2 - MAGNETISM AND SUPERCONDUCTIVITY

Magnetism is a virtually unlimited field of activity for neutron scattering studies. Applications range from well-established topics like high-temperature supraconductors, « colossal magnetoresistance » manganites, or heavy-fermion compounds, to more emerging problems : photomagnetism, magnetic structures and excitations in epitaxial films, incommensurabilities in composite systems, etc. In many cases, neutrons are unchallenged in their ability to reveal details of the time and space dependencies of relevant interactions at a microscopic level. A number of examples can be found in the following sections, which present an overview of the most significant results obtained over the last two years.

STRONGLY CORRELATED ELECTRON SYSTEMS

Spin dynamics in hole-doped high-temperature superconducting cuprates

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In order to understand electron pairing in high-temperature superconducting (SC) copper oxides, one needs first to shed light on the intrinsic nature of the CuO_2 planes on a microscopic scale. There are, for the time being, two main schools of thought. For the first one, the CuO_2 planes are characterized by a uniform 2D liquid of itinerant charge carriers in strong interaction. The second one rather assumes that a spin-charge segregation takes place in the CuO_2 planes, resulting in hole-rich nonmagnetic lines playing the role of anti-phase boundaries between hole-poor antiferromagnetic domains. The study of the spin excitation spectrum by inelastic neutron scattering (INS) can provide valuable information to discriminate between a 2D itinerant picture or a 1D "stripes" scenario. To this aim, we have developed a strategy based on : *i)* comparison with canonical stripe-ordered systems, *ii)* search for uniaxial anisotropy in orthorhombic systems, *iii)* search for hallmarks of the physics of CuO_2 planes in the dispersion of magnetic collective modes *iv)* study of the effect of inter-plane interactions on the spin dynamics in bilayer systems. Results obtained along these lines are summarized hereafter.

i) In several cuprates such as $YBa_2Cu_3O_{6+x}$, the spin excitation spectrum is characterized by the appearance, in the SC state, of an antiferromagnetic (AF) excitation whose energy scales with the critical temperature: the so-called "magnetic resonance peak". This excitation exhibits a downward dispersion and was first interpreted in terms of triplet exciton which can develop in a *d*-wave superconductor according to itinerant models. Alternatively, it has been proposed that this $S = 1$ collective mode might be reminiscent of magnons in a disordered stripe system. To test this suggestion, INS experiments were performed in the diagonal-stripe-ordered system $La_{1.69}Sr_{0.31}NiO_4$. The spin waves are actually found to merge at the AF wave vector as predicted, while their overall evolution as a function of temperature or wave vector does not match that reported for the magnetic resonance peak in cuprates, pointing to marked differences between the spin dynamics of stripe-ordered-nickelates and superconducting cuprates.

ii) The INS measurements carried out at the LLB and the ILL on almost fully detwinned $YBa_2Cu_3O_{6+x}$ single crystals revealed 2D spin fluctuations in the SC state, with an incipient 1D anisotropy at an energy well below that of the magnetic resonance peak. This result, valid from optimal doping (x = 0.9) to the underdoped regime (x = 0.66), implies that the orientation of stripes (if any) has to fluctuate. In contrast, in an itinerant picture, the anisotropy of the magnetic response should reflect the effects of orthorhombicity and a possible Pomeranchuk instability on Fermi surface (see *Highlights*: Hinkov et al.).

iii) Close examination of the upper part of the spin excitation spectrum in optimally doped $YBa_2Cu_3O_{6.95}$ has revealed an unsuspected upward dispersive resonant magnetic mode, starting near or at the magnetic resonance peak. In slightly underdoped $YBa_2Cu_3O_{6.85}$, the upward branch was found to become evanescent when approaching particular wave vectors. This phenomenon could reflect the electron-hole decay of collective spin fluctuations within the magnetic continuum in the SC state, as predicted in itinerant models. Surprisingly, the X-shape dispersion around the AF wave-vector can also be found in many stripe models.

