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A high magnetic field and very low temperature cryomagnet for neutron scattering experiments

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Résumé. — Nous décrivons un système cryomagnétique qui comprend principalement un cryostat permettant d'obtenir des températures variables et un aimant supraconducteur. Le cryostat permet d'utiliser l'aimant supraconducteur soit à 4,2 K soit à 2,17 K et d'obtenir des températures sur l'échantillon comprises entre 1,5 K et 300 K. L'aimant est réalisé par deux bobines en position d'Helmotz qui fournissent un champ vertical de 8,7 T à 4,2 K et de 10 T à 2,17 K. Un anticryostat équipé d'un réfrigérateur à dilution ³He-⁴He peut être installé qui permet d'atteindre 50 mK. Quelques résultats significatifs sont rapportés concernant UAs, CeSb, CeB₆ et TMMC.

Abstract. — A cryomagnetic system is described in which the main components are a cryostat providing variable temperatures and a superconducting magnet. The cryostat enables the magnet to operate either at 4.2 K or 2.17 K and the sample temperature to be varied from 1.5 K to 300 K. The magnet is a split coil providing a vertical magnetic field of 8.7 T at 4.2 K and 10 T at 2.17 K. A ³He-⁴He dilution refrigerator insert can be used to reach T = 50 mK. A few significative experimental results are presented concerning UAs, CeSb, CeB₆ and TMMC.

The goal was to build a cryomagnetic system which supplies a temperature from 1.5 K to 300 K and a magnetic field along a vertical axis with the highest possible value for elastic and inelastic neutron scattering experiments. The sample diameter was limited to 13 mm and the gap between the split coils is only 15 mm. The neutron beam access must be as large as possible in the horizontal plane and $+10^{\circ}$, -5° in the vertical plane. With such a geometry a magnetic field H = 10 T can be reached by using Nb₃Sn multifilamentary wires which accept a field value up to 14 T-15 T. The investment cost and the weight of the cryomagnetic system have been reduced by cooling the magnet with a superfluid helium bath at T = 2.17 K. In order to get temperatures lower than 1.5 K a dilution refrigerator insert has been built which allows to reach T = 50 mK.

A schematic view of the cryomagnetic system is given in figure 1, only the main characteristics will be reported in this paper because they have been described in more details in reference [1] excepted the very low temperature system.

1. Superconducting magnet.

The superconducting magnet, manufactured by Thor Cryogenics, is a split pair with the magnetic axis vertical. The magnet is wound from filamentary Nb₃Sn and filamentary NbTi superconductors. The windings are impregnated with epoxy resin to provide enhanced mechanical stability. The photograph (Fig. 2) shows the general aspect of the magnet. Due to the 2.17 K operation, there are only three Nb₃Sn sections in the magnet, two of which are in the top half. The remaining sections are in NbTi : three in the upper part and two in the lower part of the magnet. The forces between the two halves of the magnet (25 tons) are supported by four concentric aluminium cylinders, with a total thickness of 12 mm, which provide a horizontal access of 340° for the neutron beam. The technical specifications of the superconducting magnet are :

- Central field intensity at T = 4.2 K
- Central field intensity at T = 2.17 K
- Central field homogeneity
- Operating current at 10 T
- Energisation rate
- Inductance
- Clear bore
- Neutron access in vertical plane
- Neutron access in horizontal plane
- Approximate dimensions



Fig. 1. — Schematic view of the 10 tesla cryomagnetic system.

2. Cryostat.

The cryostat contains liquid nitrogen and helium reservoirs with the superconducting magnet at the bottom, the magnet discharge resistances being placed at the top in the helium gas.

The initial cooling of the magnet (60 kg) is performed in two stages : from 300 K to 90 K by a forced circulation of helium gas cooled at 80 K in the liquid nitrogen bath and from 90 K to 4.2 K by supplying liquid helium.

