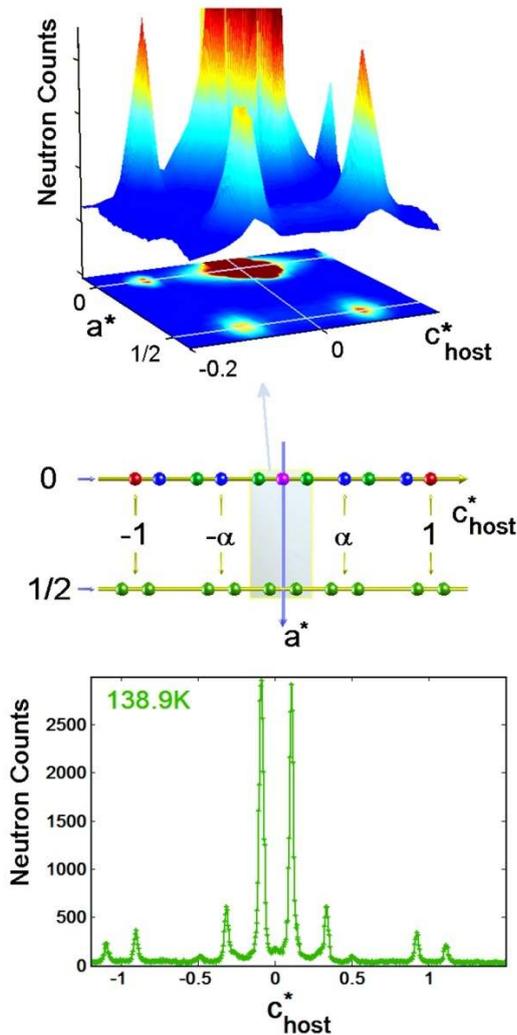


Fig. 2: Superstructure lines are generated in  $h=1/2$  (in the high symmetry hexagonal notation) at a first transition ( $T_{c1}=149\text{K}$ ). This doubling concerns only the internal dimension of the crystallographic superspace: there are no host, no guest, no common superstructure Bragg peak, but only satellites superstructure at  $138.9\text{K}$  [2].

We show that, at  $T_{c1}=149\text{K}$ , the crystal presents a structural instability towards an orthorhombic superspace with a doubling which concerns only its fourth superspace dimension [2,3]. Indeed, only satellite reflections of type  $1/2klm$  with  $l, m \neq 0$  appear in the superstructure reciprocal line (figure 2). There is thus an intermodulation of the host and guest lattices that alternates from channel to channel in the  $a$  direction, yet the average positions of the host and guest molecules are the same from channel to channel

On further cooling below  $T_{c2}=129\text{K}$ , a second phase transition appears revealing the four different types

of superstructure Bragg peaks (figure 3). So, the doubling now concerns also the mean host and guest substructures in addition to the previous doubling of the intermodulation.

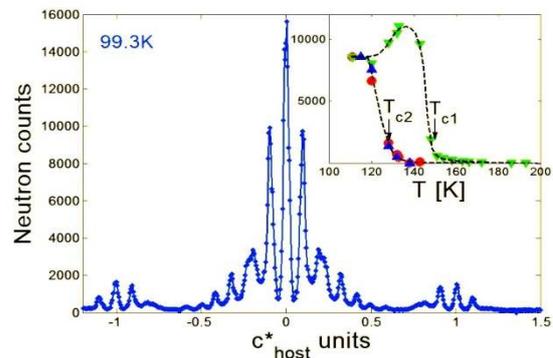


Fig 3: In *nonadecane\_urea*, a second ordered crystallographic phase appears below  $T_{c2}=129\text{K}$ . This phase is an orthorhombic superspace phase which is characterized by the appearance of host, guest and common superstructure in addition to the satellite ones already present in the first ordered phase. Whereas in the first ordered phase, the doubling concerns only the intermodulation, it concerns now also the mean host and guest substructures [2].

The unusual phase between  $149\text{K}$  and  $129\text{K}$  shows that nature can use the degrees of freedom hidden in the internal space to form states that cannot be envisaged in the usual 3D real space. This observation suggests a wider perspective for our understanding of the organisation of complex structures, manipulation of materials on the nanometric scale, sub-nano-fluidity, or translocation of polymers. In addition, these additional structural degrees of freedom of structure may couple with new dynamical ones to lead to new and tuneable properties of nanoscale and biological materials.

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## ISING VERSUS XY ANISOTROPY IN FRUSTRATED $R_2\text{Ti}_2\text{O}_7$ COMPOUNDS AS “SEEN” BY POLARIZED NEUTRONS

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The pyrochlore lattice is a model for geometrical magnetic frustration. In rare earth pyrochlore titanates  $R_2\text{Ti}_2\text{O}_7$ , frustration arises from the subtle interplay of single ion anisotropy, exchange, and dipolar interactions. Depending on the balance between these factors, one may observe spin ice ( $R=\text{Ho}, \text{Dy}$ ) [1] or spin liquid ( $R=\text{Tb}$ ) behaviors, or complex magnetic orders stabilized by first order transitions ( $R=\text{Er}, \text{Yb}, \text{Gd}$ ). A major role in the selection of the ground state is played by the single ion anisotropy that arises from the trigonal crystal electric field (CEF) at the rare earth site.

In the pyrochlore lattice, selection between Ising, Heisenberg, or XY models cannot be based, as usual, on the analysis of the macroscopic properties because of the presence of four anisotropy axes, namely, the  $\langle 111 \rangle$  axes. Then only an average over the four local axes can be measured by classical methods, and no direct information can be obtained about the anisotropy of the local magnetic susceptibility of the  $R$  ion, or of the local exchange or dipolar tensor. There is thus a clear motivation to get a direct access to these quantities and to quantify the axial or planar anisotropy in the rare earth pyrochlores.

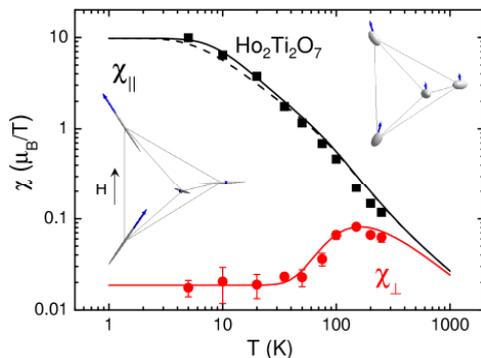


Fig. 1: Susceptibility components  $\chi_{\parallel}$  and  $\chi_{\perp}$  versus  $T$ . Dashed lines, CEF-only calculation; solid lines, calculation including effective exchange.

We have shown that the site susceptibility approach [2] developed for polarized neutron diffraction is an accurate tool for obtaining the local susceptibility tensor  $\chi$ . The measured thermal variations of the components of  $\chi$  are quantitatively compared with calculations using CEF [3]. To obtain good agreement with the data, one needs to introduce an anisotropic molecular field tensor, which takes into account both the first neighbor

exchange and the dipolar coupling [4].

Single crystals of  $\text{Ho}_2\text{Ti}_2\text{O}_7$ ,  $\text{Tb}_2\text{Ti}_2\text{O}_7$ ,  $\text{Er}_2\text{Ti}_2\text{O}_7$ , and  $\text{Yb}_2\text{Ti}_2\text{O}_7$  were grown by the floating-zone technique, using a mirror furnace. Their crystal structures were determined by zero-field neutron diffraction at 100 K and 5 K, within the space group  $Fd-3m$ .

Neutron diffraction measurements were performed at the ORPHÉE reactor of the Laboratoire Léon Brillouin. Polarized neutron flipping ratios were measured on the Super-6T2 spectrometer using neutrons of incident wavelength  $\lambda_n=1.4 \text{ \AA}$ , and on the 5C1 spectrometer ( $\lambda_n=0.84 \text{ \AA}$ ). For each compound, 100–200 flipping ratios were measured, at selected temperatures in the range 2–270 K, in a magnetic field of 1 T applied parallel to the  $[110]$  direction.

The thermal evolutions of  $\chi_{\parallel}$  and  $\chi_{\perp}$  are shown in Fig.1–4 for the four compounds. In the insets are represented the susceptibility ellipsoids at 5 K (lower) and 250 K (upper), whose axes have been scaled by temperature to compensate for the Curie-Weiss decrease, together with the magnetic moments induced by the field applied along  $[110]$ . The data were interpreted using a single ion

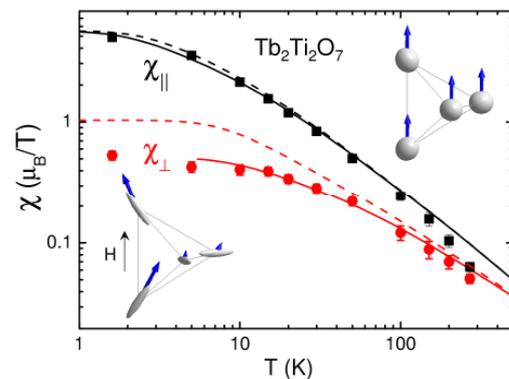


Fig. 2: Susceptibility components  $\chi_{\parallel}$  and  $\chi_{\perp}$  versus  $T$ . Dashed lines, CEF-only calculation; solid lines, calculation including effective exchange.

approach, where the trigonal symmetry CEF and Zeeman Hamiltonian is written as

$$H = \sum_{n,m} B_n^m C_n^m + g_J \mu_B \mathbf{J} \cdot \mathbf{H}$$

## HIGHLIGHTS

In order to take into account exchange and dipole-dipole interactions, we introduced an anisotropic molecular field tensor  $\{\lambda_{\parallel}, \lambda_{\perp}\}$  and assumed a field component of 1T either parallel or perpendicular to the ternary axis.

**Ho<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> (Fig. 1):** At low temperature, the ellipsoid elongations  $\chi_{\parallel}$  increase so markedly that they degenerate into needles, while  $\chi_{\perp}$  is very small, lying at the limit of experimental precision. These features are explained by the CEF level scheme of Ho<sup>3+</sup> ( $J = 8$ ,  $g_J = 5/4$ ): the ground state is very close to the extremely anisotropic non-Kramers doublet  $|J=8; J_z=\pm 8\rangle$ , which has a moment of  $10 \mu_B$  along the ternary axis and  $\chi_{\perp} = 0$ , and the first

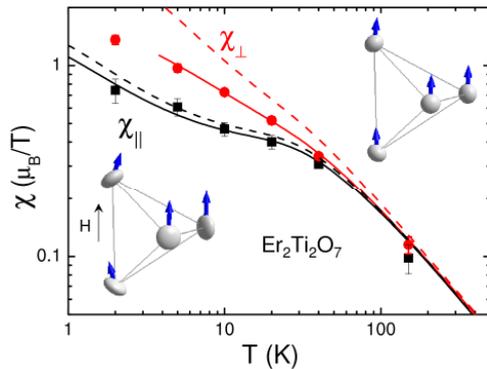


Fig. 3: Susceptibility components  $\chi_{\parallel}$  and  $\chi_{\perp}$  versus  $T$ . Dashed lines, CEF-only calculation; solid lines, calculation including effective exchange.

excited state lies at about 250 K above the ground state. For  $\chi_{\parallel}$ , introduction of a small positive (ferromagnetic) value  $\lambda_{\parallel}=0.05(1) \text{ T}/\mu_B$  improves the agreement with the data.

**Tb<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> (Fig. 2):** The CEF anisotropy of Tb<sup>3+</sup> ( $J = 6$ ,  $g_J = 3/2$ ) is considerably smaller than that of Ho<sup>3+</sup>. At room temperature, it is practically isotropic due to the relatively large number of populated CEF levels. Decreasing the temperature, it evolves progressively into an Ising type with a ratio of ellipsoid axes of the order of 10. Introducing an anisotropic  $\lambda$  tensor improves the agreement, especially for  $\chi_{\perp}$ , with the AFM values:  $\lambda_{\parallel}=-0.05(2) \text{ T}/\mu_B$  and  $\lambda_{\perp} = -1.0(2) \text{ T}/\mu_B$ . Both components of the  $\lambda$  tensor, which corresponds to an effective (exchange+dipole) low temperature interaction, are found to be of the AFM type.

**Er<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> (Fig. 3):** The anisotropy of Er<sup>3+</sup> ( $J=15/2$ ,  $g_J = 6/5$ ) is rather weak, and a spherical shape for the magnetic ellipsoids is recovered above 100 K. At low temperature,  $\chi_{\perp} > \chi_{\parallel}$ , is in agreement with the planar magnetic ordering. We find the  $\lambda$  tensor is also anisotropic and of the AFM type:  $\lambda_{\perp} = -0.45(5) \text{ T}/\mu_B$  and  $\lambda_{\parallel} = -0.15(1) \text{ T}/\mu_B$ . The in-plane effective exchange is thus stronger than its

## Structure and Phase Transitions

component along the ternary axis, which reinforces the XY character determined by the CEF anisotropy.

**Yb<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> (Fig. 4):** Yb<sup>3+</sup> ( $J=7/2$ ,  $g_J=8/7$ ) shows a planar anisotropy. Its magnetic ellipsoids are disk-shaped, like in Er<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub>, but a strong anisotropy persists up to much higher temperature because

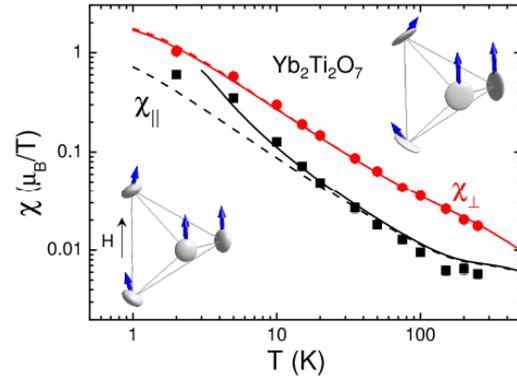


Fig. 4: Susceptibility components  $\chi_{\parallel}$  and  $\chi_{\perp}$  versus  $T$ . Dashed lines, CEF-only calculation; solid lines, calculation including effective exchange.

the excited Kramers doublets lie much higher in energy (700–1000 K). A ferromagnetic value  $\lambda_{\parallel}=2.5(5) \text{ T}/\mu_B$  is needed to reproduce  $\chi_{\parallel}(T)$ . The  $\lambda_{\parallel}$  is much stronger than  $\lambda_{\perp} = 0$ , which results in a moment, at the temperature below 0.25 K, lying along the  $\langle 111 \rangle$  axis, out of the rule of the strong CEF easy plane anisotropy.