iv) $YBa_2Cu_3O_{6+x}$ is a bilayer system (containing two CuO_2 planes per unit cell), and intra-bilayer interactions should thus lift the degeneracy of purely planar spin fluctuations. In addition to the magnetic resonance peak of odd symmetry with respect to the exchange of spin within the bilayer, a second resonant excitation of much weaker intensity and of even symmetry has recently been observed at higher energies. The asymmetric intensity of both odd and even resonant modes has been used to pinpoint the threshold of the SC spin-flip continuum at the AF wave vector, which scales with the maximum of the SC gap, as expected in the itinerant picture. On the other hand, the odd or even symmetry of the spin fluctuation implies that charge stripes should be stacked on top of each other within the bilayer: a rather unfavorable arrangement in terms of Coulomb energy.

Long- and short-range magnetic order, charge transfer and superconductivity in $YBa_2Cu_{3-x}Co_xO_7$

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In the $YBa_2Cu_{3-x}Co_xO_7$ system, Co substitution suppresses superconductivity at $x_c = 0.42$ and antiferromagnetic long-range order emerges above this concentrations. Previous measurements performed at the LLB on a single crystal (Hodges et al. Phys. Rev. B 2002, *LLB report* 2001-2002), had shown that AF order might exist in some superconducting fully oxidized crystals at low Co content ($x \sim 0.04$). The phase diagram derived from systematic powder experiments in a wide concentration range ($0.03 \leq x \leq 0.72$) did not evidence such coexistence but revealed a number of new interesting features. The Co ions substitute almost only on the charge reservoirs, the Cu1 sites, and that their charge varies from 4+ to 3+ with increasing Co content. The antiferromagnetic order survives at much higher hole doping than in related systems ($p = 0.14$ hole/Cu2 plane instead of ~ 0.04 in $YBCO_y$ and Ca-$YBCO_6$). This suggests that Co substitution does not only change the hole concentration in the Cu2 planes but also reduces the hole mobility. This interpretation is supported by the fact that the metal-insulating transition coincides with the onset of AF long-range order at x_c. The AF order shows notable differences compared to that found in $YBa_2Cu_3O_6$. In particular the unit cell is doubled along the *c* axis, with a non-zero moment on the Cu1 sites. The Co moments remain mostly disordered, forming random AF next-nearest-neighbor pairs. For $x > x_c$, these pairs compete with the ordered AF structure, which can explain the surprisingly low value of the ordered moments at the Cu1 sites.

Spin dynamics of the electron-doped high-Tc cuprate $Nd_{2-x}Ce_xCuO_4$

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i) Electron-doped versus hole-doped superconductors

The electron-doped superconductors $Nd_{2-x}Ce_xCuO_4$ share some common features with their *p*-type counterparts (typically the CuO_2 planes sandwiched by donor/acceptor rare-earth blocks). However significant differences have also been found, which may help elucidate the common mechanism for superconductivity involved in the two families. Among interesting results obtained at the LLB is the detailed characterization of the magnetic excitation spectrum as a function of energy and temperature.

ii) Low temperature magnetic excitations

Magnetic excitations are found to be confined to the magnetic 2D rod $\boldsymbol{Q} = (1/2, ½, \zeta)$, and do not show any incommensurate feature. At low temperature the magnetic excitation spectrum shows a well-defined spin gap of 2.9 meV. Above the gap there exists a continuum of excitations, in contrast to the magnetic resonance peak observed in *p*-type HTSC.

iii) Temperature dependence of the spin gap

An in-depth study of the temperature dependence of the spectrum has been done by performing constant-energy $\boldsymbol{Q}$ scans through the rod. The dynamical susceptibility below an energy of 2 meV decreases with decreasing temperature, as the sample enters the superconducting state, vanishing completely at $T = 1.6$ K. This clearly demonstrates the suppression of electronic states below 2 meV in the superconducting state. The distortion of the excitation spectrum can be described as a decrease of the spin gap when temperature is increased up to T_c. Additional measurements at lower energies, measured with $k_f = 1.97$ and 1.64 Å^{-1}, give evidence that the normal state spectrum is gapless down to very low energies. The spectrum at $T = 1.6$ K, when compared with that at $T = 26$ K, reveals a broad range of energies above the gap, between 2 and 4 meV, where the density of excitations is only partially reduced. The latter feature may be associated with the anisotropic *d*-wave gap order parameter.

