Neutron scattering/Diffusion de neutrons

Magnetic neutron diffraction under high pressure

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Available online 5 November 2007

Abstract

Applying high pressure in neutron diffraction provides a microscopic understanding of the magnetic order and magnetic interactions in materials under extreme conditions. By checking the dependence of interactions over a wide range of interatomic distances, this technique also allows testing microscopic models at ambient pressure. Recent instrumental progress in high pressure neutron diffraction now allows magnetic materials to be measured in an unprecedented range of pressures, possibly combined with very low temperatures and high magnetic fields. A few examples chosen in model ferromagnets, solid oxygen or geometrically frustrated magnets give some insight into the possibilities of this technique. ***To cite this article: I. Mirebeau, C. R. Physique 8 (2007).***
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Résumé

Diffraction magnétique de neutrons sous haute pression. L'application de hautes pressions, combinée à la diffraction de neutrons permet de comprendre au niveau microscopique l'ordre et les interactions magnétiques dans les matériaux en conditions extrêmes. En vérifiant les lois de variations des interactions dans un large domaine de distances interatomiques, cette technique permet aussi de tester la validité de modèles microscopiques à pression ambiante. De récents progrès instrumentaux en diffraction de neutrons sous haute pression offrent maintenant la possibilité de mesurer des matériaux magnétiques dans une gamme de pression inégalée, et de combiner l'application de hautes pressions à celle de très basses températures et de forts champs magnétiques. Quelques exemples choisis dans des ferromagnétiques modèles, l'oxygène solide ou des composés géométriquement frustrés donnent un aperçu des possibilités offertes par cette technique. ***Pour citer cet article : I. Mirebeau, C. R. Physique 8 (2007).***
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Keywords: Neutron diffraction; High pressure; Magnetism

Mots-clés : Diffraction de neutrons ; Haute pression ; Magnétisme

1. Introduction

Applied pressure induces a wide variety of phenomena in solid state physics. They are often associated with phase transitions, which can be well studied by neutron scattering. A change in interatomic distances may induce a change in the nature of the atomic bonding, in the crystal structure, the electronic structure, the excitation spectra, etc. In soft condensed matter and biological systems, pressure is also a very effective modulator, since low pressure may change intermolecular distances and affect molecular conformations, without changing covalent bond distances and angles. An applied pressure affects proton dynamics, yields protein denaturation or inhibits bacterial growth for instance.

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Soft materials like proteins, polymers or liquid crystals generally undergo transitions at low or medium pressures (from a few kbars to a few GPa). Such pressures can be easily combined with neutron studies, using helium gas or piston cylinders cells. Application of a medium pressure offers some advantages, such as the possibility of reaching purely hydrostatic conditions, or measuring large sample volumes required for inelastic neutron scattering. Medium pressures were used to study various materials by neutrons [1]. However, many phenomena in solid state physics require a much higher pressure range (10 GPa or above), which cannot be obtained by conventional techniques. A well-known example is the investigation of geological materials, where pressures in the Megabar range are required to simulate the thermodynamical conditions inside the Earth mantle or inside planets.

In the field of magnetism, many transitions occur under very high pressures, yielding new states of condensed matter. This article focuses on some of them. Besides their intrinsic interest, high pressure studies may also help one to understand the nature of the ambient pressure state, by allowing one to check models of interactions for a large variation of interatomic distances. Among the pressure induced phenomena, one can quote valence transitions, hybridization of magnetic moments, magnetic transitions induced by structural transitions, for instance. The list is far from being exhaustive. Pressure induced transitions also occur in compounds close to an instability threshold, allowing their magnetic state to be tuned with interatomic distances. This may be the case of some metal-insulating transitions, or of thresholds between localized and itinerant magnetism. Applied pressure also plays a major role in systems whose ground state is controlled by an energy balance between several interactions, which depend on interatomic distances in different ways. Geometrically frustrated magnets, where the ground state is highly degenerated, offer good examples of original magnetic states induced under pressure.