To reach the maximum field of 10 T a refrigerating system, based on a Joule Thomson expansion, allowed

8.7 T 10 T 1 % over 10 mm diameter and 10 mm height 96.5 A \simeq 120 mn to 10 T 28 H 32 mm + 10° - 5° 3 × 113° 230 mm long, 320 mm diameter



Fig. 2. — General view of the magnet.

us to cool down the lower part of the helium bath down to 2.17 K. So the magnet is immersed in superfluid helium and above the normal helium is stratified in temperature, then the surface is at T = 4.2 K and refilling with liquid helium is possible without any perturbation on the superfluid bath.

The liquid helium consumption rate is 0.7 l/h at T = 4.2 K and 0.9 l/h at T = 2.17 K.

3. Variable temperature system.

To get a variable temperature from 1.5 K to 300 K at the sample position an anticryostat isolated from

the liquid helium bath has been built. It contains a small liquid helium reservoir (1.2 l) which is filled from the main bath. By pumping, its temperature can be lowered down to T = 1.2 K for cooling an heat exchanger. A helium gas flow, at reduced pressure and thermalized in this heat exchanger, cools another heat exchanger connected with the sample chamber. Then the sample temperature can be changed from 1.5 K to 10 K. Above T = 10 K the anticryostat is empty and the stainless steel tube, heated at the level of the sample holder, is connected to a cold point only above the liquid helium level of the main bath. Temperature up to T = 300 K can be reached with an additional liquid helium consumption rate of 0.5 l/h.

Over the complete temperature range, the temperature stability $(\Delta T/T)$ is better than 10^{-3} .

4. Very low temperature system.

The variable temperature anticryostat can be removed and replaced by a very low temperature system which is schematized in figure 3. The cooling source is



Fig. 3. — Schematic view of the 3 He- 4 He dilution refrigerator system.

produced by a ³He-⁴He dilution refrigerator system [2] which has a power of 2.5 μ W at T = 50 mK. The rigid connexion between the mixing chamber and the sample is insured by a Al₂O₃ single crystal. The mixing chamber is set up properly with three shrouds. The thermal contact is realized by superfluid helium provided from the T = 1.5 K reservoir which is continuously supplied in liquid helium from the main bath. The calorimeter contains two indium rings and two thermal shieldings at T = 0.8 K and T =1.5 K. The different parts of this very low temperature system can be seen in the photograph given in figure 4.



Fig. 4. — General view showing the different parts of the 3 He- 4 He dilution refrigerator system.

The lowest achieved temperature with this system is T = 50 mK in zero field and T = 70 mK at H = 10 tesla.

5. Typical experimental results.

This cryomagnetic system was successfully used both at the Siloe reactor of the C.E.N. Grenoble and at ILL. A few significative results are reviewed shortly; they concerned both the program on the magnetic properties of cerium and actinide compounds and the program on non linear excitations in low dimensional magnetic systems.

UAs.

Among the uranium monopnictides (NaCl structure), UAs exhibits the most unusual magnetic phase diagram [3]. The phase diagram given in figure 5 has been determined by neutron experiments at the Siloe reactor up to H = 10 T.

A first order magnetic transition occurs at $T_{\rm N} = 124$ K, the ordering corresponds to a collinear (singlek) structure of type I ($\mathbf{k} = [001]; + - + -$) with



Fig. 5. -(H, T) magnetic phase diagram of UAs.

moments along a [001] axis. A magnetic field induces a ferrimagnetic collinear structure with a (++-) stacking sequence of (001) ferromagnetic planes.

At $T_0 = 62$ K a first order transition takes place, the ordering becomes a double-k structure of type IA $(\mathbf{k} = [00\frac{1}{2}]; + + - -)$ with magnetic moments along [110] axes within a (001) plane. Then the magnetic field flips only the moment component parallel to the field giving rise to successive ferrimagnetic sequences while the perpendicular component retains the antiferromagnetic coupling.



Fig. 6. -(H, T) magnetic phase diagram of CeSb.

CeSb.

Neutron experiments performed at the Siloe reactor up to H = 10 T allow us to establish the magnetic phase diagram of CeSb [4, 5] given in figure 6. In spite of a simple NaCl structure CeSb exhibits one of the most unusual magnetic phase diagram, three regions can be distinguished :

(i) A low temperature region where the magnetic

ordering consists of an antiferro (AF), ferri (AFF) or ferromagnetic (F) stacking of fully ordered ferromagnetic [001] planes.