In conclusion, polarized neutron diffraction allowed us to determine the local susceptibility tensor in the  $R_2\text{Ti}_2\text{O}_7$  series, inaccessible by macroscopic measurements in single crystals. Its temperature dependence in the paramagnetic phase cannot be entirely accounted for by the sole crystal field anisotropy: a molecular field tensor  $\lambda$  must be introduced, which could be important for theory.

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## CONTROL OF ROUGHNESS AT INTERFACES AND THE IMPACT ON CHARGE MOBILITY IN ALL-POLYMER FIELD-EFFECT TRANSISTORS

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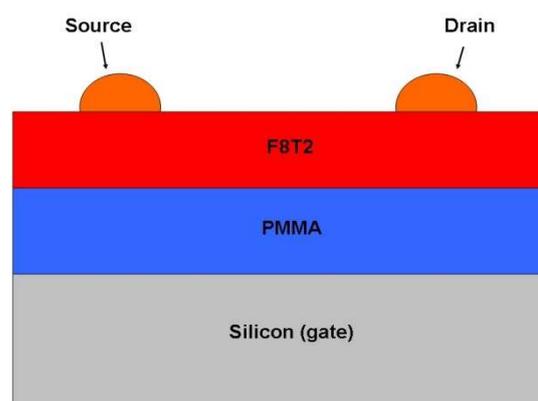
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Using conjugated polymers as the active materials in electronic and optoelectronic devices opens up the possibility of fabricating all-polymer devices using solution processing technologies. The fabrication of good quality field-effect transistors (FETs) is crucial to a number of polymer-based devices, such as active matrix displays and integrated circuits. Central to FET operation is the dielectric/semiconductor interface. Here we look at the interface between a polymer gate dielectric and a conjugated polymer, using neutron reflectivity. By using a mixed solvent (toluene/cyclohexane) to deposit the conjugated polymer directly on top of the polymer dielectric we are able to fabricate bilayer FET architectures with systematically controlled interfacial roughness, and study the impact on transistor performance.

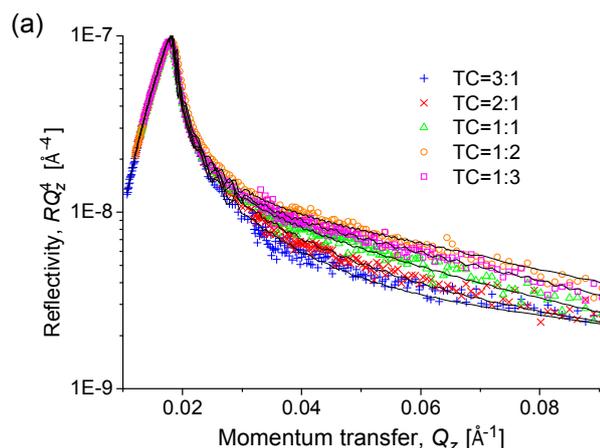
The use of conjugated organic materials as the semiconducting component, within electronic devices is an increasingly important technology. Conjugated polymers combine the promise of products with desirable properties such as flexibility, with the processing advantages of solution-based production methods (large areas at low cost). The nature of the interfaces between semiconducting polymers and other materials is of vital importance in the performance of several organic devices. This study focuses on FETs, where the interface between the polymer dielectric and the semiconducting polymer is of crucial importance, as it is adjacent to this interface that current flows within the device.

In this work we focus on a bottom gate, top contact architecture, with poly(9,9-dioctylfluorene-*alt*-bithiophene) (F8T2) as the semiconducting layer and poly(methylmethacrylate) (PMMA) as the gate dielectric (see figure 1). By dissolving the F8T2 in a miscible mixture of a good solvent (toluene) and a poor solvent (cyclohexane) for PMMA in various volume ratios, and directly spin-coating onto the PMMA layer, it is possible to control the structure of the buried F8T2/PMMA interface. By varying the ratio of the two solvents in this mixture we are able to systematically control interfacial roughness over a broad range and correlate this directly with charge mobility. FETs were also fabricated following the same procedures, but with the addition of a 300nm SiO<sub>2</sub> on top of the silicon substrate.



**Figure 1.** Schematic diagram of a bottom gate, top contact FET, showing the source, drain and gate electrodes.

Our goal in this study was to make polymer-polymer bilayers with controlled interfacial roughness. However, only specific combinations of PMMA molecular weight and solvent ratio can produce well-controlled bilayer structures. If the toluene content is too high, or the PMMA molecular weight too low, dissolution of the PMMA film by the solvent used for the deposition of the F8T2 is too rapid, and a complex laterally structured morphology is obtained. For PMMA with molecular weight Mw=313,500 g/mol we are able to produce bilayers for toluene:cyclohexane (T:C) solvent ratios ranging from 4:1 to 1:3, and it is these bilayers that we shall discuss here.



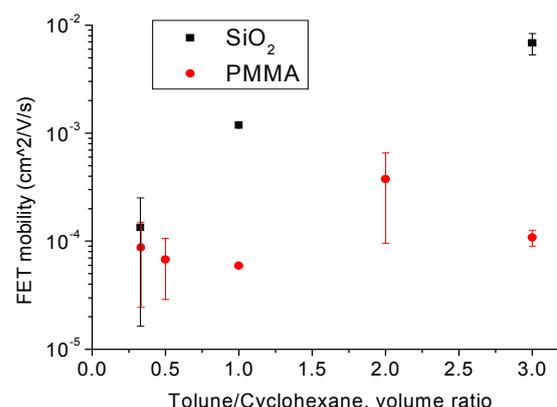
(b)

**Figure 2.** Reflectivity data (Reflectivity  $\times Q_z^4$ ) for F8T2/PMMA/silicon bilayers at various solvent ratios. Solid lines represent bilayer fits with the surface and interfacial profiles modelled as error functions.

Specular neutron reflectivity measurements were performed on several bilayers at the reflectometers EROS (LLB) and CRISP (ISIS.). The reflectivity curves for five different bilayers with T:C ratios 3:1, 2:1, 1:1, 1:2, and 1:3 are shown in figure 2. It is clear that the reflectivity systematically reduces as the toluene content rises. The root-mean-square roughnesses extracted from the fits in figure 2 decrease from approximately 3.3 nm to around 1.5 nm as the T:C ratio changes from 3:1 to 1:3. It is therefore clear that we are able to systematically control the interfacial roughness at this dielectric/semiconductor interface using solvent quality as a control parameter.

Working devices were then made from these bilayers. Figure 3 shows the field-effect mobility extracted from the F8T2/PMMA FETs and from a set of control FETs. The controls were made from the same mixed solvents but used SiO<sub>2</sub> alone as the gate dielectric. The data from the F8T2/SiO<sub>2</sub> FETs allow a measurement of mobility as a function of the solvent quality, independent of the interface roughness, which remains constant at less than 5 Å. Figure 3 shows a clear correlation between the mobility and the T:C ratio for the F8T2/SiO<sub>2</sub> transistors, with higher charge mobility for toluene-rich solvent. We believe that this is because by using a good solvent the polymer

chains are more extended in the film leading to more homogeneous electronic structure across the film, more uniform interchain packing and improved mobilities. However, in the context of the present study which is focussed on the effects of interface roughness the detailed dependence of conformation/packing of the polymer chains within the solid films on solvent ratio has not yet been investigated.



**Figure 3.** Field-effect mobility measurements for F8T2/SiO<sub>2</sub> and F8T2/PMMA FETs.

Typical mobilities in the F8T2/PMMA FETs are significantly lower than those in the F8T2/SiO<sub>2</sub> FETs, reflecting the significantly higher interface roughness at the F8T2/PMMA interfaces compared to that of F8T2/SiO<sub>2</sub>. However, in contrast to the F8T2/SiO<sub>2</sub> FETs, the mobility of the F8T2/PMMA devices in figure 3 shows relatively little dependence on solvent ratio. Therefore it appears that, to a first approximation, the effect of increasing roughness is to reduce mobility, and that this effect is sufficiently strong to cancel out the dependence of F8T2 charge mobility on conformation/packing. It therefore appears that in this particular system the two effects counteract each other and lead to a mobility which is not significantly dependent on solvent quality.

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## ANISOTROPIC REINFORCEMENT OF NANOCOMPOSITES TUNED BY MAGNETIC ORIENTATION OF THE FILLER NETWORK

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The improvement of the mechanical properties of polymer films by inclusion of mineral particles is a well known industrial practice used since the end of the XIX century. A classical example is the rubber industry for which the addition of carbon black as fillers permits to increase the elastic modulus of the pneumatics. Nevertheless, the mechanisms which govern the reinforcement properties are still not completely described by experiments and theory. More recently, the use of fillers of nanometric size and of controlled geometry like colloidal silica allowed significant advances in the understanding of these mechanisms and in the conception of innovative materials. In this context, an original approach is to try to improve and to control the specific properties of the material with an external trigger like a magnetic field. We have obtained polymer films with anisotropic mechanical properties by inclusion of magnetic particles inside the matrix which can be aggregated in a controlled way during the film processing and oriented with an external magnetic field. The orientation of the fillers during the processing condition with the field is a new approach when magnetic composites are usually exploited to modulate the mechanical properties after the synthesis.

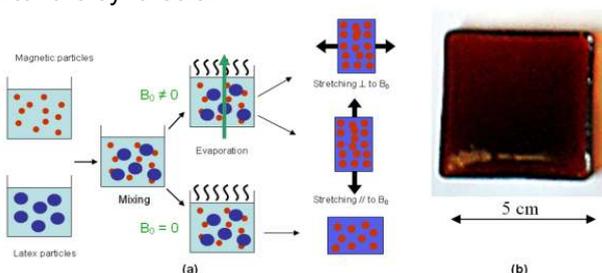


Fig. 1: (a) Principle of films processing without and with magnetic films, (b) picture of the final film.

The film synthesis was adapted from a previous work [1] on films formed by simple drying of aqueous solutions of nano-latex and of silica particles ( $D=10\text{nm}$ ). The morphology of the aggregates of particles controlled the macroscopic properties of the materials and can be modulated with the physical-chemistry conditions of the solutions before casting. Our magnetic particles (maghemite  $\gamma\text{-Fe}_2\text{O}_3$ ,  $D=10\text{nm}$ ) have been covered with a thin silica layer to realize the synthesis in the same conditions as [1] and to control the aggregation. Films have been dried without/with magnetic films (150 Gauss) (Fig. 1a). Resulting composites are macroscopically homogeneous (Fig. 1b).

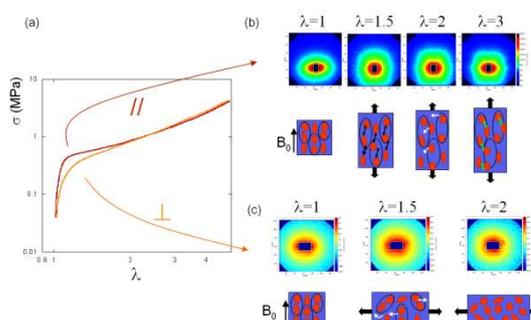


Fig. 2: (a) Stress-strain isotherm for a sample synthesized under magnetic field, stretched along the field direction or perpendicular to it, (b) 2D SANS curves for stretched sample parallel to the field (top) and perpendicular to it (bottom) as a function of the deformation, and resulting filler structure in the real space.

When the films are drying under magnetic films, the mechanical properties are very anisotropic. The reinforcement is larger along the direction parallel to the field than along the direction perpendicular to the field in the low deformation regime (figure 2). The rapport between the reinforcement factors is larger than 2! For larger deformations, the mechanical properties become similar and are close to the modulus of the pure matrix. These spectacular properties can be explained by the local structure of the magnetic particles inside the polymer matrix which has been determined with SANS. During the drying process, anisotropic aggregates are formed and oriented along the field direction. When the films are stretching along the field direction, the aggregates re-arrange and connected to form a filler network which increases strongly the reinforcement. Conversely, when the films are stretching perpendicular to the field direction, the aggregates filler network is breaking and the resulting reinforcement is lower [2].

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## SLOOKING AT MICELLISATION OF GRADIENT COPOLYMERS IN SUPERCRITICAL CARBON DIOXIDE

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Self-assembly properties of small molecules into large supramolecular assemblies have been a matter of interest for scientists for more than a century now. One might thus tend to believe that the spontaneous formation of, e.g., micelles of surfactant molecules in water, has given up all its secrets. Indeed, many possible morphologies of micelles have been discovered and characterised by soft matter scientists, in particular using small angle neutron scattering (SANS). The careful measurement of micellar properties by SANS has contributed greatly to our understanding of the multiple forces acting between the different parts of the molecules, like between the hydrophobic tails, or between the hydrophilic head groups, and their implications on micellar structure and interaction. The importance of industrial applications, like detergency, enhanced oil recovery, or mineral flotation, have also been and still are a powerful motor of this research area. Obviously, the behaviour of a surfactant system, like cleaning power, depends strongly on its capacity to take up hydrophobic, oily substances in aqueous solvents, a process naturally promoted using micelles as molecular transport vehicles.

solvating properties can be tuned by applying moderate pressures: CO<sub>2</sub> becomes supercritical (scCO<sub>2</sub>) close to room temperature (T > 31.1°C), at P > 73.8 bar. Unfortunately, molecules which are amphiphilic with respect to water do not automatically possess self-assembly properties in scCO<sub>2</sub>. One partner of this collaboration, the school of chemistry in Montpellier (ENSCM) has a long-standing record of synthesizing new types of molecules, with amphiphilic properties with respect to scCO<sub>2</sub>. The primary scope of such developments is the design of new systems having self-assembly properties in scCO<sub>2</sub>.



Fig. 2: Cloud point observation. Left: the polymer is soluble and light reflexes are transmitted through the sample. Right: the system is below the cloud point and thus opaque.