In many cases, high pressure magnetic transitions are characterized by macroscopic probes only, such as optical absorption and electrical resistivity, eventually magnetization. It is therefore a huge potential field of investigation for neutron diffraction, the only technique allowing a complete characterization of magnetic order. Due to their magnetic moments, and their wavelength close to interatomic distances, neutrons are perfectly suitable to investigate magnetism at a microscopic level. However, since the interaction of neutrons with matter is weak,—as compared with electron or X-ray synchrotron radiation—large quantities of material (typically from a few mm^3 to a few cm^3, depending on the values of the magnetic moments and the type of magnetic order), are usually needed to obtain good statistics in reasonable counting times, and to get a correct signal/background ratio. Such quantities cannot be easily pressurized. Moreover, since magnetic transitions often occur at low temperatures, below ambient of even liquid helium temperature, they might also require a magnetic field, namely sample environments which are not easily compatible with high pressures. These drawbacks limited or prevented the use of neutrons in high pressure magnetism for a long time.

The situation drastically changed in the 1980s, when the feasibility of performing neutron diffraction experiments in diamond, then in sapphire anvils cells, was demonstrated at the Kurchatov Institute of Moscow [2]. Then, high pressure was implemented to neutron experiments by two specialized teams, using very different approaches, one in Kurchatov Institute in collaboration with Laboratoire Léon-Brillouin (LLB) [3], the other in Université Pierre et Marie Curie (Paris) in collaboration with Edinburgh University [4]. A specialized diffractometer G61-Micro was built at the LLB, which is fully adapted for powder magnetic diffraction studies under high pressure. It is briefly described here. The powder diffractometer is installed on a cold source of the Orphée reactor. The long wavelength of the incoming neutrons (4–4.8 Å) provides good conditions for magnetic studies. A double stage focusing system is installed between the monochromator and the sample. Each focusing device (Fig. 1) is made of four Ni–Ti supermirrors, compressing the beam in both vertical and horizontal planes [5]. The variable focusing allows one to choose the best compromise between intensity and angular resolution. Compact specialized pressure cells with diamond or sapphire anvils known as the Kurchatov-LLB pressure cells have been developed, with wide apertures for the neutron beams. They allow measurements under several scattering geometries, and provide excellent transmission and background conditions. They are compatible with a helium cryostat (modified by inserting inner cadmium masks to reduce the background), and a dilution insert. Single crystal measurements are also available under high pressure [6], possibly combined with low temperature and magnetic field, on the spectrometer 6T2 of the LLB. The whole set up allows one to measure powder and single crystals with volumes as small as $0.001\ mm^3$, therefore to cover an unprecedented range of pressures (up to 50 GPa), which may be combined with magnetic field (up to 7 T) and low temperatures (down to 50 mK). Notice that the Paris-Edinburgh cells [4] also provide high pressures for neutron experiments in the 10 GPa range. They accept larger sample volumes, and have been adapted to perform inelastic neutron scattering measurements [7], but they are more suitable to experiments at ambient or high temperature, in zero magnetic field.
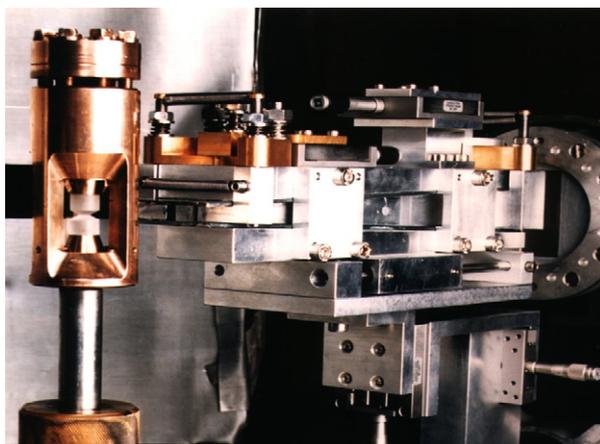
Fig. 1. Focusing device and pressure cell, installed on the high pressure powder diffractometer G61-Micro of the Laboratoire Léon Brillouin (LLB).