(ii) At high temperature and low field, the magnetic structures, antiferromagnetic in nature, contain non-magnetized [001] planes periodically distributed (AFP).

(iii) At high temperature and high field, the FPphases consist of a periodic stacking of non-magnetized layers (entropy ~ k Ln 2) and ferromagnetic layers ($m \sim 2 \mu_B$).

$\underline{\text{CeB}_6}$.

The magnetic phase diagram of CeB_6 has been investigated at the Siloe reactor up to H = 10 T [6]. The results are reported in figure 7. This work is done in collaboration with the Kasuya's group in Sendai (Japan).

In the simple cubic structure CeB_6 is a monovalent metal.

In phase I, the resistivity exhibits a typical dense Kondo behaviour.

In phase II, a strong magnetic field induces an anti-



Fig. 7. -(H, T) magnetic phase diagram of CeB₆.

ferromagnetic component associated with a wave vector $\mathbf{k} = \begin{bmatrix} \frac{111}{222} \end{bmatrix}$ indicating the existence of an antiferro quadrupolar ordering ($T_Q = 3.2$ K at H = 0).

Below $T_{\rm N} = 2.35$ K, CeB₆ orders with a double-k structure commensurate with the lattice (k = $\left[\frac{111}{442}\right]$). The moment is only about 0.3 $\mu_{\rm B}$, a strongly reduced value for a Γ_8 ground state quartet.

The surprising result is the large increase of T_Q when a magnetic field is applied.

TMMC.

The experimental studies of the linear (magnons) and non-linear (solitons) excitations in the one dimensional antiferromagnet TMMC in high field up to 10 tesla and low temperature (T < 6 K) have been performed at ILL using the three-axis spectrometer IN12 installed on a cold neutron beam [7, 8]. This research program results from a collaboration with J. P. Boucher.

In a 1D-antiferromagnet, like TMMC, the dipolar interaction gives rise to an easy plane anisotropy (XY); a magnetic field applied within this plane, i.e. perpendicular to the chain axis, leads to a system described by a sine-Gordon Hamiltonian which possesses linear and non-linear excitations. The thermodynamic of the non interactive soliton gas predicts the existence of a quasi-elastic peak with a width in q and ω depending exponentially on H/T:



Fig. 8. — Central peak in TMMC : typical energy scans performed at q = 0 and T = 2.5 K as a function of H.

and

 $\Gamma_{\omega} \sim H \exp(-\alpha H/T)$

Such a dependence is effectively observed in TMMC in medium field (H < 50 kOe) with $\alpha = 0.26$ K/kOe (Fig. 8). In higher field (H > 80 kOe) a cross over from XY to YZ solitons is observed, resulting from a competition between the applied field (H //Oy) and the anisotropy field ($\hat{H}_D \sim 80$ kOe).

The effects of the impurities give rise to a diffusive motion of solitons instead of a coherent motion. This behaviour explains very well the narrowing of the ω -width observed for all fields (Fig. 9).



Fig. 9. — Effect of impurities on the central peak in TMMC.

Finally, the study of the magnons in high field up to 10 tesla and low temperature (T = 1.4 K) (Fig. 10 and Fig. 11) gives evidence for strongly non linear effects which arise from the mixing of the spin components due to the spin canting.

6. Conclusion.

In conclusion, this cryomagnetic assembly has been proved to work successfully in a large temperature



Fig. 10. — Energy scan at q = 0 in TMMC showing the magnetic excitations in high magnetic field (10 T) and low temperature (T = 1.44 K).

range from 50 mK up to 300 K and to provide a magnetic field as large as 10 tesla. This high field value has been achieved with a moderated investment cost due to the installation of a refrigerating circuit allowing to operate the magnet at T = 2.17 K. A large number of neutron experiments have been performed with this cryomagnetic system, in particular to determine magnetic phases diagram of cerium or actinide com-



Fig. 11. — Magnetic field dependence of magnetic excitation energies at q = 0.

pounds or to study the dynamics of low dimensional magnetic systems.

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