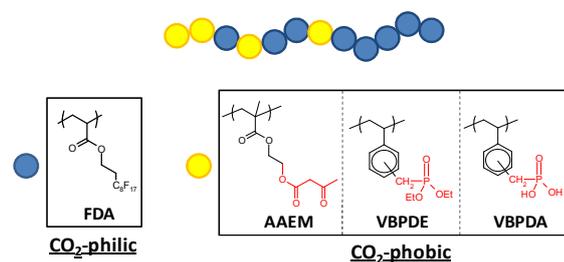


Fig.

1: Different gradient copolymers used in this study.

The natural driving force of surfactant self-assembly is amphiphilicity, i.e. it is due to the opposite behaviour of the head and tail groups with respect to water: the hydrophobic groups induce aggregation in order to minimize contact with water molecules. It is then a natural objective of surfactant studies to transpose this behaviour to other solvents, and in particular to CO<sub>2</sub>. Compared to organic solvents, CO<sub>2</sub> has many advantages: it is non toxic, cheap and environment-friendly. But most importantly, its

In this project, we have developed and tested a variety of polymeric surfactants, i.e. molecules where the CO<sub>2</sub>-philic and CO<sub>2</sub>-phobic groups are actually small polymer blocks. As compared to smaller surfactant molecules, this allows us to add additional functions to the molecule, a point to which we come back below. Three purpose-synthesized molecules are presented in Figure 1. In all cases, the CO<sub>2</sub>-philic part - poly(FDA) - is the same. These fluorinated molecules are known to be highly soluble in scCO<sub>2</sub>, a feature which can be easily tested by visual inspection of the solubility limit called the cloud-point as shown in Figure 2.

The CO<sub>2</sub>-phobic parts are represented in Figure 1 [1]. By themselves, they are virtually insoluble in

scCO<sub>2</sub>, whereas the polymeric surfactant molecules built with them and FDA are found to be extremely soluble. Polymeric CO<sub>2</sub>-surfactant molecules have become well-studied over the past two decades. A novelty with our system is that, instead of synthesizing block copolymers by a two-step process, we have prepared gradient copolymers in a one-step process. Such gradient copolymers have so far been rarely studied in CO<sub>2</sub>, and never by SANS.

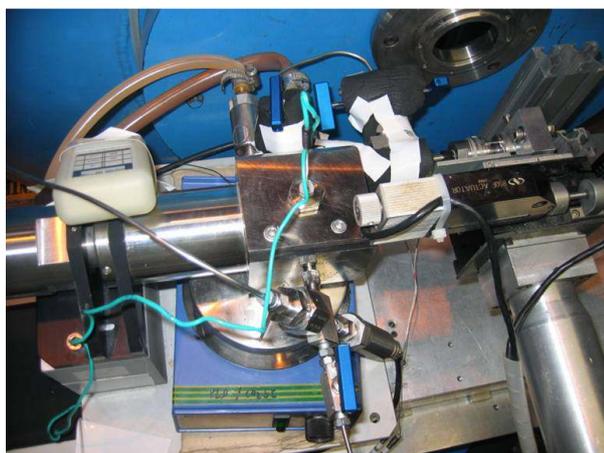


Fig. 3: Photo of the Niobium pressure cell in front of the beam-tube, and the variable volume view-cell with sapphire window on the left.

As stated above, it is crucial for applications to have micellisation in scCO<sub>2</sub>. The fundamental question we have to address is if such gradient molecules still present sufficiently strong self-assembly properties. The experimental challenge encountered in CO<sub>2</sub>-systems is that measurements have to be performed under high pressure. Therefore, we have used a pressure vessel in Niobium, which displays a neutrons transmission superior to 90% at 6 Å, allowing direct micellisation studies of our polymeric surfactant in scCO<sub>2</sub>. The experimental set-up is shown in Figure 3.

A large number of parameters had to be explored in order to obtain a comprehensive view of the aggregation properties of our gradient copolymers [2]. Besides thermodynamic parameters like pressure and temperature, we have studied self-assembly as a function of concentration, molecular weight and composition. In Figure 4, the effect of decreasing pressure is shown. The increase in intensity can be translated in a higher average aggregation number, from unimers to about three. Concomitantly, the typical size increases from about 25 to 33 Å. Summarizing this study which extended over several runs of neutron beam-time over the past years at LLB, we found that aggregation is enhanced for high concentration of polymer, low

temperatures and pressures, and for a copolymer having a high fraction of CO<sub>2</sub>-phobic units.

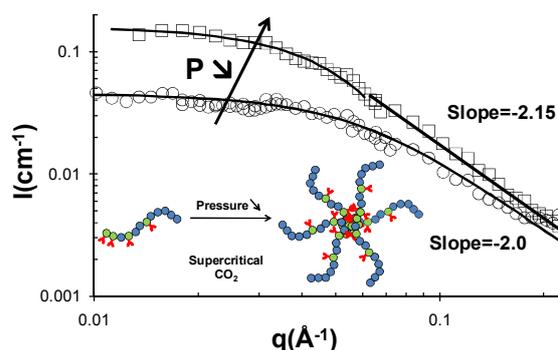


Fig. 4: Influence of pressure. SANS data for a gradient copolymer with AAEM units at C=4 %wt of polymer vs. CO<sub>2</sub>: T=40 °C, P=140 bar (□) and T=40 °C, P=210 bar (○), with the Debye fit indicating a random coil, respectively. The arrow indicates the evolution with decreasing pressure.

We have thus demonstrated that our gradient copolymer system is capable of micellisation in scCO<sub>2</sub>. From this rather academic question, an interesting connection to recent developments in green chemistry can be drawn by adding a particular function to our molecules. One of the problems of nuclear waste management is that washing contaminated industrial workers' cloths generates huge quantities of possibly radioactive water. By switching to scCO<sub>2</sub> as a solvent, one may capture radioactive ions in micelles, and then dispose of the clean CO<sub>2</sub> - thereby concentrating the ions in much smaller quantities of surfactant. This is the reason why we have added the particular functions (acetoacetoxy, phosphonate ester and phosphonic diacid represented in red in Figure 1) to the molecules depicted above. Having shown their micellisation in scCO<sub>2</sub>, it is hoped that our study will open the way to a new, 'greener' treatment of nuclear waste.

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## SELF ASSEMBLY OF FATTY ACIDS IN AQUEOUS SOLUTIONS

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The use of agricultural resources for industrial purposes will undoubtedly be one of the major challenges of the 21st century. Organic biosyntheses used in chemistry should progressively replace those coming from fossil fuels. Our work on dispersions of fatty acids and hydroxylated derivatives forms part of these efforts in that it seeks to demonstrate the potential contribution of fatty acids (which may be extracted from plants) as a new class of surface active agents. Dispersions of fatty acid and their hydroxyl derivatives are thus studied in solution in order to generate a new class of surface active agents for foaming and emulsifying properties.

However, it is known that fatty acids and their hydroxylated derivatives are insoluble in water. Our initial studies thus targeted the physicochemical conditions which would enable dispersion of these compounds. Using commercial fatty acids as model systems, we produced dispersions by using a large variety of counter-ions such as soluble organic amines (ethanolamine, lysine...). These salts made it possible to obtain homogeneous dispersions with considerable polymorphism, which formed micelles [1], vesicles [2], nanotubes [3, 4], cones [5] and torsades [6]. Preliminary studies in foams and emulsions have demonstrated that their stability differs as a function of polymorphism [7].

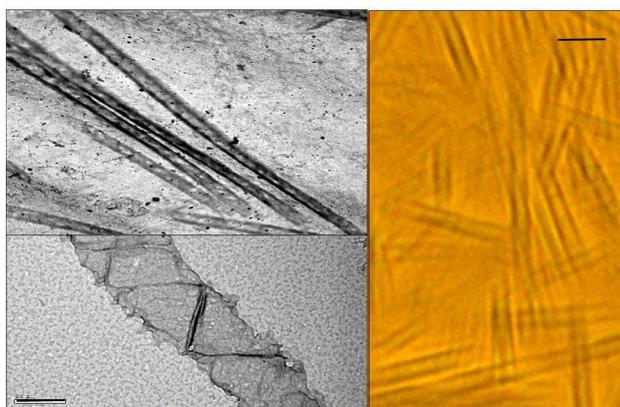


Fig. 1: TEM micrographs (left) (scale bar: 2  $\mu\text{m}$  (top) and 500 nm (bottom)) and (right) phase contrast micrograph at 52  $^{\circ}\text{C}$  of a tube dispersion (scale bar: 10  $\mu\text{m}$ )..

Here, we report on the results obtained with the nanotubes made of the ethanolamine salt of 12-hydroxy stearic acid and a novel system of sodium salts of fatty acids in the presence of guanidine chloride and show how small angle neutron diffraction (SANS) help us for the characterization of such systems.

12-hydroxy stearic acid is insoluble in water but forms tubes when combined with ethanolamine as the counter-ion<sup>3</sup>. The tubes are well visualised by classical microscopy techniques (see Fig. 1). Their external diameter is about 600 nm at room temperature. Those tubes exhibit an incredible temperature behaviour since their diameter varies by a factor ten upon heating [4]. The SANS pattern of such dispersion is depicted at Fig. 2. It shows a strong anisotropic scattering signal and 3 Bragg peaks positioned at  $Q_0$ ,  $2Q_0$  and  $3Q_0$  indicating the formation of stacked bilayers between which water is intercalated. The distance between the bilayers can then be measured from  $Q_0$ , and is the order of 350 Å at room temperature and slightly decreases with the temperature. One can also measure the bilayer thickness in the Porod regime (not shown) which returns a value of about 40 Å at low temperature and 27 Å at higher. This change indicates a phase transition from a gel to a fluid bilayer.

We now turn on aqueous dispersions made of sodium salts of saturated fatty acids in the presence of guanidine chloride (GuCl). This system was shown to exhibit a broad polymorphism by using microscopy and solid state NMR. Whereas pure sodium salts of fatty acids crystallise at low temperature, the presence of GuCl prevents this phenomenon and yields the formation of a viscous birefringent solution of membranes at low temperature (not shown) and a phase of 'anastomosis' is observed (fig. 3) at higher (in press in JCIS). One clearly sees that the fatty acids self assemble into ramified superstructures having a width of tens nm and several microns length, called 'anastomosis' which are defined as 'a network of streams that both branch out and reconnect' 8.

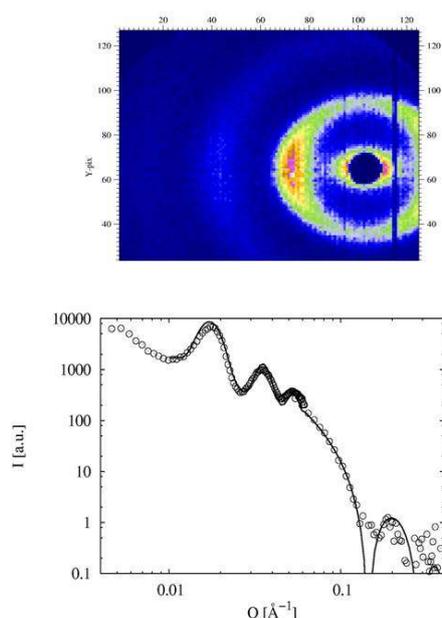


Fig. 2: SANS patterns of a tubes dispersion at room temperature. The anisotropy in the 2D signal (top) arises from the partial alignment of the tubes because of the flow when pouring the solution within the SANS cell.

We shall now investigate this system by using SANS experiments in order to determine the exact structure of those superstructures. If they are composed by nanotubes made of a bilayer of fatty acids, one should obtain oscillations or at least a typical signal in the porod regime that should give us the bilayer thickness. However, if those superstructures are made of a bundle of elongated micelles, the scattering signal should be at a power  $-4$  for low  $Q$ .

Unexpectedly, for an alkyl chain containing 20 carbons, the system now forms vesicles (Fig. 3). Clearly, the vesicle membranes are composed by fatty acid bilayers which were characterized by deuterium solid state NMR. We also need to perform SANS experiments on such vesicles for determining the characteristic dimensions. For instance, it is probable that the alkyl chains in the bilayer are interdigitated. This could be easily measured in the Porod regime.

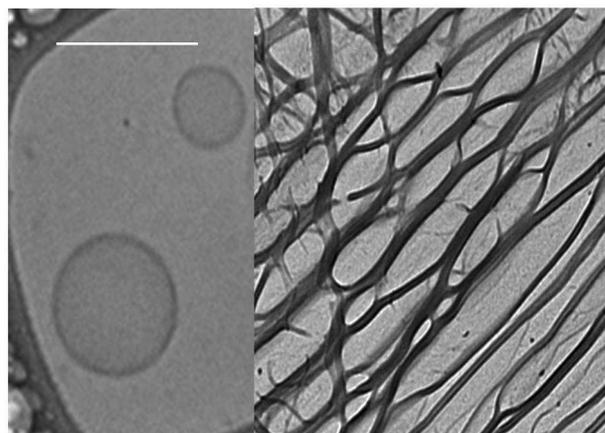


Fig. 3: TEM micrographs of two systems in the presence of GuCl. (left, scale bar 500nm) vesicles made of the sodium salt of arachidic acid (C20) and (right) the phase of anastomosis.

In summary, we have used SANS experiments for characterizing our systems made of fatty acid dispersions. That technique is very useful for determining the polymorphism and structural dimensions of the assemblies.

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## INFLUENCE OF MACROMOLECULAR CROWDING ON PROTEIN STABILITY

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Following the synthesis by the ribosome, to carry out its biological function, a protein much fold into a single, well defined conformational state: the native state. Protein folding is thus the physico-chemical process by which a polypeptidic chain undergoes a structural change from an ensemble of coil like structure up to the unique structure encoded in its amino-acid sequence. This process is fascinating and remains one of the most challenging problems of structural biology. Protein misfolding is involved in number of pathologies such as BSE an Alzheimer diseases.