The influence of an applied pressure may be compared to that of a 'chemical pressure', realized by substituting a given ion (magnetic or not) by another one with the same valency, but a different ionic size, either compressing or expanding the lattice. Taking into account the typical compressibilities of solids, the change of interatomic distances induced by chemical substitution is often comparable to that induced by a high pressure (typical substitutions allowed by phase stability yield chemical pressures in the 10 GPa range). However, the concomitant changes in magnetism and band structure may be strongly different.

In the following, the article describes a few recent experiments of magnetic studies performed in LLB. Its purpose is not to give an overview of the numerous fields which can be studied by neutrons under pressure, but simply to give precise examples showing the contribution of high pressure neutron diffraction to some basic problems of solid state physics.

2. Enhancement of ferromagnetism in the model system EuX ($X =$ S, Se, Te)

Europium monochalcogenides EuX ($X =$ O, S, Se, Te) have been model systems in solid state physics since the early 1960s, due to their simple NaCl type structure, and the presence of localized 4f Eu^{2+} magnetic moments with spin only component. EuO and EuS are the first known semiconducting ferromagnets (F), used as a reference for Heisenberg (isotropic) magnetism. EuSe is ferrimagnetic, whereas EuTe is antiferromagnetic (AF). EuX compounds allow one to test theoretical models of the magnetic interactions in semiconductors. The small radius of the 4f shell prevents direct exchange interactions. The magnetic interactions are mediated either by the 2p orbitals of the anion X (super exchange), or by the 5d or 6s Eu orbitals (indirect exchange). The remarkable change in the magnetic order from F to AF, observed when the size of the anion increases from O to Te, arises from a competition between the F interaction J_1 between first neighbors and the AF interaction J_2 between second neighbors. The different sensitivity of J_1 (indirect exchange) and J_2 (superexchange) to interatomic distances is the key point of this behavior. However, comparing different members of the family does not allow one to separate the effect of interatomic distances from the chemical nature of the anion. So fully different models of the magnetic interactions were proposed to explain ambient pressure data in EuX.

Applying high pressure allows one to check theoretical descriptions at ambient pressure. High pressure powder neutron diffraction patterns were measured in EuS and EuSe ferromagnets up to the very high pressure of 20.5 GPa, using diamond anvil cells inserted in an helium cryostat [8]. For each pressure, the integrated intensity of a given Bragg peak is recorded versus temperature, allowing the determination of the Curie temperature T_C: the intensity decreases from $T = 0$ up to T_C, while the thermal average of the ordered Eu^{2+} moment $\langle S_z^2 \rangle$ decreases, then it saturates above T_C, when only the structural contribution to the Bragg peak remains. The positions of the Bragg peaks yield the lattice constant a and the equation of state a(P). Similar measurements were performed on the antiferromagnet EuTe [9], showing the transformation from AF to F phase under pressure.