Vortex lattice studies

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Vortex lattice studies are important to understand critical currents in superconductors. Small angle neutron scattering measurements on YBaCuO samples with different densities of twin boundaries have revealed that these twin boundaries produce both a meandering of the flux lines and, as their density increases, a change in the apparent symmetry of the flux line lattice from hexagonal to square (see *Highlight*: Pautrat et al.).

Spin-ladder compounds

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The compound $Sr_{14}Cu_{24}O_{41}$ exhibits an incommesurate structural modulation resulting from a misfit between the *c* lattice parameters of the two planar components (CuO_2 chains, and $Sr_2Cu_2O_3$ ladders) of its composite structure. High-*Q*-resolution experiments on the triple-axis spectrometer 4F2 have shed light on the controversial charge-ordering transition occurring near 250 K. The results suggest that charge ordering is indeed responsible from the superstructure reflections observed below the transition, but that its modulation locks onto that of the composite structure (see *Highlights*: Braden et al.).
Hole-doping of these materials by La substitution causes pronounced effects on their magnetic properties, which may be taken as a paradigm for understanding those found in cuprates. Neutron diffraction experiments on $La_5Ca_9Cu_{24}O_{41}$ have shown correlations with a very weak value of the induced ferromagnetic component. This behavior is interpreted as a coupled spin-charge effect in which a motion of the holes along the chains is accompanied by a motion of the magnetic domain walls (see *Highlights*: Klingeler et al.).

Manganites

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Among the numerous challenges faced in understanding colossal magnetoresistance (CMR) manganites $La_{1-x}Ca_xMnO_3$, the possible existence of mixed phases has been pointed out by numerical studies. In recent inelastic neutron scattering experiments for the compositions x = 0.17 and 0.22, remarkable anomalies have been observed in the dispersion of magnetic excitations, which could be interpreted in terms of spin waves confined within finite-size, hole-poor, orbital-ordered regions (see *Highlights* : M. Hennion et al.).
Other aspects of phase coexistence have been investigated in the Pr compound $Pr_{0.67}Ca_{0.33}MnO_3$. At low temperature, this compound is insulating and residual conductivity takes place only through a « variable-range-hopping » process. It is proposed that, in this situation, a new channel of magnetic exchange interaction termed «hopping exchange » exists, leading to the segregation of a filamentary phase in which Mn spins are parallel, with (AF-coupled) spins outside the filaments playing the role of a magnetic shield. This view is supported by the observation of a fractal law with exponent 5/3, reminiscent of polymer physics, in polarized small-angle neutron scattering measurements as well as in Monte Carlo simulations (see *Highlights* : Viret et al.).

New studies have been undertaken on the bilayer manganite $(La_{0.4}Pr_{0.6})_{1.2}Sr_{1.8}Mn_2O_7$. $La_{1.2}Sr_{1.8}Mn_2O_7$, exhibits a phase transition from a paramagnetic insulating (PI) to a ferromagnetic metallic (FM) state with a CMR effect. Upon 60% Pr substitution, magnetic order and PI to FM transition are suppressed. Application of a moderate magnetic field restores a FM state with a CMR effect. Neutron scattering by a single crystal of, under a magnetic field of 5 T, has revealed a long-range and homogeneous ferromagnetic order. In the PI phase, under zero field, correlated lattice polarons have been detected. At 28 K, under 5 T, the spin wave dispersion curve determines an in-plane isotropic spin wave stiffness constant of 146 meV $Å^2$. So the magnetic field not only generates a homogeneous ferromagnetic ground state, but also restores a magnetic coupling characteristic of FM CMR manganites. Unpolarized and polarized neutron diffraction measurements have also been carried out on this compound. Magnetization density reconstruction by the maximum-entropy method, together with multipole refinement on flipping ratios, evidence the existence of two distinct field-induced ferromagnetic (FIFM) states. For $H \parallel \boldsymbol{c}$, the FIFM(c) state is characterized by the presence of a magnetic moment of 0.48(2) μ_B on the Sr site, due to Pr substitution, and by a high population of the $3z^2$-r^2 orbitals of Mn^{3+}. For $H \parallel \boldsymbol{a,b}$ no magnetization density was found at the Sr site and the x^2-y^2 orbital is slightly more populated than the $3z^2$-r^2 orbital.