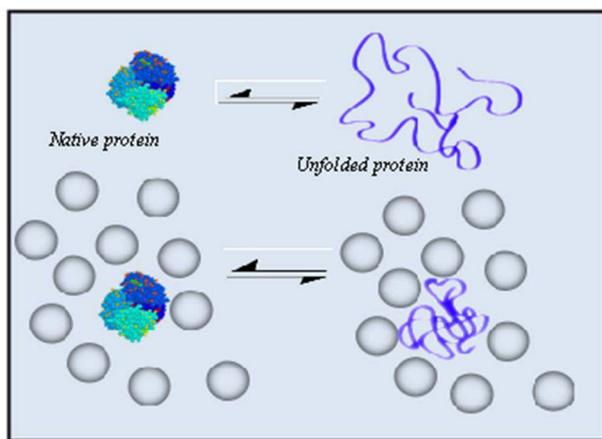


Fig. 1: Equilibrium reaction between the Unfolded ensemble and the Native state of a protein with or without crowding agent.

Experimental studies of protein folding are often (if not always) performed in diluted solutions. Under such conditions small globular proteins can fold and unfold relatively rapidly depending mostly on the nature of the solvent (presence of unfolding agents, change of temperature or pressure ...). In-vivo, under physiological conditions, the proteins are surrounded by a huge number of macromolecules different in nature, size and shape (proteins, nucleic acids, ribosome, carbohydrates ...). Each macromolecule is generally present at very low concentration, but the overall mass concentrations of macromolecules in the cytoplasm can reach values as high as 300 to 400 mg.ml<sup>-1</sup>, which correspond to volume fractions of the order of 0.2 to 0.3 [1,2]. This environment is qualified of crowded. The chemistry of life, as opposed to the biochemistry of the laboratory, will occur in a very densely packed media. For such volume fraction the protein-protein interactions play a fundamental role. These interactions can be either specific (protein associations ...) or non-specific (sterical or

electrostatic interactions). Non specific interactions are generally much weaker than specific ones.

Theoretical works [3,4] predicted that macromolecular crowding in cells could significantly influence protein stability. The predictions suggest that the equilibrium ( $N \leftrightarrow S_i$ ) between the native (N) and the unfolded states ensemble ( $S_i$ ) of proteins is shifted towards the native state due to the excluded volume effect induced by macromolecular crowding. The volume excluded to a polypeptide chain by high concentrations of rigid macromolecule would be expected to exert a compressive force reducing its average dimension. More extended conformations of the unfolded chain ensemble are predicted to be preferentially destabilized relative to more compact conformations, including the native state.

Experimental studies intending to verify whether the proteins are stabilized by excluded volume effects were performed on test macromolecules in presence of polysaccharides such as Ficoll 70 or Dextran to mimic the crowding of the cytoplasm. Fluorescence measurements [6] or enzymatic tests [7] that serve to measure protein activities showed protein stabilization (the concentration of unfolded agent needs to be higher to unfold the protein or the denaturation temperature is higher...). Such stabilization effects were also observed by using circular dichroism [7] that quantifies the amount of secondary structures of the proteins. All these experiments have unambiguously stressed protein stabilization due to macromolecular crowding, but the physical mechanism remains unknown because they cannot capture directly the change in conformation of the unfolded state and observe the decrease of its radius of gyration that is associated with the compression of the chain.

Small-angle neutron scattering (SANS) is the ideal tool to evidence such mechanism because contrast matching methods allow to suppress the macromolecular crowding agent signal and to observe the scattering of very dilute species in a huge fraction of agents mimicking the crowded cytoplasm

To achieve this, either the protein or the crowding agent must be deuterated to get enough contrast. As the protein in the unfolded state has a Gaussian chain like conformation [8], and protein deuteration is tricky (especially in rather high quantity as is useful for neutron scattering) we decided to first study the evolution of a deuterated polymer in rather good solvent in water: the polyethylene-glycol (PEG) in order to observe the compression. To obtain information on one single chain we need, for each volume fraction in macromolecular crowder, to extrapolate the information to zero polymer concentration (Zimm plot).

We have shown that macromolecular crowding strongly reduces the radius of gyration of the D-PEG (fig. 2). For macromolecular crowder and polymer chain of similar radii of gyration, at a mass fraction of F70 of  $\Phi_m \sim 0.27$ , the reduction of  $R_g$  of the chain is 30% compared to that measured in water [9].

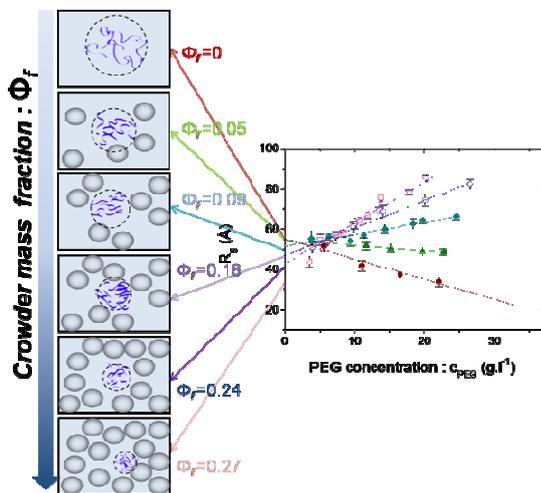


Fig. 2: Zimm plot of the evolution of the radius of gyration of a Gaussian chain when increasing the mass fraction of the crowding agent (Ficoll) up to  $\Phi_c = 0.27$ .

As can be shown on fig. 2, the crowding not only affects the radius of gyration of the chain but also the protein-protein interactions (slope of the curves).

For a homopolymer like the PEG, we observe a strong compression of the chain but not its total collapse, which would have a too large entropic cost.

We started a second step in order to mimic the situation in biological systems, by performing similar experiments on deuterated proteins. We aim to correlate the destabilization of the unfolded state with the stabilization of the protein observed with biological techniques. One can speculate that, for a heteropolymer like an amino-acid chain a total collapse is possible. For such chains the short range interactions (hydrogen bonds, hydrophobic interactions ...) would bring a sufficient enthalpic gain to compensate for the entropic lost caused by the collapse, and hence significantly stabilize the native state of the protein.

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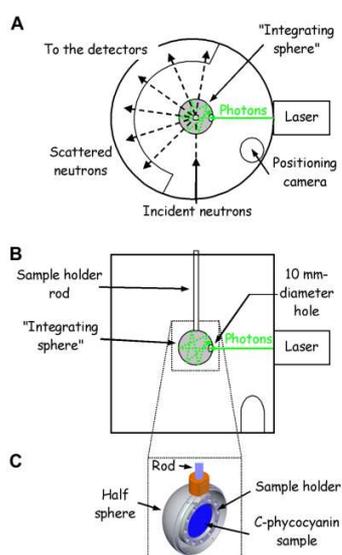
## COUPLING OF LASER EXCITATION AND INELASTIC NEUTRON SCATTERING MEASUREMENT

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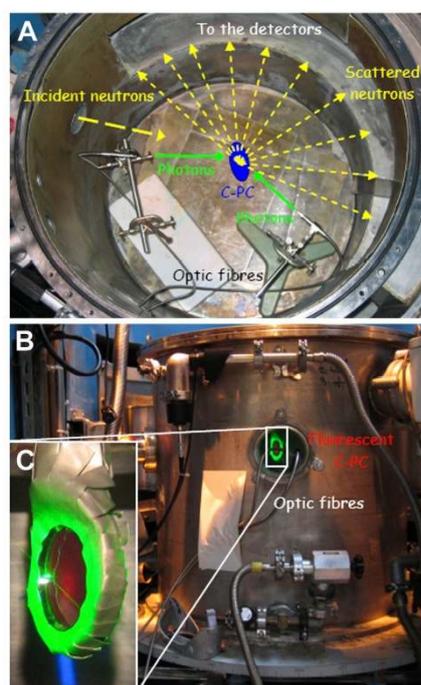
Light-harvesting antennae are pigment-protein complexes involved in light-absorption and excitation energy transfer (EET) to the so-called “reaction center” complexes, where the photochemical processes of photosynthesis take place. C-phycoerythrin (C-PE) is one component of the phycobilisome, the light-harvesting system of cyanobacteria [1]. In phycobilisomes, EET is a highly efficient key event [2-3], where light-induced dynamics of the antenna pigment/protein complexes may play a role [4-7].



**Fig. 1** Schematic experimental setup of the “laser/neutron” coupling on the time-of-flight MIBEMOL neutron spectrometer : top (A) and side (B) views. The neutron and photon beams are perpendicularly oriented. Homogeneous illumination of the protein is provided by an “integrating sphere” (a spherical and hollow Al chamber) containing the sample holder (C). Photons aim on the sample through a 10 mm-diameter hole in the sphere. Height and orientation positioning of the sphere inside the sample well is checked using a camera.

To detect fast and localized protein motions at room temperature, related to light absorption and EET in the isolated pigment/C-PC protein system, as well as to investigate the timescale of such dynamical changes, we developed a new experimental setup on the time-of-flight spectrometer MIBEMOL (LLB, France). This new “time-resolved” method was technically challenging, since we had to synchronize pulsed inelastic neutron scattering measurements with repetitive light excitations. The principle of the experimental setup is shown in Fig. 1. The laser was fixed outside the sample well of MIBEMOL

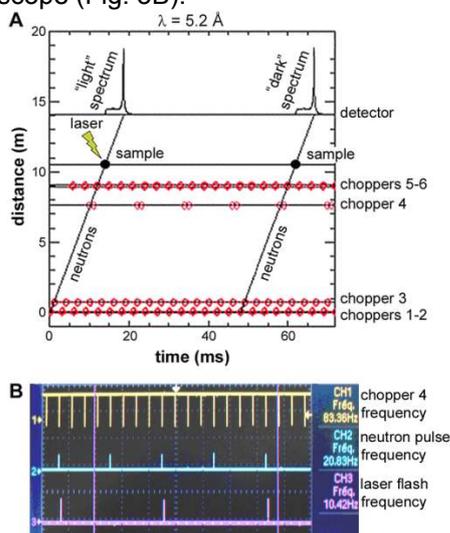
spectrometer, perpendicularly to the neutron beam and in front of the detectors (Fig. 1A). The photon beam was directly aimed on the sample through the 10 mm-diameter hole of an “integrating sphere” (Fig. 1, B and C), without any additional optical device. We used a spherical and hollow aluminium chamber, containing a highly reflective and diffusing interior coating, to illuminate the sample inside uniformly (Fig. 1C).



**Fig. 2** Top (A) and side (B) views of the sample environment on MIBEMOL neutron spectrometer during illumination experiments on C-PC. To check that the protein solution was entirely illuminated, we made direct observation of the sample, without the sphere, by using optic fibres. Inset (C): fluorescence of the C-PC (red colour) in the sample holder with the green laser beam reflected on the cadmium mask.

The “integrating sphere” was positioned in front of the laser head using a camera located inside the sample well. First, we positioned and aligned the experimental setup without the “integrating sphere” but using optic fibres illuminating both sides of the sample container (Fig. 2). In that configuration, we were able to check that the sample was entirely illuminated, since fluorescence was homogeneously emitted from the surface of the sample (Fig. 2C).

MIBEMOL data acquisition system has been modified to synchronize the laser excitation flashes (6 ns duration) with the neutron pulses ( $\sim 87 \mu\text{s}$  duration) at the sample position, in order to perform “time-resolved” measurements. Successive neutron scattering measurements (few hours duration), with or without the laser, gave access to the “light” and “dark” spectra respectively. “Light” and “dark” spectra were separated by 50 ms in the case of a 10 Hz laser frequency (Fig. 3). The quasi-simultaneous process of such “light” and “dark” relative measurements and their evolution as a function of time could be compared to measurements performed in a “double beam” spectrometer (Fig. 3A). The flash lamp pumping and the laser triggering have been controlled by a special device and checked all along the experiment by an oscilloscope (Fig. 3B).



**Fig. 3** A): Diagram of the chopper velocities on MIBEMOL spectrometer for the “laser/neutron” setup, at  $\lambda = 5.2 \text{ \AA}$  configuration ( $\sim 140 \mu\text{eV}$  resolution). The incident and scattered neutrons are represented by a solid line and typical TOF spectra have been drawn at the detector. The “light” spectrum was obtained when the beginning of the neutron test pulse ( $\sim 87 \mu\text{s}$  duration) was synchronized with the laser flash, symbolized by a lightning symbol ( $\sim 25 \text{ mJ}$ , 10 Hz,  $\lambda = 532 \text{ nm}$ , 6 ns duration). The “dark” spectrum, resulting from each neutron test pulse between two laser flashes, was considered as the reference without laser. B): Oscilloscope screen showing the neutron pulse frequency at chopper #4 (80 Hz, channel #1), the recorded neutron pulse frequency (20 Hz, channel #2), and the laser flash frequency (10 Hz, channel #3).

This procedure of alternating “light” and “dark” measurements was novel and eliminated many spurious effects that could occur in the sample during the experiment (e.g. positioning of the sample holder or undesired laser-induced heating of the sample). For the present study, no time delay has been introduced between the beginning of the laser flash and the beginning of the neutron pulse. However, for future

measurements, a time delay between the laser flash and the neutron pulse can be introduced.

Measurements have been performed at constant temperature (298 K) and pressure. The light source was a 6 ns pulsed Nd:YAG laser, at  $\lambda = 532 \text{ nm}$ , with maximum frequency and energy of 14 Hz and  $\sim 25 \text{ mJ}$ , respectively. We chose a circular Al sample holder surrounding 50 mm diameter sapphire glasses with a 0.5 mm sample thickness. Such dimensions optimized the intersection with the neutron beam. Besides, the elastic intensity level from the Al/sapphire sample holder was very weak and, in the Q-window of our measurements, no Bragg peak was observed for sapphire glass, nor Al. The Al “integrating sphere” surrounding the sample holder was also almost “transparent” for neutrons.

We obtained a set of energy-transfer spectra measured at different scattering angles ( $0.5 < Q < 2.1 \text{ \AA}^{-1}$ ). We used an incident wavelength of  $5.2 \text{ \AA}$  to have the maximum neutron flux with a reasonable resolution ( $\sim 140 \mu\text{eV}$ ). Such a resolution gives access to motions on 0.2-20 ps timescale. Data collection times were about 10-20 h to obtain sufficient statistics. In the case of C-PC, the incoherent dynamical structure factor was not significantly altered by light; 2 modes within the vibrational density of states were slightly increased upon illumination. New applications using other stimuli on sample like electric or magnetic excitations are now under development. We have in particular already undertaken to probe the dynamics of a polymer electrolyte (lithium salt embedded in a polymer matrix used as electrolytes in batteries) submitted to an electric field, so far in the 1 Hz-30 MHz range.