In Fig. 2 (left), the Curie temperatures T_C are plotted versus pressure. One observes a huge increase of T_C with increasing pressure: typically in EuS from 17 K at ambient pressure to 162 K at 22.5 GPa. Moreover this increase
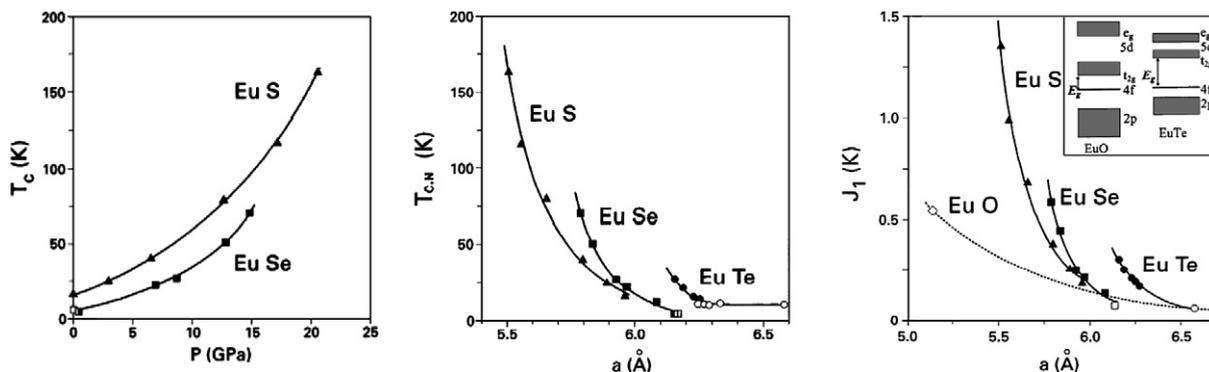
Fig. 2. Left: Curie temperature versus pressure in EuS and EuSe. Middle: ordering temperatures versus the lattice constant a in the EuX family (X = S, Se, Te). Open (filled) symbols correspond to Néel (Curie) temperatures, respectively. The half filled symbol corresponds to the triple point of the paramagnetism, ferromagnetism and antiferromagnetism. Solid lines are derived from the fit of the first neighbor exchange constant J_1. Right: first neighbour exchange constant J_1 versus lattice constant a. Filled symbols are derived from the T_C data of Ref. [8]. Open symbols correspond to J_1 values at ambient pressure. Solid (dotted) lines correspond to the variation of J_1 under applied (chemical) pressure respectively. Insert: schematic view of the electronic structure of EuX. The neutron powder measurements were performed on the diffractometer G61-Micro of the LLB.

is different for each member of the family, so that all data points do not merge in a single curve. This is shown in Fig. 2 (middle) by plotting the ordering Curie and Néel temperatures T_C and T_N versus the lattice constant a for all members of the family. An applied pressure has a much stronger effect on T_C than a chemical pressure, whose influence is evaluated by plotting the T_C values versus a at ambient pressure for all compounds.

The first neighbor interaction J_1 is deduced from T_C in the mean field approximation, assuming that J_2 is pressure independent. This is justified since in the antiferromagnetic region of the phase diagram, T_N which depends on J_2 only in the mean field approximation, is pressure independent. The increase of J_1 with applied pressure (Fig. 2 right, full lines), much stronger than with chemical pressure (dotted line), is explained by the change in the band structure (insert). J_1 is calculated with the model of indirect exchange, assuming a contribution of the anion to the gap between the 4f electron level and the conduction band. This gap is more reduced under pressure than by decreasing the anion size, where the reduction of interatomic distances is partly compensated by a decrease in the crystal field.

3. Magnetic collapse in solid oxygen under pressure

Solid oxygen is the only elementary molecular magnet. It is an antiferromagnetic insulator at ambient pressure, the ground state of the O_2 molecule being a triplet of spin $S = 1$. It transforms into a metallic superconductor at the very high pressure of 96 GPa [10]. The superconducting state is nonmagnetic, as expected from BCS superconductivity (the local magnetic fields break the Cooper pairs).

The question is "when does magnetism disappear?" The pressure–temperature phase diagram (insert Fig. 3) shows that several phases (noted α to ϵ) are stabilized before the superconducting ξ phase. The monoclinic ϵ phase, sandwiched between the antiferromagnetic orthorhombic δ phase and the ξ phase is the most intriguing. Its magnetic state remained controversial for a long time. First principle calculations based on the density functional theory (DFT) initially suggested [11] that the magnetic collapse occurs at the ϵ–ξ transition, or somewhere inside the ϵ phase, but more recent DFT models [12] proposed a nonmagnetic insulating ground state for the ϵ phase. Experimentally, the information given by optical probes about the molecular and magnetic state of ϵ-O_2 was not conclusive.