Anomalous rare-earth magnetism

i) Spin dynamics in mixed-valence systems and Kondo insulators

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Previous studies of the low-temperature spin-gap state in YbB_{12} (*LLB report* 2001-2002) had revealed a pronounced ***Q*** dependence of the sharp magnetic mode denoted *M*1 near 15 meV, with a minimum in the dispersion and a maximum in the intensity at the AF point (½, ½, ½). This behavior has been fully confirmed by new polarized-neutron experiments on IN20 (ILL), as well as the magnetic character of the second mode *M*2 (20 meV), whose ***Q*** and *T* behaviors differ markedly from those of *M*1. It is suggested that *M*1 may correspond to the spin-exciton *in-gap* mode predicted by Riseborough, i.e. a bound state of an electron-hole pair produced by strong AF correlations. Time-of-flight measurements on $Yb_{0.25}Lu_{0.75}B_{12}$ (HET ISIS) have confirmed that the spin-gap response persists far into the diluted regime, where the electronic state is unambiguously metallic, supporting the view that the spin-gap formation is based, at least partly, on a local mechanism and does not directly depend on the existence of the hybridization gap.

Previous work on Sm mixed-valence (MV) systems has been extended to another compound, $EuCu_2Si_2$, in which Eu fluctuates between the 3+ state (same electronic configuration as Sm^{2+}) and the pure-spin state Eu^{2+} ($J = S = 7/2$). The spin-orbit $J = 0 \rightarrow J = 1$ transition has been observed on isotopic (^{154}Eu) powder at ISIS (HET), with the interesting result that the excitation is sharp but split into a doublet and shifted with respect to the value expected for the free ion. A crystal-field splitting of the $J = 1$ state would be compatible with the site symmetry but quite unexpected in view of the strong degree of valence mixing. Other interpretations (exciton state, coupling to phonons, …) are currently under consideration.

ii) Magnetic polarons, p-f mixing

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Ce*X* (*X* = P, As, Sb and Bi) compounds are known to exhibit magnetic structures with two different Ce magnetic moments originated from the different crystal-filed states Γ_7 and Γ_8. This feature is explained by the magnetic-polaron effect produced by the localization of the low-density carriers and the strong *p-f* mixing between the Ce-ion Γ_8 state and the *p*-hole state of neighboring *X* ions. Inelastic neutron scattering experiments on CeSb and CeBi have revealed a new phonon existing only in the magnetically ordered phases due to the *p-f* mixing effect. The observed new phonon is a stretching mode of the Ce-*X* bond forming the *p-f* mixing orbital. This fact indicates a novel coupling between electronic states by the *p-f* mixing and the crystal lattice.

iii) Multipolar interactions in Ce-based hexaborides

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Light rare-earth hexaborides RB_6 (R = Ce, Pr, Nd) are unique examples of the interplay between different types of multipole couplings as, for instance, in the so-called "phase II" of pure CeB_6 whose description requires consideration of dipole, quadrupole, and octupole interactions. The evolution of (H, T) magnetic phase diagrams in the $(Ce,Nd)B_6$ and $(Ce,Pr)B_6$ solid solutions has been studied by both powder and single-crystal neutron diffraction. A number of new phases have been observed and characterized including, for $x_{Pr} = 0.3$, a novel regime tentatively ascribed to an incommensurate order of quadrupole moments.

Quantum magnetism, spin liquids

*i) Magnetic structure of a frustrated S = ½ square lattice (*Highlights *: Bombardi et al.)*

The controversial issue of the exact magnetic structure of Li_2VOSiO_4 has been settled by combining results of powder neutron and x-ray diffraction. It consists of a FM stacking, along the *c* axis, of collinear AF *a-b* layers. The ordered magnetic moment is larger than previously reported and compatible with the large next-nearest-neighbor exchange ($J_2/J_1 >> 1$) predicted by local density calculations.