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## NEUTRONS AND WATER STRUCTURE: THE HEAVY WATER BRIDGE

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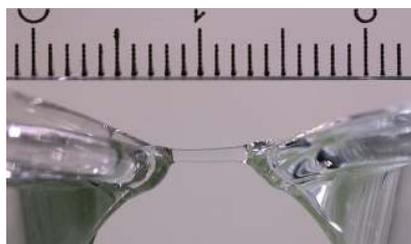
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In 1893 Sir William Armstrong reported a remarkable experiment: if a high voltage is established between two wine-glasses filled to the brim with pure water and connected by a cotton thread, a rope of water is formed and remains suspended between the lips of the two glasses. This "water bridge" can subsist for a few seconds even once the thread has been removed [1].

Recently, a group of physicists from Graz University, Austria [2] succeeded on establishing a bridge (without any thread!) between the two glasses under a voltage of 20 kV. The length of the bridge may reach values larger than 1 cm, its diameter is of the order of a few mm and the lifetime currently exceeds 1 hour. Under the effect of the electrical field, the local temperature increases and reaches 60° C, a temperature sufficient to break the bridge.



Water bridge (5mm length, 1mm diameter) between two glasses filled with heavy water, under a voltage of 15 kV.

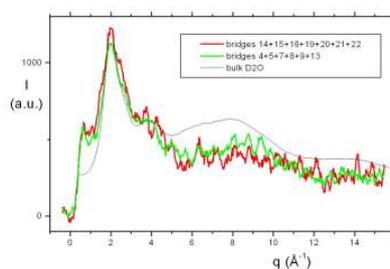
This short-lived structure due to the intense electrical field is not fully understood, although it results certainly from the unique properties of liquid water, its chemical composition, shape of the molecule and intermolecular bonds. Indeed, the role of hydrogen bonds on the general behaviour of pure water is always the object of active research because of both the fundamental aspects and importance in other domains, namely in Biology.

In order to enlighten the problem, a first measurement of the molecular structure of water inside the bridge has been performed by neutron scattering at the Laboratoire Léon Brillouin

(CEA/CNRS, reactor Orphée) at the diffractometer 7C2 for liquids and amorphous materials [3].

Generally speaking, the scattering of neutrons by nuclei gives precise information about the average local arrangements of atoms and molecules of a

liquid sample. The optimisation of the experimental conditions implies the utilisation of deuterated samples, i.e., in this case, of heavy water (D<sub>2</sub>O). This isotopic substitution has no effect on the structure of the liquid. Consequently, for the first time, a "heavy water bridge" has been successfully established between two water glasses filled with heavy water. One of the difficulties to overcome was the necessary purity of the D<sub>2</sub>O sample.



Neutron scattered intensity of the water bridge compared to that of bulk water (black line). The important differences observed at large  $q$  ( $> 4 \text{ \AA}^{-1}$ ) are not significant because they are due to the shadow of the two glasses. The first structural peak at  $2 \text{ \AA}^{-1}$  is not shifted, showing that both density and intermolecular distances are not different from those of the common liquid. Instead, the intensity at small  $q$  ( $\sim 1 \text{ \AA}^{-1}$ ) is larger what suggests complementary small angle experiments, necessary to the correct interpretation of this result [3].

The first structural peak of the scattering patterns (see figure) shows that no important differences exist between common bulk water and the water in the bridge. This first conclusion means that both density and inter-molecular distances are not different inside the "water bridge" as compared to bulk water. Although expected, this result eliminates some hypothesis suggested to explain the observed effect,

namely the eventual presence density gradients or collective molecular orientations.

Instead, the scattering at small momentum transfer ( $< 2 \text{ \AA}^{-1}$ ) is different from that of bulk water. Its intensity is larger, what is explained likely by the presence of nano bubbles of gas that reinforce the effects due to the surface tension. One cannot exclude as well, the possible local arrangement of water molecules within tiny domains analogous to those existing in magnetic structures [4].

Even if a definitive explanation of the physics of the "water bridge" remains unknown, it was shown that the experiment can be achieved with heavy water and

that the structure can be measured with reasonably good accuracy, demonstrating once more the high performance of neutron scattering on the study of substances containing light atoms. Other experiments focusing on the small angle domain are under preparation and should yield complementary results.

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## HIGHLIGHTS

La<sub>1.2</sub>Sr<sub>1.8</sub>Mn<sub>2</sub>O<sub>7</sub>. *Nature Materials* 8 (10):798-802.

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## Publications

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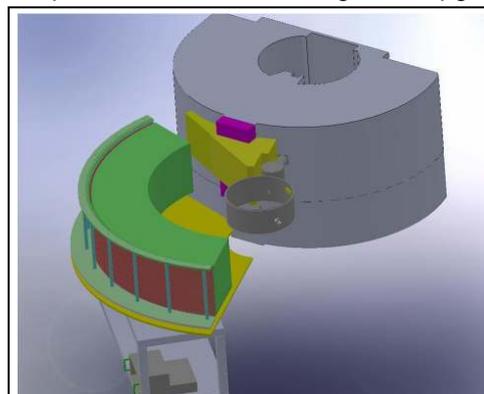
# **INSTRUMENTATION**



## INSTRUMENTATION PROGRAM CAP2015

**CAP2015** is the instrumental program of the LLB. This program will enable our instruments to stay at the best international level compared to other neutron sources. Setup within a constrain budget, all upgraded or new apparatus should be on line by 2015. This will enable the LLB to reach the end of the decade with the state of the art spectrometers. At that time, the European neutron scattering landscape will certainly be changed with the arrival of ESS and the predicted fuel evolutions for the reactors. The objective of the CAP2015 instrumental program is to give to the National neutron scattering community the instruments it deserves to be ready to assure its future at the ESS.

If this program is mainly devoted to instruments, some components of the reactor itself will also follow a continuous refurbishment process. After ageing with irradiation we have to replace some of our beam tube thimbles. Up to 2016, this program will enable us to improve the in pile part of most of our neutron beams. For the same reason, both cold sources will be replaced in 2011. Their geometry will be slightly changed to increase the flux available at large wavelength.



Schematic view of the new 7C2 diffractometer



VIP delivered at LLB

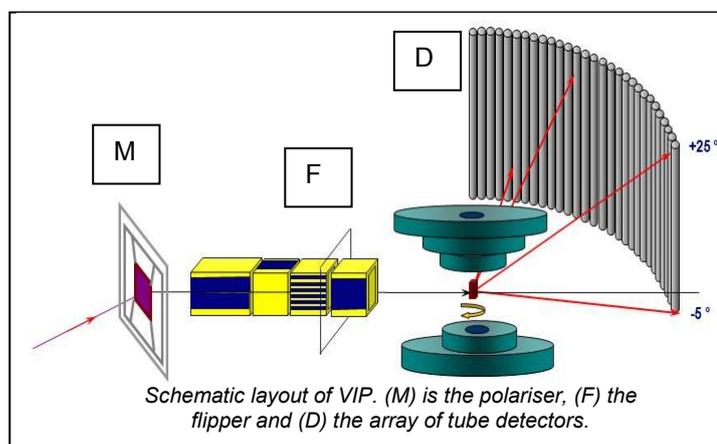
### Project management structure

Nearly half of our instruments (10 out of 22) will benefit of our Cap2015 program. Ensure the success of this program at reasonable costs is a challenging commitment for our laboratory. This is why we have reorganized our staff by creating an instrumental development group which main objective is the accomplishment of this program. This will be accompanied by setting a strong project management structure that should ensure the delivery of milestones defined within CAP2015 in time. Some of the CAP2015 projects are presented below.

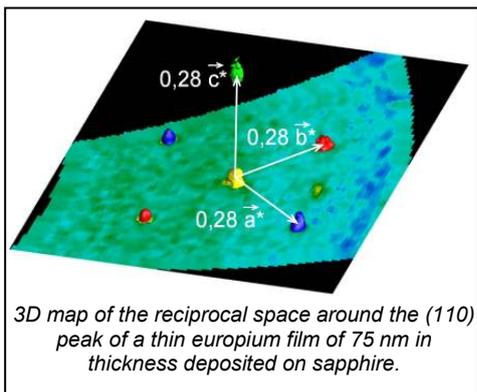
### Hot neutrons spectrometers

At present there are only two hot sources available in the world, including the one at the LLB. Since hot neutrons provide unique access to large Q, it is our duty to equip our hot beam channels with the state of the art spectrometers. Therefore two of them 7C2 and 5C1 will benefit of major upgrade. Our liquid and amorphous diffractometer 7C2 will be equipped by new detector bank. Localization electronics, 256 position sensitive detectors and mechanics have been purchased and should be delivered by the end of the year. With its large, high efficiency detectors, the new 7C2 should have its counting rates increased by a factor of more than 20 in 2011.

The polarized neutron diffractometer for single crystals 5C1 will also be replaced by **VIP** (Very Intense Polarized neutron diffractometer). All its mechanicals part, radial collimators and 64 position sensitive detectors have been delivered recently to the LLB and installed on the test station G44. Compared to the current 5C1, VIP will present a large detection area which enables fast acquisition on large crystals or measurements on very small samples.



Schematic layout of VIP. (M) is the polariser, (F) the flipper and (D) the array of tube detectors.

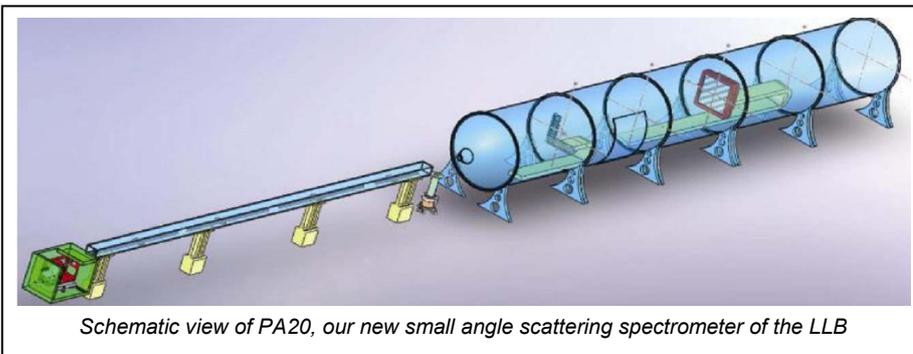


**Cold neutrons spectrometers**

Neutrons being very sensitive to hydrogen, they are extensively used for structure determination and dynamic studies in soft matter. This is due to the ability of labeling hydrogenated molecules with deuterium. In this domain, three projects of CAP 2015 should be mentioned.

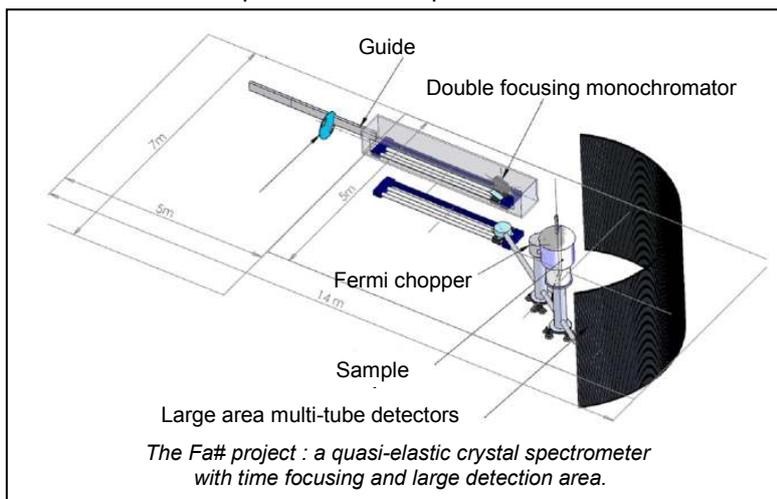
This year, a new **Very Small Angle Neutron Spectrometer**, named **TPA**, has been opened to first users. Its original conception involves an Image Plate detector with a high resolution of 0.15\*0.15mm<sup>2</sup>, and one multi-beam pinhole collimator which more than 350 tiny neutron beams converging on to the detector. Such high resolution equipments enable measurements down to Q values of 2 10<sup>-3</sup> nm<sup>-1</sup> filling the gap between neutron and light scattering.

LLB operates four standard Small Angle Neutron Spectrometers. Compared to similar instruments installed at other neutron sources, the four LLB instruments are shorter. As a consequence the same experiment needs more configurations to be performed. This takes longer time. The construction of a new spectrometer **PA20** with a 20 m collimation length has been approved. The instrument will be located at the end of guide G5 and re place the current SANS instrument PAXE. Equipped with large surfaces, high resolution, high efficiency XY detectors build in collaboration with ILL, the counting rate and Q range covered in a single measurement by this spectrometer will be increased by a factor of two compared to our current SANS. Focusing lenses will also provide additional increase of flux in some specific configurations.



Equipped with polarized neutrons, the new spectrometer will enable the study of magnetic systems. The general design of the instrument is completed and the purchases of first elements will take place in 2010. PA20 should be delivered in 2013.

After almost 30 years of successful operation, the performance of Mibémol, the LLB-Orphée Time-of-Flight (ToF) machine, is now challenged by the new generation of ToF instruments. For this reason, LLB is undertaking the design of a new ToF machine **Fa#**. Since **Fa#** will be the only ToF spectrometer at the LLB it must be competitive both in quasi-elastic and inelastic measurements, for disordered systems (soft matter) but also for crystalline samples (magnetism and solid state physics).