The answer came from high pressure neutron diffraction [13]. Such experiments are quite difficult due to the weak magnetic scattering of oxygen. Powder neutron diffraction patterns were collected up to 9.5 GPa between 1.5 K and 300 K, reaching the range of stability of the ϵ phase. The magnetic signal was separated from the structural one by measuring a pattern in the paramagnetic region. The magnetic patterns (Fig. 3 left) measured at low temperature (1.5–4 K) show the antiferromagnetic peaks of the α and δ phases up to 8 GPa. Above 8 GPa, the sample transforms into the ϵ phase and this structural change is accompanied by a complete suppression of the magnetic Bragg peaks. This clearly shows that there is no long range magnetic order in the ϵ phase. The magnetic order is recovered by releasing the pressure to 6.7 GPa, where the δ phase is stable.
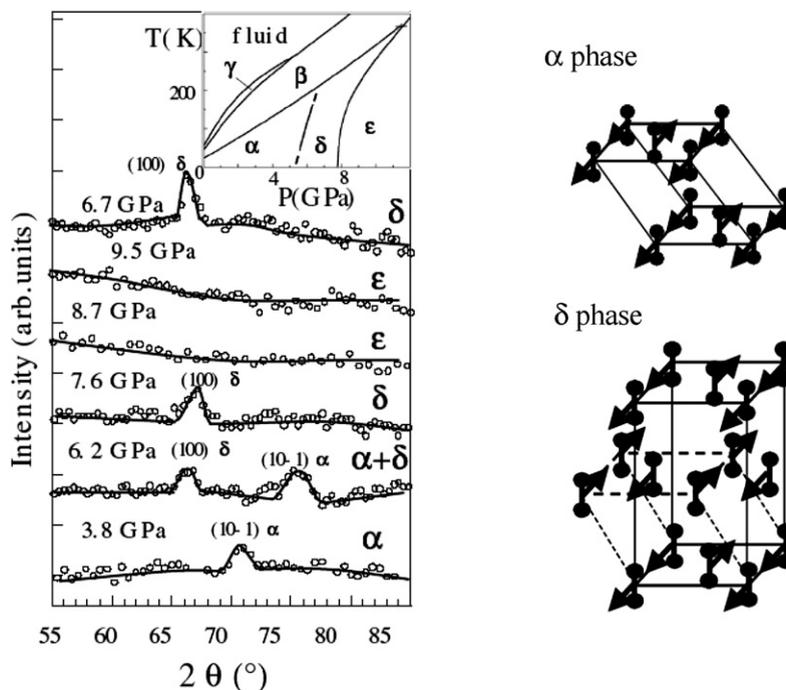
Fig. 3. Solid O_2. Left: magnetic neutron diffraction patterns measured at the lowest temperatures 1.5–4 K and several pressures. In order to separate magnetic and structural contributions, spectra measured in the paramagnetic range were subtracted. Spectra were measured with increasing pressure up to 9.5 GPa, then with releasing it to 6.7 GPa. The neutron powder measurements were performed on the diffractometer G61-Micro of the LLB. Inset: the P–T structural phase diagram of solid oxygen derived from X-ray data. Right, the magnetic structures of α and δ oxygen. Magnetic moments are shown by arrows, perpendicular to the O_2 molecules, as schematized by dumbbells.

Another interesting feature of these experiments is the observation of a new antiferromagnetic structure in the δ phase [14], different from that in the α phase (Fig. 3 right), and the discovery of a nonmagnetic region in the δ phase at temperatures above the range of stability of the ϵ phase. These observations provided a self consistent picture of the magnetism of oxygen under pressure. The exact nature of the magnetic collapse: full suppression of the magnetic moments or persistence of short range (static or dynamic) correlations, is still an open question.