*ii) Spin correlations in a dimer spin liquid (*Highlights *: Rüegg et al.)*

Elementary singlet-triplet excitations in the spin-liquid dimer ground state of $TlCuCl_3$ have been determined as a function of temperature up to $k_B T \approx J_{intra}$, the dominant AF exchange interaction. Tracing the renormalization of spin correlations at high enough temperatures provides an insight into the correct statistical description of this quantum many-body system. Excellent agreement is found with the Troyer-Tsunetsugu-Würtz mean-field theory.

MAGNETIC FILMS AND MULTILAYERS

Diffraction on thin layers and multilayers using three-axis spectrometers

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Significant results have been obtained recently in diffraction measurements on thin layers or multilayers performed on 4F1 and 4F2. The main advantages of using cold-beam three-axis spectrometers for such studies are: *i)* the high flux of cold neutrons, *ii)* the good resolution, *iii)* the background reduction provided by the analyzer, *iv)* the availability of a full polarization analysis of the scattering process. Up to now, most studies have been devoted to the determination of magnetic structures.

The recent search for materials suitable for applications in the field of so-called "spintronics", where control of the carrier spin state is required, has stimulated new demand for magnetic characterization of thin layers. Indeed a variety of new systems are now produced by such methods as beam epitaxy, sputtering, vapor deposit, etc. These systems often consist of a stacking of layers with different physical properties. For instance, ferro-magnetic layers can be sandwiched with a nonmagnetic spacer. For thin enough layers, a magnetic interlayer coupling often takes place, the mechanism of which is not yet fully understood. The existence of AF coupling between ferromagnetic layers offers the possibility to trigger the system by means of an external parameter (e.g. magnetic field or illumination). In other systems, the characteristics of an AF layer can be influenced by the magnetization of a ferromagnetic coating layer. In this case domain structuration seems to be the relevant parameter.

Neutron diffraction measurements provide valuable information on such systems because, on the one hand, they give direct evidence for the existence of AF correlations or long-range order, and on the other hand they can determine the size of magnetic domains when distances of less than about 100 nm are involved. Recent measurements on various systems (Si/Fe, Fe/Fe_2N multilayers [see *Highlights*: Szuszkiewicz et al.], NiO, MnPt, FeF_2 thin layers) have demonstrated the feasibility of such measurements on samples with an area of typically 1 cm^2 and a thickness as low as 5 nm. Improvements of sample environments are underway to extend the possibilities of such measurements.

Magnetic behavior of Eu epitaxial thin films

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Bulk Eu is known to undergo a magnetic phase transition to a helical order phase at T = 90 K. The helical axis is aligned along one of the equivalent cubic <100> directions, leading to the formation of 3 magnetic domains. Elastic neutron scattering experiments on Eu MBE thin films performed by the Nancy group, both at the LLB and NIST (USA) have shown that external stress modifies the magnetic behavior of Eu by making the domain inequivalent (destabilization of the domain with in-plane helical wave vector; rotation of the out-of-plane wave vectors initially aligned along cubic axes). Domain population can be further modified by applying a magnetic field, giving rise to magnetic irreversibility. Basing on symmetry considerations and on correlations between deformation amplitudes and magnetic effects, two types of models have been considered, involving either exchange magnetoelasticity or magnetostriction.