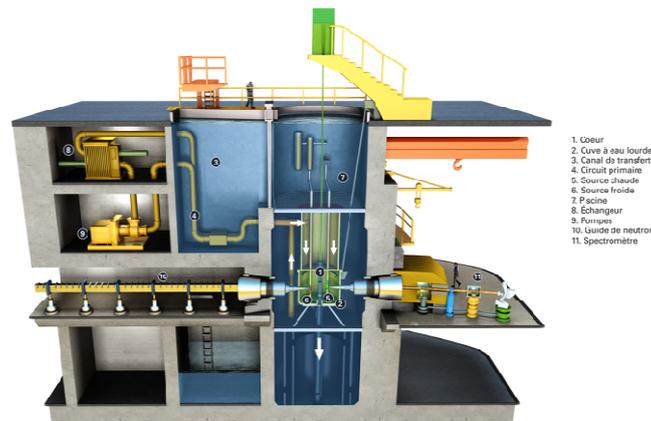


Therefore Fa# is planned to achieve high flexibility to map energy resolution in the 15 to 500 μeV range on an extended Q domain from 0.05 to 5 Å<sup>-1</sup>. The spectrometer should meet these criteria with the highest flux achievable. A direct geometry so-called “hybrid” set-up, similar to the Focus spectrometer at PSI, has been chosen. Variable Guide/Monochromator and Monochromator/Sample distances will make it possible to switch easily from time focusing to monochromatic focusing modes. This will be a key

feature to reach the best flux vs. resolution balance.

## ORPHEE REACTOR

### A- General presentation



*Schematic view of the Orphée reactor*

The Basic Nuclear Installation n°101, located at CEA Saclay Centre, comprises the experimental 14-MW thermal power ORPHEE reactor and the experimental buildings used by the teams of the the Léon Brillouin Laboratory (LLB).

The ORPHEE reactor was built between 1976 and 1980 as a, national-based, sister facility of the High Flux Reactor (RHF) of the Laue Langevin Institute (ILL) in Grenoble. The decision to build the reactor stemmed from the continuous expansion of European and international neutronic research in the 70s.

The design and construction of the ORPHEE reactor were managed by CEA and Technicatome (presently AREVA TA). ORPHEE design directly benefited from the safety, construction and operation feedback of previous generation reactors like RHF and was contemporary to the development of the PWR equipment program in France.

The reactor first went critical on december 19th 1980. The designers' projections purposely resulted into a moderate investment cost - moderate operation cost facility, ensuring both reliable operation and good safety record. The reactor has been operated circa 5300 days between start up and the end of the year 2008.

The reactor safety case was reassessed twice since 1980 (second reassessment file submitted to the French regulatory body on march 31st 2009). Due to its original design, the reactor is submitted to very limited ageing mechanisms (all of them manageable) and the initial reactor safety features remain in good adequation with up-to-date safety practices.

Since 2006, the start up of the neighbouring SOLEIL synchrotron on the Plateau of Saclay and the massive undergoing R&D development plan of the Plateau of Saclay have constituted the basis of a top-level 21st century integrated complex for the exploration of matter and physical science south of Paris.

### B- Reactor technical characteristics

ORPHEE is a « pool » type reactor.

The 14-MWth compact, light-water moderated, core provides up to  $3 \cdot 10^{14}$  n/cm<sup>2</sup>/s thermal flux in the surrounding heavy water reflector tank. Core life cycle duration is 100 EFPD. Core flow velocity is 7.5 m/s. The core and the heavy water tank are immersed in a pool filled with demineralised light water. This ensures radiological shielding and facilitates handling from above the pool.

The reactor hosts 9 horizontal channels (steaming from the heavy water tank and feeding 20 neutron beams) and 9 vertical channels (steaming mostly from the heavy water tank, and comprising 4 pneumatic channels for activation analysis and 5 irradiation pits for radio-isotope and other industrial productions).

The heavy water tank is equipped with three local moderators : two cold sources (liquid hydrogen at 20K) and one hot source (graphite at 1400 K). These provide neutrons of respectively lower and higher energy.



*Operation above Orphée's pool*

On this basis, experimental users can benefit from 8 thermal neutron beams, 8 ultra-cold neutron beams, 4 hot neutron beams.

ORPHEE neutrons are supplied to 26 experimental areas (including 19 areas with cold neutrons). The experimental areas are located around the reactor, either in the reactor building or along the neutron guides of the guides hall. One experimental area with specific radiological shielding has been designed for industrial neutronography (mostly used by the aeronautical or space industry, and in particular the European Ariane launchers).

### C- Reactor safety

The safety design of the reactor is based upon the defence-in-depth principle in order to ensure permanent control of the three main safety functions: reactivity control, residual heat removal and containment of radioactive material.

The design includes the following elements:

- Permanent reactor monitoring by a safety system using 3 completely independent channels. If necessary, 2 channels out of 3 will automatically trigger the reactor emergency shutdown (fast control rod drop by gravity).
- Once the reactor has been stopped, the residual power is removable by purely passive natural convection between the core and the reactor pool.
- The core and the core cooling circuit are located in the reactor building made of reinforced concrete. The lower section of the building is surrounded by an outer tank which purpose is to collect leaks which may occur to each pipes that exit the building.

Three impervious, resistant and stand alone barriers are placed between dangerous products and the environment. These barriers are : the reactor fuel cladding, the reactor main cooling circuit plus the reactor pool, and the reactor building.

During normal operation, the reactor building is maintained at lower pressure than outside. The reactor building has also been designed to sustain any accident which could occur to the reactor.

The supervision of the impact of the installation onto the environment is included in the Saclay Centre environmental monitoring program.

### D- Operation teams



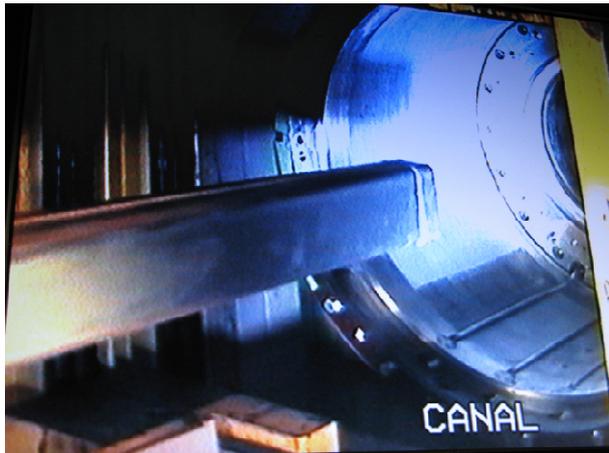
*The control room of Orphée reactor*

The ORPHEE reactor is operated by the Nuclear Energy Division of CEA (CEA/DEN), acting on behalf of the Physical Science Division of CEA (CEA/DSM), for the benefit of the experimental users of the Léon Brillouin Laboratory and of their French, European or non-European scientific visitors.

ORPHEE + LLB operations are performed as a joint CEA and CNRS undertaking and submitted to a joint annual review by the Physical Science Division of CEA and CNRS.

ORPHEE operation is performed by a 60 people team, about half of which work on a shift basis. This facility benefits from the support of the radiological protection teams and security teams of CEA Saclay Centre.

More generally, the facility is located inside an integrated nuclear research Centre which was an historic cradle of French reactor technology and nowadays hosts up-to-date theoretical and support teams in the fields of core operation, chemistry, nuclear materials and mechanics.

**E- 2008 review**

*Extraction of irradiated 4F thimble in 2008*

The year 2008 was characterised by 189 days of operation, corresponding to a 95% availability factor. Specific ten-yearly operations were carried out, notably replacement of most of the reactor batteries and inspection of most of the pressurised equipments.

The 2008 summer outage enabled the replacement of the 4F horizontal channel inside the heavy water tank. This operation, which took place in the framework of the management of irradiated components, was a first of kind since reactor start up and followed a preparatory operation during the year 2006 summer outage.

The irradiated 4F thimble was extracted, then immediately split into sections. The new thimble was put in place and has given satisfaction to experimental users since the restart of september 2008. The 4F operation also demonstrated the technical capability to perform the further replacements of irradiated horizontal channels which are scheduled from 2011 on.

As regards further heavy maintenance and upgrading activities: the contract to manufacture the replacement cold sources of the reactor (to be put into operation before 2011) and the contract to re-manufacture the electronic processing devices of the radiation protection system (to be put into operation at summer 2009 and then summer 2010) were concluded.

The other main issue of the year 2008 was the preparation and the completion of the work on the 2<sup>nd</sup> safety reassessment of the reactor.

To this aim, a major non-destructive inspection programme was carried out all along the year 2008 (eg. inspection of the natural convection check valves upstream of the core).

In parallel, substantial safety study work was carried out in association with, and with notable support of, the CEA specialists from the Nuclear Energy Division technical departments of the Saclay Centre.

The resulting work underwent CEA internal review and control at the end of the year 2008 and beginning of the year 2009. Results of these actions confirmed the very satisfactory technical and safety condition of the reactor after 28 years of operation.



# **FIGURES AND FACTS**



## IGOR GONCHARENKO: A PIONEER IN HIGH-PRESSURE NEUTRON DIFFRACTION.

I. Mirebeau and J.-M. Mignot

*Laboratoire Léon Brillouin, CEA/CNRS  
CEA-Saclay, France*



Igor Goncharenko was born in Novosibirsk (USSR) on 2 June 1965. He disappeared in a scuba diving accident in the Red Sea, near to Hurghada (Egypt), on 4 November 2007. After completing high school in Novosibirsk, Igor went to Moscow in 1982 and started engineering studies. He graduated in Advanced Studies at the Institute of Physics and Technology in Moscow in 1988. He prepared his thesis on the 'crystal structure and equation of state of hydrides under pressure' during the years 1988–1991, and then became an associate researcher at the Russian Scientific Centre 'Kurchatov Institute' of Moscow. When Igor started his Ph.D. work, the Kurchatov Institute had already a high expertise in high-pressure neutron diffraction, developing diamond anvil cells adapted to neutron beams. The main limitation of those devices was the very small sample size, which restricted the neutron studies to samples with high-scattering cross-sections. During the years 1986–1990, Igor developed new sapphire-anvil pressure cells, reaching 7–8 GPa with larger sample volumes. This technique, published in 1988, was successfully used later on at several neutron centers in Russia, France and Japan.

Igor started to collaborate with the Laboratoire Léon Brillouin (LLB, Saclay, France) in 1991, when an agreement was signed between Jean Rossat-Mignod (LLB) and Victor Somenkov (KI), to combine the Russian expertise in high pressures with the potential of a high-luminosity neutron source. For several years, Igor spent about half of his time in France and LLB through temporary contracts before obtaining a position at the Centre

National de la Recherche Scientifique (CNRS), the main French research organization in 1995. He obtained his second degree of the Doctorate in Physics (the highest scientific diploma in Russia, which gives the right to direct scientific studies) in 1999.

High-pressure diffraction requires very small samples, down to 0.0001 mm<sup>3</sup>, and therefore much smaller than the samples usually measured in neutron experiments. While at LLB, Igor developed a specific spectrometer, dedicated to high-pressure neutron powder diffraction, by implementing focusing devices with supermirrors on a diffractometer already equipped with a multidetector, and installed on a cold neutron source. These devices increased the neutron flux by one order of magnitude. A specific shielding was also implemented to reduce the background. These achievements allowed him to perform neutron experiments under pressures up to 51 GPa, a world record established in 1997 which still holds today. Igor also developed specialized high pressure cells, available for single crystals and powders, which could be combined with very low temperatures (down to 50 mK) and high magnetic fields (up to 7 T). In the last few years, he designed a hybrid high-pressure cell, which he used both for X-rays and neutrons, in combined pioneering experiments performed both at the synchrotron source of the ESRF and at the LLB.

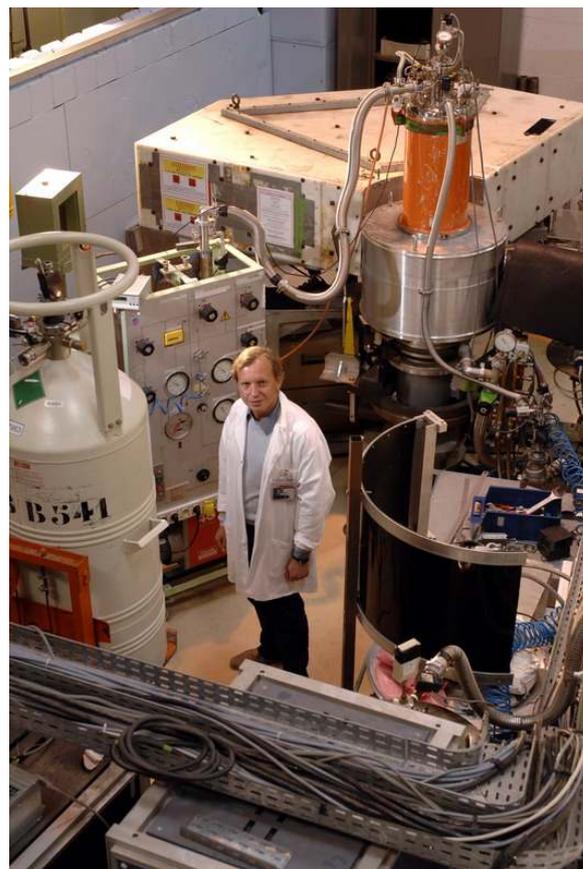
Building on these technical achievements, Igor performed numerous pioneering studies of crystal and magnetic structures under high pressures, bringing answers to long-standing questions in solid-state physics.

He directed, or co-directed, several Ph.D. works on these subjects. In the field of magnetism, he studied model ferromagnetic and antiferromagnetic Heisenberg systems like EuS, EuSe or MnO, as well as heavy-fermion and mixed-valence compounds. He also investigated frustrated magnets, like the Laves phases and their hydrides, which show a transition from localized to itinerant magnetism, tuned by pressure. In the geometrically frustrated pyrochlores called ‘spin liquids’, ‘spin ices’ or ‘spin glasses’, he discovered novel types of orders under pressure. He determined high-pressure magnetic structures in solid oxygen and the nature of its mysterious epsilon phase, a precursor to superconductivity. His contribution to crystal structures was also of great significance, with the recent determination of the broken-symmetry phase of solid deuterium, using both neutron and X-ray diffraction, and the structure of a pressure-induced hydrogen dominant metallic state in an aluminium hydride under extreme pressure, in the megabar range.



*High pressure cells developed by Igor Goncharenko for neutron/X-ray scattering.*

The work of Igor Goncharenko was widely recognized and appreciated among the international community, both in neutron and high-pressure science. This is shown by the numerous invited talks he presented in the last few years, as well as by his many articles in top-level journals (two letters in Nature and a dozen letters in Physical Review Letters, Europhysics Letters and the Physical Review, among others). In 1996, Igor was elected a member of the High-Pressure Commission of the International Union of Crystallography, and re-elected in 1999. He also took part in the organization of the scientific programs of several International Conferences, in France, Russia, United States, Japan or Great Britain.