4. Unusual succession of magnetic phases in Laves phases

The Laves compounds $R\mathrm{Mn}_2$ (R = rare earth) of cubic structure, show very rich magnetic phase diagrams due to the combination of several features. The pyrochlore-like Mn lattice, made of corner sharing tetrahedra, is geometrically frustrated for antiferromagnetic first neighbor interactions between isotropic moments, whereas the diamond-like lattice of the rare earth is not frustrated. The interplay of R and Mn magnetism leads to complex magnetic structures with magnetic and nonmagnetic Mn sites [15–17]. The Mn magnetism is unstable, depending on the interatomic distance between first neighbor Mn pairs, with a threshold of instability at a critical distance of 2.67 Å. When Mn moments start to be unstable, R–Mn interactions can induce local Mn moments on some Mn sites, releasing the frustration at the same time. By using chemical and applied pressure, one can cross the instability threshold for Mn magnetism and vary the relative contributions of R and Mn magnetism to the energy balance. This stabilizes successively several magnetic phases under pressure.

The $\mathrm{Ho}(\mathrm{Mn}_{0.9}\mathrm{Al}_{0.1})_2$ compound shows such a succession of magnetic phases. In HoMn_2 the distance between first neighbour Mn atoms is situated just below the critical distance, and Mn moments are induced on some sites by the Ho ones, so that magnetic and nonmagnetic Mn planes alternate, in a canted antiferromagnetic structure (Fig. 4 right), as also observed for DyMn_2 [17,18]. Al substitution of some Mn sites expands the lattice and stabilizes localized and frustrated Mn moments [19]. In $\mathrm{Ho}(\mathrm{Mn}_{0.9}\mathrm{Al}_{0.1})_2$ at ambient pressure, the Mn–Mn distance of 2.67(1) Å is just in the range of the instability threshold. Short range antiferromagnetic correlations coexist with incommensurate and ferromagnetic order (Fig. 4, top left).
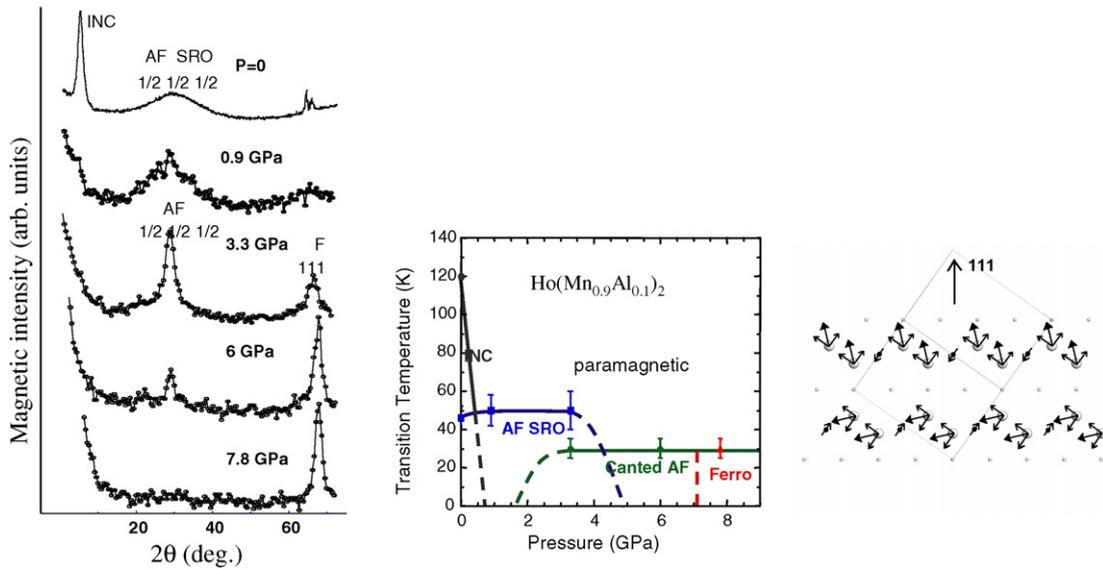
Fig. 4. $Ho(Mn_{0.9}Al_{0.1})_2$. Left: magnetic neutron diffraction patterns at 1.5 K. Spectra in the paramagnetic phase have been subtracted. At ambient pressure, a short range antiferromagnetic (AF SRO) and a magnetic incommensurate (INC) phase coexist. With increasing pressure, these phases disappear and a long range canted antiferromagnetic phase (Canted AF), then a ferromagnetic (F) phase are successively stabilized. Middle: the magnetic phase diagram under pressure. Right: the canted structure of $DyMn_2$ from [18], showing magnetic and nonmagnetic Mn sites. Dy and Mn atoms are shown by large and small spheres respectively. Magnetic moments are symbolized by arrows. For the Dy atoms, the ferro and antiferromagnetic components of the magnetic moments are also shown (thin arrows). The neutron powder measurements were performed on the diffractometer G61-Micro of the LLB.