Magnetic exchange interactions in hexagonal MnTe

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The Mn element is involved in many magnetic systems, either as a constituent or as a dopant. The resulting magnetic properties depend on the details of exchange interactions, which are very sensitive to the crystal structure of the compound. This property is actually used on a phenomenological basis to modify the properties of semiconducting materials, with a significant renewal due to the possibility of growing novel systems by such methods as, e.g., beam epitaxy or sputtering. In pure systems one can achieve an accurate

determination of magnetic exchange interactions and anisotropies through the analysis of the spin-wave dispersion. Single-crystal inelastic neutron scattering is the only technique providing direct access to the spin-wave spectrum, from which an insight can be gained into the microscopic mechanisms responsible for the magnetic exchange.
Such an analysis has already been performed for cubic (zinc-blende structure) MnTe obtained by beam epitaxy. MnTe naturally crystallizes in the NiAs hexagonal structure, and is magnetically ordered (type-I AF) up to ~ 310 K. Cubic MnTe, on the other hand, orders in a type-III antiferromagnetic structure below $T_N \approx 67$ K. This difference gives us an opportunity to determine a new set of exchange interactions complementing those previously determined for the cubic phase.
A single crystal of volume ~ 30 mm^3 was grown at the Institute of Physics in Warsaw. Spin-wave measurements were performed on the thermal-beam three-axis spectrometer 2T, and a complete set of dispersion curves has been determined at $T = 15$ K, along the [100], [001], and [101] directions of the reciprocal lattice. The determination of exchange integrals and anisotropies is in progress and will involve exchanges at least up to third nearest neighbors.

Neutrons surface scattering techniques applied to the study of magnetic thin films

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In the last two years, a number of scattering techniques have been applied to the study of thin films. Besides the classical specular reflectivity technique (PRISM), Grazing Incidence Small Angle Neutron Scattering (GISANS) has been implemented on the new spectrometer PAPYRUS (see chapter on Technical and Instrumental Developments). Direct diffraction has been applied to characterize the atomic structure of magnetic films as thin as 10 nm. Using this panel of techniques, one can now probe the structure of thin films in an extremely wide range of Q vectors spanning over 5 orders of magnitude:

- Off-specular reflectivity (OSR) $6\times10^{-5} < Q < 6\times10^{-3}$ nm^{-1}
- Specular reflectivity (SR) and GISANS $6\times10^{-2} < Q_\perp$ and $Q_{//} < 1$ nm^{-1}
- Diffraction $1 < Q < 20$ nm^{-1}

This makes it possible to cover a broad range of topics ranging from magnetic order in epitaxial thin films (FeF_2, Fe_2O_3, NiO, MnPt) or magnetism at interfaces (Fe/FeN, Fe/Si, GaAs/GaMnAs, Gd/Cr, Gd/V, Fe_3O_4/Fe_2O_3, FeF_2/Fe…), to thin-film magnetic nanostructures (Fe nanodots, magnetic aggregates) and magnetic domains (Fe/FeF_2, FePd).
These type of measurements are illustrated by measurements on FePd thin films mixing different types of magnetic anisotropies and domain structures.
Besides the above developments of surface scattering techniques, significant effort is now devoted to improving sample environments for magnetic studies (high fields, wide temperature range).

MOLECULAR MAGNETISM, PHOTOMAGNETISM

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High-spin molecular clusters are of particular interest because they behave as “single-molecule magnets”. The magnetic ground state ($S = 10$) of the high-spin cluster $[(tacn)_6Fe_8O_2(OH)_{12}]$ has been characterized by polarized neutron diffraction, in collaboration with D. Gatteschi’s group. The relative strengths of competing intramolecular magnetic interactions are clearly established from the experimental spin distribution.
In the field of photomagnetism significant progress has been made in the study of structural changes induced by the switching from the low-spin state to the photo-excited high-spin state. The nuclear structures in the ground and the photo-induced states of the spin-transition complex $[Fe^{II}(ptz)_6](BF_4)_2$ have recently been investigated on the Laue neutron diffractometer VIVALDI at the ILL. An in-situ photo-excitation setup was installed especially for that experiment, in collaboration with G. McIntyre. Photo-induced structural changes were clearly evidenced in this complex, for which the magnetization density in the photo-excited state was determined by polarized neutron diffraction. Structure determinations are underway.
Promising results have also been obtained in powder neutron diffraction under light illumination. Partial photo-excitation of a powder sample of a Fe^{II} spin-transition complex has been obtained with the help of a new setup installed on the G4.1 diffractometer. This should prove quite useful for studies of photo-induced magnetic ordering.