*Igor Goncharenko in front of G61 in September 2007.*

Only a few people knew that Igor was not just a great scientist, but also a passionate scuba diver. Actually, he loved sports challenges almost as much as scientific ones. Over the years, he acquired professional skills and, when he disappeared in the Red Sea, he was approaching the amazing depth of 200 m (the 1999 world record). Igor was an outstanding experimentalist, and a researcher with rare tenacity and tremendous working power. When working with him, scientific miracles started to seem natural. He always applied great care to all his activities, whether scientific, technical or sporting, but never took himself too seriously. Everyone remembers his simplicity and sense of humor. His death was a terrible shock for his wife Lada, his parents Nina and Nicolai and family, and a big loss for his friends and colleagues and for the scientific community as a whole. He will be missed by all of us.

*High Pressure Research*  
 Vol. 28, No. 2, June 2008, 141–142  
 e-mail: [isabelle.mirebeau@cea.fr](mailto:isabelle.mirebeau@cea.fr)

## SCIENTIFIC AWARDS

J-M Zanotti, K. Lagrenée, D. Lebeugle



The 21<sup>st</sup> of October 2008, at the "Institut de France", Jean Salençon, Vice-President of the National Academy of Sciences, handed over the "Grand Prix Michel Gouilloud Schlumberger" to **Jean-Marc Zanotti**. This prize awards outstanding research from a young (<40 years-old) researcher in the domains of Earth Sciences (Geology or Geophysics) applied to Oil Industry. The award was granted for Jean-Marc Zanotti's works on the **dynamics of confined/interfacial water**.



In 2009, the jury of the SFN Thesis Prize chaired by M. Leduc, President of the SFP, has distinguished **Karine Lagrenée** for her thesis defended in October 2008 at the Université Paris Sud. This thesis, co-directed by M Daoud (SPEC) and J-M. Zanotti (LLB), was dedicated to the "**Dynamic study of Polymers under quasi-uniaxial confinement**".

The 2008 SFN thesis prize was awarded to **Delphine Lebeugle** for her work on the "**Coupling between ferroelectric polarisation and magnetic structure in BiFeO<sub>3</sub> single crystals**" (SPEC, CEA-Saclay). The prize was officially delivered on May 28 during the half day JDN Conference. The multi-disciplinary jury was chaired by Jean Pierre Hanssen, Liquid State theorist, currently at Cambridge University.

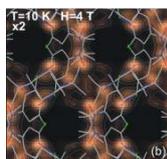
## MEETINGS AND WORKSHOPS

### 10th SXNS conference (2-5<sup>th</sup> July 2008)

The 10th SXNS conference was jointly organized by SOLEIL, the French synchrotron source, and the Laboratoire Léon Brillouin, the French neutron source from 2nd to 5th of July, 2008 nearby Paris. This series, started in 1989 in Marseille, brings together researchers studying surfaces and interfaces of solid, liquid, biological and soft matter via neutron or X-ray (either hard, soft, or EUV) scattering techniques.



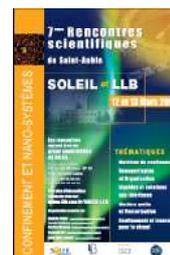
### Position Sensitive Detectors for Single Crystal Diffraction (PSD4SC, 12-14<sup>th</sup> Nov. 2008)



This first Discussion Meeting concerning data collection and treatment from "Position Sensitive Detectors for Single Crystal Diffraction (PSD4SC)" was held from 12th Nov to 14th Nov 2008 at Les Loges en Josas. The discussions, led by A. Goukassov, focused on Data Treatment and storage, involved representatives from all major European neutron centres involved in Single Crystal Diffraction Studies.

### 7<sup>èmes</sup> Rencontres Scientifiques de Saint-Aubin (12-13<sup>th</sup> March 2009)

In the context of collaboration between research infrastructures of the plateau de Saclay, LLB and the Soleil light source hold since 2005 thematic meetings around the use of synchrotron/neutron radiation for the study of condensed matter. The 7<sup>th</sup> LLB-Soleil meeting, in 2009, was devoted to the contribution of neutron and synchrotron techniques to the study of nanosystems formed by ions, molecules and molecular assemblies at the interfaces or confined at the nanometric scale.



### XVIII Int. Conf. on "Horizons in Hydrogen Bond Research"(14-18<sup>th</sup> Sept. 2009)



The conference was organized around **3 main topics: structure, dynamics and function of molecular and supramolecular objects** of increasing complexity (small building blocks, biological systems and materials). The congress focussed on the complementary of relevant experimental and theoretical techniques (time-dependent UV/VIS and vibrational spectroscopy, microwave, X-ray and neutron crystallography, inelastic neutron scattering, liquid and solid state NMR, but also theoretical chemistry, numerical simulations; photoinduced processes, isotope substitution).

### Sample Environment JRA progress meeting (29-30<sup>th</sup> Oct. 2009)

The first meeting of the *Sample Environment* JRA (Joint Research Activity, part of the NMI3 Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy - EU Framework Programme FP7) was organized at the LLB (J.-M. Mignot and B. Annighöfer) on October 29-30, 2009. It gathered 18 participants from six leading international neutron facilities. Presentations and discussions covered the main topics of the project : development of innovative high-pressure cells (for both inert gases and hydrogen), high-temperature furnaces, and gas absorption control systems.



## TRAINING AND EDUCATION

### *HERCULES Courses (23-27 Mar. 2009)*

This five-weeks course is designed to provide training for students, postdoctoral and senior scientists from European and non-European universities and laboratories, in the field of **Neutron and Synchrotron Radiation for condensed matter studies** (Biology, Chemistry, Physics, Materials Science, Geosciences and Industrial applications). In addition to lectures, all full time participants carry out practicals or tutorials in the European Facilities (ILL and ESRF) at Grenoble and, French facilities (SOLEIL and LLB) in Paris area.

<http://hercules.grenoble.cnrs.fr>

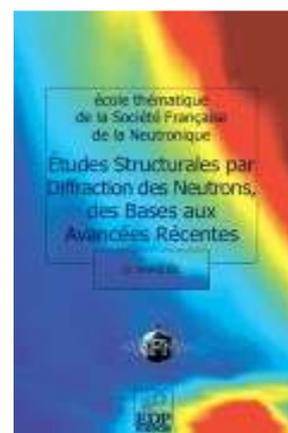


### *FANs du LLB 2008 (2-5 Feb. 2009)*

After a late restart of the Orphée reactor in late 2008, the FANs du LLB 2008 were postponed to 2-5 February 2009. During this session, the 18 trainees focused mainly on Powder Diffraction and Small Angle Neutron Scattering.

### *FANs du LLB 2009 (23-26 Nov. 2009)*

The second session of 2009, from 23 to 26 November, welcome 25 trainees from various Universities and Research Institutions. During 4 days, they add a first opportunity to discover and practice Single Crystal and Powder diffraction, Small Angle Neutron Scattering, Inelastic scattering...



### *Journées de la neutronique (23-29 May 2009)*

The 17th « Journées de la Neutronique » and « Rossat-Mignot days », co-organised by the LLB, ILL and LCVN, took place from 23 to 29 mai 2009 in La Grande Motte. This year, the meeting was untitled « Neutrons and Soft Matter ». As usual, all the teaching documents will be gathered in a book that will be available free of charges in electronic form on the web site of EDP Editions:

<http://www.neutron-sciences.org>.

**PRATICAL FOR UNDERGRADUATES STUDENTS**

*Students from Rennes-I University (left) and Bordeaux I University (right) at work in LLB.*

**Rennes-I University – European Master MaMaSELF (27 Nov. 2008)**

The visit, organized by W. Paulus from Rennes-I University, gathered about 50 students of 1<sup>st</sup> and 2<sup>nd</sup> year of Material Sciences Master. Together with them came also European students from the Master “Erasmus Mundus” MaMaSELF (Materials Science exploiting European Large Scale Facilities).

<http://etudes.univ-rennes1.fr/mamaself>).

**Bordeaux-I University – European Master FAME (26-28 Nov. 2008 & 25-27 Nov. 2009)**

On 2008 and 2009, the LLB welcome respectively 13 and 17 students of Masters M2 «Physics and Chemistry” and “Material Sciences” from Bordeaux-I University, and from European Master FAME. During their stay, they were initiated to Powder diffraction, Quasi-elastic Neutron Scattering and Inelastic Neutron scattering on G4.2, Mibémol and G4.3 spectrometers.

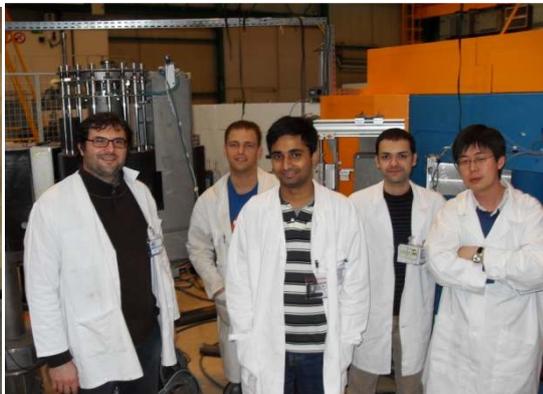
These practical trainings were supervised by doctoral fellows of LLB, and came in complement to lessons on neutrons scattering provided during the semester by A. Desmedt (ISM, Talence), M. Josse and A. Toulemonde (ICMCB, Pessac).

**Ecole Centrale (11-12<sup>th</sup> Dec. 2008 & 10-11<sup>th</sup> Dec. 2009)**

In 2008 and 2009, a dozen of students in 3<sup>rd</sup> year at the Ecole Centrale de Paris came at LLB for practical work on neutron scattering organized by B. Gillon (LLB) and J-M. Gillet (ECP). The themes proposed were “Powder diffraction” (G41, G. André), « Single crystal diffraction » (6T2, A. Bataille) and “Small angle neutron scattering” (PACE, J. Jestin).

**Paris VI : (14-17<sup>th</sup> Dec. 2008)**

In last December, LLB-Orphée welcome 4 students from the International Master of Applied Physics “NanoMat”, co-organized by Paris VI, Uppsala and Roma 3 Universities for practical work on Inelastic Neutron Scattering on G4.3, trained by V. Balédent, thesis student at the LLB.



*Students from Ecole Centrale (left) and Paris-VI University (right) at work in LLB.*

*DOCTORAL FORMATION AT LLB*

	Title of the Thesis	Funding	Examination/ University
 <b>CALLIGARI</b> Paolo	Adaptation of proteins to hot seabeds environment : The case of initiation factor 6, studied by molecular simulation and neutron scattering.	ILL/LLB	18/12/2008 (PARIS 6)
 <b>LAGRENE</b> Karine	Dynamics of electrolyte polymers under quasi uniaxial confinement	CEA	21/10/2008 (PARIS 6)
 <b>VINAS</b> Jérôme	Hybride (polymers-silica particles) materials: Synthesis and characterisation.	Region	29/02/2008 (AIX-MARSEILLE 1)
 <b>CHEVIGNY</b> Chloé	(Polymers-Grafted particles) composites : from synthesis in colloid solution to the study of macroscopic properties.	CEA	12/10/2009 (PARIS 11)
 <b>JOUAULT</b> Nicolas	Silica-Polymers nanocomposites : structure of charges, mechanical reinforcement, conformation of chains et evolution under deformation.	Region	03/11/2009 (PARIS 6)
 <b>LE CŒUR</b> Clémence	Influence of cytoplasmic molecular crowding on the stability and diffusion of proteins.	CEA	08/03/2010 (PARIS 7)
 <b>MAURER</b> Thomas	Magnetism of anisotropic nano-objects : magnetic and neutron studies of Co <sub>1-x</sub> Ni <sub>x</sub> nanowires.	CNRS-CEA	10/11/2009 (PARIS 11)
 <b>TENCE</b> Sophie	Magnetic structures and properties of hydrides and magnetocaloric compounds based on rare eaths.	Region	30/09/2009 (BORDEAUX 1)