Magnetic order drastically changes under pressure [20], which brings the sample well below the instability threshold (Fig. 4). A low pressure ($P < 0.9$ GPa) suppresses the incommensurate satellite seen at ambient pressure. The broad peak due to short range correlations disappears more gradually, above 4 GPa. Instead, Bragg peaks start to grow both at (1/2 1/2 1/2) and (1 1 1) peak positions, showing the onset of long range order, which corresponds to a canted antiferromagnetic structure similar to that in $HoMn_2$ and $DyMn_2$. When pressure increases further, the (1/2 1/2 1/2) peak connected with the canted structure disappears. Finally at 7.8 GPa, the single (1 1 1) peak shows a simple collinear ferromagnetic order, which occurs between Ho moments when the Mn ones collapse. The magnetic phase diagram (Fig. 4 middle) shows the variation of the transition temperatures with pressure. The overall decrease of the transition temperatures reflects a change from dominant Mn magnetism to R magnetism, the interactions between 4f electrons being much weaker than between 3d ones.

This experiment shows that at low Al content, the main effect of Al substitution is the lattice expansion, which can be reversed by applied pressure. The succession of magnetic phases under pressure results from a change in the energy balance of the spin interactions, from dominant Mn–Mn interactions, frustrated by the geometry, to competing R–Mn and R–R ones, and finally to R–R interactions only. These changes are associated with changes in the nature of the Mn magnetism, from a localized to an induced then to a nonmagnetic state.