## NEW PEOPLE



**Pascal LAVIE**  
Instrumentation Group  
3D-Modelisation  
of new spectrometers



**Sergey KLIMKO**  
Instrumentation Group  
Polarised neutrons  
techniques



**Julien ROBERT**  
Groupe 3 axes  
Highly correlated  
electrons



**Nancy LINDER**  
Soft matter  
Mesoporous materials  
Synthesis, Chemistry



**Fabrice AUDONNET**  
Biology/Disordered systems  
Confined media,  
Thermodynamics



**Olivier CASTELNAU**  
Interface/Materials Group  
Texture-Strain studies.



**Veronique ARLUISON**  
Biology/Disordered systems  
Protein-Nucleic acid interaction,  
Bacterial division

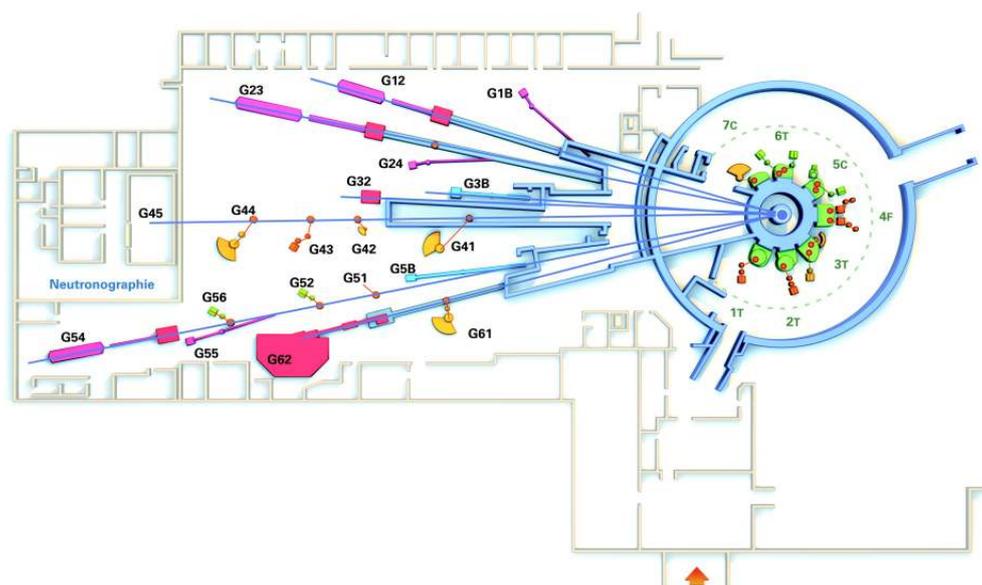


**Arnaud LAVERDUN**  
Informatics  
Interface with spectrometers

**SPECTROMETERS  
AND  
ACCES TO BEAMTIME**



## GENERAL LAYOUT OF THE SPECTROMETERS



	SPECTROMETERS OPEN TO USERS	CONTACTS
	<b>Powder diffractometers</b>	
3T2	Florence Porcher	<a href="mailto:florence.porcher@cea.fr">florence.porcher@cea.fr</a>
G4.1	Gilles André	<a href="mailto:gilles.andre@cea.fr">gilles.andre@cea.fr</a>
G6.1	Isabelle Mirebeau	<a href="mailto:isabelle.mirebeau@cea.fr">isabelle.mirebeau@cea.fr</a>
	<b>Single crystal diffractometers</b>	
5C1	Béatrice Gillon	<a href="mailto:beatrice.gillon@cea.fr">beatrice.gillon@cea.fr</a>
5C1	Alain Cousson	<a href="mailto:alain-f.cousson@cea.fr">alain-f.cousson@cea.fr</a>
6T2	Arsen Goukassov	<a href="mailto:arsen.goukassov@cea.fr">arsen.goukassov@cea.fr</a>
	<b>Diffuse scattering instrument</b>	
7C2	Brigitte Beuneu	<a href="mailto:brigitte.beuneu@cea.fr">brigitte.beuneu@cea.fr</a>
	<b>Small-angle scattering instruments</b>	
G1.2	Didier Lairez	<a href="mailto:didier.lairez@cea.fr">didier.lairez@cea.fr</a>
G2.3	Alain Lapp	<a href="mailto:alain.lapp@cea.fr">alain.lapp@cea.fr</a>
G5.4	José Teixeira	<a href="mailto:jose.teixeira@cea.fr">jose.teixeira@cea.fr</a>
G5bis	Sylvain Désert	<a href="mailto:sylvain.desert@cea.fr">sylvain.desert@cea.fr</a>
G5.5	Grégory Chaboussant	<a href="mailto:gregory.chaboussant@cea.fr">gregory.chaboussant@cea.fr</a>
	<b>Diffractometers for material science studies</b>	
6T1	Marie-Hélène Mathon	<a href="mailto:marie-helene.mathon@cea.fr">marie-helene.mathon@cea.fr</a>
G5.2	Vincent Klosek	<a href="mailto:vincent.klosek@cea.fr">vincent.klosek@cea.fr</a>
	<b>Reflectometers</b>	
G3bis	Fabrice Cousin	<a href="mailto:fabrice.cousin@cea.fr">fabrice.cousin@cea.fr</a>
G2.4	Frédéric Ott	<a href="mailto:frederic.ott@cea.fr">frederic.ott@cea.fr</a>
	<b>Triple-axis instruments</b>	
1T	Daniel Lamago / Yvan Sidis (CRG Instrument Karlsruhe/LLB)	<a href="mailto:daniel.lamago@cea.fr">daniel.lamago@cea.fr</a> / <a href="mailto:yvan.sidis@cea.fr">yvan.sidis@cea.fr</a>
2T	Philippe Bourges	<a href="mailto:philippe.bourges@cea.fr">philippe.bourges@cea.fr</a>
4F1	Sylvain Petit	<a href="mailto:sylvain.petit@cea.fr">sylvain.petit@cea.fr</a>
4F2	Daniel Petitgrand	<a href="mailto:daniel.petitgrand@cea.fr">daniel.petitgrand@cea.fr</a>
	<b>Quasi-elastic instruments</b>	
G6.2	Jean-Marc Zanotti	<a href="mailto:jean-marc.zanotti@cea.fr">jean-marc.zanotti@cea.fr</a>
G1bis	Stéphane Longeville	<a href="mailto:stephane.longeville@cea.fr">stephane.longeville@cea.fr</a>
	<b>Neutron radiography</b>	
G4.5	Guy Bayon	<a href="mailto:guy.bayon@cea.fr">guy.bayon@cea.fr</a>

## THE LLB-ORPHEE NEUTRON SCATTERING AND IMAGING INSTRUMENTS

### Powder diffractometers

- 3T2** "Thermal neutrons" 2-axis (50 detectors) high resolution, mainly for nuclear structure determination.
- G4.1** "Cold neutrons" 2-axis (multidetector 800 cells) high flux, mainly for magnetic structure determination
- G6.1** "Cold neutrons" 2-axis (multidetector 400 cells) with long wavelength ( $\sim 5\text{\AA}$ ) and high flux, for the study of very small powder samples ( $< 1\text{mm}^3$ ). Very high pressure cell available (40 GPa).

### Single crystal diffractometers

- 5C1** "Hot neutrons" 2-axis with lifting arm, polarized neutrons, magnetic field (8 Tesla) for spin-density maps determination
- 5C2** "Hot neutrons" 4-circle for nuclear structure determination.
- 6T2** "Thermal neutrons" 2-axis, lifting arm and 4-circle, mainly for magnetic structure determination. 12 Tesla magnetic field available, 2D detector.

### Diffuse scattering instruments

- 7C2** "Hot neutrons" 2-axis (multidetector 640 cells) for local order studies in liquid or amorphous systems. Cryostat and furnace available (1.2K to 1300°C).

### Small-angle scattering instruments

- G1.2** "Cold neutrons" (annular detector, 30 rings) for study of large scale structures in isotropic systems (mainly polymers and colloids).
- G2.3** "Cold neutrons" (X-Y detector, 128x128 cells) for study of large scale structures (10 to 500 Å) in anisotropic systems (polymers under stress, metallurgical samples, vortex in superconductors).
- G5.4** "Cold neutrons" (X-Y detector, 64x64 cells) for multipurpose studies of large scale structures.
- G5bis** Very Small Angle Neutrons Scattering spectrometer
- G5.5** "Cold neutrons" (X-Y detector, 64x64 cells) ; polarized beam ; large scale magnetic structures ; contrast variation by nuclear polarization of protons.

### Diffractometers for material science studies

- 6T1** "Thermal neutrons" 4-circle for texture determination.
- G5.2** "Cold neutrons" 2-axis for internal strain determination in bulk samples with spatial resolution  $\sim 1\text{mm}^3$ .

### Reflectometers

- G3bis** "Cold neutrons" reflectometer operating in time-of-flight mode for multipurpose surface studies.
- G 2.4** "Cold neutrons" reflectometer with polarized neutrons and polarization analysis for the study of magnetic layers.

### Triple-axis instruments

- 1T** "Thermal neutrons" high-flux 3-axis instrument with focussing monochromator and analyser, mainly devoted to phonon dispersion curves measurements. Very high pressure cell (100 Kbar) available. CRG Instrument operated in collaboration between the INFP Karlsruhe and the L.L.B
- 2T** "Thermal neutrons" high-flux spectrometer with focussing monochromator and analyser, mainly devoted to spin-waves and magnetic excitations studies (1.5 to 80 meV).
- 4F1** "Cold neutrons" high flux 3-axis instruments with double monochromator and analyzer, mainly devoted to the study of low-energy (15 $\mu\text{eV}$  to 4meV) magnetic excitations. Polarized neutrons and polarization analysis option available.

### Quasi-elastic instruments

- G62** "Cold neutrons" high resolution ( $\sim 15\mu\text{eV}$  at 10Å) time-of-flight instrument for the study of low energy excitations, mainly in disordered systems.
- G1bis** "Cold neutrons", high resolution and high flux spin-echo instrument. It can study, in a large Q range, slow dynamics of large molecules in biology or long relaxation times like in glassy transition (Fourier times  $\sim 20\text{ns}$ )

### Neutron Radiography

- G4.5** Imaging technique : white beam facility for non-destructive control or dynamics imaging.

[http://www-llb.cea.fr/fr-en/spectros\\_p.php](http://www-llb.cea.fr/fr-en/spectros_p.php)

## AUXILLIARY SERVICES AVAILABLE

Laboratories for sample preparation:

- Chemistry laboratory
- Biological laboratory

Technical help for:

- Vacuum/Cryogenics
- Cryostat, Furnace (0.1 – 2000 K)
- High pressures (up to 10 GPa)
- High magnetic fields (up to 10 T)
- Mechanics

## ACCESS TO BEAMTIME



LLB has been selected in the frame of the European Community – Access activities of the Neutron scattering and Muon spectroscopy Integrated Infrastructure Initiative (NMI3) which supports access to neutron beams for the selected user teams, travel and subsistence fees of visiting scientists. The program is opened to E.C. users and to scientists of the associated states.

<http://idb.neutron-eu.net/facilities.php>

*Beamtime access is free of charge for any experimentalist from the French Scientific community. LLB takes in charge the expenses (travel and stay) of 2 people during the experiment.*

*Beamtime on the 23 open-access spectrometers can be requested by submission of:*

- **An experimental application to a Selection Committee (Normal Procedure)**  
*This procedure is open to any public/industrial researcher that is interested in using neutron scattering for his research. Results should be free to be totally or partially published in a Scientific Review.*

**DEADLINE FOR APPLICATION: May 1<sup>st</sup> and November 1<sup>st</sup>**

<http://www-llb.cea.fr/en/fr-en/proposal.php>

- **An experimental application to the Directors (Exceptionnal)**  
*This special procedure should only be used exceptionally for hot topics, confidentiality reasons or if an anomaly in the review procedure is suspected. The delay between the acceptance decision and the realization of the experiment is shortened to the minimum.*

***There is no deadline for such propositions, which are examined all along the year.***

<http://www-llb.cea.fr/en/fr-en/proposal.php>

- **A fast access application**

*This procedure allows a rapid access (1 to 2 months delay) to the spectrometers in order to perform a short experiment (1 day max.). It can be used for feasibility tests, sample characterization, obtaining complementary results...*

***There is no deadline for such propositions, which are examined all along the year.***

<http://www-llb.cea.fr/en/fr-en/prop-rap.php>

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## SELECTION COMMITTEES

Proposals are examined by 5 Selection Committees. Each is composed of 10 to 12 senior scientists that are nominated by the management of LLB for 3 years. At least half of them do not belong to the LLB and 2 or 3 are coming from foreign institutes.

For each spectrometer, LLB gives a beam-time available which is shared out by the committee; each proposal gets a grade A or B or C.

A : The experiment must be done and the committee allocates a beam-time

B : The experiment might be done if there is some extra beam-time,

C : The experiment is refused on scientific arguments.

Selection Committees are asked to take care of the educational duty of the LLB when proposal comes from new young searcher.

### SELECTION COMMITTEES: SCIENTIFIC FOCUS AND SUB-FOCUS

#### Theme I Chemical physics, biological systems

- I.01 Polymers and Supramolecular Structures
- I.02 Water, aqueous solutions, polyelectrolytes, surfactants
- I.03 System of biological interest, Biophysics
- I.04 Colloids, nanostructures
- I.05 Gels, composite materials
- I.06 Other...

#### Theme II Crystallographic and magnetic structures

- II.01 Crystalline structures
- II.02 Phases transitions
- II.03 Magnetic Structures
- II.04 High pressures (on powders)
- II.05 Other...

#### Theme III Magnetism: Single-crystal systems and thin layers

- III.01 Magnetic thin layers
- III.02 Spin density
- III.03 Systems with strong quantum correlations
- III.04 Extreme conditions (strong fields, high pressures)
- III.05 Magnetic nanosystems
- III.06 Other...

#### Theme IV Disordered Systems, nanostructured materials and materials

- IV.01 Liquid and amorphous structures
- IV.02 Dynamics of disordered systems
- IV.03 Thin film materials
- IV.04 Nanostructured materials, precipitation, cavities,...
- IV.05 Crystallographic textures
- IV.06 Strains and residual stresses
- IV.07 Other...

#### Theme V Excitations

- V.01 Magnons
- V.02 Superconductivity
- V.03 Coupling spin-network
- V.04 Dynamics in frustrated systems
- V.05 Polarized neutrons with polarization analysis
- V.06 Phonons
- V.07 Other...

## LLB Reviewing committees (Autumn 2009)

### COLLEGE 1 : Physico-Chemistry, Biological systems

*Organisers : G. Carrot, N. Malikova*

LLB representatives	French users	European users
J. Jestin D. Lairez	C. Chassenieux E. Dubois P. Fontaine R Schweins	W. Hauessler R. V. Klitzing P. Stepanek [Chairman] M. Sferrazza M. Weik

### COLLEGE 2 : Magnetic and nuclear structures

*Organisers : F. Porcher, F. Damay*

LLB representatives	French users	European users
J.-M. Kiat I. Mirebeau	M.-H. Lemée-Cailleau G. Rousse M. Josse	G. Heger [Chairman] J. Alonso L. Chapon

### COLLEGE 3 : Magnetism : Single crystals and thin films

*Organisers : A. Bataille, J. Robert*

LLB representatives	French users	European users
B. Gillon	K. Dumesnil E. Janod O. Mentré	J. Campo [Chairman] K. Temst

### COLLEGE 4 : Disordered systems and Materials

*Organisers : V. Klosek, M.H. Mathon*

LLB representatives	French users	European users
F. Audonnet	J.-L. Bechade [Chairman] D. Morineau P. Vajda	B. Frick M. Fitzpatrick

### COLLEGE 5 : Excitations

*Organisers : P. Bourges, D. Petitgrand*

LLB representatives	French users	European users
S. Petit H. Moudden	M. Boehm L.-P. Regnault P. Foury	P. Link [Chairman] J. Hlinka, D. Reznik

# Un logo pour LLB/Orphée



Logos created by members of Orphée-LLB for the competition "A new logo for LLB-Orphée"

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