5. Pressure induced crystallization of a spin liquid

Geometrically frustrated pyrochlores $R_2T_2O_7$, where R^{3+} is a rare earth ion and T^{4+} a transition metal or sp ion, are chemically ordered, but their inability to satisfy all magnetic pairwise interactions simultaneously prevents them from ordering magnetically on a large scale, as for standard Curie or Néel orders. They show exotic types of short range magnetic orders, which have been called spin liquids, spin ices or spin glasses, due to their analogies with the usual states of matter [21]. Among them $Tb_2Ti_2O_7$ has been one of the most studied, showing a spin liquid state, namely short range correlations between fluctuating Tb^{3+} magnetic moments [22] down to the lowest measured temperature of 50 mK. The reason why $Tb_2Ti_2O_7$ does not order, in spite of strong antiferromagnetic interactions, has remained a mystery for a long time and was recently attributed to the influence of quantum fluctuations [23]. Surprisingly this spin liquid "crystallizes", i.e. undergoes long range antiferromagnetic order, under pressure [24].
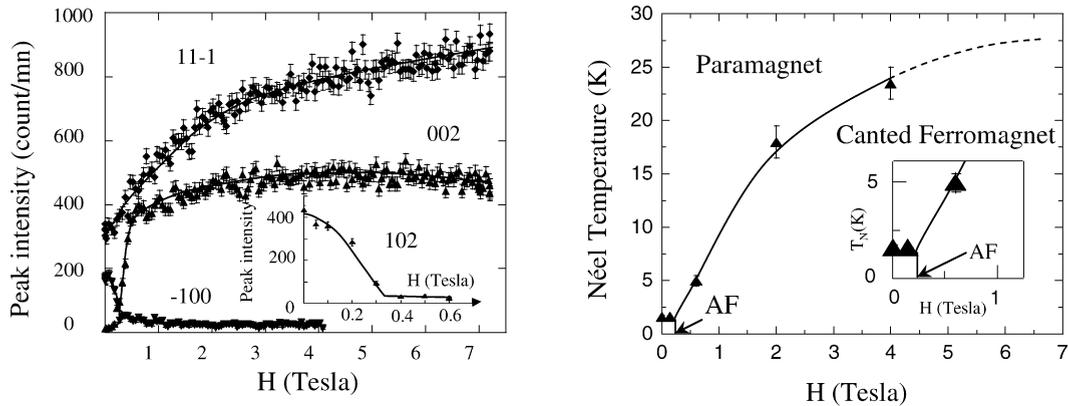
Fig. 5. $Tb_2Ti_2O_7$. Left: variation of the intensity of the magnetic Bragg peaks at 0.12 K with the applied field H in the pressure-induced ordered state (isotropic pressure of 2.4 GPa combined with a uniaxial pressure of 0.3 GPa along the [011] axis). The decrease of the $(-1\,0\,0)$ peak and the increase of the $(1\,1\,-1)$ and $(0\,0\,2)$ peaks show the change from antiferromagnetic to canted ferromagnetic order. Right: the pressure- field magnetic phase diagram. From ref. [25]. The neutron single crystal measurements were performed on the spectrometer 6T2 of the LLB.

$Tb_2Ti_2O_7$ spin liquid therefore behaves somewhat like Helium, the well-known quantum liquid which remains liquid at ambient pressure down to $T = 0$ due to the zero point motion of He atoms, but solidifies under pressure thanks to the enhancement of He–He pair interactions. In $Tb_2Ti_2O_7$, the reason for the onset of the pressure-induced magnetic order was clarified by single crystal measurements [25]. They showed that a uniaxial pressure (or stress) was necessary, breaking the symmetry of the competing magnetic interactions, and tuning the compound in an ordered state. The combination of three extreme conditions of high pressure, low temperature and high magnetic field also showed that the pressure-induced antiferromagnetic order could be transformed in a canted ferromagnetic one with applying a magnetic field (Fig. 5). Spin liquid fluctuations still persist in the ordered states.

6. Conclusions

The above examples provide a short insight into the numerous ways offered by neutron experiments under pressure to study new magnetic states, induced in extreme conditions. Other examples can be found for instance in helical magnets, heavy fermions with quantum critical points, unconventional superconductors, intermediate valence compounds, materials of geological interest, or magnetic dimer systems. The huge field of investigations, which is not limited to magnetism, has given rise to devoted instruments. An ambitious project is going in the steady state reactor Orphée, with the aim at reaching the megabar pressure range. A specialized instrument (PEARL) also exists in the pulsed neutron source ISIS, and some instruments are foreseen on the future European spallation source ESS, using time of flight geometries. Besides neutron diffraction, inelastic neutron scattering experiments are now widely performed in the medium pressure range (around 1 GPa), and a few also in the 10 GPa range at ambient temperature. Finally, the combination of neutron diffraction with other probes under pressure [26] such as synchrotron X-ray diffraction, EXAFS, magnetization, electrical resistivity, optical or infrared absorption, Mössbauer spectroscopy, and even muon spin rotation for medium pressures, is now widely used to reach a complete understanding of the crystal and magnetic states induced by pressure in condensed matter.

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